Addendum 03 – QAPP

Smoky Canyon Mine
Remedial Investigation/Feasibility Study
Revised Section 2.0, Sampling and Analysis Plan

Quality Assurance Project Plan (QAPP)

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APPROVAL PAGE

QUALITY ASSURANCE PROJECT PLAN FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

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SMOKY CANYON MINE
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

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Quality Assurance Project Plan (QAPP) Attachments

1 SVL Analytical Supporting Documents

2 Columbia Analytical Services Supporting Documents

3 Smoky Canyon RI/FS Preliminary Data Usability Screening Summary Table Template and Data Usability Checklist

LIST OF ACRONYMS

ASTM	American Society for Testing and Materials
BLM	Bureau of Land Management
COC	Chain of Custody
COPCs	Chemicals of Potential Concern
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
COC	Chain of Custody
CVAA	Cold Vapor Atomic Absorption
DQOs	Data Quality Objectives
FSP	Field Sampling Plan
HASP	Health and Safety Plan
ICB	Initial Calibration Blank
ICPMS	Inductively Coupled Plasma-Mass Spectrometer
ICV	Initial Calibration Verification
IDEQ	Idaho Department of Environmental Quality
LCS	Laboratory Control Sample
LLC	Limited Liability Company
MDC	minimum detectable concentration
MDL	Method Detection Limit
mg/L	milligrams per liter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NIST	National Institute of Standards and Technology
NTU	nephelometric turbidity unit
ORP	Oxygen Reduction Potential
PARCC	precision, accuracy, representativeness, comparability and completeness
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RI/FS	Remedial Investigation/Feasibility Study
RL	Reporting Limit

RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedures
SVL	SVL Analytical
USEPA	United States Environmental Protection Agency
USEPA CLP	USEPA Contract Laboratory Program
USFS	United States Department of Agriculture Forest Service
USFWS	United States Fish and Wildlife Service

2.0 QUALITY ASSURANCE PROJECT PLAN

2.1 Introduction

The purpose of this Quality Assurance Project Plan (QAPP) is to describe the quality assurance and quality control (QA/QC) policies and procedures used with data collection and evaluation conducted in support of the Smoky Canyon Remedial Investigation/Feasibility Study (RI/FS). This QAPP is one part of the three-part Sampling and Analysis Plan (SAP), prepared by Formation Environmental, LLC (Formation) for the J.R. Simplot Company (Simplot) in support of the RI/FS. The SAP is comprised of this QAPP (Section 2.0), a Field Sampling Plan (FSP; Section 3.0), and a Health and Safety Plan (HASP; Section 4.0).

The QAPP describes the measures that shall be employed during the Smoky Canyon RI/FS to assure that data generated are of a known and defensible quality in relation to the overall objectives of the investigation. The QAPP was prepared in accordance with United States Environmental Protection Agency (USEPA) guidance on Quality Assurance Project Plans (USEPA, 2002; EPA QA/G-5), and is comprised of four basic project plan elements:

- project management;
- data generation and data acquisition;
- data validation and usability; and
- assessment and oversight.

The four subsections that follow provide the four USEPA project plan elements (USEPA, 2002), and each presents the topics applicable to that element with appropriate Site-specific content, as needed for planning the Smoky Canyon RI/FS.

2.2 Project Management

This section addresses project administrative functions and project concerns, goals, and approaches to be followed during implementation of the Smoky Canyon RI/FS.

2.2.1 Problem Definition/Background

A 2009 Settlement Agreement and Administrative Order on Consent (Settlement Agreement/CO) entered into by Simplot, the U.S. Department of Agriculture, Forest Service (USFS), USEPA, and Idaho Department of Environmental Quality (IDEQ) established an RI/FS process for this Site, with BLM, U.S. Fish and Wildlife Service (USFWS), and Shoshone-Bannock Tribes participating as Support Agencies. The Settlement Agreement/CO also defines the Site, presents a summary of the Site history, identifies a general project schedule, and provides a preliminary list of chemicals of potential concern (COPCs) for each media type. The

information provided by that agreement and the extensive previous investigation work performed for this Site allows for a well defined statement of the problem to be addressed by this RI/FS, which is presented in the Data Quality Objectives section of the RI/FS Work Plan (Formation, 2011; refer to Section 6.0).

The overarching goals of the RI/FS are to:

- Determine the nature and extent of contamination and any risks to human health or the environment caused by the release or threatened release of contaminants at or from the Site; and

- Determine and evaluate alternatives for remedial action to respond to or remedy any unacceptable risks.

Ultimately, the RI/FS process will provide the basis for the USFS to select a final remedy for the Site.

2.2.2 Project Description

The RI/FS Work Plan (Formation, 2011) presents the rationale and scope of data collection activities planned to achieve the goals listed above. The data collection activities detailed in the Work Plan are intended to augment existing data and/or fill gaps identified in the historical data that will be used in the remedy selection.

The data collection activities associated with the RI/FS are described in detail in the Field Sampling Plan (Section 3) and include the following tasks:

1. Installation of one new deep monitoring well in the Wells Formation aquifer;

2. Collection of groundwater samples from monitoring wells and water supply wells;

3. Collection of surface water samples from streams, springs, seeps and ponds;

4. Measurement of groundwater levels and stream flows;

5. Collection of sediment samples from streams and ponds;

6. Collection of soil and overburden samples;

7. Collection of vegetation tissue samples and description of terrestrial vegetation communities;

8. Collection of other terrestrial and aquatic tissue samples (riparian vegetation, terrestrial insects/small mammals, benthic macroinvertebrates, fish) in support of the Baseline Human Health and Ecological Risk Assessment; and

9. Analysis of the various sample types for the parameters identified in the FSP, including the COPCs listed in Attachment B to the Settlement Agreement/CO (Table 1-2).

Data collected through these field investigations will be used to characterize the nature and extent of COPC distribution from source areas at the Site to the surrounding environmental media. These data will also be used to describe the fate and transport of COPCs and identify exposure routes for human and ecological receptors. Baseline risk assessment will be conducted to evaluate the existing risks to human health and to local ecological receptor communities, including risks from exposures that may occur in the future if no action is taken at the Site (although the baseline ecological risk assessment will focus on local receptor communities, migratory populations and individual threatened and endangered species, if present, will also be evaluated). The results from this investigation will be used to make recommendations for remedial actions that may be needed to protect human health or the environment.

Any appropriate and required scope modifications or additions may be accommodated by revisions to the SAP documents followed by receipt of written approval from the USFS Remedial Project Manager.

2.2.3 Project Organization

The RI/FS is being conducted by Simplot in accordance with the 2009 Settlement Agreement/CO entered into with the USFS, USEPA, and IDEQ. The USFS is the Lead Agency for the RI/FS.

Simplot is responsible for implementation of the RI/FS in accordance with the Settlement Agreement/CO. Simplot responsibilities include preparation of project planning documents; collection of data needed to complete the RI/FS and baseline risk assessments; routine data reporting to the USFS and supporting agencies; data analysis and interpretation as needed to complete Site characterization, risk assessment, and development and evaluation of remedial alternatives; and generation of draft and final RI Reports and FS Reports.

Simplot's project team for the Smoky Canyon Mine RI/FS currently includes:

- Formation Environmental LLC (Formation), Boulder, CO (environmental services contractor);
- SVL Analytical, Kellogg, ID (chemical analysis laboratory);
- Columbia Analytical Services (CAS), Kelso, WA (chemical analysis laboratory); and

- TLI Solutions, Inc., Golden, CO (data validation contractor).

Simplot may also contract with other consultants and contractors as needed to complete the RI/FS, subject to USFS approval of qualifications. Simplot's selection of contractors to support the RI/FS will be subject to the review and approval of each contractor's qualifications by the Lead Agency.

An organizational chart showing the overall organization of the Simplot project team is provided as Figure 1-1. Simplot's **RI/FS Program Manager** is Alan Prouty. The **Program Manager** oversees scheduling and management of all technical and non-technical aspects of the project (e.g., field activities, data collection, data analysis, report preparation, scheduling, costing) and serves as primary point of contact with agency representatives. The primary responsibilities of key personnel on the project team are described as follows:

Formation RI/FS Project Representative (Steve Werner, Formation)

Oversees scheduling and management of all technical and non-technical aspects of the project (e.g., field activities, data collection, data analysis, report preparation, scheduling, costing). Reports to the Simplot Program Manager (Alan Prouty) and USFS Remedial Project Manager (Mary Kauffman). Directs Field Investigations Manager and Project QA Manager. Ensures that all field personnel understand the scope of work including QA/QC requirements. Responsible for ensuring that the sampling methods and data analyses reflected in the SAP meet the objectives of the RI/FS Work Plan. Reviews and approves project plans and all project deliverables.

Field Investigations Manager (Fred Charles, Formation)

Plans and supervises sampling and other field activities and obtains necessary permits. Schedules and manages various field tasks (e.g., sample collection, measurements, data collection) and is responsible for sample transport to the laboratory. Makes certain that the QAPP, HASP, and field investigations (via the FSP) are implemented. Responsible to the RI/FS Project Representative for implementation of field sampling activities, QA/QC measures, and health and safety program requirements defined in the SAP. The Field Investigations Manager is also responsible for ensuring that field staff have appropriate, hands-on training.

Project QA Managers (Kathy Tegtmeyer/Laura Clayton, Formation)

Project QA Managers are responsible for coordinating the development and approval of the QAPP and its supporting procedures and for maintaining the current, approved version of the QAPP for use on the project. The QA Manager participates in the review and approval of all project deliverables, assists with establishing laboratory contracts, acts as a day-to-day liaison with the laboratories, directs field and laboratory audit

activities, coordinates any subsequent corrective and preventive actions, if needed, and communicates regularly with the Simplot Program Manager and Formation Project Representative regarding any laboratory or data validation concerns. The QA Manager will also oversee data validation efforts and coordinate the resolution of any necessary corrective actions resulting from data validation activities, including any quality issues that may be resolved during field activities (i.e., resampling to replace unusable samples).

Laboratory Project Manager(s) (SVL and CAS)

Reviews QAPP and ensures laboratory resources are available, reviews final analytical reports produced by the laboratory, coordinates scheduling of laboratory analyses, and supervises in-house chain-of-custody procedures. SVL and CAS have been selected as the analytical laboratories to support this project.

Simplot Mine Manager (Scott Lusty, Simplot Smoky Canyon Mine)

Oversees scheduling and management of all on-Site aspects of the project and provides necessary resources for conduct of on-Site data collection, testing, or construction activities related to the RI/FS.

Field Supervisors (Patrick Lennberg and Jim Jonas, Formation) **and Field Technicians**

The field supervisor(s) and technicians performing sampling and data collection may be either Simplot employees or employees of USFS-approved contractors. All field staff must have hands-on training in the use of the Standard Operating Procedures included in the FSP Attachment as well as other appropriate experience.

2.2.4 Training Requirements

Field personnel shall be trained in the requirements of the SAP in a project meeting prior to the initiation of field activity. All personnel shall read the SAP documents, including this QAPP, prior to the start of field work and shall acknowledge that they have read the documents at the time of the project meeting. In addition, prior to conducting sampling activities, the Field Investigations Manager, or designee, shall review field procedures and sampling requirements in order to better ensure that samples are collected and handled according to FSP and QAPP requirements. One hard copy of the current approved version of the entire SAP shall be maintained for ready-reference purposes in the field vehicle or field office. All field team members shall have access to *.pdf format files of the complete SAP through their personal laptop computers.

2.2.5 Quality Objectives and Criteria for Measurement Data

Measurement performance criteria are established for each field and laboratory measurement parameter. Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for accuracy, precision, representativeness, comparability, and completeness. The definitions of accuracy, precision, representativeness, comparability, and completeness are provided below along with the acceptance criteria for data collected in support of the RI/FS.

2.2.5.1 Purpose of QAPP

The objectives of this QAPP are to assure that the precision and accuracy of program data are known and documented; that sample collection, analysis, and reporting are complete; and that samples are representative of tested environmental media. This plan also provides guidance for documentation of information collected in the field, including field quality control data, maintenance of documented sample custody, and documentation of laboratory analytical procedures and quality control data for data verification and validation.

2.2.5.2 Data Quality Objectives

The data quality objectives (DQOs) for this program are described in the Smoky Canyon Mine RI/FS Work Plan (Formation, 2011). Consistent with USEPA guidelines (USEPA, 2006), the DQOs, describe the systematic planning of data collection activities to assure that the proper type, quality, and quantity of data are collected. The DQOs shall be fulfilled by implementation of these quality assurance and quality control activities during data collection in support of the RI/FS:

- following specific sampling designs (refer to FSP);
- adherence to standardized procedures for field measurements, sampling, sample handling, and sample chain of custody (COC);
- collection and analyses of field and laboratory QC samples, as specified on Table 2-11 (discussed in Section 2.3.4.1) and Table 2-12 (discussed in Section 2.3.4.2);
- analyses of samples in accordance with standard method protocols selected to meet the project's measurement performance goals (Section 2.2.5.3) and detectability requirements (Section 2.3.3.2);
- implementation of laboratory-specific preventative maintenance measures;
- data review and reduction by the laboratories;
- third party data validation; and
- quality auditing and corrective/preventative action processes, as described in the FSP and this QAPP.

2.2.5.3 Measurement Performance Criteria - Definitions

The definitions of precision, accuracy, representativeness, comparability, and completeness (PARCC) are provided below along with the acceptance criteria for data collected in support of the RI/FS. Equations for calculation of precision, accuracy, and completeness are also provided in Table 2-1.

Precision

Precision is the level of agreement among repeated measurements of the same characteristic. There are two general forms of uncertainty. The first is the random error component of the data collection process. The second is inherent stochastic variability, which cannot be eliminated but can be described.

Data precision is assessed by determining the agreement between replicate measurements of the same sample and/or measurements of duplicate samples. The overall random error component of precision is a function of the sampling and analytical precision and is assessed by the analysis of field duplicates. The analytical precision is determined by the analysis of field duplicates by laboratories and by replicate analyses of the same sample. An analytical duplicate is the preferred measure of analytical method precision. When analytes are present in samples at concentrations below or near the quantitation limit, precision may be evaluated using duplicate analyses of laboratory prepared samples such as duplicate laboratory control samples (LCS/LSCD) and duplicate laboratory matrix spike samples (MS/MSD).

Precision can be measured as relative percent difference (RPD) or as relative standard deviation (RSD; also known as a coefficient of variation). Formulae for both are presented in Table 2-1.

Accuracy

Accuracy is the degree of difference between the measured or calculated value and the true value. It is a measure of the bias or systematic error of the entire data collection process. Potential sources of systematic errors include:

- sample collection methods;
- physical or chemical instability of the samples;
- interference effects during sample analysis;
- calibration of the measurement system; and,
- contamination.

Data accuracy or analytical bias may be evaluated by the analysis of laboratory control samples (LCS) and/or matrix spike (MS) samples, with results expressed as a percentage recovery

measured relative to the true (known) concentration (refer to Table 2-1 for percent recovery calculations).

Field equipment and ambient blanks and laboratory blanks may be analyzed to assess artifacts introduced during sampling, transport, and/or analysis that may affect the accuracy of the data. In addition, initial and continuing calibration verification samples (ICV and CCVs) and initial and continuing calibration blanks (ICB and CCB) may be used to verify that the sample concentrations are accurately measured by the analytical instrument throughout the analytical run.

Representativeness

Data representativeness is defined as the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or environmental conditions. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. Representativeness of samples shall be achieved through the careful selection of sampling locations and methods. The sampling program described in the FSP has been designed to provide samples that are representative of the medium being sampled as well as a sufficient number of samples to meet the project DQOs.

Comparability

Data comparability is defined as the measure of the confidence with which one data set can be compared to another. Comparability is a qualitative parameter but must be considered in the design of the sampling plan and selection of analytical methods, quality control protocols, and data reporting requirements.

Completeness

Completeness refers to the amount of useable data produced during a sampling and analysis program. The procedures established in this QAPP are designed to ensure, to the extent possible, that data shall be valid and usable. To achieve this objective, every effort shall be made to collect each required sample and to avoid sample loss.

2.2.5.4 Measurement Performance Goals

This section identifies numerical goals for precision, accuracy, and completeness for the various environmental media. Failure to meet these goals shall be considered in the data validation process described in Section 2.4.

Precision

Precision shall be determined on field data and laboratory analysis data by the analysis of field duplicates, laboratory replicates, matrix spike and matrix spike duplicate results and evaluation of the RPD for these various paired measurements. The RPD goals for measures of precision associated with the COPC analysis methods are presented in Tables 2-2 through 2-5.

Accuracy

Sampling accuracy shall be determined by the collection and analysis of field ambient blanks and equipment blanks, at the frequencies described in Section 2.3.4.

Laboratory accuracy is determined by the analysis of calibration and method blanks, calibration verification samples, laboratory control samples or standard reference materials, and matrix spike samples. Method blank goals shall be that blanks contain less than the reporting limit for each target parameter. Accuracy goals for the specific laboratory analysis methods that will be relied on to generate data for the RI COPCs are summarized in Tables 2-2 through 2-5.

Representativeness

Representativeness is addressed by the description of the sampling techniques and the rationale used to select the sampling locations. Sampling methods are established by the standard operating procedures (SOPs) provided in the FSP Attachment. Sample representativeness is also evaluated using the RPDs for field duplicate results and by a review of the results of field blanks (i.e., ambient blanks for water matrices only and equipment blanks as appropriate to sampling methods).

Representativeness of individual sample analyses will be described on the basis of results obtained from associated laboratory quality control samples. The representativeness of sample analyses will be considered acceptable as long as any detectable concentrations of analytes in associated field and method blanks are less than the reporting limit.

Comparability

Comparability shall be ensured by analyzing samples obtained in accordance with appropriate SOPs and the referenced standard laboratory analysis methods. All data should be calculated and reported in units consistent with standard reporting procedures so that the results of the analyses can be compared with those of other laboratories, if necessary. In general, data shall be reported in milligrams per liter (mg/L) for water matrices and mg/kg (with the moisture basis specified) for solid matrices.

Completeness

The project's completeness goals are 95 percent for groundwater, surface water, sediment and soil analyses and 90 percent for tissue analyses.

2.2.6 Documents and Records

This section describes the management of project documents and records, including this QAPP.

2.2.6.1 Field Logbooks

Documentation of observations in the field provides information on conditions at the time of sampling and a permanent record of field activities. Field observations and data collected during routine monitoring activities will be recorded with waterproof ink in a permanently bound weatherproof field log book with consecutively numbered pages, or on field forms associated with the individual SOPs referenced by the FSP. Field forms for recording various types of sampling and measurement activities include sampling of surface water, groundwater, fish tissue, sediment, soil, and vegetation and collection of groundwater depth-to-water and surface water discharge measurements. The appropriate forms for sampling of various media are located in the applicable SOP (refer to the FSP and FSP Attachment). The Field Documentation SOP (JRS SOP No. 1) also provides instructions for recording field activities at the time of field measurements or sample collection.

Field notebook and data sheet entries will, at a minimum, include the information listed below:

- Project name and number;
- Sample location;
- Data and time of sample collection;
- Sample identification numbers;
- Description of sample (sample matrix or species);
- Number of samples collected;
- Field measurements;
- Field observations and weather conditions;
- Personnel present;
- Sampler's signature; and
- Field filtration activities, if performed.

In addition, other ancillary information shall be recorded, including:

- personnel and/or other visitors to the sampling site(s);

- weather conditions;
- presence of livestock or wild game; and
- any unusual events.

Changes or deletions in the field book or on the data sheets will be recorded with a single strike mark through the changed entry, with the sampler's initials and the date recording the new entry. All entries must remain legible. Sufficient information should be recorded to allow the sampling event to be reconstructed without having to rely on the sampler's memory.

Completed field forms and logbooks will be copied to the project's quality records (refer to Section 2.2.6.4), in addition to copies of outgoing COCs and sample shipping documents.

2.2.6.2 COC Records

Documentation of sample custody must be maintained. Information on the custody, transfer, handling, and shipping of samples shall be recorded by field personnel on a COC form as specified in JRS SOP No. 2 (FSP Attachment), and as described in greater detail in Section 2.3.2 below.

A COC form shall be completed for each set of samples collected daily and shall contain the following information:

- sampler's signature and affiliation;
- program name and identification number;
- date and time of collection;
- sample identification number and matrix;
- analyses requested;
- number of containers;
- signature of persons relinquishing custody, dates, and times;
- signature of persons accepting custody, dates, and times;
- method of shipment; and,
- shipping papers/waybill identification number (as appropriate).

A copy of each as-transmitted COC form shall be retained in the program quality records (refer to Section 2.2.6.4).

2.2.6.3 Analytical Laboratory Records

Results received from the laboratory will be documented both in report form and in an electronic format. Original hard copy deliverables and electronic files received from laboratories will be

maintained with the program quality records, as described below. Section 2.4 presents the project's laboratory reporting requirements in detail. The hard-copy deliverable ("data package" or "report") issued to Simplot and Formation will include data necessary to complete validation of laboratory results in accordance with specifications included in Section 2.4.

2.2.6.4 Program Quality Records

Program quality records are defined as completed, legible documents that furnish objective evidence of the quality of items or services, activities affecting quality, or the completeness and quality of data. These records shall be organized and managed by Formation and shall include, at a minimum:

- copies of all bound field logbooks;
- copies of all field documentation forms;
- field copies and original (laboratory) copies of all COC forms;
- incoming and outgoing program correspondence (letters, telephone conversation records, faxes, and e-mail messages);
- copies of all laboratory agreements and amendments thereto;
- as-received laboratory data packages (hard copy and electronic);
- complete laboratory data validation packages;
- documentation of field and/or laboratory audit findings and any corrective actions;
- draft and final versions of all reports; and,
- draft and final delivered versions of the RI/FS report and supporting procedures such as statistical analyses, numerical models, etc.

The other documentation included in the program's quality records include the approved FSP and QAPP, any approved revisions or addendums to the FSP and QAPP, and SOPs referred to for field data collection with any updates, revisions, or addendums to those SOPs approved by the RI/FS Project Representative and Field Investigations Manager to address specific conditions encountered at the Site during the field investigations.

2.3 Data Generation and Acquisition

The elements in this section address management of data generation and acquisition activities.

2.3.1 Sampling Design

A detailed description of the sampling design for each task, including the schedule for sample frequency, is contained in the FSP (Section 3.0). The FSP also describes in detail the procedures that will be used to collect each sample type planned for the RI/FS. The SOPs

included in the FSP Attachment, and listed in Table 1-3, are more detailed descriptions of those procedures, and they also provide information on field documentation and QA activities for the sampling team.

2.3.1.1 Sampling Locations and Frequencies

Comprehensive RI/FS sampling activities are summarized in Table 2-6, and consist of surface water, sediment, groundwater, soil, vegetation, and other biological tissue sampling at various locations. The number and types of samples that will be collected and sampling locations are detailed in Tables 3-1, 3-4, 3-5 and 3-8 of the FSP and summarized in Table 2-6.

2.3.1.2 Sampling Methods

The Standard Operating Procedures for sample collection of various matrices included in the FSP Attachment are listed in SAP Table 1-3.

2.3.2 Sample Handling, Preservation, and Custody

This section describes sample handling requirements and chain of custody procedures from the sample collection step through laboratory analysis and ultimate disposal.

2.3.2.1 Sample Labeling

Each sample that is collected in the field will be labeled for future identification. Sample labels may be filled out as completely as possible by a member of the sampling team prior to the start of the day's field sampling activities. Samples will be labeled with all necessary information on pre-printed waterproof labels using waterproof ink. An example label is included as Figure 2-1. At a minimum, each sample label shall contain the following information:

- location identification;
- sample identification number (including codes for site location, sample matrix, and sample type, described in further detail below);
- date and time of sample collection;
- analyses required;
- method of preservation, if used;
- sample matrix; and
- sample depth, if applicable.

Each sample shall be assigned a unique sample identification number. These numbers are required for tracking the handling, analysis, and verification or validation status of all samples collected during monitoring. Each sample identification number will identify the sampling

location and type of sample. Sample identification numbers will be assigned using several codes as follows:

Sampling Event - Location - Media and Type - Number

SC0510-LSV2C-FT013

The first field in the identification number identifies the general sampling location and time period. For example, samples collected in May 2010 will all have the prefix “SC0510.”

The second field in the identification number identifies the location of the sample. For example, LSV-2C is a designated sampling location in the Lower Sage Valley. Location identifiers for most locations have already been established and are included in the SAP figures. The location identifiers established by the SAP will be used whenever they are available.

The third field has three parts. The first part is a two- or three-letter acronym that identifies the sample matrix type. The matrix types are defined as:

GW: groundwater

SW: surface water

SD: sediment

SL: soil (or overburden)

VG: terrestrial vegetation tissue

FT: fish tissue

ITT: terrestrial macroinvertebrate/insect tissue

ITA: aquatic benthic macroinvertebrate tissue

MT: small mammal tissue

The second part of the third field is comprised of a single digit describing the intended sample use. These sample use codes and include:

0: primary sample

2: field duplicate sample

3: equipment rinsate or QA/QC blank sample

Note that additional codes may be added as the project proceeds. The additions will be communicated immediately to the field staff and data management team.

The third and final part of the third field is a two-digit number unique to the specific sample. Numbers will begin with 01 and increase consecutively as sampling tasks are implemented.

For example, SC0510-LSV2C-FT013, is a primary fish tissue sample collected from location LSV-2C in the Lower Sage Valley in May 2010 with the sequential number 13 (i.e., the thirteenth fish sample collected at that site).

Samples will be immediately labeled in the field and sample numbers shall be recorded at the time of sampling in field notes and on field data collection forms.

2.3.2.2 Sample Containers, Preservation, and Holding Times

Sample Containers

For aqueous sample matrices (i.e., groundwater, surface water, rinsates, etc.), the laboratory will provide new, certified pre-cleaned, prepared sample containers appropriate to the list of analyses to be requested and as specified on Table 2-7. For the other matrices, samples may be collected in containers supplied by the sampling contractor, in accordance with each sampling task in the FSP and appropriate sample collection SOPs (also refer to Table 2-7).

Sample Preservation and Storage

Samples are preserved in order to prevent or minimize chemical changes that could occur during transit and storage. Sample containers containing appropriate preservative are used to ensure preservation immediately upon sample collection. The contracted laboratories will provide containers and appropriate preservatives (i.e., “pre-preserved” containers), as needed for the analyses to be requested.

Aqueous samples (groundwater, surface water, equipment rinsates) submitted for metals/metalloids analyses, as well as some other analyses, require preservation upon collection, as specified in Table 3-3 of the FSP and Table 2-7. Preservation requirements are associated with the individual analyses to be performed and the referenced analytical methods.

Solid samples (sediment, soil, overburden) typically do not require preservation other than temperature control during storage and transfer to the laboratory. Tissue samples may also be frozen for storage and shipping. Descriptions of sample preservation requirements for solid matrices and biologic tissues are summarized in Table 3-3 (refer to Field Sampling Plan) and Table 2-7.

Sample Holding Times and Analyses

Sample holding times are established to minimize chemical changes in a sample prior to analysis and/or extraction. A holding time is defined as the allowable time between sample collection and analysis recommended to ensure accuracy and representativeness of analysis results, based on the nature of the analyte of interest and chemical stability factors.

Immediately after collection, samples shall be placed in field coolers with wet ice and/or blue ice. If there is no likelihood that a holding time will be violated, samples may be transferred to a locked refrigerator for one or more days of storage prior to shipping to a laboratory. Transfer to the laboratory for analysis should be prompt to minimize the possibility of exceeding holding times. Prompt delivery of biological tissue samples to the laboratory is critical in order to minimize risk of decomposition.

Holding times for the chemical constituents for which samples will be analyzed are summarized in Table 2-7. Failure to conduct analyses within the required holding times may potentially require the qualification of associated analytical results and shall prompt appropriate corrective and preventive action measures as outlined in Section 2.4.4.

2.3.2.3 Sample Handling and Chain of Custody

Sample Handling and Shipping

After collection, sample labels will be completed (refer to Section 2.3.2.1 above), and the samples will be placed on ice in an insulated cooler. The sample bags or jars will be placed in re-closeable freezer-type plastic storage bags. Each sample container will be carefully packaged in a shipping container, typically an ice chest, with Styrofoam® peanuts, vermiculite or other packing material to prevent breakage during shipment. A labeled temperature blank may also be included with each cooler shipped, if temperature-sensitive samples were collected. Ice placed in the cooler will be double-bagged to prevent leakage of water. The coolers will be taped shut.

Chain of Custody

After samples have been collected, they will be maintained under strict chain-of-custody protocols. The field sampling personnel will complete a chain-of-custody (COC) form (refer to JRS SOP No. 2, FSP Attachment) for each shipping container (i.e., cooler, ice chest or other container) of samples to be delivered to the laboratory for analysis. The sampler is responsible for initiating and filling out the COC form. The COC for a shipping container will list only those samples in that shipping container. Information contained on the triplicate, carbonless COC form will include the following:

- Project number;

- Date and time of collection;
- Sample identification number;
- Sample matrix;
- Analyses requested;
- Number of containers/bags for each sample;
- Sample preservation;
- Field filtration, if applicable;
- Sampler's signature and affiliation;
- Signature of persons relinquishing custody, dates, and times;
- Signature of persons accepting custody, dates, and times;
- Method of shipment;
- Shipping air bill number (if the samples are shipped);
- Condition of samples and cooler temperature upon receipt by laboratory; and
- Any additional instructions to the laboratory.

Any documentation, including COCs, placed inside the cooler during sample shipment, should be placed inside a re-closeable plastic bag.

The sampling personnel whose signature appears on the COC is responsible for the custody of the samples from the time of sample collection until custody of the samples is transferred to a designated laboratory, a courier, or to another project employee for the purpose of transporting the sample to the designated laboratory. The sample is considered to be in custody when the sample is: (1) in the direct possession of the sample custodian; (2) in plain view of the sample custodian or (3) is securely locked in a restricted-access area by the sample custodian. Custody is transferred when both parties to the transfer complete the portion of the COC under "Relinquished by" and "Received by." Signatures, printed names, company names, dates and times are required. Upon transfer of custody, the sampling personnel who relinquished the samples will retain the third sheet (pink copy) of the COC. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the COC. Copies, receipts and carbons of Bills of Lading will be retained as part of the permanent documentation in the project file. It is not necessary for courier personnel to sign the COC.

When the analytical laboratory receives the samples, the COC will be immediately signed along with the date and time of receipt. The top sheet (white copy) or a copy of the COC may be returned with the final analytical report. The laboratory will follow appropriate chain-of-custody procedures when shipping any samples to a subcontracted laboratory for analysis. A copy of all inter-lab COCs will be included with the final analytical report.

Laboratory Sample Handling and Storage

Upon receipt by the laboratory, the samples will be inspected for sample integrity and proper preservation, including temperature. The COC will be reviewed to verify completeness. Any discrepancies between the COC and sample labels and any problems or questions noted upon sample receipt will be communicated immediately to the Formation QA Manager. The laboratory shall provide the Formation QA Manager with a copy of the COC, and associated sample-receipt information, within 2 working days of receipt of samples. The sample-receipt information routinely provided will include: sample receipt date, sample ids transcribed from the COCs, sample matrix type, list of analyses to be performed for each sample, and verification of sample temperatures and preservation requirements. Broken custody seals, damaged sample containers, sample labeling discrepancies between container labels and the COC form, and analytical request discrepancies shall be noted on the COC form. The Formation QA Manager shall be notified of any such problems; discrepancies or nonconformances shall be resolved and addressed prior to the samples being released to the laboratory for analysis.

The laboratory will store the samples in a specially designated area, which is clean and maintained at the appropriate preservation temperature, if necessary. The laboratory will be responsible for following their internal custody procedures from the time of sample receipt until sample disposal. At a minimum, the following procedures shall also be in place for laboratory storage of samples:

- samples and extracts shall be stored in a secure area controlled by the laboratory's designated sample custodian;
- samples shall be removed from the shipping container and stored in their original containers unless damaged; damaged samples shall be disposed in an appropriate manner after notifying the Formation QA Manager, and authorization to dispose is received and documented;
- whenever samples are removed from storage, removal shall be documented;
- sample transfers shall be documented on internal COC records;
- samples and extracts shall be stored after completion of analyses in accordance with contractual requirements; and
- samples shall not be stored with standards or sample extracts.

2.3.3 Analytical Methods

Samples will be prepared and analyzed using standard laboratory procedures and methods according to performance criteria identified in the following sections.

2.3.3.1 Sample Preparation

The analytical parameters and targeted method detection limits and/or quantitation limits and analytical methods for the RI/FS sample analyses are specified in the matrix-specific tables included with the FSP (Section 3.0). A copy of the appropriate sample-analysis and method table, by sample type and sampling task, will be included in each batch of samples submitted to the laboratory for analyses to accurately document the analyses being requested.

Sample preparations shall be in accordance with the USEPA SW-846 method specifications included in Table 2-7 as well as standard laboratory practices. Sample preparation methods for non-standard matrices, including vegetation and biologic tissue samples, will be performed as described in the approved laboratory Quality Management Plan and Table 2-7. QAPP Attachment 1 provides the Quality Management Plan for SVL Analytical. QAPP Attachment 2 provides the Quality Management Plan and relevant laboratory SOPs for preparation of non-standard sample types for Columbia Analytical Services.

Solid matrices such as sediment, soil, and vegetation shall be homogenized prior to analysis. Sediment, soil, vegetation, and tissue results typically shall be reported on a dry-weight basis. Percent moisture will also be reported for tissue samples to allow for future conversion of dry weight to wet weight concentrations, if necessary.

2.3.3.2 Target Analyses and Methods

<u>Water Matrices</u>

Tables 3-2 and 3-6 of the FSP list the target analytes and analytical methods for groundwater and surface water samples, respectively, collected to support the RI/FS.

Historically, water samples collected at the Site have been analyzed for selenium using a gaseous-hydride-generation, atomic absorption method (Standard Method 3114C). The FSP allows for selenium analyses by either ICP-MS, in accordance with EPA Method 6020, or the gas-hydride atomic absorption method (SM 3114C). Analyses performed using SM 3114C will maintain consistency and comparability with historical data. Water samples will be analyzed for other metals and metalloids using EPA 6010B (ICP), EPA 6020 (ICP-MS), and EPA 7470A (cold-vapor atomic absorption).

These methods, and the other methods specified in the FSP for water analyses, provide quantitative results at levels that are appropriate for the intended uses of surface water and groundwater data. The more protective of the national water quality criteria or State of Idaho water quality standards listed on Table 2-8 will serve as preliminary screening values for evaluating the nature and extent of surface water and groundwater contamination for each of

the COPCs. The other screening levels listed on the table are risk-based concentrations that will be used in either the human health or ecological risk assessment.

Surface water and groundwater monitoring parameters are listed on Table 2-8 with regulatory and risk-based screening values for these parameters in surface water and groundwater. The reporting limits and method detection limits (MDLs) are less than these screening levels with a few exceptions. The arsenic reporting limit (0.003 mg/L) and MDL (0.0005 mg/L) are greater than the USEPA Regional Screening Level for arsenic in tap water of 0.0000045 mg/L. The antimony reporting limit (RL) (0.003 mg/L) is higher than the EPA Regional Screening Level for antimony in tap water (0.0015 mg/L), but the MDL (0.00022 mg/L) is lower than the screening level. The chromium VI reporting limit (0.01 mg/L) and MDL (0.005 mg/L) are higher than the USEPA Regional Screening Level for residential tap water (0.000043 mg/L). For chromium VI, the EPA Method that is specified, EPA 7199 (ion chromatography) is the EPA Method that provides the lowest MDL available for chromium VI. Analyses for chromium VI will be performed on surface water samples only when the total chromium result for a previous sample is >/= 0.011 mg/L.

Soil, Sediment, and Tissue Matrices

Tables 3-7 and 3-9 of the FSP list the target analytes and analytical methods for soil and sediment, and biologic tissue samples, respectively. Historically, tissue samples have been analyzed for selenium using a modified version of EPA Method 7742, a borohydride-reduction, atomic absorption method. Due to the complex sample matrix (i.e., homogenized and digested tissue), Simplot's contracted laboratory for tissue analyses, Columbia Analytical Services, recommends that this method continue to be used for tissue analyses conducted in support of the RI/FS. According to Columbia Analytical Services, Method 7742 provides superior accuracy to Method 6020 for selenium analyses of tissue matrices. Tissue samples collected to support the RI/FS will be analyzed for selenium in accordance with EPA Method 7742 and Table 2-5. The other COPCs in tissue samples will be analyzed using EPA methods 6010B, 6020, 7471A and in accordance with those methods and Tables 2-2 through 2-4.

The methods specified in the FSP for analyses of soil and sediment samples for the RI COPCs provide quantitative results at levels that are appropriate for the intended uses of those data. Soil and sediment COPC concentration data will be used to describe the nature and extent of COPC contamination and also to evaluate human health and ecological risks. COPCs are listed on Table 2-9 with human health and ecological risk-based screening values for these parameters in soil. The screening levels listed in Table 2-9 provide conservative criteria (i.e., low concentrations) for the purpose of selecting sample analysis methods for COPCs in soil and sediment.

The reporting limits (RLs) and MDLs for methods selected for soil and sediment COPC analyses are less than the risk-based screening levels listed in Table 2-9 with two exceptions. The RL for antimony (0.3 mg/Kg) is greater than the soil screening level (SSL) for mammalian exposures

(0.27 mg/Kg), but the MDL for antimony (0.011 mg/Kg) is less than the SSL. Because the laboratory will report quantitative values to the MDL (refer to Section 3.4 below), antimony results reported between the MDL and RL will be available for comparison to the SSL value. The RL for silver (0.5 mg/Kg) is the same as the sediment lowest effect level (LEL) for silver, but the MDL (0.02 mg/Kg) is lower than the LEL. The RL for arsenic is close to, but does not exceed, the residential SSL for evaluation of risks to human health. The analysis method selected for antimony, arsenic, and silver (ICP-MS, EPA Method 6020) is among the standard EPA analytical methods that provide the lowest available detection limits for these parameters.

The methods specified in Table 2-10 for analyses of biological tissue and vegetation samples for the RI COPCs provide quantitative results at concentration levels appropriate for the anticipated uses of these data. There are no risk-based screening levels for COPCs in tissue or vegetation. Instead, the COPCs are listed on Table 2-10 with target detection limits that are calculated as one-tenth the USEPA Ecological Soil Screening Levels (EcoSSLs), which serve as conservative criteria for the purpose of selecting the sample analysis methods.

Columbia Analytical determines their MDLs for analyses of tissue samples in accordance with the MDL protocols described in CAS SOP ADM-MDL, which is included in QAPP Attachment 2 for reference. In the case of MDL development for analyses of metals/metalloids in tissue samples, the solid blank material used is a Teflon chip that is free of the analytes of interest.

All of the RLs listed on Table 2-10 for tissue and vegetation sample analyses are less than the target detection limits for those sample types. The only the MDL higher than a target detection limit is the MDL for antimony, which is higher than the target minimum detection limit for evaluating mammalian-receptor exposures. The analysis method selected for antimony in tissue and vegetation samples is one of the standard EPA methods that provides lowest available detection limits for this parameter of interest.

2.3.4 Quality Control

There is potential variability in any sample collection, analysis, or measurement activity. This section describes checks that will be performed to evaluate that variability.

2.3.4.1 Field Quality Control Samples

Field quality control samples are introduced into the measurement process to provide information on transport, storage and field handling biases and on field sampling precision. Equipment rinsate blanks, field ambient blanks, and field duplicate samples will be collected. The equipment rinsate and field ambient blanks may be identified to the laboratory so that they are not used for preparation of an analytical duplicate or matrix spike sample. Descriptions and frequencies of these QC samples are provided below.

Field Blank Samples

Two types of field blanks will be collected: equipment rinsate blanks and ambient blanks. An equipment rinsate consists of analyte-free reagent-grade water (e.g., American Society for Testing and Materials [ASTM] Type II) poured through the sampling equipment, collected in a clean sampling bottle, and preserved as needed. Equipment rinsate samples may be used to demonstrate that sampling devices have been adequately cleaned between uses and provide for representative samples. An ambient blank consists of analyte-free, reagent-grade water poured into an unused, clean sample container at the field sampling location, and preserved as needed. Equipment rinsates and ambient field blanks are analyzed blindly as regular field samples, and they are both analyzed for the same suite of analytical parameters as the associated samples.

Analyses of equipment rinsates and field ambient blanks quantify any artifacts introduced into the sample during collection. Potential sources of bias or cross-contamination include sampling gloves and sampling equipment that may incidentally come into contact with the sample.

Equipment rinsate samples will be collected at rate of 1 per every 20 field samples and at a minimum of one per day for waters, sediment, soil, and tissue sampling when sampling equipment is reused at multiple sampling locations (see Table 2-11). In the absence of equipment rinsate blanks for water samples that are collected without re-using sampling equipment, field ambient blanks will be collected at a rate of 1 per 20 water samples (see Table 2-11).

Field Duplicates

Field duplicates are collected to measure the sampling and analytical variability associated with the sample results. Duplicate samples are usually collected simultaneously with or immediately after the corresponding original samples have been collected, depending on the sample type and medium and consistent with detailed instructions in the relevant SOPs for sample collection. In all cases, the same sampling protocol is used to collect the original sample and the field duplicate sample. The field duplicate is analyzed for the same suite of analytical parameters as the original sample. There are no USEPA criteria for evaluation of field duplicate sample comparability; however, the relative percent difference (RPD) between the original sample and field duplicate can be calculated for each parameter and compared to the project's precision goal. Field duplicate RPDs greater than the project-specified precision goal indicates a high variability associated with the sampling and analysis methods used.

For groundwater, surface water, sediments, and soil samples, field duplicates will be collected at a rate of 1 per 20 samples or a minimum of one per day, by matrix and by sample type.

Table 2-11 summarizes the minimum required frequencies for the field QC samples.

2.3.4.2 Laboratory Quality Control Samples

Laboratory quality control samples are introduced into the measurement process to evaluate laboratory performance and sample measurement bias. Control samples may be prepared from environmental samples or be generated from standard materials in the laboratory. The appropriate type and frequency of laboratory QC samples will be dependent on the sample matrix, analytical method, and the specifications included in Tables 2-2 through 2-5 and Table 2-12. Laboratory QC samples will be analyzed in addition to the calibration samples with each QC batch.

Table 2-12 summarizes the minimum required frequencies for the laboratory QC samples. A laboratory method blank, laboratory control sample, analytical duplicate, and a pair of matrix spike samples should be run in each laboratory QC batch with a frequency of one each per 20 field samples. If less than 20 field samples are submitted, then one set of these three QA/QC samples should still be run per batch. Field staff responsible for collection and shipping of samples to the laboratory shall designate the samples to be used for laboratory QC analyses on the COC forms. In the event that such instructions are not included, the laboratory shall always utilize samples submitted from the Smoky Canyon RI/FS for preparation of laboratory duplicates and matrix spike samples used for batch QC analyses.

<u>Method Blanks</u>

Method blanks shall be used for the laboratory processes. A method blank is a volume of deionized water that is carried through the entire sample preparation and analysis procedure. The method blank volume or weight shall be approximately equal to the sample volumes or sample weights being processed. Method blanks are used to monitor interference caused by constituents in solvents and reagents and on glassware and other sampling equipment.

Project target analytes must not be detected in laboratory method blanks at concentrations greater than the Reporting Limit (RL). Method blank contamination, if found, will be addressed in accordance with the response actions given in Tables 2-2 through 2-5, as appropriate to the analytical methods. Method blanks will be evaluated during the data validation process, and associated sample results may be qualified on the basis of blank contamination.

<u>Laboratory Control Samples and Standard Reference Materials</u>

A laboratory control sample (LCS), or a blank spike, is an aqueous or solid control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the program samples. An LCS is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix for a stock solution that is different than that used for the calibration standards. The LCS is the primary indicator of process control used to demonstrate whether the sample preparation and

analytical steps are in control, apart from sample matrix effects. LCS samples will be run with all water, soil, and sediment samples at the frequencies specified herein.

Reference materials, known as Standard Reference Materials (SRMs) or Certified Reference Materials (CRMs), are homogeneous and stable materials for which target analyte concentrations have been determined with a very high degree of certainty. SRMs and CRMs are obtained from an outside entity such as the National Institute of Standards and Technology (NIST) in the US or the National Research Council of Canada (NRC-Canada). SRMs/CRMs can serve the same function as LCSs for analyses of non-standard sample matrices such as vegetation and fish tissue. Analyses of SRMs/CRMs are used to demonstrate whether sample preparation and analytical steps are in control for a matrix that is the same or similar to these types of non-standard sample matrices. Appropriate SRMs will be identified by the laboratory for analyses with terrestrial and aquatic vegetation, fish tissue, macro-invertebrate tissue, and small mammal tissue samples at the frequencies specified in Tables 2-2 through 2-5 and Table 2-12.

Analytical Duplicates

Analytical duplicates are samples that are split at some step in the measurement process and then carried through the remaining steps of the process. Duplicate analyses provide information on the precision of the operations involved.

- Analytical duplicates are a pair of subsamples from a field sample that are taken through the entire preparation and analysis procedure; any difference between the results indicates the precision of the entire method in the given matrix.

- Under certain method protocols (refer to Tables 2-2 through 2-5), the matrix spike is duplicated, to provide a matrix spike duplicate, and serves as the analytical duplicate sample.

Analyses of analytical duplicates and/or matrix spike duplicates monitor the precision of the analytical process.

Matrix Spikes

A matrix spike is prepared by adding an analyte to a subsample of a field sample before sample preparation and analysis. For multi-analyte methods, a representative suite of the analytes is used in the matrix spike. From the concentrations of the analyte in the spiked and unspiked samples, a percent recovery is calculated. Many samples show matrix effects in which other sample components interfere with the determination of the analyte. The value of the percent recovery indicates the extent of the interference.

Laboratory matrix spike samples are used to evaluate potential sample matrix effects on the accurate quantitation of an analyte using the prescribed analytical method. Percent recoveries of target analytes from matrix spike samples should fall within the prescribed control limits. Matrix interference and other effects may cause low or high percent recoveries in investigative samples; matrix effects may be noted at the same time that recoveries from laboratory control samples indicate acceptable method performance.

Site-specific samples need to be used for MS/MSDs. Field sampling personnel will collect extra volume and designate on the COC forms the samples that are to be used for the MS/MSD. Every effort will be made to ensure that these samples are representative of the general sample matrix of samples collected on that sampling data. Equipment rinsate and field ambient blank samples are not designated for MS/MSD.

The laboratories will be instructed to use spike concentrations that are consistent with criteria provided in the National Functional Guidelines for Inorganic Data Validation (EPA, 2004) and any specific instructions provided in the referenced analytical methods. Note that due to the wide range of COPC concentrations anticipated in most of the sample matrices submitted to the lab, the target spike concentrations may not always be achieved for all samples in a given sample delivery group.

Performance Evaluation Samples

Program-specific laboratory performance evaluations via performance evaluation samples are not anticipated as part of this Smoky Canyon Mine RI/FS, but may be performed if analytical or validation exercises indicate the presence of potential laboratory QA issues.

2.3.5 Instrument/Equipment Calibration and Maintenance

In order to ensure continual quality performance of any instruments or equipment, calibration and maintenance shall be performed and recorded as described in this section.

2.3.5.1 Field Equipment

Preventative maintenance of field equipment will include routine inspection and either calibration or testing at specified in the relevant SOP or manufacturer's instructions.

All field equipment will be cleaned and safely stored between each use, and any routine maintenance recommended by the equipment manufacturer will also be performed. Equipment will be inspected and the calibration checked (if applicable) before it is transported to a field setting for use. Equipment will be inspected before use and field instruments that fail calibration requirements will be tagged as "nonfunctional" or "defective" and returned to the manufacturer or other supplier for repair or replacement.

Water quality parameters to be measured during groundwater and surface water sampling include pH (standard units), temperature (°C), oxidation-reduction potential (redox potential or ORP, mV), conductivity or specific-conductance (μS/cm), nephelometric turbidity unit (NTU), ferrous and ferric iron, and dissolved oxygen (DO, mg/L). All field equipment designed to provide these measurements require daily calibration prior to use to ensure that the accuracy and reproducibility of the results are consistent with the manufacturer's specifications and the project's data needs.

Field equipment shall be calibrated using the standards specified or provided by the equipment manufacturer. Atmospheric oxygen shall be used to calibrate the dissolved oxygen element. Calibration standard usage prior to and after field activities shall be according to the manufacturer instructions. Instructions for performance and documentation of calibration for such field equipment are provided in JRS SOP No. 5 (Water Quality Sampling), MFG SOP No. 17 (Field Measurement of Dissolved Oxygen), and MFG SOP No. 13 (Field Measurement of Oxidation-Reduction Potential) (FSP Attachment).

2.3.5.2 Laboratory Equipment

Instruments used by the laboratory will be maintained in accordance with the laboratory's Quality Assurance Plan and method requirements. All analytical measurement instruments and equipment used by the laboratories shall be controlled by a formal calibration and preventive maintenance program. In addition, each laboratory's preventive maintenance program shall include the following, as a minimum:

- a listing of the instruments and equipment;
- the frequency of maintenance considering manufacturer's recommendations and previous experience with the equipment; and
- a file for each instrument containing a list of spare parts maintained, external contracts, and a listing of the items to be checked or serviced during maintenance.

The laboratory will keep maintenance records and make them available for review, if requested, during laboratory audits. Laboratory preventative maintenance will include routine equipment inspection and calibration at the beginning of each day or each analytical batch, per the laboratory's internal SOPs, the specifications included in Tables 2-2 though 2-5 and method requirements.

<u>Calibration Methods</u>

Physical and chemical calibrations shall be performed within each laboratory as specified by the EPA Methods, instrument manufacturer's guidelines, and this project's calibration requirements for the requested EPA methods, which are summarized in Tables 2-2 through 2-5. When laboratory measurement instruments do not meet the calibration criteria of the laboratory's Quality Assurance Plan and/or EPA method, then the instrument will not be used for analysis of

samples submitted under this QAPP. Calibration records and demonstration of acceptable calibration results will be required elements of the laboratory's data reporting. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing QC activities. These records will be filed at the location where the work is performed and will be subject to QA audit.

Calibration procedures for a specific laboratory instrument will consist of initial calibration (blank and standards), initial calibration verification (ICV) and continuing calibration verification (CCV). All analyses will be governed by the specifications provided in Tables 2-2 through 2-5, and appropriate calibration procedures and frequencies can be found in each of these tables. Calibration and quality control sample procedures for trace metals analysis by EPA Method 6020 (inductively coupled plasma-mass spectrometer [ICPMS]) are provided in Table 2-2. Calibration and quality control sample procedures for trace metals analysis by EPA Method 6010B (inductively coupled plasma [ICP]) are provided in Table 2-3. Calibration and quality control sample procedures for mercury analysis by EPA Method 7470A/7471A (cold vapor atomic absorption [CVAA]) are provided in Table 2-4. Table 2-5 presents calibration and quality control sample and calibration criteria for selenium analyses by EPA Method 7742 and Standard Method 3114C.

2.3.6 Acceptance Requirements for Supplies and Consumables

All supplies and consumables received for a project (e.g., sample bottles, calibration standards) will be checked for damage and other deficiencies that would affect their performance. All inspections should be documented and a copy of the inspection should be kept in the project's file.

2.3.7 Criteria for Use of Existing, Non-Direct Measurement Data

The general categories of intended data uses for the RI/FS include:

- RI/FS scoping;
- Identification of data needs and development of preliminary (pre-RI) conceptual site model;
- Characterization of sources;
- Characterization of the nature and extent of COPC distribution;
- Description of COPC fate and transport pathways and evaluation of temporal or spatial trends;
- Development and evaluation of remedial alternatives; and
- Assessment of baseline ecological and human health risks.

Previous investigations of Site conditions provide a large quantity of existing chemical and other measurement data that are relevant to each of these general categories of data uses. This section describes the specific criteria that will be used to evaluate and potentially accept existing data, i.e., non-direct measurement data, for the different uses listed above. The most stringent criteria for acceptance of existing (non-measurement) data will be applied to data that are being considered for use in risk assessment and more specifically for use in completing baseline exposure calculations. The data usability criteria for baseline exposure calculations are defined below in Section 2.3.7.1. The usability criteria for the other data uses listed above are described in Section 2.3.7.2.

2.3.7.1 Criteria for Acceptance of Existing Data for Use in Risk Assessment Exposure Calculations

All data used to complete exposure calculations for risk assessment, whether existing data or data collected in accordance with the RI/FS SAP, will be subject to the quality assurance and quality control requirements specified by this QAPP. Any existing, chemical data sets identified by risk assessors as relevant for exposure calculations will be subject to review to confirm that either (1) appropriate supporting documentation is available to allow for validation of data in accordance with specifications described in Section 2.4.4.2 for Stage IIB (USEPA, 2009), or higher, validation protocols or (2) data have already been validated in accordance with those specifications.

For those data sets with sufficient supporting information and data validation already completed, the existing data validation records will be reviewed to confirm that data quality has been evaluated and described in general accordance with the project's measurement criteria identified above in Sections 2.2.5.3 and 2.2.5.4 and Tables 2-2 through 2-5 of this plan. Qualifiers previously applied to individual results will be reviewed to confirm that the criteria for data qualification were consistent with the criteria presented in this plan, USEPA's 2004 National Functional Guidelines for Inorganic Data Review (USEPA, 2004), and USEPA protocols for Stage IIB validation (USEPA, 2009), at a minimum.

For data sets with sufficient supporting information but incomplete data validation, data validation will be completed and documented in accordance with Tables 2-2 through 2-5 and Section 2.4.4.2 of this plan and consistent with National Functional Guidelines for Inorganic Data Review (USEPA, 2004) and USEPA Stage IIB validation protocols (USEPA, 2009).

Upon completion of these review and validation steps, as appropriate for each data set, the validated data and any assigned data validation qualifiers will be incorporated into the project's electronic database, and the data will be identified therein as usable for quantitative risk assessment (i.e., Level 4 data, see below). A final analysis and determination of data usability for baseline risk assessment will be conducted by the ultimate data users, the risk assessors, upon completion of new data collection and compilation of new and existing data. That final

analysis will be performed in accordance with USEPA Guidance for Data Usability in Risk Assessment (USEPA, 1992) and Section 2.4.5 of this plan.

2.3.7.2 Criteria for Acceptance of Existing Data for Other RI/FS Data Uses

Previous investigations of Site conditions provide environmental data that are relevant to the Site-characterization objectives of the RI/FS. The Preliminary Data Usability Summary Table provided in QAPP Attachment 3 presents the sources of existing data, or non-direct measurement data, that have been identified as relevant and potentially usable for the RI/FS. Nearly all of the existing data sources listed in QAPP Attachment 3 provide Site-specific chemical data or other measurement data that was collected in accordance with planning and quality control documents reviewed and approved by the USFS and/or one or more of the RI/FS support agencies (e.g., BLM, EPA, IDEQ, etc.) and originally intended for use in characterizing Site conditions. The supporting information available to describe the quality of data included in each of these data sets typically includes detailed records of field and laboratory procedures, complete field and laboratory data documentation and quality control records, and final reports that provide descriptions of data quality and/or data validation.

Existing Site-specific, chemical data and associated documentation will be reviewed to ensure that the type and quality of the data are appropriate for the specific types of RI/FS data uses. For each of the individual data sources listed in the Preliminary Data Usability Screening Summary Table (QAPP Attachment 3), this process will include review of the original planning documents; all available documentation regarding sample collection, handling, and quality control procedures; laboratory analysis methods and quality control procedures; and previous data quality reviews or data validation completed by other investigators or data users, if any.

The data usability review process described above will be consistently documented for each data set using the Data Usability Review Checklist, provided in QAPP Attachment 3, to guide completion of a final Data Usability Screening Summary Table. One or more data usability checklists will be completed for each of the evaluated data sets, and the checklist will be used to guide entry of required review information on the Data Usability Screening Summary Table. At the end of the usability review completed for each data set, the data will be assigned a data usability level number (Level 0, 1, 2, 3a, 3b, or 3c) for use in tracking appropriate data uses in the project's database. Any Level 3 data evaluated for use in risk assessment and found usable, as described above in Section 2.3.7.1, will be assigned Level 4. Data usability levels and corresponding data uses are defined in the table below.

Database queries made to support the objectives of the RI/FS will include a specification for data usability level such that each data set ultimately assembled from existing data for use in appropriate RI/FS Site characterization activities will be of known quality.

Data Usability Levels and Appropriate Data Uses

Data Usability Level	Appropriate Data Uses
Level 0	Not usable for RI/FS
Level 1	RI/FS scoping
Level 2	Development of preliminary conceptual site model; evaluation of temporal or spatial trends; identification of site characterization and risk assessment data needs.
Level 3a (data quality not reviewed)	Limited to Level 2 data uses until data quality review completed; available for Level 3b data uses after data quality review completed.
Level 3b (reviewed but not validated)	Characterization of nature and extent of contamination, source characterization, identification of transport pathways. Usable for baseline risk assessment upon completion of project-specified validation.
Level 3c (reviewed and validated)	Same as Level 3b.
Level 4 = Level 3 data that have been validated in accordance with project validation protocols and meet project DQOs for risk assessment data uses.	Same as Level 3b and also baseline risk assessment (primary lines of evidence).

2.3.8 Data Management

The program quality records will be maintained by Simplot's contractor, Formation in their Boulder, CO office. These records, either electronic or hard copy in form, shall include:

- Project work plans with any approved modifications, updates, and addendums;
- Project sampling and analysis plans, including the QAPP, with any approved modifications, updates, and addendums;
- Field documentation;
- COC records;
- Laboratory documentation (results received from the laboratory will be documented both in report form and in an electronic format);
- Data validation reports;
- Data Summary Reports submitted to USFS; and
- Final project reports/deliverables.

Hard-copy field and laboratory records shall be maintained in the project's central data file, where original field and laboratory documents are filed chronologically for future reference. These records are also scanned to produce electronic copies in *.pdf format. The electronic

versions of these records are maintained on Formation's central server system with backup scheduled on a daily basis.

All project records listed above shall be maintained on file by either Simplot, Formation, or another Simplot-designated and USFS-approved contractor for a minimum of 10 years after completion of the RI/FS work, consistent with the requirements of the 2009 Settlement Agreement/CO for the RI/FS (refer to Section 12 of the Settlement Agreement/CO).

A key element of the project's data management process is maintenance of an electronic database that is used to store relevant environmental sampling data, including existing data considered usable to support the RI/FS (i.e., non-direct measurement data), in a consistent, readily retrievable format. Microsoft® Access will be used for the data structure and query support, and a designated Database Manager will ensure the security and integrity of electronically stored data. The project's electronic database will be maintained on a central server system with data backup scheduled on a daily basis.

The project database serves as a source of data for the data presentation and analysis tasks performed to support the RI/FS. The database contains information describing chemical and physical parameters for surface water, groundwater, soil/overburden material, and sediment samples, as well as surface water flow and groundwater elevation measurements. Sample information and results for analyses of vegetation, fish, and other terrestrial and aquatic biota are also included. The database incorporates, at a minimum, sample collection information (e.g., sample identification, location, date and time of sample collected, matrix) and laboratory analytical fields specified in the project EDD requirements (Table 2-13).

Prior to incorporation of field and laboratory data into the project database, the data and supporting documentation shall be subject to appropriate review, as described below in Section 2.4, to ensure the accuracy and completeness of original data records. Field data that has been reviewed in a hard-copy format will be entered into electronic data files for upload to the project database. All manual data entry into an electronic format will be reviewed by a separate party before such data are incorporated into the project's database (see Section 2.4.1). Laboratory EDDs and related data packages will be reviewed as part of the data validation process, as described in Sections 2.4.2 and 2.4.4.

Following these review steps, field and laboratory electronic data files will be imported to the project database. Electronic files received from the data validators (refer to Table 2-14) will also be imported to maintain result qualifiers, and related information, for reference by all data users. The USEPA flags, Reason Codes, and final, qualified data will be uploaded from electronic files that the data validators populate (see Table 2-14) and return to Simplot/Formation, as discussed in Section 2.4. Standardized data import formats and procedures will be used to upload both field and laboratory data into the electronic database. At this time, standardized station identifiers, parameter names, numerical formats, and units of measure are applied to the original information to facilitate comparability across all datasets and within the database.

2.4 Data Review, Validation and Usability

The following sections address the final project checks conducted to confirm that the data obtained meet the project objectives and to estimate the effect of any deviations on data usability.

2.4.1 Field Data Review

Raw field data shall be entered in field notebooks; and/or sample collection record forms, which shall be reviewed for completeness by the Field Investigations Manager or Field Supervisor at the end of each day. The overall quality of the field data from any given sampling round shall be further evaluated during the process of data reduction and reporting.

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Field data review will include verification that QC checks and calibrations are recorded properly in the field logbooks and/or data sheets and that any necessary and appropriate corrective actions were implemented and recorded. Such data will be written into field logbook and/or data sheets immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, the appropriate Field Supervisor will proof the field logbooks and/or data sheets to determine whether any transcription errors have been made by the field crew. If transcription errors have been made, the appropriate Field Supervisor and field crew will address the errors to provide resolution.

Field measurement data will be entered into electronic files for import to the project's database. Data entries will be made from the reviewed field data sheets or logbooks, and all data entries will be reviewed by a separate party before the electronic file is provided to the database manager. Electronic files of field measurement data will be maintained as part of the project's quality records.

2.4.2 Laboratory Data Review

Internal laboratory data reduction procedures will be according to the laboratory's Quality Management Plan. At a minimum, paper records shall be maintained by the analysts to document sample identification number and the sample tag number with sample results and other details, such as the analytical method used (SOP #), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. These records shall be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these records by the laboratory QA Manager takes place prior to final data reporting to Simplot.

QC data (e.g., laboratory duplicates, LCS, SRMs, MSs, and MSDs) will be compared to the specifications included in Tables 2-2 through 2-5. Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the laboratory QA Manager for review. If approved, data are logged into the project database format. The laboratory shall appropriately flag unacceptable data in the data package.

2.4.2.1 Laboratory Data Reporting Requirements

The laboratories shall prepare hard copy data packages for transmittal of results and associated quality control information to Simplot and Formation in general accordance with the following instructions, which are based on the USEPA's CLP Statement of Work. Deviations from these specifications may be acceptable provided the hard-copy report presents all of the requested types of information in an organized, consistent, and readily reviewable format. Laboratories providing hard copy data reports for this project shall be responsible for reviewing the following requirements, notifying Formation of any differences between their reports and these requirements, and confirming the acceptability of their intended report content and format with Formation before any laboratory data reports are generated for this project.

Each hard-copy report will be paginated and organized with a table of contents. A cross reference that correlates the client or field identification as provided on the chain-of-custody document with the laboratory's sample identification will be included.

For each batch of sample results consisting of 20 or fewer samples analyzed together and sharing common QC data, the laboratory data will be presented on a form equivalent to the USEPA Contract Laboratory Program (USEPA CLP) "Form 1" (see below). Case narratives will be prepared which will include information concerning data that fell outside laboratory acceptance limits, and any other anomalous conditions encountered during sample analysis.

CLP Form 1 contains all required data for field samples. The Form 1 (or equivalent reporting mechanism) will provide the following information:

- Field sample identification;
- Laboratory sample identification;
- Sample result, with appropriate units, method detection limit, and reporting limit. [COPC concentrations equal to or greater than the MDL will be reported. Concentrations between the MDL and reporting limit will be flagged by the laboratory. Parameters that are not detected or not present at concentrations equal to or greater than the MDL are flagged by the laboratory as "U" and interpreted to be not detected at a value equal to or greater than the MDL. Any non-detected value ("U" flagged) will be reported with its reporting limit and MDL.];
- Sample collection and receipt dates;
- Sample preparation date/time;

- Analysis date/time;
- Dilution factor;
- Preparation batch number or identification;
- Analysis batch number or identification;
- Sample matrix and instrument;
- Percent moisture determination; and
- For solid-matrix samples, identify basis of reporting (i.e., wet-weight or dry-weight basis).

The following additional information will be provided with the Form 1s, as applicable for the reported analytical methods. QC batch will be clearly associated with each sample (on the CLP Form specified, or an equivalent reporting mechanism):

- Case narrative;
- Chain-of-custody;
- Summary of all field sample results (Form 1s, or equivalent, as described above);
- Sample results and preparation blank;
- Initial calibration verification (ICV), and continuing calibration verification (CCV);
- Initial calibration blanks (ICB), continuing calibration blank (CCB), and preparation blanks;
- Inductively coupled plasma (ICP) interference check sample (CLP Form IVA-IN) or inductively coupled plasma-mass spectrometer (ICPMS) interference check sample;
- Matrix spike (MS), and when applicable matrix spike duplicate (MSD), sample recovery and, when applicable, MS/MSD relative percent difference (RPD);
- Post-digest spike sample recovery;
- Laboratory duplicate precision;
- Laboratory control sample (LCS) recovery;
- ICP and ICPMS serial dilution percent differences;
- MDLs;
- ICP interelement correction factors;
- ICP and ICPMS linear ranges;
- Preparation log;
- Analysis run Log;
- ICPMS tunes;
- ICPMS internal standards relative intensity summary;
- Sample log-in sheet; and
- Deliverables inventory sheet.

In addition to this standard data package, the laboratory shall deliver a "Level 4" data package, as detailed below. The Level 4 Data Package includes all items specified above plus instrument raw data and/or documentation of the following:

- Calibration standards (including source, preparation date).
- Blanks (ICB, CCB, and preparation).
- ICV, CCV standards.
- Interference check samples.
- Serial dilution samples.
- LCS.
- Diluted and undiluted samples.
- Dilution factors.
- Sample volumes.
- Laboratory duplicates.
- Matrix spikes (source, concentration, volume).
- Post-digest spikes (source, concentration, volume).
- Method of standard addition results.
- Instrument identification.
- Analysis date and time.
- Integration time (cold vapor atomic absorption [CVAA] only).
- All inorganic methods: full raw data printouts from instruments.
- Full run log for each analysis. and
- ICPMS to include: internal standard recoveries, tune data (atomic mass unit [amu] and peak width), and molecular interference check data.

2.4.2.2 Laboratory Electronic Data Deliverable

Each hard copy data package, as described above, shall be accompanied by an electronic data deliverable (EDD) prepared by the laboratory. The content and format of laboratory electronic data deliverables (EDDs) are specified in Table 2-13. Additional laboratory QC data can be included in the EDD as long as the data fields specified in Table 2-13 are also maintained.

EDDs will be cross checked against corresponding hard-copy data reports to confirm consistency in results reported in these two separate formats. This cross check will take place as part of the data validation process described in Section 2.4.4.

2.4.3 Specific Quality Control Assessment Procedures

The accuracy, precision, completeness, and representativeness of analytical data will be described relative to the project's control limits through a process of field and laboratory data quality review and data validation. Results from these reviews will be documented in routine Data Summary Reports prepared for all data users, including the USFS and supporting agencies, and any qualification of the data resulting from that review will also be incorporated into the project's electronic database so that all data users are aware of any uncertainties associated with individual results.

2.4.4 Data Validation

Data validation is the process of verifying that qualitative and quantitative information generated relative to a given sample is complete and accurate. Data validation procedures shall be performed for both field and laboratory operations as described below.

2.4.4.1 Evaluating Field Data

The results of field quality control sample analyses associated with each laboratory data package will be reviewed to allow for evaluation of equipment blanks and other field QC samples and further indications of the data quality. If a problem is identified through the review of field QC data, all related field samples will be identified, and if possible, corrective actions can be instituted and documented. If data are compromised due to a problem identified via field QC sample review, appropriate data qualifications will be used to identify the data for future data users.

The handling, preservation, and storage of samples collected during the sampling program will be monitored on an on-going basis. The project laboratories will document sample receipt including proper containers and preservation at the time samples are logged into their individual laboratory. The sample receipt records (a required data package deliverable) as well as the COC documentation will also be assessed during third-party data validation. Sample handling, storage or preservation problems identified during data validation will result in appropriate qualification of data.

2.4.4.2 Evaluating Laboratory Chemistry Data

The purpose of chemistry data validation is to verify that the data are of known quality, technically valid, defensible, and usable for their intended purpose. The objectives of the third-party data validation process will be to:

- Verify completeness of data packages and corresponding EDDs;
- Assess compliance to project specific procedures and programs;

- Evaluate system process control through review of control charts (if applicable);
- Verify that no systematic errors exist within the data sets;
- Assess field QC samples to determine if sampling has adversely impacted the reported results and, therefore, usability;
- Assess both method and laboratory performance through tabulation of QC outliers; and
- Provide measures of data quality in terms of precision, accuracy, and completeness so that overall usability can be determined.

Data validation will be performed by a 3rd party using the general protocols and processes described in the following documents, as applicable to the method calibration and QC limits specified on Tables 2-2 through 2-5 and to the extent possible when certain non- CLP methods are used:

- Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (NFG; USEPA, 2004); and
- Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (USPEA, 2009).

Data validation for the Smoky Canyon RI/FS project will be performed using a "tiered" approach. One-hundred percent (100%) of the data packages will be evaluated and qualified for all quantitative QC elements (e.g., spike recoveries, method and field blank contamination, duplicate sample %RSD, and instrument stability and performance [e.g., initial and continuing calibration results, instrument tuning and internal standard areas]) using hard-copy summary forms (described above). This validation of 100% of the data is considered Tier 1, and it is equivalent to a “Stage 2B Validation,” as defined in the USEPA guidance for labeling externally validated data (USEPA, 2009).[1] Specific QC elements that will be reviewed during the Tier 1/Stage IIB validation include:

- Presence and completeness of COC and sample receipt documentation;
- Sample Index (correlation of field sample ID to laboratory sample ID);
- Laboratory Case Narrative (method deviations and QC anomalies);
- Analytical holding times;
- Method blank;
- Matrix spike recoveries;
- Matrix spike/matrix spike duplicate RPD values;
- Field duplicate RPD values;
- Laboratory Duplicate RPD values;
- Summaries of initial and continuing Calibration;

[1] EPA, 2009. Page 6: “A verification and validation based on completeness and compliance checks of sample receipt conditions and BOTH sample-related and instrument-related QC results…”

- Summaries of instrument blanks (e.g., initial calibration blank, CCB, if specified in method);
- Review of reagent/preparation blanks (inorganics);
- Review of Laboratory Control Standards (LCS);
- Instrument stability and performance (e.g., serial dilution); and
- Summaries of internal standards.

Tier 2 of the project's data validation is equivalent to an "USEPA CLP Level IV" validation and essentially the same as "Stage 4 Validation" (USEPA, 2009).[2] The Tier 2/Stage 4 validation includes all of the Tier 1 elements as well as a complete evaluation of all the raw data. Tier 2/Stage 4 validation will be performed for a minimum of 20% of the data.

Additional quality indicators evaluated during Tier 2 include:

- Completeness of laboratory documentation for sample receipt, sample analysis, and sample result reporting;
- Overall documentation practices;
- Raw data for instrument stability and performance (e.g., mass tuning, etc.);
- Interference check samples (ICP analysis);
- Serial dilutions (ICP analysis);
- Analytical spikes (graphite furnace analysis);
- Recalculation of results from raw data; and
- Transcription check (from raw data to final results).

The overall project minimum of 20% of the data packages will undergo Tier 2 validation. The data packages selected for Tier 2 validation will be representative of the project as a whole so as to include the various sample media and analysis methods conducted in support of the RI/FS. Since the laboratory is producing full "CLP-like" data packages for all data, as specified above for the hard copy data packages, and all data packages will be submitted to the data validation contractor, the selection of packages for Tier 2 is blind to the laboratory.

Data Validation Worksheets will be completed for both tiers of validation. These worksheets will be developed by the third-party data validator and subject to final review and approval of the USFS. The worksheets will indicate data qualifiers applied to individual results and reasons for application of those qualifiers. Definitions of the data qualifiers that may be applied to individual results as a result of data validation are as follows:

[2] Ibid, page 7: "A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, recalculation checks, AND the review of actual instrument outputs…"

U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

R The result is unuseable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample.

UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

The following “Reason Codes” will be applied as applicable to the validated data:

1 Holding Time

2 Sample Preservation (including receipt temperature)

3 Sample Custody

4 Missing Deliverable

5 ICPMS Tune

6 Initial Calibration

7 Initial Calibration Verification

8 Continuing Calibration Verification

9 Low-Level Calibration Check Sample

10 Calibration Blank

11 Laboratory or Preparation Blank

12 ICPMS or ICP Interference Check Standard

13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery

14 Laboratory Control Sample Precision

15 Laboratory Duplicate Precision

16 Matrix Spike or Matrix Spike Duplicate Recovery

17 Matrix Spike/Matrix Spike Duplicate Precision

18 ICPMS or ICP Serial Dilution

19 ICPMS Internal Standard

20 Field Replicate Precision

21 Equipment Rinsate Blank

22 Linear Range Exceeded

23 Other reason

24 Result is less than the minimum detectable concentration (MDC)

25 Result is less than two times the error

Formation will provide an EDD to the third-party data validator to populate (specifications are provided in Table 2-14). The validator will add the following data to that EDD upon completion of validation, and the updated EDD will be returned to Formation for incorporation into the project's database:

Field Header "USEPA Flag": Populate with USEPA flags specified above and in template reports.

Field Header "Reason Code": Populate with all applicable Reason Codes as specified above and in template reports.

Field Header "Final Result": Populate with the final, qualified result, including any adjustment based on blank contamination.

The 3rd party data validator will perform a Manual Validation, as defined in the USEPA guidance for labeling externally validated data (USEPA, 2009), on the hard copy data packages generated by the laboratories and provided by Formation.

2.4.5 Data Usability

Laboratory packages summarizing the data generated for this investigation will be validated as described above. Once validated, the data will be loaded into a project database managed by Formation. Data usability will be determined by Formation risk assessors based on the results of data validation and overall comparison to DQOs. Formation will determine whether data is usable for risk assessment based on the specifications in USEPA guidance for risk assessments (USEPA, 1992).

2.4.6 Measurement Data Analysis and Reporting

Measurement data will be reported in consistent units for each sample matrix to maintain comparability and facilitate data analyses. Concentrations in liquid samples shall be expressed in terms of weight per unit volume such as milligram per liter (mg/L). Concentrations of solid matrices shall be expressed in terms of weight per unit weight with the moisture basis specified such as milligram per kilogram (e.g., mg/kg wet wt). In addition, soil, vegetation, fish tissue whole-body, and sediment concentrations shall be converted to a dry-weight basis using the percent solids or percent moisture of the sample. The number of significant figures in the field and laboratory data presented in the final report shall be consistent with the limits of uncertainty inherent in the measurement or analytical method.

Statistical analyses and other evaluations may be performed that consider the validated data set, as well as the original detected values for parameters with results below the minimum reporting limit. Statistical methods may include published methods found in statistical handbooks, textbooks, and USEPA or other agency statistical guidance documents.

2.5 Assessment and Oversight

Assessments of data collection and reporting activities are designed to verify that sampling and analyses are performed in accordance with the procedures established in the FSP and QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits. Internal audits will be performed by Simplot, Formation, or a contracted laboratory. External audits will be performed by the Lead Agency or supporting agencies. Procedures used to conduct internal and external audits shall be consistent with those described in *USEPA Guidance on Technical Audits and Related Assessments* (EPA QA/G-7; USEPA, 2000).

Performance and systems audits of field and laboratory data collection and reporting procedures are described in this section. Data assessments, such as data verification and validation, were presented in Section 2.4.

2.5.1 Field Performance and System Audits

At a minimum, Formation's QA Manager, or designee, shall conduct an onsite systems and performance audit of field sampling practices during the first two weeks of field data collection activities. Any non-conformances observed in the audit shall be documented and resolved (see Section 2.4.3). Additional systems audits or surveillance may be conducted during the remaining field investigations conducted for the RI/FS at the discretion of the Formation Project Representative or Formation QA Manager. At least one field audit per field season is recommended.

2.5.1.1 Internal Field Audits

Internal audits of field activities including sampling and field measurements, will be conducted by the Formation QA Manager, or designee. These audits will verify that procedures established in the FSP and QAPP, including referenced SOPs, are being followed.

The internal field audits (systems and performance audits) will include examination of field measurement and sampling records and field instrument operating records; sample collection, handling, decontamination (Section 3.2 of the FSP), and packaging activities; and documentation of sampling activities in compliance with the established procedures for each field activity audited. Follow-up audits may be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the investigation. The results of field audits will be documented. The completed field audit report will be kept on file by the Formation QA Manager. After a field audit is conducted, the results of the audit will be shared by the auditor with the field teams prior to additional sampling to enhance sampling performance where applicable.

Findings of these audits will be summarized in an audit report that is given to the Formation Project Representative, Field Investigations Manager, and appropriate Field Supervisor in charge of the audited activities. The audited party will submit a reply addressing each finding cited in the report, the corrective action (if necessary) to be taken, and a schedule for implementation. The Field Investigations Manager is responsible for ensuring that corrective actions are taken.

2.5.1.2 External Field Audits

External field audits may be conducted by representatives from the Agencies. External field audits may be conducted at any time during the field operations. These audits may or may not be announced and are at the discretion of the Agencies.

External field audits will be conducted according to the field activity information presented in the field SOPs or in the sampling procedures outlined in the FSP. Results of the external field audit may document the need for a change to procedures in the FSP and/or QAPP and result in the need for an amendment to the FSP and/or QAPP.

2.5.2 Laboratory Performance and Systems Audits

2.5.2.1 Internal Laboratory Audits

The internal laboratory audit will be conducted by the QA Officer at each laboratory utilized for the investigation. Audits will be performed in accordance with the laboratory's Quality Management Plan.

The internal laboratory system audits will be conducted on an annual basis while the internal lab performance audits will be conducted on a quarterly basis, or as specified in the laboratory's Quality Management Plan.

The internal laboratory system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc. The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The QA Officer from each laboratory utilized for this investigation will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance.

2.5.2.2 External Laboratory Audits

An external laboratory audit may be conducted by representatives from the Agencies at any time. An external laboratory audit may be conducted prior to the initiation of the sampling and analysis activities. These audits may or may not be announced, may be conducted at any time and are at the discretion of the Agencies.

External laboratory audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis. Typically, the external laboratory audit will be conducted in the lab

so that the staff may be questioned regarding laboratory procedure. A recently produced sample data package will be compared with their SOP to ensure compliance with applicable standards.

2.5.3 Corrective Actions

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out-of-QC performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment.

Nonconforming equipment, items, activities, conditions and unusual incidents that could affect data quality and attainment of the project's quality objectives will be identified, controlled and reported in a timely manner. For the purpose of this QAPP, a nonconformance is defined as a malfunction, failure, deficiency, or deviation that renders the quality of an item unacceptable or indeterminate in meeting the project's quality objectives.

Corrective action in the laboratory may occur prior to, during and after initial analyses. If the analytical results from laboratory QC samples fall outside of the measurement performance criteria, the laboratory should initiate corrective actions immediately. If the laboratory cannot correct the situation that caused the nonconformance and an out-of-control situation continues to occur or is expected to occur, then the laboratory will immediately contact the Formation QA Manager and request instructions regarding how to proceed with sample analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the Laboratory QA Manager to approve the implementation of corrective action. These conditions may include dilution of samples, additional sample extract cleanup, automatic re-injection/reanalysis when certain QC criteria are not met, etc.

Completion of any corrective action should be evidenced by data once again falling within prescribed measurement performance criteria. If an error in laboratory procedures or sample collection and handling procedures cannot be found, the results will be reviewed by the Formation QA Manager and Formation RI/FS Project Representative to assess whether reanalysis or re-sampling is required.

Any corrective actions taken will be documented in writing by either the Laboratory QA Manager or the Formation QA Manager and reported to the Formation RI/FS Project Representative. Corrective action records will be included in the program's quality records.

2.5.4 Corrective Action during Data Validation and Data Assessment

The Formation QA Manager may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include re-sampling by the field team, reanalysis of samples by the laboratory, or re-submission of data packages with corrected clerical errors. The appropriate and feasible corrective actions are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded, etc.). Corrective actions of this type will be documented by the Formation QA Manager.

2.5.5 Quality Assurance Reports to Management

The deliverables associated with the tasks identified in the RI/FS will contain QA discussions in which data quality information collected during the task is summarized. Those reports will be the responsibility of the Formation RI/FS Project Representative and QA Manager.

The QA discussions will contain, on a routine basis, the results of field and laboratory audits, information generated on the achievement of specific DQOs and a summary of any corrective actions that were implemented and their immediate results on the project. Detailed references to any QAPP modifications will also be highlighted.

2.6 QAPP References

Formation Environmental LLC, 2011. Final (Rev03) Remedial Investigation/Feasibility Study Work Plan, Smoky Canyon Mine Site, prepared for J.R. Simplot Company. May.

USEPA, 1992. Guidance for Data Useability in Risk Assessment, Publication 9285.7-09A, Office of Emergency and Remedial Response, PB92-963356.

USEPA, 2000. Guidance on Technical Audits and Related Assessments, EPA QA/G-7.

USEPA, 2002. Guidance for Quality Assurance Project Plans EPA QA/G-5. EPA 240-R-02-009. December.

USEPA, 2004. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. EPA 540-R-04-004. October.

USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G-4, EPA/240/B-06/001. February.

USEPA, 2009. USEPA Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use. EPA 540-R-08-005. January.

QUALITY ASSURANCE PROJECT PLAN

TABLES

Table 1-2. Smoky RI/FS Preliminary List of RI COPCs and Analytical Parameters

Groundwater	Surface Water	Sediment and Soil	Vegetation/ Biota
Field Measurements			
Groundwater Elevation	Discharge		
Temperature	Temperature		
pH	pH		
Specific conductance	Specific conductance		
Dissolved oxygen	Dissolved oxygen		
Turbidity	Turbidity		
ORP	ORP		
Ferrous Iron			
Ferric Iron			
Metals (Total and Dissolved for Aqueous and Total for Solids)			
Aluminum	Aluminum	Aluminum	Aluminum
Antimony	Antimony	Antimony	Antimony
Arsenic	Arsenic	Arsenic	Arsenic
Barium	Barium	Barium	Barium
Beryllium	Beryllium	Beryllium	Beryllium
Boron	Boron	Boron	Boron
Cadmium	Cadmium	Cadmium	Cadmium
Chromium	Chromium	Chromium	Chromium
	Chromium III/VI		
Cobalt	Cobalt	Cobalt	Cobalt
Copper	Copper	Copper	Copper
Iron	Iron	Iron	Iron
Lead	Lead	Lead	Lead
Manganese	Manganese	Manganese	Manganese
Mercury	Mercury	Mercury	Mercury
Molybdenum	Molybdenum	Molybdenum	Molybdenum
Nickel	Nickel	Nickel	Nickel
Selenium, total recoverable	Selenium, total recoverable	Selenium	Selenium
Silver	Silver	Silver	Silver
Thallium	Thallium	Thallium	Thallium
Uranium	Uranium	Uranium	Uranium
Vanadium	Vanadium	Vanadium	Vanadium
Zinc	Zinc	Zinc	Zinc
Other Analyses			
Chloride	Hardness		
Nitrate/Nitrite as N	Nitrate/Nitrite as N		
Sulfate			
Total Dissolved Soids	Total Dissolved Solids		
Total Organic Carbon			
Total Suspended Solids	Total Suspended Solids		

TABLE 1-3

SMOKY CANYON MINE RI/FS

LIST OF STANDARD OPERATING PROCEDURES FOR THE RI

SOP Number	Rev. No.	Title
JRS SOP No. 1	3	Field Documentation
JRS SOP No. 2	2	Sample Custody, Packaging, and Shipment
JRS SOP No. 3	3	Storage and Disposal of Soil, Drilling Fluids, and Water Generated During Field Work
JRS SOP No. 4	3	Groundwater Sampling and Water Level Measurements at Monitoring Wells and Piezometers
JRS SOP No. 5	2	Water Quality Sampling
JRS SOP No. 6	4	Surface Water Discharge Measurement
JRS SOP No. 7	3	Equipment Decontamination
JRS SOP No. 8	5	Collection of Fish Tissue for Chemical Analysis
JRS SOP No. 10	7	For Conducting Electrofishing Surveys
JRS SOP No. 11	4	Vegetation Cover and Community Measurement
JRS SOP No. 14	1	Sediment Sampling for Chemical Analysis
JRS SOP No. 15	0	Benthic Macroinvertebrate Tissue and Community Sampling
JRS SOP No. 25	1	Terrestrial Invertebrate Tissue Sampling
JRS SOP No. 26	1	Small Mammal Sampling
JRS SOP No. 27	1	Soil Sampling for Inorganic Compounds

TABLE 1-3

SMOKY CANYON MINE RI/FS

LIST OF STANDARD OPERATING PROCEDURES FOR THE RI (continued)

SOP Number	Rev. No.	Title
JRS SOP No. 28	0	Terrestrial Vegetation Sampling
MFG SOP No. 4JRS	1	Supervision of Exploratory Borings
MFG SOP No. 13	1	Field Measurement of Oxidation-Reduction Potential (ORP)
MFG SOP No. 17	1	Field Measurement of Dissolved Oxygen (DO)
NewFields SOP No. 7	2	Monitoring Well Development
NewFields SOP No. 23	2	Installation of Deep Bedrock Monitoring Wells and Piezometers

Table 2-1

Precision, Accuracy and Completeness Calculation Equations

Characteristic	Formula	Symbols
Precision (as relative percent difference, RPD)	$RPD = \frac{\lvert x_i - x_j \rvert}{\left(\frac{x_i + x_j}{2}\right)} \times 100$	x_i, x_j: replicate values of x
Precision (as relative standard deviation, RSD, otherwise known as coefficient of variation)	$RSD = \frac{s}{\bar{x}} \times 100$	s: sample standard deviation x: sample mean
Accuracy (as percent recovery, R, for samples without a background level of the analyte, such as reference materials, laboratory control samples, and performance evaluation samples)	$R = \frac{x}{t} \times 100$	x: sample value t: true or assumed value
Accuracy (as percent recovery, R, for measurements in which a known amount of analyte, a spike, is added to an environmental sample)	$R = \frac{x_s - x}{t} \times 100$	x_s: value of spiked sample x: value of unspiked sample t: true or assumed value
Completeness (as a percentage, C)	$C = \frac{n}{N} \times 100$	n: number of valid data points produced N: total number of samples taken

Table 2-2

Summary of Calibration and QC Procedures for EPA Method 6020 (ICPMS)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
MS tuning sample	Prior to initial calibration, solution as specified by lab's SOP.	Mass calibration < 0.1 amu from the true value; r ≥ 0.995. Stability: RSD < 5% for at least five replicate analyses.	Retune instrument then reanalyzing tuning solution.	Per Section II of ICP-MS NFG, except substitute with method/lab acceptance limits.	Per Section II and Table 11 in NFG, using professional judgment.
Initial calibration (ICAL) for all target analytes (minimum one standard and a blank)	Daily initial calibration prior to sample analysis	Calibration blank plus 1 or more non-zero standards; a minimum of 3 replicate integrations are required and the average shall be used.	Correct problem then repeat initial calibration.	Per Section III of ICP-MS NFG.	Per Section III and Table 13 in NFG
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for calibration and from a second source)	All analytes within ± 10% of expected value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Per Section III of ICP-MS NFG.	Per Section III and Table 13 in NFG
Initial Calibration Blank (ICB)	After ICV	Absolute value ≤ 2x MDL for each analyte. If (2x MDL) > CRQL, then use the absolute value ≤ CRQL as the criteria instead.	Correct problem and reanalyze.	Per Section IV of ICPMS NFG, except U at detected value if result between MDL and CRQL.	Per Section IV and Table 14 in NFG, except U at detected value if result between MDL and CRQL.
CRQL Check Standard (CRI)	Daily, after ICAL, after every 20 samples and at end of each analysis run.	The analyte(s) within ± 30% of expected value for all analytes except Co, Mn and Zn (± 50%).	Correct problem then reanalyze.	Per Section III of ICP-MS NFG.	Per Section III and Table 13 in NFG
Interference Check Solution A & AB (ICS-A & ICS-AB)	At the beginning of an analytical run (not before the ICV) and immediately followed by a CCV/CCB.	ICS-A and ICS-AB: ±3xCRQL or ±20% (whichever is greater).	Correct problem and reanalyze.	Per Section V of ICP-MS NFG.	Per Section V and Table 15 in NFG
Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence (at a mid-calibration range concentration)	The analyte within ± 10% of expected value	Correct problem then repeat CCV and reanalyze all samples since last successful CCV.	Per Section III of ICP-MS NFG.	Per Section III and Table 13 in NFG
Continuing Calibration Blank (CCB)	Before beginning a sample run, after every 10 samples, and at end of the analytical sequence	Absolute value ≤ 2x MDL for each analyte. If (2x MDL) > CRQL, then use the absolute value ≤ CRQL as the criteria instead.	Correct problem then reanalyze calibration blank and previous 10 samples.	Per Section IV of ICPMS NFG, except U at detected value if result between MDL and CRQL.	Per Section IV and Table 14 in NFG, except U at detected value if result between MDL and CRQL.

Table 2-2

Summary of Calibration and QC Procedures for EPA Method 6020 (ICPMS)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Method Blank (or preparation blank)	One per analytical batch	Absolute value ≤ CRQL for each analyte	If absolute value is >CRQL all sample results (excluding field blanks) must be ≥10x the blank concentration. Otherwise, all samples associated with the blank and <10x blank concentration must be redigested and reanalyzed.	Per Section IV of ICPMS NFG, except U at detected value if result between MDL and CRQL.	Per Section IV and Table 14 in NFG, except U at detected value if result between MDL and CRQL.
Laboratory Control Sample (LCS) or Standard Reference Material (SRM) for all analytes	One LCS/SRM per analytical batch	Aqueous/Soil/Sed LCS: 80% - 120% or vendor-specified control limits (but not wider than 80-120% recovery). Tissue SRM: vendor-specified control limits.	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS/SRM and all samples in the preparation batch.	Per Section VI of ICP-MS NFG.	Per Section VI and Table 16 in NFG, using appropriate control limits
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 20 samples per matrix - not to be performed using a field blank. MSDs not required for tissues.	Laboratory-determined control limits (but not wider than 75-125% recovery and RPD < 20). MS/MSD recoveries are not applicable if the sample concentration (used for spiking) is >4x the spike concentration.	Flag associated sample results and perform post-digestion spike addition (not required if only MSD is outside limits).	Per Section VIII of ICP-MS NFG.	Per Section VIII and Table 18 in NFG.
Post-digestion spike addition	If MS/MSD fails	Recovery within laboratory-determined control limits (but not wider than 75-125%).	Perform dilution test.	Per Section VIII of ICP-MS NFG.	Per Section VIII and Table 18 in NFG.
Analytical duplicate sample	One duplicate per every 20 samples per matrix	RPD <20% if sample and duplicate concentrations ≥5xCRQL. If sample and/or duplicate concentration <5xCRQL the control limit will be a difference of ±CRQL.	Flag associated sample results.	Per Section VII of ICP-MS NFG for aqueous samples. For soil, sediment and tissue samples, control limits of RPD<35% (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL) may be used based on professional judgment.	Per Section VII and Table 17 in NFG for aqueous samples. For soil, sediment and tissue samples, control limits of RPD<35% (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL) may be used based on professional judgment.

Table 2-2

Summary of Calibration and QC Procedures for EPA Method 6020 (ICPMS)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Field duplicate sample	Per Table 3-11 of QAPP	Not applicable	Not applicable	Per Section XI of ICP-MS NFG.	NFGs do not provide specific guidelines. Project control limits will be RPD<20% for aqueous samples (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±CRQL (if sample and/or duplicate <5xCRQL). Project control limits will be RPD<50% for soil/sediment samples (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL). For field duplicates outside project control limits, J/UJ flag field duplicate pair results and use professional judgment regarding flagging other samples in SDG.
Split samples (fish samples only)	To be determined	Not applicable	Not applicable	Not applicable	An RPD<50% will be used as a project control limit. Use professional judgment for flagging (R/J/UJ).
Serial dilution (SD) test	One SD sample per every 20 samples	Fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50xMDL.	Flag associated sample results.	Per Section IX of ICP-MS NFG.	Per Section IX and Table 19 in NFG
Internal Standaards (IS')	Every sample; internal standards as specified by method and lab's SOP.	60% - 125% of intensity in the calibration blank.	Dilute by a factor of two and re-analyze. If IS recoveries still out, report undiluted results.	Per Section X of ICP-MS NFG, though specific internal standards that are used (and the number used) will be determined by the lab.	Per Section X and Table 20 in NFG
Concentrations between the MDL and CRQL	All samples	Not applicable	Flag as estimated value ("B" flag)	Not applicable	Not applicable

Note that specific QC procedures may vary based on the laboratory that performs the analyses.
[a] National Functional Guidelines (NFG) for Inorganic Data Review (USEPA, 2004).
[b] Refer to NFG for detailed evaluation protocols.
MDL - Method detection limit
CRQL - Contract Required Quantitation Limit. May be referred to as "PQL" - Practical Quantitation Limit or "RL" - Reporting Limit.
RPD - Relative percent difference

Table 2-3

Summary of Calibration and QC Procedures for EPA Method 6010B (ICP)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Initial calibration (ICAL) for all target analytes (minimum one standard and a blank)	Daily initial calibration prior to sample analysis	Calibration blank plus 1 or more non-zero standards	Correct problem then repeat initial cal bration.	Per Section II of ICP NFG.	Per Section II and Table 3 in NFG
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for calibration and from a second source)	All analytes within ± 10% of expected value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Per Section II of ICP NFG.	Per Section II and Table 3 in NFG
Initial Calibration Blank (ICB)	After ICV	Absolute value ≤ 2x MDL for each analyte. If (2x MDL) > CRQL, then use the absolute value ≤ CRQL as the criteria instead.	Correct problem and reanalyze.	Per Section III of ICP NFG, except U at detected value if result between MDL and CRQL.	Per Section III and Table 4 in NFG, except U at detected value if result between MDL and CRQL.
CRQL Check Standard (CRI)	Daily, after ICAL, after every 20 samples and at end of each analysis run.	The analyte(s) within ±30% of expected value except for Sb, Pb and Tl (± 50%).	Correct problem then reanalyze.	Per Section II of ICP NFG.	Per Section II and Table 3 in NFG
Interference Check Solution A & AB (ICS-A & ICS-AB)	At the beginning of an analytical run	ICS-A and ICS-AB: ±2xCRQL or ±20% (whichever is greater).	Correct problem and reanalyze ICS-A and ICS-AB.	Per Section IV of ICP NFG.	Per Section IV and Table 5 in NFG
Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence (at a mid-calibration range concentration)	The analyte within ± 10% of expected value	Correct problem then repeat CCV and reanalyze all samples since last successful CCV.	Per Section II of ICP NFG.	Per Section II and Table 3 in NFG
Continuing Calibration Blank (CCB)	Before beginning a sample run, after every 10 samples, and at end of the analytical sequence	Absolute value ≤ 2x MDL for each analyte. If (2x MDL) > CRQL, then use the absolute value ≤ CRQL as the criteria instead.	Correct problem then reanalyze cal bration blank and previous 10 samples.	Per Section III of ICP NFG, except U at detected value if result between MDL and CRQL.	Per Section III and Table 4 in NFG, except U at detected value if result between MDL and CRQL.
Method Blank (or preparation blank)	One per analytical batch	Absolute value ≤ CRQL for each analyte	If absolute value is >CRQL all sample results (excluding field blanks) must be ≥10x the blank concentration. Otherwise, all samples associated with the blank and <10x blank concentration must be redigested and reanalyzed.	Per Section III of ICP NFG, except U at detected value if result between MDL and CRQL.	Per Section III and Table 4 in NFG, except U at detected value if result between MDL and CRQL.

Table 2-3

Summary of Calibration and QC Procedures for EPA Method 6010B (ICP)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Laboratory Control Sample (LCS) or Standard Reference Material (SRM) for all analytes	One LCS/SRM per analytical batch	Aqueous/Soil/Sed LCS: 80-120% or vendor-specified control limits (but not wider than 80-120% recovery; except for aqueous Sb and Ag which may have other control limits). Tissue SRM: vendor-specified control limits.	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS/SRM and all samples in the preparation batch.	Per Section V of ICP NFG.	Per Section V and Table 6 in NFG.
Matrix Sp ke/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 20 samples per matrix - field blanks may not be used. MSDs not required for tissues.	Laboratory-determined control limits (but not wider than 75-125% recovery and RPD < 20). MS/MSD recoveries are not applicable if the sample concentration (used for spiking) is >4x the spike concentration.	Flag associated sample results and perform post-digestion spike addition.	Per Section VII of ICP NFG.	Per Section VII and Table 8 in NFG.
Post-digestion sp ke addition	If MS/MSD fails	Recovery within laboratory-determined control limits (but not wider than 75-125%).	Perform dilution test.	Per Section VII of ICP NFG.	Per Section VII and Table 8 in NFG.
Analytical duplicate sample	One duplicate per every 20 samples per matrix	RPD <20% if sample and duplicate concentrations ≥5xCRQL. If sample and/or duplicate concentration <5xCRQL the control limit will be a difference of ±CRQL.	Flag associated sample results.	Per Section VI of ICP NFG for aqueous samples. For soil, sediment and tissue samples, control limits of RPD<35% (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL) may be used based on professional judgment.	Per Section VI and Table 7 in NFG for aqueous samples. For soil, sediment and tissue samples, control limits of RPD<35% (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL) may be used based on professional judgment.

Table 2-3

Summary of Calibration and QC Procedures for EPA Method 6010B (ICP)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Field duplicate sample	Per Table 3-11 of QAPP	Not applicable	Not applicable	Per Section IX of ICP NFG.	NFGs do not provide specific guidelines. Project control limits will be RPD<20% for aqueous samples (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±CRQL (if sample and/or duplicate <5xCRQL). Project control limits will be RPD<50% for soil/sediment samples (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL). For field duplicates outside project control limits, J/UJ flag field duplicate pair results and use professional judgment regarding flagging other samples in SDG.
Split samples (fish samples only)	To be determined	Not applicable	Not applicable	Not applicable	An RPD<50% will be used as a project control limit. Use professional judgment for flagging (R/J/UJ).
Serial dilution (SD) test	One SD sample per every 20 samples.	Fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50xMDL.	Flag associated sample results.	Per Section VIII of ICP NFG.	Per Section VIII and Table 9 in NFG.
Concentrations between the MDL and CRQL	All samples	Not applicable	Flag as estimated value ("B" flag)	Not applicable	Not applicable

Note that specific QC procedures may vary based on the laboratory that performs the analyses.
[a] National Functional Guidelines (NFG) for Inorganic Data Review (USEPA, 2004).
[b] Refer to NFG for detailed evaluation protocols.
CRQL - Contract Required Quantitation Limit. May be referred to as "PQL" - Practical Quantitation Limit or "RL" - Reporting Limit.
MDL - Method detection limit
RL - Reporting limit

Table 2-4

Summary of Calibration and QC Procedures for EPA Method 7470A/7471A (CVAA)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Initial cal bration (ICAL) for all target analytes (minimum five standards including a blank)	Daily initial calibration prior to sample analysis	Blank plus 4 or more calibration concentrations, correlation coefficient (r) ≥ 0.995	Correct problem then repeat initial calibration.	Per Section II of Mercury NFG.	Per Section II and Table 23 in NFG.
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for cal bration and from a second source)	All analytes within ± 20% of expected value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Per Section II of Mercury NFG.	Per Section II and Table 23 in NFG.
Initial Calibration Blank (ICB)	After ICV	Absolute value ≤ 2x MDL for each analyte. If (2x MDL) > CRQL, then use the absolute value ≤ CRQL as the criteria instead.	Correct problem and reanalyze.	Per Section III of Mercury NFG, except U at detected value if result between MDL and CRQL.	Per Section III and Table 24 in NFG, except U at detected value if result between MDL and CRQL.
CRQL Check Standard (CRI)	Daily, after ICAL, after every 20 samples and at end of each analysis run.	Within ± 30% of expected value.	Correct problem then reanalyze.	Per Section II of Mercury NFG.	Per Section II and Table 23 in NFG.
Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence (at a mid-calibration range concentration)	The analyte within ± 20% of expected value	Correct problem then repeat CCV and reanalyze all samples since last successful CCV.	Per Section II of Mercury NFG.	Per Section II and Table 23 in NFG.
Continuing Calibration Blank (CCB)	Before beginning a sample run, after every 10 samples, and at end of the analytical sequence	Absolute value ≤ 2x MDL for each analyte. If (2x MDL) > CRQL, then use the absolute value ≤ CRQL as the criteria instead.	Correct problem then reanalyze calibration blank and previous 10 samples.	Per Section III of Mercury NFG, except U at detected value if result between MDL and CRQL.	Per Section III and Table 24 in NFG, except U at detected value if result between MDL and CRQL.

Table 2-4

Summary of Calibration and QC Procedures for EPA Method 7470A/7471A (CVAA)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Method Blank (or preparation blank)	One per analytical batch	Absolute value ≤ CRQL	If absolute value is >CRQL all sample results (excluding field blanks) must be ≥10x the blank concentration. Otherwise, all samples associated with the blank and <10x blank concentration must be redigested and reanalyzed.	Per Section III of Mercury NFG, except U at detected value if result between MDL and CRQL.	Per Section III and Table 24 in NFG, except U at detected value if result between MDL and CRQL.
LCS/SRM	One LCS/SRM per analytical batch.	Aqueous LCS: 80-120% or vendor-specified or laboratory-determined control limits (but not wider than 80-120% recovery). Solid LCS/SRM: vendor-specified control limits.	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS/SRM and all samples in the preparation batch.	Per Section IV of Mercury NFG.	Per Section IV and Table 25 in NFG.
Matrix Spike/Matrix Sp ke Duplicate (MS/MSD)	One MS/MSD per every 20 samples per matrix - field blanks may not be used. MSDs not required for tissues.	Laboratory-determined control limits (but not wider than 75-125% recovery and RPD < 20). MS/MSD recoveries are not applicable if the sample concentration (used for spiking) is >4x the spike concentration.	Flag associated sample results and perform post-digestion spike.	Per Section VI of Mercury NFG.	Per Section VI and Table 27 in NFG.
Post-digestion sp ke addition (tissue only)	If MS/MSD fails	Recovery within laboratory-determined control limits (but not wider than 75-125%).	Perform dilution test.	Not applicable	Use professional judgment.

Table 2-4

Summary of Calibration and QC Procedures for EPA Method 7470A/7471A (CVAA)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Analytical duplicate sample	One duplicate per every 20 samples per matrix	RPD <20% if sample and duplicate concentrations ≥5xCRQL. If sample and/or duplicate concentration <5xCRQL the control limit will be a difference of ±CRQL.	Flag associated sample results.	Per Section V of Mercury NFG for aqueous samples. For soil, sediment and tissue samples, control limits of RPD<35% (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL) may be used based on professional judgment.	Per Section V and Table 26 in NFG for aqueous samples. For soil, sediment and tissue samples, control limits of RPD<35% (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL) may be used based on professional judgment.
Field duplicate sample	Per Table 3-11 of QAPP	Not applicable	Not applicable	Per Section VII of Mercury NFG.	NFGs do not provide specific guidelines. Project control limits will be RPD<20% for aqueous samples (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±CRQL (if sample and/or duplicate <5xCRQL). Project control limits will be RPD<50% for soil/sediment samples (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL). For field duplicates outside project control limits, J/UJ flag field duplicate pair results and use professional judgment regarding flagging other samples in SDG.
Split samples (fish samples only)	To be determined	Not applicable	Not applicable	Not applicable	An RPD<50% will be used as a project control limit. Use professional judgment for flagging (R/J/UJ).

Table 2-4

Summary of Calibration and QC Procedures for EPA Method 7470A/7471A (CVAA)

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section [a]	Data Validation Qualification [b]
Serial dilution (SD) test (tissue only)	One SD sample per every 20 samples.	Fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50xMDL.	Flag associated sample results.	Not applicable	NFGs do not provide specific guidelines. If difference >10%, flag all detected results as estimated (J).
Concentrations between the MDL and RL	All samples	Not applicable	Flag as estimated value ("B" flag)	Not applicable	Not applicable

Note that specific QC procedures may vary based on the laboratory that performs the analyses.

[a] National Functional Guidelines (NFG) for Inorganic Data Review (USEPA, 2004).
[b] Refer to NFG for detailed evaluation protocols.
CRQL - Contract Required Quantitation Limit. May be referred to as "PQL" - Practical Quantitation Limit or "RL" - Reporting Limit.

MDL - Method detection limit
RL - Reporting limit

Table 2-5

Summary of Calibration and QC Procedures for EPA Method 7742 and SM3114C

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Procedure	Data Validation Qualification
Initial calibration (ICAL) for all target analytes	Daily initial calibration prior to sample analysis	5 or more calibration concentrations including a blank, correlation coefficient (r) ≥ 0.995.	Correct problem then repeat initial cal bration.	Evaluate r against control limits.	Any problems must be corrected. Samples may not be analyzed without a valid ICAL.
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for calibration and from a second source)	All analytes within ± 10% of expected value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Evaluate percent recovery.	Any problems must be corrected. Samples may not be analyzed wuntil the cal bration has been verified.
Initial Calibration Blank (ICB)	After ICV	Absolute value ≤ 2x MDL for each analyte. If (2x MDL) > CRQL, then use the absolute value ≤ CRQL as the criteria instead.	Correct problem and reanalyze.	Evaluate blank result against criterion and compare to sample concentrations.	Use Table 24 in NFG as guidance, except U at detected value if sample result between MDL and CRQL
Low-level cal bration check standard	Daily, after ICAL (at a concentration ≤ CRQL)	The analyte within ± 30% of expected value	Correct problem and reanalyze.	Evaluate percent recovery.	%R <70% = UJ (non-detects) and J (detects). %R > 130% = J (detects).
Continuing Cal bration Verification (CCV)	After every 10 samples and at the end of the analysis sequence (at a mid-calibration range concentration)	The analyte within ± 10% of expected value	Correct problem then repeat CCV and reanalyze all samples since last successful CCV.	Evaluate percent recovery.	%R < 90% = UJ (non-detects) and J- (detects). %R > 110% = J+ (detects).
Continuing Cal bration Blank (CCB)	Before beginning a sample run, after every 10 samples, and at end of the analytical sequence	Absolute value ≤ 2x MDL for each analyte. If (2x MDL) > CRQL, then use the absolute value ≤ CRQL as the criteria instead.	Correct problem then reanalyze calibration blank and previous 10 samples.	Evaluate blank result against criterion and compare to sample concentrations.	Use Table 24 in NFG as guidance, except U at detected value if sample result between MDL and CRQL
Method Blank (or preparation blank)	One per analytical batch	Absolute value ≤ CRQL	Correct problem. Corrective actions determined by lab which may including system cleaning, re-extraction and reanalysis.	Evaluate blank result against criterion and compare to sample concentrations.	Use Table 24 in NFG as guidance, except U at detected value if sample result between MDL and CRQL

Table 2-5

Summary of Calibration and QC Procedures for EPA Method 7742 and SM3114C

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Procedure	Data Validation Qualification
Laboratory Control Sample (LCS) or Standard Reference Material (SRM)	One LCS/SRM per analytical batch	Aqueous/Soil/Sed LCS: 85-115% or vendor-specified control limits (but not wider than 85-115% recovery). Tissue SRM: vendor-specified control limits.	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS/SRM and all samples in the preparation batch.	Evaluate %R against control limits.	%R < control limit = UJ (non-detects) and J- (detects). %R > control limit = no flag (non-detects) and J+ (detects). Use professional judgment for LCS/SRM recoveries that are well outside of control limits - R flags may be necessary.
Analytical spikes; Method of Standard Addition (MSA) - method 7742 only	One analytical spike per sample	Recovery >40%	Correct problem and reanalyze.	Verify analytical spikes were analyzed and recoveries > 40%.	Use professional judgment; possibly requesting reanalysis of sample.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 20 samples per matrix - field blanks may not be used. MSDs are not required for tissues.	Laboratory-determined control limits (but not wider than 75-125% and RPD < 20). MS/MSD recoveries are not applicable if the sample concentration (used for spiking) is >4x the spike concentration.	Flag associated sample results and perform post-digestion spike (method 3114C only). Post-digestion spikes not applicable to method 7742 since MSA used.	Evaluate %R against control limits.	%R < 30% = R (non-detects) and J- (detects). %R >30% but < control limit = UJ (non-detects) and J- (detects). %R > control limit = no flag (non-detects) and J+ (detects).
Post-digestion spike addition	If MS/MSD fails (method 3114C only)	Recovery within laboratory-determined control limits (but not wider than 75-125%).	Perform dilution test.	Evaluate %R against control limits.	See dilution test.
Analytical duplicate sample	One duplicate per every 20 samples per matrix	RPD <20% (aqueous samples) or < 30% (soils/tissues) if sample and duplicate concentrations ≥5xCRQL. If sample and/or duplicate concentration <5xCRQL the control limit will be a difference of ±CRQL.	Flag associated sample results.	Evaluate RPD against control limits. For validation purposes for soils/sediment/tissues, control limits of RPD<35% (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL) may be used based on professional judgment.	RPD > control limit or ±CRQL criteria exceeded = UJ (non-detects) and J (detects).

Table 2-5

Summary of Calibration and QC Procedures for EPA Method 7742 and SM3114C

Quality Control Check	Minimum Frequency	Lab Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Procedure	Data Validation Qualification
Field duplicate sample	Per Table 3-11 of QAPP	Not applicable	Not applicable	Evaluate RPD against project control limits. Project control limits will be RPD<20% for aqueous samples (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±CRQL (if sample and/or duplicate <5xCRQL). Project control limits will be RPD<50% for soil/sediment samples (if sample and duplicate concentrations are ≥5xCRQL) or a difference of ±2xCRQL (if sample and/or duplicate <5xCRQL).	For field duplicates outside project control limits, J/UJ flag field duplicate pair results and use professional judgment regarding flagging other samples in SDG.
Split samples (fish samples only)	To be determined	Not applicable	Not applicable	Evaluate %RPD.	An RPD<50% will be used as a project control limit. Use professional judgment for flagging (R/J/UJ).
Serial dilution (SD) test	One SD sample per every 20 samples (method 3114C only). SDs not applicable to method 7742 since MSA used.	Fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50xMDL.	Flag associated sample results.	Evaluate % difference.	If difference >10%, flag all detected results as estimated (J).
MDL Study	At initail set-up and subsequently once per 12-month periof or quarterly MDL verification checks.	Detection limits established shall be ≤ 1/2 the RL in 40CFR, Part 136 Appendix B.	Run MDL verification check at higher level and set higher MDL or re-conduct MDL study.	Not applicable	Not applicable
Concentrations between the MDL and RL	All samples	Not applicable	Flag as estimated value ("B" flag)	Not applicable	Not applicable

Note that specific QC procedures may vary based on the laboratory that performs the analyses.
CRQL - Contract Required Quantitation Limit. May be referred to as "PQL" - Practical Quantitation Limit or "RL" - Reporting Limit.
MDL - Method detection limit

Table 2-6

Summary of RI/FS Sampling Activities

Sampling Task	Sample Type	Planned Number of Primary Samples
Groundwater Investigations (see Table 2-1)	Groundwater from monitoring wells	24-40 (samples specific to RI, other monitoring programs are ongoing)
	Groundwater collected at springs.	26 samples
Surface Water Investigations (see Table 2-4)	Surface water from streams and seeps	90-120
		(50 locations, plus additional seeps or springs as identified, sampled two times a year for one year)
	Surface water from detention basins	52 samples poss ble, less than 10 samples anticipated
Sediment Investigations (see Table 2-4)	Sediment from streams and springs	18 stream sediment samples
	Sediment from detention ponds	52 detention pond sediment samples
Soil/Overburden Investigations (see Tables 2-5 and 2-8)	Soil/overburden disposal areas	70 surface soil samples
	Soil from Sage Valley	10+ surface soil samples
	Soil from seep areas	3 surface soil samples
	Solids from riparian areas	6 surface soil samples
Vegetation Investigations (see Tables 2-5 and 2-8)	Vegetation from overburden disposal areas	85 composite vegetation samples, 10 samples each of forbs, grasses, and woody species composites
	Vegetation from Sage Valley	10+ composite vegetation samples, at least 1 sample of up to 4 culturally significant plant species
	Vegetation from seep areas	3 composite vegetation samples, 3-6 samples of up to 4 culturally significant plant species
Baseline Eco Risk Investigations (see Tables 2-5 and 2-8)	Riparian vegetation	15 composite vegetation samples
	Terrestrial invertebrates and small mammals (ODAs, seeps, riparian)	48 invertebrate samples
		48 small mammal samples
	Fish	fish samples from 16 locations
	Benthic macroinvertebrates	benthic macroinvertebrate samples from 16 locations

Table 2-7 (QAPP Addendum 01, May 2011)

Requirements for Sample Preservation and Preparation Techniques, Sample Volumes, and Holding Times

Parameter	Referenced Method	Sample Preparation Method	Preservative[1]	Minimum Sample Volume	Maximum Holding Time (Days)
Solid Matrices (Sediment, Soil, Vegetation, Biological Tissue)					
Metals and metalloids (COPCs)	EPA 6010B and EPA 6020 (ICP and ICP-MS)	Total Digestion-hot plate (M3050B) for sediment, soil and vegetation; closed vessel digestion for non-vegetation tissues	None	5 g	180
Mercury	EPA 7471A	Preparation per method 7471A	None	5 g	28
Selenium	EPA 7742 Modified or SM 3114C, AA-Hydride	Total Digestion-methods 3050B and SM 3114B for sediment and soil; method 3050B for vegetation; closed vessel digestion for non-vegetation tissues	None	5 g	180
pH	USDA No. 60 [21A]	Saturated Paste (USDA No. 60[2])	None	20 g	ASAP
Percent Solids for Soil, Sediment, and Fish	CLPSOW390, Part F, D-98, Freeze-Dry (CAS SOP/NOAA Status & Trends)	None	None	10 g	NA
Total Organic Carbon	ASTM D4129 (modified)	None	None	20 g	28
Fish (and other aquatic tissue) Sample Preparation	EPA 600/4-81-055	Fish Tissue Pulverization	None	Entire sample	NA
Moisture Content for Plant Tissue	M209F, Gravimetric-60C	None	None	Entire sample	NA
Plant-Tissue Sample Preparation	Homogenization (CAS SOP)	Plant Tissue Pulverization	None	Entire sample	NA
Sediment Sample Preparation	USDA No.1 ASA No.9 15-4.2.2; ASTM D422	Air Dry at 34°C and Sieve-2.0mm	None	Entire sample	NA
Water Matrices (Surface Water, Groundwater)					
Metals and metalloids (COPCs), Hardness	EPA 6010B and 6020 (ICP and ICP-MS)	Field filtered (dissolved), Hot Plate Digestion 3005A (6010B) or 3020A (6020)	HNO_3	500 mL	180
		Unfiltered (total); Hot Plate Digestion 3005A (6010B) or 3020A (6020)	HNO_3	500 mL	180
Chromium VI (surface water only)[3]	EPA 7199[3]	Field filtered (dissolved); extract per method 7199	None	50 mL	1 (24 hours)[3]
Mercury	EPA 7470A	Field filtered (dissolved), preparation per method 7470A	HNO3	50 mL	28
		Unfiltered (total); preparation per method 7470A	HNO3	50 mL	28
Major cations (Ca, Mg, K, Na)	EPA 6010B	Field filtered (dissolved), Hot Plate Digestion 3005A	HNO_3	250 mL	180
Chloride, Sulfate	EPA 300.0 (Ion Chromatography)	None	None	50 mL	28
Nitrate+nitrite, as N	EPA 353.2	None	H_2SO_4	50 mL	28
Alkalinity (Alkalinity, Bicarbonate Carbonate, Hydroxide), TDS, TSS	SM2320B (Titration), SM 2540C (TDS), SM 2540D (TSS)	None	None	100 mL	14 (Alkalinity) 7 (TDS, TSS)
Total Organic Carbon	SM 5310B	None	H_2SO_4	50 mL	28
Selenium[2]	SM3114C (AA-Hydride)[2]	Field filtered (dissolved), Digestion per method SM3114B	HNO_3	25 mL	180
		Unfiltered (total); Digestion per method SM3114B	HNO_3	25 mL	180

[1]In addition to the preservation listed, all samples shall be maintained at 4 ° ± 2°C or frozen (biological tissues) following collection and during shipment to the lab.

[2]Specific parameters are media or sample type specific, see appropriate table in Field Sampling Plan.

[3]Chromium speciation will only be run if a previous result for total chromium was ≥0.011 mg/L.

Table 2-8

Achievable Laboratory Limits, Regulatory Standards, and Risk-Based Screening Values
Surface Water and Groundwater Analysis Parameters

Analysis Method	Monitoring Parameter [1]	Units	Achievable Laboratory Limits [2]		State of Idaho Standards				EPA MCL[6]		EPA Regional SL[7]	Health Comparison Values of Drinking Water[8]		National Recommended Water Quality Criteria - Aquatic Life[9, p]		Aquatic Life Secondary Values	
					Ground Water[3,s]	Surface Water [4]	Aquatic Life [5]										
			RL	MDL			Acute	Chronic	Primary	Secondary[s]	Tap Water	Child	Adult	Acute	Chronic	Chronic	Source
EPA 6020A	Antimony	mg/L	0.003	0 00022	0 006	0.0056	- -	- -	0 006	- -	0.0015	0 004	0.010	- -	- -	0 24	[10]USEPA 1986
	Arsenic	mg/L	0.003	0 0005	0.05	0 010	0 34[a]	0.15[a]	0 01	- -	0 000045[b]	0 003	0.010	0.34	0.15	- -	
	Barium	mg/L	0.001	0 00003	2	- -	- -	- -	2	- -	0.73	- -	- -	- -	- -	0.44[N]	[11]MDEQ 2009 - FCV
	Beryllium	mg/L	0.0002	0 000029	0 004	- -	- -	- -	0.004	- -	0.0073	0.02	0.07	- -	- -	0.01[N]	[11]MDEQ 2009 - FCV
	Cadmium	mg/L	0.0002	0 000024	0 005	- -	0.0013[c]	0.0006[c]	0 005	- -	0.0018	0 002	0.007	0.0020[d]	0 00025[d]	- -	
	Chromium (total)	mg/L	0.0015	0 00023	0.1	- -	0 57[c,f]	0 074[c,f]	0.1	- -	5.5 [e]	0.1	0.1	0.57[d,f]	0 074[d,f]	- -	
	Cobalt	mg/L	0.001	0 000013	- -	- -	- -	- -	- -	- -	0.0011	0.1	0.4	- -	- -	0.10	[11]MDEQ 2009 - FCV
	Copper	mg/L	0.001	0 000073	1.3	- -	0.017[c]	0.011[c]	1.3 [v]	1 0	0.15	0.1	0.4	0 013[d]	0.0090[d]	- -	
	Lead	mg/L	0.003	0 000053	0 015	- -	0.065[c]	0.0025[c]	0 015 [v]	- -	- -	- -	- -	0.065[d]	0.0025[d]	- -	
	Manganese	mg/L	0.001	0 000021	(0.05)	- -	- -	- -	- -	0.05	0 088	- -	- -	- -	- -	1 65[N,p]	[12]CDPHE 2009
	Molybdenum	mg/L	0.001	0 0001	- -	- -	- -	- -	- -	- -	0 018	0.05	0 2	- -	- -	3.2[N]	[11]MDEQ 2009 - FCV
	Nickel	mg/L	0.001	0 00011	- -	0 61	0.47[c]	0.052[c]	- -	- -	0 073	0 2	0.7	0.47[d]	0 052[d]	- -	
	Selenium	mg/L	0.003	0 00024	0.05	0.17	0.020[g]	0 005[f,g]	0.05	- -	0 018	0.05	0 2	notes[h,k]	0.0050[k]	- -	
	Silver	mg/L	0.0001	0 000019	(0.1)	- -	0.0034[c]	- -	- -	0.10	0 018	0.05	0 2	0.0032[d]	- -	- -	
	Thallium	mg/L	0.001	0 000023	0 002	0.00024	- -	- -	0.002	- -	- -	- -	- -	- -	- -	0 0072	[11]MDEQ 2009 - FCV
	Uranium	mg/L	0.001	0 0000081	- -	- -	- -	- -	0.03	- -	0 0022[r]	0.03	0.03	- -	- -	1.5[N,p]	[12]CDPHE 2009
	Vanadium	mg/L	0.0015	0 0003	- -	- -	- -	- -	- -	- -	0.018[i]	0.03	0.1	- -	- -	0.012	[11]MDEQ 2009 - FCV
	Zinc	mg/L	0.005	0 00048	(5)	7.4	0.12[c]	0.12[c]	- -	5	1.1	3	10	0.12[d]	0.12[d]	- -	
EPA 6010B	Aluminum	mg/L	0.1	0 05	(0.2)	- -	- -	- -	- -	0 05 - 0 2	3.7	20	70	- -	- -	0.087	[10]USEPA 1986
	Boron	mg/L	0.05	0 02	- -	- -	- -	- -	- -	- -	0.73	0.1	0.4	- -	- -	5 00	[11]MDEQ 2009 - FCV
	Calcium	mg/L	0.05	0 02	- -	- -	- -	- -	t	t	t	- -	- -	- -	- -	- -	
	Iron	mg/L	0.1	0 02	(0.3)	- -	- -	- -	- -	- -	2.6	- -	- -	- -	- -	1 00	[10]USEPA 1986
	Magnesium	mg/L	0.1	0 02	- -	- -	- -	- -	t	t	t	- -	- -	- -	- -	q	q
	Potassium	mg/L	0 5	0.1	- -	- -	- -	- -	t	t	t	- -	- -	- -	- -	q	q
	Sodium	mg/L	0 5	0 05	- -	- -	- -	- -	t	t	t	- -	- -	- -	- -	q	q
EPA 7470A	Mercury	mg/L	0 0002	0 00006	0 002	- -	- -	- -	0.002	- -	0 0011[l]	- -	- -	0 0014	0 00077	- -	
EPA 7199	Chromium VI	mg/L	0.001	0.0002	- -	- -	0.016[a]	0 011[a]	- -	- -	0.000043[b]	- -	- -	0.016	0.011	- -	
SM 3114C	Selenium	mg/L	0.002	0.0002	0.05	0.17	0.020[g]	0 005[g]	0.05	- -	0 018	- -	- -	notes[h,k]	0.0050[k]	- -	
EPA 300 0	Chloride	mg/L	0.2	0.1	(250)	- -	- -	- -	t	250	t	- -	- -	- -	- -	q	q
	Sulfate	mg/L	1	0.5	(250)	- -	- -	- -	- -	250	- -	- -	- -	- -	- -	q	q
EPA 353 2	Nitrate /Nitrite as N	mg/L	0.05	0.025	10 [10/1]	- -	- -	- -	10 [10/1] [m]	- -	[58/0.37] [m]	- -	- -	- -	- -	- -	
SM 2450C	TDS	mg/L	10	5	(500)	- -	- -	- -	- -	500	- -	- -	- -	- -	- -	1134[o]	[13]Chapman et al. 2000
SM 2540D	TSS	mg/L	5	2.5	- -	- -	- -	- -	- -	- -	- -	- -	- -	- -	- -	- -	

Table 2-8

Achievable Laboratory Limits, Regulatory Standards, and Risk-Based Screening Values
Surface Water and Groundwater Analysis Parameters

Notes:

[1] The project- or event-specific target parameter list will be established prior to the sampling event; samples may or may not be analyzed for all listed parameters.

[2] RLs and MDLs are subject to change based on the laboratory capabilities at the time of sample submittal.

[3] State of Idaho Ground Water Quality Rule (IDAPA 58.01.11); secondary standard in parentheses.

State of Idaho Surface Water Quality for Domestic Water Supply Use (IDAPA 58.01.02).

[5] State of Idaho Surface Water Quality for Aquatic Life (IDAPA 58.01.02); Acute Criteria (CMC) and Chronic Criteria (CCC).

[6] USEPA primary and secondary Maximum Contaminant Level (MCL), National Primary Drinking Water Regulations, EPA (http://www.epa.gov/safewater/contaminants/index.html); 9/11/2009.

[7] Residential tap water screening levels from USEPA, 2009a. Regional Screening Levels for Chemical Contaminants at Superfund Sites. Developed by Oak Ridge National Laboratory under an Interagency Agreement with EPA.

Available at http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm; updated December 2009. (Carcinogenic effect Target Risk = 10^{-6}, Non-carcinogenic effect Hazard Quotient = 0.1)

[8] Public Health Assessment: Southeast Idaho Phosphate Mining Resource Area: Bannock, Bear Lake, Bingham, and Caribou Counties, Idaho EPA Facility ID: IDN001002245 (U.S. Department of Health and Human Services, Public Health Services, Agency for Toxic Substances and Disease Registry, 2006). Non-cancer effects only.

[9] Freshwater standards from U.S. Environmental Protection Agency (USEPA). 2009. National Recommended Water Quality Criteria (NRWQC) for Priority Pollutants. EPA Office of Water, Office of Science and Technology (4304T). Available at http://www.epa.gov/waterscience/criteria/wqcriteria.html. Updated December 2, 2009; Acute Criteria (CMC) and Chronic Criteria (CCC)

[10] U.S. Environmental Protection Agency (EPA). 1986. Quality Criteria for Water 1986 ("The Gold Book"). EPA 440/5-86-001. May 1, 1986.

[11] Michigan Department of Environmental Quality (MDEQ). 2009. Freshwater Chronic Values (FCV) from Rule 57 Water Quality Values based on Rule 323.1057 (Toxic Substances) of the Part 4. Water Quality Standards gives procedures for calculating water quality values to protect humans, wildlife and aquatic life. http://www.michigan.gov/deq/0,1607,7-135-3313_3686_3728-11383--,00.html. Updated December 11, 2009.

[12] Colorado Department of Public Health and Environment (CDPHE) Water Quality Control Commission (WQCC). 2007. Reg. Number 32. Classifications and Numeric Standards for the Arkansas River System, updated February 2009. Available at http://www.cdphe.state.co.us/regulations/wqccregs/100232arkansasriverbasinnew.pdf.

[13] Chapman, P.M., H. Bailey, and E. Canaria. 2000. Toxicity of Total Dissolved Solids Associated with Two Mine Effluents to Chironomid Larvae and Early Lifestages of Rainbow Trout. Environmental Toxicology and Chemistry: Vol. 19, No. 1, pp. 210–214.

[a] Criteria for these metals are expressed as a function of the water effect ratio, WER, as defined in Subsection 210.03.c.iii of IDAPA 58.01.02

[b] Reporting limit and MDL are greater than screening value.

[c] Aquatic life criteria for these metals are expressed as a function of total hardness (mg/L as calcium carbonate), the pollutant's water effect ratio (WER) as defined in Subsection 210.03.c.iii of IDAPA 58.01.02 and multiplied by an appropriate dissolved conversion factor as defined in Subsection 210.02. For comparative purposes only, the values displayed in this table are shown as dissolved metal and correspond to a total hardness of one hundred (100) mg/L and a water effect ratio of one (1.0).

[d] The freshwater criterion for this metal is expressed as a function of hardness (mg/L) in the water column. The value given here corresponds to a hardness of 100 mg/L. Criteria values for other hardness may be calculated from the following:

CMC (dissolved)=$\exp\{m_A[\ln(\text{hardness})]+b_A\}$ (CF), or CCC (dissolved) = $\exp\{m_C[\ln(\text{hardness})]+b_C\}$ (CF).

[e] Value is for chromium III.

[f] Criterion is expressed as total recoverable (unfiltered) concentration.

[g] Selenium values are 0.005 mg/L for riparian habitat use, 0.050 mg/L for domestic animal drinking water use, and 0.201 mg/L for transitory wildlife drinking water.

[h] The CMC = 1/[(f1/CMC1)+(f2/CMC2)] where f1 and f2 are the fractions of total selenium that are treated as selenite and selenate, respectively, and CMC1 and CMC2 are 0.1859 mg/L and 0.01282 mg/L, respectively.

[i] Value is for vanadium and compounds

[k] This recommended water quality criterion for selenium is expressed in terms of total recoverable metal in the water column. It is scientifically acceptable to use the conversion factor (0.996- CMC or 0.922-CCC) that was used in the GLI (60FR15393-15399, March 23, 1995; 40CFR132 Appendix A) to convert this to a value that is expressed in terms of dissolved metal.

[l] Value is for inorganic mercury salts

[m] Values in brackets are the individual MCLs values for nitrate/nitrite. Nitrate+Nitrite RSL is based on the lesser of the Nitrate RSL and the Nitrite RSL.

[N] Hardness based threshold derived using 100 mg/L CaCO3

[o] No observed effects level of chironomids

[p] Metals are stated as dissolved unless otherwise specified.

[q] No criteria available, but considered as a component in total dissolved solids (TDS)

[r] Uranium RSL was recalculated using the oral reference dose of 0.0006 mg/kg/day as used by the USEPA Office of Drinking Water to develop the revised uranium MCL; USEPA, 2000. National Primary Drinking Water Regulations; Radionuclides; Final Rule. Federal Register, December 7, 2000 (Volume 65, Number 236), page 76707-76753.

[s] Secondary standards are non-enforceable guidelines regulating contaminants that may cause cosmetic effects (such as skin or tooth discoloration) or aesthetic effects (such as taste, odor, or color) in drinking water; these standards are not toxicity-based.

[t] Calcium, chloride, iodine, magnesium, phosphorus, potassium, and sodium are classified as non-toxic essential minerals and do not have RSL or MCLs. Chloride does have a secondary MCL.

[V] Copper and lead MCLs are action levels

CWA - Clean Water Act

IDAPA Idaho Administrative Protection Agency'

IDEQ - Idaho Department of Environmental Quality

mg/L - milligrams per liter

na - not applicable to this method

TDS - total dissolved solids

TOC- total organic carbon

TSS - total suspended solids

WER - water effect ratio

Table 2-9

Achievable Laboratory Limits and Risk-Based Screening Values
Soil and Sediment Parameters

Media Types	Laboratory Measurement	Method[1]	Reporting Limit (RL) (mg/Kg)	Method Detection Limit (MDL)[1] (mg/Kg)	Soil			Sediment					
					HH SOIL SCREENING LEVELS (mg/Kg)[2]	ECO SOIL SCREENING LEVELS (mg/Kg)[3]		SQAGS[4]	NOAA SQuiRTs[5]				PNEC[6]
					Residential	Avian	Mammalian	TEC	LELs	SELs	UETs	ARCS	
Soil/Sediment	Aluminum	6010B	8	1.8	7,700	NA	NA	NA	NA	NA	NA	25500	NA
	Antimony	6020	0.3	0.011	3.1	NA	0 27	NA	NA	NA	3	NA	NA
	Arsenic	6020	0.3	0.014	0.39	43	46	9 8	6	33	17	NA	NA
	Barium	6010B	1	0.2	1,500	NA	2000	20	NA	NA	NA	NA	NA
	Beryllium	6020	0.02	0 0034	16	NA	21	NA	NA	NA	NA	NA	NA
	Boron	6010B	4	0.48	1,600	NA	NA	NA	NA	NA	NA	NA	NA
	Cadmium	6020	0.02	0.006	7.0	0.77	0 36	1	0.6	10	3	NA	NA
	Chromium	6010B	1	0.2	12,000 [a]	26	34	43	26	110	95	NA	NA
	Cobalt	6010B	2	0.4	2.3	120	230	50	NA	NA	NA	NA	NA
	Copper	6010B	2	0.5	310	28	49	32	16	110	86	NA	NA
	Iron	6010B	6	1.5	5,500	NA	NA	NA	20000	40000	40000	NA	NA
	Lead	6010B	2	0.4	400 [b]	11	56	36	31	250	127	NA	NA
	Manganese	6010B	4	1	180	4,300	4,000	NA	460	1100	1100	NA	NA
	Mercury	7471A	0 033	0.006	2.4 [c]	NA	NA	0.18	0.2	2	0.56	NA	NA
	Molybdenum	6010B	1.5	0.5	39	NA	NA	NA	NA	NA	NA	NA	NA
	Nickel	6010B	2.5	0.5	160	210	130	23	16	75	43	NA	NA
	Selenium	6020*/7742	0.3	0.011	39	1.2	0 63	NA	NA	NA	NA	NA	NA
	Silver	6020	0.5	0 02	39	4.2	14	1	0.5	NA	4.5	NA	NA
	Thallium	6020	0.1	0 0015	NA	NA	NA	NA	NA	NA	NA	NA	NA
	Uranium	6020	0.1	0.00029	24 [d]	NA	NA	NA	NA	NA	NA	NA	100
	Vanadium	6020	0.15	0.025	39 [e]	7.8	280	NA	NA	NA	NA	NA	NA
	Zinc	6010B	5	1	2,400	46	79	NA	NA	NA	NA	NA	NA

Notes:

NA = No screening level available.
SQAGS - sediment quality assessment guidelines
SQuiRTs - screening quick reference tables
TEC - Threshold Effect Concentration
LELs- Lowest Effect Levels
SELs - Severe Effect Levels
UETs - Upper Effect Levels
ARCS - Assessment and Remediation of Contaminated Sediments
PNEC - Predicted No-Effect Concentration

[1] Specific methods, RLs, and MDLs are subject to change based on the laboratory capabilities at the time of sample submittal.

[2] Residential soil screening levels from USEPA, 2009a. Regional Screening Levels for Chemical Contaminants at Superfund Sites. Developed by Oak Ridge National Laboratory under an Interagency Agreement with EPA. Available at http://www.epa gov/reg3hwmd/risk/human/rb-concentration_table/index htm; updated December 2009. (Carcinogenic effect TR = 10^{-6}, Non-carcinogenic effect HQ = 0.1) RSL will be used both soils and sediments.

[3] Eco Soil Screening Levels from USEPA Eco SSL guidance serve as conservative estimates of minimum detection limits; U.S. Environmental Protection Agency (EPA). 2005. Guidance for Developing Ecological Soil Screening Levels (EcoSSLs) Office of Solid Waste and Emergency Response (OSWER), OSWER Directive 9285.7-55. Published November 2003, Revised November 2005 and subsequent contaminant-specific EcoSSL documents.

[4] MacDonald, D.D., C.G. Ingersoll, D E. Smorong, R.A. Lindskoog, G. Sloane, and T. Biernacki. 2003. Development and Evaluation of Numerical Sediment Quality Assessment Guidelines (SQAGs) for Florida Inland Waters. Florida Department of Environmental Protection, Tallahassee, FL.

[5] U.S. National Oceanic and Atmospheric Association (NOAA). 2008. Screening Quick Reference Tables (SQuiRTs). NOAA Office of Response and Restoration Division, NOAA OR&R Report 08-1, Seattle, WA. Available at http://response.restoration noaa gov/book_shelf/122_NEW-SQuiRTs.pdf.

[6] Sheppard, S.C., M.I. Sheppard, M.O. Gallerand, B. Sanipelli. 2005. Derivation of ecotoxicity thresholds for uranium. Journal of Environmental Radioactivity 79(1):55-83.

[a] Value is for chromium III

[b] Lead's soil screening concentration is based on blood lead modeling and not calculated the way other RSLs have been calculated. Therefore, the Lead RSL is used unmodified, as reported in USEPA (2010).

[c] Value is for mercury, inorganic salts

[d] RSL were recalculated using the oral reference dose of 0 0006 mg/kg/day as used by the EPA Office of Drinking Water to develop the revised uranium MCL (EPA 2000).

[e] Value is for vanadium and compounds

Table 2-10

Achievable Laboratory Limits and Risk-Based Screening Values
Plant and Animal Tissue

Media Types	Laboratory Measurement	Method[1]	Reporting Limit (RL) (mg/Kg)	Method Detection Limit (MDL)[1] (mg/Kg)	Target Detection Limits (=EcoSSLs/10) (mg/Kg)[2]	
					Avian	Mammalian
Terrestrial and Riparian Vegetation	Aluminum	6020	2	0.4	NA	NA
	Antimony	6020	0.05	0.02	NA	0.027
	Arsenic	6020	0.5	0.06	4.3	4.6
	Barium	6020	0.05	0.005	NA	200
	Beryllium	6020	0.02	0.003	NA	2.1
	Boron	6010B	2	0.4	NA	NA
	Cadmium	6020	0.02	0.004	0.077	0.036
	Chromium	6010B	0.5	0.2	2.6	3.4
	Cobalt	6020	0.02	0.003	12	23
	Copper	6020	0.1	0.08	2.8	4.9
	Iron	6010B	2	0.7	NA	NA
	Lead	6020	0.05	0.009	1.1	5.6
	Manganese	6020	0.05	0.03	430	400
	Mercury	7471A	0.02	0.002	NA	NA
	Molybdenum	6020	0.05	0.02	NA	NA
	Nickel	6020	0.2	0.03	21	13
	Selenium	7742	0.1	0.03	0.12	0.063
	Silver	6020	0.02	0.008	0.42	1.4
	Thallium	6020	0.02	0.003	NA	NA
	Uranium	6020	0.02	0.006	NA	NA
	Vanadium	6010B	1	0.3	0.78	28
	Zinc	6020	0.5	0.2	4.6	7.9
Terrestrial and Aquatic Receptor Tissue	Aluminum	6020	2	0.9	NA	NA
	Antimony	6020	0.05	0.02	NA	0.027
	Arsenic	6020	0.5	0.06	4.3	4.6
	Barium	6020	0.05	0.009	NA	200
	Beryllium	6020	0.02	0.004	NA	2.1
	Boron	6010B	1	0.2	NA	NA
	Cadmium	6020	0.02	0.005	0.077	0.036
	Chromium	6010B	0.2	0.08	2.6	3.4
	Cobalt	6020	0.02	0.002	12	23
	Copper	6020	0.1	0.03	2.8	4.9
	Iron	6010B	2	0.4	NA	NA
	Lead	6020	0.02	0.005	1.1	5.6
	Manganese	6020	0.05	0.02	430	400
	Mercury	7471A	0.02	0.002	NA	NA
	Molybdenum	6020	0.05	0.02	NA	NA
	Nickel	6020	0.2	0.02	21	13
	Selenium	7742	0.1	0.05	0.12	0.063
	Silver	6020	0.02	0.02	0.42	1.4
	Thallium	6020	0.02	0.002	NA	NA
	Uranium	6020	0.02	0.002	NA	NA
	Vanadium	6010B	0.2	0.07	0.78	28
	Zinc	6020	0.5	0.08	4.6	7.9

Notes:
[1] Specific methods, RLs, and MDLs are subject to change based on the laboratory capabilities at the time of sample submittal.

[2] Eco Soil Screening Levels from USEPA Eco SSL guidance serve as conservative estimates of minimum detection limits; U.S. Environmental Protection Agency (EPA). 2005. Guidance for Developing Ecological Soil Screening Levels (EcoSSLs). EPA Office of Solid Waste and Emergency Response (OSWER), OSWER Directive 9285.7-55. Published November 2003, Revised November 2005 and subsequent contaminant-specific EcoSSL documents.

NA = No screening level available.

Table 2-11

Required Frequencies for Field QC Samples

Sample Type	Method	Equipment Rinsate Blank	Field Ambient Blank	Field Duplicate
Surface Water Groundwater	6010B 6020 7470A SM3114C 300	1/20 samples or 1 per day if < 20 samples per day	1 / 20 samples if equipment is not re-used	1 / 20 samples Or At least 1 per day by sample type
Sediment Soil Tissue*	6010B 6020 7471A 7742 SM3114C	1/20 samples or 1 per day if < 20 samples	--	1 / 20 samples Or At least 1 per day by sample type

*Field duplicates are not required for vegetation and other tissue samples.

Table 2-12

Required Frequencies for Laboratory QC Analyses

Sample Type	Method	Method Blank	Laboratory Control Sample	Analytical Duplicate	Matrix Spike/Matrix Spike Duplicate Pair
Surface Water Groundwater	6010B 6020 7470A SM3114C	1/20	1/20	1 / 20 recommended	1 pair/ 20
Sediment Soil	6010B 6020 7471A SM3114C	1/20	1/20	1/20	1 pair/ 20
Tissue	6010B 6020 7471A 7742	1/20	1/20 use Standard Reference Material	1/20	1 MS/20

Table 2-13

EDD Specifications for the Laboratory

Lab EDD Fields	Description
COCSampleID	Field sample Identification number
SampleDate	Date sample collected
SampleTime	Time sample collected
PreparationMethod	Preparation method number
AnalyticalMethod	Analytical method number
Matrix	Sampling matrix
TorDAnalysis	Total or dissolved analysis (filtered or unfiltered sample)
Basis	(Wet/dry for biota samples)
Analyte	Parameter label
Result	Measured concentration
Units	Units of measure
DetLimit	Detection limit
DetLimitType	Detection limit type (e.g., MDL or IDL)
ReportingLimit	Reporting limit
LabQualifier	Parameter value qualifier
Dilution	Dilution factor
LabName	Lab name
SDGNumber	Lab Sample Delivery Group (SDG) number
LabSampleID	Lab sample identification number
ReceivedDate	Date sample received by laboratory
AnalysisDate	Data sample analyzed by laboratory
Laboratory QC identification number	ID for any lab QC analyses reported with EDD
CAS#	Compound name

EDD - Electronic data deliverable

IDL - Instrument detection limit

MDL - Method detection limit

SDG - Sample delivery group

Table 2-14

EDD Specifications for Data Validators

Validator EDD Fields	Description
SampleName	Field sample identification number
Date	Date sample collected
Time	Time sample collected
AnalyticalMethod	Analytical method number
OriginalMatrix	Sampling matrix
FiltStat	Filtered or unfiltered sample
Basis	(Wet/dry for biota samples)
OriginalAnalyte	Parameter label
OriginalValue	Measured concentration
OriginalUnit	Units of measure
DetLimit	Detection limit
DetLimitType	Detection limit type (e.g., MDL or IDL)
CRDL	Contract required detection limit
LabQualifier	Parameter value qualifier
Dilution	Dilution factor
LabName	Lab name
JobID	Lab sample delivery group (SDG) number
LabSampleID	Lab sample identification number
ReceivedDate	Date sample received by laboratory
AnalysisDate	Date sample analyzed by laboratory
AnalyteNotes	Compound name
USEPA Flag	USEPA validation qualifier
Reason Code	Reason for validation qualifier
Final Result	Final qualified result, with any adj for blank contamination
ValStatus	Validation status
ValPerson	Validator
ValProtocol	Validation protocol
ValNotes	Validation notes

IDL - Instrument detection limit

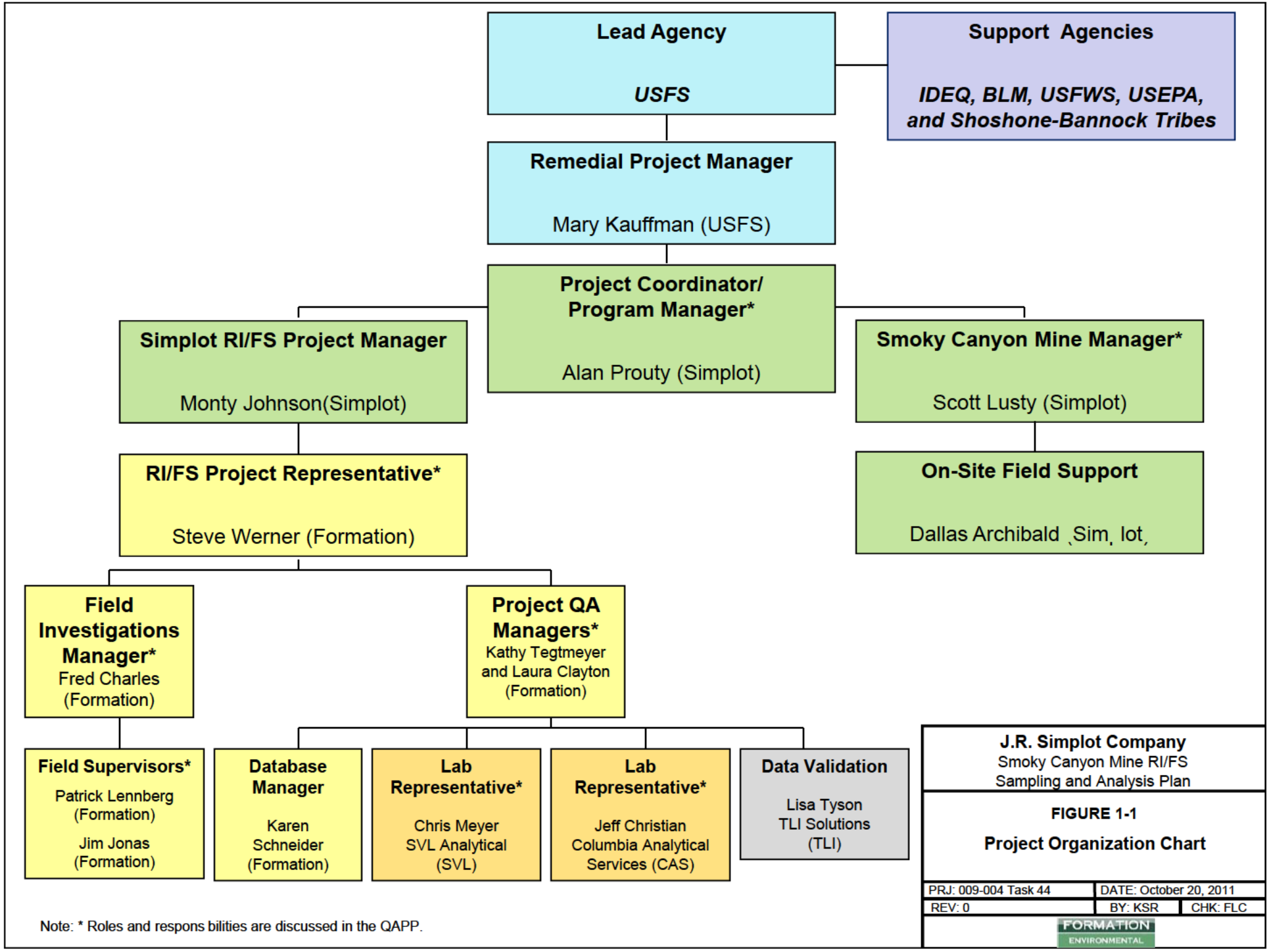
MDL - Method detection limit

SDG - Sample delivery group

USEPA - U.S. Environmental Protection Agency

QUALITY ASSURANCE PROJECT PLAN

FIGURES


Lead Agency
USFS
Support Agencies
IDEQ, BLM, USFWS, USEPA, and Shoshone-Bannock Tribes
Remedial Project Manager
Mary Kauffman (USFS)
Project Coordinator/ Program Manager*
Alan Prouty (Simplot)
Simplot RI/FS Project Manager
Monty Johnson(Simplot)
Smoky Canyon Mine Manager*
Scott Lusty (Simplot)
RI/FS Project Representative*
Steve Werner (Formation)
On-Site Field Support
Dallas Archibald ¸Sim¸ lot¸
Field Investigations Manager*
Fred Charles (Formation)
Project QA Managers*
Kathy Tegtmeyer and Laura Clayton (Formation)
Field Supervisors*
Patrick Lennberg (Formation)
Jim Jonas (Formation)
Database Manager
Karen Schneider (Formation)
Lab Representative*
Chris Meyer SVL Analytical (SVL)
Lab Representative*
Jeff Christian Columbia Analytical Services (CAS)
Data Validation
Lisa Tyson TLI Solutions (TLI)
Note: * Roles and respons bilities are discussed in the QAPP.
J.R. Simplot Company
Smoky Canyon Mine RI/FS
Sampling and Analysis Plan
FIGURE 1-1
Project Organization Chart
PRJ: 009-004 Task 44
DATE: October 20, 2011
REV: 0
BY: KSR
CHK: FLC
FORMATION ENVIRONMENTAL

SampleID: SC0510-LSV2C-FT013
Date/Time: /
Matrix: Tissue
Analysis: Total Metals, % Solids

Preservative: None Lab QC:
Depth: -

J.R. SIMPLOT COMPANY
SMOKY CANYON MINE RI/FS
SAMPLING AND ANALYSIS PLAN

FIGURE 2-1

EXAMPLE
SAMPLE LABEL

PRJ: 0442-004-900.44	DATE: OCT 7, 2009	
REV: 0	BY: KMS	CHK: KSR

FORMATION ENVIRONMENTAL, LLC

QUALITY ASSURANCE PROJECT PLAN (QAPP) ATTACHMENTS

ATTACHMENT 1
SVL Analytical Supporting Documents

Quality Assurance Management Plan

Quality Manual

SVL ANALYTICAL, INC.
P.O. Box 929
One Government Gulch
Kellogg, Idaho 83837
208-784-1258
FAX 208-783-0891

March 2008

President and CEO Wayne R. Sorensen	Date
Laboratory Director John R. Kern	Date
Technical Director Kirby L. Gray	Date
Quality and Systems Manager Brandan A. Borgias, Ph.D.	Date
Quality Assurance Coordinator Michael Desmarais	Date
Supervisor Inorganic Instrument Department Danny Sevy	Date
Supervisor Classical Chemistry Department James L. Hodge	Date

Additional Signatories

_____	_____
Client Services Manager G. Christine Meyer	Date
_____	_____
Deputy Technical Director Nan S. Wilson	Date
_____	_____
Deputy Technical Director Larry Drew, Ph.D.	Date

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1.0 QUALITY POLICY STATEMENT

SVL Analytical, Inc. (SVL) recognizes that an effective quality system is paramount to providing analytical data that is legally defensible, technically accurate, and scientifically meaningful.

The emphasis of SVL's Quality Manual (QM) is to define control procedures for receipt, handling, and storage of samples; preparation and storage of standards; calibration and maintenance of analytical equipment; performance of analytical methods; and the generation, review, and reporting of analytical data.

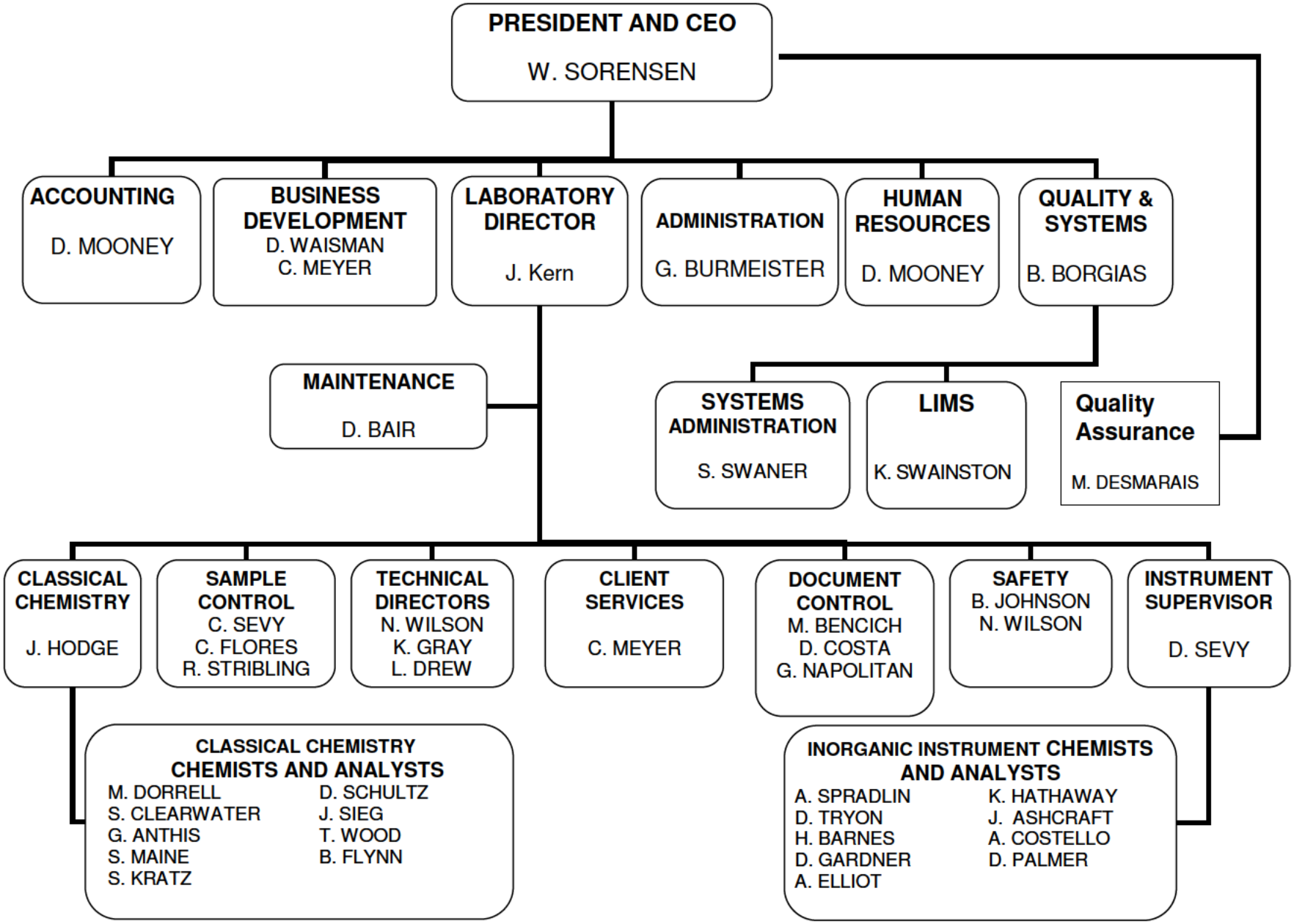
At SVL, quality assurance begins with the definition of Data Quality Objectives (DQO) and continues on through data reporting. Control procedures are defined for every step of the program as detailed in SVL's Standard Operating Procedures (SOPs). SVL realizes that without these controls in all phases of the analytical process, data become suspect and hence of less value to our clients. Therefore, SVL is committed to providing data of the highest quality, usability, and defensibility for every project undertaken.

SVL's Management ensures that this Quality Manual complies with all applicable NELAC Quality System Standards and sees that it is reviewed annually and revised as needed.

2.0 ORGANIZATION AND STRUCTURE

The organizational structure of SVL follows a traditional scheme of management with a few modifications. The President/CEO is at the top of the chain of command followed immediately by the Laboratory Director, Quality and Systems Manager, Business Development, Human Resources, Administration and Accounting. The Quality Assurance Coordinator reports directly to the President/CEO. Technical Directors, Client Services, Sample Control, Classical Chemistry Department, Inorganic Instrument Department, Safety, Document Control and Maintenance Department report to the Laboratory Director. Systems Administrator and LIMS Chemist report to the Quality and Systems Manager.

2.1 Organization Chart



2.2 Employee List

Position	Employee	Degree	Years of Lab Experience
President and CEO	Wayne Sorensen	BS 1962	41
Laboratory Director	John R. Kern	MS 1982	25
Business Development/Safety Officer	Blake Johnson	PhD 1971	23
Quality and Systems Manager	Brandan A. Borgias	PhD 1985	27
Document Control Officer	Melba Bencich		27
Client Services Manager	G. Christine Meyer		29
Business Development Manager	Dave Waisman	MS 1985	14
Technical Director	Kirby L. Gray	BS 1972	23
Deputy Technical Director/Safety Officer	Nan Wilson	BS 1996	12
Deputy Technical Director	Larry Drew	PhD 1973	5
Supervisor Inorganic Instrument	Danny Sevy		20
Supervisor Classical Chemistry	James L. Hodge		41
Systems Analyst	Scott Swaner		5
LIMS Chemist	Kale Swainston	BS 1998	5
Accounting and Human Resources	Donella Mooney		17
Quality Assurance Coordinator	Michael Desmarais	BS 1995	11
Ion Chromatography Analyst	Ann Costello	BS 1971	21
ICP Spectroptomist	Anne L. Spradlin	BA 1983	22
ICP Analyst	David Tryon		4
ICP and ICP-MS Chemist	Dianne Gardner	BA 1987	4
ICP-MS and GFAA Analyst	Kevin Hathaway		20
CVAA Analyst	Judy Ashcraft		38
Chemist	Stephanie Clearwater	BS 2000	6
Chemist	Brian Flynn	MS 2003	2
Chemist	Melissa Dorrell	BS 2004	2
Chemist	Sherry Maine	MS 2004	7
Chemist	Aleisha Elliott	BS 2007	1
Analyst	Dean Palmer	BS 1979	10
Analyst	Gordon Anthis		17
Analyst	Traci Wood		2
Analyst	Debbie Schultz		6
Analyst	Heidi Barnes		5
Analyst	Sheila Kratz		3
Analyst	Jennifer Sieg		1
Sample Control Officer	Crystal Sevy		5
Sample Receiving	Cindy Flores		4
Sample Receiving	Robin Stribling		2
Document Control	Dianne Costa		1
Document Control	Geri Napolitan		2
Maintenance	Dan Bair		1
Receptionist	Gloria Burmeister		5

3.0 JOB DESCRIPTIONS

3.1 Laboratory Director

The Director supervises day-to-day operations of the laboratory. Responsible for monitoring standards of performance in quality control and quality assurance, and for monitoring the validity of the analyses performed and data generated in the laboratory. The Director holds a weekly staff meeting to discuss client and technical issues.

3.2 Quality and Systems Manager

The Systems Manager supervises operations of the Information Technology groups. The Systems Manager uses Excel, Crystal Reports and other database programs to develop and maintain client reports and electronic data deliverables.

3.3 Department Supervisor

Department heads supervise the day-to-day operations of our analytical departments. They are responsible for department safety and analyst training. They are also responsible for review of out-going analytical data.

3.4 Quality Assurance Coordinator (QAC)

The QAC is responsible for implementation of the quality system. The QAC manages the performance evaluation sample program and conducts laboratory audits. The QAC obtains and maintains laboratory accreditations, reviews and approves SOPs, and conducts staff training in integrity and quality systems.

3.5 Document Control Officer (DCO)

DCO is responsible for the generation and the retention of analytical reports and records, including but not limited to Chains-of-Custody and sample shipping documents. DCO is also responsible for delivering electronic data deliverables.

3.6 Sample Control Officer (SCO)

SCO is responsible for sample receipt, job creation/verification, and sample storage.

3.7 Technical Director

Technical Directors provide technical support to laboratory staff and provide final reviews of analytical data packages.

4.0 APPROVED LABORATORY SIGNATORIES

The Laboratory Director, John Kern, Quality and Systems Manager, Brandan Borgias, and Technical Director, Kirby Gray and Deputy Technical Directors, Larry Drew and Nan Wilson, and Department Supervisors Jim Hodge and Danny Sevy are approved laboratory signatories for analytical reports. QA/QC Coordinator Michael Desmarais has report generation privileges.

5.0 RECORDS AND DOCUMENT CONTROL

5.1 Standard Operating Procedures (SOPs)

The Quality Assurance Coordinator (QAC) retains the master copies of SOPs. Controlled copies are distributed to individual laboratories as appropriate and electronic copies are available on the laboratory's computer network. All SOPs are scheduled for review each year. Electronic copies are available on the laboratory network on the date of the Coordinator's review with promulgation two weeks after that date. When a revision is created, the previous version is removed from the master file and affected laboratories, with a copy retained for the SOP archive file.

5.2 Quality Manual (QM)

The QAC retains the master copy of the QM. The QM is scheduled for review annually or when revisions are needed. Management makes copies available to Accrediting Authorities, laboratory staff and clients as needed. When a revision is created, previous versions are removed from use, and a copy is retained in the QM archive file.

5.3 Analytical Data

The Document Control Officer (DCO) retains analytical data, including calibration records and quality control, for five years, unless a longer period is required by contract.

5.4 Training Records

The QAC maintains records of analyst training and proficiency.

5.5 Performance Evaluation Samples

The QAC maintains records of analysis of performance evaluation samples and the reports associated with the analyses.

5.6 External and Internal Audits

The QAC retains records of external and internal audits.

5.7 Corrective Action Reports

Are kept electronically and filed by hardcopy.

5.8 Laboratory Logbooks

SVL controls the issue, use, and closure of laboratory logbooks. The process is described in SOP SVL 2017. Examples of logbooks may include: the conductivity of laboratory water, preparation of reagents and standards, preparation of samples, calibration of balances, calibration of micropipets, volumetric pipets, repipettors, maintenance of instruments, and temperatures of ovens and refrigerators. The QAC assigns and archives logbooks.

5.9 Chain of Custody

The DCO is in charge of chain-of-custody retention, they are currently held for five years, unless a longer time is required by contract. Records of C.O.C.s, sample log-in and job creation are maintained in SVL's LIMS. LIMS files are backed-up 3 times a day and electronically archived for five years.

5.10 Analytical Reports

The DCO retains photocopies of analytical reports for five years, unless a longer time is required by contract. Electronic copies are stored

within the LIMS database and are archived for seven years. Archived analytical reports are stored in a secured environment to protect them from damage.

5.11 Backup and Storage of Electronic Data

5.11.1 Electronic Data Collection: Currently the backup server is protected with an administrative password, which is changed every 6 months; it is in control of the IT Systems Analyst.

5.11.2 Archives of Electronic Data: Data files that reside on the SVL file servers are backed up on a daily basis and kept onsite for 90 days: a full backup of the data files residing on the server is done monthly and sent to an offsite storage facility for 7 years. All software used to recover data files is also stored at the offsite facility.

5.11.3 Offsite Backup Storage: A secure offsite facility is maintained to house the electronic data collected by the current backup system.

6.0 TRACEABILITY OF MEASUREMENTS

6.1 Chemicals and Reagents

SVL uses reagent grade or better chemicals. Some equivalent grades are "Fisher Trace Metals", "Baker Instra-Analyzed", "Baker A.C.S.", "Baker Analyzed", "Fisher A.C.S.", and "Fisher Certified". SVL requires a certificate of analysis or purity for stock calibration standards.

SVL records the preparation of reagents and standards in controlled logbooks or electronically in the LIMS. The initials of the preparer, the date prepared, the lot number and amount of stock materials, the final volume, the matrix, and the expiration date are all recorded. Preparation instructions are included in the SOPs for the analytical methods.

SVL labels containers of prepared reagents and standards with their contents, a unique reference number, date prepared, disposal (expiration) date and a perceived hazard warning.

SVL routinely obtains reference standards from commercial sources. These standards are used to check and document the concentration of calibration standards and validate method QC requirements.

SVL stores reagents and standards separately from samples.

6.2 Water

The primary reagent water in the laboratory is furnished by a reverse osmosis system followed by a micropore filter with an ion-exchange resin cartridge. This satisfies the specifications of ASTM Type II water. When Type I (16.67 MΩ-cm) water is required, SVL uses a four-cartridge ion-exchange system. SVL measures and records the resistivity and conductivity of the laboratory water each weekday.

7.0 TEST METHODS

7.1 Analyses Performed by SVL

SVL routinely performs the following analytical methods.

ANALYTE	METHOD	TECHNIQUE
Aluminum	EPA 200.7, SW846 6010B	ICP
Antimony	EPA 200.7, SW846 6010B	ICP
Antimony	EPA 200.8, SW846 6020	ICPMS
Arsenic	EPA 200.7, SW846 6010B	ICP
Arsenic	EPA 200.8, SW846 6020	ICPMS
Barium	EPA 200.7, SW846 6010B	ICP
Barium	EPA 200.8, SW846 6020	ICPMS
Beryllium	EPA 200.7, SW846 6010B	ICP
Beryllium	EPA 200.8, SW846 6020	ICPMS
Boron	EPA 200.7, SW846 6010B	ICP
Boron	EPA 200.8, SW846 6020	ICPMS
Cadmium	EPA 200.7, SW846 6010B	ICP
Cadmium	EPA 200.8, SW846 6020	ICPMS
Calcium	EPA 200.7, SW846 6010B	ICP
Chromium	EPA 200.7, SW846 6010B	ICP
Chromium	EPA 200.8, SW846 6020	ICPMS
Chromium, Hexavalent	ASTM D-1687, SM 3500 CR D	Colorimetry
Cobalt	EPA 200.7, SW846 6010B	ICP
Cobalt	EPA 200.8, SW846 6020	ICPMS
Copper	EPA 200.7, SW846 6010B	ICP
Copper	EPA 200.8, SW846 6020	ICPMS
Gallium	EPA 200.7, SW846 6010	ICP
Gold	EPA 231.2	GFAA
Iron	EPA 200.7, SW846 6010B	ICP
Lanthanum	EPA 200.7, SW846 6010B	ICP
Lead	EPA 200.7, SW846 6010B	ICP
Lead	EPA 200.8, SW846 6020	ICPMS
Lithium	EPA 200.7, SW846 6010B	ICP

ANALYTE	METHOD	TECHNIQUE
Magnesium	EPA 200.7, SW846 6010B	ICP
Manganese	EPA 200.7, SW846 6010B	ICP
Manganese	EPA 200.8, SW846 6020	ICPMS
Mercury	EPA 245.1, SW846 7470A, 7471A	CVAA
Molybdenum	EPA 200.7, SW846 6010B	ICP
Molybdenum	EPA 200.8, SW846 6020	ICPMS
Nickel	EPA 200.7, SW846 6010B	ICP
Nickel	EPA 200.8, SW846 6020	ICPMS
Potassium	EPA 200.7, SW846 6010B	ICP
Scandium	EPA 200.7, SW846 6010B	ICP
Selenium	SM 3114C	Hydride AA
Selenium	EPA 200.7, SW846 6010B	ICP
Selenium	EPA 200.8, SW846 6020	ICPMS
Silica	EPA 200.7	ICP
Silver	EPA 200.7, SW846 6010B	ICP
Silver	EPA 200.8, SW846 6020	ICPMS
Sodium	EPA 200.7, SW846 6010B	ICP
Strontium	EPA 200.7, SW846 6010B	ICP
Thallium	EPA 200.7, SW846 6010B	ICP
Thallium	EPA 200.8, SW846 6020	ICPMS
Tin	EPA 200.7, SW846 6010B	ICP
Titanium	EPA 200.7, SW846 6010B	ICP
Uranium	EPA 200.8	ICPMS
Vanadium	EPA 200.7, SW846 6010B	ICP
Vanadium	EPA 200.8, SW846 6020	ICPMS
Zinc	EPA 200.7, SW846 6010B	ICP
Zinc	EPA 200.8, SW846 6020	ICPMS
Acidity	SM 2310 B	Automated Titration
Alkalinity	SM 2320 B	Automated Titration
Ammonia	EPA 350.1	Automated Colorimetry
Bromide	EPA 300.0	Ion Chromatography
Chemical Oxygen Demand	EPA 410.4	Colorimetry
Chloride	EPA 300.0	Ion Chromatography
Color	SM 2120 B	Colorimetry
Conductivity	EPA 120.1	Wheatstone Bridge
Corrosivity	SM 2330 B	Langelier Index
Cyanide, Total	EPA 335.4, SW 846 9012B	Automated Colorimetry
Cyanide, Free	SM 4500 CN (F)	Ion Specific Electrode
Cyanide, WAD	SM 4500 CN I	Automated Colorimetry
Cyanide, Available	OIA 1677	Amperometry
Fluoride	EPA 300.0	Ion Chromatography
Hardness	SM 2340B, Ca as $CaCO_3$ by 200.7	ICP Sum
Ignitability	SW846 1010	Pensky-Martens
Nitrate	EPA 300.0	Ion Chromatography
Nitrate + Nitrite	EPA 353.2	Automated Colorimetry
Nitrate + Nitrite	EPA 300.0	Ion Chromatography
Nitrite	EPA 300.0	Ion Chromatography
Nitrite	EPA 353.2	Automated Colorimetry
Odor	SM 2150B	Sniff Panel
ortho-Phosphate	SM 4500 P E, 300.0	Colorimetry, IC

ANALYTE	METHOD	TECHNIQUE
pH (aqueous)	SM 4500-H^+ B	Electrometric
pH (soil)	EPA 9045C, EPA 9045D	Electrometric
Paste pH	ASA Monograph 9	Electrometric
Phosphate, Total	SM 4500 P E	Persulfate Digestion
Residue, Filterable (TDS)	SM 2540 C	Gravimetric
Residue, Non Filterable (TSS)	SM 2540 D	Gravimetric
Settleable Solids	SM 2540 F	Volumetric
Specific Conductance	EPA 120.1, SM 2510 B	Wheatstone Bridge
Sulfate	EPA 300.0	Ion Chromatography
Sulfide	SM 4500 S^{-2} F	Titrimetric
Surfactants (MBAS)	SM 5540 C	Colorimetry
Total Solids	SM 2540 B	Gravimetric
Total Kjeldahl Nitrogen	EPA 351.2, SM 4500 NH_3D	Colorimetry
Total Organic Carbon	SM 5310 B	Combustion
Total Volatile Solids	EPA 160.4	Gravimetric
Turbidity	EPA 180.1	Nephelometric
TCLP (Toxicity Characteristic Leaching)	SW846 1311	Extraction
SPLP (Synthetic Precipitation Leaching)	SW846 1312	Extraction
STLC (Soluble Threshold Limit Concentration)		Extraction
MWMP (Meteoric Water Mobility)	ASTM E2242-02	Extraction
CA-WET (California Waste Extraction Test)		Extraction
CEC (Cation Exchange Capacity)	SW846 9081	
Textural Analysis (Particle Size)	ASA “Methods of Soil Analysis” Number 9, Part 1	
Specific Gravity		Displacement
TOM/TOC	USDA, HB60(24)	
ANP (Acid Neutralization Potential)		Titration
ABA (Acid Base Account)	ASTM E1915-05	LECO
Total Sulfur + Sulfur Forms	ASTM E1915-05	LECO
Total Carbon	ASTM E1915-05	LECO
Arsenic Speciation	K.S. Subramanian et al.	Graphite Furnace
Iron Speciation	HACH-8146	Colorimetry
TKN	ASA “Methods of Soil Analysis” Number 9	
Gradation		Sieving
Loss on Ignition	Soil & Plant Analysis Council	Gravimetric
Percent Silica	ASTM 2795	Colorimetry
Tot Suspended Particulates	40CFR 50, App B amend 12/6/82	Gravimetric
Flash Point	SW-846 1010, ASTM D93-80	Closed Cup

7.2 References

Methods for Chemical Analysis of Water and Wastes, revised March 1983, EPA-600/4-79-020.

Methods for the Determination of Metals in Environmental Samples Supplement I, EPA/600/R-94/111, May 1994

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW 846), Third Edition, Update III, December 1996.

Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992

Standard Methods for the Examination of Water and Wastewater, 19th Edition, 1995

Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1999

ASTM Book of Standards, part 31

Soil Testing and Plant Analysis, 3rd Edition, Soil Sciences Society of America, 1990

American Society of Agronomy, "Methods of Soil Analysis" Number 9, Parts 1 and 2

U.S. Department of Agriculture, Handbook #60

U.S. Department of the Interior, Bureau of Reclamation, Procedure for Determining Moisture, Ash, and Organic Content of Soil, USBR 5430-89

8.0 NEW WORK

The Business Development group discusses new work with clients before the work is received. If the work being requested involves tests not usually performed by SVL, the project is discussed with Department Supervisors to determine if the work can be accepted. Occasionally SVL receives a work order with no prior notification that requests unusual tests, or tests to be conducted in a time frame not suitable for the work requested. When this

occurs, the Sample Control Officer reviews the job with Client Services and/or Department Supervisors to determine if the work can or should be accepted. Routine work from established clients normally is not reviewed with the clients before jobs are set up, unless there is a problem with sample integrity or information on the Chain-of-Custody.

SVL reviews and makes available to the LIMS, a client's requested work order. A schedule can be derived for the work that has been received; this allows the staff to plan workloads and to track jobs. A Laboratory/Technical Director or Client Services member shall review all work orders. Adjustments to work schedules and staff deployment are made based upon the workload. Department Supervisors keep equipment and supplies on hand for routine work and for many non-routine tests as well.

8.1 Sample Acceptance Policy

8.1.1 Samples received at SVL will be accepted for testing if the following criteria are met at the time of sample receipt:

A proper SVL or client Chain of Custody will accompany the sample shipment and must be completed in full, including but not limited to; the client's name, address, phone/fax numbers, contact person, unique sample identification of individual samples, sample locations (if applicable), date and time of collection, collector's name, preservative type, sample matrix, filtered or unfiltered, number of bottles, analytes and/or tests to be performed, method of analysis, and any comments concerning sample specifics or QC requirements.

The use of correct sample containers (with proper preservation) for the sample matrices collected and ensuring that sufficient sample volume is provided for the tests requested (including extra volumes for QC requirements).

Accurate labeling of sample bottles using coded, water resistant labels and permanent ink, with said labels being cross referenced with information contained in the Chain of Custody.

Adherence to holding time requirements as required by test or method requested.

8.1.2 In the event that a sample is received in non-compliance with this policy, the sample in question will be segregated and the client notified by telephone or email. The client may direct SVL to continue on with analysis of the non-conforming sample(s). Non-conformity will be noted on the Sample Receipt/Chain of Custody and within the Final Report.

8.1.3 New clients will be informed of this policy through Client Services or Sample Receiving. They will be provided with a copy of the Quality Manual (hard bound or electronically) or a hand out on sample acceptance (located in SVL's waiting room or in Sample Receiving).

Current clients will receive these notices if they bring in samples that do not meet SVL's requirements.

9.0 CALIBRATION

9.1 Thermometers

Calibrating thermometers is described in SOP SVL 1004.

An outside company calibrates SVL's NIST-certified thermometers.

SVL calibrates in-house liquid-in-glass thermometers against a NIST-certified thermometer. Digital thermometers are calibrated against a NIST-certified thermometer. The thermometers are then labeled with a correction factor.

9.2 Balances

Servicing and calibrating balances is described in SOP SVL 1025.

An outside company services and calibrates SVL's balances.

SVL checks the calibration of a balance before each day of use with at least two weights traceable to a NIST traceable standard. For analytical balances, the measured weight must agree with the certified weight within 0.1%. Balances that fail the criterion are checked with Class-1 weights. If they still fail, they are removed from service.

9.3 Balance Weights

Calibrating balance weights is described in SOP SVL 1025.

An outside company calibrates SVL's set of Class-1 weights, with Reference Standards Traceable to NIST.

SVL uses Class-1 weights to certify the Class-4 weights used for the daily calibration of balances.

9.4 Micropipets

The calibration of micropipets is described in SVL SOP 1026.

SVL checks the calibration of variable-volume micropipets each day of use. Fixed-volume micropipets are checked quarterly. The mean of three measured volumes must agree with the expected value within 3%. Micropipets that fail this criterion are repaired or removed from service.

9.5 Repipettors

The calibration of repipettors is described in SVL SOP 1026.

SVL checks the calibration of repipettors quarterly. The measured volume must agree with the expected value within 3%. Repipettors that fail this criterion are repaired or removed from service.

9.6 Refrigerators

SVL records the temperature of sample, standard, and reagent storage refrigerators each weekday. The process is described in SVL SOP 2004. The temperature must lie in between 0º and 6ºC. If a temperature is outside this criterion, the temperature is recorded again after one hour. If the temperature is still outside the acceptance range, samples, standards, and reagents are transferred to alternate refrigerators or coolers.

9.7 Ovens

SVL records the temperature of ovens for drying solids each weekday. The required temperature is stated in the applicable SOP.

9.8 Inductively Coupled Plasma Mass Spectrometer (ICP-MS)

SVL calibrates its ICP-MS in accordance with EPA methods 200.8 and 6020. Five calibration standards and a calibration blank are analyzed at the beginning of a sequence. The software creates a linear calibration curve that must have a correlation coefficient of at least 0.995. An Initial Calibration Verification (ICV) from a secondary source follows to verify the calibration. An Initial Calibration Blank (ICB) indicates the system is clean. Analysis of a Continuing Calibration Verification (CCV) and a Continuing Calibration Blank (CCB) follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4111.

9.9 Inductively Coupled Plasma Spectrometer (ICP)

SVL calibrates ICPs in accordance with EPA methods 200.7 and 6010B. A single calibration standard and a calibration blank are analyzed at the beginning of a sequence. A standard at the reporting limit is analyzed to verify that the instrument will detect a response at that level. An Initial Calibration Verification (ICV) from a secondary source follows to verify the calibration. An Initial Calibration Blank (ICB) indicates the system is clean. Analysis of a Continuing Calibration Verification (CCV) and a Continuing Calibration Blank (CCB) follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4102.

9.10 Graphite Furnace Atomic Absorption Spectrometer (GFAA)

SVL calibrates it's GFAA in accordance with EPA method 231.2. for gold and K.S. Subramanian et al. for arsenic speciation. Three calibration standards and a calibration blank are analyzed at the beginning of a sequence. Perkin-Elmer instruments create a linear calibration curve that must have a correlation coefficient of at least 0.995. An Initial Calibration Verification (ICV) from a secondary source follows to verify the calibration. An Initial Calibration Blank (ICB) indicates the system is clean. Analysis of a Continuing Calibration Verification (CCV) and a Continuing Calibration Blank (CCB) follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4115.

9.11 Mercury Analyzer (CVAA)

SVL calibrates its CVAA in accordance with EPA methods 245.1, 7470A, and 7471A. Six calibration standards and a calibration blank are analyzed at the beginning of a sequence. The instrument creates a linear calibration curve that must have a correlation coefficient of at least

0.995. An Initial Calibration Verification (ICV) from a secondary source follows to verify the calibration. An Initial Calibration Blank (ICB) indicates the system is clean. Analysis of a Continuing Calibration Verification (CCV) and a Continuing Calibration Blank (CCB) follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4010.

9.12 Flame Atomic Absorption Spectrometer (FLAA)

SVL calibrates FLAAs in accordance with analytical method requirements.

9.13 Ion Chromatograph (IC)

SVL calibrates ICs in accordance with EPA method 300.0. Five calibration standards and a calibration blank are analyzed. The instrument creates a linear or quadratic calibration curve that must have a correlation coefficient of at least 0.995 An Initial Calibration Verification (ICV) from a secondary source follows to verify the calibration. An Initial Calibration Blank (ICB) indicates the system is clean. A Continuing Calibration Verification (CCV), and a Continuing Calibration Blank (CCB) follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4122.

9.14 Flow-Injection Auto Analyzer (FIA)

SVL calibrates FIAs in accordance with EPA methods 335.4 (Total Cyanide), 350.1 (Ammonia), 353.2 (Nitrate and Nitrite), 9012B (Total Cyanide), and Standard Methods 4500-CN-I (WAD Cyanide), and method OI 1677 (Amperometric Cyanide). A minimum of five calibration standards and a calibration blank are analyzed at the beginning of each analytical sequence. The instrument software creates a linear or quadratic calibration curve that must have a correlation coefficient of at least 0.995. A Laboratory Control Sample (LCS) and an Initial Calibration Verification (ICV) from a secondary source verifies the calibration curve. An Initial Calibration Blank (ICB) indicates the system is clean. Analysis of a Continuing Calibration Verification (CCV) and a Continuing Calibration Blank (CCB) follow after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOPs SVL 4012, SVL 4099, SVL 4048, SVL 4075, and SVL 4101.

9.15 Total Organic Carbon Analyzer (TOC)

SVL calibrates TOC analyzers in accordance with SM 5310 B. Three calibration standards for total carbon and three calibration standards for inorganic carbon are analyzed to prepare a calibration curve that must have a correlation coefficient of at least 0.995. A Continuing Calibration Verification (CCV) is analyzed at the beginning of each analytical sequence, after every ten samples and at the end of the analytical sequence. The acceptance criteria are defined in SOP SVL 4116.

9.16 UV/Visible Spectrophotometers (UV/VIS)

SVL calibrates its UV/Visible spectrophotometer in accordance with the applicable published methods. A minimum of three calibration standards and a calibration blank are analyzed at the beginning of each analytical sequence. The calibration curve must have a correlation coefficient of at least 0.995.

9.17 pH and Ion Selective Electrode Meters (ISE)

SVL calibrates pH and ISE meters in accordance with the applicable published methods. For TKN, SVL uses an Excel spreadsheet to create a calibration curve of potential (mV) versus log of concentration.

10.0 SAMPLING, SAMPLE RECEIVING, AND STORAGE

10.1 Sampling

SVL does not conduct sampling. Sampling procedures and contamination in the field are beyond SVL's control. SVL recommends the following procedures to its clients.

Sample preservation is critical for sample integrity. Chemical and biological reactions may occur that begin to change some chemical species upon sample collection. Unfortunately, for most samples, immediate analysis is neither economically feasible nor logistically possible. Although no chemical preservative exists that is valid for every parameter, SVL strongly recommends the preservation methods, container type, sample size and estimated maximum holding times for collection of water and wastewater samples summarized in Table 1.

Solid samples are best preserved by cooling the sample to a range between 0 and 6 C°.

Table 1

Analysis	Volume Required (mL)	Container	Preservative	Holding Time
Color	50	P,G	Cool to ≤ 6 °C	48 Hours
Conductance	100	P,G	Cool to ≤ 6°C	28 Days
Hardness	100	P,G	HNO_3 to pH<2	6 Months
Odor	200	G only	Cool to ≤ 6°C	24 Hours
pH	25	P,G	None Required	* ASAP
Temperature	1000	P,G	None Required	* ASAP
Turbidity	100	P,G	Cool to ≤ 6 °C	48 Hours
Filterable Residue (TDS)	100	P,G	Cool to ≤ 6 °C	7 Days
Non-Filterable Residue (TSS)	100	P,G	Cool to ≤ 6 °C	7 Days
Total Residue	100	P,G	Cool to ≤ 6 °C	7 Days
Volatile Residue	100	P,G	Cool to ≤ 6 °C	7 Days
Settleable Matter	1000	P,G	Cool to ≤ 6 °C	48 Hours
Dissolved Metals	200	P,G	Filter on site; HNO_3 to pH<2	6 Months
Total Metals	100	P,G	HNO_3 to pH<2	6 Months
Chromium (VI)	200	P,G	Cool to ≤ 6 °C	24 Hours
Mercury, Dissolved	100	P,G	Filter; HNO_3 to pH<2	28 Days
Mercury, Total	100	P,G	HNO_3 to pH<2	28 Days
Acidity	100	P,G	Cool to ≤ 6 °C	14 Days
Alkalinity	100	P,G	Cool to ≤ 6 °C	14 Days
Bromide	100	P,G	None Required	28 Days
Chloride	50	P,G	None Required	28 Days
Cyanide	500	P,G	Cool to ≤ 6 °C; NaOH to pH>12	14 Days
Fluoride	300	P	None Required	28 Days
Ammonia	400	P,G	Cool to ≤ 6 °C H_2SO_4 to pH<2	28 Days
Total Kjeldahl Nitrogen	500	P,G	Cool to ≤ 6 °C H_2SO_4 to pH<2	28 Days
Nitrate plus Nitrite	100	P,G	Cool to ≤ 6 °C H_2SO_4 to pH<2	28 Days
Nitrate	100	P,G	Cool to ≤ 6 °C	48 Hours
Nitrite	50	P,G	Cool to ≤ 6 °C	48 Hours

Analysis	Volume Required (mL)	Container	Preservative	Holding Time
Ortho-Phosphate Dissolved	50	P,G	Filter on site; Cool to ≤ 6 °C	48 Hours
Total Phosphate	50	P,G	Cool to ≤ 6 °C; H_2SO_4 to pH<2	28 Days
Total Dissolved Phosphate	50	P,G	Filter on site; Cool to ≤ 6 °C; H_2SO_4 to pH<2	28 Days
Silica	50	P only	Cool to ≤ 6 °C	28 Days
Sulfate	50	P,G	Cool to ≤ 6 °C	28 Days
Sulfide	500	P,G	Cool to ≤ 6 °C add 2 mL zinc acetate plus NaOH to pH>9	7 Days
COD	50	P,G	Cool to ≤ 6 °C H_2SO_4 to pH<2	28 Days
Total Organic Carbon	25	P,G	Cool to ≤ 6 °C H_2SO_4 or HCl to	28 Days
Phenolics	500	G only	Cool to ≤ 6 °C H_2SO_4 to pH<2	28 Days
MBAS	250	P,G	Cool to ≤ 6 °C	48 Hours

* pH and Temperature should be measured in the field whenever possible. They are subject to rapid change. Measurements of pH and Temperature made in the laboratory will almost always be out of holding time.

SVL has formed alliances with other laboratories for the analysis of organic parameters. The recommended containers and preservatives are

Analysis	Amount Required	Container	Preservative	Holding Time Until Extraction	Holding Time After Extraction Until Analysis
Mercury, Low Level**					
524.2 (Volatile Organic Compounds)	3x40mL vials	G,T	Cool to ≤ 6 °C; HCl to pH<2	14 days	NA
608 (Pesticides and/or PCBs)	3 L	amber G,T	Cool to ≤ 6 °C	7 days	40 days
624 (Volatile Organic Compounds)	3x40mL vials	G,T	Cool to ≤ 6 °C; HCl to pH<2	14 days	NA
625 (Semi-volatile Organic Compounds)	3 L	amber G,T	Cool to ≤ 6 °C	7 days	40 days
1664 Hexane Extractable Materials	2L	G only	Cool to ≤ 6 °C H_2SO_4 or HCl to pH<2	28 days	NA
8081A (Pesticides)	8 oz (soil) 1L (aqueous)	amber G,T	Cool to ≤ 6 °C	14 days 7 days	40 days

Analysis	Amount Required	Container	Preservative	Holding Time Until Extraction	Holding Time After Extraction Until Analysis
Mercury, Low Level**					
8082 (PCBs)	8 oz (soil) 1 L (aqueous)	G,T	Cool to ≤ 6 °C	14 days 7 days	40 days
8260B (Volatile Organic Compounds)	4 oz (soil) 3x40mL (aq)	G,T	Cool to ≤ 6 °C; HCl to pH<2	14 days	NA
8270C (Semi-volatile Organic Compounds)	8 oz (soil) 1 L (aqueous)	amber G,T	Cool to ≤ 6 °C	14 days	40 days
8015 (TPH-Gasoline)	4 oz (soil) 3x40 mL (aq)	amber G,T	Cool to ≤ 6 °C; HCl to pH<2	14 days	35 days
8015AZ ***	8 oz (soil)	G,T	Cool to ≤ 6 °C	48 hours	14 days for extraction and analysis
8260BAZ***	4 oz (soil)	G,T	Cool to ≤ 6 °C	48 hours	NA
8015 (TPH-Diesel Motor Oil)	1 L (aq) 8 oz (soil)	amber G,T	Cool to ≤ 6 °C: HCl to pH<2	14 days	40 days

** Call for sampling and hold time requirements.
*** TPH 8015AZ and 8260AZ (soils) have a 48 hour hold time before extraction.

10.1 Sampling Cont'd

Field blanks allow for identification of systematic and random sample contamination that may result from the sampling equipment, storage containers, sampling agents, or chemicals added to preserve samples. Field blanks consist of a sample container of distilled or deionized water with the appropriate chemical preservative. Preservation, filtration, storage, handling, and analysis are performed as if the field blanks were samples. To achieve accurate and meaningful data, field blank containers should be filled with analyte-free water and the appropriate preservative at the sampling site.

Sources of sample contamination include unclean sample containers and filters; impure solvents and reagents; and use of cleaning products inappropriate for the proposed analysis. Hair, tobacco smoke, and dust also are appreciable sources of contamination, so sampling should be conducted in as careful a manner as possible.

Before filtering samples for dissolved parameters, the filter paper should be rinsed with de-ionized or distilled water and with a small portion of

sample. The filtration apparatus should also be rinsed with de-ionized or distilled water between samples. Handle filter paper only on the edge, using appropriate forceps (plastic for trace metals analysis).

Use the proper sample container for the parameter specified. Samples for trace metals analysis must not come into contact with any metallic surface; samples for organic analysis must not come into contact with any plastic surface.

Sampling personnel should complete a chain-of-custody form that documents sample identification, sampling date and time, matrix type, number of sample containers, type of preservation, whether samples have been filtered, and the parameters to be analyzed.

10.2 Sample Receiving and Storage

SOPs SVL 2001, SVL 2003, and SVL 2004 describe sample receiving, job creation, and sample storage, respectively.

SVL takes a temperature reading for sample shipping containers (coolers) upon receipt and opening. Each sample is checked for visible damage and the presence of an intact custody seal. SVL gives each group of samples a unique job number (e.g., "100001"). This job number remains with the samples throughout the analytical process. Each sample is assigned a unique, sequential identification number. Samples are labeled with a bar code (containing sample and job numbers) before being stored in a secure area.

Samples that require refrigeration are stored in walk-in coolers (which are kept between 0ºC and 6°C), except during times of sample preparation or analysis. Samples that do not require refrigeration are stored in a sample storage annex. The laboratory does not refrigerate soil samples that were received without refrigeration. Samples are retained by SVL for a minimum of 30 days (or longer if required by the client) after a data report is issued to the client. At the end of the specified period, samples are returned to the client or discarded in an appropriate manner.

Analysts use the LIMS to provide a custody log of sample movement during preparation and analysis. Analysts are responsible for logging the samples in to their custody. They assume accountability for the sample(s) while they are in their possession. When use of the sample is complete, analysts must scan samples back into the appropriate home

location or another analyst may assume custody by scanning/logging the sample into their possession in the LIMS.

10.3 Sub-sampling

Sub-sampling is described in SOP SVL 2018.

10.4 Sample Disposal and Hazardous Waste

Procedures for sample disposal are described in SOP SVL 1001. Disposal procedures follow federal and state regulatory requirements. SVL's hazardous waste program is described in SOP SVL 1008.

11.0 EQUIPMENT AND INSTRUMENTS

SVL uses the following instruments to generate analytical data and to calibrate other instruments.

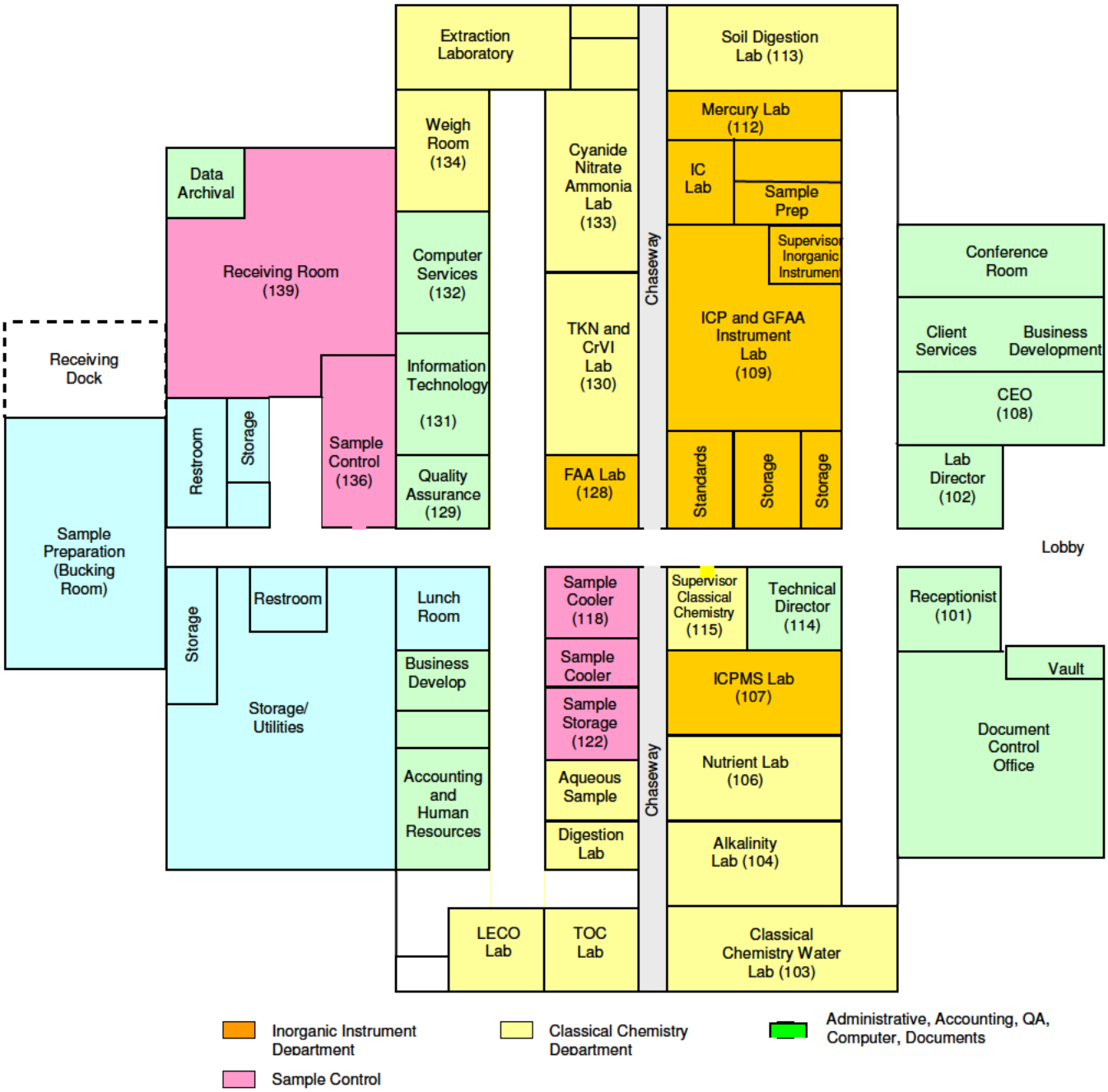
11.1 SVL performs instrument maintenance as recommended by the manufacturer. SVL maintains service contracts with vendors for its major analytical instrumentation. Maintenance logbooks are kept to provide a record of major and minor repairs; as well as, preventative maintenance.

11.2 The analysts and supervisors will determine if a repair has created a need to update instrument MDLs, linear ranges, calibrations etc.

INSTRUMENT	MANUFACTURER	MODEL	SERIAL NUMBER
Spectrometer (ICP-MS)	Perkin-Elmer	ELAN 5000	W0660402
ICP-MS Auto Sampler	ESI	SCFAST	X2-070106
Spectrometer (ICP) Optima 1	Perkin-Elmer	Optima 4300	077N0061602
Spectrometer (ICP) Optima 5	Perkin-Elmer	Optima 5300	077N5011902
Spectrometer (ICP) Optima 6	Perkin-Elmer	Optima 5300	077N6062101
Spectrometer (ICP) Optima 7	Perkin-Elmer	Optima 5300	077C8011601
ICP Auto Sampler	ESI	SCFAST	X2-060502
Atomic Absorption Spectrometer with Graphite Furnace	Perkin-Elmer	Analyst 600	601S3090501
Atomic Absorption Spectrometer with Vapor Generation Assembly	Varian	AA 55B	EL03048142
Atomic Absorption Spectrometer with Vapor Generation Assembly	Varian	SpectrAA 20	9101123
Mercury Analyzer with Autosampler	CETAC	M-6000A	029907MAS

INSTRUMENT	MANUFACTURER	MODEL	SERIAL NUMBER
Ion Chromatograph	Dionex	ICS90	4090417
Ion Chromatograph	Dionex	DX-100	921517
Ion Chromatograph	Dionex	4000i	14421
Automated Flow Analyzer with Autosampler	Alpkem	FS3000	843-1604-758
Micro Distillation unit	Lachat	ID 001	A2000-828
MIDI Distillation Units	BSL		
Ammonia Distillation Unit	Andrews Glass		
Ammonia/N analyzer	Astoria Pacific	A2	200104
Auto sampler	Astoria Pacific	W311	4632A11096
Auto Titrator with Autosampler	Metrohm	Titrino 751GPD	1751.0010.08208
Auto Titrator with Autosampler	Metrohm	Titrino 809 Titrando	18090010-07108
UV/Visible Spectrophotometer	Genesys	10	205G261004
UV/Visible Spectrophotometer	Spectronic	501	0283085
Turbidimeter	Hach	2100	95041453
COD Reactor	Hach	COD	930900009554
COD Reactor	Hach	COD	971100016584
pH/Ion Meter	Corning	450	1246
pH/Ion Meter	Corning	150	2173
Ion Meter	Thermo	9606BNWP	
pH Meter	Accumet	AB15	AB92314557
pH Meter	Thermo	Symphony SB20	6081
pH Meter	Thermo	Symphony SB20	5712
pH Meter	Beckman		224148
Dissecting Microscope	Nikon	104	
Polarizing Microscope	Nikon	106	
Conductance Meter	Accumet	35636-30	AB 92315548
Elemental Analyzer	LECO	SC632	3208
Elemental Analyzer	LECO	SC444	3616
Carbon Analyzer (TOC)	Shimadzu	TOC-5000A	3701168A
Carbon/Nitrogen Analyzer (TOC)	Shimadzu	TOC-VCSH-N	37401162
Semi-Micro Balance	Mettler	AE-240	K89952
Semi-Micro Balance	Mettler	AE-240	G43270
Filter Balance	Mettler	AJ100	N09817
Analytical Balance	Sartorius	AC121S	41007209
Analytical Balance	Ohaus	Explorer	F2221120252601
Analytical Balance	Ohaus	AR2140 Adventurer	H2131203121033P
Analytical Balance	Ohaus	AR1530 Adventurer	1203200181P
Analytical Balance	Ohaus	N1D110 Navigator	1122352966
Analytical Balance	Ohaus	AS 513	8028301193
Thermometer	HBI	68ºC to 86ºC	4B1321
Thermometer	Ertco	-20ºC to 110ºC	5283
Thermometer	Ertco	0º C to 202ºC	I94-245

12.0 FACILITIES



12.1 SVL is an analytical laboratory specializing in the performance of tests and methods used in the characterization of environmental and mining samples. Since 1972, SVL has analyzed water, soil, sediment, sludge, oil, paint, rock, animal tissue, vegetation, air filters, and other sample types. SVL occupies a modern 25,000 square foot laboratory facility architecturally designed and specifically organized to ensure efficient operation and meet the needs of a large capacity analytical laboratory. Building access, security and safety features have been carefully considered. Access through the outside laboratory entrance and to internal areas is limited to laboratory staff and other essential personnel. Visitors are escorted during their stay at SVL.

13.0 STANDARD OPERATING PROCEDURES

SVL performs work in accordance with the requirements of its SOPs. SVL's SOPs are listed below and describe all aspects of its work performance including Safety and Quality Assurance (1000 Series), Sample and Document Management (2000 Series) and Inorganic Analysis (4000 Series).

SOP NUMBER	DESCRIPTION
SVL 1001	SAMPLE DISPOSAL
SVL 1002	WRITING AND REVISING STANDARD OPERATING PROCEDURES
SVL 1004	CALIBRATING THERMOMETERS
SVL 1005	INTERNAL QUALITY ASSURANCE AUDITS
SVL 1007	SOIL STERILIZATION
SVL 1008	DISPOSAL OF HAZARDOUS WASTE
SVL 1010	TRAINING
SVL 1011	PERFORMING AN MDL STUDY
SVL 1015	PROCUREMENT, RECEIVING, AND SUBCONTRACTING
SVL 1017	RECORDS RETENTION AND PROTECTION
SVL 1019	CORRECTIVE ACTION
SVL 1020	CALIBRATION FOR ANALYTICAL METHODS
SVL 1021	MANUAL INTEGRATION
SVL 1022	IN-HOUSE ACCEPTANCE LIMITS,CONTROL CHARTS AND TRENDING
SVL 1023	SOFTWARE VERIFICATION
SVL 1025	CALIBRATING BALANCES
SVL 1026	CALIBRATING MICROPIPETS, REPIPETTORS, AND GLASSWARE
SVL 1027	CLIENT SERVICES
SVL 1028	CALCULATIONS FOR ANALYTICAL METHODS
SVL 1029	PERFORMANCE TESTING SAMPLES

SOP NUMBER	DESCRIPTION
SVL 1030	INITIAL, PERIODIC AND AFTER-MAINTENENANCE CHECKS
SVL 1031	COMPUTER AND INFORMATION SECURITY POLICY
SVL 2001	SAMPLE RECEIVING
SVL 2003	SVL JOB CREATION
SVL 2004	SAMPLE STORAGE AND SECURITY
SVL 2006	DATA CORRECTIONS
SVL 2007	CASE FILE ASSEMBLY
SVL 2007-ILMO5.4	CASE FILE ASSEMBLY ILOM5.4
SVL 2009	DATA REVIEW
SVL 2013	DATA PACKAGE PRODUCTION
SVL 2015	LEVEL 3 – CLP DATA PACKAGE
SVL 2017	LOGBOOK CONTROL
SVL 2018	PREPARATION AND SUBSAMPLING OF EARTH, ROCK, AND TISSUE SAMPLES
SVL 4010	DETERMINATION OF MERCURY (CVAA)
SVL 4010-ILMO5.4	DETERMINATION OF MERCURY (CVAA) BY ILMO5.4
SVL 4012	TOTAL CYANIDE BY MIDI DISTILLATION FOLLOWED BY AUTOMATED COLORIM
SVL 4012-ILMO5.4	TOTAL CYANIDE BY MIDI DISTILLATION FOLLOWED BY ILMO5.4
SVL 4013	GLASSWARE WASHING FOR CLASSICAL CHEMISTRY AND TRACE METALS
SVL 4021	FILTER DIGESTION
SVL 4022	PERCENT SOLIDS/PERCENT MOISTURE
SVL 4024	COLOR
SVL 4025	CONDUCTIVITY
SVL 4026	TURBIDITY (METHOD 180.1)
SVL 4028	PH
SVL 4029	SPECIFIC GRAVITY
SVL 4031	ACIDITY
SVL 4032	SULFIDES BY TITRATION
SVL 4034	TOTAL DISSOLVED SOLIDS AND SUSPENDED SOLIDS
SVL 4035	TOTAL AND VOLATILE SOLIDS
SVL 4037	METHYLENE BLUE ACTIVE SUBSTANCES
SVL 4040	TOTAL PHOSPHORUS (AQUEOUS SAMPLES)
SVL 4042	ORTHO-PHOSPHATE (AS P)
SVL 4043	CHEMICAL OXYGEN DEMAND
SVL 4044	TOTAL ORGANIC MATTER
SVL 4045	TOTAL KJELDAHL NITROGEN
SVL 4048	NITRATE/NITRITE AS N: AUTOMATED CADMIUM RE REDUCTION
SVL 4049	CATION EXCHANGE CAPACITY BY METHOD 9081

SOP NUMBER	DESCRIPTION
SVL 4056	FREE CYANIDE BY METHOD 4500-CN F
SVL 4060	LOSS ON IGNITION (SVL METHOD)
SVL 4061	DETERMINATION OF ACID GENERATING POTENTIAL (AGP), ACID NEUTRALIZATION POTENTIAL (ANP), AND ACID BASE ACCOUNTING (ABA)
SVL 4065	METEORIC WATER MOBILITY EXTRACTION
SVL 4068	SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP)
SVL 4070	TOTAL SUSPENDED PARTICULATES
SVL 4075	WAD CYANIDE BY MIDI DISTILLATION FOLLOWED BY SEMI-AUTOMATED COLORIMETRY
SVL 4078	SAMPLE DIGESTION FOR TOTAL METALS IN AQUEOUS SAMPLES FOR ICP-MS (EPA METHOD 3020A)
SVL 4079	SAMPLE DIGESTION FOR TOTAL METALS IN AQUEOUS SAMPLES FOR ICP (3010A)
SVL 4080	SAMPLE DIGESTION FOR TOTAL RECOVERABLE METALS IN AQUEOUS SAMPLES FOR ICP (3005A)
SVL 4081	HEXAVALENT CHROMIUM
SVL 4082	ARSENIC SPECIATION (ASIII AND ASV)
SVL 4084	DETERMINATION OF ALKALINITY AND pH USING THE AUTOTITRATOR
SVL 4093	CASSETTE FILTER DIGESTION
SVL 4094	SAMPLE DIGESTION FOR METALS IN SOILS (EPA METHOD 3050B)
SVL 4095	FLASHPOINT PENSKY-MARTENS CLOSED TESTER
SVL 4096	pH DETERMINATION FOR SOILS AND PASTE
SVL 4097	TOTAL SULFUR, TOTAL CARBON
SVL 4099	AMMONIA BY SEMI-AUTOMATED COLORIMETRY
SVL 4101	ANALYSIS OF AVAILABLE CYANIDE BY FLOW INJECTION AND AMPEROMETRY (METHOD 1677)
SVL 4102	ANALYSIS OF METALS BY METHODS 6010B AND 200.7 USING THE PERKIN-ELM OPTIMA ICP
SVL 4102-ILMO5.4	ANALYSIS OF METALS BY ILMO5.4 USING THE PERKIN-ELM OPTIMA ICP
SVL 4105	SELENIUM BY HYDRIDE
SVL 4106	SAMPLE DIGESTION FOR TOTAL RECOVERABLE METALS IN AQUEOUS SAMPLES BY ICP (200.2)
SVL 4107	SAMPLE DIGESTION FOR TOTAL METALS IN AQUEOUS SAMPLES BY ICP AND GFAA (40CFR136 APPENDIX C 9.3)
SVL 4108	SAMPLE PREPARATION FOR ANALYSIS OF DIRECT ANALYSIS, DRINKING WATER,DISSOLVED AND POTENTIALLY DISSOLVED METALS IN AQUAEOUS SAMPLES
SVL 4111	ANALYSIS OF METALS BY ICPMS (METHOD 200.8)
SVL 4111-ILMO5.4	ANALYSIS OF METALS BY ICPMS (METHOD 200.8) BY ILMO5.4
SVL 4112	ANALYSIS OF METALS BY ICPMS (METHOD 6020)
SVL 4114	TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)
SVL 4116	TOTAL ORGANIC CARBON
SVL 4118	CALIFORNIA WASTE EXTRACTION TEST (CA-WET)

SOP NUMBER	DESCRIPTION
SVL 4119	PREPARATION OF QC SOLUTIONS FOR METALS ANALYSIS
SVL 4120	TOTAL NITROGEN
SVL 4121	DETERMINATION OF THRESHOLD ODOR NUMBER (TON) SM 2150B
SVL 4122	INORGANIC ANIONS BY CHROMATOGRAPHY USING THE DIONEX DX 100 AND ICS-90
SVL 4123	ASTM D-2795 AND D-3682-78 SOLID SILICA
SVL 4124	OPERATION OF PERKIN/ELMER GFAA: ANALYSIS OF GOLD BY GRAPHITE FURNACE

13.1 Deviations

Occasionally, a deviation from an SOP is required to generate an accurate result for a given test or client. This may occur when a client specifically requires a modification, or when the sample matrix interferes with the analysis. The Laboratory Director or a Department Supervisor may authorize a deviation. The analyst documents details of the deviation from the SOP on the instrument raw data printout or the job bench sheet.

14.0 QUALITY CONTROL

14.1 Quality Control Parameters

SVL uses a number of quality control parameters to validate calibration, and to measure contamination, accuracy, and precision. Each SVL SOP defines the parameters required for the method being used.

14.1.1 Blanks

Method Blank Is an aliquot of analyte-free water that is put through all the steps of a specific method along with the samples. It is sometimes called a Laboratory Reagent Blank.

Field Blank Randomly selected sample container that is filled with analyte-free water and the appropriate chemical preservative in the field.

Trip Blank A specific type of field blank. A trip blank is not opened in the field. It is a check on sample contamination originating from sample transport, shipping, and site conditions.

The acceptance criterion for a blank may be set by the published method or by client Data Quality Objectives (DQOs). In the absence of these directives, the acceptance criterion may be set at the reporting limit.

14.1.2 Matrix Spike

Is an aliquot of sample to which a known amount of analyte has been added prior to sample preparation or digestion. It is a measure of the effect of the sample matrix on the analytical method. It is sometimes called the "Laboratory Fortified Matrix".

The recovery is calculated by:

$$\% \text{ Recovery} = 100 \times (MS - S) / SA$$

Where the MS = Spiked Sample Result
S = Sample Result
SA = Spike Added

The laboratory uses in-house statistical acceptance criteria. In the absence of in-house criteria, acceptance criteria for the matrix spike recovery may be determined by the published method, by client DQOs, or set at 75 to 125%, if the spike added is greater than some fraction of the concentration in the un-spiked sample, specified in the appropriate SVL SOP.

14.1.3 Analytical Spike or Post-Digestion Spike

Is an aliquot of sample to which a known amount of analyte has been added after sample preparation. It is a measure of the effect of the matrix on a digestate or extract.

14.1.4 Laboratory Control Sample (LCS)

Is a solution or material of known concentration that is analyzed to evaluate the accuracy of a method. It is sometimes called a Laboratory Fortified Blank.

The laboratory uses in-house statistical acceptance criteria. In the absence of in-house criteria, acceptance criteria for the LCS may be determined by the published method, by the manufacturer of the standard, or by client DQOs.

14.1.5 Sample Duplicate

A second aliquot of a sample treated exactly the same through preparation and analysis. The Relative Percent Difference (RPD) between the values of the duplicates is a measure of the precision of the analytical method.

$$RPD = 100 \times | S - D | / [(S + D)/2]$$

The laboratory uses in-house statistical acceptance criteria. In the absence of in-house criteria, the acceptance criterion for the RPD is usually set at 20% if the concentration in the sample is greater than five times the reporting limit. There is no acceptance criterion if the sample concentration is less than five times the reporting limit.

14.1.6 Matrix Spike Duplicate (MSD)

A second matrix spike (MSD), is treated exactly the same as the first matrix spike (MS) through preparation and analysis. The RPD between the recovery values is a measure of the precision of the analytical method.

$$RPD = 100 \times | MSD - MS | / [(MSD + MS) / 2]$$

14.1.7 Interference Check Sample (ICS)

A sample with known concentrations of elements used to determine if the inter-element correction factors of the ICP are valid.

14.1.8 Initial Calibration Verification (ICV)

A standard usually made from a different source than the calibration standards. It is analyzed immediately after the calibration to determine the validity of the calibration standards.

14.1.9 Continuing Calibration Verification (CCV)

A calibration standard analyzed after every ten samples, and at the end of an analytical sequence to verify that the calibration is still valid.

14.1.10 Initial Calibration Blank (ICB)

A matrix matched deionized water sample ran to prove the system is clean with no carry-over.

14.1.11 Continuing Calibration Blank (CCB)

A matrix matched deionized water sample ran to prove the system is clean with no carry-over.

14.2 Control Charts

Control charts are created using Element (LIMS) for method blanks, duplicates and matrix spikes. The process is defined in SOP SVL 1022. A standard X bar control chart is used to plot MS and DUP RPD results. Upper and lower warning limits of ±2s (where s equals standard deviation) and upper and lower control limits of ±3s are calculated with no fewer than 20 measurements in a 6 month period. Method defaults are used when not enough points are generated during a 6 month period.

14.2.1 LCS control charts are developed using a spread sheet that incorporates all LCS data and follows the above parameters.

14.3 Acceptance Limits

Acceptance limits for quality control parameter recoveries may be set by published analytical methods, or may be calculated statistically in-house. Individual SOPs provide the accepted recoveries for each method. The procedure for calculating in-house limits is described in SOP SVL 1022.

14.4 General Frequency of Quality Control Checks

For those methods that do not have published QC requirements, SVL will use the following QC and frequency if applicable per batch of 20 samples:

Method or Instrument Blanks at a frequency of 5%.

Laboratory Fortified Blank or LCS at a frequency of 5%.

Duplicates at a frequency of 10%.

Matrix Fortified Samples at a frequency of 10%.

Continuing Calibration Verification every ten samples.

Continuing Calibration Blank every ten samples.

14.5 Uncertainty of Measurement

SVL uses control charting as a means of determining when selected parameters are out of control. Warning and unacceptable control limits are defined at 2 and 3 sigma, respectively. SVL uses its LIMS to collate the information from its database and construct control charts every six months.

Almost all approved methods used at SVL contain a section on precision and bias. Random uncertainties that are systemic cannot be determined statistically and can only be estimated by a trained analyst. Uncertainty represents a bias associated with analytical measurements. The presence and magnitude of bias can be determined by assessment of SVL's control sample results.

SVL reports out data to 3 significant numbers, with the number of decimal places determined by the sensitivity of the method.

15.0 CORRECTIVE ACTION

The SVL Corrective Action Program is defined in SVL SOP 1019.

When a QC parameter fails acceptance criteria during the course of analysis, the Analyst or Supervisor resolves the problem before reporting data. The Supervisor may arrange for service or repair of instrumentation, if needed.

Any employee may initiate a Corrective Action Report (CAR) to support the quality system. Typical reasons are the need for an SOP revision, overdue MDL study, overdue training, incorrect data reduction or review, improper instrument calibration, or incorrect analytical method.

If there is a non-acceptable result in a Performance Evaluation Sample, the QAC documents the failure as a CAR and works with the Analysts and Supervisors to discover the cause. If there are findings from an internal or external audit, the QAC issues a CAR to appropriate staff members so they can prepare a Corrective Action Plan.

15.1 Preventative Action

A "Preventative Action" is a pro-active process for dealing with a problem before it happens. It is taken to eliminate the cause of an undesirable situation in order to prevent its occurrence rather than a

reaction to the identification of a problem or nonconformity. These actions are taken to reduce the probability that a potential problem will occur. They may also include contingencies to reduce the seriousness should a future problem occur. Subjects for "Preventative Action" may be implemented to address a weakness in the Quality System that is not yet causing nonconformities and can be initiated internally or externally (client complaints). The focus for preventative actions should be to avoid creating nonconformities, but may also lead to improved laboratory efficiencies.

SVL uses a Preventative Action Report to document ideas, plans or actions whether developed internally or externally. These reports are audited at a future date to ensure that the changes sought have been effective.

16.0 COMPLAINTS

The Business Development group strives to resolve all complaints from clients regarding analytical reports or service. Client Services contacts the appropriate Director, or Department Supervisor to investigate and resolve any issues. Actions may include reanalysis of samples or an explanation of technical issues that relate to an analytical result.

16.1 If a client requests a re-analysis, a SVL Reanalysis Request Form must be filled out. The form includes: SVL Work Order, Date, Requested By, Batch, Client Receive the Data, Reason for Request, Date Reanalyzed, Analyst, Sample Numbers, Data Qualifiers Assigned, Final Review, was the Original Data Invalid and any Comments.

16.2 Reports will have a case narrative or qualifiers. Multi-analyte samples will have all of the analytes re-ran for that method. Any re-issued reports will have both values contained within the new report.

17.0 TRAINING

SVL conducts training in legal and ethical responsibilities for all staff members, different training sessions are provided to staff members annually. New employees will be given training within a week of their hire date.

SVL Management and Supervisors train staff members in laboratory safety. At a minimum this consists of an annual review of the Chemical Hygiene Plan. It also includes seminars on important safety issues throughout the year.

Staff members also receive training in the quality system and QM. At a minimum this consists of an annual review of the QM.

Department Supervisors ensure that staff is adequately trained to perform the analyses assigned to them. The process is defined in SOP SVL 1010. Training includes, as appropriate, quality control requirements, instrument operation, instrument maintenance, software operation, reading the published method, reading the applicable SVL SOPs, successful analysis of a performance evaluation sample, and completion of the Initial Demonstration of Capability (IDOC). When an IDC is not defined by the analytical method, the completion of a method detection limit study may be substituted. Upon completion of training a Demonstration of Capabilities Certificate is placed within their personal file.

SVL Management defines the required elements for training for analytical methods. A Supervisor or a fully trained analyst provides training, when possible. If no fully trained analyst exists, an analyst may learn a new analysis by reading the appropriate method and instrument manual, then performing an IDOC.

During the training period, an analyst may produce data for clients under the supervision of a fully trained analyst. The Department Supervisor or a fully trained analyst must review and sign all trainee work produced.

17.1 To document continued proficiency, an analyst must perform one of the following tasks annually:

17.1.1 Successfully analyze a blind performance sample.

17.1.2 Complete another IDOC.

17.1.3 Successfully analyze four consecutive LCSs.

18.0 ETHICS AND CONFIDENTIALITY

18.1 SVL is committed to providing its clients with accurate and defensible data and meeting all client requirements for data quality and integrity. To achieve our commitment, and as a condition for employment with

SVL, all employees agree to follow SVL's policy regarding ethics and data integrity characterized but not limited to the items listed below.

18.1.1 All reported data, including dates and times, shall represent actual values obtained and are not modified or manipulated in any manner which is not allowed for in the referenced method.

18.1.2 There will be no misrepresentation of another analyst's identity.

18.1.3 Altering the contents of logbooks and/or data sheets to misrepresent data is prohibited.

18.1.4 Altering any operating procedures or QC to make data "fit" is prohibited.

18.1.5 Failing to comply with standard operating procedures without proper documentation and approval from the appropriate supervisor and/or QAC is prohibited.

18.1.6 Any attempt to misrepresent data or events as they actually occur in the course of data production, review or reporting is prohibited.

18.1.7 Deleting files, whether electronic or hard copy of raw data that was used in a reported value is prohibited.

18.1.8 Engaging or being a party to any practice that ultimately misrepresents data or narratives in any way is prohibited.

18.2 SVL has established a zero-tolerance policy for improper, unethical, or illegal activities. Improper actions are defined as unapproved deviations from contract-specific or method-specific analytical practices. They may be intentional or unintentional. Unethical or illegal actions are defined as the deliberate falsification of analytical or quality assurance results where failed method or contractual requirements are made to appear acceptable. Some examples of improper, unethical, or illegal practices are listed below:

18.2.1 Improper use of manual integrations to meet calibration or method quality control criteria.

18.2.2 Intentional misrepresentation of the date or time of analysis.

18.2.3 Falsification of results to meet method requirements.

18.2.4 Reporting results without analysis.

18.2.5 Selective exclusion of data to meet quality control criteria (dropping calibration points).

18.2.6 Unwarranted manipulation of computer software.

18.2.7 Improper alteration of analytical conditions (changing voltages or run times).

18.2.8 Misrepresentation of quality control samples (not preparing them as samples).

18.2.9 Intentionally reporting results from one sample for those of another.

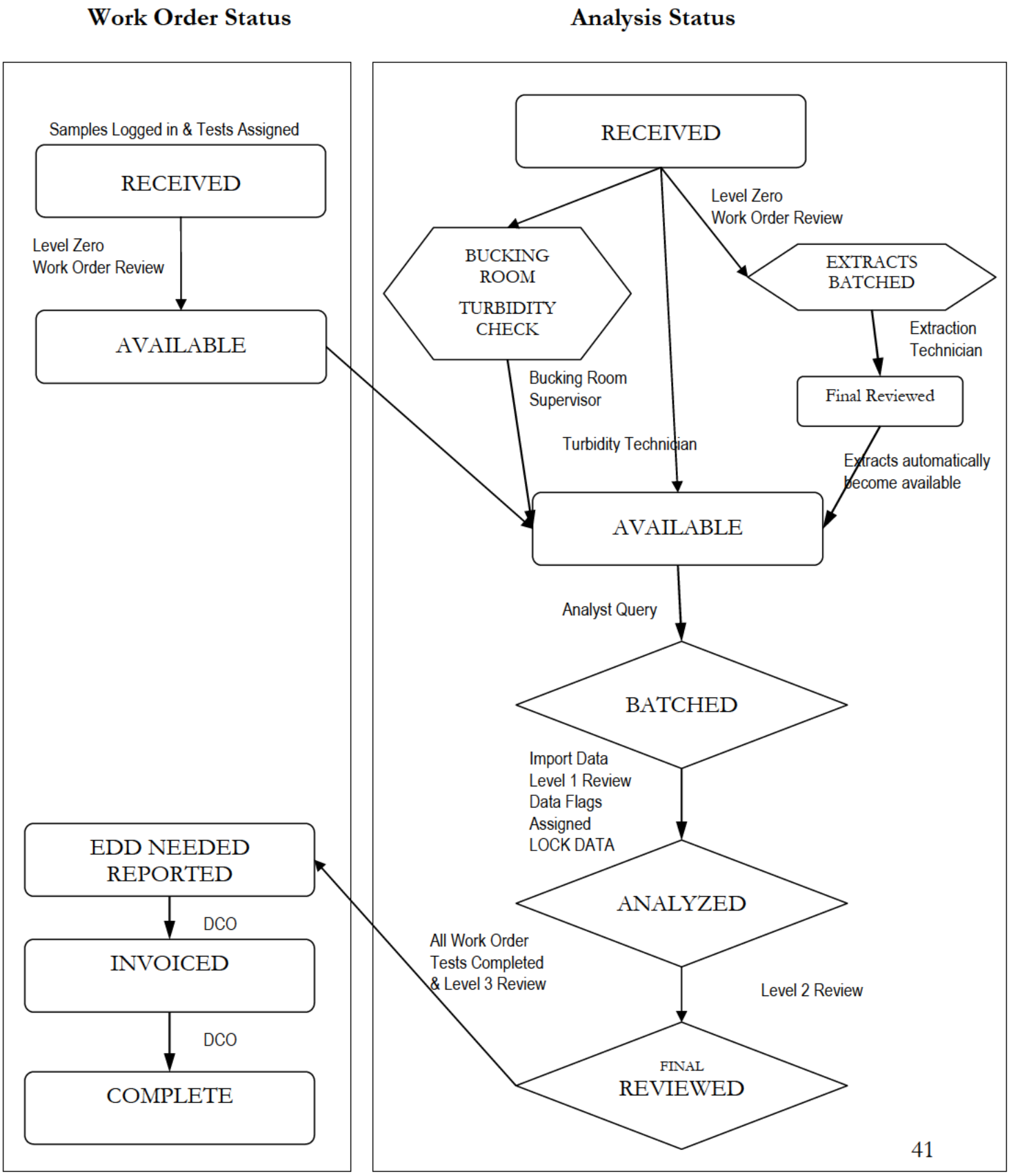
18.2.10 Reporting calibration or quality control data not linked to the reported samples.

19.0 DATA REVIEW

SVL uses a three-tier system for data review via the LIMS system. The first level is conducted by the analyst, the second level by a peer or supervisor, the third by a signatory, DCO, Technical Director or the Laboratory Director. Reviews take place within the LIMS system (which uses a system of locks to assure data is secure from accidental corruption). The process is governed by SOP SVL 2009.

In the case that erroneous data does leave the lab, the Laboratory Director or Client Services will contact the affected clients as soon as all of the facts are available. SVL will work with the clients in seeking a new or alternative strategy to meet the client's needs.

19.1 Data Review Flow Chart



20.0 REPORTING

SVL has a single standard report format for nearly all results (SVL_Sample) from ELEMENT. This includes a case narrative, sample report and QC report.

Reports are available in a number of routine and custom hardcopy formats. Electronic Data Deliverables (EDD) can be provided in ASCII, spreadsheet, and database formats, including EQWin, GIS/Key, and EnviroData Solutions. If a client has a specific format, we are usually able to provide data that will merge into their previous records.

Data that will be used to create EPA CLP-like deliverable packages are loaded into a third party data review and reporting system (MARRS) that generates all the forms required for a full data package. SVL has the capability of providing both hardcopy and EDDs. EDDs are available in standard EPA CLP formats, as well as popular spreadsheet and database files.

21.0 AUDITS

21.1 Performance Evaluation Program

SVL participates in two WS, two SOIL, and two WP Performance Evaluation Studies each year. SVL submits the second WP Study to meet the DMRQA requirements of our clients. The PE samples are logged in as single-blinds and ran as if they were normal samples in all aspects.

21.2 Internal System Audits

The Quality Assurance Coordinator (QAC) conducts a minimum of one internal system audit per year. The audit provides an overview of the implementation of procedures and policies set forth in the laboratory's Quality Manual and SOPs. System audits (that may be limited in scope) may be undertaken at any time in response to external audits, corrective actions, or at the request of the Laboratory Director.

The QAC prepares an internal audit plan based on information garnered from previous audits both internal and external, Corrective Action Reports (CARs), method changes, new instrumentation and requests or complaints from clients. The internal audit plan may define participating auditors, any applicable documents, the audit schedule, and

scope of laboratory activities to be audited. The QAC may use written checklists and/or quizzes to assess the analyst's knowledge of the Quality Manual, methods and current SVL SOPs.

The QAC will interview the analyst(s) and conduct reviews of records, logbooks, and data packages.

At the close of the audit, a post-audit meeting is held to discuss the audit findings. The auditor can close a finding during this discussion if the laboratory staff can satisfactorily demonstrate that the finding is inappropriate or easily remedied.

The QAC will deliver the report to the Laboratory Director. The report will contain at a minimum the following parameters: Date and location of the audit, personnel involved in the audit, laboratory operations audited, any minor or major findings that require corrective action (major findings require the issuance of a CAR), the auditor's summation and any quizzes taken by the analysts.

21.3 Data Audits

The QAC performs a data audit of several data packages each year. Data audits can also be triggered by audits, CARs or requests from the Laboratory Director. The purpose of data audits is to alert the QAC to any errors, chronic problems or trends that may be developing.

22.0 MANAGEMENT REVIEW

The Management of SVL conducts a review of the adequacy of the quality system and QM annually. The reviews takes into account reports from supervisory personnel, recent internal audits, external audits, the results of PE samples, changes to the volume or type of work undertaken, feedback from clients, and CARs. Conclusions are incorporated into any revisions to the QM and in improvements in laboratory operations.

23.0 SUBCONTRACTING AND PURCHASING

Prior to subcontracting work to another laboratory, the Laboratory Director or Client Services ensures that the subcontracted laboratory is NELAP accredited, or is certified by the appropriate state, for the tests to be subcontracted. Management also verifies that the laboratory has an active Quality Assurance Program (QAP) that meets SVL's and clients DQOs. This may be

accomplished by obtaining a copy of the subcontracting laboratory's QAP, or equivalent document. The Laboratory Director or Client Services advises the client that the work is being subcontracted.

SVL ensures that purchase orders contain the required technical and quality specifications prior to release. If a catalog specifies technical and quality criteria (like the grade or purity), reference to a catalog number is deemed satisfactory.

SVL tests reagents and standards prior to analyzing samples and reporting data. New reagents will be used in Method Blank and LCS preparations; if the QC requirements are met then those reagents are deemed to be acceptable. Standards will be diluted so as to fit into the current linear range of the instrument; they will be accompanied by a Method Blank and LCS to ensure that the standard is of sufficient quality and passes the grade and purity criteria as put forth by the manufacturer (SVL SOP 1015).

24.0 TRANSFER OF ANALYTICAL REPORTS, RECORDS and SAMPLES

In the event that SVL Analytical, Inc. (SVL) goes out of business or there occurs a transfer of ownership, the following plans will apply.

All current clients and past clients going back 5 years, longer if bound by contract, will be contacted by registered mail, return receipt requested, at their current or last known address, and made aware of the permanent closure or transfer of ownership of SVL.

Clients will be requested to respond in writing by return mail, fax or email within 10 business days with the instructions as to the final disposition (in the case of closure) or as to how they wish to proceed with the new ownership concerning their reports, records and/or samples, including work that is in progress.

Options for the client may include complete transfer of all reports, records and samples to their business location, or, complete destruction of all documents and samples. SVL does not take ownership of client samples at any time or under any circumstances, and title to all reports, records and samples resides with the client. SVL will not be responsible for disposal of hazardous materials.

Methods of reports and records transfer may be by hard copy purge file, hard copy reports only, or by electronic data deliverables (EDD) for all date

accessible and stored in SVL's database. No customized EDDs will be available.

Should a client decide to stay with the new ownership, any business relationship between the two parties will constitute a new relationship independent of any involvement by SVL. The maintenance of reports and records, and the completion of the work in progress (but not completed by SVL) shall be under the sole control of the new owner. SVL will be relinquished from any and all responsibilities concerning the business relationship between the parties.

25.0 GLOSSARY

Accuracy - The degree of agreement of a measured value with the true or expected value of the quantity of concern.

Aliquot - A portion of a sample.

Analytical Spike - An aliquot of sample to which a known amount of analyte has been added after sample preparation. It is a measure of the effect of the matrix of a digest or extract. It is sometimes known as a post-digestion spike.

Bias - A systematic error inherent in a method or caused by some idiosyncrasy of the measurement system. Temperature effects, extraction efficiencies, contamination, mechanical losses, and calibration errors create bias. Bias may be either positive or negative.

Blank - An artificial sample designed to monitor the introduction of contamination into the process. For aqueous samples, reagent water is used as a blank matrix.

Calibration Blank - The zero-concentration standard analyzed as part of a calibration curve.

Method Blank - Is an aliquot of analyte-free water that is put through all the steps of a specific method along with the samples. It is sometimes called a Laboratory Reagent Blank.

Field Blank - Randomly selected sample container that is filled with analyte-free water and the appropriate chemical preservative in the field.

Trip Blank - Is a specific type of field blank. A trip blank is not opened in the field. It is a check on sample contamination from the time the container is

sealed at the lab or supplier and verifies the container integrity during sample transport and during its time on site (it should always be with sampling group).

Blind Sample - A sample submitted for analysis whose concentration is unknown to the analyst.

Calibration - Comparison of an instrument response with a standard or a certified instrument. Commonly it is performed with a set of known standards plotted versus a response.

Completeness - The percentage of measurements that meet quality control acceptance criteria for requested determinations. Percentage completeness is defined by client DQOs.

Continuing Calibration Verification (CCV) - A calibration standard analyzed after every ten samples, and at the end of an analytical sequence, to verify that the calibration is still valid.

Continuing Calibration Blank (CCB) - A matrix matched deionized water sample ran to prove the system is clean with no carry-over.

Control Chart - A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

Double Blind Sample - A sample known by the submitter but submitted to an analyst in such a way that its identification as a check sample is unknown.

Duplicate Sample - Second aliquot of a sample, treated exactly the same through preparation and analysis. The RPD between the values of the duplicates is a measure of the precision of the analytical method.

Homogeneity - The degree to which a property or substance is evenly distributed throughout a material.

Initial Calibration Verification (ICV) - A standard usually made from a different source than the calibration standards. It is analyzed immediately after the calibration to determine the validity of the calibration standards.

Instrument Detection Limit (IDL) - The smallest concentration detectable on a specific instrument. It is statistically determined by analysis of at least seven replicates of a blank that has not been digested.

Interference Check Sample (ICS) - A sample with known concentrations of elements used to determine if the inter-element correction factors of the ICP are accurate.

Initial Calibration Blank (ICB) - A matrix matched deionized water sample ran to prove the system is clean with no carry-over.

Laboratory Control Sample (LCS) - A solution or material of known concentration that is analyzed to evaluate the accuracy of a method. Sometimes it is called a Laboratory Fortified Blank.

Laboratory Fortified Blank (LFB) - Another term for a laboratory control sample.

Laboratory Fortified Matrix (LFM) - Another term for a matrix spike.

Laboratory Reagent Blank (LRB) - Another term for a method blank.

Matrix Spike (MS) - An aliquot of sample to which a known amount of analyte(s) has been added. It provides information about the effect(s) of the sample matrix on the analytical method. The spike is added prior to preparation. Sometimes it is called a "Laboratory Fortified Matrix".

Matrix Spike Duplicate (MSD) - A second matrix spike, treated exactly the same as the first matrix spike through preparation and analysis. The RPD between the values is a measure of the precision of the analytical method.

Mean - The sum of all observations divided by the number of observations.

Method Detection Limit (MDL) - The smallest concentration detectable with 99% certainty on an instrument by a specific method. It is statistically determined by analysis of seven replicates of a low-level standard, prepared in the same way as a sample.

Precision - The degree of agreement of independent measurements under specified conditions.

Quality Assurance - A system of activities used to ensure defined standards of quality.

Quality Control - A system for verifying and maintaining the desired level of accuracy and precision of an analytical method.

Relative Standard Deviation (%RSD) - The Standard Deviation divided by the Mean and multiplied by 100.

Reporting Limit (RL) - The smallest concentration usually reported for an analyte. It is usually at least three times the Method Detection Limit.

Standard Operating Procedure (SOP) - A written procedure that defines a laboratory operation or analytical method.

Sub-sample - A portion taken from a sample.

Standard Deviation - Is the positive square root of the variance. A measure of the average spread around the mean.

Variance - The value approached by the average of the sum of the squares of deviations of individual measurements from the mean. Mathematically, it may be expressed as:

$$\frac{\sum (X_i - m)^2}{n} \rightarrow \sigma^2 \text{ as } n \rightarrow \infty$$

Ordinarily, only its estimate s^2 can be known.

$$s^2 = \frac{\sum (x_1 - \bar{x})^2}{n - 1}$$

25.0 CERTIFICATIONS

SVL maintains certification for analysis of drinking water in the following states:

Arizona
California
Colorado
Idaho
Montana
Nevada
Washington
Wyoming

SVL maintains certification for analysis of environmental samples in the following states:

Arizona
California
Nevada
Washington

NELAC Certification Awarded – Primary Accreditation Florida

25.1 Copies of the Scopes of Accreditation can be located at www.svl.net .

QUALITY ASSURANCE PROJECT PLAN (QAPP) ATTACHMENTS

ATTACHMENT 2
Columbia Analytical Services Supporting Documents

Carbon, Total Organic in Soil (SOP GEN-ASTM)
Selenium Analysis by Borohydride Reduction Atomic Absorption (SOP MET-7742)
Vegetation Sample Preparation (SOP MET-TISP)
Biologic-Tissue Sample Preparation and Digestion (SOP MET-TDIG)
MDL Determination (SOP ADM-MDL)
Standard Reference Materials Used During Analyses of Vegetation and Biological Tissue Samples
Quality Assurance Management Plan

STANDARD OPERATING PROCEDURE

CARBON, TOTAL ORGANIC IN SOIL

GEN-ASTM
Revision 6
October 18, 2007

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Approved By: _____ Supervisor — 10/18/07 Date

_____ QA Manager — 10-18-07 Date

_____ Laboratory Manager — 10/18/07 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: N Date: 10/1/08
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

CARBON, TOTAL ORGANIC IN SOIL

1. SCOPE AND APPLICATION

1.1. This procedure is applicable to the determination of Total Organic Carbon (TOC) using ASTM method D4129-82, modified for soil and sediment matrices (Puget Sound Estuary Program and Lloyd Kahn). Total organic carbon is a measure of the total amount nonvolatile, partially volatile and particulate organic compounds in a sample. Sample should be treated to remove inorganic carbon (carbonates, bicarbonates, free CO_2 etc.), prior to analysis, as these compounds will interfere with true readings.

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1.2. This method is applicable to all soils and sediments and most matrices that can be dried and shatter-boxed to a fine powder.

1.3. Results are reported as percent (%) carbon, and the applicable range is the MDL - 100%. The Method Reporting Limit (MRL) for TOC on soils is 0.05%, dry weight basis. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The Method Detection Limit (MDL) has been determined at 0.02%.

2. METHOD SUMMARY

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2.1. Samples are combusted in an oxygen atmosphere to convert organic and inorganic forms of carbon to CO_2. The combustion temperature is selected to completely oxidize all carbon forms. The combustion product gases are swept through a barium chromate catalyst/scrubber to ensure that all of the carbon is oxidized to CO_2. Other potentially interfering product gases such as SO_2, SO_3, HX, and NO_x are removed from the gas stream in a series of chemical scrubbers. The CO_2 is then swept to the coulometer where it is detected by automatic, coulometric titration, with coulometric end point indication.

2.2. The coulometer cell is filled with a partially aqueous medium containing ethanolamine and a colorimetric indicator. When a gas stream passes through the solution, CO_2 is quantitatively absorbed. CO_2 reacts with the ethanolamine to form a strong titratable acid which caused the indicator to fade. The titration current automatically turns on and electrically generates base to return the solution to its original color.

3. DEFINITIONS

3.1. Analysis Batch - Samples are analyzed in a set referred to as an analysis batch. The batch begins with calibration/standardization followed by QC analyses and samples. The batch ends when the QC analyses and set of samples has been completed.

3.2. Method Blank - The method blank is an artificial sample (empty boat) designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.3. Laboratory Control Sample (LCS) - A standard of known TOC concentration which is used to ensure that the analysis produces an accurate measurement of TOC in samples analyzed in the batch.

4. INTERFERENCES

4.1. Acidic and other gases, including SO_2, SO_3, H_2S, HCl, HBr, HI, Cl_2, and NO_x can be effectively removed using scrubbers such as KI, Ag_2SO_4, $AgNo_3$, and MnO_2.

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4.2. Volatile organics may be lost in the decarbonization process.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

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5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4. Disconnect teflon tubing from furnace at check valve whenever system is not in use or when O_2 flow is turned off or furnace temperature is reduced. If the carbon cathode solution should be siphoned through a failed check valve into the magnesium perchlorate scrubber potentially explosive DMSO-perchlorate could be formed.

5.5. Do not attempt to combust large samples of organic or other materials that will react with pure oxygen. Such samples can cause the pyrolysis tube to explode.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

Samples can be collected in glass or plastic containers. Samples are preserved by storage at 4±2°C. Samples are analyzed within 28 days of collection.

7. APPARATUS AND EQUIPMENT

7.1. Induction furnace, Coulometrics Incorporated.

7.2. Analytical balance, 0.1mg accuracy.

7.3. Desiccator.

7.4. Quartz combustion boats.

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7.5. Sample scoop.

7.6. Porcelain dishes.

7.7. Glass ladles and miscellaneous laboratory glassware,

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Standards

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8.1.1. Urea - 20% carbon. Use 10 μg.

8.1.2. Nutrients in Soil, purchased standard with a known TOC value (typically ERA #542). Use 50 mg for LCS.

8.2. Reagents

8.2.1. Hydrochloric acid, 50% and 10%.

8.2.2. Carbon Cathode Solution. Dimethyl Sulfoxide; DMSO. Purchased from Coulometrics Inc. as a prepared solution. Used for coulometer solution.

8.2.3. Anode Solution. Dimethyl Sulfoxide and potassium iodide. Purchased from Coulometrics Inc. as prepared solution.

8.2.4. Manganese dioxide. Gas scrubber solution.

8.2.5. Potassium Hydroxide. Gas scrubber solution.

8.2.6. Potassium Iodide. Anode chemical.

8.2.7. Magnesium Perchlorate desiccant

9. RESPONSIBILITIES

9.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

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9.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the *SOP for Documentation of Training*, is also the responsibility of the department supervisor/manager.

10. PREVENTIVE MAINTENANCE

Maintenance is performed as follows:

Maintenance Item	Frequency
Cell	Clean daily with methanol and water to clean frit
Mg Perchlorate Scrubber	change daily
KOH Scrubber	change monthly
NOX scrubber	change as needed
Repack Precombustion Column	as needed
Repack Combustion Column	as needed

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11. PROCEDURE

11.1. Sample Preparation.

11.1.1. Turn furnace on to #5 (≈1000°C). Allow furnace to warm-up for about 1/2 hours. Turn on oxygen to ≈ 5 psi and 75 to 125 ml/min at flowmeter.

11.1.2. Clean quartz boats. Scrape out old sample and rinse boats with DI water. Place boats in crucible and muffle for at least 10-15 minutes. Remove boats and place in desiccator until ready for use.

11.1.3. Samples should be dried at 70°C and homogenized prior to analysis.

11.1.4. As a rule, the darker (or closer to black) a sample is, the more carbon it contains. Place a small portion of sample on a watch glass. Add 1 drop of 10% HCl. Watch for effervescence or bubbling. If bubbles are present, the sample contains inorganic carbon (CO_3). If sample bubbles, reduce sample size to prevent sample from bubbling out of boat. If sample is dark, wood product or sludge reduce sample volume to 5 → 10mg. Normal sample volume = 50mg. After boats are loaded with sample add 1 to 2 drops 10% HCl. Place boats in 70°C oven to dry. If samples bubbled when acid was added, add 1 to 2 drops more acid and dry at 70°C. Continue acidifying and drying until samples no longer bubble. Place samples in desiccator until ready for analysis.

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11.2. Apparatus Preparation.

11.2.1. Fill cell with carbon cathode solution to 100 → 125 ml, drop in stir bar. Place cell top on snug.

11.2.2. Cover bottom of anode cell with KI. About 2 small scoops.

11.2.3. Add carbon anode solution to cell such that when anode is inserted in the anode cell, the anode solution level is the same as the cathode solution level.

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11.2.4. Place cell in coulometer cell holder.

11.2.5. Turn on detector lamp and stir plate. (Power on)

11.2.6. Turn adjust knob to 122 (all the way to the right) then turn back down to 100. Rotate cell until maximum transmittance is obtained.

11.2.7. With oxygen bubbling to cell and maximum transmittance obtained, turn on the current to the anode and cathode. The carbon cathode solution will begin to titrate to a blue color.

11.2.8. Change Magnesium Perchlorate desiccant daily.

11.2.9. The instrument is now ready to run.

11.3. Calibration and Standardization.

11.3.1. Burn both ladles for five minutes each to remove any residual TOC.

11.3.2. Establish baseline.

11.3.2.1. After placing ladles in sample inlet, allow system to purge for 1 minute.

11.3.2.2. Burn three boats empty five minutes each. The average of the three runs is the baseline.

11.4. Analysis.

11.4.1. Place one platinum or quartz boat in a ladle. Place the ladle in the sample inlet and purge for 1 minute. Simultaneously insert the sample into the furnace, press the reset button on the coulometer and start the timer for five minutes.

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11.4.2. After five minutes, obtain a reading from the instrument. Remove the ladle from the furnace. (Occasionally, a high sample may require longer than 5 minutes to complete the titration).

11.4.3. Load the other ladle with the next platinum (or quartz) boat. Remove the ladle in use from the inlet port and insert the next ladle.

11.4.4. Repeat steps 10.4.1 through 10.4.3 until all samples are analyzed.

12. QA/QC REQUIREMENTS

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12.1. Initial Precision and Recovery Validation

The precision and accuracy of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS's are prepared and analyzed. The RSD should be <20% and average recovery must be 85-115%.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Analyze a minimum of seven spiked blank replicates at a level near the MRL. Follow the procedures starting in Section 11 to analyze the samples. Refer to the CAS *SOP for The Determination of Method Detection Limits and Limits of Detection*.

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described below and in the *SOP for Sample Batches*. Project-specific batching protocols may also be required.

12.3.1. LCS - An LCS must be analyzed with each batch of 20 or fewer samples. Analyze 50mg of the purchased standard (see 8.1.2) is used. The acceptance criteria for this LCS is ± 15% of the true value.

12.3.2. Method Blank - Burn one empty boat per batch of 20 or fewer samples. Method Blank must be <0.05% carbon.

12.3.3. CCV (Continuing Calibration Verification) - A CCV must be analyzed every tenth analysis. Analyze ~10mg urea. The CCV must be 18.0% - 22.0% carbon.

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12.3.4. CCB (Continuing Calibration Blank) - A CCB must be analyzed following every CCV.

12.3.5. Sample duplicate - One sample per batch of 20 or fewer samples must be analyzed in duplicate. Duplicates should be 20% RPD, if > five times the MRL.

12.3.6. Matrix Spike - One spike must be analyzed with each batch of 20 or fewer samples. The acidified sample will be spiked with a known amount of urea.

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12.3.7. See Table 1 for a summary of acceptance criteria and corrective actions.

13. DATA REDUCTION AND REPORTING

13.1. Calculate % carbon as follows:

$$\%Carbon = \frac{(Gross\ reading\ - baseline\ \mu g)(0.1)}{mg\ sample\ analyzed}$$

13.2. For duplicate analyses, calculate relative percent difference as follows:

$$RPD = \frac{S_1 - S_2}{Avg} * 100$$

where S1 = Sample with higher value
S2 = Sample with lower value
Avg = Average of the two sample values

13.3. Calculate percent recovery as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

13.4. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified above. Average, RPD, spike level and spike recovery are entered on spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

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13.5. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

13.6. Refer to the *SOP for Laboratory Data Review Process* for general guidelines for data review.

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13.7. Reporting

13.7.1. Total organic carbon is reported as % carbon, normally on a dry weight basis. Results may be reported on an as received basis.

13.7.2. The Method Reporting Limit is 0.05% carbon, on a dry weight basis.

13.7.3. Report all results to three significant figures.

13.7.4. Bench sheets are labeled "Total Organic Carbon, TOC". These benchsheets, located in Appendix I, should be in use at all times during TOC analysis.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for Nonconformity and Corrective Action for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method is validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

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It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

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17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

17.3. This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. REFERENCES

18.1. Coulometrics Inc. Instruction Manual, Model 5020.

18.2. EPA Method Modified 415.1.

18.3. Total Organic Carbon, Method 9060, EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, September 1986, Revision 0.

18.4. Total Organic Carbon(TOC), Conventional Sediment Variables, Puget Sound Estuary Program, March 1986.

18.5. Determination of Total Organic Carbon in Sediment, Lloyd and Kahn, U.S.E.P.A Region II, July 1998.

18.6. ASTM Method D4129-88

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TABLE 1

Summary of Corrective Actions

Method Reference	Analysis	Control Item	Acceptance Criteria	Corrective Action
ASTM Method D4129-82	TOC (Soil)	Urea	±10%	Re-analyze all samples affected.
		Benzoic Acid	± 15%	Re-Analyze.
		Method Blank	<0.05%	Re-analyze. If still high, clean boats and start over.
		Sample Duplicate	20% RPD	Analyze a triplicate. Homogenize again and reanalyze.
		Sample Spike	75-125%	Re-analyze.

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APPENDIX I

BENCHSHEETS

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Columbia Analytical Services, Inc.

Service Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

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Acid Purge Time: 1 minute **Reading Time:** 5 minutes **TOC % =** $\frac{\text{(Net Reading)(µg 0.1)}}{\text{mg Sample Injected}}$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

CCV1 =97 CCV2 =

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C REC%=

Comments :

X= RSD= SPK%REC=

Analyzed By:	Date:
Reveiwed By:	Date:

TOC.XLT

Columbia Analytical Services, Inc.

Service Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

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Acid Purge Time: 1 minute **Reading Time:** 5 minutes

$$\text{TOC \%} = \frac{(\text{Net Reading})(\mu g\ 0.1)}{\text{mg Sample Injected}}$$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C % Rec =

Comments :

Analyzed By:	Date:
Reveiwed By:	Date:

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
				AVG.

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TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
CCV-1				
CCB-1				
LCS				Avg
MB				
CCV-2				
CCB-2				
CCV-3				
CCB-3				
CCV-4				
CCB-4				
CCV-5				
CCB-5				

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Columbia Analytical Services, Inc.

Service Request #: K7112 / K7040 / K7669 | Method: 9060s / PSEP (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) | Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon
CCV-1	6.8	1328.3	7.3	1321.0	19.4
CCB-1	50.0	6.8	7.3	-0.5	< 0.05
LCS-1	47.4	198.4	7.3	191.1	0.40
MB-1	50.0	7.2	7.3	-0.1	< 0.05
LCS-2	47.8	201.1	7.3	193.8	0.41
LCS-3	56.1	247.8	7.3	240.5	0.43
LCS-4	46.1	238.5	7.3	231.2	0.50
LCS-5	47.5	196.5	7.3	189.2	0.40
LCS-6	46.2	194.2	7.3	186.9	0.40
LCS-7	47.0	200.7	7.3	193.4	0.41
LCS-8	46.0	214.9	7.3	207.6	0.45
LCS-9	47.1	194.8	7.3	187.5	0.40
LCS-10	45.6	203.5	7.3	196.2	0.43
CCV2	10.7	2074.1	7.3	2066.8	19.3
CCB-2	50.0	9.3	7.3	2.0	< 0.05

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Acid Purge Time: 1 minute **Reading Time:** 5 minutes **TOC % =** (Net Reading)(µg 0.1) / mg Sample Injected

CCV : Urea Baker (lot #A17584) ID#: TOCS/1-10-J TV = 20.0%C

CCV1 = 97 CCV2 = 97

LCS: ERA Cat#: 542 Lot#: D060-542 ID#:PO3/2-26E TV = 0.34%C

Comments :

LCS 1 to 10 $\bar{x}$ = 0.42 RSD = 8%

ERA QC Performance Acceptance limits for TOC are 436 to 6370 mg/kg

MATERIAL STANDARDIZED TO 0.42%. Use as TV for this lot LWP 8-29-08

Analyzed By:		Date: 8/26/08	Time: 0900
Reveiwed By:		Date: 8/28/08	

TOC.XLT

STANDARD OPERATING PROCEDURE

SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION

MET-7742

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Revision: 2

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Approved By: _____ Supervisor — 1/9/06 Date

_____ QA Manager — 1-9-06 Date

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_____ Laboratory Manager — 1/9/06 Date

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SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION

1. SCOPE AND APPLICATION

1.1. This Standard Operating Procedure (SOP) describes the procedure used for the analysis of Selenium by borohydride reduction atomic absorption using EPA Method 7742 or Standard Method 3114B. This procedure describes the analysis procedures used to determine the analyte concentration and reporting limits listed. The sample preparation procedures are described in sample preparation SOPs MET-3010A, MET-3050B, and MET-TDIG.

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1.2. This procedure is used to determine Selenium in water, soil, and tissue matrices. The procedure may be applied to other miscellaneous sample matrices providing that the analyst demonstrates the ability of the procedure to give data of acceptable quality in that matrix. The Method Reporting Limits (MRLs) are presented in Table 1. Method Detection Limits (MDLs) that have been achieved are also given.

2. METHOD SUMMARY

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2.1. Samples are prepared according to the nitric acid digestion procedure described in Method 3010 for aqueous and extract samples, the nitric/peroxide/hydrochloric acid digestion procedure described in Method 3050 for sediments, soils, and sludges, and the nitric/closed vessel digestion for tissues. Excess peroxide is removed by evaporating samples to near-dryness at the end of the digestion followed by dilution to volume and degassing the samples upon addition of urea. The selenium is converted to the +4 oxidation state during digestion in HCl. Selenium is then converted to its volatile hydride using hydrogen produced from the reaction of the acidified sample with sodium borohydride in a continuous-flow hydride generator.

2.2. The volatile hydrides are swept into, and decompose in, a heated quartz absorption cell located in the optical path of an atomic absorption spectrophotometer. The resulting absorption of the lamp radiation is proportional to the selenium concentration..

3. DEFINITIONS

3.1. Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc...) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2. Independent Calibration Verification (ICV) - Initial calibration verification standards that are analyzed after initial calibration with newly prepared standards but prior to sample analysis, in order to verify the validity of the standards used in calibration. The ICV standards are prepared from a materials obtained from a source different from that used to prepare calibration standards.

3.3. Matrix Spike (MS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis.

3.4. Duplicate Sample (DUP) A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

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3.5. Standard Curve - A standard curve is a calibration curve that plots concentrations of a known analyte standard versus the instrument response to the analyte. A linear regression calibration model is used. The appropriate criteria for assessing the validity of the calibration curve must be followed prior to quantitation of target analytes in actual sample analyses.

3.6. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

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3.7. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.8. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into subsequent sample analyses.

4. INTERFERENCES

4.1. Very high (>1000 mg/L) concentrations of cobalt, copper, iron, mercury, and, nickel can cause analytical interferences through precipitation as reduced metals and associated blockage of transfer lines and fittings.

4.2. Traces of peroxides left following the sample work-up can result in analytical interferences. Peroxides must be removed by evaporating each sample to near-dryness

followed by reacting each sample with urea and allowing sufficient time for degassing before analysis.

4.3. Even after acid digestion, flame gases and organic compounds may remain in the sample. Flame gases and organic compounds can absorb at the analytical wavelengths and background correction should be used.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

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5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

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6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Aqueous samples are typically collected in plastic containers. Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4 ± 2°C from receipt until analysis.

6.2. Non-aqueous samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until analysis. Non-aqueous samples should be analyzed as soon as possible following sampling.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

7.1.2. Concentrated Hydrochloric Acid (HCl)

7.1.3. 4% Sodium Borohydride ($NaBH_4$). 20g $NaBH_4$ plus 2g of sodium hydroxide dissolved in 500mL of reagent water.

7.1.4. Urea (H_2NCONH_2)

7.1.5. Reagent and grade

7.2. Standards

7.2.1. Stock standard solutions may be purchased from a number of vendors. All standards purchased from vendors must be traceable to NIST or A2LA certified reference materials. Purchased standards are typically prepared at a concentration of 1000 ppm, and are prepared in 500 mL plastic bottles. The vendor-assigned expiration date is used.

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7.2.2. A 1000 ppb intermediate stock standard is prepared by pipetting 0.100 mL of 1000 ppm stock standard, plus 1.0 mL of concentrated HCl, into a 100 mL volumetric and diluting to volume with reagent water.

7.2.3. Calibration standards are prepared by pipetting 0.050, 0.100, 0.500, 1.00, and 1.50 mL of 1000 ppb intermediate stock standard into a series of five 100 mL volumetric flasks. 50 mL of concentrated HCl is added to each flask and they are diluted to volume with reagent water. The final concentrations of the calibration standards are 0.5, 1.0, 5.0, 10, and 15 ppb.

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7.2.4. The ICV is prepared at 10 ppb as described above. The 1000 ppb intermediate stock used for the ICV is prepared from a source independent of the original 1000 ppm purchased standard.

8. APPARATUS AND EQUIPMENT

8.1. Varian SpectrAA-20 Atomic Absorption Spectrophotometer.

8.1.1. Varian VGA-76 Vapor Generator

8.1.2. Quartz Absorbance Cell

8.1.3. Selenium Hollow Cathode Lamp

8.2. Digestion Hot-Block capable of maintaining 70°C

8.3. 50 mL Centrifuge Tubes

8.4. 150 mL Beakers

8.5. Hot Plates capable of maintaining 70°C

8.6. 100 mL Volumetric Flasks

8.7. (Describe other apparatus and equipment).

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, description of maintenance, date, and name. The log should contain a reference to return to analytical control.

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9.2. Typical preventive maintenance measures include, but are not limited to, the following items:

- Cleaning the nebulizer and burner head
- Cleaning the gas liquid separator
- Inspection of the hollow cathode and deuterium lamp conditions

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10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in Methods 7742 and 3114B, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water samples are first prepared by EPA Method 3010A (CAS SOP; MET-3010A), soil samples by EPA Method 3050B (CAS SOP; MET-3050) with modifications noted below, and tissue samples by closed vessel digestion (CAS SOP; MET-TDIG).

Note: The 3050B soil digestion is modified as follows: After the final peroxide addition (i.e. before the final reduction stage) add 5.0mL of concentrated hydrochloric acid and reduce the digestate volume to less than 5.0mL, but not to dryness. After cooling, dilute the digestate to 100mL with reagent water.

11.1.2. The sample digestates are then prepared for hydride analysis by aliquoting 10 mL of the digestate into a 50 mL centrifuge tube. For soil digestates 0.200 g of Urea is next added. Finally, 10 mL of concentrated HCl is added. The centrifuge caps are loosely place on the tubes and the samples are heated in the digestion hot-block at 70°C of 30 minutes. After cooling the sample volumes are diluted to 20 mL as needed.

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11.2. Standard Preparation

11.2.1. Working standards are prepared in 100 mL volumetric flasks by aliquoting 0, 0.05, 0.1, 0.5, 1.0, and 1.5 mL of 1000 ug/ L intermediate standard into 150 mL beakers containing approximately 30 mL of reagent water. The appropriate amounts of concentrated HCl and/or HNO_3 are added in order to replicate the matrix of the initial 3010A, 3050B, or tissue digests. An additional 50 mL of concentrated HCl is added and the beakers are covered with a watchglass and heated on a hotplate at 70°C of 30 minutes. After cooling the standards are quantitatively transferee to 100 mL volumetrics and diluted to volume with reagent water.

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11.2.2. An Independent Calibration Verification (ICV) standard is prepared by aliquoting 1.0 mL of a 1000 ug/L intermediate standard prepared from a different source than the intermediate used for the calibration standards. This standard is then processed as described above.

11.3. Instrument Setup

Varian-1 is used in conjunction with the VGA-76 hydride generator, an external peristaltic pump, and burner head mounted quartz cell. The sample and blank (50% HCl) flows are set at 4.2 mL/min and the borohydride (0.5% Sodium Borohydride) flow at 2.1 mL/min. The instrument is optimized to wavelength of 196.0 nm, with a slit width 1.0, and D_2 background correction turned on. The burner is ignited and the system is allowed to warm up for 10 minutes prior to starting the analysis.

CAUTION: The hydride of selenium is very toxic. Precautions must be taken to avoid inhaling the gas.

11.4. Calibration and Analysis

11.4.1. The sampling tube is placed into the calibration blank, allowed to come to equilibrium, then analyzed. The remaining calibration standards are analyzed similarly in ascending order. After the calibration curve is complete the r value is calculated. If the r value is not $\geq$ 0.995 then the calibration is rejected and must be re-analyzed.

11.4.2. Immediately following the calibration the ICV solution is analyzed followed in order by an Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), Continuing Calibration Blank (CCB), and the MRL check standard (i.e. the low calibration standard). ICV and CCV recoveries must fall within ± 10% of their true value, and ICB and CCB results must be less than the 0.5 ug/L.

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11.4.3. Once the above calibration and QC check standards have been successfully run the samples are ready to be analyzed. After a maximum of 10 samples have been analyzed an additional set of CCV and CCB standard must be analyzed using the criteria described above. If one or both of these standards are out of control the problem must be corrected, the instrument recalibrated, the initial QC check standards analyzed, and the samples following the last compliant CCV/CCB check re-analyzed.

11.5. List any matrix modifiers or reducing agents used in the analysis on the associated raw data

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12. QUALITY CONTROL

12.1. Initial Precision and Recovery Validation

The precision of the procedure must be validated before sample analysis, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (water or soil) samples at a level near, or below the MRL. Follow the analysis procedures in Section 11 to analyze the samples.

12.2.2. Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to the CAS *SOP for The Determination of Method Detection Limits.* The MDL study should be done annually.

12.3. Method Blank

A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis.

12.4. Lab Control Sample (LCS)

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12.4.1. The laboratory control sample for water samples is composed of analyte-free water into which Selenium is spike. The laboratory control sample for soils and tissues consists of an appropriate reference material (e.g. ERA Lot 246 for soils, NRCC Dolt-3 for tissues). The LCS is designed to monitor the accuracy of the procedure. The concentration of the spike in the LCS matrix should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

12.4.2. A lab control sample (LCS) must be prepared and analyzed with every batch of 20 (or fewer) samples. Calculate the LCS recovery as follows:.

$$\%R = X/TV \times 100$$

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Where X = Concentration of the analyte recovered
TV = True value of amount spiked

The acceptance criterion for waters is 85-115%. Acceptance limits for other reference materials is specific to the material used. If the LCS fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis.

12.5. Matrix Spike

A matrix spike (MS) must be prepared and analyzed with every batch of 20 (or fewer) samples. The MS is prepared by adding a known volume of the matrix spike solution to the sample and determining the spiked sample concentration. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

The acceptance limits for the MS are 75-125% for waters, 60-130 for soils and tissues. If the MS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or reextraction and reanalysis.

12.6. A Duplicate sample is prepared and analyzed one per batch, or per 20 samples. Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{|R1 - R2|}{(R1 + R2) / 2} \times 100$$

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Where R1= result for the sample
R2= result for the sample duplicate

The RPD criteria is <20% for water samples and <30% for soils. If not, redigest the sample batch. Determine if the sample is non-homogenous and redigest if it is homogenous.

12.7. Prior to preparation of samples, blanks should be analyzed to determine possible interferences from sample handling steps, reagents, or glassware. If the blanks show contamination, the source of the contamination should be isolated and minimized.

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13. DATA REDUCTION AND REPORTING

13.1. The concentration of the analyte(s) in the sample digest (Cex) is calculated using the calibration curve. The concentration of analytes in the original samples is computed using the following equations:

Aqueous Samples:

$$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where Cex	=	Concentration in digestate in µg/mL
Vf	=	Final volume of extract in mL
D	=	Dilution factor
Vs	=	Volume of sample digested, liters

Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

Where	Cex	=	Concentration in digestate in µg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	W	=	Weight of sample digested in grams. The wet or dry weight may be used, depending upon the specific client requirements.

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13.2. Sample concentrations are reported when all QC criteria for the analysis has been met. Reported results not meeting QC criteria must be qualified with a standard CAS footnote.

13.3. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details.

13.4. Reporting

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13.4.1. Reports are generated in the MARRS or Harold reporting softwares by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). The compiled data is also used to create EDDs.

13.4.2. As an alternative, reports are generated using Excel© templates located in R:\ICP\FORMS. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The results are then transferred, by hand or electronically, to the templates.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

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It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

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17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. TRAINING OUTLINE

18.1. Review literature (see references section). Review the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.2. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of approximately two weeks. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file.

19. REFERENCES

19.1. *EPA Method 7742, Revision 0, 1994*

19.2. *Standard Method 3114B, 20th Edition*

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TABLE 1

TARGET ANALYTES, MRLs, and MDLs

Analyte	Method Detection Limit		Method Reporting Limit	
	Water (ug/L)	Soil (mg/kg)	Water (ug/L)	Soil (mg/kg)
Selenium	0.3	0.02	1.0	0.1

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STANDARD OPERATING PROCEDURE

for

TISSUE SAMPLE PREPARATION

SOP Code: MET-TISP
Revision: 6

December 31, 2009

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Approved By: _____ Supervisor — 12/18/09 Date

_____ QA Manager — 12/18/09 Date

_____ Laboratory Manager — 12/18/09 Date

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Standard Operating Procedure for Tissue Sample Preparation

1. PURPOSE

This standard operating procedure describes procedures for the initial preparation of tissue samples prior to sample analysis. Customer-specific contracts or statement of works (SOWs) with alternate procedures will take precedence over this SOP.

2. APPLICABILITY

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This SOP is intended to provide guidance for the preliminary preparation of tissue samples prior to the sample aliquoting and analytical preparation described in individual analytical SOPs. The procedures described in this SOP also apply to compositing and subsampling of tissue samples for analyses to be subcontracted.

3. DEFINITIONS

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3.1. Sample: The material presented to the laboratory for analysis or testing.

3.2. Sample Aliquot: A representative part or portion of a sample for analysis which is a fraction of the whole sample. See subsampling also.

3.3. Compositing: The process by which sample aliquots from two or more samples are united to form a combined sample which is subsequently analyzed.

3.4. Subsampling: The process by which a representative portion is obtained from a whole sample.

3.5. Service Request: The service request (SR) is a document prepared at the time of sample receipt and summarizes sample analysis and reporting instructions about a customer's sample(s).

3.6. QAPP: Quality Assurance Project Plan document provided by the client specifically written for their project.

3.7. VOC Analyses: Volatile organic compounds (VOC) analyses, including halogenated and aromatic volatile organic compounds and gasoline range organics (GRO) analyses.

3.8. Non-VOC Analyses: Any analysis other than a VOC analysis.

4. DISCUSSION

4.1. Tissue samples are inherently heterogeneous requiring special considerations in order to obtain a truly representative sample aliquot for analysis. This SOP provides guidance for handling tissue samples prior to the sample preparation steps described in analytical SOPs. This SOP applies to samples delivered to the lab in whole body form or in the form of pre-dissected tissues.

4.2. The sample handling strategy must consider:

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- what analyses are to be performed (metals, organics, or both, and VOC or non-VOC),
- how much sample is available
- are the analyses to be performed on individual samples or composite homogenates,
- are the analyses to be performed on whole body, edible portions or specific organs, and
- are any of the analyses going to be subcontracted which may require subsampling.

4.3. Proper preparation and handling of tissue samples is required to obtain a representative sample, avoid contamination, and to ensure loss of sample and target constituents is minimized.

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5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

Refer to the determinative method.

7. APPARATUS AND EQUIPMENT

Note: Refer to the Procedure section for specific equipment used based on the determinative analysis to be performed. The use of implements and surfaces may vary depending on the analyses to be performed.

7.1. Hobart Food Chopper, or comparable device.

7.2. Tissumizer.

7.3. Waring blender, or similar device.

7.4. Freeze-drier, Labonco or equivalent.

7.5. Glass or PTFE cutting boards.

7.6. Knives and cutting implements – refer to Procedure section.

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7.7. Standard laboratory glassware (beakers, scintillation vials, etc.)

7.8. VOA vial – pre-cleaned, 40ml with Teflon-lined cap.

7.9. Pre-cleaned glass jars with PTFE lined lids, various sizes.

7.10. Gloves: Should be talc free and of non-contaminating materials.

7.11. Heavy duty aluminum foil.

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8. RESPONSIBILITIES

8.1. Sample custodians, together with project chemists and department supervisors, are responsible for documenting any required sample preparation (including the percent solids or percent lipids determination if required) on the service request. All personnel preparing tissue samples should be familiar with the contents of this document prior to commencing work.

8.2. Tissue sample preparation is to be performed only by lab analysts instructed in the proper handling techniques outlined in this SOP. It is the responsibility of the analyst to perform this procedure to complete all documentation required for data review.

9. PREVENTIVE MAINTENANCE

9.1. No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.

9.2. For organics samples, polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials.

10. PROCEDURE

10.1. Sample Login

Any special sample handling must be noted on the service request and on a label attached to the sample itself. During sample receiving, a sample custodian must follow the procedures described in Section 10.1.1.

10.1.1. Tissue Samples with Limited Quantity

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An assessment of the required sample quantity should be made by the project chemist when taking delivery of the sample. This assessment must take into consideration the tests, the required detection limits and the necessary quality assurance samples. If the quantity of sample given to the laboratory is insufficient for the analyses requested, the sample custodian will, along with the project chemist estimate the total amount of sample available. A "LIMITED SAMPLE VOLUME" tag is attached to the sample on which is recorded the estimated sample quantity.

In some cases it may be beneficial to perform sample preparations as described in this SOP prior to estimating the sample amount. In this case, the analyst prepring the sample will provide the project chemist with an estimate of the amount available.

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The project chemist must determine if limited sample quantity exists and set the priorities for the analyses and, if possible, estimate the quantity of sample to be *used for each test*. This information is to be documented and placed in the project file and on the service request to communicate to the laboratory staff. For example, 8080 use 10 g; metals use 1g.

NOTE. Samples that are quantity limited and require multiple analyses must be identified as soon as possible. Optimally, this should happen during sample login; however, discovery at any time should trigger appropriate actions as described in Section 10.1.1.

10.2. Sample Homogenization

This section outlines the steps for preparing homogenous samples of whole fish, edible fish (fillets), edible shellfish, worm composite homogenates, eggs, and plant tissues.

10.2.1. Samples for Organics Analyses:

Equipment used for the processing of tissue samples for organics analyses should be of stainless steel, anodized aluminum, glass or polytetrafluorethylene (PTFE).

Polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials. Filleting should be done on glass or PTFE cutting boards that are cleaned properly between samples or on cutting boards that are covered with heavy duty aluminum foil (hexane rinsed) that is changed between samples. Tissue should be handled with precleaned, high quality, corrosion-resistant stainless steel instruments. Fillets or homogenate should be stored in cleaned glass jars of suitable dimensions with PTFE lined lids. If the sample is to be analyzed for VOCs, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40ml voa vial and labelled "FOR VOA ANALYSIS ONLY".

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Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, hexane, and DI water. Precleaned, certified sample containers may be used without further cleaning. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone and the hexane rinsing of the aluminum foil. Exposure to solvent vapors must be minimized.

10.2.2. Samples for Metals Analyses:

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Equipment used in the processing of samples for metals analyses should be of PTFE, ceramic, polypropylene or polyethylene. Filleting should be performed on PTFE cutting boards which are cleaned after each sample. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Tissue should be stored in glass jars with PTFE lined lids.

Prior to sample handling, utensils, cutting boards and containers should be washed in a detergent hot water solution, rinsed with tap water, 25% HCL (except metal utensils), and DI water. Precleaned, certified sample containers may be used without further cleaning.

10.2.3. Samples for both Metals and Organics Analyses:

If the sample is to be prepared for both organics and metals, care must be taken to use equipment and cleaning procedures that are non-contaminating for both.
Quartz, ceramic, glass and PTFE are recommended materials for sample processing equipment. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Glass or PTFE cutting boards should be used.

If the sample is to be analyzed for VOC's, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40ml voa vial and labelled "FOR VOA ANALYSIS ONLY".

Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, acetone, methanol, or hexane (as appropriate), and DI water. Precleaned, certified sample containers may be used without further cleaning. Non-metallic surfaces and utensils should also be rinsed with 25% HCL followed by DI water. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone. Exposure to solvent vapors must be minimized.

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10.2.4. Sample Preparation

Each tissue sample may be homogenized in the original glass bottle container if there is sufficient space to allow thorough mixing. If homogenization is not achievable in the original container, place the entire sample contents into a clean glass jar. Generally, liquids contained in the container are to be considered part of the sample. If the sample requires size reduction prior to homogenization, chop the sample into the 1-2" chunks using a titanium or stainless steel bladed knife. Large samples may require the use of industrial food processors such as a Hobart Food Chopper, or comparable device. Size-reduced chunks of tissue are thoroughly homogenized to a paste-like consistency using a Tissumizer, Waring blender, or similar device until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

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10.2.4.1. Whole Fish Tissue

Samples may be frozen in the field or in the laboratory. While still partially frozen, rinse the fish with DI water to remove extraneous materials and liquids. Cut the fish into appropriate size chunks and mechanically macerate the sample using cutting tools appropriate for the size of the sample and the analysis type. If necessary, process fish tissue chunks through the Hobart Food Chopper. To ensure thorough mixing, divide the ground sample into quarters, mix opposite quarters and then mix halves. Homogenize sample using a Tissumizer or Waring blender until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

10.2.4.2. Edible Fish Tissue

If the client or QAPP indicates that only edible tissue be analyzed, the fish must be filleted. If the sample arrives prefilleted, the sample tissue may be frozen before processing. If the sample is not yet filleted, the sample should remain chilled until the filleting is completed. Freezing can result in the contamination of edible tissues from the bursting of internal organs. Fish having ruptured internal organs should be noted on the prep benchsheet and the Project Chemist consulted. Rinse the fish with DI water to remove extraneous materials and liquids. Remove scales from scaled fish or skin from non-scaled fish. Rinse the fish again prior to filleting. A separate or clean cutting board should be used for filleting. Gloves should be changed between samples. Carefully remove the fillets from the carcass by following the steps outlined in Appendix A. Care should be taken to avoid contaminating fillet with inadvertent puncture of internal organs. Cut the fillet tissue into appropriate size chunks and mechanically macerate the sample using cutting and grinding tools appropriate for the size of the sample and the analysis type. Proper selection of maceration equipment must consider the potential contaminants, sample size/volume and amount of tissue likely to be lost in using the equipment.

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Divide the ground sample into quarters, mix opposite quarters and then mix halves. Again homogenize the sample using an appropriate blending mixer. Continue repeating this process until the sample is truly homogenous and no chunks of tissue remain. Freeze sample until ready for extraction.

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10.2.4.3. Shellfish Tissue

Shellfish should be frozen as soon as possible after receipt by the laboratory unless samples can be prepared within 48 hours of sampling. Edible portions of various shellfish are described below and resection described in Appendix B. Thawing of frozen shellfish samples should be kept to a minimum during tissue removal to avoid loss of liquids. Shellfish should be rinsed with DI water prior to tissue removal to dislodge external debris. When multiple organisms constitute a single sample, the edible tissues are collected, composited and homogenized.

10.2.4.3.1. Bivalve mollusks (oysters, clams, mussels, and scallops).

Bivalves are typically prepared by severing the adductor muscle, prying open the shell, and removing all of the soft tissue. The soft tissue includes viscera, meat, and body fluids.

10.2.4.3.2. Crabs

Edible tissue includes all leg and claw meat, back shell meat and body cavity meat. Internal organs generally are removed. If the crab is soft shelled, the entire crab is used in the sample.

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10.2.4.3.3. Shrimp and Crayfish - Edible tissue includes the tail meat.

10.2.4.3.4. Lobster - Edible tissue includes the tail and claw meat.

10.2.4.4. Worms

Samples are typically supplied to the lab in sample jars containing multiple organisms. Liquid and specimens constitute the entire sample and are blended together typically in the sample container. When a worm sample containing dirt particles or significant amounts of water is encountered, the technician should contact the project chemist to seek guidance from the client.

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10.2.4.5. Eggs

Avian eggs are typically removed from the shell and blended. Aquatic eggs are blended including the soft shell.

10.2.4.6. Internal Organs Extraction

Organs such as livers or kidneys must be identified and removed by an experienced sample technician following clear written resection procedures or other guidance provided by the client.

10.2.4.7. Plant Tissue

Plant tissue should be handled using the size reduction, homogenization and implement cleaning steps outlined in Sections 10.2.1, 10.2.2, 10.2.3, and 10.2.4. Where these procedures are inappropriate, specific written procedures or guidance from the client is recommended.

If drying is requested by the client or is project-specified, a subsample for mercury analysis is taken from the wet sample, then the plant tissue is dried at 60°C prior to homogenization.

10.2.4.8. Small Mammals and Rodents

10.2.4.8.1. There are two primary concerns in working with small mammals and rodents: safety and sample homogenization.

10.2.4.8.2. Small mammals are potential carriers of lethal viruses, such as hantavirus and rabies, and bacteria that can be contracted through inhalation or direct contact. Typically, these organisms are excreted in the feces and distributed on the air as the fecal matter dries. During the sample preparation process, tissue is typically freeze-dried in order to calculate a percent solids value and to analyze for metals. As such, it is possible to increase the potential for dispersion of the bacteria or viruses after the sample is homogenized and processed. Prior to processing, all samples should be stored frozen and shipped to a subcontractor for irradiation. Minimum irradiation dosage is 3 - 4 Mrads. The samples should be frozen and shipped next day priority in an insulated cooler packed on ice or dry ice. Use enough ice to ensure the samples remain frozen until return of the samples (use a three day time line in estimating the amount of ice needed to ensure samples remain frozen). Care should be take to minimize air space in the cooler thereby maximizing insulating potential. It is recommended that the cooler be chilled prior to packing.

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10.2.4.8.3. Prior to sample homogenization, instructions should be received from the client regarding the processing of the hide. For organics, it is recommended that the hides be left on the carcass and the entire sample be homogenized. For metals, there is a potential for accumulation in the hair. As a non-digestible portion of the rodent, inclusion of the hair may result in a high bias if the data is to be used in estimating bioaccumulation up the food chain. Skinning may be a preferred alternative when metals are the primary chemicals of concern.

10.2.4.8.4. Homogenization should be done while the carcass is still partially frozen.

10.2.4.8.5. If the hide is to be included in the homogenization, snip the feet from the animal using stainless steel scissors.

10.2.4.8.5.1. The tail should be removed if it will prevent complete homogenization of the sample (e.g., the tail of a mouse or rat may result in incomplete homogenization and should not be included with the sample). Remove seeds, grasses, and mud from the hide.

10.2.4.8.6. If the hide is to be removed from the carcass, make an incision through the skin on the back of the neck (do not cut into the muscle). In most cases, the hide can be removed by pulling the incision horizontally along the back in one direction, and over the ears, head and snout in the opposite direction. The eyes are usually lost during this procedure. Continue to skin the animal by peeling the hide over the hind legs, off the underside of the animal, and around the front legs. The hide is removed at the hind legs and the snout. Care should be taken not to tear the connective tissue under the hide. Fat should be scraped from the hide when possible and included with the sample. Rinse the skinned carcass with DI water to remove any hair or dirt that has accumulated during the skinning procedure.

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10.2.4.8.7. Homogenize the sample using a stainless steel Waring blender. Select a blender cup that is sized in accordance with the amount of sample to be homogenized. That is, small samples should be homogenized using small blender cups. This will improve the overall homogenization and recovery of the sample. Continue to mix the sample into a paste like consistency. Make sure no chunks of muscle, hide, or bone are distributed in the sample. Transfer the sample paste to a glass jar for storage and freeze. The new sample container is labeled "homogenized", initialed, and dated.

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10.2.5. General Provision for Handling Large Sample Mass

In some cases, large specimens will be received by the laboratory for homogenization prior to chemical analysis. For the purpose of this SOP, 'large' is defined as requiring preliminary size reduction to allow sequential processing of the sample. Sub-samples of the whole specimen should be cut to a size appropriate for the blender, mixer, or grinder that will be used. After each individual fraction is processed, the homogenized material is added to a reservoir large enough to hold all fractions as they accumulate. The reservoir will be constructed of a material suitable for the analytical application as defined under Section 10.2.3. For very large specimens (i.e. >20 pounds), high grade stainless steel containers are used (large bowls or small drums).

Blending of the combined fractions to achieve a whole homogenous material is achieved via manual mixing. In general, this is accomplished using a high grade stainless steel paddle or spoon of appropriate size (i.e. relative to the whole homogenate). Very large specimens (i.e. >20 pounds) generally require secondary processing through the grinder, particularly when large amounts of skin, bone, and/or cartilage is present. In these cases, the Hobart grinder is generally used.

10.3. Compositing

Each sample is to be logged in and receive a lab code. Additionally, the sample composite also is assigned a lab code. The compositing process is to be performed by trained staff. It is to be performed in an area free of contamination. It is imperative that the samples are treated in a manner consistent with the requirements of the tests to be performed on the composited sample. Compositing of homogenates should be performed according to this SOP or specific instructions provided by the client.

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10.3.1. Documentation

The analyst preparing the composite will document

- that homogenization was done before removing an aliquot,
- the quantity of each (field or discrete) sample used for the composite,
- the date and time of compositing, and
- any other pertinent observations.
- An example of a data sheet that can be used for this purpose is given in Appendix C.

10.3.2. Tissue Samples with Limited Quantity

Samples and sample composites that are quantity limited will be handled by the same procedure as described in Section 10.1.1

10.3.3. Compositing Procedure

10.3.3.1. Each tissue sample is first homogenized as per instructions in Section 10.2.

10.3.3.2. An equal weight of sample aliquot from each of the homogenized samples is weighed into a clean glass sample bottle. The amount to be weighed of each sample will depend upon the number of analyses to be performed on the composite and if the quantity of any individual sample is limited.

10.3.3.3. The mixture of the individual sample aliquots is thoroughly homogenized in the glass container. The composite sample bottle is labeled with:

- the name of the composite,
- the lab code of the composite,
- the analyst's initials
- the date of composite preparation
- The composite sample and the remaining (individual, discrete) samples are stored frozen until analysis.

10.3.4. Tissue Samples Requiring VOC Analyses

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A separate aliquot of the composite homogenate should be placed in 40ml voa vial container for later analysis by the VOA department. Each container should be labelled with the lab identifier, date, initials, and "FOR VOA ANALYSIS ONLY". To minimize losses of volatile constituents, the sample should be kept as cold as possible, the work should be completed as quickly as possible, and the VOA vial filled to the top to minimize head space.

10.4. Subsampling

The sample is first thoroughly homogenized as per Section 10.2. A sample aliquot is removed and placed into a clean glass container of appropriate size and labelled as follows:

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- the name of the sample,
- the lab code of the sample,
- "homogenized" written on the label,
- the purpose of the sub-sample (e.g. "dioxin subsample")
- the analyst's initials
- the date.

10.5. Freeze-Drying

10.5.1. Depending on project specifications, samples may require freeze-drying. Freeze-drying may be performed on a separate portion of sample to determine % Freeze-Dried Solids, or may be done on the analytical subsample for certain tests. The analyst should obtain direction from the supervisor and/or Project Chemist.

10.5.2. Weigh 5-8 g of sample (wet weight) into a scintillation vial. Freeze the sample for at least 2 hours.

10.5.3. Remove the sample from the freezer and place in the freeze drier for at least 24 hours, or longer if necessary for the particular sample matrix.

10.5.4. Record the measurements on the benchsheet in Appendix C.

11. QA/QC REQUIREMENTS

11.1. A rinsate blank should be prepared to accompany each batch of tissue samples. The blank is comprised of a collection of DI water rinses of cleaned equipment (knives, cutting boards and mixers/grinders) **prior** to the commencement of sample batch preparation. If contamination of the samples is suspected, the rinsate blank is extracted and analyzed for contaminants. The rinsate blank should be labeled with the extraction date and the associated SR numbers and stored at 4 C. In the event that contamination is suspected, the rinsate blank can be analyzed to confirm the presence of contaminants in the tissue preparation process.

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11.2. A homogenization blank is prepared to determine if the homogenization equipment was effectively cleaned between samples. Some project quality plans may require homogenization blanks between each sample. Following the blending of a tissue sample decontaminate the Hobart mixer (model HCM62) by following these steps:

- Wash the bowl, blade assembly, and lid with soap and hot water.
- Rinse all parts with dionized water.
- Move to fume hood and hexane rinse all parts.
- Allow excess hexane to evaporate.

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11.2.1. Reassemble the mixer and make ready for the next sample.

11.2.2. Fill the bowl with dionized water and turn the mixer on for the approximately average time used for the type of samples being processed.

11.2.3. Aliquot the dionized water to bottles appropriate for the testing being conducted and preserve accordingly. If insufficient sample volume is produced for the required testing, repeat the procedure after the next tissue sample is homogenized.

12. RECORDS

Sample handling documentation must include information about sample homogenization (was it done or not), compositing, and subsampling. The data benchsheets in Appendix C provide a means for recording this information. This sample handling information is to be filed in the project file with the raw data.

13. REFERENCES

Kateman and L. Buydens, *Quality Control in Analytical Chemistry*, Second Edition, John Wiley & Sons, Inc., New York, NY, 1993: Chapter 2 on Sampling and especially sections 2.5 (Sample Quality) and 2.7 (Handling of Samples).

Guidance For Assessing Chemical Contaminant Data For Use In Fish Advisories; Volume 1; Fish Sampling and Analysis, 3rd Edition; USEPA Office of Water; EPA 823-B-00-007; Nov 2000.

Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound; Tetra Tech, Inc.; final report TC-3991-04 Recommended Guidelines for Measuring Organic Compounds in Puget Sound Sediment and Tissue Samples Revision April 1996.

PCB's and Mirex In Fish Tissue and Clams New York State Department of Health Wadsworth Center For Laboratories and Research; Albany, N.Y. 10/6/81

Draft Method 1613-Tissue; Determination of PCDDs and PCDFs in Fish and Other Tissue Using Method 1613; USEPA Office of Water June 1993.

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14. CHANGES SINCE THE LAST REVISION

14.1. Reformatted section 3.
14.2. Sec 4.1 corrected spelling.
14.3. Sec 11.2 is new.

APPENDIX A

Fish Filleting Procedure

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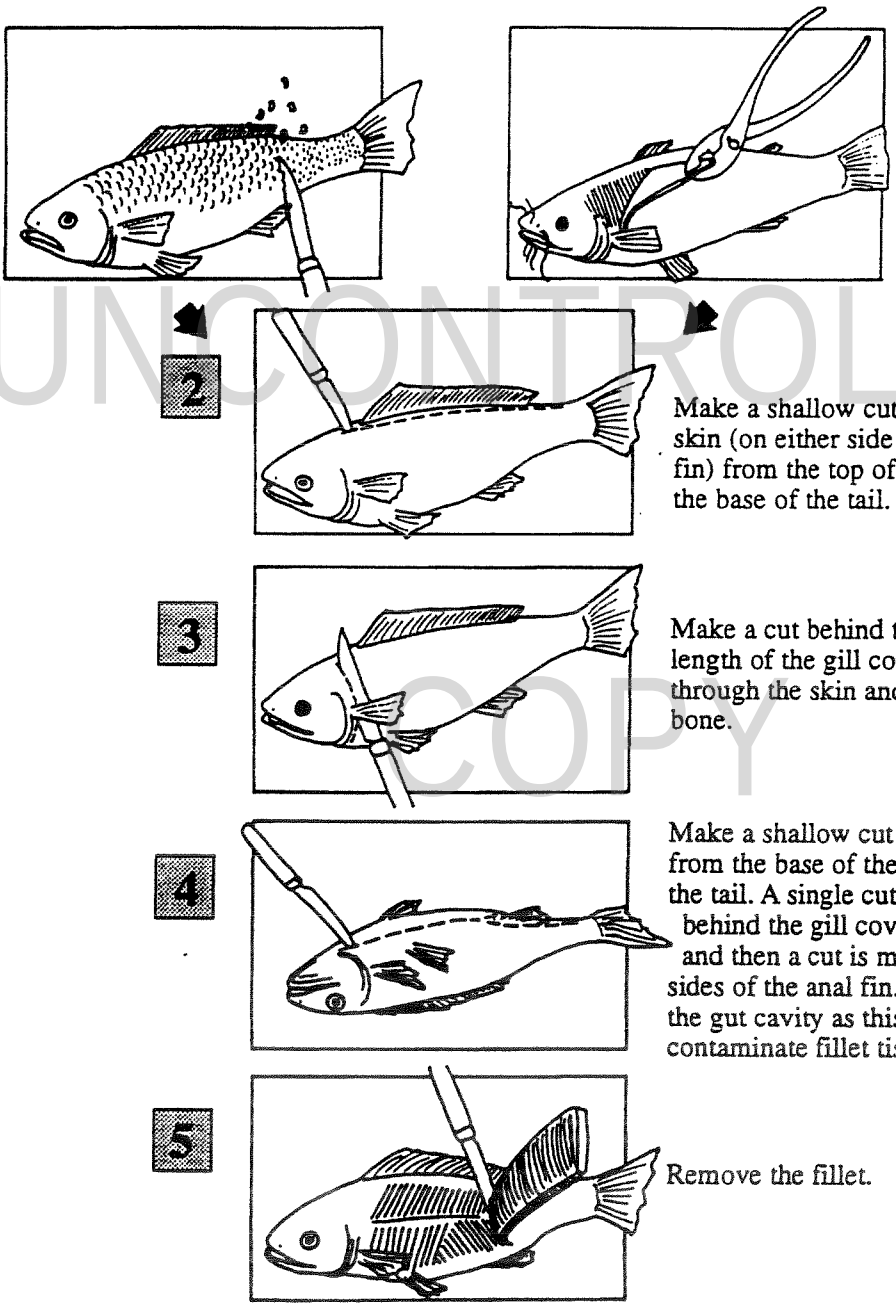
1 **Scaled Fish**

After removing the scales (by scraping with the edge of a knife) and rinsing the fish:

1b **Scaleless Fish**

Grasp the skin at the base of the head (preferably with pliers) and pull toward the tail.



Note: This step applies only for catfish and other scaleless species.

2 Make a shallow cut through the skin (on either side of the dorsal fin) from the top of the head to the base of the tail.

3 Make a cut behind the entire length of the gill cover, cutting through the skin and flesh to the bone.

4 Make a shallow cut along the belly from the base of the pectoral fin to the tail. A single cut is made from behind the gill cover to the anus and then a cut is made on both sides of the anal fin. Do not cut into the gut cavity as this may contaminate fillet tissues.

5 Remove the fillet.

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Source: U.S. EPA, 1991d.

Figure 7-3. Illustration of basic fish filleting procedure.

APPENDIX B

General Procedure for Removing Edible Tissues From Shellfish

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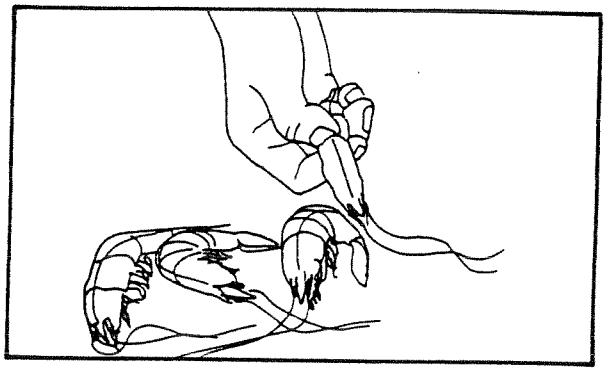
Heading, peeling and deveining shrimp

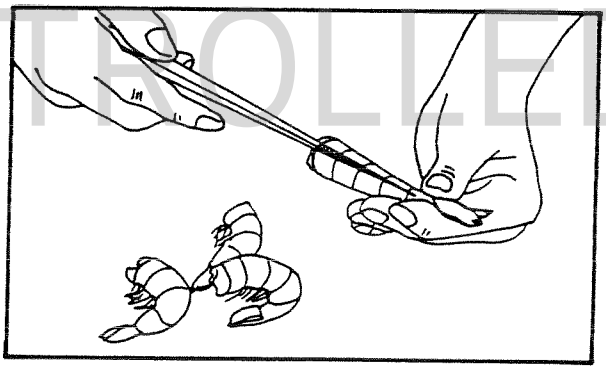
1

To head a shrimp, hold it in one hand. With your thumb behind shrimp head, push head off. Be sure to push just the head off so that you do not lose any meat.



2

If using a deveiner, insert it at head end, just above the vein.



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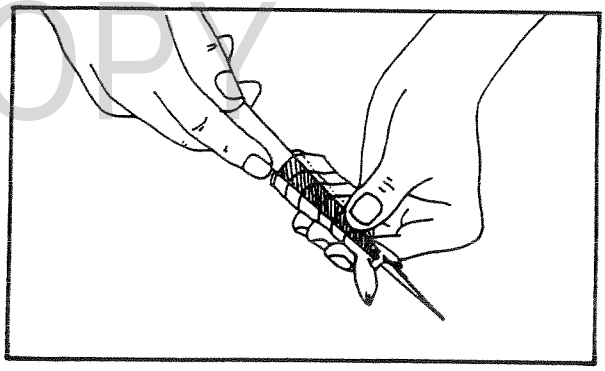
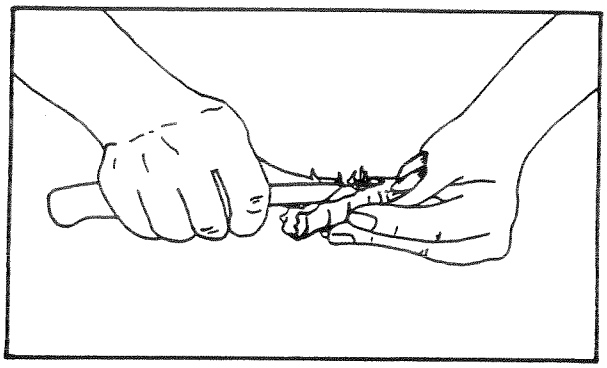
3

Push through shrimp to the tail and split and remove shell. This removes vein at the same time.



4

If you prefer to use a paring knife, shell shrimp with your fingers or knife. Then use knife to gently remove vein.

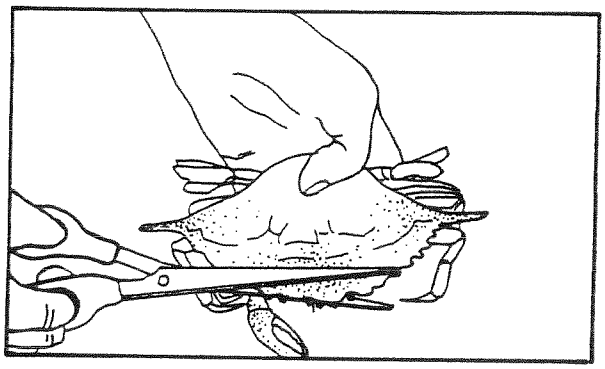


Source: UNC Sea Grant Publication UNC-SG-88-02

Cleaning soft-shell crabs

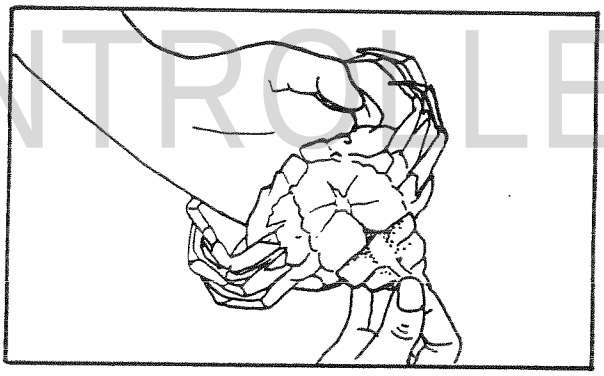
1

Hold crab in one hand and cut across body just behind eyes to remove eyes and mouth.



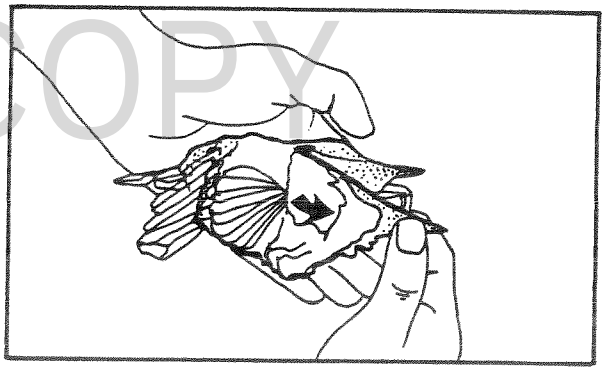
2

Turn crab on its back. Lift and remove apron and vein attached to it.



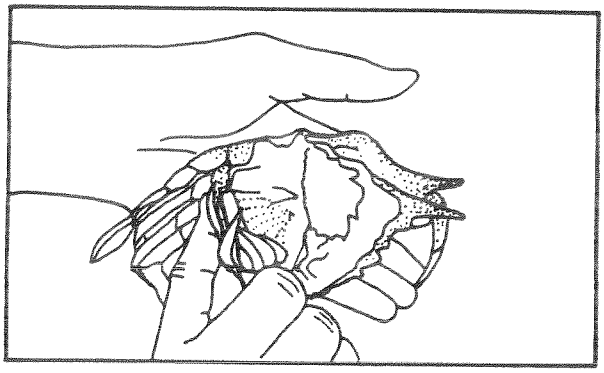
3

Turn crab over and lift one side of top shell.



4

With a small knife, scrape off grayish-feathery gills. Repeat procedure on other side.



Source: UNC Sea Grant Publication UNC-SG-88-02

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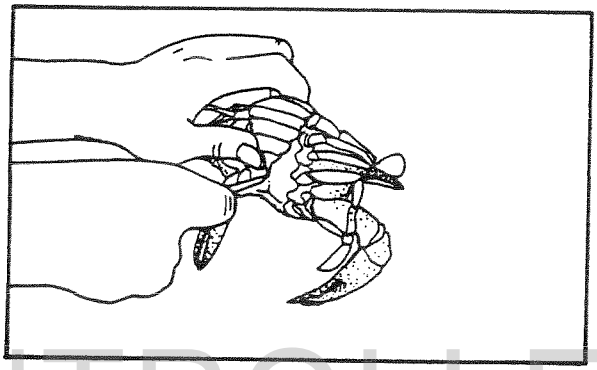
Cleaning hard-shell crabs

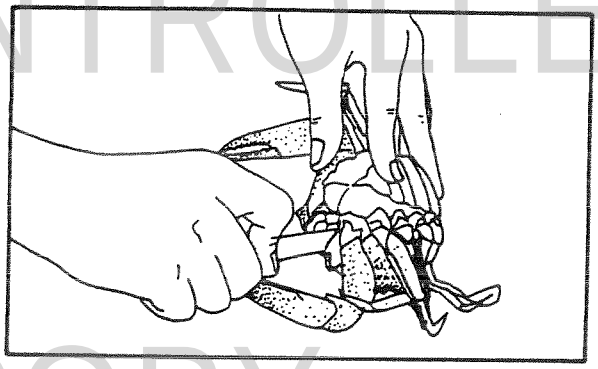
1

Hold crab in one hand. Turn crab over and stab straight down at point of apron with a knife.



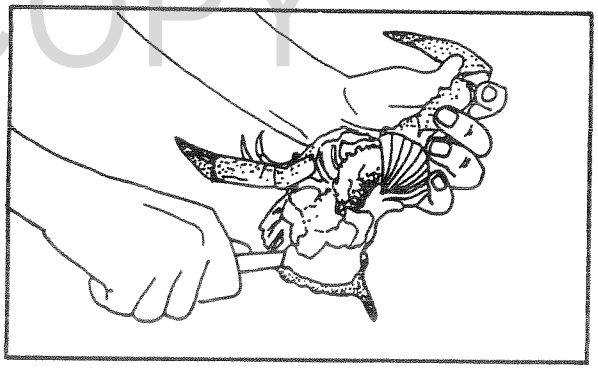
2

Make two cuts from this point to form a V-pattern that will remove mouth.



3

Do not remove knife after making second cut. Firmly press crab shell to cutting surface without breaking back shell. With other hand, grasp crab by legs and claws on the side where you are holding knife, and pull up. This should pull crab body free from back shell.



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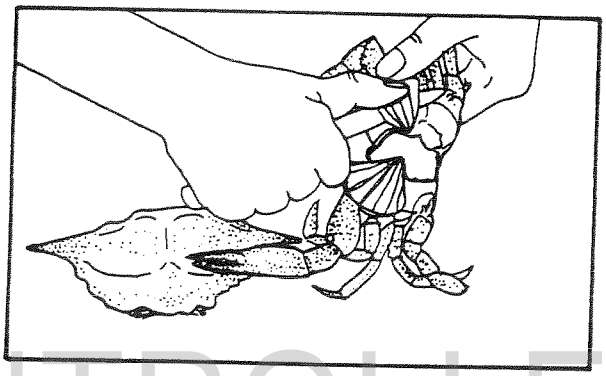
4

Remove gray, feathery gills, which are attached just above legs. Cut and scrape upward to remove gills.



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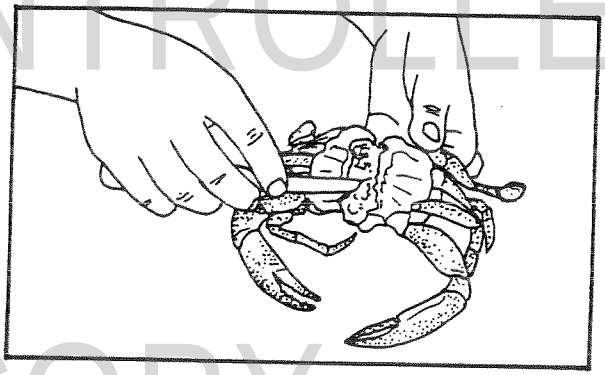
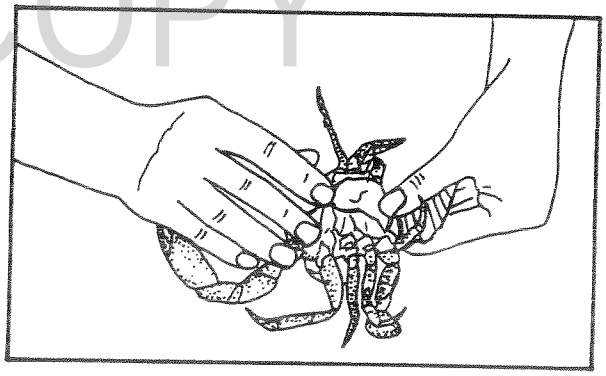
5

Remove all loose material—viscera and eggs—from body cavity.



6

If apron did not come loose with shell, remove it.

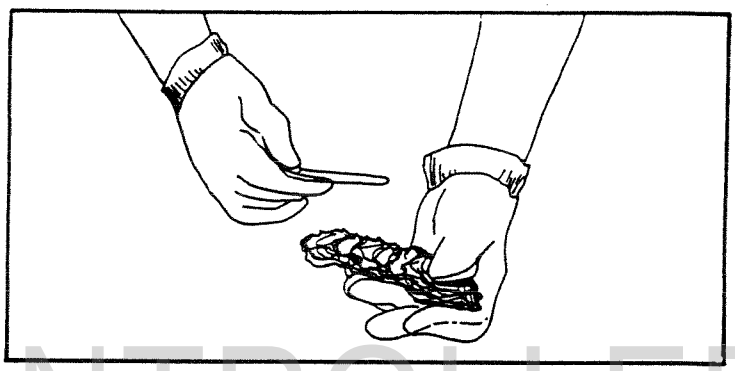


Source: UNC Sea Grant Publication UNC-SG-88-02

Shucking oysters

1

Oyster shells are especially sharp; be sure to wear gloves to protect your hands. Chip off a small piece of shell from the thin lip of the oyster until there is a small opening.



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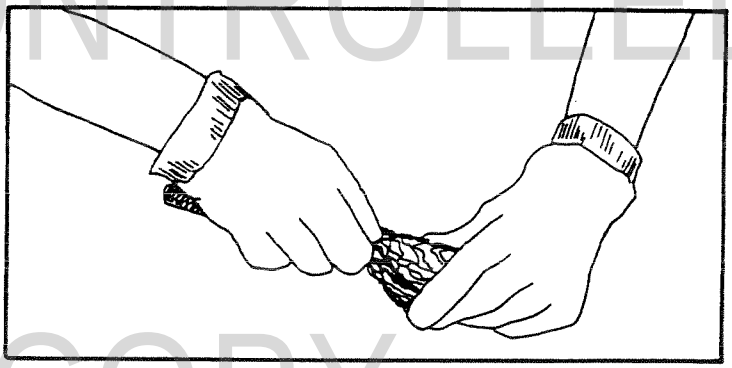
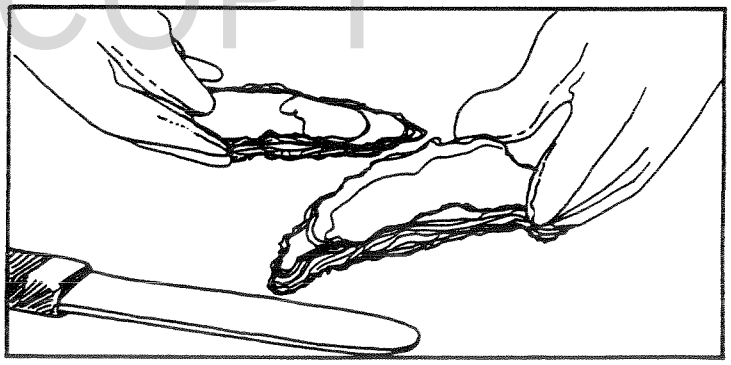
2

Insert knife blade into the opening and cut muscle free from top and bottom shells.



3

Remove oyster meat from the shell.

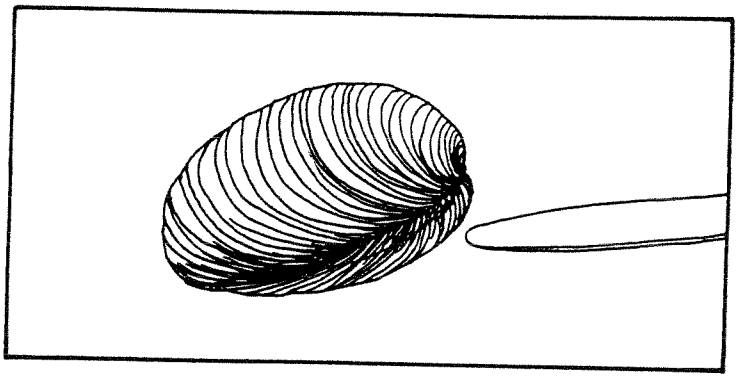


Source: UNC Sea Grant Publication UNC-SG-88-02

Shucking clams

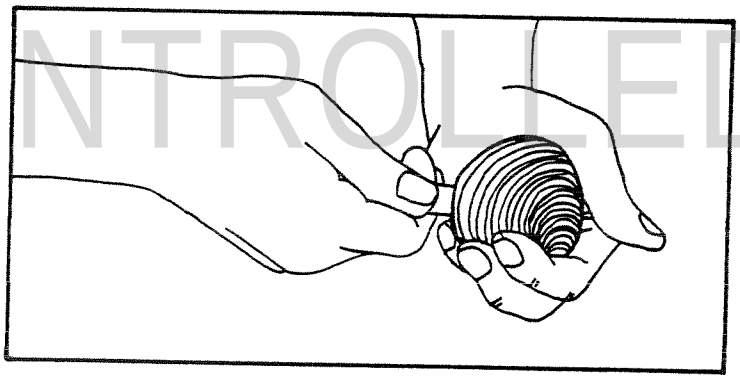
1

In the back of clam near the hinge is a black ligament. Toward the front where ligament ends is a weak spot. Insert your knife at this spot.



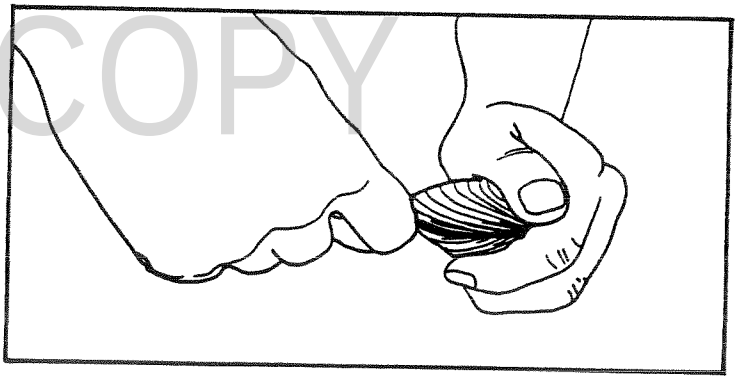
2

Inside are two muscles. Run the knife around the shell to sever both muscles.



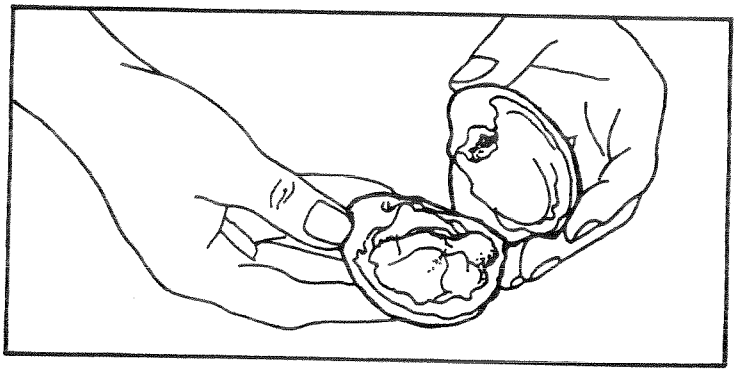
3

Now insert the knife blade into the front of the shell and separate the two shells.



4

Scrape the meat free from the top and bottom shell.



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APPENDIX C

Compositing and Freeze-Drying Bench Sheets

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COLUMBIA ANALYTICAL SERVICES, INC.

Service Request #: _____

Analysis For: Freeze Dried Solids

Lab Code	Wet Weight (g)	Tare (g)	Tare + Dry Wt.(g)	Dry Weight (g)	% Total Solids

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Date/Time in Freeze Dryer:_____ Date/Time out of Freeze Dryer:_____

Balance ID:_____ Date Balance checked:_____

Comments:_____

x = RPD

Analyst:	Date:
Reviewed By:	Date:

Columbia Analytical Services, Inc.

Service Request Number(s):

TISSUE HOMOGENIZATION DATA

Laboratory ID	Weight of Sample* (g)	Tare of Jar (g)		Matrix	Number per Sample	

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Comments:

* The weight of sample is after homogenization.

Balance ID: Date Balance Checked:

Analyst:	Date:
Reviewed:	Date:

Columbia Analytical Services, Inc.

Service Request Number(s):

TISSUE WHOLE BODY WEIGHT, LENGTH, and HOMOGENIZATION

Laboratory ID	Weight of Sample* (g)	Tare of Jar (g)		Length of Sample (in)	Matrix	

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Comments:

* The weight of sample Is after homogenization.

Balance ID: Date Balance Checked:

Analyst:	Date:
Reviewed:	Date:

Columbia Analytical Services, Inc.

Service Request Number(s):

Analysis for:
Pesticides PCB's Lipids SVO PAH Butyltins Dioxins SUB

ALIQUOT DATA

Service Request #		Wet Wt. (g)		Tare Wt. (g)		Matrix

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Comments:

Balance ID:	Date Balance Checked:
Analyst:	Date:
Reviewed:	Date:

STANDARD OPERATING PROCEDURE

for

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS

SOP No.: MET-TDIG

UNCONTROLLED

Revision: 2
March 25, 2010

Approved by: _____ Supervisor — 3/12/10 Date

_____ QA Manager — 3/12/10 Date

_____ Laboratory Manager — 3/12/10 Date

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COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS

1. SCOPE AND APPLICATION

This procedure describes techniques used for sample preparation and acid digestion of biological tissue samples. This procedure is applicable to the analysis of biological tissue for heavy metals. The procedure provides a convenient and efficient digestion/dissolution technique which allows for the simultaneous or sequential analysis of the sample for metals. The digestates may be analyzed by graphite furnace atomic absorption (GFAA), Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). The procedure includes, but is not restricted to, the metals listed in Table 1.

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2. METHOD SUMMARY

A representative tissue sample is lyophilized, blended, then sub-sampled for conventional oven digestion. Oxidation is brought about by the use of concentrated nitric acid in a Teflon closed vessel. The digestate is then analyzed for metallic constituents by GFAA, ICP-OES, or ICP-MS methods

3. DEFINITIONS

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3.1. Method Blank (MB) - The method blank or Preparation Blank (PB) is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.2. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.3. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at levels specified by a project analysis plan.

3.4. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

4. INTERFERENCES

Refer to the determinative method for a discussion of interferences.

5. SAFETY

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5.1. Follow all CAS safety practices as described in the CAS Safety Manual.

5.2. Each chemical compound or reagent should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

5.3. Nitric Acid is extremely corrosive. Care should be taken while working with this chemical. Personal protective equipment shall include safety glasses (with side shields), gloves and a lab coat. Follow normal precautions as per the CAS Safety Manual.

6. SAMPLE, COLLECTION, PRESERVATION AND STORAGE

Samples are typically collected in plastic containers and iced or refrigerated at 4 ± 2°C from time of collection until preparation or analysis. Sample may be frozen at ≤-10°C or as specified by project requirements.

7. APPARATUS AND EQUIPMENT

7.1. Lyophilizing apparatus, LABCONCO Model 7948040 freeze dryer.

7.2. Conventional laboratory oven capable of precise temperature control at 105°C.

7.3. Analytical balance capable of weighing to 1 mg.

7.4. 50 mL graduated poly tubes.

8. STANDARDS AND REAGENTS

8.1. Reagent water - ASTM Type I or Type II water

8.2. Concentrated nitric acid.

8.3. 1,000 ppm stock metal standards for preparation of spike solution(s) to include analyte(s) of interest.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for the freeze drying instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

9.2. Routine cleaning of the sample handling and digestion apparatus is necessary. Refer to the SOP for Metals Laboratory Glassware Cleaning.

9.3. Record all maintenance and monitoring activities in a laboratory notebook. All entries must be consistent with the SOP, *ADM-DATANTRY*.

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10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP *Documentation of Training* (ADM-TRANDOC), is also the responsibility of the department supervisor/manager.

10.3. When new or unfamiliar situations develop, it is also the responsibility of the employee to immediately notify the section supervisor, project manager, or the laboratory director for quick resolution of all issues. Due to the nature of the work, it is important that all work be documented, department supervisors, project managers and when necessary, the client, be contacted immediately when questions arise.

11. PROCEDURE

11.1. Obtain a representative tissue sample that will yield ~ 300 mg of freeze-dried solids.

Note: This is designed to be a general guideline. Approximately 300 mg of dry sample is typically required to obtain the desired detection limits that often are necessary for tissue analysis.

11.2. Slice the sample into thin pieces prior to filling the drying vessel. Homogenize the sample before freeze drying.

11.3. Cap the vessel and freeze the sample in a conventional freezer. Note: Using the Labconco Model 7948040 freeze dryer, the samples do not need to be frozen prior to freeze drying.

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11.4. When the sample is frozen, remove from freezer and follow the manufacturer's instructions for operation of the freezer dryer.

11.5. Blend the dry solids to obtain a homogeneous sample. The sample may be stored dry until digestion.

11.6. Sample Digestion

11.6.1. Transfer 300 mg of dried sample, weighed to the nearest 0.1 mg, to a 50 mL decomposition vessel.

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11.6.2. Add 4.5mL concentrated nitric acid to the vessel.

11.6.3. Follow the manufacturer's instructions for the use of the decomposition vessels. Place the vessel in a conventional oven at 105°C for a minimum of 24 hours. If using the conventional oven option, monitor oven temperatures for each batch and record this data onto the appropriate benchsheet.

11.6.4. Cool the vessel, open to relieve pressure and vent the gases. If Antimony, Silver or Tin are target analytes, then add 10 ml of deionized water to the cooled vessel. Re-cap and tighten the vessel. Return to the oven for an additional hour.

11.6.5. Cool the vessel, open to relieve pressure and vent the gasses. Transfer the sample to a volumetric container and dilute to 30 ml. The sample is ready for analysis.

12. QA/QC REQUIREMENTS

12.1. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project

specific quality assurance plans (QAPP). For example projects managed under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories*. General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)*. General QC Samples are:

12.1.1. Prepare one blank per digestion batch, or per 20 samples, or per SDG, whichever is more frequent. Analyze at least one preparation blank per group of samples digested. Refer to the SOP for *Sample Batches.*

12.1.2. Prepare duplicates at 5% frequency or one per batch, whichever is more frequent.

12.1.3. Prepare one laboratory control samples (LCS) and a duplicate laboratory control sample (DLCS) at 5% frequency or one of each per batch, whichever is more frequent.

12.1.4. Prepare spikes at 5% frequency or one per batch, whichever is more frequent. Spikes should be added directly to the dry sample. Spike solutions should be multi-element with analyte concentrations high enough to minimize volume added to sample.

12.1.5. Analyze a standard reference material (SRM) at 5% frequency or one per batch, whichever is more frequent. Standard Reference Material (SRM's) should be representative of the tissue sample being analyzed.

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13. REPORTING

13.1. Refer to the SOP for *Data Reporting and Report Generation* for reporting guidelines.

14. DATA REVIEW AND ASSESSMENT

14.1. Digestion data sheets, including weights and volumes used are completed and a batch lot number is assigned and attached to the data sheet. The Manufacturer's lot numbers for the reagents used are added to the digestion data sheet. (See appendix A)

14.2. Spiking sheets are completed including all spike data and volumes of spiking solutions used. (See appendix A)

14.3. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative.

15. CORRECTIVE ACTION

15.1. Refer to the SOP for *Corrective Action*. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

15.1.1. Handling out-of-control or unacceptable data

On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures; specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

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15.1.2. Documentation of nonconformity must be done using a Corrective Action Report (NCAR) when: a) corrective action is not taken or not possible b) corrective action fails to correct an out-of-control problem.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

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16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS Environmental Health and Safety Manual.

17. TRAINING

17.1. Training outline (See the SOP for *Documentation of Training*)

17.1.1. Read and understand the SOP/Method. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the trainee is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst. This incremental step in training shall be documented at each

step by use of the Training Plan or a modified version of the Training Plan outlined in the SOP for *Documentation of Training.*

17.1.3. Perform an initial precision and recovery (IPR) study for tissue samples. Summaries of the IPR are reviewed and signed by the Technical Director. Along with the completed Training Plan, copies are forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the SOP *for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

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18. REFERENCES

Recommended Guidelines for Measuring Metals in Puget Sound Marine Water, Sediment, and Tissue Samples; April 1997.

19. CHANGES SINCE THE LAST REVISION

19.1. Removed microwave digestion procedure.
19.2. 10.2. Added comment regarding sample homogenization before drying.
19.3. 10.3. Inserted comment about freezing sample before freeze drying.
19.4. 6.1. Updated Make/Model number of the freeze dryer.
19.5. 11.4. Clarified Sample Batch information; included reference to the Sample Batches SOP.
19.6. 10.7.4. Added a digestion step, per request.
19.7. Appendix A to include updated bench sheets.
19.8. 11.5. Deleted final sentence of the paragraph.
19.9. Updated references to to Administrative SOPs throughout the document.
19.10. Added the Definitions Section to the SOP.
19.11. Updated Safety Section.
19.12. Included Preventive Maintenance Section.
19.13. Added Training Section
19.14. Added Corrective Action, Reporting, and Data Review and Assessment Section.
19.15. Updates throughout the document to reflect current NELAC requirements.

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Table 1

Selected Elements and Analysis Procedures for Tissue Samples

Element	GFAA	ICP-OES	ICP-MS
Aluminum		√	√
Antimony		√	√
Arsenic	√		√
Barium		√	√
Beryllium		√	√
Boron		√	
Cadmium	√	√	√
Calcium		√	
Chromium		√	√
Cobalt		√	√
Copper		√	√
Iron		√	
Lead	√	√	√
Lithium		√	
Magnesium		√	
Manganese		√	√
Molybdenum		√	√
Nickel		√	√
Potassium		√	
Phosphorus		√	
Selenium	√		
Silver		√	√
Sodium		√	
Strontium		√	
Thallium	√	√	√
Tin		√	√
Vanadium		√	
Zinc		√	√

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Operation of the Freeze Drier Labconco 7948040

Operation Checklist

The following checklist should be followed after each use of the Stoppering Tray Dryer.

1. Wipe out the interior of the Stoppering Tray Dryer chamber with a paper towel to remove any moisture or debris.
2. Wipe the interior of the collector chamber (base unit)of the freeze dry system with a paper towel to remove any accumulated moisture.
3. Check the collector chamber drain hose on the freeze dry system (base unit) to ensure that the hose is free of moisture and that the drain plug is securely installed.
4. Using a paper towel, wipe the freeze dry system collector chamber (base unit) lid gasket and the Stoppering Tray Dryer door gasket to remove any dirt and contaminants that could cause a vacuum leak. Vacuum grease is not required on the door gasket or collector lid gasket to obtain proper vacuum seal.

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Starting the Freeze Dry cycle

1. On the left side of the Stoppering Tray Dryer unit flip the black switch to the on position. On the left side of the base unit flip the black switch to the on position. On the base unit that the condensing coil is located push the manual button. The collector temperature must reach -40°C before the vacuum pump can be turned on.

2. Place the containers holding the pre-frozen samples onto the trays in the Stoppering Tray Dryer unit on top of the base unit. When closing the door pick up left hand bottom corner of door while closing. Turn the door handle all the way to the right. The door should be covering the entire black gasket attached to the Stoppering tray unit. Make sure that the vacuum release valve on Stoppering Tray Dryer unit is turned to the closed position. Press the mode (see attached diagram) button until the green light is on for Automatic on the Stoppering Tray Dryer unit. Press the run stop button on the Stoppering Tray Dryer unit P1 will appear in the display and the amber led button by the run stop button will light. Once the Stoppering Tray Dryer unit reaches -40°C it will hold at that temperature for one hour and forty eight minutes. After one hour and forty eight minutes the vacuum pressure in the display should decrease. If the vacuum is not decreasing press the vacuum on the collector unit button and open and close the door lifting up the bottom left hand corner of the door while closing. Turn the door handle all the way to the right. Press the vacuum button to restart the vacuum.

3. When the vacuum pressure (mBar) is the same on the Stoppering tray dryer and the base unit the samples have finished the freeze drying process (typically after 48 hours, depending on mass of tissue). Press the vacuum button on the collector unit to turn the vacuum pump off. Flip the switch on the side of the base unit to off. Flip the switch on the side of the Stoppering Tray Dryer unit to off. Open the chamber by moving the vacuum release control on the front of the

Stoppering Tray Dryer unit to the open position. When the sound of air through the back fill port is no longer audible, the chamber door is ready to open.

Alarms

A number of unusual events may occur during a lyophilization procedure that can adversely effect the operation of the Stoppering Tray Dryer. If an event occurs, the alarm indicator will flash and the beeper will sound. The beeper will automatically mute itself after one minute. The specific alarm can be identified observing the display. The following "out of specification" conditions will initiate an alarm:

System Temperature Variations
Once the system temperature has stabilized for 20 minutes, if the manual set point temperature or automatic hold temperature varies more than ±2°C as measured by the system temperature sensor, the Red Alarm indicator and word "TMP" on the display will flash until the end of the run.

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Vacuum
Once the system vacuum is low and stabilize at a point where it changes less that 0.020mBar in 5 minutes if the vacuum changes more than 0.500mBar , the red Alarm indicator and the word "VAC" on the display will flash until the end of the run.

System Temperature Set Point
If during a ramp mode the system temperatures stabilize without reaching the set point temperature, the control will enter the next Hold mode. The Red Alarm indicator will flash and the program indicator "Px" on the display will flash until the end of the run.

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Power Failure
If a power failure occurs while a run in is progress, the Red Alarm indicator and Run/Stop indicator will flash when the power in restored. Once the power is restored, the process will continue as programmed until completion. Pressing Run/Stop cancels the flashing warning.

Vacuum Pump

The oil in the Vacuum pump should be checked before every use. It must be changed if it is cloudy, shows particles or is discolored. The useful life of the vacuum pump oil can be extended if the vacuum pump is operated for an extended period of time after a freeze dry run.

Changing the Vacuum pump oil:

Pump oil is located in the ICPMS Instrument Laboratory.

1. Make sure the Vacuum pump is turned off
2. Remove the front cover of the bottom unit of the freeze drying unit by pressing up on the metal clasp located on the bottom of the front cover in the middle. Use a small screwdriver.
3. Slide the Pump out far enough to allow a 1 L plastic container to placed under the drain on the front of the Vacuum pump.
4. Remove the Grey cap on top of the Vacuum pump.
5. Remove the Grey plug on the bottom of the Vacuum pump.
6. After all of the used oil has drained out of the pump, pour a small amount of the unused oil into the vacuum pump with the drain plug removed to rinse out the oil reservoir. Place the used oil in the **Used Oil** container located in the ICPMS Instrumentation Laboratory.
7. Place the drain plug on the vacuum pump (hand tighten) fill the pump up with approx 400 mls of oil the vi should be 1 inch from the top mark on the outside of the vacuum pump. Screw the grey cap back onto the vacuum pump (hand tighten).

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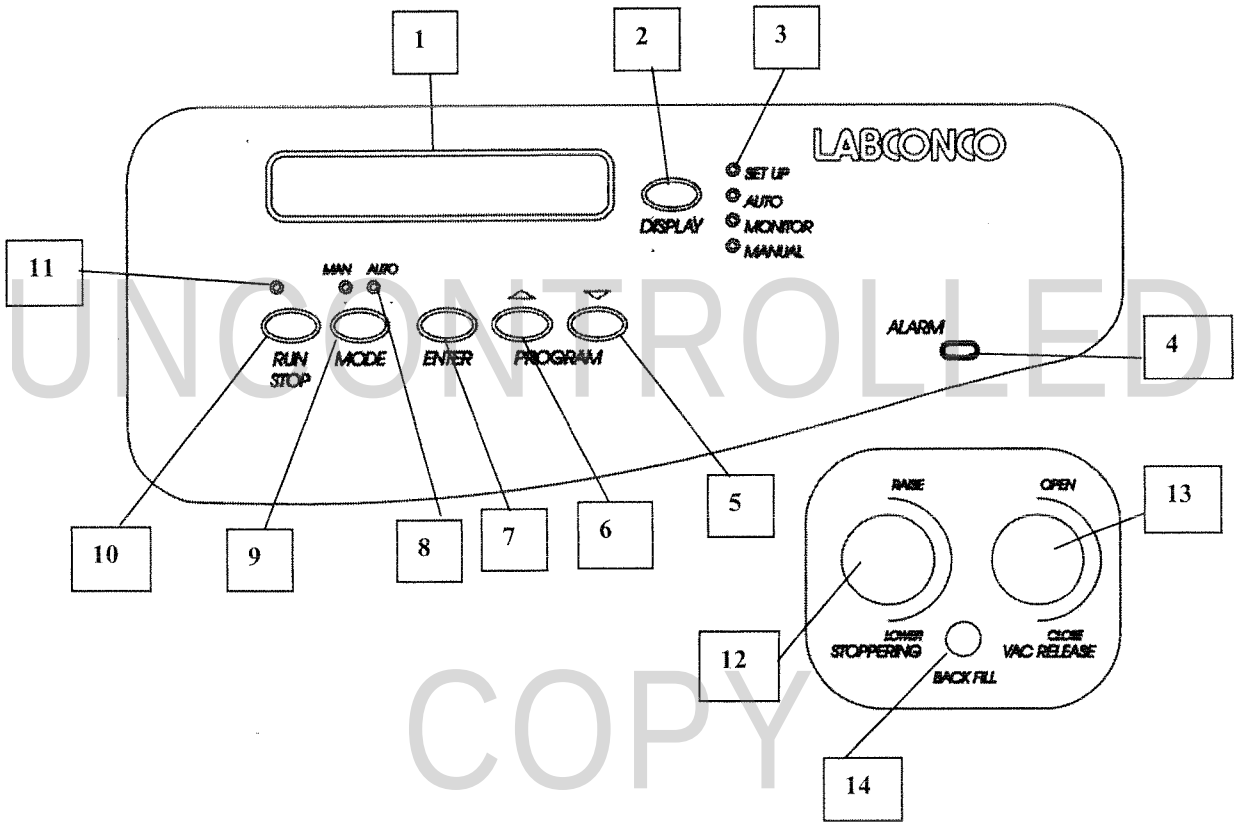
Stoppering Tray Dryer Controls

The control panel for the Stoppering Tray Dryer is shown with a description about its function.



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1. **Display**. Displays all necessary programming and operational data. Provides prompts to aid in programming.

2. **Display Selector**. Changes format of display from "monitor' to "auto" to "manual" to "set up" screen.

3. **Display Indicators**. Amber lamps indicate which display "screen" is shown.

4. **Alarm Indicator**. Red LED indicates that a system alarm has occurred.

5. **Decrease Switch**. Used in programming to decrease a parameter set point.

6. **Increase Switch**. Used in programming to increase a parameter set point.

7. **Enter Switch**. Used in programming to enter a selected set point into memory.

8. **Mode Indicator**. Amber lamp indicates whether the control is set to operate either in the automatic or manual mode.

9. **Mode Selector Switch**. Selects the mode of operation – either manual or automatic.

10. **Run/Stop Switch**. Initiates the start or stop of the lyophilization process.

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11. **Run/Stop Indicator**. Amber lamp burns steadily while freeze drying is in progress and turns off if either the programmed cycle is completed or the process is terminated in mid cycle. The amber lamp flashes if freeze drying is in progress and an out-of-specification condition exists.

12. **Stoppering**. Controls the stoppering up and down movement. The mechanism will function only while the chamber is under vacuum. The stoppering action should be closely monitored when operating this control.

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13. **Vacuum Release**. Vents the chamber so the chamber door can be opened. This control can also vent gas into the chamber when the gas is properly connected to the backfill port.

14. **Back Fill**. A regulated tank of gas may be connected to the port to allow the introduction of gas into the chamber after freeze drying. The port accepts 1/8" tubing.

! Ethylene Oxide is not recommended for use in this Tray Dryer for decontamination because of its hazardous and corrosive properties.

15. **Power Switch**. Turns all power to Tray Dryer ON or OFF. Not shown. Located on left side of cabinet.

APPENDIX A

Benchsheets

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COLUMBIA ANALYTICAL SERVICES, INC.

Service Request #: _____

Analysis For: Freeze Dried Solids

Lab Code	Wet Weight (g)	Tare (g)	Tare + Dry Wt.(g)	Dry Weight (g)	% Total Solids
NRCC DORM-3	(If Applicable)				96.1%
NRCC TORT-2	(If Applicable)				94.7%

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Date/Time in Freeze Dryer:_____ Date/Time out of Freeze Dryer:_____

Balance ID:_____ Date Balance checked:_____

Comments:_____

_____

_____

_____

_____

x = RPD

Analyst:	Date:
Reviewed By:	Date:

Columbia Analytical Services
Metals Tissue Digestion Sheet

Service Request Number(s) :

Star Lims Run No.: | Analysis for : ICP ICP-MS GFAA

Method : Tissue _____ | other: _____

Sample	Initial Weight (g)	freeze Dry	Wet	Final Volume (ml)	Matrix

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Time Digestion Started:_____**Oven Temp:**_____ **Time Digestion Ended:**_____

Lot # Acids Used: HNO3 _____ **Oven Temp:**_____

LCS: Dorm-3 (96.1% Solids), Tort-2(94.7% Solids) **Balance I.D.:** _____

SPIKE INFO

K-MET SS1,_____**mls added**

K-MET SS2,_____**mls added**

K-MET SS4,_____**mls added**

K-MET SS3, _____**mls added**

K-MET SS5,_____**mls added**

Additional spikes:_____

Comments: _____

Analyst _____ **Date** _____

Reviewer_____ **Date** _____

TissueDig.xls
3/1/2010

Service Request #_____

Q.C. Sample #_____

Circle type of digest: GFAA ICP FAA ICP-MS Other:_____ Initials / Date:_____/_____

Circle type of sample: Soil Water Misc. Sludge Oil Other:_____

Solution Name	Element	mLs of 1000ppm Solution	Final Volume	Solution Conc. mg/L	Enter mls Added	
K-MET SS1 SS1-MET1-81-B	HNO3	50.0	1000ml	-		Expires: 05/01/2010
	Al	100*	1000ml	200		
	Ag	100*	1000ml	5		
	Ba	100*	1000ml	200		
	Be	100*	1000ml	5		
	Cd	100*	1000ml	5		
	Co	100*	1000ml	50		
	Cr	100*	1000ml	20		
	Cu	100*	1000ml	25		
	Fe	100*	1000ml	100		
	Pb	100*	1000ml	50		
	Mn	100*	1000ml	50		
	Ni	100*	1000ml	50		
	Sb	50	1000ml	50		
	V	100*	1000ml	50		
	Zn	100*	1000ml	50		
K-MET SS2 SS4-MET1-76-B	HNO3	25.0	500ml	-		Expires: 4/2010
	As	2.0	500ml	4		
	Cd	2.0	500ml	4		
	Pb	2.0	500ml	4		
	Se	2.0	500ml	4		
	Tl	2.0	500ml	4		
	Cu	2.0	500ml	4		
K-MET SS3 MET1-82-C	HNO3	25.0	500ml	-		Expires: 05/2010
	As	50.0	500ml	100		
	Se	50.0	500ml	100		
	Tl	50.0	500ml	100		
K-MET SS4 MET1-82-B	HNO3	25	500ml	-		Expires: 7/30/2010
	B	50	500ml	100		
	Mo	50	500ml	100		
K-MET SS5 SS7-MET1-78-H	HNO3	10.0	200ml	-		Expires: 3/29/10
	K**	20	200ml	1000		
	Na**	20	200ml	1000		
	Mg**	20	200ml	1000		
	Ca**	20	200ml	1000		
K-MET GFLCSW (MET1-80-J)	HNO3	10.0	1000ml	-		Expires: 07/01/10
	As, Pb, Se, Tl	5.0	1000ml	2.5		
	Cd	-	-	1.25		
	Cu	2.5	1000ml	2.5		
K-MET QCP-CICV-1 (MET1-79-E)	Ca, Mg, Na, K	no dilution	-	2500		Expires: 10/01/10
	Al, Ba	no dilution	-	1000		
	Fe	no dilution	-	500		
	Co, Mn, Ni, V, Zn	no dilution	-	250		
	Cu, Ag	no dilution	-	125		
	Cr	no dilution	-	100		
	Be	no dilution	-	25		
K-MET QCP-CICV-2 (MET1-80-N)	Sb	no dilution	-	500		Expires: 12/10
K-MET QCP-CICV-3 (MET1-80-O)	As, Pb, Se, Tl	no dilution	-	500		Expires: 12/1/10
	Cd	no dilution	-	250		

* Denotes volume of mixed stock standard.

** Denotes 10,000 ppm individual stock standards.

Standard	mls of standard	ppm	Logbook #	Exp. Date

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STANDARD OPERATING PROCEDURE

for

Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation

SOP Code: ADM - MDL

Revision: 9

Effective Date: September 28, 2009

Approved by: _____ 9-8-09
Quality Assurance Director — Date

_____ 9-9-09
President — Date

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Annual review of this SOP has been performed and the SOP still reflects current practice.	DOCUMENT CONTROL
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Standard Operating Procedure for Performing Method Detection Limit Studies and Establishing Limit of Detection and Quantitation

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1.0 PURPOSE

This standard operating procedure (SOP) describes the procedure for the determination of method detection limits (MDLs) and limits of detection (LOD). The procedures for establishing the limits of quantitation are also included.

2.0 APPLICABILITY

2.1 The procedure described in this SOP is designed to be applicable to a wide variety of sample types ranging from reagent (blank) water or wastewater containing the analyte, to solids (such as soil) containing the analyte, to the analyte in a gaseous matrix. The MDL for an analytical procedure will vary as a function of sample matrix. This SOP requires a complete, specific, well-defined, and written analytical procedure (i.e., an SOP). It is essential that all sample-processing steps of the analytical procedure are included in the determination of the MDL; that is, all the steps that a sample is processed through, from sample preparation to analytical completion, must be included in the MDL determination. The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample. This SOP for the determination of MDLs was designed to be applicable to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device or instrument-type independent.

2.2 The procedures described in this SOP are intended to meet the requirements of the NELAC 2003 Quality System Standard and the Department of Defense *Quality System Manual* (4.1) with regard to limits of detection and limits of quantitation.

3.0 DEFINITIONS

3.1 Method Detection Limit (MDL)

3.1.1 The MDL is the minimum concentration of a substance or analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

3.1.2 The Calculated MDL (MDL_C) is the MDL as calculated in Section 6.1.11 and will typically contain two or more significant figures.

3.1.3 The Reported MDL (MDL_R) is the MDL that is used for reporting purposes. MDLs for <u>organic analytes</u> will be reported with two significant figures. MDLs for <u>inorganic analytes</u> will be reported with either one or two significant

figures depending upon the number of significant figures in the analytes' method reporting limit.

3.2 Limit of Detection (LOD)

3.2.1 The Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and is laboratory dependent.

3 2.2 For non DOD applications LOD = MDL_R For DOD the LOD is the spike concentration a whi h he in tia d tection limit and LOD verif cation are succe sfully pe formed

3.3 Limit of Quantitation (LOQ) - The Limit of Quantitation (LOQ) is the minimum level, concentration, or quantity of a target analyte that can be reported with a specific degree of confidence. The LOQ is equivalent to the method reporting limit (MRL).

3.4 Analytical Procedure - The Analytical Procedure is the written, step-by-step description of the operation by which samples are processed in order to obtain the concentration of an analyte in a sample.

3.5 Spike Level - The spike level is the known concentration of analyte that is added to a matrix for the determination of the MDL or LOD.

3.6 Interferences - Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of known or unknown species (inte ferent tha hinde an accurate analy is of th arg t analyte( ).

3.7 Matrix

3.7.1 When the matrix analyzed is <u>aqueous</u> (includes ground water, surface water, waste water, drinking water, etc.), analyte-free reagent water is to be used.
When the matrix analyzed is <u>solid</u> (includes soil, sand, tissue, or other solid materials), analyte-free soil, sand, tissue, or a suitable material is to be used. When he matrix analyzed i <u>gaseous</u> (i e., air or emi sions), an analyte-free, inert gas (such as zero grade a r or ultrapure helium or nitrogen) s to be used.

3.7.2 If the analysis is performed on a matrix for which there is not available an appropriate or similar, analyte-free matrix (such as, metals analysis on soil samples), the MDL analysis will be done as prescribed by the SOP for the analysis except the sample (weight) will be omitted; that is, the analysis will be done on all the reagents but without addition of any sample.

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4.0 DISCUSSION

4.1 The MDL is a property of the analytical procedure, sample matrix, and measurement system (e.g., an instrument if one is used in the analytical procedure). The MDL is a statistic. It is an estimate that includes both the systematic and random errors that are an inherent part of the analytical procedure. The MDL for a given analyte will be unique for the sample's matrix and may be different than the MDLs shown in published methods. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

4.2 The relative uncertainty of an analytical measurement increases as the measured value approaches the MDL and at the MDL the uncertainty in the measured value may be 100% or greater.

4.3 The MDL procedure in this SOP is based upon the procedure described in *40 CFR Part 136, Appendix B* (Reference 9.1).

5.0 RESPONSIBILITIES

5.1 It is the responsibility of the laboratory Quality Assurance Program Manager (QA PM), and department managers and supervisors to schedule MDL determinations as necessary to meet the requirements of this SOP. It is the responsibility of the QA PM to track the status of MDLs, DOD LODs, laboratory LOQs, and their verifications in order to maintain compliance with this SOP and various accreditation programs.

5.2 Completed MDL determinations are to be reviewed by the department manager or supervisor and approved by the QA PM before they are implemented. The QA PM is responsible for maintaining the MDL file described in Section 8.0

6.0 PROCEDURE

6.1 Performing the MDL study

6.1.1 General requirements

6.1.1.1 MDLs are to be determined for each analyte and for each matrix. This SOP describes procedures for determining MDLs for the generic matrices aqueous, solid, and gaseous. MDLs for specific matrix types may be adapted from the procedures in this SOP.

Note: For aqueous MDL determinations, the default procedure for determining the MDL is the 'spiked blank' procedure. Sample matrix or sample-specific MDLs are performed only when required by project or program.

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6.1.1.2 All sample processing steps in the analysis procedure shall be included in the determination of the MDL. MDL shall be generated using all preparatory and cleanup procedures routinely used on samples.

6.1.1.3 For PCB Aroclors an MDL study is required for PCB Aroclors 1016 and 1260 only, unless required by accreditation program or project. However, MDL/LOD/LOQ verifications must be performed for all Aroclors being reported.

6 1.1.4 An MDL study is not required for any analyte for which spiking solutions or qu li y contr l sampl s are not ava lable; e.g., temperatur . Also, under NELAC and DOD program , an MDL study is ot required when results are not reported below the MRL/LOQ.

6.1.2 Frequency of MDL determination and verifications

6.1.2.1 An MDL study shall be performed initially; i.e., when the procedure is first put into production, and the LOD and LOQ established prior to sample analyses.

6.1.2.2 An MDL study shall be performed at the frequency specified in the applicable method or as specified by an accrediting authority. For example, some state accrediting programs may require annual MDL studies.

6.1.2.3 A new MDL determination is to be performed each time there is a change in the test method that affects how the test is performed, or when a change in instr m ntation occurs tha affects the s nsitivity of th analy is.

6.1.2.4 MDL/LOD Verification – If an MDL study is not performed annually, an MDL/LOD verification shall be performed annually on every instrument used to perform a particular analysis. **Note:** This verification is required quarterly for DOD accredited tests.

6.1.3 Instruments - If more than one instrument is used for the same analytical procedure, he replicate samples should be analyzed on each instrument to ensure there is no instrument bias. Under some pecific custome con racts and for some programs, instrument-specific MDLs may be required. There are two options for complying with this requirement:

- Analyze the replicate samples on each instrument used for the analytical procedure and calculate the MDL_C for each instrument. The MDL_R will be the largest of the several MDL_C's; or

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- Analyze the replicate samples on each instrument used for the analytical procedure and calculate a single MDL_C using all the values from each instrument. A minimum of five values is needed from each instrument. For example, if two instruments are used, there would be a minimum of two times five or ten values to be used to calculate the MDL_C. Make sure to use the appropriate Student's t-statistic that corresponds to the number of values used to calculate the standard deviation.

  Note: This option may not be acceptable under some specific customer contracts or for some programs, such as the DOD quality systems for environmental laboratories.

6.1.4 Estimation of the MDL - Use one of the following guides to help estimate the MDL.

6.1.4.1 The concentration value that corresponds to an instrument signal-to-noise ratio in the range of 2.5 to 5.

6.1.4.2 The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

6.1.4.3 That region of the calibration curve where there is a significant change in sensitivity, i.e., a break in the slope of the calibration curve.

6.1.4.4 Instrumental limitations.

6.1.5 Aqueous Blank MDLs

6.1.5.1 Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at or above the MDL of each analyte of interest.

6.1.5.2 Prepare a minimum of 7 (preferably 8 to 12) analyte-spiked reagent water samples at a concentration that is 3 to 5 times the estimated MDL.

6.1.5.3 Analyze the analyte-spiked reagent water samples by processing them through the **entire** analytical procedure. Make all computations according to the directions prescribed in the analytical procedure with the final results reported in the same units as used for water samples. Proceed to calculation of the MDL.

6.1.6 Aqueous Sample MDLs

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6.1.6.1 Analyze the aqueous sample by processing it through the **entire** analytical procedure

6.1.6.2 Calculate the analyte conc ntrat on

6.1.6.2.1 If the measured concentration of the analyte is in the recommended range of 3 to 5 times the estimated MDL, proceed to Section 6.1.6.3.

6 1 6.2.2 If the measured concentration of the analyte is less than the re ommende 3 o 5 times the estimate MDL, add a known amo nt of analy e to bring the concentration of an lyte between 3 to 5 times the estimated MDL and proceed to Section 6.1.6.3.

6.1.6.2.3 If the measured concentration of the analyte is greater than 5 times the estimated MDL, either obtain another sample with a lower concentration of analyte in the same matrix, or the sample may be used as is for determining the MDL if the analyte concentration does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical procedure changes as the analyte concentration increases from the MDL; hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations. Proceed to Section 6.1.6.3.

6 1.6.3 Prepare and analyze a minimum of 7 (preferably 8 to 12) aliquots of the aque us sampl by proc ssing them through the **entire** analyt cal proc dure. Ma e all computations according o the di ecti ns prescribed in the analytical procedure with the final results reported in the same units as used for water samples. Proceed to calculation of the MDL.

6.1.7 Solid Blank MDLs

6.1.7.1 Prepare a solid material (e.g., soil, sand, tissue, Na_2SO_4, Teflon chips, or other appropr ate materia) that is free of analyte

6.1.7.2 Prepare a minimum of 7 (preferably 8 to 12) analyte-spiked solid samples at a concentration that is 3 to 5 times the estimated MDL. The same weight of analyte-spiked solid is substituted for the sample weight in the analytical procedure.

6.1.7.3 Analyze the analyte-spiked solid samples by processing them through the **entire** analytical procedure.

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Make all computations according to the directions prescribed in the analytical procedure with the final results reported in the same units as used for solid samples. Proceed to calculation of the MDL.

6.1.8 Solid Sample MDLs

6.1.8.1 Analyze the solid sample by processing it through the **entire** analytical procedure.

6.1.8.2 Calculate the analyte concentration

6.1.8.2. If the measured concentration of the analyte is in the recommended range of 3 to 5 times the estimated MDL, proceed to Section 6.1.8.3.

6.1.8.2.2 If the measured concentration of the analyte is less than the recommended 3 to 5 times the estimated MDL, add a known amount of analyte to bring the concentration of analyte between 3 to 5 times the estimated MDL and proceed to Section 6.1.8.3.

6.1.8.2.3 If the measured concentration of the analyte is greater than 5 times the estimated MDL, either obtain another sample with a lower concentration of analyte in the same matrix, or the sample may be used as is for determining the MDL if the analyte concentration does not exceed 10 times the MDL of the analyte in soil. The variance of the analytical procedure may change as the analyte concentration changes from the MDL; hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

6.1.8.3 Prepare and analyze a minimum of 7 (preferably 8 to 12) aliquots of the soil sample by processing them through the **entire** analytical procedure. Make all computations according to the directions prescribed in the analytical procedure with the final results reported in the same units as used for solid samples.
Proceed to calculation of the MDL.

6.1.9 Gaseous Blank MDLs

6.1.9.1 Using an appropriate sample container (e.g., Tedlar® bag or SUMMA® passivated canister) and appropriate analyte-free inert gas (such as zero-grade air or ultrapure nitrogen), prepare a minimum of 7 (preferably 8 to 12) analyte-spiked inert gas samples at a concentration that is 3 to 5 times the estimated MDL.

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6.1.9.2 Analyze the an lyte-spiked iner gas amples prepared in Section 6.8.1 by processing them through he **entire** nalytical procedure. Make all computations according to the directions in the analytical procedure with the final results reported in the same units as used for air samples. Proceed calculation of the MDL.

6.1.10 Rejection of Replicate Sample Results

6 1.10.1 A replicate sample result may only be rejected if there is an assignable caus for not using that result. A signable causes in lude, but are not limi ed to, r plicate ample preparation error, instrument malfunction, bad injection or purge, and internal standard(s) missing or response uncharacteristically high or low. The cause for rejecting the replicate sample result must be documented in the MDL data package.

6.1.10.2 For multi-analyte analyses, if a replicate sample result is rejected for an assignable cause, results for all the analytes from that sample are to be rejected; that is, "picking and choosing" analyte results from a sample is not permitted.

6.1.11 Calculation of MDL_C

6.1.11.1 Determine the standard deviation, *s*, of the replicate sample results.

$$s = \sqrt{\frac{\sum_{n=1}^{i} (x_i - \overline{x})^2}{n - 1}}$$

where $\overline{x} = \frac{\sum_{n=1}^{i} x_i}{n}$

Multiply the standard deviation obtained above times the appropriate one-sided 99% Student's t-statistic, which is found in the following table.

$MDL_C = s \times$ {appropriate Student's t-statistic}

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No. of Samples (n)	Student's t-statistic	Degrees of Freedom (n - 1)
7	3 143	6
8	2.998	7
9	2.896	8
10	2.821	9
11	2.764	10
12	2.718	11
13	2 681	12
4	2.650	13
5	2.624	14
16	2.602	15
17	2.583	16
18	2.567	17
19	2.552	18
20	2.539	19
21	2.528	20

6.1.12 Determination of MDL_R

The Reported MDL (MDL_R) is the calculated MDL rounded **up** to the appropriate number of significant figures.

6.2 Evaluation of the Quality of the MDL Study

6.2.1 The quality f the MDL i eval at d using the following criteria.

6.2.1.1 Spike Level - The spike level is **too low** if the MDL_C is greater than the spike level. The spike level is **too high** if the spike level is greater than <u>ten</u> times the MDL_C.

6.2.1.2 Percent Relative Standard Deviation (%RSD) - The %RSD should not exceed 35, where the %RSD is equal to the standard deviation (*s*) divided by the average of he spike recoveries times 100

$$\%RSD = (s \div \bar{x})\ 100$$

6.2.1.3 Percent Spike Recovery - The spike recovery should be what is typically obtained for that analyte from the analytical procedure; i.e., a 40% spike recovery for an analyte is too low if the method normally recovers 80% or more for that analyte.

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6.2.1.4 At least one of the criteria in Sections 6.2.1.2 and 6.2.1.3 must be true. If the MDL does not meet these criteria, then the study should be repeated, adjusting the spike level appropriately. Additionally, the MDL_R needs to be verified as described in following sections.

7.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

7.1 Matrices - MDLs shall be generated for all applicable matrices.

7.2 Preparatory and clean-up procedures - MDLs shall be generated for all preparatory and clean up procedures routinely used on samples.

7.3 Analysis of MDL study replicates

7.3.1 No fewer then 7 replicate samples can be used; 8 to 12 replicate samples is preferred.

7.3.2 The replicate samples do not have to all be analyzed in the same analytical batch on the same day. In fact, it is preferred to spread out the replicate samples among several analytical batches analyzed on several days to increase the contribution of the day-to-day variability. For drinking water compliance tests, sample preparation and analyses for the MDL calculation should be made over a period of at least three days to include day-to-day variation as an additional source of error.

7.4 Review and Approval - Completed MDL determinations are to be reviewed by the supervisor of the analysis. The QA PM will review and approve the MDL determination before it is implemented.

7.5 MDL/LOD verification, non-DOD analysis (annually)

7.5.1 The MDL/LOD verification is required for NELAC accredited tests for which results will be reported below the lower end of the calibration range (i.e. below the MRL/LOQ).

7.5.2 Following the completion of MDL study, verify the validity of the MDL_R by confirming the qualitative identification of the analyte in a MDL/LOD verification QC sample in each applicable matrix. The verification sample should contain the analyte at approximately 2 - 3 times the MDL_R for single analyte tests and 1 - 4 times the MDL_R for multiple analyte tests. This verification must be performed on every instrument that is used for the analysis of samples and reporting of data.

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7.5.3 The verification sample is processed through the entire analytical process. The verification shall be acceptabl f it produces a response that is at least three times above the instrument's noise evel and can be qualitatively identified (ion abundance, 2nd column confirmed retention time, pattern recognition, etc.). If the MDL verification check fails, the cause is to be determined and documented, and additional MDL verification checks shall be performed at a higher level to set a higher MDL, or a new MDL study shall be performed and the verification repeated.

7.5.4 Once the verification is completed, the MDL_R is then used for data evaluation and reporting

7.6 LOD verifica ion – DoD accre ited ana ys s (quarte ly)

7.6.1 The LOD verification is required for DOD accredited tests for which results will be reported below the lower end of the calibration range (i.e. below the LOQ).

7.6.2 Following the completion of MDL study, determine the LOD by confirming the qualitative identification of the analyte in a LOD verification QC sample in each applicable matrix. The verification sample should contain the analyte at approximately 2 - 3 times th MDL_C for single analyte tests and 1 - 4 times the MDL_C for multiple analyte tests. This verification must be performed on every instrument that is used for the analysis of samples and reporting of data.

7.6.3 The verification sample is processed through the entire analytical process. The verification shall be acceptable if it produces a response that is at least three times above the instrument's noise level and can be qualitatively identified (ion abundan e, nd column c nfirmed retention time, pattern recognition, etc.). If the LOD verific ti n check f ils, the cause is o be determine and documented, and additional LOD verification checks (two are required) shall be performed at a higher level to set a higher LOD, or a new MDL study shall be performed and the LOD verification repeated.

7.6.4 Once the verification is completed, the concentration of the LOD verification sample establishes the LOD for data evaluation and reporting. The MDL is *not* used for reporting for DOD projects unless project-spec fied.

7.7 Determining and verifying the limit of quantitation, non-DOD analyses (annually)

7.7.1 The LOQ (MRL) is initially se by identifying the lowest concentration point on the calibration curve, putting that concentration through the sample calculation formulae using the sample weights, volumes, dilutions, etc used to calculate sample results.

7.7.2 The LOQ/MRL must be at a level above the MDL_R.

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7.7.3 The LOQ (MRL) is th n confirmed by perfo ming an LOQ verification by successfully ana yzing a QC sample containing the analyte at 1-2 times the claimed LOQ. This must be d ne in each pplicable matrix. The verification is acceptable if the following conditions are met:

- The analyte is qualitatively verified as per LOD verification.
- The recovery of each analyte is within the established criteria, or project DQOs, for accuracy.

If the LOQ verification check fails the cause is to be dete mined and documented, and addition l LOQ ve ification ch cks shall be p rforme at a highe level to set a higher LOQ, or a new MDL study shall be performed and the LOQ verification repeated.

7.7.4 Once the verification is completed, the LOQ/MRL is then used for data evaluation and reporting.

Note: The LOQ verifica ion is not required in the following cases:

- For 'non-spikable' tests or for tests where QC samples are not available or appropriate, or
- If during routine analysis the accuracy and precision is evaluated at the LOQ/MRL (i.e. batch QC LCS is run at the LOQ/MRL).

7.8 Determining a d ve ifying the limit of qu ntitatio – DOD a credited analyses (quarterly)

7.8.1 The LOQ is ni ially set by identifying the lowe t concent ation poin on the calibration curve (or higher), putting that concentration through the sample calculation formulae using the sample weights, volumes, dilutions, etc used to calculate sample results.

7.8.2 The LOQ must be at a level at least 3 times the DOD LOD earlier established.

7.8 3 The LOQ is then con irmed by p rforming an LOQ verification by successfully analyzing a QC samp e conta ning the analyte at 1-2 times the claimed LOQ. This must be done in each applicable matrix. The verification is acceptable if the following conditions are met:

- The analyte is qualitatively verified as per LOD verification.

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- The recovery of each analyte is within the established criteria, or project DQOs, for accuracy. Initially, the criteria will be the laboratory's statis ical LCS limits for the test. As LOQ verification points are accumulat d, they will be us d to establish limits for subsequent verifications.
- Using from 2 to 4 LOQ verification points, the ongoing %RSD is calculated to demonstrate the precision at the LOQ.

If the LOQ verification check fails, the cause is to be determined and documented, and additional LOQ verification checks shall be performed at a higher level to set a highe LOQ, or a new LOD establi hed and the LOQ veri ication repeated.

7.8.4 Once the verification is completed, the LOQ is then used for data evaluation and reporting.

Note: The LOQ verification is not required for 'non-spikable' tests or for test where QC samples are not available or appropriate. Also, the LOQ verification is not required if during routine analysis the accuracy and precision is evaluated at the LOQ/MRL (i.e. batch QC LCS is run a the LOQ/MRL).

8.0 RECORDS

8.1 The data for the MDL determination is summarized in an Excel spreadsheet or similar format (e.g. Stealth or LIMS outputs). The spreadsheet contains programmed cells to automatically calculate the data once individual analysis results are entered. In addition to the summarized data, the spreadsheet will contain approval lines and all applicable header information to ident fy the MDL tudy

8.2 The summary and t e eference t the loc tion of he r w da a are to be fil d in a readily available file of MDLs, either hardcopy or electronically. This file is to be located both in the department performing the analytical procedure and in a centralized location for MDLs from the entire laboratory.

9.0 REFERENCES

9.1 *40 CFR Part 136 Appendix B*, Defin tion and Procedure for the Determination of the Method Detection Limit--Revision 1.11

9.2 *SOP for Significant Figures*, ADM-SIGFIG

9.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, September 1986 and as amended by Updates I, II, IIA, IIB, III, IIIA/B, IV

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9.4 *Standard Methods for the Examination of Water and Wastewater*, APHA/AWWA/WEF, 19th Edition, 1995, Method 1030E; 20 h Edition 1998, Method 1030C; 21st Edition, 2005, Method 1030C.

9.5 National Environmental Laboratory Accreditation Conference (NELAC), 2003 Quality Systems Standard, Section 5.5.9.2.a)4); Appendix C, Section C.3.1.; and Appendix D, Section D.1.2.1

9.6 *Department of Defense Quality Systems Manual for Environmental Laboratories* (DoD QSM) Final Version 3 January 2006, Appendices C and D; Final Version 4.1, April 2009, Appendice C and D

9.7 *Manual for the Certification of Laboratories Analyzing Drinking Water*, US EPA, 5th Edition, January 2005 and Supplement, June 2008.

10.0 CHANGES FROM PREVIOUS REVISION

Title page	Revised title.
All pages	Removed footnotes, revised numbering to follow standard Word template
All pages	Moved some sec ions, minor wording/format changes for improved readability
Section 1.0	Revised to add LOQ
Section 2.0	Removed redundant previous last paragraph, added new 2nd paragraph.
Section 3.2	Revised to clarify new DOD LOD definition
Section 3.3	Revised to add MRL line from prior 3.1.2
Section 4.0	Revised and removed last paragraph. Removed redundant section 4.3.
Section 5 0	Revised to add/c ar fy responsibiliti s
Sec ion 6.1.	Added 'd fault' not for aqueous MDLs
Section 6.1.2	Move from previo s 6 13 for improved ow
Section 6.1.3	Revised
Section 6.2	Removed non-DoD vs DOD MDL evaluations.
Section 6.2.1	Revised, delete last sentence
Section 6.2.3	Revised and moved part of procedure to more applicable Section 7
Section 6.12.5	Revised and moved part of procedure to more applicable Section 7
Section 6.14	Revised and moved part of procedure to more applicable Section 7
Section 7	R arranged to improve flow/r adability. NOTE: Several significant changes
Section 7.5	Revised to add non-DOD vs DOD details.
Section 7.6	Revised to add non-DOD vs DOD details.
Section 7.7	New section
Section 8.0	Revised
Section 9.0	Removed ADM-BATCH reference, updated EPA and DOD references
Figure 1	Removed
Appendix A	Removed

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Plant Tissue SRMs:

	NIST SRM 1573a Tomato Leaves (mg/Kg)	Preliminary Acceptance Limits (mg/Kg)	NIST SRM 1575a Pine Needles (mg/Kg)	Preliminary Acceptance Limits (mg/Kg)	European Commission IRMM BCR-402 White Clover (mg/Kg)	Preliminary Acceptance Limits (mg/Kg)
Aluminum	598 ± 12	469-732	580 ± 30	440-732		
Antimony						
Arsenic						
Barium			6.0 ± 0.2	4.6-7.4		
Beryllium						
Cadmium			0.233 ± 0.004	0.183-0.284		
Calcium	50500 ± 900	39700-61700	2500 ± 100	1920-3120		
Chromium	1.99 ± 0.06	1.54-2.46				
Cobalt	0.57 ± 0.02	0.44-0.71			0.178 ± 0.008	0.136-0.223
Copper	4.7 ± 0.14	3.65-5.81	2.8 ± 0.2	2.1-3.6		
Iron	368 ± 7	289-450	46 ± 2	35-58		
Lead			0.167 ± 0.015	0.122-0.218		
Magnesium	12000*	9600-14400	1060 ± 170	712-1480		
Manganese	246 ± 8	190-305	488 ± 12	381-600		
Mercury	0.034 ± 0.004	0.024-0.046	0.0399 ± 0.0007	0.0314-0.0487		
Molybdenum					6.93 ± 0.19	5.39-8.54
Nickel	1.59 ± 0.07	1.22-1.99	1.47 ± 0.10	1.10-1.88		
Potassium	27000 ± 500	21200-33000	4170 ± 80	3270-5100		
Selenium					6.70 ± 0.25	5.16-8.34
Silver						
Sodium	136 ± 4	106-168	63 ± 1	50-74		
Thallium						
Vanadium						
Zinc	30.9 ± 0.7	24.2-37.9	38 ± 2	29-48		

* Uncertified value

Aquatic Tissue SRMs:

	NRCC TORT-2 Lobster Hepatopancreas (mg/Kg)	CAS Acceptance Limits (mg/Kg)	NIST SRM 2976 Mussle Tissue (mg/Kg)	Preliminary Acceptance Limits (mg/Kg)
Aluminum	-	-	134 ± 34 *	80-202
Antimony	-	-	-	-
Arsenic	21.6	15.8-28.1	13.3 ± 1.8	9.2-18.1
Barium	-	-	-	-
Beryllium	-	-	-	-
Cadmium	26.7	20.9-32.8	0.82 ± 0.16	0.53-1.18
Calcium	-	-	7600 ± 300 *	5840-9480
Chromium	0.77	0.5-1.1	0.50 ± 0.16 *	0.27-0.79
Cobalt	0.51	0.34-0.72	-	-
Copper	106	77-139	4.02 ± 0.33	2.95-5.22
Iron	105	74-142	171.0 ± 4.9	133-211
Lead	0.35	0.18-0.58	1.19 ± 0.18	0.81-1.64
Magnesium	-	-	5300 ± 500 *	3840-6960
Manganese	13.6	9.9-17.8	33 ± 2 *	25-42
Mercury	0.27	0.17-0.40	0.061 ± 0.0036	0.046-0.078
Molybdenum	0.95	0.68-1.26	-	-
Nickel	2.50	1.85-3.23	0.93 ± 0.12 *	0.65-1.26
Potassium	-	-	9700 ± 500 *	7360-12200
Selenium	5.63	3.97-7.56	1.80 ± 0.15	1.32-2.34
Silver	-	-	-	-
Sodium	-	-	35000 ± 1000 *	27200-43200
Thallium	-	-	-	-
Vanadium	1.65	1.46-2.2	-	-
Zinc	180	139-223	137 ± 13	99.2-180

* Non-Certified Reference Values

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

1317 South 13th Avenue
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(360) 577-7222

January 12, 2007

Approved by:

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DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

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3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is an employee-owned professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, and other material.

It is a policy at CAS that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities. Policies and procedures are established in order to meet the quality objectives of clients, accrediting authorities, and certifying organizations. The Quality System is established to meet the requirements of the National Environmental Laboratory Accreditation Conference (NELAC).

CAS maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

CAS is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, CAS also operates laboratories in California, Florida, New York, Arizona, and Texas.

The information in this document has been organized according to the format described in *EPA Requirements for Quality Management Plans, EPA QA/R-2,* USEPA, 2001; and *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* USEPA, 2001.

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at CAS is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of CAS:

> "The mission of Columbia Analytical Services, Inc., is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

In support of this mission, our QA program addresses all aspects of laboratory operations, including laboratory organization and personnel, standard operating procedures, sample management, sample and quality control data, calibration practices, standards traceability data, equipment maintenance records, method proficiency data (such as method detection limit studies and control charts), document control/storage and staff training records.

4.1 Facilities and Equipment

CAS features over 25,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, CAS minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals “clean room” sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Water Chemistry & General Chemistry Laboratories
- Semi-volatile Organics Sample Preparation Laboratories (3)
- Gas Chromatography/High Performance Liquid Chromatography Laboratory

- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Semi-volatile Organics Drinking Water Laboratory
- Volatile Organics Laboratory
  - Separate sample preparation laboratory
  - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water System
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.2 Technical Elements of the Quality Assurance Program

The Quality Assurance Program provides a platform on which technical operations are based. The program provides laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained. This includes method approvals and audit administration. In addition, internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, and preventative maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.3 Operational Assessments

There are a number of methods used to assess the laboratory and its daily operations. In addition to the routine quality control (QC) measurements to measure quality, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients. On-time performance, report quality, training, and Quality Assurance are a few of the items that are used to assess performance from an external perspective. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

CAS utilizes a number of different methods to ensure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results. All Requests for Proposal (RFP) documents are reviewed by the Project Chemist and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Plans (QAPPs) if available. A weekly status meeting is also conducted with the laboratory staff by the Client Services Manager to inform the staff of the status of incoming work, future projects, or project requirements.

4.4 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The procedures described in the SOP include distribution, tracking, filing, and copyrighting of CAS controlled documents. The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled CAS documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

CAS maintains a records system that ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH).*

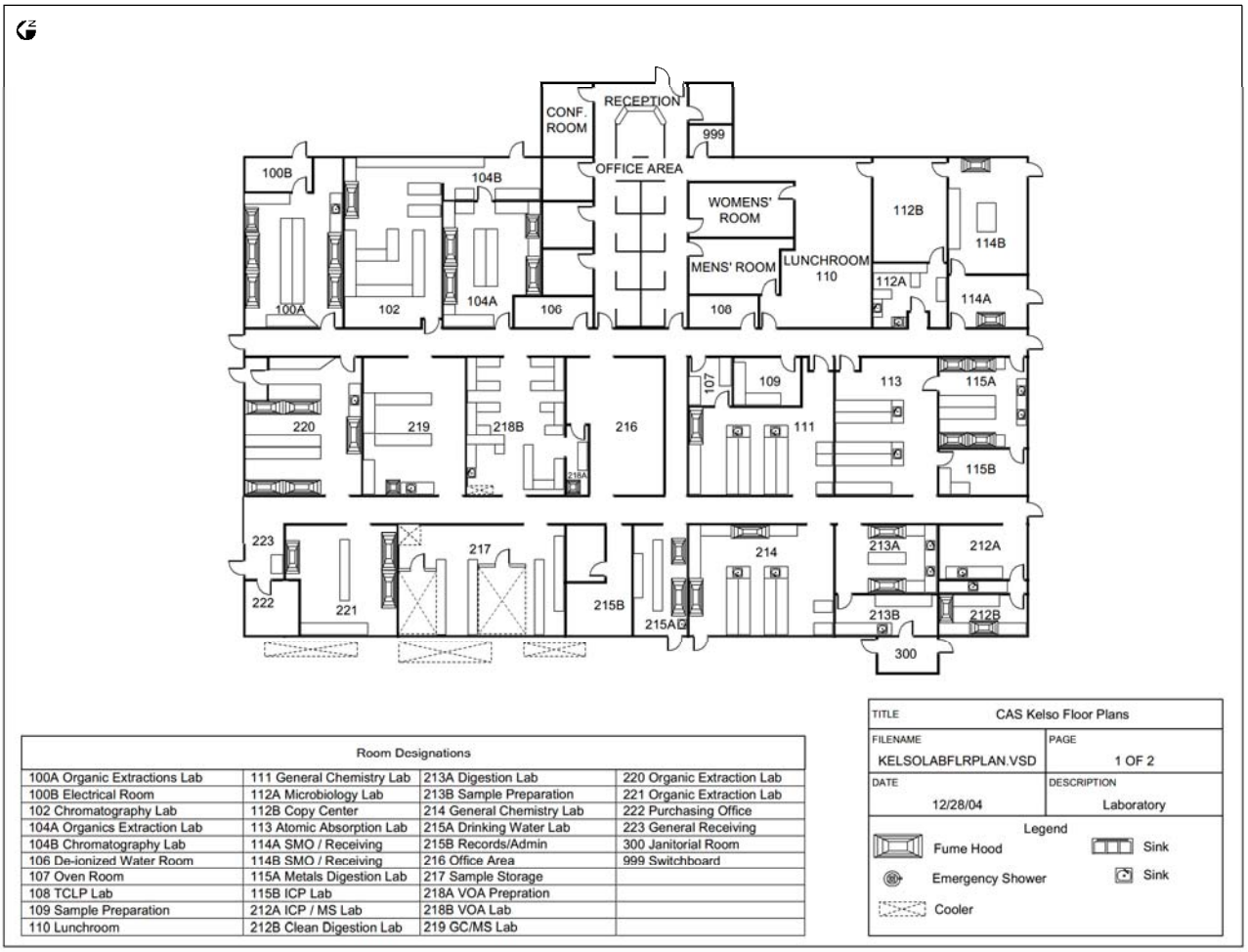
4.5 Subcontracting

Analytical services are subcontracted when CAS/Kelso needs to balance workload or when the requested analyses are not performed by CAS/Kelso. Subcontracting is only done with the knowledge and approval of the client. Subcontracting to another CAS laboratory is preferred over external-laboratory subcontracting. Further, sub-contracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories (ADM-SUBLAB).* The Quality Assurance Director is responsible for qualifying and oversight of subcontract laboratories.

4.6 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned (by the laboratory user) as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The procedures for purchasing and procurement are described in the *SOP for Purchasing through CAS Purchasing Agent (SOP ADM-PUR).* Also, refer to section 10.4 for a discussion of reference materials.

Figure 4-1
CAS/Kelso Laboratory Floor Plan



Room Designations			
100A Organic Extractions Lab	111 General Chemistry Lab	213A Digestion Lab	220 Organic Extraction Lab
100B Electrical Room	112A Microbiology Lab	213B Sample Preparation	221 Organic Extraction Lab
102 Chromatography Lab	112B Copy Center	214 General Chemistry Lab	222 Purchasing Office
104A Organics Extraction Lab	113 Atomic Absorption Lab	215A Drinking Water Lab	223 General Receiving
104B Chromatography Lab	114A SMO / Receiving	215B Records/Admin	300 Janitorial Room
106 De-ionized Water Room	114B SMO / Receiving	216 Office Area	999 Switchboard
107 Oven Room	115A Metals Digestion Lab	217 Sample Storage	
108 TCLP Lab	115B ICP Lab	218A VOA Prepration	
109 Sample Preparation	212A ICP / MS Lab	218B VOA Lab	
110 Lunchroom	212B Clean Digestion Lab	219 GC/MS Lab	

5.0 PROFESSIONAL CONDUCT AND ETHICAL PRACTICES

One of the most important aspects of the success of CAS is the emphasis placed on the integrity of the data provided and services performed. To promote product quality, employees are required to comply with certain standards of conduct and ethical practices. The following examples of CAS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action. Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.

- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible. Employee discipline is progressive in its severity and each situation is handled individually in that the discipline is designed to fit the circumstances. Potential disciplinary actions may include a verbal warning, written warning, a second written notice (more severe and more strongly worded than a warning), suspension without pay, demotion, or termination.

- It is the responsibility of all CAS employees to safeguard sensitive company and client information. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action.

All employees are required to sign and adhere to the requirements set forth in the CAS *Confidentiality and Conflicts of Interest Employee Agreement* and the CAS *Commitment to Excellence in Data Quality Policy.* All employees receive in-house ethics training and are periodically reminded of their data quality and ethical conduct responsibilities.

CAS makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the CAS Employee Handbook. This includes the CAS Ombudsman Program, the CAS Open Door Policy, and the use of flexible work hours. Operational assessments are regularly made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads (Section 4.3). Procedures for subcontracting work are established, and within the CAS laboratory network additional capacity is typically available for subcontracting, if necessary.

6.0 ORGANIZATION AND RESPONSIBILITIES

The CAS/Kelso staff, consisting of approximately 110 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

CAS is committed to providing an environment that encourages excellence. Everyone within CAS shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 6-1 lists the CAS/Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.
- The responsibility of the **Quality Assurance Manager** (QAM) is to oversee implementation of the quality program and to coordinate QA activities within the laboratory. The QAM works with laboratory production units to establish effective quality control and assessment plans. The QAM has the authority to stop work in response to quality problems. The QAM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and coordinating the annual review of each SOP; maintaining QA records such as metrological records, archived logbooks, PT sample results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; performing internal QA audits; preparing QA activity reports; etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the CAS Quality Assurance Director. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

  The Chief Quality Officer (CQO) is responsible for the overall QA program at all the CAS laboratories. The CQO is responsible for ensuring that annual internal audits are performed at each CAS laboratory; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of CAS-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Organics Department Manager (for the Laboratory Director) and the QA Director or Laboratory Director (for the QA manager).
- The **Environmental Health and Safety Officer** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to CAS' EH&S Director.
- The **Client Services and Sample Management Office Manager** is responsible for the Client Services Department (customer services/project chemists, and Electronic Data Deliverables group) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.
- The **Project Chemist** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the CAS laboratory and administrative staff to ensure that client-specific needs are understood, and that the services CAS provides are properly executed and satisfy the requirements of the client.
- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within the department. Each **Department Manager and Supervisor** has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.
- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.
- **Information Technology** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 6-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	28	Laboratory Director
Lee Wolf, B.S.	21	Quality Assurance Manager
Lynda Huckestein, B.S.	18	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	17	Inorganics Department Manager
Todd Poyfair, B.S.	15	Gas Chromatography and Petroleum Hydrocarbons Department Manager
Jeff Grindstaff, B.S.	18	Volatiles and Semivolatiles GC/MS Department Manager
Jim Smith, B.S.	19	Organics Drinking Water Department Manager
Eileen Arnold, B.A.	25	Environmental Health and Safety Officer
Paul Gowan, B.A.	20	Technical Information Specialist
Gary Ward, M.S.	31	CAS Information Technology Director CAS Chief Quality Officer
Steve Vincent, B.S.	31	CAS President

7.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. CAS management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major automated data processing equipment.

7.1 Software Quality Assurance Plan

CAS has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the CAS Software Quality Assurance Plan (SQAP). The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of configuration management plan are policies for controlling the software version that is in use in the laboratory.

7.2 IT Support

The local CAS Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, CAS corporate IT provides support for network-wide systems. CAS also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

7.3 Information Management Systems

CAS has various systems in place to address specific data management needs. The CAS Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system is used to establish and define sample identification, analysis specifications, and provide a means of sample tracking. This system is used during sample login to generate the internal Service Request. The Service

Request provides a summary of client information, sample information, required analyses, work instructions, deliverable requirements and other necessary information provided on the chain of custody. The LIMS also is the basis for valuable sample tracking mechanisms used throughout the laboratory. Laboratory analysts generate responsibility reports from the LIMS and perform internal chain of custody via the LIMS.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain) dedicated to this function. This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. Another server is dedicated to data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats, including Staged Electronic Data Deliverable (SEDD) format.

7.4 Backup and Security

CAS laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non-CAS access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. CAS uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. Email attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems.

8.0 SAMPLE MANAGEMENT

8.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. CAS recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

CAS uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 18 for complete citations). The container, preservation and holding time information for these references is summarized in Table 8-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for container, preservation and holding time information for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

CAS routinely provides sample containers with appropriate preservatives for our clients. The containers are purchased as precleaned to a level 1 status, and conform to the requirements for analytical samples established by the USEPA. Certificates of analysis for the sampling containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container. Figure 8-1 shows the chain-of-custody form routinely used at CAS and included with sample kits. For large sample container shipments, the containers may be

shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to CAS. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. CAS keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. CAS also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When CAS ships environmental samples to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures for maintaining the chain of custody of the sample(s) (SOP *for Chain of Custody for Sample Transfer between Laboratories* [SOP ADM-COC]), proper packaging and shipment, specification of proper methodology, etc. Blue or gel ice is the only temperature preservative used by CAS, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures are established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received. Complete documentation of all sample storage is maintained in order to preserve the integrity of the samples.

Once samples are delivered to the CAS sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 8-2 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);
- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.

- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Chemist and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Chemist for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are kept refrigerated until they undergo analysis, unless otherwise specified. CAS stores samples in various refrigerators or freezers, depending on the type of analysis and the matrix of the sample. CAS has five walk-in refrigerators which house the majority of sample containers received at the laboratory. In addition to the walk-in refrigerators, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. These refrigerators are segregated by matrix type (soil or water) and method of analysis. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. CAS also has six sub-zero freezers capable of storing samples at -20° C; these are primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit used at CAS is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

CAS adheres to the method-prescribed or project-specified holding times for all analyses. In order to comply with holding time requirements, the sampling date and time are entered into the LIMS system at the time of sample receipt and login. Each analyst then monitors holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. In order to report adherence to holding time requirements, the date and time analyzed is printed or written on the analytical raw data. For analyses with a holding time prescribed in hours it is essential that the sample collection time is provided, so that holding time compliance can be demonstrated based on the analysis time. If not, the sample collection time is assumed as the earliest in the day (i.e. the most conservative).

Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature for 30 days, soil samples are retained at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Environmental Health and Safety Manual.* All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

8.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN)*. Figure 8-1 is a copy of the chain-of-custody form routinely used at CAS.

Facility security and access is important in maintaining the integrity of samples received at CAS/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The CAS facility is equipped with an alarm system and CAS employs a private security firm to provide nighttime and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC).*

8.4 Project Setup

The analytical method(s) to be used for sample analysis are chosen based on the client's requirements. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Chemist will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Chemist ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the LIMS generated Service Request. To communicate and specify project-specific requirements, a Tier V form (Figure 8-3) is used and accompanies the service request form.

Table 8-1
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total	W, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (BOD)	W	P,G	Cool, 4°C	48 hours
Bromate	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (COD)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride – Method 300.0	W, DW	P,G	None Required	28 days
Chloride – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Chlorine, Total Residual	W, DW	P,G	None Required	24 hours
Chlorite	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI – Method 7196A	W	P,G	Cool, 4°C	24 hours
Color	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	W, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron	W, DW	G Amber	Cool, 4°C	24 hours
Fluoride – Method 300.0	W, DW	P,G	None Required	28 days
Fluoride – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Hardness	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrate – Method 300.0	W, DW	P,G	Cool, 4°C	48 hours
Nitrate – Method 353.2	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrate – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite – Method 353.2	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite – Method 300.0	W, DW	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitrite – Method 353.2	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrite – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Orthophosphate – Method 365.3	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe)	W, DW	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Perchlorate	W, DW	P,G	Protect from temp. extremes	28 days
Phenolics, Total	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Elemental	W	G Only	Cool, 4°C	48 hours
Phosphorus, Total	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	W	P,G	Cool, 4°C	7 days
Residue, Settleable	W	P,G	Cool, 4°C	48 hours
Residue, Volatile	W	P,G	Cool, 4°C	7 days
Silica	W	P Only	Cool, 4°C	28 days
Specific Conductance	W, DW	P,G	Cool, 4°C	28 days
Sulfate – Method 300.0	W, DW	P,G	Cool, 4°C	28 days
Sulfate – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Sulfide	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite	W	P,G	None Required	24 hours
Surfactants (MBAS)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin	W	P,G	Cool, 4°C	28 days
Temperature	W	P,G	None Required	Analyze immediately
Turbidity	W, DW	P,G	Cool, 4°C	48 hours
Metals				
Metals, except CrVI and Mercury	W, DW	P,G	HNO_3 to pH<2	6 months
	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Chromium VI – Method 7195	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
	S	P,G	Cool, 4°C	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Organic Tests				
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (TOC)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (TOX)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (AOX)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction
Volatile Organics				
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days
Purgeable Halocarbons	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
EDB and DBCP	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Alcohols and Glycols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phenols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phthalate Esters	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrosamines	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitroaromatics and Cyclic Ketones	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Polynuclear Aromatic Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Haloethers	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Resin and Fatty Acids	W	G, Teflon-Lined Cap	NaOH to pH $\geq$10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Drinking Water Organics				
Purgeable Organics	DW	G, Teflon-Lined Septum Cap,	Ascorbic Acid, HCl to pH$\leq$2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP	DW	G, Teflon-Lined Septum Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Carbamates, Carbamoyloximes	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH<3; 80 mg/L $Na_2S_2O_3$ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/4omL NaS; Cool, <6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH$\leq$ 2; Cool, 4°C	14 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Diquat and Paraquat	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$ if Res.Cl., Cool, 4°C,	7days until extraction; 21 days after extraction
Endothall	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$, Cool, 4°C	14 days
Haloacetic Acids	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH$\leq$ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)				
Mercury	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction
Semivolatile Organics	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction

a See Section 18.0 for sources of holding time information.
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass
d For chlorinated water samples
e The maximum holding time is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

Figure 8-1
Chain of Custody Form

Columbia Analytical Services INC.
An Employee-Owned Company

CHAIN OF CUSTODY

1317 South 13th Ave. • Kelso, WA 98626 • (360) 577-7222 • (800) 695-7222x07 • FAX (360) 636-1068

SR#: _____

PAGE _____ OF _____ COC # _____

PROJECT NAME
PROJECT NUMBER
PROJECT MANAGER
COMPANY/ADDRESS
PHONE # | FAX #
SAMPLER'S SIGNATURE

SAMPLE I.D.	DATE	TIME	LAB I.D.	MATRIX	NUMBER OF CONTAINERS	Semivolatile Organics by GC/MS 625 ☐ 8270 ☐ 8270LL ☐	Volatile Organics 624 ☐ 8260 ☐	Hydrocarbons (*see below) Gas ☐ Diesel ☐ Oil ☐ ☐ Fuel Fingerprint (FIQ) ☐ NW-HCID Screen	Oil & Grease/TRPH 1664 HEM ☐ 1664 SGT ☐	PCB's Aroclors ☐ Congeners ☐	Pesticides/Herbicides 608 ☐ 8081A ☐ 8141A ☐ 8151A ☐	Chlorophenolics - 8151M Tri ☐ Tetra ☐ PCP ☐	PAHS 8310 ☐ SIM ☐	Metals, Total or Dissolved (See list below)	Cyanide ☐ Hex-Chrom ☐	pH, Cond., Cl, SO_4, PO_4, F, NO_2, NO_3, BOD, TSS, TDS (circle)	NH_3-N, COD, Total-P, TKN, TOC, DOC (circle) NO_2+NO_3	TOX 9020 ☐ AOX 1650 ☐ 506 ☐						REMARKS

REPORT REQUIREMENTS

___ I. Routine Report: Method Blank, Surrogate, as required
___ II. Report Dup., MS, MSD as required
___ III. Data Validation Report (includes all raw data)
___ IV. CLP Deliverable Report
___ V. EDD

INVOICE INFORMATION

P.O. # _____
Bill To: _____

TURNAROUND REQUIREMENTS

____ 24 hr. ____ 48 hr.
____ 5 Day
____ Standard (10-15 working days)
____ Provide FAX Results

_____
Requested Report Date

Circle which metals are to be analyzed:

Total Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

Dissolved Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

*INDICATE STATE HYDROCARBON PROCEDURE: AK CA WI NORHTWEST OTHER:_____ (CIRCLE ONE)

SPECIAL INSTRUCTIONS/COMMENTS:

RELINQUISHED BY:	RECEIVED BY:	RELINQUISHED BY:	RECEIVED BY:
Signature / Date/Time	Signature / Date/Time	Signature / Date/Time	Signature / Date/Time
Printed Name / Firm	Printed Name / Firm	Printed Name / Firm	Printed Name / Firm

RCOC #1 04/02

Figure 8-2

Columbia Analytical Services Inc.
Cooler Receipt and Preservation Form

Project/Client_____Work Order K060_____

Cooler received on _____ and opened on _____by_____

1.	Were custody seals on outside of coolers?	Y	N
	If yes, how many and where?_____		
2.	Were seals intact and signature & date correct?	Y	N
3.	Is the shipper's airbill available and filed? If no, record airbill number:_____	Y	N
4.	COC# _____ _____ _____ _____		
	Temperature of cooler(s) upon receipt: _____ _____ _____ _____		
	Temperature Blank: _____ _____ _____ _____		
5.	Were custody papers properly filled out (ink, signed, etc.)?	Y	N
6.	Type of packing material present_____		
7.	Did all bottles arrive in good condition (unbroken)?	Y	N
8.	Were all bottle labels complete (i.e analysis, preservation, etc.)?	Y	N
9.	Did all bottle labels and tags agree with custody papers?	Y	N
10.	Were the correct types of bottles used for the tests indicated?	Y	N
11.	Were all of the preserved bottles received at the lab with the appropriate pH?	Y	N
12.	Were VOA vials checked for absence of air bubbles, and if present, noted below?	Y	N
13.	Did the bottles originate from CAS/K or a branch laboratory?	Y	N
14.	Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?	Y	N
15.	Was C12/Res negative?	Y	N

Explain any discrepancies:_____
_____
_____
_____
_____
_____

RESOLUTION:_____

Samples that required preservation or received out of temperature:

Sample ID	Reagent	Volume	Lot Number	Bottle Type	Rec'd out of Temperature	Initials

Figure 8-3
Tier V Form

Client :

Project Chemist :

Project Name :

Service Request :

Project Number :

SMO LimsTemplate ID :

Project Description :

QAPP/SOW Information :

Reporting

Tier Level :

PDF:

Report to :

In result field use :

EDD :

Flagging Requirements :

Other Requirements :

Sample Considerations

Sample Limitations :

Sample Prep/Analysis :

Non-Standard Holdtimes :

Historical Data :

Comments :

9.0 ANALYTICAL PROCEDURES

CAS employs methods and analytical procedures from a variety of sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 18.0. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by CAS is described in SOPs specific to each method. A list of SOPs and NELAC-accredited methods are given in Appendix E. Further details are described below.

9.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

CAS maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *Making Entries into Logbooks and onto Benchsheets* SOP (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

9.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

9.3 Modified Procedures

CAS strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

9.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that CAS has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples, are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.

2) All (field) samples in a batch are of the same matrix.

3) The QC samples to be processed with the (field) samples include:

   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b) Laboratory Control Sample (a.k.a. Laboratory Fortified Blank)

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.

4) A single lot of reagents is used to process the batch of samples.

5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.

7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

9.5 Specialized Procedures

CAS not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are reductive precipitation metals analysis, specialized GC/MS analyses, and ultra-low level pesticides and PCBs.

9.6 Sample Cleanup

CAS commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

10.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at CAS are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Records are maintained to provide traceability of reference materials.

Any item of the equipment which has been subjected to overloading or mishandling, or has been shown by verification or otherwise to be defective; is taken out of service until it has been repaired. The equipment is placed back in service only after verifying by calibration that the equipment performs satisfactorily. An evaluation of the effect of this defect on previous calibrations or tests is made and documented appropriately.

Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. For NELAP accredited methods, the concentration of calibration verification standards are varied within the calibration range periodically.

10.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators. Bound record books are kept which contain daily-recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC).* The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a professional metrology organization on an annual basis.

10.2 Analytical Balances

Analytical balances are serviced on a semi-annual basis by a professional metrology organization. New certificates of calibration for each balance are issued to the laboratory on a semi-annual basis. The calibration of each analytical balance is checked by the user each day

of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels within the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The weights are recertified using NIST traceable standards by a professional metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC.

10.3 Water Purification System

The water purification system is designed to produce deionized water with specific resistivity no less than 18 megohms-cm at 25°C, meeting specifications for Type I water, as described in *Standard Methods for the Examination of Water and Wastewater (SM1080)*. The system is monitored continuously for conductivity and resistivity with an on-line meter, which is recorded daily in a bound record book. The accuracy of the meter is verified annually. Deionizers are rotated and replaced on a regular schedule, and are monitored by an indicator light on the unit. The status of the system is monitored and recorded daily in a bound record book following a written SOP. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. Water for microbiology is checked at a point downstream of the purification system at a tap in the laboratory, and the monitoring documented.

10.4 Source and Preparation of Standard Reference Materials

All analytical measurements generated at CAS are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type. This information is provided to the client when requested.

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. CAS relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

10.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

10.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

10.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be “dropped” from the resulting calibration curve.

10.8 GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. Compounds selected as system performance check compounds (SPCCs) must show a method-specified response factor in order for the calibration to be considered valid. Calibration check compounds (CCCs) must also meet method specifications for percent difference from the multipoint calibration. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.10 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be "dropped" from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at CAS include total phenolics, phosphates, surfactants and tannin-lignin.

10.11 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be "dropped" from the resulting calibration curve. Standard CAS acceptance limits are used to evaluate the calibration curve prior to sample analysis.

10.12 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a 5-point calibration curve. Initial calibration points cannot be "dropped" from the resulting calibration curve. A correlation coefficient of $\geq$ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and bromide. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

10.13 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

10.14 Ion-selective electrode

The method-prescribed number of standards (typically two for Standard Methods and EPA wastewater methods) are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

10.15 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP for Checking Pipet Calibration*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

10.16 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

11.0 QUALITY CONTROL

A primary focus of Columbia Analytical Services Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis of field samples, acceptable method performance is established by performing demonstration of capability analyses and performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. CAS has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the methodology or are statistically derived based on the laboratory's actual historical data obtained from the various QC measurements for each analytical method. The Quality Control objectives are defined below.

11.1 Quality Control Objectives

11.1.2 Demonstration of Capability - Where required by mandatory test method, regulation, or accreditation protocols, a demonstration of capability (DOC) is made prior to using any test method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control reference material or quality control sample is obtained. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results or MDL study results may be used to meet this requirement, provided acceptance criteria is met.

11.1.3 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or

caused by an artifact of the measurement system (e.g., contamination). CAS utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

11.1.4 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

11.1.5 Control Limits - The control limits for accuracy and precision originate from two different sources: For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the Quality Assurance Manager. The new control limits replace the previous limits and data is assessed using the new values. The current acceptance limits for accuracy and precision are available from the laboratory and on the accompanying CD-ROM. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

11.1.6 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. CAS has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. These include the *SOP for Subsampling and Compositing of Samples* and the *SOP for Tissue Sample Preparation*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

11.1.8 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The CAS objective is to generate data of known quality with the highest level of comparability, completeness, and usability to the client. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using specified data qualifiers where necessary.

11.2 Method Detection Limits and Method Reporting Limits

Method Detection Limits (MDL) for analytical methods routinely performed at CAS/Kelso are determined annually, thus may change slightly from year to year. The MDLs are determined by following the *SOP for the Determination of Method Detection Limits,* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAC and DoD protocols, the validity of derived MDLs is verified using MDL verification samples. The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. the lower limit of quantitation). Therefore, analyses are calibrated to the MRL, or lower. To take into account day-to-day fluctuations in instrument sensitivity, analyst performance, and other factors, the MRL is established at three times the MDL (or greater). The current MDLs and MRLs are available from the laboratory.

11.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below. In addition, a number of other quality control processes that may impact analytical results are also described below.

11.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is either analyte-free water or analyte-free soil (when available), subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute may be used instead. The method blank is analyzed to demonstrate that the analytical system itself is not contaminated with the analyte(s) being measured. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, below half of the MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

11.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

11.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

11.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

11.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in the calibration. Once it is determined that there is no reference material defect or systematic error in preparation of the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

11.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

11.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

11.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

11.3.9 Laboratory Control Samples (a.k.a. Laboratory Fortified Blanks)

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is(are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples. The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

11.3.10 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is(are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

11.3.11 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$\textit{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

11.3.12 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

11.3.13 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

11.3.13 Control Charting

The generation of control charts is routinely performed at CAS. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

11.3.14 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at CAS undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at CAS; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

CAS reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the *SOP for Laboratory Data Review Process.*

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the “before” and “after” integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (SOP ADM-INT).

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.

- The sample meets all of the following requirements:

    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.

    2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods

    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).

    2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.

- GC/MS Methods - Two criteria are used to verify identification:

    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.

    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Review and Validation

The integrity of the data generated is assessed through the evaluation of the results of the calibrations and analysis of QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below. Details are provided in applicable SOPs. The numerical criteria for evaluation of these QC samples are listed within each method-specific Standard Operating Procedure. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAC Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, AFCEE QAPP protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.

- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications, or a new initial calibration is performed. For DoD projects, the concentration of these two consecutive must be varied as required by the DoD QSM, Version 3.

- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.

- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) it is verified that the result is within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. For sample and sample duplicate analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly and qualified. The MRL must meet project requirements.

- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. It is verified that the result is within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly and qualified. The MRL must meet project requirements.

- Surrogate Results (Organic) – Following sample analysis and calculations the percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable and other sample evaluation is complete, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.

- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.

- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. The source of the problem is identified and, depending on the source of the problem, the LCS and the associated batch is reanalyzed or re-prepared and reanalyzed.

- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. CAS maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Reporting and Report Generation* addresses the flagging and qualification of data. The CAS-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the project chemist to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Chemist verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Chemist accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the CAS client.

12.5 Documentation

CAS maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the *SOP for Data Archiving*.

12.5.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

12.5.2 Documentation of Batch-related QC and Calibration Data

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

12.6 Deliverables

In order to meet individual project needs, CAS provides several levels of analytical reports. Basic specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) to following specialized deliverables:

- ADEC – Alaska Department of Conservation specified data package
- ACOE/HTRW – Army Corps of Engineers HTRW specified data package and reporting requirements
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, CAS provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 12-1
Descriptions of CAS Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results and acceptance criteria for applicable organic methods
5. Chain of custody documents
6. Dates of sample preparation and analysis for all tests

Tier II and IIA. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Tier IIA also includes Laboratory Control Sample (LCS) result(s) with calculated recovery and including associated acceptance criteria

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or Quality Control check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Summary forms for associated QC and calibration parameters
6. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Results and spectra of TIC compounds will be included upon request.

Tier IV. CLP-Level Data Validation Package.

A complete Data Validation Package containing all sample results, quality control and calibration results, and raw data necessary to fulfill all deliverable requirements of an EPA Contract Laboratory Program (CLP) data package.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of CAS/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of CAS/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which CAS/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of CAS/Kelso are conducted regularly by the Quality Assurance Manager. The internal audit procedures are described in the *SOP for Internal Audits*. The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that systems, technical operations, hardcopy data, and electronic data are assessed.
- Hardcopy report audits – minimum of 3 per quarter.
- Electronic audit trail reviews – each applicable instrument per quarter.

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within 30 days. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the 30 day corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices and files, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

13.2 Performance Audits

CAS/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. CAS routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the CAS Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a possible cause for the deficiency, and corrective action is taken and documented.

Table 13-1
Current CAS Performance and System Audit Programs

Federal and National Programs

- Naval Facilities Engineering Service Center
  Validated Laboratory for NFESC Parameters
- U.S. Air Force, Air Force Center for Environmental Excellence (AFCEE)
  Approved Laboratory for AFCEE Projects
- U.S. Army Corps of Engineers - MRD, HTRW Center of Expertise
  Validated Laboratory for HTRW parameters
- National Environmental Laboratory Accreditation Conference (NELAC) Accredited

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory, Lab I.D. UST040
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of Arkansas, Department of Environmental Quality
  Certified Environmental Laboratory, Lab I.D. 88-0637
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Colorado, Department of Public Health and Environment
  Certified Drinking Water Laboratory
- State of Florida, Department of Health
  Primary NELAC Accreditation No. E87412
- State of Hawaii, Department of Health
  Certified Drinking Water Laboratory
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Indiana, Department of Health
  Certified Drinking Water Laboratory, Lab I.D. C-WA-01
- State of Louisiana, Department of Environmental Quality
  Accredited Environmental Laboratory, Lab I.D. 3016
- State of Louisiana, Department of Health and Hospitals
  Accredited Drinking Water Laboratory, Lab I.D. LA070008
- State of Maine, Department of Human Services
  Certified Environmental Laboratory, Lab I.D. WA0035
- State of Michigan, Department of Environmental Quality
  Certified Drinking Water Laboratory, Lab I.D. 9949
- State of Minnesota, Department of Health
  Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nevada, Division of Environmental Protection
  Certified Drinking Water Laboratory, Lab I.D. WA35
- State of New Jersey, Department of Environmental Protection
  Accredited Environmental Laboratory, Lab I.D. WA005

Table 13-1 (continued)

State and Local Programs (continued)

- State of New Mexico, Environment Department
  Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
  Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
  Accredited Environmental Laboratory, Lab I.D. WA200001
- State of Pennsylvania Department of Environmental Protection
  Registered Environmental Laboratory
- State of South Carolina, Department of Health and Environmental Control
  Certified Environmental Laboratory, Lab I.D. 61002
- State of Utah, Department of Health, Division of Laboratory Services
  Accredited Environmental Laboratory
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
  Accreditation No. C1203
- State of Wisconsin, Department of Natural Resources
  Accredited Environmental Laboratory, Lab I.D. 998386840

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at CAS (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at CAS contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at CAS before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Equipment records also include a copy of the manufacturer's manual(s) and dates and results of calibrations.

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at CAS. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D.

This inventory or “parts list” also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix D for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives outlined in Section 11, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist may be notified in order to ascertain if contact with the client is necessary.

Problems with analysis, as well as the corresponding corrective actions taken, are documented on Nonconformity and Corrective Action Reports (See Figure 15-1) following the requirements in the *SOP for Nonconformity and Corrective Action Documentation* (SOP No. ADM - NCAR). This form is utilized to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

Corrective action due to a performance audit or a check sample problem is initiated by the Quality Assurance Manager; the affected laboratory supervisors and managers are promptly informed of performance audit results requiring corrective action.

Figure 15-1

Nonconformity and Corrective Action Report

NONCONFORMITY

N&CA Report No. _____

PROCEDURE (SOP or METHOD): EVENT DATE: _____

EVENT: ☐ Missed Holding Time ☐ QC Failure ☐ Lab Error (spilled sample, spiking error, etc.)
☐ Method Blank Contamination ☐ Login Error ☐ Project Management Error
☐ Equipment Failure ☐ Unacceptable PT Sample Result ☐ Other (describe):

SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED

DETAILED DESCRIPTION

ORIGINATOR: _____ DATE: _____

PROJECT CHEMIST(s): _____ NOTIFIED BY: _____ DATE: _____

CORRECTIVE ACTION AND OUTCOME

Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent its reoccurrence. Include Project Chemist instructions here.

Is the data to be flagged in the Analytical Report with an appropriate qualifier? ☐ **No** ☐ **Yes**

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____ Date: _____
Comments:

QA PM Verification and Approval of Corrective Action _____ Date: _____
Comments:

Customer Notified by ☐ Telephone ☐ Fax ☐ E-mail ☐ Narrative ☐ Not notified
Project Chemist Verification and Approval of Corrective Action _____ Date: _____
Comments: (Retain record)

Page 1 of 1
Original: Customer File Photocopies: Supervisor and QA PM N&CA Report 2004.doc 3/26/2004

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by CAS personnel at all levels of the organization. Information flow and feedback mechanisms are designed so that analysts, supervisors and managers are aware of quality assurance issues in the laboratory. Analysts performing routine testing are responsible for generating a Data Quality Report (DQR), or similar form, with every analytical batch they process. This report contains explicit documentation of the various controls that must be met during the analysis. This report also allows the analyst to provide appropriate notes and/or a case narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and corrected if possible.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness. The project chemist must also review the entire body of data to ensure that any and all client-specified objectives were successfully achieved. A case narrative may be written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists as required (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QAM provides the Laboratory Director with quarterly reports that summarize the various QA/QC activities that occurred during the previous quarter. The report addresses such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

Any operational or quality assurance problems noted by the Laboratory Director are then addressed during the senior staff operations meetings with all appropriate department managers. The Laboratory Director also performs an annual documented review of the laboratory quality system to identify any necessary changes or improvements to the quality system or quality assurance policies.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at CAS are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at CAS when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at CAS. Safety training begins with the reading of the *Environmental Health and Safety Manual.* Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer. Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s).

Each employee participates in Ethics training, which is part of the CAS Improper Practices Prevention Program. CAS also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The "CAS University" education system, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

A training plan is developed for each Standard Operating Procedure. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of proficiency. Where the analyst performs the entire procedure, a generic training plan may be used. In cases where work cells are used, a training plan specific to the work cell is established.

17.1 Initial Demonstration of Capability

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).

- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision. (For use of this option, LCSs must be from "second-source" standard materials independent of the calibration standards materials.).

- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.

- Where one of the three above is not possible, special requirements are as follows:

    - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
    - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
    - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 17-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

17.2 Continuing Demonstration of Proficiency

A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind PT sample analyses using the test method, or a similar test method using the same technology.

- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.

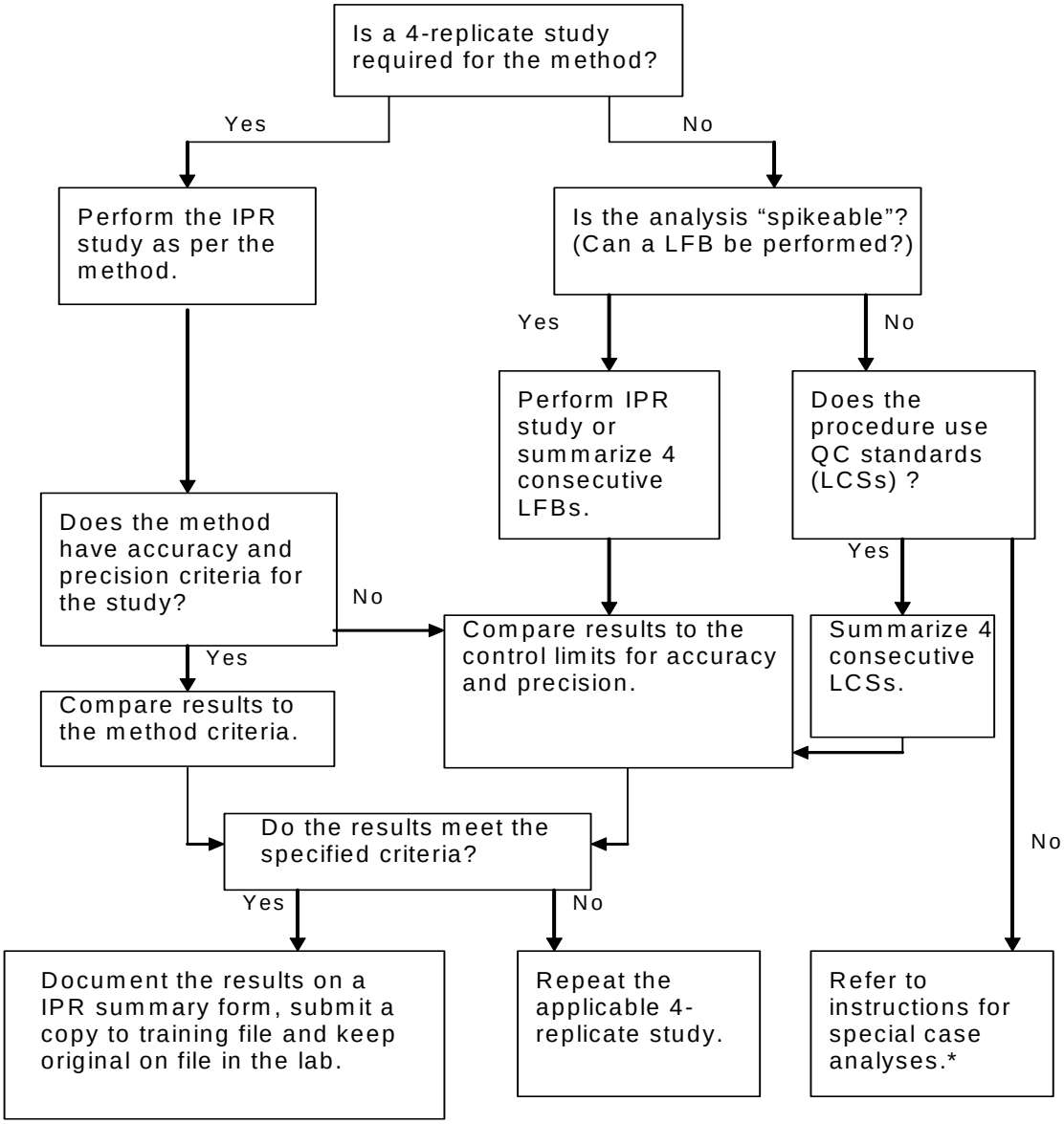
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.

- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

17.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and CAS resumes. A database is used to record the various technical skills and training acquired while employed by CAS. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Training (SOP No. ADM-TRANDOC).*

Figure 17-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per NELAC requirements
*Total Settleable Solids: Successful PT sample analysis and duplicate results with RPD<10%.
*Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
* Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES

The analytical methods used at CAS generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at CAS are taken from the following references:

- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Proposed Update IV, and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4. Unless otherwise specified, the most current published version is used.

- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).

- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).

- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplements.

- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.

- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.

- *Standard Methods for the Examination of Water and Wastewater*, 16th Edition (1985); 17th Edition (1989); 18th Edition (1992); 19th Edition (1995). See Introduction in Part 1000.

- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.

- 40 CFR Part 141, National Primary Drinking Water Regulations.

- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.

- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).

- Annual Book of ASTM Standards, Part 31, Water.
- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM01.8, OLM02.0, OLM03.1, OLM03.2, OLM04.2, and OLM04.3.
- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0, ILM04.1, and ILM05.2.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).
- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).
- *Identification and Listing of Hazardous Waste,* California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).
- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).
- National Environmental Laboratory Accreditation Conference (NELAC), 2003 Quality Standards.
- *Department of Defense Quality Systems Manual for Environmental Laboratories*, Final Version 2 (June 2002) and Final Version 3 (January 2006).

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

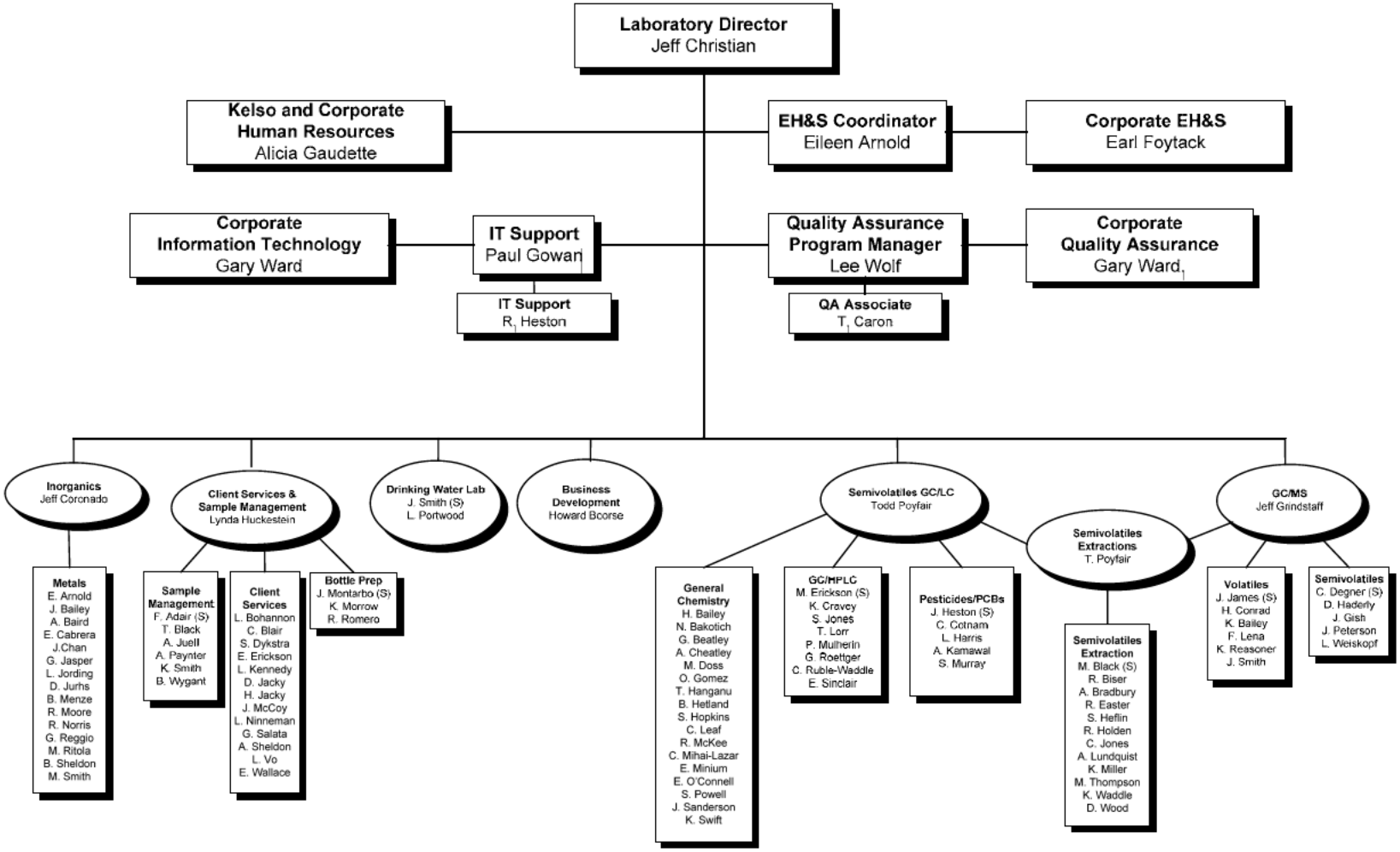
Quality Assurance Manual	1/10/07
Software Quality Assurance Plan	7/11/05
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	AppSignatories.pdf
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
Quality Control Acceptance Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001
TECHNICAL STANDARD OPERATING PROCEDURES	
SOP TABLE OF CONTENTS	SOPLIST.DOC
ADMINISTRATIVE STANDARD OPERATING PROCEDURES	
ADMINISTRATIVE - CORPORATE	**FILE NAME**
CHAIN OF CUSTODY FOR SAMPLE TRANSFER BETWEEN LABORATORIES	ADM-COC
CHECKING NEW LOTS OF CHEMICALS FOR CONTAMINATION	ADM-CTMN
CONTROL LIMITS	ADM-CTRL_LIM
DEALING WITH COMPLAINTS	ADM-CMPLT
DOCUMENT CONTROL	ADM-DOCCTRL
DOCUMENTATION OF TRAINING	ADM-TRANDOC
ELECTRONIC DATA AUDITING	ADM-E_DATAUDIT
MAKING ENTRIES INTO LOGBOOKS AND ONTO BENCHSHEETS	ADM-DATANTRY
MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS	ADM-INT
NONCONFORMITY AND CORRECTIVE ACTION	ADM-NCAR
PREPARATION OF STANDARD OPERATING PROCEDURES	ADM-SOP
PURCHASING THROUGH CAS PURCHASING AGENT	ADM-PUR
QUALIFICATION OF SUBCONTRACT LABORATORIES	ADM-SUBLAB
SAMPLE BATCHES	ADM-BATCH
SIGNIFICANT FIGURES	ADM-SIG.FIG
THE DETERMINATION OF METHOD DETECTION LIMITS AND LODs	ADM-MDL

ADMINISTRATIVE – LOCAL LABORATORY	**FILE NAME**
ARMY CORPS OF ENGINEERS HTRW PROJECT MANAGEMENT	ADM-HTRW
CHECKING PIPET CALIBRATION	ADM-CPIP
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	ADM-ECP
CONTROL CHARTING QUALITY CONTROL DATA	ADM-CHRT
DATA ARCHIVING	ADM-ARCH
DATA REPORTING AND REPORT GENERATION	ADM-RG
ELECTRONIC DATA BACKUP AND ARCHIVING	ADM-EBACKUP
FACILITY AND LABORATORY CLEANING	ADM-FACL
INTERNAL QUALITY ASSURANCE AUDITS	ADM-IAUD
LABORATORY DATA REVIEW PROCESS	ADM-DREV
METHOD DETECTION LIMIT DOCUMENTATION AND CONTROL	ADM-MDLC
PROJECT MANAGEMENT	ADM-PCM
REAGENT LOGIN AND TRACKING	ADM-RLT
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC
THERMOMETER CALIBRATION	ADM-TCAL
SAMPLE MANAGEMENT SOPS	**FILE NAME**
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC

APPENDIX B

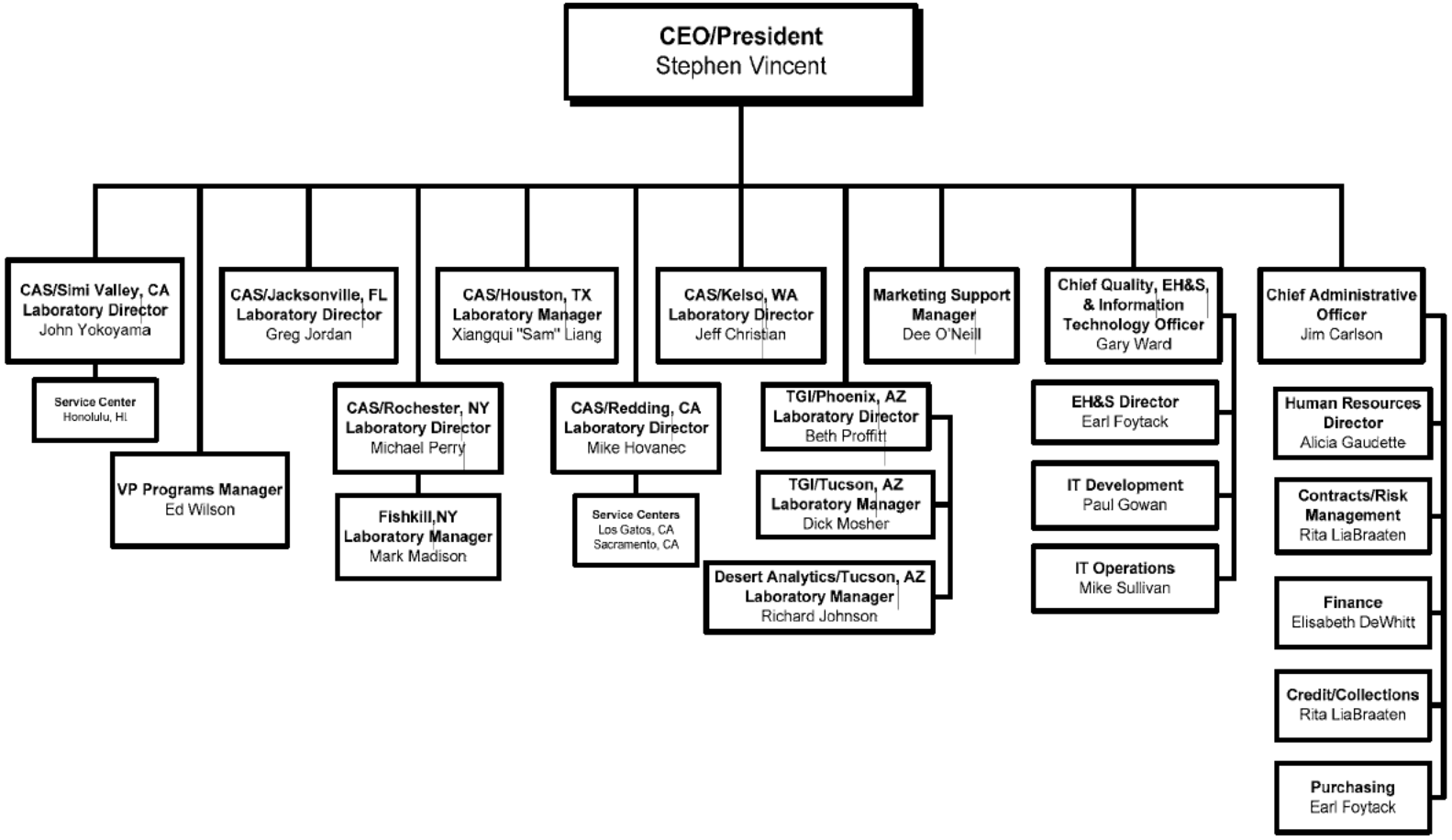
ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

Columbia Analytical Services, Inc.
Kelso, Washington Laboratory Organization



Revised 1/12/07

Columbia Analytical Services, Inc.
Laboratory Division Organization



JEFFREY D. CHRISTIAN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

VICE PRESIDENT/NW REGIONAL DIRECTOR – 1996 to Present

Responsibilities

Responsible for all phases of laboratory operations at the Kelso (WA) and Redding (CA) facilities, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory (i.e. serves as the Kelso Laboratory Director, 1993-present). Also responsible for additional duties acquired as a member of the Columbia Analytical Services Holdings, Inc., Board of Directors.

Experience

Laboratory Director, Kelso Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance.

Operations Manager, Kelso Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts.

Project Chemist and Manager, Metals Analysis Laboratory, *Columbia Analytical Services, Kelso, Washington,* 1989-1992. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories.

Scientist, *Weyerhaeuser Technology Center, Federal Way, Washington,* 1986-1989. Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods.

Lead Technician, Metals Lab, *Weyerhaeuser Technology Center, Federal Way, Washington*, 1981-1986. Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing.

Research Assistant, *ITT Rayonier, Olympic Research Division, Shelton, Washington,* 1978-1981. Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry.

Education

B.S., Chemistry, *Evergreen State College, Olympia, Washington,* 1993.
ICP/MS Training Course, *VG-Elemental,* 1992.
Coursework, *Pacific Lutheran University, Tacoma, Washington.* 1988-1989.
Coursework, *Tacoma Community College, Tacoma, Washington.* 1970-1971, 1988-1989.
Perkin-Elmer Advanced Furnace, *Norwalk, Connecticut,* 1986.
CERTIFICATION, Chemistry, *L.H. Bates Technical, Tacoma, Washington,* 1978.
Coursework, *Central Washington University, Ellensburg, Washington.* 1969-1970.

Publications/ Presentations

On request.

LEE E. WOLF

1988 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER IV, KELSO QUALITY ASSURANCE MANAGER – 2002 to Present

Responsibilities

Responsible for the overall coordination of the laboratory QA program, and for ensuring that established quality objectives are met. Responsible for Quality Assurance function, including the Quality Assurance Manual, certifications, documenting SOPs, and maintaining performance evaluation records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits. Provides audit responses and initiates any changes in procedures resulting from an audit. Coordinate the analysis of performance evaluation samples required for certification/accreditation programs. Report and review results for these analyses. Conduct internal audits and make recommendations for corrective action.

Experience

Scientist IV, Kelso Quality Assurance Manager, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-2002. Duties primarily as listed above.

Project Chemist/Principal Organic Scientist, *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1996. Responsibilities included GC and GC/MS method development and special projects coordination. Acts as technical advisor to the GC and GC/MS laboratories and GC/MS interpretation specialist and CLP organics specialist. Also responsible for Project Chemist functions, including management and coordination of projects for clients, identifying client needs, and preparation of data reports.

Semi-VOA Department Manager, *Columbia Analytical Services,* 1988-1994. Responsibilities included overall management of the Semi-VOA department. Oversee the operation of Semi-VOA GC/MS, data review and reporting and related QA/QC function. Also responsible for supervision of staff, including training, scheduling, and other personnel issues. Beginning in 1992, increased responsibilities to include Project Chemist functions for organics EPA-SAS and other clients. This involved scheduling projects for clients, identifying client needs, and preparing data reports.

GC/MS Chemist, *U.S. Testing Co., Richland, Washington,* 1985-1988. Responsibilities included GC and GC/MS analysis of water and soil samples for volatiles and Semi-VOA by EPA protocol, including Methods 8240, 8270 and CLP. Coordinated extraction and GC-GC/MS areas to manage sample/data flow through the lab. Experience also with pesticide/PCB analysis by EPA Methods 8080 and CLP. Responsible for development of analysis methods for non-routine pesticides and herbicides and performed HPLC analysis.

Laboratory Assistant, *Eastern Washington University, Cheney, Washington,* 1985. Responsibilities included supervision and instruction of organic chemistry labs. Experience with GC and IR operation. Responsible for lab safety.

Chemist Assistant, *Spokane County Air Pollution Control Authority, Spokane, Washington,* 1984. Responsibilities included gathering and analyzing air samples for CO content using IR equipment.

Education

Documenting Your Quality System, *A2LA Short Course, Las Vegas, Nevada,* 1998.
Internal Laboratory Audits, *A2LA Short Course, Las Vegas, Nevada,* 1998.
Mass Spectra Interpretation, *ACS Short Course, Denver, Colorado,* 1992.
BS, Chemistry, Minor in Geology, *Eastern Washington University, Cheney, Washington,* 1985.

Publications/ Presentations

On Request.

Affiliations

American Chemical Society.

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

CLIENT SERVICES MANAGER IV – 1998 to Present

Responsibilities

Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Personally responsible for approximately 1.5 million dollars of client work annually performing technical project management and client service. Provides technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Chemist, *Columbia Analytical Service, Inc., Kelso, Washington,* 1992-1998. Primary responsibilities included technical project management and client service in areas of pulp & paper, marine services, mining, and DOD. Also responsible for providing technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory

Project Chemist and Department Manager, General Chemistry Laboratory, *Columbia Analytical Services, Inc.,* 1989-1992. Responsible for management of the General Chemistry laboratory for routine wastewater, bioassay, and microbiological analyses. Also responsible for supervision of staff, data review, and reporting.

Analyst III, *Columbia Analytical Services, Inc., Kelso, Washington,* 1989. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and TKN, in addition to miscellaneous wet chemistry analyses.

Microbiologist/Chemist, *Coffey Laboratories, Portland, Oregon,* 1983. Coliform analysis; water chemistry.

Laboratory Assistant, *Oregon State University, Corvallis, Oregon,* 1983. Wheat spike dissection and tissue culture.

Education

BS, Microbiology, *Oregon State University, Corvallis, Oregon,* 1983.

JEFFREY A. CORONADO

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER IV, INORGANICS DEPARTMENT MANAGER – 2001 to Present

Responsibilities

Primary responsibilities include management of the Metals laboratory department. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the Metals department. Also responsible for departmental budgets, method development efforts, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Metals Department Manager, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-2001. Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence..

Supervisor, GFAA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1989-1992. Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.

Education

Field Immunoassay Training Course, *EnSys Inc*., 1995.
Winter Conference on Plasma Spectrochemistry, *San Diego, California*, 1994.
ICP-MS Training Course, *VG-Elemental*, 1992.
BS, Chemistry, *Western Washington University, Bellingham, Washington,* 1988.
BA, Business Administration, *Western Washington University, Bellingham, Washington,* 1985.

JEFFREY A. GRINDSTAFF

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER III, GC/MS VOA AND SEMI-VOA LABORATORIES – 1997 to Present

Responsibilities

Primary responsibilities include management of the GC/MS SemiVoa and VOA laboratory departments. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the GC/MS departments. Also responsible for departmental budgets, method development efforts, and resource allocation. Also performs GC/MS maintenance and troubleshooting.

Documentation of Demonstration of Capabilities is available for review.

Experience

Manager, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1997. Responsible for supervision of GC/MS VOA staff, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems.

Scientist III, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1994. Responsibilities included scheduling workload, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and customer service.

Chemist, *Enseco-CRL, Ventura, California,* 1990-1991. Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses.

GC/MS Chemist, VOA Laboratory *Coast-to-Coast Analytical Service, San Luis Obispo, California,* 1990-1991. Responsible for standard preparation for VOA analyses and instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants..

Education

Mass Selective Detector Maintenance, *Hewlett-Packard Education Center,* 1993.
Interpretation of Mass Spectra I, *Hewlett-Packard Analytical Education Center,* 1992.
B.S., Chemistry, *California Polytechnic State University, San Luis Obispo, California,* 1989.
A.A., Liberal Arts, *Allan Hancock College, Santa Maria, California.* 1986

Publications/ Presentations

Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, Florida. 1996

Affiliations

American Chemical Society. 1989

TODD N. POYFAIR

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER III, SVG LABORATORIES – 2001 to Present

Responsibilities

Primary responsibilities include management of the GC, HPLC, and General Chemistry laboratory departments. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the these departments. Also responsible for departmental budgets, method development efforts, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Supervisor/Manager, General Chemistry Department, *Columbia Analytical Services, Inc., Kelso, Washington,* 1995-2001. Responsibilities included supervision, management, and training of General Chemistry staff. Also responsible for workload coordination, data review, reporting, and instrument maintenance within the General Chemistry department.

Project Chemist, Client Services Group, *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsibilities included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to acted as liaison for all client-related activities within CAS.

Scientist II, General Chemistry Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included the review and summarization of pH, alkalinity, conductivity, turbidity, hardness, and CODs.

Scientist I, **General Chemistry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992. Responsibilities included analysis of Total Organic Halogens, Chemical Oxygen Demand, Sulfides, Ammonia, TKN, Nitrate/Nitrite by Lachat, and Cyanide.

Analyst III, **General Chemistry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1992. Responsibilities included analysis of pH, Conductivity, Alkalinity, Turbidity, and Oil and Grease.

Education

BS, Chemistry, *Portland State University, Portland, Oregon,* 1991.
BA, German, *Portland State University, Portland, Oregon,* 1990.
COURSEWORK, *Brigham Young University, Provo, Utah.* 1982-1983 & 1985-1986.

JAMES R. "JIM" SMITH

2001 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER I, DRINKING WATER LABORATORY SUPERVISOR – 2002 to Present

Responsibilities

Primary responsibilities include management of the Semivolatiles Drinking Water laboratory department. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the department. Also responsible for departmental budgets, method development efforts, and resource allocation. Project management of Drinking water accounts. Operation of Varian GC/MS, Agilant GC/ECD and Agilant HPLC.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Manager III, *Columbia Analytical Services, Inc., Kelso, Washington,* 2001-2002. Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Extensive technical experience with the various GC and GC/MS method allow for detailed technical review of organics projects. Also has extensive experience coordinating drinking water and sediment projects. Currently responsible for coordination of approximately $200,000 of analyses in the laboratory on a monthly basis. Current large clients include Bechtel (Navy work), which involves numerous groundwater-monitoring projects, the Port of Seattle and URS. Also responsible for various storm water studies for Bremerton Naval Shipyard.

Director, Trace Organics and Project Manager, *Amtest, Inc., Redmond, Washington,* 1987-2001. Responsible for project management, client contact, data review, and writing reports. Additional responsibilities pertained to supervision of the trace organics department and running the GC/MS system. Performed various methods by GC/MS for volatiles, semi-volatiles and by GC for pesticides, PCBs, herbicides, and fuels (LUST). Also performed hazardous waste characterization including completion of waste profile forms.

Education

BS, Chemistry, *Rochester Institute of Technology, Rochester, New York,* 1985.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFTEY OFFICER – 1994 to Present

Responsibilities

Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Chemist, Client Services Group, Kelso Health and Safety Officer, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1994. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Scientist IV, Metals Laboratory, Health and Safety Officer, *Columbia Analytical Services, Inc., Kelso, Washington, 1987-1992.* Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Chemist, *Dow Corning Corporation, Springfield, Oregon,* 1986-1987. Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon.

Chemist, *Ametek, Inc., Harleysville, Pennsylvania,* 1982-1985. Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques.

Chemist, *Janbridge, Inc., Philadelphia, Pennsylvania,* 1978-1982. Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.

Education

BA, Chemistry, *Immaculata College, Immaculata, Pennsylvania,* 1977.

Affiliations

American Chemical Society, Member since 1987.

PAUL GOWAN

1994 TO PRESENT

CAS Holdings Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

IT MANAGER II, 2002 to Present

Responsibilities

Identify and plan IT requirements by interacting with management personnel to identify current and long-term user objectives. Assist in developing and tracking the IT computer capital budget. Provide electronic data deliverable (EDD) and LIMS system guidance to management personnel throughout the company. Ensure the effective utilization of computer systems. Train users as required. Establish IT policies, standards, practices, and specifications. Participate in the LIMS planning group.

Plan and supervise IT department staffing, organization, hardware and software acquisitions to meet requirements. Acquire, develop, and maintain a skilled staff through effective performance review and career development programs. Develop and maintain relationships with local and national vendors of computer hardware, software, and telephone equipment.

Experience

KELSO IT MANAGER, *Columbia Analytical Services, Inc., Kelso, Washington,* 1999-2002. Responsible for all IT efforts related to the CAS laboratory in Kelso, WA, including, but not excluded to computing equipment specification, purchasing and maintenance; network infrastructure; software development; strategic planning for future IT initiatives; budget preparation performance reviews, and career growth planning for IT staff.

TECHNICAL INFORMATION SPECIALIST, *CAS Holdings,Inc., Kelso, Washington,* 1994-1999. Primary responsibilities included support automation of HP chemstation and Enviroquant; support continued development of unified organic laboratories; support and development of project-specific data deliverables, technical assistance in Information Technology at Kelso to meet CAS IT objectives as related to LIMS.

Organics Section Manager, *Anametrix, Inc., San Jose, California,* 1992-1994. Responsibilities included managing the GC/MS and GC/Pesticide Departments, following protocols of the Department of Defense NEESA contracts (demanding stringent QA/QC and "Level D" data package submittals). Primary responsibilities included supervisor training and development, budget preparation and maintenance, performance reviews, data review, continuing research on environmental trends, SOP generation and updates, method development, capital equipment evaluation for laboratories, and project management.

GC/MS Program Manager, *Anametrix, Inc., San Jose, California,* 1988-1992. Responsibilities included supervision of five chemists, two Finnigan 4000 GC/MS Systems, and three HP 5971 GC/MS Systems. Primary responsibilities were to maintain high productivity and insure that the GC/MS department generated legally defensible data. Also responsible for sample scheduling and tracking, instrument maintenance and troubleshooting, analyst training and review, client interfacing, and purchasing.

GC/MS Analyst, *Anametrix, Inc., San Jose, California,* 1986-1988. Responsibilities included analyzing for VOA and Semi-VOA priority pollutants using EPA Methods 624/625 and 8240/8370.

Education

BA, Biochemistry, *San Jose State University, San Jose, California,* 1986.

GARY K. WARD

2001 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

VICE PRESIDENT, CHIEF QUALITY, SAFETY, AND ETHICS OFFICER – 2001 to Present

Responsibilities

Responsibilities include directing and managing the overall corporate-wide quality systems, ethics and safety programs for all CAS facilities, as well as strategic planning, marketing, business development, and information technology. Responsible for all interaction and liaison with government entities involving quality, technical and operational issues.

Experience

Deputy Director, Laboratory Standards, *Intertek Testing Services, Houston, Texas,* 1998-2001. Responsibilities included professional standards/quality assurance for 240 laboratories in 93 countries, involving laboratory tests ranging from petroleum products and environmental samples to toys, textiles, and building products. Resolution of issues with a variety of governments, agiences, and companies with particular focus on interactions with the US EPA. Was previously responsible for all operations of over 100 labs in the Americas, ranging from Canada to South America, including duties to improve quality, raise profits and revenues, and implement a LIMS.

Director, Technical Operations, *Environmental Health Laboratories, South Bend, Indiana,* 1995-1998. Responsibilities included operations and quality assurance of the laboratory. Directed, administered and coordinated activities of the lab in accordance with goals and objectives of the company. Responsible for the R&D program, laboratory throughput and financial performance, and implementation of the new LIMS system.

Executive Scientist, *Quanterra (Enseco), Arvada, Colorado,* 1987-1995. Responsibilities included providing expertise and experience in laboratory analysis and operations to the entire laboratory system. Duties included implementation of network-wide LIMS as well as coordination of the Technology, QA, IS, and Operations groups. As Director of Technology and Quality Assurance was responsible for management of the R&D program, Quality Assurance program, and Environment, Health and Safety program throughout the Enseco lab system. Direct reports were all QA managers, safety managers, and chief scientists from each of the 13 laboratories.

Deputy Branch Chief, *U.S. Environmental Protection Agency*, 1983-1987. Responsibilities included providing expertise to entire Superfund program ranging from lab analytical services to sampling. Duties involved managing the CLP program as well as the Superfund R&D program. As CLP National Program Manager was responsible for development and implementation of CLP analytical protocols, administration of contracts for over 100 laboratories throughout the country, and liasion with contract divisions, other EPA programs, and enforcement. Responsible for development and implementation of disk deliverables, automated contract screening, as well as writing new protocols for specific methods such as ICP/MS and for EPA methods such as included in SW846, 3rd Edition. Duties also included coordination of the annual CLP conferences.

Education

MS, Chemical Oceanography, *RSMAS, University of Miami, Miami, Florida,* 1973.
BS, Chemistry, *Loyola University, Los Angeles, California,* 1970.

Publications, Presentations. And Affiliations

Mr. Ward has a number of publications and presentations, and is affiliated with several professional organizations. For a list of these, please contact CAS.

STEPHEN W. VINCENT

1986 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

PRESIDENT, CAS HOLDINGS INC. – 1986 to Present

Responsibilities

Responsible for the overall growth and profitability of the CAS laboratory network. This includes establishing and implementing long-range objectives, plans, and policies, and representing the company with its major customers, technical community, and the public.

Experience

Laboratory Manager, *Weyerhaeuser Company, Federal Way, Washington,* 1979-1986. Responsibilities involved all phases of technical and administrative management. This included management of organic, inorganic, and microbiological analyses and management of capital; an annual operating budget of approximately $2 million; management of thirty staff members; contract procurement, and project management. Projects included an EPA Inorganic CLP contract; an EPA acid rain deposition contract; a contract with the Fish and Wildlife Service to measure trace organic contaminants in animal tissues; and others.

Analytical Chemist, *Weyerhaeuser Company, Longview, Washington,* 1975-1979. Responsibilities: Method development, routine analysis and supervision for the Weyerhaeuser Multi-Region Support Lab. Responsible for setting up a company-wide laboratory audit, round robin, and quality assurance program.

Education

Market Strategy for Technology Based Companies, *Executives Program, Stanford University*. 1994.
Advanced Technical Management Program, *University of California at Los Angeles, Department of Business, Engineering and Management,* 1991.

Completion of Coursework for MS, Pulp and Paper Technology, *University of Washington, Seattle, Washington,* 1984.
Post Graduate Coursework, Engineering and Management, *University of California at Los Angeles, Graduate School of Engineering and Applied Science, Los Angeles, California,* 1981.
BS, Oceanography, *University of Washington, Seattle, Washington,* 1974.

Publications/ Presentations

Mr. Vincent has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.

Affiliations

American Chemical Society.
Technical Association of the Pulp and Paper Industry.

APPENDIX C

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (9):			
Precisa and Mettler models	1988-2000	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
Calorimeters (2):			
Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	15
Colony Counter - Quebec Darkfield	1988	LM	4
Conductivity Meters (2):			
YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5):			
COD (4)	1987, 1989	LM	3
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	5
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	7
Drying Ovens (11):			
Shel-Lab and VWR models	1988 - 2003	LM	15
Flash Point Testers (2):			
ERDCO Setaflash Tester	1991	LM	4
Petroleum Systems Services	2005	LM	4
Flow-Injection Analyzers (2):			
Lachat Quik-Chem AE	1990	LM	5
Bran-Leubbe	2002	LM	4
Ion Chromatographs (3)			
Dionex 2000i with Peaknet Data Systems	1988	LM	3
Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Ion Selective Electrode Meters (5)			
Fisher Scientific Accument Model 50	1997	LM	6
Fisher Scientific Accument Model 25	1993	LM	6
Fisher Scientific Accument Model 20	2000	LM	6
Orion Model 920A	1990	LM	6
Corning pH/ion Meter Model 135	1992	LM	6
Microscopes (2):			
Bausch & Lomb	1988	LM	1
Swift	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	15
pH Meters (2):			
Fisher Scientific Accument Model 20	1993	LM	6
Fisher Scientific Accument Model AR25	2005	LM	6

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Shatter Box - GP 1000	1989	LM	5
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	7
Total Organic Carbon (TOC) Analyzers (2)			
Coulemetrics Model 5012	1997	LM	3
O-I Corporation Model 1010	2002	LM	3
Total Organic Halogen (TOX) Analyzers (3):			
Mitsubishi TOX-Sigma	1995	LM	4
Mitsubishi TOX-100 (2)	2001	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	8
UV-Visible Spectrophotometers (2):			
Hitachi 100-40 Single Beam	1986	LM	5
Beckman-Coulter DU520	2005	LM	5
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991	LM	13
Water Baths/Incubators (6):			
Hach Model 15320 Incubator	1986	LM	15
Precision Model L-6 (2)	1989, 1990	LM	15
VWR 1540	1991	LM	15
Fisher 11-680-626M Incubator	1992	LM	15
Fisher Isotemp Incubator	2001	LM	15

METALS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (6)			
Various Mettler AE 200 analytical balance	1990	MM	12
Various Mettler models (5)	1988	MM	12
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA Zeeman/220 AA w/Data Systems (2)	2000	LM	3
CETAC Mercury Analyzer	2000	LM	2
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	2
Atomic Fluorescence Spectrophotometer			
Brooks-Rand Model III (2)	1996, 2005	LM	3
Leeman Mercury Analyzer (1)	2006	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (2) - Labconco	1992, 2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) - Thermo Jarrell Ash Model 61E	1988	LM	4
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES): Thermo Jarrell Ash, Model IRIS	2000	MM	4
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG PQ-S	1997	MM	3
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	3
Muffle Furnace - Thermolyne Furnatrol Model 53600 (2)	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5

SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
Analytical Balance (3)			
Mettler BB240	1987	MM	12
Satorious B610 (2)	1999, 2000	MM	12
Aspirator pumps - Labconco Cole Parmer (1)	1994	LM	7
Centrifuges (2):			
Adams Model DYNAC	1986	LM	12
Sorvall Model GLC-1	1988	LM	12
Drying Oven - Fisher Model 655 G	1991	LM	7
Evaporators (10):			
Organomation N-Evap (5)	1989-90,1998-2001	LM	12
Organomation S-Evap (5)	1989-1991	LM	12
Extractors: Lab-Line Multi-Unit Extraction Heaters (60)	1987-1992	LM	12
Extractors (64):			
Continuous Liquid/Liquid Extractors (24)	1991	LM	10
Branson Model 450 Sonifier (2)	1991	LM	4
Tekmar Sonifier (2)	1994	LM	4
Soxhtherm (36)	2000	LM	5
Gel Permeation Chromatography (GPC) (4)			
ABC single column (2)	1998, 1999	LM	4
ABC Autoprep 1000	1995	LM	4
J2 Scientific	2005	LM	4
Muffle Furnace - Parflow MIC 6000	1994	LM	12
Solid Phase Extractors (8) – Dionex SPE-Dex 4790	2003, 2006	LM	6
Vacuum Pump - Edwards	1992	LM	8

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler AT 250	1989	MM	5
Chromatography Data Systems (12)			
HP Enviroquant (8)	1994-2002	LM	5
Thruput Target (4)	1998-2000	LM	5
Gas Chromatographs (13):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (7)	1990 – 1995	LM	5
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	5
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (4)	2001, 2005	LM	5
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	MM	5

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	6
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	2
Semivolatile GC/MS Systems (8):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	MM	4
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1990,1994	MM	4
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	MM	4
Agilent 6890/5973 with ATAS Optic3 LVI and 7683 Autosampler (1)	2004	MM	4

PETROLEUM HYDROCARBONS GC/HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
SAMPLE PREPARATION			
Analytical Balance (3)			
Mettler BB300	1991	MM	6
Mettler BB240	1994	MM	6
Mettler AE166	1994	MM	6
Aspirator pumps – GAST (2)	2002, 2004	LM	6
Drying Oven - Fisher Model 630F	1991	LM	6
Evaporators (2):			
Organomation N-Evap	1990	LM	6
Organomation S-Evap	1991	LM	6
Extractors (4):			
Sonic Horns (2): Branson, Ultrasonics, Fisher Models	1991-1994	LM	6
INSTRUMENTATION			
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	6
Gas Chromatographs (5):			
Hewlett-Packard 5890 Series II with PID/PID/FID:	1991	LM	3
Tekmar LSC-2000 Purge and Trap Concentrator	1991	LM	3
Dynatech Archon 5100 Autosampler	1992	LM	3
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FID Detectors (2)	1990 - 1995	LM	3
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (2)	2001, 2005	LM	3
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	3
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	3
High-Performance Liquid Chromatograph/Mass Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS with Thermo Surveyor HPLC and Autosampler	2005	MM	2

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance			
Mettler PE 160	1989	MM	6
Baxter Vortex Mixer	1989	LM	6
Extractors (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	2
TCLP Extractor - Tumbler (12 position)	1989	LM	2
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	6
Drying Ovens (2):			
Narco 420	1989	LM	6
VWR 1305 U	1991	LM	6
Sonic Water Bath - Branson Model 2200	1989	LM	6
Volatile GC/MS Systems (8):			
Agilent 5890/5970 (2)	1989	MM	4
Tekmar 3000 Purge and Trap Concentrator	1995	LM	4
Dynatech ARCHON 5100 Autosampler	1996	LM	4
Agilent 5890/5970	1999	MM	4
EST Encon Purge and Trap Concentrator	2002	LM	4
Dynatech ARCHON 5100 Autosampler	1999	LM	4
Agilent 5890/5971	1991	MM	4
Tekmar 3000 Purge and Trap Concentrator	2001	LM	4
Dynatech ARCHON 5100 Autosampler	1995	LM	4
Agilent 5890/5972A	1993	MM	4
Tekmar 3000 Purge and Trap Concentrator	1995	LM	4
Dynatech ARCHON 5100 Autosampler	1996	LM	4
Agilent 6890/5973 (2)	2001, 2007	MM, LM	4
Tekmar 3100 Purge and Trap Concentrator	2001, 2007	LM	4
Varian Archon Autosampler	2001, 2007	LM	4
Agilent 6890/5973	2005	MM	4
Tekmar Velocity Purge and Trap Concentrator	2005	LM	4
Tekmar Aquatech Autosampler	2005	LM	4

DRINKING WATER ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB300	1991	MM	2
Extractors (5) – Horizon SPE-DEX Solid Phase Extractor	2003	LM	2
Aglinet Enviroquant Chromatography Data Systems (2)	2003	LM	2
Varian Saturn Chromatography Data System	2003	LM	2
Evaporator - Organomation N-Evap	2003	LM	2
Agilent 1100 HPLC w/post-column derivitization: UV/Fluoescence detectors Pickering PCX-5200 Post-column derivitization unit	2003 2003 2003	LM LM LM	2 2 2
Agilent 6890N GC/ECD system: Dual micro-ECD detectors Agilent autosampler	2003 2003 2003	LM LM LM	2 2 2
Varian Ion trap GC/MS: Varian 3800 GC w/CP8400 autosampler Varian 3900 GC Varian Saturn 2100T mass spectrometer	2003 2006 2003 2003	LM LM LM LM	2 2 2 2

PHARMACEUTICAL TESTING LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Thermo High-Performance Liquid Chromatograph with Diode Array UV Detector	2005	MM	2
Thermo FTIR	2005	MM	4
Viscometer	2006	LM	1
Analytical Balance - Mettler AB104-5 and AT-250	2004	MM	4
Incubator – VWR 1510E	2004	LM	2
Karl Fisher Titrators (2) – Mettler DL38 and DL39	2004	MM	3
Melting point apparatus – Optimelt	2004	LM	3
Refractometer – Reichert Abbe Mark II	2004	LM	2
Rotary evaporator – Labonco/Cole-Parmer aspirator pump	2004	LM	3
Rotary shakers – Thermolyne Rotomix (2)	2004	LM	2
Vacuum oven – Precision Model 19 w/GAST aspirator	2004	LM	3
Water baths (3) – VWR Models 1212, 1235 and Labline 18002	2004	LM	2
Gas Chromatographs (2): Hewlett-Packard 5890 Series II with TCD/FID detector Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FID Detectors	 1988 1990	 LM LM	 3 3

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMS Sample Manager using Oracle 10g DBMS running on Redhat Advanced Server 3.0 (Linux) platform connected/linked on a frame relay WAN environment	1994-2004	LM	NA
2 - Network Servers Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 Advanced Server, 1 for Applications running Windows 2003 Advanced Server. Data acquisition capacity at 65GB with redundant tape and disk arrays.	1994 - 2004	LM	NA
Approximately 50+ HP and Dell Laserjet printers (various types including IIIs, 4s, 5s, 8150s, 4000s, 4050s, 4250, 8150s, W5300s)	1991 - 2004	LM	NA
Approximately 130 Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2004	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97.	1996 - 2004	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Outlook Web Access interface. Microsoft Outlook 2003.	1994 - 2006	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Facsimile Machines 9600 - 33600 Baud, (2) Brother 4750e, 15 ppm; (1) Brother MFC 1970MC, 2ppm; (1) Canon CFX-L4000	1991 - 2004	LM	NA
Copiers/Scanners (2) Konica 7085, 85ppm, 20GB; (2) Konica 7155; (1) Konica 7035, BizHub. The 7085s and one 7155 are accessible via LAN for network scanning.	2000 - 2004	LM	NA
Dot Matrix Epson FX-880, LQ-1050, LX-300	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, StarLIMS reporting software systems.	1998 - 2004	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures Clean coils Check coolant	Daily Annually Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured Sash operation Change filters Inspect fan belts	Quarterly As needed Annually Annually
Ovens	Clean Record temperatures	As needed or if temperature outside lim. Daily, when in use
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures Wash with disinfectant solution	Daily, morning and evening When water is murky, dirty, or growth appears
Autoclave	Check sterility Check temperature Clean	Every month Every month When mold or growth appears
Analytical Balances	Check alignment Check calibration Clean pans and compartment	Before every use Daily After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride ISE	Store in storage solution	Between uses
Ammonia ISE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero Check digestion/condensation vessels Clean digestion chamber Clean permeation tube Clean six-port valves Clean sample pump Clean carbon scrubber Clean IR cell	Weekly Each use Every 2000 hours, or as needed Every 2000 hours, or as needed Every 200 - 2000 hours, or as needed Every 200 - 2000 hours, or as needed Every 200 - 2000 hours, or as needed Every 2000 - 4000 hours, or as needed

Instrument	Activity	Frequency
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed
Flow Injection Analyzer	Check valve flares	Each use
	Check valve ports	Each use
	Check pump tubing	Each use
	Check light counts	Each use
	Check flow cell flares	Quarterly
	Change bulb	As needed
	Check manifold tubing	Each use
	Check T's and connectors	Each use
Ion Chromatographs	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro-photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency
ICP - MS	Check argon dewar	Daily
	Check water level in chiller	Daily
	Complete instrument log	Daily
	Replace peristaltic pump tubing	Daily
	Clean sample and skimmer cones	As needed
	Clean RF contact strip	As needed
	Inspect nebulizer, spray chamber, and torch	Clean as needed
	Clean lens stack/extraction lens	As needed
	Check rotary pump oil	Monthly
	Change rotary pump oil	Every six months
Gel-Permeation Chromatographs	Clean and repack column	As needed
	Backflush valves	As needed
High Pressure Liquid Chromatographs	Backflush guard column	As needed
	Backflush column	As needed
	Change guard column	As needed when back pressure too high
	Change column	Annually or as needed
	Change in-line filters	As needed
	Leak check	After column maintenance
	Change pump seals	As needed
	Change pump diaphragm	Annually
	Clean flow cell	As needed
	Fluorescence detector check	Daily
	Diode array absorbance check	Daily
Gas Chromatographs, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean FID	Weekly or as needed
	Clean ECD	Quarterly or as needed
	Leak test ECD	Annually

Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Annually or as needed
	Change septum	Daily, when in use
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications
Purge and Trap Concentrators	Change trap	Every four months or as needed
	Change transfer lines	Every six months or as needed
	Clean purge vessel	Daily
Gas Chromatographs, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean PID lamp	As needed
	Clean FID	As needed
	Change ion exchange resin	Every 60 days
	Replace nickel tubing	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Annually or as needed
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications

APPENDIX E

SOP LIST AND LIST OF NELAC ACCREDITED METHODS

COLUMBIA ANALYTICAL SERVICES, INC. , KELSO, WA.
STANDARD OPERATING PROCEDURES TABLE OF CONTENTS
January 11, 2006

SOP NAME	FILE NAME	REV #
ARMY CORPS OF ENGINEERS HTRW PROJECT MANAGEMENT	ADM-HTRW	1
CHECKING PIPETTE CALIBRATION	ADM-CPIP	4
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	ADM-ECP	0
CONTROL CHARTING QUALITY CONTROL DATA	ADM-CHRT	1
DATA ARCHIVING	ADM-ARCH	3
DATA REPORTING AND REPORT GENERATION	ADM-RG	5
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	ADM-DOD	0
ELECTRONIC DATA BACKUP AND ARCHIVING	ADM-EBACKUP	0
FACILITY AND LABORATORY CLEANING	ADM-FACL	0
INTERNAL QUALITY ASSURANCE AUDITS	ADM-IAUD	5
LABORATORY DATA REVIEW PROCESS	ADM-DREV	4
METHOD DETECTION LIMIT DOCUMENTATION AND CONTROL	ADM-MDLC	2
PROJECT MANAGEMENT	ADM-PCM	6
REAGENT LOGIN AND TRACKING	ADM-RLT	2
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC	8
COLIFORM, FECAL	BIO-9221FC	5
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D	1
COLIFORM, TOTAL	BIO-9221TC	4
COLIFORM, TOTAL (DRINKING WATER)	BIO-9221DW	3
COLILERT COMPLETED TEST VERIFICATION OF E. COLI IN MUG CULTURES	BIO-CCT	0
COLILERT P/A	BIO-COLI	2
ENTEROLERT	BIO-ENT	0
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B	5
HEPTEROTROPHIC PLATE COUNT	BIO-HPC	3
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC	10
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN	0

EPA CLP ORGANICS ANALYSES	CLP_ORGA	1
ADDITION OF SPIKES AND SURROGATES	EXT-SAS	5
AUTOMATED SOXHLET EXTRACTION	EXT-3541	3
CONTINUOUS LIQUID - LIQUID EXTRACTION	EXT-3520	10
CONTINUOUS LIQUID - LIQUID EXTRACTION FROM AQUEOUS SOURCE SAMPLING IMPINGERS	EXT-3520IMP	0
DIAZOMETHANE PREPARATION	EXT-DIAZ	4
FLORISIL CLEANUP	EXT-FLOR	1
ORGANIC EXTRACTIONS GLASSWARE CLEANING	EXT-GC	2
MASS BALANCE DETERMINATION OF TETRACHLOROETHYLENE AND ACETONE IN POLLEN PRODUCTION	EXT-MASSBAL	0
MEASURING SAMPLE WEIGHTS AND VOLUMES FOR ORGANIC ANALYSIS	EXT-WVOL	2
PREPARATION OF ANHYDROUS SODIUM SULFATE , MARTIX SAND, SODIUM CHLORIDE, AND POTASSIUM CARBONATE	EXT-SULF	2
PRESSURIZED FLUID EXTRACTION	EXT-3545	5
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510	7
SOLID PHASE DISPERSION IN TISSUES	EXT-SPD	0
SOLID PHASE EXTRACTION	EXT-3535	2
SOXHLET EXTRACTION	EXT-3540	8
ULTRASONIC EXTRACTION	EXT-3550	9
WASTE DILUTION EXTRACTION	EXT-3580	1
ACIDITY	GEN-305.2	1
ALKALINITY TOTAL	GEN-310.1	5
AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE	GEN-350.3	5
AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1	6
AUTOFLUFF	GEN-AUTOFLU	0
BIOCHEMICAL OXYGEN DEMAND	GEN-405.1	8
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109	0
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU	1
CARBON, TOTAL ORGANIC IN SOIL	GEN-ASTM	5
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M	0
CHEMICAL OXYGEN DEMAND	GEN-COD	5

CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)	GEN-325.3	3
CHLORINE, TOTAL/FREE RESIDUAL	GEN-330-4	1
COLOR	GEN-110.2	2
COLOR, NCASI	GEN-NCAS	1
CONDUCTIVITY IN WATER AND WASTES	GEN-COND	8
CORROSIVITY TOWARDS STEEL	GEN-CORR	1
CYANIDE EXTRACTION OF SOLIDS AND OILS	GEN-9013	0
CYANIDE, WEAK ACID DISSOCIABLE	GEN-CNWAD	0
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300_1	2
DISSOLVED SILICA	GEN-370.1	0
FERROUS IRON IN WATER	GEN-FeII	2
FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020	5
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE	5
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM	1
GLASSWASHING FOR INORGANIC ANALYSES	GEN-WASH	3
GRAVIMETRIC SULFATE	GEN-375.3	1
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650	2
HALIDES, ADSORBABLE ORGANIC (AOX) - SM 5320B	GEN-5320B	1
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M	2
HALIDES, TOTAL ORGANIC (TOX)	GEN-9020	7
HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION	GEN-5050	2
HARDNESS, TOTAL	GEN-130.2	4
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HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD	1
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26	2
ION CHROMATOGRAPHY	GEN-IONC	9
MBAS	GEN-425.1	2
MERCURY IN COAL SAMPLE PREPARATION BY PARR BOMB COMBUSTION	GEN-HGPREP	0
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NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN	8
ORTHOPHOSPHORUS BY FLOW INJECTION ANALYSIS - SM 4500P-F	GEN-4500P-F	0
OXYGEN CONSUMPTION RATE	GEN-O2RATE	0
PARTICLE SIZE DETERMINATION	GEN-PSP	4
PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM	0
PERCHLORATE BY ION CHROMATOGRAPHY	GEN-314-0	10
Ph IN SOIL AND SOLIDS	GEN-Phs	7
Ph IN WATER	GEN-Phw	7
PHENOLICS, TOTAL	GEN-420.1	10
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3	8
POST DIGESTION DETERMINATION OF TOTAL KJELDAHL NITROGEN BY SEMIAUTOMATED COLORIMETRY	GEN-TKNAA	0
SETTEABLE SOLIDS	GEN-160.5	2
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-160.1	6
SOLIDS, TOTAL SUSPENDED (TSS)	GEN-160.2	6
SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES	GEN-160.4	4
SPECIFIC GRAVITY	GEN-SPGRAV	0
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS	1
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2	1
SULFIDE, TITRIMETRIC (IODINE)	GEN-376-1	0
SULFIDES, ACIDS VOLATILE	GEN-AVS	5
SULFIDE, METHYLENE BLUE	GEN-376-2	0
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SULFITE	GEN-SO3	1
TANNIN AND LIGNIN	GEN-5550	4
THIOCYANATE	GEN-THIOCN	0
TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION	GEN-335	11
TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY	GEN-9076	1
TOTAL ORGANIC CARBON IN WATER	GEN-TOC	7
TOTAL SOLIDS	GEN-160.3	10
TOTAL SULFIDE BY PSEP	GEN-S2PS	0
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030M	8

TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS	1
TURBIDITY MEASUREMENT	GEN-TURB	3
ULTIMATE BOD	GEN-UBOD	0
CATION-EXCHANGE CAPACITYOF SOILS (SODIUM ACETATE) - METHOD 9081	MET-9081	1
CLOSED VESSEL OIL DIGESTION	MET-3051M	0
DETERMINATION OF LEAD BY FLAME ATOMIC ABSORPTION (FAA)	MET-7420	1
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020	8
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICP.MS	10
DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES	MET-ICP	18
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FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRIC ANALYSES	MET-FAA	1
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MERCURY IN LIQUID WASTE	MET-7470A	9
MERCURY IN SOLID OR SEMISOLID WASTE	MET-7471A	10
MERCURY IN WATER	MET-245.1	10
MERCURY IN WATER BY OXIDATION, PURGE&TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY	MET-1631	7
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METALS DIGESTION	MET-3005A	3
METALS DIGESTION	MET-3010A	8
METALS DIGESTION	MET-3020A	10
METALS DIGESTION	MET-3050	8
METALS DIGESTION	MET-7195	5
METALS DIGESTION - CLP	MET-DIG	8
METALS LABORATORY GLASSWARE CLEANING	MET-GC	2
METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630S	0
METHYL MERCURY IN TISSUE BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630T	0
METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630W	0
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SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT	0
SAMPLE PREPARATION OF AQUEOUS SAMPLES BY "CLEAN" TECHNIQUES	MET-ACT	3

SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS	MET-TDIG	1
SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7742	2
TISSUE SAMPLE PREPARATION	MET-TISP	4
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS	4
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC	0
ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS	PHC-WIDRO	0
ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS	PET-SVF	10
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	PET-GRO	7
GRAVIMETRIC DETERMINATION OF HEAXANE EXTRACTABLE MATERIAL (1664)	PET-1664	5
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD	7
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT	7
SAMPLE DISPOSAL	SMO-SDIS	6
SAMPLE RECEIVING	SMO-GEN	22
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC	10
ALDEHYDES BY HPLC	SOC-8315A	4
BUTYLTINS	SOC-BUTYL	7
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL	5
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CHLORINATED PHENOLICS IN WASTE WATER BY IN-SITU	SOC-1653A	5
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M	7
CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES	SOC-CONF	4
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GG/ECD	SOC-8082C	7
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141	9
DIMP	SOC-DIMP	5
DMD SYNTHESIS	SOC-DMD	1
EXTRACTION METHOD FOR ORGANOTINS IN SEDIMENTS, WATER, AND TISSUE	SOC-OSWT	5
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GLYCOLS	SOC-8015M	7
HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES	SOC-9902	1
HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901	2
METHANOL IN PROCESS LIQUIDS	SOC-9403	3
MONOCHLOROACETIC ACID BY GC-ECD	SOC-MCA	4
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8330	10
NITROGLYCERIN AND PETN BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8332	5
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-NITG	1
N-NITROSODIMETHYLAMINE BY GC/MS	SOC-NDMA	1
N-NITROSAMINES BY GC/MS/MS	SOC-521	0
N-NITROSODIMETHYLAMINE BY GC/MS/MS	SOC-521M	0
ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC	SOC-OALC	1
ORGANOCHLORINE PESTICIDES AND PCBs (METHOD 608)	SOC-608	4
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PICRIC ACID AND PICRAMIC ACID BY HPLC	SOC-PICRIC	2
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SOC-8270P	5
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	SOC-8310	11
REMOVAL OF SULFUR USING COPPER	SOC-3660	3
RESIN AND FATTY ACIDS BY GC/MS	SOC-85.01	3
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-625	4
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C	8
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE	SOC-8270L	3
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SOC-8270S	4
SEMI-VOLATILE ORGANICS SCREENING	SOC-SCR	2
SILICA GEL CLEANUP	SOC-3630C	0
SULFURIC ACID CLEANUP	SOC-3665	1
TENTATIVELY IDENTIFIED COMPOUNDS BY GCMS ON EXTRACTS FROM AQUEOUS SOURCE SAMPLING IMPINGERS	SOC-IMPTIC	0

1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC	SVD-504	4
CARBAMATES AND CARBAMOYLOXIMES IN WATER BY POST-COLUMN DERIVITIZATION HPLC	SVD-531 -1	4
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515_4	4
DIQUAT AND PARAQUAT BY HPLC	SVD-549	3
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ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1	3
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AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 602	VOC-602BTEX	2
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 8021	VOC-8021BTEX	4
PURGE AND TRAP FOR AQUEOUS SAMPLES	VOC-5030	3
PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM	VOC-5035	5
SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES	VOC-BVOC	3
TENTATIVELY IDENTIFIED COMPOUND ANALYSIS BY DIRECT INJECTION GCMS ON EXTRACTS FROM AQUEOUS SOURCE SAMPLING IMPINGERS	VOC-IMPTIC	0
VOA STORAGE BLANKS	VOC-BLAN	4
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-524.2	10
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-624	8
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-8260B	10
WA-DOH DRINKING WATER PROTOCOL	VOC-WA.DOH	2
ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)	VOC-ZHE	5
SOP NAME - PHARMACEUTICAL TESTING SOPS	**FILE NAME**	**REV #**
PHARMACEUTICAL TEST METHOD SOPs		
DETERMINATION OF ORGANIC VOLATILE IMPURITIES (OVI's) IN PHARMACEUTICAL EXCIPIENTS BY GC	PHC-POVI	0
FDA EXTRACTABLES	PHC-FDAEX	0
GC-FID IMPURITIES IN METHYLSULFONYLMETHANE (MSM)	PHM-MSM	0
AMINO ACID ASSAY IN PHARMACUETICAL MANUFACTURING MATERIALS USING HPLC	SOC-AALC	0
TIN AS STANNOUS FLUORIDE IN CHOLETEC BY ICP-AES	PHM-SnCholetec	0
PHARMACEUTICAL EQUIPMENT SOPs		

OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO AB104-S ANALYTICAL BALANCE	PHM-IN01	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE VWR VACUUM OVEN	PHM-IN02	2
OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO DL38 TITRATOR	PHM-IN03	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE HEWLETT PACKARD 5890 GAS CHROMATOGRAPH	PHM-IN04	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE HEWLETT PACKARD 1050 SERIES HPLC	PHM-IN05	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO DL39 COULOMETRIC TITRATOR	PHM-IN06	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE SRS MPA100 AUTOMATED MELTING POINT SYSTEM	PHM-IN07	2
OPERATION, CALIBRATION, AND MAINTENANCE OF THE SUN INSTRUMENTS TYPE WXG-D DISK POLARIMETER	PHM-IN08	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE REICHERT ABBE MARK II MODEL 10480 REFRACTOMETER	PHM-IN09	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE LAB-LINE MODEL 18002 UTILITY HEATED WATER BATH	PHM-IN10	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE Brookfield DV-III Ultra Programmable Rheometer	PHM-IN11	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE THERMOE ELECTRON NICOLET 4700 FTIR	PHM-IN12	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO AT250 ANALYTICAL BALANCE	PHM-IN13	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The PERKIN ELMER AANALYST 200 FLAME ATOMIC ABSORPTION SPECTROMETER (Flame Only)	PHM-IN14	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE VARIAN SPECTRA AA 220ZGFAA SPECTROMETER	PHM-IN15	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE THERMO IRIS ADVANTAGE ICP-AES	PHM-IN17	1
OPERATION, CALIBRATION, AND MAINTENANCE OF The PERKIN ELMER AANALYST 200 FLAME AA AND FIAS 100 FOR CVAA MERCURY	PHM-IN18	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The VWR 1540 INCUBATOR	PHM-IN19	1
OPERATION, CALIBRATION, AND MAINTENANCE of the BECKMAN COULTER DU500 UV/Vis SPECTROPHOMETER	PHM-IN20	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The FISHER ISOTEMP INCUBATOR	PHM-IN21	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The NAPCO 6300 INCUBATOR	PHM-IN23	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The VWR 2020 INCUBATOR	PHM-IN24	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The HANNA AUTOTITRATOR	PHM-IN25	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO SEVENMULTI ELECTROCHEMICAL ANALYTICAL METER	PHM-IN26	0
PHARMACEUTICAL QUALITY ASSURANCE SOPs		
CONTROLLED SUBSTANCES	PHM-CSUB	1
VALIDATION OF PHARMACEUTICAL AND NUTRACEUTICAL TESTING METHODS	PHM-QA01	0
INSTRUMENT QUALIFICATION AND VERIFICATION	PHM-QA02	3
PHARMACEUTICAL TESTING DOCUMENTATION AND RECORDKEEPING	PHM-QA03	3
DETERMINATION OF TEST SPECIFICATIONS	PHM-QA04	0
EVALUATING AND SELECTING TEST METHODS FOR PHARMACEUTICAL AND NUTRACEUTICAL TESTING	PHM-QA05	2

OUT OF SPECIFICATION (OOS) INVESTIGATION AND RETESTING	PHM-QA06	2
CHANGE CONTROL	PHM-QA07	2
DATA MANAGEMENT OF PHARMACEUTICAL TESTING RECORDS	PHM-QA08	0
PHARMACEUTICAL PERSONNEL TRAINING	PHM-QA09	0
VALIDATION OF SOFTWARE USED FOR PHARMACEUTICAL TESTING	PHM-QA10	0
DATA SYSTEMS ACCESS AND AUDIT TRAILS	PHM-QA11	0
QUALITY CONTROL OF PHARMCEUTICAL TESTING DATA	PHM-QA12	0
SOP NAME - CORPORATE ADMINISTRATIVE SOPS MAINTAINED BY CORPORATE QA	**FILE NAME**	**REV #**
CHAIN OF CUSTODY FOR SAMPLE TRANSFER BETWEEN LABORATORIES	ADM-COC	1
CHECKING NEW LOTS OF CHEMICALS FOR CONTAMINATION	ADM-CTMN	2
CONTROL LIMITS	ADM-CTRL_LIM	4
DEALING WITH COMPLAINTS	ADM-CMPLT	3
DETERMINATION OF METHOD DETECTION LIMITS AND LIMITS OF DETECTION	ADM-MDL	6
DOCUMENT CONTROL	ADM-DOCCTRL	5
DOCUMENTATION OF TRAINING	ADM-TRANDOC	9
ELECTRONIC DATA AUDITING	ADM-E_DATAAUDIT	1
ESTIMATION OF UNCERTAINTY OF MEASUREMENTS	ADM-UNCERT	2
MAKING ENTRIES INTO LOGBOOKS AND ONTO BENCHSHEETS	ADM-DATANTRY	6
MANAGERIAL REVIEW OF THE LABORATORY"S QUALITY SYSTEM	ADM-MGMTRVW	1
MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS	ADM-INT	2
NONCONFORMITY AND CORRECTIVE ACTION	ADM-NCAR	4
PREPARATION OF ELECTRONIC DATA FOR ORGANIC ANALYSES ELECTRONIC DATA AUDITS	ADM-EDATA	2
PREPARATION OF STANDARD OPERATING PROCEDURES	ADM-SOP	6
PROFICIENCY TESTING SAMPLE ANALYSIS	ADM-PTS	0
PURCHASING THROUGH CAS PURCHASING AGENT	ADM-PUR	1
QUALIFICATION OF SUBCONTRACT LABORATORIES OUTSIDE OF CAS NETWORK	ADM-SUBLAB	2
SAMPLE BATCHES	ADM-BATCH	7
SIGNIFICANT FIGURES	ADM-SIG.FIG	6

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,3-Trichloropropane	EPA 504.1	Group II Unregulated Contaminants	NELAP	7/17/2003
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	7/17/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	7/17/2003
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
2,4,5-T	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
2,4-D	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
3-Hydroxycarbofuran	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDD	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDD	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDE	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDE	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDT	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDT	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/17/2003
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acetochlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Acifluorfen	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Alachlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Aldicarb (Temik)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldicarb sulfone	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldicarb sulfoxide	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldrin	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldrin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Alkalinity as CaCO3	SM 2320 B	Primary Inorganic Contaminants	NELAP	10/8/2001
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Aluminum	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Antimony	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Arsenic	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Atrazine	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Benzo(a)pyrene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Bromate	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Bromide	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Bromoacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Bromochloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/17/2003
Bromodichloromethane	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Bromoform	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Butachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Butyl benzyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001

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Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Carbaryl (Sevin)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Carbofuran (Furaden)	EPA 531.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Chlorate	EPA 300.1	Secondary Inorganic Contaminants	NELAP	12/23/2005
Chlordane (tech.)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	10/8/2001
Chlorite	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Chloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Chloroform	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	7/17/2003
Conductivity	SM 2510 B	Primary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.7	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.8	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Cyanide	EPA 335.4	Primary Inorganic Contaminants	NELAP	10/8/2001
Dacthal (DCPA)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dalapon	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
DCPA di acid degradate	EPA 515.4	Group I Unregulated Contaminants	NELAP	7/17/2003
DCPA mono acid degradate	EPA 515.4	Group I Unregulated Contaminants	NELAP	7/17/2003
Di(2-ethylhexyl)adipate	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dibromoacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Dibromochloromethane	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

Laboratory Scope of Accreditation

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Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dicamba	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Dichloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Dieldrin	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Dieldrin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Diethyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Dimethyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Di-n-butyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Di-n-octyl phthalate	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Diquat	EPA 549.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Endothall	EPA 548.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Endrin	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Endrin	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
EPTC (Eptam, s-ethyl-dipropyl thio carbamate)	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Fluoride	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Fluoride	SM 4500 F-C	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Glyphosate	EPA 547	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor epoxide	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor epoxide	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Heterotrophic plate count	SM 9215 B	Microbiology	NELAP	7/17/2003
Hexachlorobenzene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Isophorone	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

Laboratory Scope of Accreditation

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State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Lead	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	10/8/2001
Methomyl (Lannate)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Methoxychlor	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Methoxychlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Metolachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Metribuzin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Molinate	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrate	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrate	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Orthophosphate as P	SM 4500-P F	Primary Inorganic Contaminants	NELAP	10/8/2001
Oxamyl	EPA 531.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Paraquat	EPA 549.2	Synthetic Organic Contaminants	NELAP	12/23/2005
PCBs	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Pentachlorophenol	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Pentachlorophenol	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Perchlorate	EPA 314.0	Primary Inorganic Contaminants	NELAP	7/17/2003
pH	EPA 150.1	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
Picloram	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Propachlor (Ramrod)	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
sec-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

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Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Selenium	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Silica as SiO2	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Silver	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Silver	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Silvex (2,4,5-TP)	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Simazine	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Sulfate	EPA 300.0	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
Terbacil	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	11/18/2004
Thallium	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Total coliforms & E. coli	SM 9223 B	Microbiology	NELAP	10/8/2001
Total dissolved solids	SM 2540 C	Secondary Inorganic Contaminants	NELAP	10/8/2001
Total haloacetic acids	EPA 552.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Total nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Total nitrate-nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Toxaphene (Chlorinated camphene)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Trichloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Turbidity	EPA 180.1	Secondary Inorganic Contaminants	NELAP	10/8/2001
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001

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Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

Laboratory Scope of Accreditation

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State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,4,6-Tetrachlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001

Laboratory Scope of Accreditation

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State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	10/8/2001
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Sec-butyl-4,6-dinitrophenol (DNBP, Dinoseb)	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	10/8/2001
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,4,5-Trichlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,5-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,6-Trichlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,6-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,5,6-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001

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E87412
Columbia Analytical Services, Inc. - WA
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Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 625	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	7/17/2003
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	7/17/2003
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Adsorbable organic halogens (AOX)	EPA 1650	General Chemistry	NELAP	10/8/2001
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	10/8/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/23/2005
Aluminum	EPA 200.7	Metals	NELAP	10/8/2001
Aluminum	EPA 200.8	Metals	NELAP	10/8/2001
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Aluminum	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Amenable cyanide	EPA 335.1	General Chemistry	NELAP	10/8/2001
Ammonia as N	EPA 350.1	General Chemistry	NELAP	10/8/2001
Ammonia as N	EPA 350.3	General Chemistry	NELAP	10/8/2001
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 200.7	Metals	NELAP	10/8/2001
Antimony	EPA 200.8	Metals	NELAP	10/8/2001
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003
Antimony	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Aramite	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Arsenic	EPA 200.7	Metals	NELAP	10/8/2001
Arsenic	EPA 200.8	Metals	NELAP	10/8/2001
Arsenic	EPA 200.9	Metals	NELAP	10/8/2001
Arsenic	EPA 6010	Metals	NELAP	10/8/2001
Arsenic	EPA 6020	Metals	NELAP	7/1/2003
Arsenic	EPA 7060	Metals	NELAP	10/8/2001
Arsenic	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Azinphos-methyl (Guthion)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Barium	EPA 200.7	Metals	NELAP	10/8/2001
Barium	EPA 200.8	Metals	NELAP	10/8/2001
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Barium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Benzene	EPA 624	Volatile Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Benzidine	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	10/8/2001
Beryllium	EPA 200.8	Metals	NELAP	10/8/2001
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
Beryllium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
beta-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	10/8/2001
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003

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Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Bolstar (Sulprofos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Boron	EPA 200.7	Metals	NELAP	10/8/2001
Bromide	EPA 300.0	General Chemistry	NELAP	10/8/2001
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	EPA 624	Volatile Organics	NELAP	10/8/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cadmium	EPA 200.7	Metals	NELAP	10/8/2001
Cadmium	EPA 200.8	Metals	NELAP	10/8/2001
Cadmium	EPA 6010	Metals	NELAP	10/8/2001
Cadmium	EPA 6020	Metals	NELAP	7/1/2003
Cadmium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Calcium	EPA 200.7	Metals	NELAP	10/8/2001
Calcium	EPA 6010	Metals	NELAP	10/8/2001
Calcium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chemical oxygen demand	EPA 410.1	General Chemistry	NELAP	10/8/2001
Chemical oxygen demand	EPA 410.2	General Chemistry	NELAP	12/23/2005
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloride	EPA 300.0	General Chemistry	NELAP	10/8/2001
Chloride	EPA 325.3	General Chemistry	NELAP	10/8/2001
Chlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003

Laboratory Scope of Accreditation

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State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chloroform	EPA 624	Volatile Organics	NELAP	10/8/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorpyrifos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chromium	EPA 200.7	Metals	NELAP	10/8/2001
Chromium	EPA 200.8	Metals	NELAP	10/8/2001
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	7/1/2003
Chromium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Chromium VI	EPA 7195	Metals	NELAP	7/1/2003
Chromium VI	EPA 7196	General Chemistry	NELAP	7/1/2003
Chrysene	EPA 625	Extractable Organics	NELAP	10/8/2001
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chrysene	EPA 8310	Extractable Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Cobalt	EPA 200.7	Metals	NELAP	10/8/2001
Cobalt	EPA 200.8	Metals	NELAP	10/8/2001
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Cobalt	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Color	EPA 110.2	General Chemistry	NELAP	10/8/2001
Conductivity	EPA 120.1	General Chemistry	NELAP	10/8/2001
Copper	EPA 200.7	Metals	NELAP	10/8/2001
Copper	EPA 200.8	Metals	NELAP	10/8/2001
Copper	EPA 6010	Metals	NELAP	10/8/2001
Copper	EPA 6020	Metals	NELAP	7/1/2003
Copper	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Coumaphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Demeton-o	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Diazinon	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorovos (DDVP, Dichlorvos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diesel range organics (DRO)	CA-LUFT	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/28/2003
Diesel range organics (DRO)	NWTPH-Dx	Extractable Organics	NELAP	7/1/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Disulfoton	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003

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Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ethanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Ethoprop	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fecal coliforms	SM 9221 E	Microbiology	NELAP	10/8/2001
Fensulfothion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 625	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Fluoride	EPA 300.0	General Chemistry	NELAP	10/8/2001
Fluoride	EPA 340.2	General Chemistry	NELAP	10/8/2001
Formaldehyde	EPA 8315	Extractable Organics	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/23/2005
Gasoline range organics (GRO)	CA-LUFT	Extractable Organics	NELAP	7/1/2003
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	7/17/2003
Gasoline range organics (GRO)	NWTPH-Gx	Extractable Organics	NELAP	7/1/2003
Hardness	EPA 130.2	General Chemistry	NELAP	10/8/2001
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ignitability	EPA 1020	General Chemistry	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	EPA 200.7	Metals	NELAP	10/8/2001
Iron	EPA 6010	Metals	NELAP	7/1/2003
Iron	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Isophorone	EPA 625	Extractable Organics	NELAP	10/8/2001
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isosafrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Kjeldahl nitrogen - total	EPA 351.4	General Chemistry	NELAP	10/8/2001
Lead	EPA 200.7	Metals	NELAP	10/8/2001
Lead	EPA 200.8	Metals	NELAP	10/8/2001
Lead	EPA 200.9	Metals	NELAP	10/8/2001
Lead	EPA 6010	Metals	NELAP	10/8/2001
Lead	EPA 6020	Metals	NELAP	7/1/2003
Lead	EPA 7421	Metals	NELAP	10/8/2001
Lead	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Magnesium	EPA 200.7	Metals	NELAP	10/8/2001
Magnesium	EPA 6010	Metals	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Magnesium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Malathion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Manganese	EPA 200.7	Metals	NELAP	10/8/2001
Manganese	EPA 200.8	Metals	NELAP	10/8/2001
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/1/2003
Manganese	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Mercury	EPA 1631	Metals	NELAP	10/8/2001
Mercury	EPA 245.1	Metals	NELAP	10/8/2001
Mercury	EPA 7470	Metals	NELAP	10/8/2001
Mercury	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Merphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methanol	NCASI 94.03	Volatile Organics	NELAP	10/8/2001
Methanol	NCASI 99.01	Volatile Organics	NELAP	10/8/2001
Methapyrilene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	10/8/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylene chloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Mevinphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Molybdenum	EPA 200.7	Metals	NELAP	10/8/2001
Molybdenum	EPA 200.8	Metals	NELAP	10/8/2001
Molybdenum	EPA 6010	Metals	NELAP	10/8/2001
Molybdenum	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Naphthalene	EPA 625	Extractable Organics	NELAP	10/8/2001
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Naphthalene	EPA 8310	Extractable Organics	NELAP	7/1/2003
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.7	Metals	NELAP	10/8/2001
Nickel	EPA 200.8	Metals	NELAP	10/8/2001
Nickel	EPA 6010	Metals	NELAP	10/8/2001
Nickel	EPA 6020	Metals	NELAP	7/1/2003
Nickel	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	10/8/2001
Nitrate as N	EPA 353.2	General Chemistry	NELAP	7/17/2003
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	10/8/2001
Nitrite as N	EPA 300.0	General Chemistry	NELAP	10/8/2001
Nitrite as N	EPA 353.2	General Chemistry	NELAP	7/17/2003
Nitrobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/1/2003
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Oil & Grease	EPA 1664	General Chemistry	NELAP	10/8/2001
Orthophosphate as P	EPA 365.3	General Chemistry	NELAP	10/8/2001
o-Toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Parathion, ethyl	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
pH	EPA 150.1	General Chemistry	NELAP	10/8/2001
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
Phenacetin	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 625	Extractable Organics	NELAP	10/8/2001
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phorate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phosphorus, total	EPA 365.3	General Chemistry	NELAP	10/8/2001
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	EPA 200.7	Metals	NELAP	10/8/2001
Potassium	EPA 6010	Metals	NELAP	10/8/2001
Potassium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Residual free chlorine	EPA 330.4	General Chemistry	NELAP	10/8/2001
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	10/8/2001
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	10/8/2001
Residue-settleable	EPA 160.5	General Chemistry	NELAP	10/8/2001
Residue-total	EPA 160.3	General Chemistry	NELAP	10/8/2001
Residue-volatile	EPA 160.4	General Chemistry	NELAP	10/8/2001
Ronnel	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Safrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.7	Metals	NELAP	10/8/2001

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Selenium	EPA 200.8	Metals	NELAP	10/8/2001
Selenium	EPA 200.9	Metals	NELAP	10/8/2001
Selenium	EPA 6010	Metals	NELAP	10/8/2001
Selenium	EPA 7740	Metals	NELAP	10/8/2001
Selenium	EPA 7742	Metals	NELAP	7/17/2003
Selenium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Silver	EPA 200.7	Metals	NELAP	10/8/2001
Silver	EPA 200.8	Metals	NELAP	10/8/2001
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Silver	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sodium	EPA 200.7	Metals	NELAP	10/8/2001
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Sodium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Stirofos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Sulfate	EPA 300.0	General Chemistry	NELAP	10/8/2001
Sulfide	EPA 376.1	General Chemistry	NELAP	10/8/2001
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Thallium	EPA 200.7	Metals	NELAP	10/8/2001
Thallium	EPA 200.8	Metals	NELAP	10/8/2001
Thallium	EPA 200.9	Metals	NELAP	10/8/2001
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	EPA 7841	Metals	NELAP	7/1/2003
Thallium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Tin	EPA 200.7	Metals	NELAP	7/17/2003
Titanium	EPA 200.7	Metals	NELAP	7/17/2003
Tokuthion (Prothiophos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Toluene	EPA 624	Volatile Organics	NELAP	10/8/2001

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Toluene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Total coliforms	SM 9221 B	Microbiology	NELAP	10/8/2001
Total cyanide	EPA 335.4	General Chemistry	NELAP	7/17/2003
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Total cyanide	ILM04.1-Exhibit D	General Chemistry	NELAP	7/1/2003
Total hardness as CaCO3	EPA 200.7	Metals	NELAP	10/8/2001
Total organic carbon	EPA 415.1	General Chemistry	NELAP	10/8/2001
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/1/2003
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	EPA 1664	General Chemistry	NELAP	10/8/2001
Total Petroleum Hydrocarbons (TPH)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	NWTPH-HCID	Extractable Organics	NELAP	7/1/2003
Total phenolics	EPA 420.1	General Chemistry	NELAP	10/8/2001
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	10/8/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	10/8/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloronate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Trichlorosyringol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Turbidity	EPA 180.1	General Chemistry	NELAP	10/8/2001
Uranium	EPA 200.8	Metals	NELAP	10/8/2001
Vanadium	EPA 200.7	Metals	NELAP	10/8/2001
Vanadium	EPA 200.8	Metals	NELAP	10/8/2001
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Vanadium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Xylene (total)	EPA 624	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8021	Volatile Organics	NELAP	7/1/2003
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Zinc	EPA 200.7	Metals	NELAP	10/8/2001
Zinc	EPA 200.8	Metals	NELAP	10/8/2001
Zinc	EPA 6010	Metals	NELAP	10/8/2001
Zinc	EPA 6020	Metals	NELAP	7/1/2003
Zinc	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	7/17/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	7/17/2003
1-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/17/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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E87412
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Kelso, WA 98626

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	10/8/2001

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Hexanone	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitropropane	EPA 8260	Volatile Organics	NELAP	7/17/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Sec-butyl-4,6-dinitrophenol (DNBP, Dinoseb)	EPA 8270	Extractable Organics	NELAP	10/8/2001
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	7/17/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	10/8/2001
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	10/8/2001
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Acetone	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acetonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acetophenone	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	10/8/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aluminum	EPA 6010	Metals	NELAP	10/8/2001
Aluminum	EPA 6020	Metals	NELAP	10/8/2001
Aluminum	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Aniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
Anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Antimony	EPA 6010	Metals	NELAP	10/8/2001
Antimony	EPA 6020	Metals	NELAP	10/8/2001
Antimony	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Aramite	EPA 8270	Extractable Organics	NELAP	10/8/2001
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	10/8/2001

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Arsenic	EPA 7060	Metals	NELAP	10/8/2001
Arsenic	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Azinphos-methyl (Guthion)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Barium	EPA 6010	Metals	NELAP	10/8/2001
Barium	EPA 6020	Metals	NELAP	10/8/2001
Barium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Benzene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Benzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzoic acid	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	10/8/2001
Beryllium	EPA 6010	Metals	NELAP	10/8/2001
Beryllium	EPA 6020	Metals	NELAP	10/8/2001
Beryllium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
beta-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	10/8/2001
Bolstar (Sulprofos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Bromobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	10/8/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Cadmium	EPA 6010	Metals	NELAP	10/8/2001

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Cadmium	EPA 6020	Metals	NELAP	10/8/2001
Cadmium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Calcium	EPA 6010	Metals	NELAP	10/8/2001
Calcium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Carbazole	EPA 8270	Extractable Organics	NELAP	10/8/2001
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	10/8/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chloride	EPA 300.0	General Chemistry	NELAP	2/17/2006
Chloride	EPA 9056	General Chemistry	NELAP	7/17/2003
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chloroprene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlorpyrifos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chromium	EPA 6010	Metals	NELAP	10/8/2001
Chromium	EPA 6020	Metals	NELAP	10/8/2001
Chromium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Chromium VI	EPA 7195	Metals	NELAP	10/8/2001
Chromium VI	EPA 7196	General Chemistry	NELAP	10/8/2001
Chrysene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Chrysene	EPA 8310	Extractable Organics	NELAP	10/8/2001
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	10/8/2001
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Cobalt	EPA 6010	Metals	NELAP	10/8/2001
Cobalt	EPA 6020	Metals	NELAP	10/8/2001
Cobalt	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Copper	EPA 6010	Metals	NELAP	10/8/2001
Copper	EPA 6020	Metals	NELAP	10/8/2001
Copper	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Corrosivity (pH)	EPA 1110	General Chemistry	NELAP	10/8/2001
Coumaphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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Demeton-o	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Demeton-s	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Diazinon	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dibromomethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dichlorovos (DDVP, Dichlorvos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Diesel range organics (DRO)	CA-LUFT	Extractable Organics	NELAP	10/8/2001
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/17/2003
Diesel range organics (DRO)	NWTPH-Dx	Extractable Organics	NELAP	10/8/2001
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/17/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dimethoate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Disulfoton	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
EPN	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Ethoprop	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	7/17/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	10/8/2001
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Fensulfothion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Fenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Fluoride	EPA 300.0	General Chemistry	NELAP	2/17/2006
Fluoride	EPA 9056	General Chemistry	NELAP	7/17/2003
Formaldehyde	EPA 8315	Extractable Organics	NELAP	10/8/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Gasoline range organics (GRO)	CA-LUFT	Extractable Organics	NELAP	10/8/2001
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	7/17/2003
Gasoline range organics (GRO)	NWTPH-Gx	Extractable Organics	NELAP	10/8/2001
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Ignitability	EPA 1020	General Chemistry	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Iron	EPA 6010	Metals	NELAP	10/8/2001
Iron	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Isophorone	EPA 8270	Extractable Organics	NELAP	10/8/2001
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Isosafrole	EPA 8270	Extractable Organics	NELAP	10/8/2001
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Lead	EPA 6010	Metals	NELAP	10/8/2001
Lead	EPA 6020	Metals	NELAP	10/8/2001
Lead	EPA 7421	Metals	NELAP	10/8/2001
Lead	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Magnesium	EPA 6010	Metals	NELAP	10/8/2001
Magnesium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Malathion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Manganese	EPA 6010	Metals	NELAP	10/8/2001
Manganese	EPA 6020	Metals	NELAP	10/8/2001
Manganese	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Mercury	EPA 7470	Metals	NELAP	10/8/2001
Mercury	EPA 7471	Metals	NELAP	10/8/2001
Mercury	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Merphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methapyrilene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/17/2003
Methyl parathion (Parathion, methyl)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Mevinphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Molybdenum	EPA 6010	Metals	NELAP	10/8/2001
Molybdenum	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Naphthalene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Naphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001

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Naphthalene	EPA 8310	Extractable Organics	NELAP	10/8/2001
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Nickel	EPA 6010	Metals	NELAP	10/8/2001
Nickel	EPA 6020	Metals	NELAP	10/8/2001
Nickel	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Nitrate	EPA 9056	General Chemistry	NELAP	7/17/2003
Nitrite	EPA 9056	General Chemistry	NELAP	7/17/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	10/8/2001
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	7/17/2003
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Oil & Grease	EPA 1664	General Chemistry	NELAP	10/8/2001
Oil & Grease	EPA 9071	General Chemistry	NELAP	10/8/2001
o-Toluidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
Parathion, ethyl	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
p-Dioxane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/17/2003
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
pH	EPA 9040	General Chemistry	NELAP	10/8/2001
pH	EPA 9045	General Chemistry	NELAP	7/17/2003
Phenacetin	EPA 8270	Extractable Organics	NELAP	10/8/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Phenanthrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Phenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
Phorate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Potassium	EPA 6010	Metals	NELAP	10/8/2001
Potassium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	10/8/2001
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Pyridine	EPA 8270	Extractable Organics	NELAP	10/8/2001
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Ronnel	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Safrole	EPA 8270	Extractable Organics	NELAP	10/8/2001
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Selenium	EPA 6010	Metals	NELAP	7/17/2003
Selenium	EPA 7740	Metals	NELAP	10/8/2001
Selenium	EPA 7742	Metals	NELAP	7/17/2003
Selenium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Silver	EPA 6010	Metals	NELAP	10/8/2001
Silver	EPA 6020	Metals	NELAP	10/8/2001
Silver	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Sodium	EPA 6010	Metals	NELAP	10/8/2001
Sodium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Stirofos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Styrene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Sulfate	EPA 300.0	General Chemistry	NELAP	2/17/2006
Sulfate	EPA 9056	General Chemistry	NELAP	7/17/2003
Sulfide	EPA 9030/9034	General Chemistry	NELAP	7/17/2003
Sulfotepp	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	7/17/2003
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	7/17/2003
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	10/8/2001

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Thallium	EPA 6010	Metals	NELAP	7/17/2003
Thallium	EPA 6020	Metals	NELAP	10/8/2001
Thallium	EPA 7841	Metals	NELAP	10/8/2001
Thallium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Tokuthion (Prothiophos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Toluene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Toluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Total cyanide	ILM04.1-Exhibit D	General Chemistry	NELAP	10/8/2001
Total organic carbon	EPA 9060	General Chemistry	NELAP	10/8/2001
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	10/8/2001
Total Petroleum Hydrocarbons (TPH)	NWTPH-HCID	Extractable Organics	NELAP	10/8/2001
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	10/8/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichloronate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Vanadium	EPA 6010	Metals	NELAP	10/8/2001
Vanadium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	10/8/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8021	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Zinc	EPA 6010	Metals	NELAP	10/8/2001
Zinc	EPA 6020	Metals	NELAP	10/8/2001
Zinc	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001

Laboratory Scope of Accreditation

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State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003

Laboratory Scope of Accreditation

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State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
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Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	7/1/2003
Arsenic	EPA 7060	Metals	NELAP	7/1/2003
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cadmium	EPA 6010	Metals	NELAP	7/1/2003
Cadmium	EPA 6020	Metals	NELAP	7/1/2003
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	7/1/2003
Chromium VI	EPA 7196	Metals	NELAP	7/1/2003
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cobalt	EPA 6010	Metals	NELAP	7/1/2003

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Kelso, WA 98626

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Copper	EPA 6010	Metals	NELAP	7/1/2003
Copper	EPA 6020	Metals	NELAP	7/1/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Iron	EPA 6010	Metals	NELAP	7/1/2003
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Lead	EPA 6010	Metals	NELAP	7/1/2003
Lead	EPA 6020	Metals	NELAP	7/1/2003
Lead	EPA 7421	Metals	NELAP	7/1/2003
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/1/2003
Mercury	EPA 7471	Metals	NELAP	7/1/2003

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Molybdenum	EPA 6010	Metals	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nickel	EPA 6010	Metals	NELAP	7/1/2003
Nickel	EPA 6020	Metals	NELAP	7/1/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Selenium	EPA 6010	Metals	NELAP	7/1/2003
Selenium	EPA 7740	Metals	NELAP	7/1/2003
Selenium	EPA 7742	Metals	NELAP	7/1/2003
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	EPA 7841	Metals	NELAP	7/1/2003
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Zinc	EPA 6010	Metals	NELAP	7/1/2003
Zinc	EPA 6020	Metals	NELAP	7/1/2003

APPENDIX F

ADDITIONAL AGENCY-SPECIFIC DOCUMENTS

QUALITY ASSURANCE PROJECT PLAN (QAPP) ATTACHMENTS

ATTACHMENT 3
Smoky Canyon RI/FS Preliminary Data Usability Screening Summary Table Template and Data Usability Checklist

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision)

Table 1. Pre-Site Investigation Data (2000-2003)

Project Name	Data Collected By	Data Collection Objective/Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group IDs	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Panels B and C SEIS Soil and Vegetation Investigation	JBR Environmental	Data were collected to determine the concentrations of selenium and other metals in soil and vegetation in reclaimed areas, develop a site-specific relationship between the concentrations of metals in soil and vegetation, compare selenium concentrations in different vegetation species, and provide additional background information on selenium concentrations in undisturbed areas adjacent to the mine.	JBR Environmental Consultants, Inc., 2000. Study Plan for Soil and Vegetation Sampling at the Smoky Canyon Mine, prepared for J.R. Simplot Company, July 18, 2000 (Appendix A of the Data Report).	JBR Environmental Consultants, Inc., 2001. Data Report for Soil/Growth Medium and Vegetation Sampling at the J.R. Simplot Smoky Canyon Mine, prepared for J.R. Simplot Company, Afton, WY, by JBR Environmental Consultants, Inc., Sandy, UT, June 2001.	Soil	Summer 2000	F	F	SVL	P	95149 95151 95243 95272 95286 95336 95339 95356	Cadmium, copper, manganese, molydenum, nickel, vanadium, zinc (EPA 6010B); cation exchange capacity (SM 9081), pH (SM 9045), selenium (EPA 7740), extractable selenium (SM 3114B)	MDL	NP	A total of 33 soil samples were collected from 29 quadrants in six separate reclamation areas and four quadrants in background areas. One composite soil sample was collected from each randomly located 100-foot by 100-foot quadrant. Samples were taken from the entire rooting depth, not just the top two inches of soil.	Soil samples were not analyzed for all RI/FS target analytes. COCs, field forms and lab data reports provided as an appendix to the report. Although the report provides an evaluation of LCS, preparation blanks, duplicates, MS and field duplicates, there is no QC report.	Partial lab data packages for review	
					Vegetation	Summer 2000	F	F	SVL	P	95150 95244 95245 95274 95337 95355 95388	Cadmium, copper, manganese, molydenum, nickel, vanadium, zinc (EPA 6010B); selenium (EPA 7740)	MDL	NP	Vegetation samples were collected from the same quadrants as the soil samples to allow geochemical comparison. One composite vegetation sample was collected from each quadrant. Samples of the four dominant vegetation species present at each reclamation area were collected for a separate species-specific analysis.	Vegetation samples were not analyzed for all RI/FS target analytes. COCs, field forms and lab data reports provided as an appendix to the report. Although the report provides an evaluation of LCS, preparation blanks, duplicates, MS and field duplicates, there is no QC report.	Partial lab data packages for review	
Chemical and Isotopic Investigation - Groundwater/Surface Water	Mayo and Associates	Characterize groundwater chemistry and flow directions in aquifers in the vicinity of Smoky Canyon Mine, including an evaluation of sources of water to groundwater discharged at Hoopes Spring and lower Smoky spring.	None on file. Samples collected by Maxim Technologies and Mayo/JBR in accordance with procedures for baseline resource studies.	Mayo, A., 2001. Chemical and Isotopic Investigation of Groundwater Flow Systems in the Vicinity of Smoky Canyon Mine, Idaho, and Results of Solute Transport Modeling, prepared for JBR Environmental by Alan Mayo, March 12, 2001.	Surface water Groundwater	2000	NP	NP	Northern Analytical Laboratories, BYU, and Univ. of Miami	NP	NP	Major ions, fluoride, total phosphorus in accordance with baseline study methods. Deuterium and oxygen-18 (mass spectrometry), tritium and carbon-14 (counting methods).	RL	NP	Vicinity of Panels A, B, C, and downstream to Tygee Creek drainage below tailings impoundments; lower Smoky springs and Hoopes Spring	Surface water and groundwater samples were not analyzed for all RI/Fs target analytes.	No original lab data packages for review	
Panels B and C SEIS Water Resources Baseline Study	Maxim Technologies	Characterize baseline surface water, sediment, and groundwater quality in the vicinity of Smoky Canyon Mine and support supplemental environmental impact assessment performed by USFS and BLM to assess potential impacts from planned Panels B and C mining activities.	Maxim Technologies, Inc., 2000. Preliminary Environmental Baseline Study Plan for Smoky Canyon Mine Panels B & C SEIS, prepared for J.R. Simplot Company, Afton, WY, March 2000.)	Maxim Technologies, Inc., 2000. Final Water Resources Baseline Study Report, First, Second and Third Quarter 2000, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, November 2000. Maxim Technologies, Inc., 2001. Revised Final Water Resources Baseline Report, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, April 2001.	Surface Water Groundwater	2000	NP	NP	Northern Analytical Laboratories	NP	NP	Major ions, nutrients, TSS, TDS, alkalinity, hardness, fluoride, nitrate,nitrite, total phosphorus, ortho-phosphorus, dissolved aluminum, antimony, arsenic, barium, beryllium, boron, cadmium, chromium, copper, iron, lead, manganese, mercury, nickel, selenium, silver, thallium, vanadium, zinc (methods not provided in report)	PQL	P	Vicinity of Panels A, B, C, and downstream to Tygee Creek drainage below tailings impoundments.	Surface water and groundwater samples were not analyzed for all RI/Fs target analytes. Data tables and data validation report provided in the revised final report.	Data validation summaries; No original lab data packages for review	
					Sediment	2000	NP	P	Energy Laboratories (metals) Maxim, Helena, MT (grain size)	NP	NP	Aluminum, antimony, arsenic, barium, beryllium, boron, cadmium, chromium, copper, iron, lead, manganese, mercury, nickel, selenium, silver, thallium, vanadium, zinc (EPA 6000/7000); grain size distribution (ASTM D422 and D1140)	PQL	P	Vicinity of Panels A, B, C, and downstream to Tygee Creek drainage below tailings impoundments.	Sediment samples were not analyzed for all RI/Fs target analytes. Data tables and data validation report provided in the revised final report.	No original lab data packages for review	
Panels B and C SEIS Soil and Vegetation Resources Baseline Study	Maxim Technologies	Characterize soil and vegetation resources in the vicinity of Smoky Canyon Mine and support supplemental EIS for Panels B and C.	Maxim Technologies, Inc., 2000. Preliminary Environmental Baseline Study Plan for Smoky Canyon Mine Panels B & C SEIS, prepared for J.R. Simplot Company, Afton, WY, March 2000.)	Duane Noel for Maxim Technologies Inc., 2001. Revised Draft Baseline Study for Smoky Canyon Mine Panels B & C Soil Resources, Caribou County, Idaho, prepared for J.R. Simplot Company, Smoky Canyon Mine, March 2001. Maxim Technologies, Inc., 2002. Final Baseline Study for Smoky Canyon Mine Panels B & C Vegetation Resources, prepared for J.R. Simplot Company, Smoky Canyon Mine, Afton, WY by Maxim Technologies, Inc., Boise, ID, March 13, 2002.	Soil	2000	F	F	Northern Analytical Laboratories and Energy Laboratories	P	NP	Cadmium, copper, manganese, molybdenum, nickel, vanadium, zinc (EPA 6020), selenium (EPA 7742), organic matter (S-8.10), potassium available (S-5.10), sand, silt, clay, texture (S-14.10), coarse fragment content (D421), cation exchange capacity (S-10.20), electrical conductivity (S-1.20), saturation (S-1.00), pH (S-1.10), calcium, magnesium, sodium, sodium adsoprtion ratio (S-1.60); trace elements (metals) were also analyzed by water extractable methods (EPA 6010/6020) and by saturated paste methods (EPA 6010B/6020)	PQL	NP	Soil samples were collected at 22 locations for each soil type or soil mapping unit. Several sample swere collected at each location, one for each soil horizon.	Field soil description forms, COCs and analytical data reports provided as an attachment to the final report.	No original lab data packages for review	
					Soil	2000	F	NP	Northern Analytical Laboratories	P	NP	Cadmium, copper, manganese, molybdenum, nickel, vanadium, zinc (EPA 6020), selenium (EPA 7742), organic matter (S-8.10), potassium available (S-5.10), sand, silt, clay, texture (S-14.10), coarse fragment content (D421), cation exchange capacity (S-10.20), electrical conductivity (S-1.20), saturation (S-1.00), pH (S-1.10), calcium, magnesium, sodium, sodium adsoprtion ratio (S-1.60)	PQL	NP	Soil and vegetation samples were collected in one riparian area and nine upland or forrested areas. Composite samples were collected from at least five sampling quadrants at each location. Locations were chosen based on potential for selenium and other contaminant transport via surface or subsurface flow.	COCs and analytical data reports provided as an attachment to the final report.	No original lab data packages for review	
					Vegetation	2000	F	NP	Northern Analytical Laboratories	P	NP	Cadmium, copper, manganese, molybdenum, nickel, vanadium, zinc (EPA 6020), selenium (EPA 7742)	PQL	NP	See above.	COCs and analytical data reports provided as an attachment to the final report.	No original lab data packages for review	
Area B Baseline Ecological Risk Assessment	MFG for Simplot	Data were collected for a screening level exposure assessment at the Smoky Canyon Mine. COPCs for ecological receptors at the tailings pond facility for which screening-level exposure exceeded NOAELS were evaluated in the baseline risk assessment. The baseline assessment for avian receptors focused on risks from chromium and selenium. For mammalian receptors, the baseline assessment focused on selenium and vanadium.	MFG, Inc., 2002. Smoky Canyon Mine Area B (Tailings Pond Area) Baseline Ecological Risk Assessment Work Plan, prepared for J.R. Simplot Company by MFG Inc., September 2002.	MFG, Inc., 2003. Draft Smoky Canyon Mine Area B (Tailings Ponds Area) Baseline Ecological Risk Assessment Report, prepared for J.R. Simplot Company by MFG Inc., July 2003. MFG, Inc., 2004. Smoky Canyon Mine Area B (Tailings Impoundments) Final Tailings Impoundment Recommendation Report, prepared for J.R.Simplot Company by MFG Inc., January 2004.	Fish tissue	Spring Summer 2002	P	P	ACZ	P	L37411 L38208	Aluminum, barium, chromium, cobalt, nickel, vanadium, zinc (EPA 6010B); arsenic, cadmium, lead, silver (EPA 6020); mercury (EPA 7471), selenium (EPA 7742)	MDL PQL	F	Four fish samples were collected from each tailings pond in June; three species from TP1 and one species from TP2. Six fish samples were collected in August, all from TP1.	Fish tissue samples were not analyzed for all RI/FS target analytes. SDG L37411 is missing the COC, data report, QC summary and raw data. SDG L38208 includes the COC, data report and QC summary but no raw data.	Data validated; one partial lab data package available for review.	
					Bird tisssue	Summer 2002	NP	F	ACZ	P	L38212 L38214 L38216 L38863 L38864 L38865	Aluminum, barium, chromium, cobalt, nickel, vanadium, zinc (EPA 6010B); arsenic, cadmium, lead, silver (EPA 6020); mercury (EPA 7471), selenium (EPA 7742)	MDL PQL	F	Eight waterfowl samples were collected in August and four waterfowl samples were collected in September from the tailings pond area. Samples were split into components: pelt, liver and carcass for analysis.	Bird tissue samples were not analyzed for all RI/FS target analytes. Raw data for SDGs L38212, L38214 and L38216 is provided in a separate folder. SDGs L38863, L38864 and L38865 are missing COCs, data reports, QC summaries and raw data.	Data validated; partial lab data packages for review.	
					Vegetation	Summer 2002	NP	F	ACZ	NP	L38839 L38840 L38842 L38843	Aluminum, barium, chromium, cobalt, nickel, vanadium, zinc (EPA 6010B); arsenic, cadmium, lead, silver (EPA 6020); mercury (EPA 7471), selenium (EPA 7742)	MDL PQL	F	Terrestrial plants: Six composite samples of herbaceous vegetation and six composite samples of woody browse vegetation were collected from the margin of each tailings pond. Aquatic plants: Six composite rooted and six composite algae samples were collected from the margins and the center of TP1. Four composite rooted and six composite algae samples were collected from TP2.	Vegetation samples were not analyzed for all RI/FS target analytes. SDGs L38839, L38840, L38842 and L38843 are missing COCs, data reports, QC summaries and raw data. Data validation reports iare available.	Data validated but original lab data packages not available for review.	

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision)

Table 1. Pre-Site Investigation Data (2000-2003)

Project Name	Data Collected By	Data Collection Objective/Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group IDs	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Area B Baseline Ecological Risk Assessment (continued)	MFG for Simplot	Data were collected for a screening level exposure assessment at the Smoky Canyon Mine. COPCs for ecological receptors at the tailings pond facility for which screening-level exposure exceeded NOAELS were evaluated in the baseline risk assessment. The baseline assessment for avian receptors focused on risks from chromium and selenium. For mammalian receptors, the baseline assessment focused on selenium and vanadium.	MFG, Inc., 2002. Smoky Canyon Mine Area B (Tailings Pond Area) Baseline Ecological Risk Assessment Work Plan, prepared for J.R. Simplot Company by MFG Inc., September 2002.	MFG, Inc., 2003. Draft Smoky Canyon Mine Area B (Tailings Ponds Area) Baseline Ecological Risk Assessment Report, prepared for J.R. Simplot Company by MFG Inc., July 2003.	Aquatic insect tissue	Summer 2002	NP	F	ACZ	NP	L38862	Aluminum, barium, chromium, cobalt, nickel, vanadium, zinc (EPA 6010B); arsenic, cadmium, lead, silver (EPA 6020); mercury (EPA 7471), selenium (EPA 7742)	MDL PQL	F	Five grab composite samples were collected from TP1. Although similar invertebrates were observed in TP2, insufficient biomass was available for analysis.	Insect samples were not analyzed for all RI/FS target analytes. SDG L38862 is missing the COC, data report, QC summary and raw data. Data validation report is available.	Data validated but original lab data packages not available for review.	
				MFG, Inc., 2004. Smoky Canyon Mine Area B (Tailings Impoundments) Final Tailings Impoundment Recommendation Report, prepared for J.R.Simplot Company by MFG Inc., January 2004.	Terrestrial insect tissue	Summer 2002	NP	F	ACZ	NP	L38861	Aluminum, barium, chromium, cobalt, nickel, vanadium, zinc (EPA 6010B); arsenic, cadmium, lead, silver (EPA 6020); mercury (EPA 7471), selenium (EPA 7742)	MDL PQL	F	Terrestrial insect samples were collected by sweep netting along the tailings pond margins. Three composite samples were collected from TP1. Due to the lack of vegetative cover, sweeps from TP2 margins produced insufficient biomass for analysis.	Insect samples were not analyzed for all RI/FS target analytes. SDG L38861 is missing the COC, data report, QC summary and raw data. Data validation report is available.	Data validated but original lab data packages not available for review.	
					All tissue	Fall 2002	NP	NP	ACZ	P	L39509	Selenium (EPA 7742) reanalyzed for all tissue samples using dilutions	MDL PQL	F	Tissue samples from SDGs L38839, L38840, L38842, L38861, L38862, L38864 and L38865 were reanalyzed.	SDG L39509 includes the data report and a QC summary but no raw data.	Partial lab data package for review	
Area B Groundwater and Other Environmental Media Investigations	MFG for Simplot	Characterize site conditions and identify any COPC transport pathways from the tailings impoundments.	MFG, Inc., 2002. Smoky Canyon Mine Area B (Tailings Pond Area), Groundwater and Other Environmental Media Investigations Work Plan, prepared for J.R. Simplot Company by MFG, Inc., November 2002.	MFG, Inc., 2003. Draft Smoky Canyon Mine Area B (Tailings Ponds Area) Groundwater and Other Environmental Media Investigation Report, prepared for J.R. Simplot Company by MFG Inc., September 2003.	Surface water Groundwater	2003	F	F	ACZ	P	L39727 L39728	Aluminum, arsenic, barium, cadmium, chromium, cobalt, copper, lead, nickel, silver, vanadium, zinc (200.8); mercury (245.5); selenium (SM 3114C); Ra-226 (903.0); Ra-228 (904.0)	MDL PQL	F	Smoky Creek and Tygee Creek drainages in the vicinity of the two tailings impoundments.	Samples not analyzed for all RI/FS target analytes. SDG L39727 missing original COC.	Partial lab data package for review	
					Sediment Tailings	2003	F	F	ACZ	P	L39748 L39749 L39750	Aluminum, barium, chromium, cobalt, nickel, vanadium, zinc (EPA 6010B); arsenic, cadmium, lead, silver (EPA 6020); mercury (EPA 7471), selenium (EPA 7742); Ra-226 (EPA 9315); Ra-228 (EPA 9320)	MDL PQL	F	Smoky Creek and Tygee Creek drainages in the vicinity of the two tailings impoundments.	Samples identified as sediment are tailings collected from the two impoundments. Samples not analyzed for all RI/FS target analytes.	Partial lab data package for review	
Smoky Canyon Mine Pre-Site Investigation Surface Water Investigation	MFG for Simplot	Surface water data were collected to characterize the loading of COPCs to Sage Creek during snowmelt runoff and during low flow conditions, describe COPC concentrations in surface water relative to applicable water quality standards, identify areas of discrete seepage or surface runoff from overburden disposal areas, characterize seep flow, and characterize COPC-load contribution to surface water from overburden disposal areas.	MFG, Inc., 2002. Spring 2002 Surface Water Quality Sampling and Analysis Plan, prepared for J.R. Simplot Company by MFG, Inc., April 19, 2002.	MFG, Inc., 2002. Spring 2002 Surface Water Quality Data Report, Smoky Canyon Mine, prepared for J.R. Simplot Company by MFG Inc., October 1, 2002.	Surface water	Spring 2002	F	F	ACZ	P	L36911	Aluminum, barium, boron, calcium, cobalt, iron, magnesium, manganese, potassium, sodium, vanadium, zinc (EPA 200.7); arsenic, cadmium, chromium, copper, lead, nickel, silver (EPA 200.8); selenium (SM 3500), mercury (EPA 245.1), alkalinity (SM 2320B), chloride (EPA 325.1), fluoride (SM 4500F-C), hardness (SM 2340B), nitrate/nitrite, nitrite (EPA 353.2); ammonia (EPA 350.1), phosphorous (EPA 365.1), TDS (EPA 160.1), TSS (EPA 160.2), sulfate (EPA 375.3)	MDL PQL	F	Surface water samples were collected at six locations within the northern portion of Sage Valley north of Sage Creek, four locations in Sage Creek within the lower portion of Sage Valley, one location below the area of discharge for Hoopes Spring, one location on the Sage Creek irrigation diversion ditch in Sage Valley, and from six seep and overburden runoff stations.	Surface water samples were not analyzed for all RI/FS target analytes. SDG L36911 includes the COC, data report and QC summary but no raw data.	Partial lab data package for review	
			MFG, Inc., 2002. Fall 2002 Surface Water Quality Sampling and Analysis Plan, Smoky Canyon Mine, prepared for J.R. Simplot Company by MFG Inc., October 1, 2002.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine, Area A, prepared for J.R. Simplot Company by NewFields Boulder, July 2005.	Surface water	Fall 2002	F	F	ACZ	P	L39011 L39012	Aluminum, barium, boron, calcium, cobalt, iron, magnesium, manganese, potassium, sodium, vanadium, zinc (EPA 200.7); arsenic, cadmium, chromium, copper, lead, nickel, silver (EPA 200.8); selenium (SM 3114C), mercury (EPA 245.1), alkalinity (SM 2320B), chloride (EPA 325.1), fluoride (SM 4500F-C), hardness (SM 2340B), nitrate/nitrite, nitrite (EPA 353.2); ammonia (EPA 350.1), phosphorous (EPA 365.1), TDS (EPA 160.1), TSS (EPA 160.2), sulfate (EPA 375.3)	MDL PQL	P	Surface water samples were collected from the 13 Sage Valley and Sage Creek locations described above, six seep and overburden runoff locations, and three additional stations near Hoopes Spring, South Fork Sage Creek Springs, and from the toe drain below the TP2 dam.	Surface water samples were not analyzed for all RI/FS target analytes. SDGs L39011 and L39012 include COCs, data reports and QC summaries but no raw data. SDG L39012 has not been validated.	Partial lab data packages for review	
			MFG, Inc., 2003. Spring 2003 Surface Water Quality Sampling and Analysis Plan, prepared for J.R. Simplot by MFG Inc., May 2003.	Letter Report from T. Drexel (MFG) to B. Winegar and J. Cunningham (Simplot), August 12, 2003, Smoky Canyon Mine Spring 2003 Surface Water Monitoring Event . Letter Report from T. Drexel (MFG) to L. Hamann (Simplot), June 6, 2003, Spring 2003 South Fork Sage Creek Gain-Loss Survey. NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine, Area A, prepared for J.R. Simplot Company by NewFields Boulder, July 2005.	Surface water	Spring 2003	F	F	ACZ	P	L41359 L41360 L41379 L41380 L41731 L41845	Aluminum, barium, calcium, iron, magnesium, manganese, potassium, sodium, vanadium, zinc (EPA 200.7); arsenic, cadmium, chromium, copper, lead, nickel, silver (EPA 200.8); selenium (SM 3114C), alkalinity (SM 2320B), chloride (EPA 325.1), fluoride (SM 4500F-C), hardness (SM 2340B), nitrate/nitrite, nitrite (EPA 353.2); ammonia (EPA 350.1), phosphorous (EPA 365.1), TDS (EPA 160.1), TSS (EPA 160.2), sulfate (EPA 375.3)	MDL PQL	F	Surface water samples were collected from 26 locations in Upper and Lower Pole Canyon Creek, North Fork Sage Creek, Upper and Lower Sage Creek, Sage Valley irrigation diversion ditch, Hoopes Spring, South Fork Sage Creek, Crow Creek, and Panels A, D and E.	Surface water samples were not analyzed for all RI/FS target analytes. SDGs L41359, L41360, L41379, and L41845 include COCs, data reports and QC summaries but no raw data. SDGs L41380 and L41731 are missing COCs, data reports, QC summaries and raw data.	Partial lab data packages for review	
Smoky Creek Gain-Loss Survey	MFG for Simplot	Surface water flows measured and samples collected upstream and downstream of lower Smoky Springs with samples also submitted for stable isotopic analyses. This work was required under the Panels B and C Record of Decision, and data were reported to USFS/BLM by Smoky Canyon Mine.	MFG, Inc., 2002. Draft Comprehensive Environmental Monitoring Program Plans, prepared for J.R. Simplot Company, Smoky Canyon Mine, November 2002.	Letter report to John Cunningham, Simplot, from T. Drexel, MFG, dated June 26, 2003, Re: Spring 2003 Smoky Creek Gain-Loss Survey, Smoky Canyon Mine. Letter report to J. Cunningham, Simplot, from K. Tegtmeyer, MFG, dated October 21, 2004, Re: Spring 2003 Smoky Creek Gain-Loss Survey, Smoky Canyon Mine: Isotope Data Report.	Surface water	Spring 2003	F	F	BYU	P	NP	Deuterium and oxygen-18 by mass spectrometry methods	NA (precison of isotopic ratios provided)	NA	Smoky Creek between haul road crossing and lower Smoky Ck surface water monitoring station LSm.	Non-standard water quality analysis methods; mass spectrometry methods used with appropriate quality assurance/quality control protocols specific to method.	Limited QC data and no raw data in laboratory reports.	

F = Full
P = Partial
NP = Not present/Not provided
NA = Not applicable

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision

Table 2. Site Investigation Data (2003-2004)

Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group IDs	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Smoky Canyon Mine Site Investigation	MFG and NewFields for Simplot	Data were collected for the Smoky Canyon Mine Area A Site Investigation to identify the primary sources of selenium and other COPCs released to the environment; describe the mechanisms for releases of selenium and other COPCs from these sources; characterize the current extent of transport of selenium and other COPCs from identified sources; and identify pathways of exposure for a range of ecological receptors. Data were also used to identify the potential for human exposure and to evaluate the baseline ecological and human health risks. Specific objectives for each media are described below.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Surface water	Fall 2003 Winter 2004 Spring 2004 Summer 2004	F	P	SVL	F	107843 107844 107845 108410 108436 108438 108478 109604 110942 110943 111023 111031 111060 111107 111424 112307 112345 112348 112350 113661 113663 113687 114099 114541	Aluminum, barium, calcium, chromium, copper, iron, magnesium, manganese, nickel, potassium, sodium, vanadium, zinc (EPA 200.7); cadmium (EPA 213.2), lead (EPA 239.2), mercury (EPA 245.1), arsenic (EPA 206.2), silver (EPA 272.2), selenium (SM 3114C), alkalinity (SM 2320), chloride, fluoride, sulfate (EPA 300.0); nitrate/nitrite (EPA 353.2), ortho-phosphate (EPA 365.2); ammonia (EPA 350.1), TSS (EPA 160.2), TDS (EPA 160.1), TOC (EPA 415.1), hardness (SM 2340B)	MDL	F	Surface water samples were collected during six monitoring events from 36 locations in Smoky Creek, Roberts Creek, Pole Canyon Creek, Sage Creek, Hoopes Spring, South Fork Sage Creek, North Fork Sage Creek and North Sage Valley, Lower Sage Valley, Crow Creek, Tygee Creek, and Panels A, C, D and E. Samples collected during two of the events (Fall 2002, Spring 2003) are covered under SCM Pre-Site Investigation Surface Water Monitoring.	Surface water samples were not analyzed for all RI/FS target analytes. The SDGs from SVL contain the COCs, data reports, QC data and raw data. Data validated in accordance with SI QAPP.	Complete lab data packages for review.	
		Surface water data were collected to monitor water quality during a non-drought or above normal runoff period and better define surface water/groundwater interactions. Data from gain/loss surveys were used to evaluate specific stream segments for gaining/losing conditions.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Seep/spring surface water	Fall 2002 Spring 2004	F	F	ACZ SVL	P	L39011 110942	Aluminum, barium, calcium, chromium, copper, iron, magnesium, manganese, nickel, potassium, sodium, vanadium, zinc (EPA 200.7); cadmium (EPA 213.2), lead (EPA 239.2), mercury (EPA 245.1), arsenic (EPA 206.2), silver (EPA 272.2), selenium (SM 3114C), alkalinity (SM 2320), chloride, fluoride, sulfate (EPA 300.0); nitrate/nitrite (EPA 353.2), ortho-phosphate (EPA 365.2); ammonia (EPA 350.1), TSS (EPA 160.2), TDS (EPA 160.1), TOC (EPA 415.1), hardness (SM 2340B)	MDL	F	Surface water samples were collected from four locations in Sage Valley; one identified as a potential sping/wetland area and the other three identified as areas of potential groundwater discharge to ponds.	Surface water samples were not analyzed for all RI/FS target analytes. The SDG from ACZ (selenium speciation analyses) contains the COC, data report and QC summary but no raw data. The SDG from SVL contains the COC, data report, QC data and raw data. Data validated in accordance with SI QAPP.	Complete lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Detention basin storm water	Spring 2003 Summer 2004	F	F	ACZ SVL	P	L41379 L41845 112345 112348 112350 112393 114532	Aluminum, barium, calcium, chromium, copper, iron, magnesium, manganese, nickel, potassium, sodium, vanadium, zinc (EPA 200.7); cadmium (EPA 213.2), lead (EPA 239.2), mercury (EPA 245.1), arsenic (EPA 206.2), silver (EPA 272.2), selenium (SM 3114C), alkalinity (SM 2320), chloride, fluoride, sulfate (EPA 300.0); nitrate/nitrite (EPA 353.2), ortho-phosphate (EPA 365.2); ammonia (EPA 350.1), TSS (EPA 160.2), TDS (EPA 160.1), TOC (EPA 415.1), hardness (SM 2340B)	MDL	F	A total of 15 surface water samples were collected from three detention basins around A Panel, seven detention basins around D Panel and five detention basins around E Panel.	Storm water samples were not analyzed for all RI/FS target analytes. The ACZ SDGs (selenium speciation analyses) contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Complete lab data packages for review.	
		Sediment data were collected to describe the extent of sediment transport from source areas and determine sediment quality conditions around the mine. Data were also used to characterize the quality of surficial materials that may be used as substrate for vegetation during reclamation.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Detention basin sediment	Summer 2004	F	F	SVL	F	112345 112400 112401	Cadmium, chromium, copper, nickel, vanadium, zinc, phosphorous (EPA 6010B); selenium (SM 3114C)	MDL	F	Sediment samples were collected from 30 detention basins. Five subsamples of sediment were collected from across the basin footprint area. The five subsamples were combined and homogenized to make one composite sample for each basin.	Sediment samples were not analyzed for all RI/FS target analytes. The SDGs contain COCs, data reports, QC summaries and raw data. Data validated in accordance with SI QAPP.	Complete lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Stream sediment	Summer 2004	F	F	SVL	F	112372	Cadmium, chromium, copper, nickel, vanadium, zinc, phosphorous (EPA 6010B); selenium (SM 3114C)	MDL	F	Sediment samples were collected from 15 locations in Tygee Creek, Upper and Lower Smoky Creek, Roberts Creek, Upper and Lower Pole Creek, Lower Sage Valley, Upper and Lower Sage Creek, Hoopes Spring, Upper and Lower South Fork Sage Creek and North Sage Valley.	Sediment samples were not analyzed for all RI/FS target analytes. The SDG contains the COC, data report, QC data and raw data. Data validated in accordance with SI QAPP.	Complete lab data package for review.	
		Groundwater data were collected to define the nature and extent of COPCs in groundwater and better understand groundwater transport of COPCs from source areas. The groundwater data were also used to determine the relative importance of hydrostratigraphic flow units for COPC transport and the relationship of the shallow flow system to surface water features.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Groundwater	4Qtrs Fall 2003 to Fall 2004	F	F	ACZ SVL	P	L45740 L45896 L47011 107799 107804 107805 107843 107844 107845 108478 108479 109603 110941 111107 111757 112392 112590 113662 113867 113869 114099 114532 114541	ACZ - Selenium total, selenium dissolved, selenium organic, selenium (IV), selenium (VI) (SM 3114B); SVL - Aluminum, barium, beryllium, boron, calcium, chromium, copper, cobalt, iron, magnesium, molybdenum, nickel, potassium, sodium, vanadium, zinc (EPA 200.7); cadmium (EPA 213.2), lead (EPA 239.2), mercury (EPA 245.2), arsenic (EPA 206.2), silver (EPA 272.2), antimony (EPA 204.2), thallium (EPA 279.2), selenium (SM 3114C), alkalinity (SM 2320), chloride, fluoride, sulfate (EPA 300.0); nitrate/nitrite (EPA 353.2), phosphorous,ortho-phosphate (EPA 365.2); ammonia (EPA 350.1), TSS (EPA 160.2), TDS (EPA 160.1), pH (EPA 150.1), turbidity (EPA 180.1), specific conductivity (EPA 120.1), hardness (SM 2340B)	MDL	F	Groundwater samples were collected from 1 well in the pit backfill and overburden disposal areas; 8 wells in the shallow flow system in the Pole Canyon area, Hoopes Spring area, South Fork Sage Creek drainage, and the northern, central and southern portions of Sage Valley; 4 wells in the Wells Formation in the Pole Canyon area, Hoopes Spring area, South Fork Sage Creek drainage and the Smoky Canyon/C Panel area; and 2 water supply wells along Smoky Creek south of the mine offices. Samples were also collected from one domestic well and three springs used as domestic water supplies.	Domestic wells were sampled only in fall 2003. Not all samples sent to SVL were analyzed for all the analytes/methods listed. ACZ SDGs (selenium speciation analyses) contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Complete lab data packages for review.	

Table 2. Site Investigation Data (2003-2004)

Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group IDs	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Smoky Canyon Mine Site Investigation (continued)	MFG and NewFields for Simplot	Near-surface soil, overburden soil and roadway materials samples were collected to describe the extent of COPC transport to soil from source areas and overburden areas, evaluate the suitability of existing soils on overburden disposal areas to support reclamation vegetation, and assess the exposure pathways from soil to ecological receptors.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Overburden area soil	Summer 2004	F	F	SVL	F	112346 112349 112352 112368 112370 112371 112375 112376	Cadmium, chromium, copper, nickel, vanadium, zinc (EPA 6010B); selenium (SM 3114C), TOC (Leco), pH (ASA M9), total organic matter (H60#24), nitrate/nitrite as N (EPA 353.1), TKN nitrogen (EPA 351.4)	MDL	P	A total of 60 soil samples were collected from 30 locations along transects that cross the A Panel disposal area, Pole Canyon disposal area, D Panel backfill area and D Panel disposal area, and 52 samples were collected from 26 locations on native soils on either side of the overburden disposal areas. Half of the samples were from the top 2 inches of soil and the other half from the root zone. Each of these samples was a composite of five subsamples collected from five randomly selected locations within a 100-foot by 100-foot grid.	Soil samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports, QC data and raw data. The data validation reports are for metals only; data validated in accordance with SI QAPP.	Complete lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Seep area soil	Summer 2004 Fall 2004	F	P	SVL	F	112352 112353 112370 112376 112393 112400 114539	Cadmium, chromium, copper, nickel, vanadium, zinc (EPA 6010B); selenium (SM 3114C), TOC (Leco), pH (ASA M9), total organic matter (H60#24), nitrate/nitrite as N (EPA 353.1), TKN nitrogen (EPA 351.4)	MDL	P	A total of 44 soil samples were collected from 22 locations around the D and E seep areas and detention basin DP-10, half from the top 2 inches of soil and the other half from the root zone. Each of these samples was a composite of five subsamples collected within a 20-foot by 20-foot area.	Soil samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports, QC data and raw data. The data validation reports are for metals only. Data validated in accordance with SI QAPP.	Complete lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Sage Valley soil	Summer 2004	F	F	SVL	F	112373 112374 112377	Cadmium, chromium, copper, nickel, vanadium, zinc (EPA 6010B); selenium (SM 3114C), TOC (Leco), pH (ASA M9), total organic matter (H60#24), nitrate/nitrite as N (EPA 353.1), TKN nitrogen (EPA 351.4)	MDL	P	Twenty soil samples were collected at 1000-foot intervals in a square sampling grid within northern Sage Valley where land was irrigated from either Pole Canyon or Sage Creek. Subsamples were collected from five random pits within a 100-foot by 100-foot area. The subsamples were combined and homogenized to form composite samples.	Soil samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports, QC data and raw data. The data validation reports are for metals only. Data validated in accordance with SI QAPP.	Complete lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Roadway soil	Summer 2004	F	F	SVL	F	112374 112377	Cadmium, chromium, copper, nickel, vanadium, zinc (EPA 6010B); selenium (SM 3114C), TOC (Leco), pH (ASA M9), total organic matter (H60#24), nitrate/nitrite as N (EPA 353.1), TKN nitrogen (EPA 351.4)	MDL	P	Twelve samples were collected at 1000-foot intervals along the Haul Road between A Panel and Sage Creek where the roadway was constructed of overburden materials.	Soil samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports, QC data and raw data. The data validation reports are for metals only. Data validated in accordance with SI QAPP.	Complete lab data packages for review.	
		Terrestrial vegetation sampling was conducted to provide a more complete understanding of the extent of COPC uptake by terrestrial vegetation from soil that contains elevated concentrations of COPCs and the resultant exposure pathway to terrestrial ecological receptors.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Overburden area terrestrial vegetation	Summer 2004	F	F	ACZ	P	L47707 L47711 L47721 L47722 L47723 L47730 L47731 L47733 L47755 L47756 L47759 L47762 L47764 L47769 L47773 L47774 L47779 L47787 L47789 L47791 L47792	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), % solids (CLPSOW390, part F, D-98)	MDL	F	A total of 30 vegetation samples were collected from 30 locations along transects that cross the A Panel disposal area, Pole Canyon disposal area, D Panel backfill area and D Panel disposal area, and 26 vegetation samples were collected from 26 locations on native soils on either side of the overburden disposal areas. Vegetation samples were co-located with surface and root zone soil samples and were collected within a 100-foot by 100-foot grid.	Vegetation samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Seep area terrestrial vegetation	Summer 2004	F	F	ACZ	P	L47755 L47760 L47761 L47764 L47771 L47775 L47786	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), % solids (CLPSOW390, part F, D-98)	MDL	F	Terrestrial vegetation samples were collected from 17 locations around the five D and E Panel seep areas. Vegetation samples were collected at reference, seep-saturated and intermittently saturated sites at each of the five seep areas.	Vegetation samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Sage Valley terrestrial vegetation	Summer 2004	F	F	ACZ	P	L47774 L47776 L47780 L47788 L47793 L47795 L47796 L47797	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), % solids (CLPSOW390, part F, D-98)	MDL	F	Terrestrial vegetation samples were collected from 20 locations within northern Sage Valley at the same locations used for soil sampling.	Vegetation samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data packages for review.	
		Specific data were collected for the Baseline Ecological Risk Assessment to identify the nature and extent of COPC concentrations within site biota including riparian vegetation, aquatic biota, terrestrial insects and small mammals.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company,	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Riparian vegetation	Summer 2004	F	P	ACZ	P	L47028 L47029 L47769 L47775 L47790 L47791 L47798	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), % solids (CLPSOW390, part F, D-98)	MDL	F	Riparian vegetation samples were collected from 14 of the 15 stream riparian areas from which aquatic tissue samples were also collected. Separate samples of forbs, browse, forage and grass species were collected from multiple locations within each area.	Vegetation samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company,	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Aquatic vegetation	Summer 2004	F	N	ACZ	P	L47014	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), % moisture (EPA 209F)	MDL	F	Aquatic vegetation was collected at 15 locations in 13 reaches. Periphyton were collected from in-stream substrates at 13 locations.	Aquatic vegetation samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data package for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company,	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Aquatic insect tissue	Summer 2004	F	F	ACZ	P	L47012	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), % moisture (EPA 209F)	MDL	F	Aquatic insects were collected from identified stream segments at 12 locations.	Aquatic insect samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data package for review.	

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision

Table 2. Site Investigation Data (2003-2004)

Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group IDs	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Smoky Canyon Mine Site Investigation (continued)	MFG and NewFields for Simplot	Specific data were collected for the Baseline Ecological Risk Assessment to identify the nature and extent of COPC concentrations within site biota including riparian vegetation, aquatic biota, terrestrial insects and small mammals.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company,	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Fish tissue	Summer 2004	F	P	ACZ	P	L47030 L47031	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), % solids (CLPSOW390, part F, D-98)	MDL	F	Fish sampling was conducted at 15 locations around the site. All fish were weighed, measured and identified. Several fish from each location were retained for tissue samples analysis.	Fish tissue samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data packages for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Terrestrial insect tissue	Summer 2004	F	F	ACZ	P	L47600	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), % solids (CLPSOW390, part F, D-98)	MDL	F	Terrestrial invertebrates were trapped at 17 sites; 12 sites located along the terrestrial vegetation sampling transects and 5 sites at seep locations.	Terrestrial insect tissue samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data package for review.	
			MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	Mammal tissue	Summer 2004	F	F	ACZ	P	L47045 L47046 L47047	Cadmium, selenium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B)	MLD	F	Small mammals were trapped at 17 sites; 12 sites located along the terrestrial vegetation sampling transects and 5 sites at seep locations.	Mammal tissue samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports and QC summaries but no raw data. Data validated in accordance with SI QAPP.	Partial lab data packages for review.	
		Data were collected to re-evaluate the results from the three locations previously grab-sampled by the USFS, which were chosen based on the presence of black shale at the surface.	MFG Inc., 2003, Smoky Canyon Mine (Area A) Site Investigation Work Plan, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 22, 2003. MFG Inc., 2003. Field Sampling Plan, J.R. Simplot Company, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, August 28, 2003.	Letter Report from T. Drexel (MFG) to J. Jones, D. VandeWeg, S. Gewe, and B. Stout (USFS), Feb. 17, 2004, Fall 2003 Smoky Canyon Mine October 2003 Panel D Soil Sampling. NewFields, 2005. Final Site Investigation Report for Smoky Canyon Mine Area A, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, July 2005.	D Panel overburden area soil	Fall 2003	F	F	SVL	F	108483	Cadmium, chromium, copper, nickel, vanadium, zinc (EPA 6010B); selenium (SM 3114C), TOC (Leco), pH (ASA M9), total organic matter (H60#24), nitrate/nitrite as N (EPA 353.1), TKN nitrogen (EPA 351.4)	MDL	P	Soil samples were collected at 3 locations sampled by the USFS where black shale was present at the surface of the overburden disposal area (2003 USFS splits). Two samples were collected at each location, one grab sample and one composite sample collected from five randomly selected locations within a 100-foot by 100-foot grid.	Soil samples were not analyzed for all RI/FS target analytes. SDG 108483 contains the COC, data report, QC data and raw data. The data validation reports are for metals only. Data validated in accordance with SI QAPP.	Complete lab data package for review.	
Smoky Canyon Mine Site Investigation - USFS Splits	USFS	Data were collected to determine the concentrations of metals and selenium in soil and vegetation in E Panel seep areas.	No work plan available. Split samples collected by USFS in accordance with sampling methods described in Smoky Canyon Mine Site Investigation Field Sampling Plan.	None on file.	Soil Vegetation	Summer 2004	NP	NP	Green Analytical	NP	NP	Soil - Barium, zinc (EPA 6010B0, selenium (EPA 7742); Vegetation - Cadmium (EPA 6020), chromium, copper, nickel, vanadium, zinc (EPA 6010B), selenium (SM 3114C)	NP	NP	Soil and vegetation samples were collected at two locations near E Panel seep areas (2004 USFS splits).	Soil samples were not analyzed for all RI/FS target analytes. Data were provided by USFS in a hard copy table.	No original lab data packages for review.	
Smoky Canyon Mine Routine Water Quality Monitoring Porgram	TRC for Simplot	The Environmental Monitoring Plan for surface water and groundwater addressed the monitoring requirements under the SEIS and Panels B and C ROD and the IDEQ Consent Order Agreement.The plan also provided the data needed to identify changes in water quality associated with mining operations and assessed the effectiveness of mitigation measures for the Panels B and C operations.	MFG Inc., 2002. Draft Comprehensive Environmental Monitoring Program Plans, prepared for J.R. Simplot Company, Smoky Canyon Mine, November 2002.	J.R. Simplot Company, 2004. Annual Environmental Monitoring Report - CY2003, Smoky Canyon Mine Environmental Monitoring Program. J.R. Simplot Company, 2005. Annual Environmental Monitoring Report - CY2004, Smoky Canyon Mine Environmental Monitoring Program. J.R. Simplot Company, 2006. Annual Environmental Monitoring Report - CY2005, Smoky Canyon Mine Environmental Monitoring Program	Surface water	2003 2004 2005	F	P	SVL	F	111023 111031 112307 113661 113663 113687 116970 116971 117847 119013 119068 119121 120268	Aluminum, barium, calcium, chromium, copper, iron, magnesium, manganese, nickel, potassium, sodium, vanadium, zinc (EPA 200.7); arsenic, cadmium, lead, silver (EPA 200.8), mercury (EPA 245.1), selenium (SM 3114C), alkalinity (SM 2320), chloride, fluoride, sulfate (EPA 300.0); nitrate/nitrite (EPA 353.2), ortho-phosphate (EPA 365.2); ammonia (EPA 350.1), TSS (EPA 160.2), TDS (EPA 160.1), TOC (EPA 415.1), hardness (SM 2340B)	MDL	NP	Surface water samples were collected once/year at Upper South Fork Sage Creek, Upper Sage Creek, Upper Pole Creek, Upper Smoky Creek, Upper Tygee Creek and East Tygee Creek; twice/year at Lower South Fork Sage Creek, Lower Sage Creek, Lower Pole Creek, Lower Sage Valley, and Hoopes Spring; and three times/year at Mid-Smoky Creek, Lower Smoky Spring, Lower Smoky Creek, Upper Roberts Creek and Lower Tygee Creek.	Surface water samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports, QC data and raw data.	Complete lab data packages for review.	
			MFG Inc., 2002. Draft Comprehensive Environmental Monitoring Program Plans, prepared for J.R. Simplot Company, Smoky Canyon Mine, November 2002.	J.R. Simplot Company, 2004. Annual Environmental Monitoring Report - CY2003, Smoky Canyon Mine Environmental Monitoring Program. J.R. Simplot Company, 2005. Annual Environmental Monitoring Report - CY2004, Smoky Canyon Mine Environmental Monitoring Program. J.R. Simplot Company, 2006. Annual Environmental Monitoring Report - CY2005, Smoky Canyon Mine Environmental Monitoring Program	Groundwater	2003 2004 2005	P	P	SVL	P	107843 107844 107845 109163 109603 110941 111107 112392 113867 114532 116284 116971 117108 117625 117847 118827 119289 119484 120268	Antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, manganese, thallium (EPA 200.8); mercury (EPA 245.1); cadmium, manganese (EPA 200.8); calcium, magnesium, potassium, sodium, vanadium, zinc (EPA 200.7); selenium (SM 3114C); chloride, fluoride, sulfate (EPA 300.0); nitrate/nitrite (EPA 353.2); orthophosphate (EPA 365.2); alkalinity (SM 2320B), TDS (EPA 160.1), TSS (EPA 160 2)	MDL PQL	P	Groundwater samples were collected from the TP2 toe drain seep quarterly, the Culinary Well as specified by IDEQ, Panels B and C overburden disposal area wells quarterly, and the Consent Order well screened in the Wells Formation aquifer north of Panel C quarterly.	Field measurements are not available, therefore, the data are Level 2. Groundwater samples were not analyzed for all RI/FS target analytes. The SDGs contain the COCs, data reports, QC data and raw data. Some COCs are missing from the files. The data validation reports are for metals only.	Different levels of completeness (full and partial) for lab data packages.	

F = Full

P = Partial

NP = Not present/Not provided

NA = Not applicable

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision

Table 3. Post-Site Investigation Data (2004-2009)

Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group ID	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Panels F and G Baseline Studies (Water Resources)	Maxim Technologies and Tetra Tech	Baseline environmental data were collected to support preparation of an EIS for the Smoky Canyon Mine Manning and Deer Creek Lease Areas (Panels F & G). Biotic data collected were used to evaluate fisheries and aquatic resources including the potential for accumulation of selenium and cadmium in macroinvertebrates and fish. Surface water and groundwater data were used to develop a conceptual hydrologic model and a preliminary evaluation of water quality.	Maxim Technologies, 2003. Baseline Data Collection Plan of Study for Water Resources, Deer Creek and Manning Creek Phosphate Lease Areas, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID by Maxim Technologies, Inc., Helena, MT, April 11, 2003.	Maxim Technologies, Inc., 2004. Baseline Technical Report: Water Resources, Maning and Deer Creek Phosphate Lease Areas (Panels F & G), Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, March 12, 2004.	Surface water	2004 2005	NP	NP	SVL	NP	112996 113661 117043 117105 119609	Aluminum, barium, boron, calcium, cobalt, iron, magnesium, manganese, potassium, sodium, vanadium, zinc (EPA 200.7); antimony, arsenic, beryllium, chromium, copper, lead, nickel, selenium, silver, thallium (EPA 200.8); selenium IV and VI speciation (SM 3114B), bicarbonate as CaCO3, carbonate as CaCO3 (SM 2320B); chloride (EPA 325.2), fluoride (EPA 340.2), sulfate (EPA 375.3), nitrate as N, nitrate + nitrite as N (EPA 353.2); ammonia as N (EPA 350.1), ortho-phosphorous, total phosphorous (EPA 365.1); alkalinity (SM 2320B), hardness (SM 2340B), oil & grease (EPA 413.1), pH (EPA 150.1), specific conductivity (EPA 120.1), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	NP	The Water Resources study area included the perennial stream portions of South Fork Sage Creeek, South Fork Deer Creek, North Fork Deer Creek, main stem of Deer Creek, Manning Creek, Wells Canyon and portions of Crow Creek. The Water Resources study area included South Fork Sage Creek, Manning Creek, Deer Creek including the North and South Forks, Wells Canyon, Stewart Canyon, Diamond Creek, Crow Creek, Lamb Canyon, Clear Creek, Warm Creek and White Dugout Creek.		No original lab data packages on file for review	
					Groundwater	2004 2005	NP	NP	SVL	NP	111757 111859 113869 114099 117043 117105 117107	Aluminum, barium, boron, calcium, cobalt, iron, magnesium, manganese, potassium, sodium, vanadium, zinc (EPA 200.7); antimony, arsenic, beryllium, chromium, copper, lead, nickel, selenium, silver, thallium (EPA 200.8); selenium IV and VI speciation (SM 3114B), bicarbonate as CaCO3, carbonate as CaCO3 (SM 2320B); chloride (EPA 325.2), fluoride (EPA 340.2), sulfate (EPA 375.3), nitrate as N, nitrate + nitrite as N (EPA 353.2); ammonia as N (EPA 350.1), ortho-phosphorous, total phosphorous (EPA 365.1); alkalinity (SM 2320B), hardness (SM 2340B), oil & grease (EPA 413.1), pH (EPA 150.1), specific conductivity (EPA 120.1), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	NP			No original lab data packages on file for review	
Panels F and G Baseline Studies (Fisheries and Aquatic Resources)	Maxim Technologies and Tetra Tech	Surface water, sediment, fish tissue and aquatic insect tissue samples were collected to provide additional information on baseline selenium and cadmium levels in streams, compare selenium and cadmium levels among biotic and abiotic elements, and relate fish age with selenium and cadmium concentration.	Maxim Technologies, Inc., 2003. Plan of Study, Baseline Data Collection Plan of Study, Fisheries and Aquatic Resources, Manning Creek and Deer Creek Phosphate Lease Areas (Panels F and G), Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID by Maxim Technologies, Inc., Helena, MT, October 17, 2003.	Maxim Technologies, Inc., 2004. Baseline Technical Report: Fisheries and Aquatic Resources, Manning Creek and Deer Creek Phosphate Lease Areas (Panels F & G), Caribou County, Idaho, prepared for J.R. Simplot Company, Pocatello, ID, February 2004.	Fish tissue	2003	NP	NP	SVL	P	108095 108096	Selenium (SM 3114C), cadmium (6010)	MDL RL	NP	The Fisheries and Aquatic Resources study area included the perrential stream portions of South Fork Sage Creeek, South Fork Deer Creek, North Fork Deer Creek, main stem of Deer Creek, Manning Creek, Wells Canyon and portions of Crow Creek. The Water Resources study area included South Fork Sage Creek, Manning Creek, Deer Creek including the North and South Forks, Wells Canyon, Stewart Canyon, Diamond Creek, Crow Creek, Lamb Canyon, Clear Creek, Warm Creek and White Dugout Creek.	Fish tissue samples were not analyzed for all RI/FS target analytes. Data are also reported in Table 2 of the 2006 Second Addendum to the Baseline Technical Report.	Complete lab data packages on file with SRM tissue analyses included.	
				Maxim Technologies, 2005. Addendum to Baseline Technical Report: Fisheries and Aquatic Resources (Panels F & G), January.	Surface water	2006	NP	NP	SVL	NP	121070	Selenium (SM 3114C), cadmium (EPA 200.8)	MDL	NP	See above for description of Water Resources study area.	Surface water samples were not analyzed for all RI/FS target analytes.	No original lab data packages on file for review	
				Maxim Technologies, 2006. Second Addendum to Baseline Technical Report: Fisheries and Aquatic Resources (Panels F & G), March 2006	Sediment	2006	NP	NP	SVL	NP	121065	Selenium (SM 3114C), cadmium (EPA 200.8)	MDL	NP	See above for description of Fisheries and Aquatic Resources study area.	Sediment samples were not analyzed for all RI/FS target analytes.	No original lab data packages on file for review	
					Fish tissue	2006	NP	NP	SVL	NP	121056 121057 121059 121063 121064	Selenium (SM 3114C), cadmium (6010)	MDL	NP		Fish tissue samples were not analyzed for all RI/FS target analytes.	No original lab data packages on file for review	
					Aquatic insect tissue	2006	NP	NP	SVL	NP	121081	Selenium (SM 3114C), cadmium (6010)	MDL	NP		Aquatic insect tissue samples were not analyzed for all RI/FS target analytes.	No original lab data packages on file for review	

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision

Table 3. Post-Site Investigation Data (2004-2009)

Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group ID	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Smoky Canyon Mine Expanded Spring 2006, High-Flow, Surface Water Monitoring	Simplot and TRC	Surface water data were collected during a period of typical high-flow associated with a typical spring, snowmelt runoff period. Previous surface water monitoring conducted in support of the SI took place during relatively dry years (2002-2005).	MFG, 2003, Smoky Canyon Mine Area A, Final Site Investigation Work Plan, prepared for J.R. Simplot Company, July.	NewFields, 2006. Technical Memorandum, Water Quality Monitoring Data Report, May-June 2006, Smoky Canyon Mine Area A, prepared for J.R. Simplot Company, Pocatello, ID, July 18.	Surface water	Spring 2006	F	P	SVL	F	122512 122612 122800 122839 122875 122877 122901 122914 122950 122963 123621 124464 124677 125397	Antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, manganese, thallium (EPA 200.8); fluoride, nitrate as N, nitrite as N, sulfate (EPA 300.0); mercury (EPA 245.1), orthophosphate as P (365.2), selenium (SM 3114C), TDS (EPA 160.1), TSS (EPA 160.2)	MDL CRDL	P	Surface water samples were collected from 36 locations in Smoky Creek, Roberts Creek, Pole Canyon Creek, Sage Creek, Hoopes Spring, South Fork Sage Creek, North Fork Sage Creek and North Sage Valley, Lower Sage Valley, Crow Creek, Tygee Creek, and Panels A, C, D and E.	Surface water samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validation conducted for a subset of data packages in accordance with SI QAPP.	Complete lab data packages for review	
			MFG, 2003, Smoky Canyon Mine Area A, Site Investigation Sampling and Analysis Plan, prepared for J.R. Simplot Company, August.		Groundwater	Spring 2006	F	P	SVL	F	121925 122512 122847 122873 123261 123612 124677 125397 125565	Antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, manganese, thallium (EPA 200.8); fluoride, nitrate as N, nitrite as N, sulfate (EPA 300.0); mercury (EPA 245.1), orthophosphate as P (EPA 365.2), selenium (SM 3114C), TDS (EPA 160.1), TSS (EPA 160.2)	MDL CRDL	P	Groundwater samples were collected from 1 well in the pit backfill and overburden disposal areas; 8 wells in the shallow flow system in the Pole Canyon area, Hoopes Spring area, South Fork Sage Creek drainage, and the northern, central and southern portions of Sage Valley; 4 wells in the Wells Formation in the Pole Canyon area, Hoopes Spring area, South Fork Sage Creek drainage and the Smoky Canyon/C Panel area; and 2 water supply wells along Smoky Creek south of the mine offices. Samples were also collected from one domestic well and three springs used as domestic water supplies.	Groundwater samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validation conducted for a subset of data packages in accordance with SI QAPP.	Complete lab data packages for review	
Smoky Canyon Mine Routine Water Quality Monitoring Program	Simplot, TRC and NewFields	Data collected for the Smoky Canyon Mine Routine Water Quality Monitoring are used to identify changes in water quality resulting from overburden handling and long-term disposal and to demonstrate that surface water and groundwater quality continue to meet applicable water quality standards.	NewFields, 2006. Comprehensive Environmental Monitoring Program Plan, Smoky Canyon Mine, prepared for J.R. Simplot Company Smoky Canyon Mine, Afton, WY, revised draft October 2006.	J.R. Simplot Company, 2007, Annual Environmental Monitoring Report - CY2006, Smoky Canyon Mine Environmental Monitoring Program, 2007. NewFields. 2007a. Technical Memorandum, Water Quality Monitoring Data Report, Fall 2006 Water Quality Monitoring Data Report. Prepared for J.R. Simplot Company, January 29. NewFields. 2007b. Technical Memorandum No. 2, Evaluation of Recent Water Quality Trends at Hoopes Spring and South Fork Sage Creek Springs. Prepared for J.R. Simplot Company, February 22.	Surface water	2006 2007 2008	F	F	SVL	P	122839 122873 122875 122877 125985 126030 126056 129344 129412 129455 129456 129478 129479 131535 131546 131547 131556 131559 W802487 W802495 W802526 W802528 W8I0276 W8I0307 W8I0310 W8J0084	Barium, manganese, vanadium, zinc (EPA 200.7), arsenic, cadmium, chromium, copper, lead, nickel, silver (EPA 200.8), selenium (EPA 270.3 or 200.8), hardness (EPA 130.2), alkalinity (EPA 310.1), sulfate (EPA 375.4), nitrate as N (EPA 353.2), TDS (EPA 160.1), TSS (EPA 160.2); LT-3 only sodium (EPA 200.7), chloride (EPA 325.2)	MDL CRDL	P	Samples for routine surface water quality were collected from 22 locations in Smoky Creek, Pole Creek, Hoopes Spring, South Fork Sage Creek Spring, South Fork Sage Creek, Sage Creek, Roberts Creek, Tygee Creek, D and E Panel seeps, and tailings impoundment TP2.	Surface water samples were not analyzed for all RI/FS target analytes. Most SDGs contain COCs, data reports, QC data and raw data. Some COCs are missing.	Different levels of completeness (full and partial) for lab data packages.	
				J.R. Simplot Company, 2007, 2008, and 2009. Annual Environmental Monitoring Report - CY2006, CY2007, CY2008 Smoky Canyon Mine Environmental Monitoring Program, March-April 2007, 2008, and 2009. NewFields. 2007a. Technical Memorandum, Water Quality Monitoring Data Report, Fall 2006 Water Quality Monitoring Data Report. Prepared for J.R. Simplot Company, January 29, 2007. NewFields. 2007b. Technical Memorandum No. 2, Evaluation of Recent Water Quality Trends at Hoopes Spring and South Fork Sage Creek Springs. Prepared for J.R. Simplot Company, February 22, 2007.	Groundwater	2006 2007 2008	F	P	SVL	P	122512 122847 123621 124677 125397 125565 126084 129908 129898 129938 131838 131856 131951 131959 132009 W801857 W802983 W803502 W803648 W804381 W8I0146 W8I0147 W8I0243 W8K0018 W8J0084 W8J0530 W8L0259	Calcium, iron, magnesium, potassium, sodium (EPA 200.7), aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, silver, thallium, uranium, vanadium, zinc (EPA 200.8), mercury (EPA 245.2), chloride (EPA 325.2), sulfate (EPA 375.4), TDS (EPA 160.1), TSS (EPA 160.2), nitrate/nitrite as N (EPA 353.2), hardness (EPA 130.2), gross alpha, gross beta (SW-846 9310 Mod)	MDL CRDL	P	Samples for routine groundwater quality monitoring were collected from Wells Fm monitoring wells in lower Pole Canyon (GW-16) and near Hoopes Spring (GW-18). Sampling was performed at the Consent Order Well and the Culinary Well as part of the Panels B and C Consent Order water quality monitoring program (see below). Limited groundwater monitoring was performed at the Industrial Well (Wells Fm). Groundwater samples were also collected at GW-15 (Pole Canyon alluvial deposits), GW-17 (colluvial slope deposits), GW-21 (South Fork Sage Creek alluvial deposits), GW-22 (Sage Valley deep alluvial deposits) and GW-12 (alluvial deposits in the Tygee Creek drainage below the TP2 dam).	Groundwater samples were not analyzed for all RI/FS target analytes. Most SDGs contain COCs, data reports, QC data and raw data. Some COCs are missing.	Different levels of completeness (full and partial) for lab data packages.	
			NewFields, 2009. Comprehensive Environmental Monitoring Program Plan, Smoky Canyon Mine, prepared for J.R. Simplot Company Smoky Canyon Mine, Afton, WY, updated in April 2009.	pending	Surface water	2009	F	F	SVL	F	W9F0035 W9F0082 W9F0139 W9F0177 W9F0180 W9I0250 W9I0311 W9I0739 W9I0785 W9J0052 W9K0535 W9K0582 W9K0583	Barium, manganese, vanadium, zinc (EPA 200.7), arsenic, cadmium, chromium, copper, lead, nickel, silver (EPA 200.8), selenium (EPA 270.3 or 200.8), hardness (EPA 130.2), alkalinity (EPA 310.1), sulfate (EPA 300.0 or 375.4), nitrate+nitrite as N (EPA 353.2), TDS (EPA 160.1), TSS (EPA 160.2); LT-3 only sodium (EPA 200.7), chloride (EPA 325.2)	MDL	NP	Samples for routine surface water quality were collected from 45 locations in Crow Creek, Deer Creek, Manning Creek, North Fork Deer Creek, Hoopes Spring, overburden disposal area seeps, Pole Canyon Creek, Roberts Creek, Sage Creek, Smoky Creek, South Fork Sage Creek, South Fork Deer Creek, Stewarts Canyon, Tygee Creek, tailings ponds and Wells Canyon.	Surface water samples were not analyzed for all RI/FS target analytes.	Complete lab data packages for review	

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Smoky Canyon Mine Routine Water Quality Monitoring Program (continued)	Simplot, NewFields and Formation	Data collected for the Smoky Canyon Mine Routine Water Quality Monitoring are used to identify changes in water quality resulting from overburden handling and long-term disposal and to demonstrate that surface water and groundwater quality continue to meet applicable water quality standards.	NewFields, 2009. Comprehensive Environmental Monitoring Program Plan, Smoky Canyon Mine, prepared for J.R. Simplot Company Smoky Canyon Mine, Afton, WY, updated in April 2009.	pending	Groundwater	2009	F	F	SVL	F	W9F0082 W9F0139 W9F0177 W9J0152 W9J0313 W9J0471 W9K0579	Chromium, manganese (EPA 200.7), cadmium (EPA 200.8), zinc (EPA 200.7 or 200.8), selenium (EPA 270.3/200.8/ SM 3114C), sulfate (EPA 300.0 or 375.3), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	NP	Samples for routine groundwater quality monitoring are collected from 14 wells including GW-16, GW-18 (Wells Fm), GW-15 (Pole Canyon alluvial deposits), GW-17 (colluvial slope deposits), GW-21(South Fork Sage Creek alluvial deposits), GW-22 (Sage Valley alluvial deposits), Industrial Well (Wells Fm), Culinary Well (Rex Chert & Wells Fm), Consent Order Well (Wells Fm), MC-MW-1, DC-MW-5 (Wells Fm), MW-F1, MW-F-1A (Wells Fm near Panel F).	Groundwater samples were not analyzed for all RI/FS target analytes.	Complete lab data packages for review	
Panels B and C Consent Order Water Quality Monitoring Program (continued)	TRC Simplot	Data collected for the Panels B and C Consent Order Water Quality Monitoring Program are used to identify impacts to surface water and groundwater resources in the vicinity of Panels B and C.	MFG, 2002. IDEQ Panels B and C Consent Order Water Quality Monitoring Plan, prepared for J.R. Simplot Company Smoky Canyon Mine, Afton, WY, April 9, 2002.	J.R. Simplot Company, 2006, Annual Environmental Monitoring Report - CY2005, Smoky Canyon Mine Environmental Monitoring Program.	Surface water	2005 2006	P	P	SVL	F	119068 122875 125985 126056	Barium, chromium, manganese, nickel, vanadium, zinc (EPA 200.7), arsenic, cadmium, copper, lead, silver (EPA 200.8), selenium (EPA 270.3 or 200.8), hardness (EPA 200.7), nitrate as N (EPA 353.2), ammonia as N (EPA 350.3), orthophosphorous (EPA 365.1), sulfate (EPA 375.4), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	P	Surface water samples were collected from Upper, Middle and Lower Smoky Creek, Lower Smoky Spring, Roberts Creek, Upper and Lower Tygee Creek, East Tygee Creek and from runoff catch basins and runoff recharge areas associated with disposal of seleniferous overburden from Panels B and C.	Surface water samples were not analyzed for all RI/FS target analytes. SDGs contain the COCs, data reports, QC data and raw data.	Complete lab data packages for review	
				J.R. Simplot Company, 2007, Annual Environmental Monitoring Report - CY2006, Smoky Canyon Mine Environmental Monitoring Program, 2007.	Groundwater	2005 2006	P	P	SVL	F	119289 122512 125397	Antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, manganese, thallium (EPA 200.8); fluoride (EPA 340.2), nitrate as N, nitrite as N (EPA 353.2); mercury (EPA 245.2), orthophosphate as P (365.1), selenium (EPA 270.3 or 200.8), sulfate (EPA 375.4), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	P	Groundwater samples were collected from the Culinary Well and Consent Order Well.	Groundwater samples were not analyzed for all RI/FS target analytes. SDGs contain the COCs, data reports, QC data and raw data.	Complete lab data packages for review	
				J.R. Simplot Company, 2008, Annual Environmental Monitoring Report - CY2007, Smoky Canyon Mine Environmental Monitoring Program, April.	Surface water	2007 2008	F	F	SVL	F	129412 129455 129456 130522 131535 131782 W802487 W802495 W804105 W8K0186 W8K0189	Barium, manganese, sodium, vanadium, zinc (EPA 200.7), arsenic, cadmium, chromium, copper, lead, nickel, silver (EPA 200.8), selenium (EPA 270.3 or 200.8), alkalinity (EPA 310.1), chloride (EPA 325.2), nitrate as N (EPA 353.2), sulfate (EPA 375.4), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	P	Surface water samples were collected from Upper, Middle and Lower Smoky Creek, Lower Smoky Spring, Upper Roberts Creek, Upper and Lower Tygee Creek, East Tygee Creek and from runoff catch basins and runoff recharge areas associated with disposal of seleniferous overburden from Panels B and C.	Surface water samples were not analyzed for all RI/FS target analytes. SDGs contain the COCs, data reports, QC data and raw data. Metals are reported to the MDL. CRDLs and PQLs are also reported. Non-metals are reported to a reporting limit.	Complete lab data packages for review	
				J.R. Simplot Company, 2008, Annual Environmental Monitoring Report - CY2007, Smoky Canyon Mine Environmental Monitoring Program, April.	Groundwater	2007 2008	F	P	SVL	F	129908 131047 132009 131838 W801857 W802983 W8I0243	Antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, manganese, thallium (EPA 200.8); fluoride (EPA 340.2), nitrate as N, nitrite as N (EPA 353.2); mercury (EPA 245.2), orthophosphate as P (365.1), selenium (EPA 270.3 or 200.8), sulfate (EPA 375.4), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	P	Groundwater samples were collected from the Culinary Well and Consent Order Well.	Groundwater samples were not analyzed for all RI/FS target analytes. SDGs contain the COCs, data reports, QC data and raw data.	Complete lab data packages for review	
					Groundwater	Fall 2008	F	F	SVL	F	W8J0530 W8K0131	Selenium (SM 3114C)	MDL CRDL/PQL	F	See above.	SDGs contain the COCs, data reports, QC data and raw data.	Complete lab data packages for review	
					Groundwater	Fall 2008	F	F	SVL	F	W8K0018	Selenium (SM 3114C/EPA 200.8)	MDL CRDL/PQL	F	See above.	Samples from SDG W8J0530 reanalyzed for selenium.	Complete lab data package	
				pending, for inclusion in CY2009 Smoky Canyon Mine Environmental Monitoring Report	Surface water	2009	P	NP	SVL	F	W9C0446 W9F0082 W9H0619	Barium, chromium, manganese, vanadium, zinc (EPA 200.7), arsenic, cadmium, copper, lead, nickel, silver (EPA 200.8), selenium (EPA 270.3 or 200.8), hardness (EPA 200.7), nitrate as N (EPA 353.2), ammonia as N (EPA 350.3), orthophosphorous (EPA 365.1), sulfate (EPA 375.4), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	NP	Surface water samples were collected from Upper, Middle and Lower Smoky Creek, Lower Smoky Spring, Upper Roberts Creek, Upper and Lower Tygee Creek, East Tygee Creek and from runoff catch basins and runoff recharge areas associated with disposal of seleniferous overburden from Panels B and C.	SDGs contain the COCs, data reports, QC data and raw data.	Complete lab data packages for review	
					Groundwater	2009	P	NP	SVL	F	W9C0534 W9F0082 W9G0061 W9G0066 W9L0234 W9L0397	Antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, manganese, thallium (EPA 200.8); fluoride (EPA 340.2), nitrate as N, nitrite as N (EPA 353.2); mercury (EPA 245.2), orthophosphate as P (365.1), selenium (EPA 270.3 or 200.8), sulfate (EPA 375.4), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	NP	Groundwater samples were collected from the Culinary Well and Consent Order Well.	SDGs contain the COCs, data reports, QC data and raw data.	Complete lab data packages for review	
D and E Panel Pilot Studies	NewFields and Simplot	Data collected for the D Panel and E Panel Pilot Studies used to support the EECA by evaluating the effectiveness of chert covers on the D-P10 catch basin and the seep at the E Panel overburden disposal area.	NewFields, 2004. Pilot Study Work Plan, E-Panel External Overburden Disposal Area, Covering Seep Surface Flow Area, prepared for J.R. Simplot Company, Pocatello, ID, August 17, 2004. NewFields, 2004. Pilot Study Work Plan, D-Panel Catch Basin DP-10, Elimination of Catch Basin and Covering to Prevent Wildlife Contact with Water, prepared for J.R. Simplot Company, Pocatello, ID, August 17, 2004.	Letter Report from S. Werner, K. Tegtmeyer, and R. Rawlins, NewFields, to M. Kauffman, USFS, April 21, 2008, D-Panel Catch Basin D-P10 Pilot Study Effectiveness Monitoring - Summary of Vegetationh Sampling and Observations in October 2007. Letter Report from S. Werner, K. Tegtmeyer, and R. Rawlins, NewFields, to M. Kauffman, USFS, April 21, 2008, E-Panel External Overburden Disposal Area Pilot Study (Covering Seep Flow Area) Effectiveness Monitoring - Summary of Vegetationh Sampling and Observations in October 2007.	Vegetation	2007	F	F	CAS	F	K0709238 K0709239	Selenium (EPA 7742)	MDL MRL	NP	Vegetation samples were collected at five locations downgradient of the chert cover at catch basin D-P10 and one location downgradient of the chert covered area at E Panel.	Vegetation samples were not analyzed for all RI/FS target analytes. The SDGs are complete.	Complete lab data packages for review	
IDEQ Total Maximum Daily Loading (TMDL) Baseline Monitoring	IDEQ	Data collected as part of the IDEQ Total Maximum Daily Loading (TMDL) Baseline Monitoring used to assess water quality impacts from phosphate mining operations and help prioritize and implement remedial actions in affected streams.	None on file.	Tetra Tech EMI Inc., 2007, Water Quality Sampling for Metals - Blackfoot River and Tributaries, Selected Bear River Tributaries and Selected Salt River Tributaries, prepared for IDEQ.	Surface water	2007	NP	NP	ACZ	NP	L62586 L62587 L62588 L62589 L62591 L63019 L63024 L64142	Calcium, magnesium, zinc (EPA 200.7); cadmium, chromium, copper, lead, nickel, silver, vanadium (EPA 200.8); selenium (SM 3114B), phosphorous (EPA 365.1), alkalinity (SM 2320B), TOC (EPA 415.1), hardness (SM2340B), nitrate/nitrite (EPA 353.2), TSS (EPA 160.2)	MDL PQL	NP	Surface water samples were collected from 17 locations in the Blackfoot River drainage, 4 locations in the Bear River drainage, 4 locations in the Salt River drainage, and 1 location in the Blackfoot River downstream of the Government Dam Road Bridge.	Surface water samples were not analyzed for all RI/FS target analytes. SDGs were not provided.	No original lab data packages on file for review	

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision

Table 3. Post-Site Investigation Data (2004-2009)

Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group ID	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Site-Specific Selenium Standard (Field Studies)	NewFields and Simplot	Data used to document and evaluate the current chemical, biological and physical conditions in Hoopes Spring and locations downstream to evaluate whether or not existing selenium concentrations are adversely affecting the quality of the fish population and aquatic community. Ultimately, this field data will be used with lab data to support development of a Site-Specific Selenium Standard (SSSS) that is protective of the aquatic community.	NewFields, 2007. Final Work Plan – Field Monitoring Studies for Developing a Site-Specific Selenium Criterion, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, April 27, 2007.	NewFields and HabiTech, Inc., 2009, Final Data Report – Fall 2006 - Fall 2008 Field Monitoring Studies for Developing a Site-Specific Selenium Criterion, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, by NewFields Boulder LLC and HabiTech, Inc., Laramie, WY, May 15.	Surface water	2006 2007 2008	F	F	SVL	F	125261 125326 129269 131272 W802427 W802511 W803298 W8I0078	Selenium (SM 3114C), alkalinity (SM 2320B), phosphorous (EPA 365.2 or SM 4500PE), nitrate as N, sulfate (EPA 300.0), TOC (EPA 415.1 or SM 5310B)	Se MDL/PQL Others RL/MDL	F	Surface water, sediment, periphyton, benthic invertebrates and fish samples were collected from up to 11 locations in fall 2006, spring and fall 2007, and spring and fall 2008. Samples were collected from reference locations in South Fork Tincup Creek, several locations along Crow Creek and Deer Creek upstream of Sage Creek, Hoopes Spring, several locations along Lower Sage Creek downstream of Hoopes Spring, and several location along Crow Creek downstream of Sage Creek. Benthic community, fish population and community, and physical habitat assessments were also conducted.	Surface water samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validated in accordance wit the study's Work Plan QAPP.	Complete lab data packages for review	
					Sediment	2006 2007 2008	F	F	SVL	F	125258 129271 131271 W802422 W802512 W803676 W8I0075	Selenium (SM 3114C)	MDL PQL/CRDL	F		Sediment samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages for review	
					Aquatic vegetation	2006	F	F	ACZ	P	L58819	Selenium (EPA 6020)	MDL PQL	F		Aquatic vegetation samples were not analyzed for all RI/FS target analytes. SDG L58819 does not have raw data. Data validated in acordance with the study's work plan QAPP.	Complete lab data packages (except 1) for review	
					Aquatic vegetation	2007 2008	F	F	CAS	F	K0708093 K0710463 K0804254 K0806057 K0808686	Selenium (EPA 7742)	MDL MRL	F		Aquatic vegetation samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages for review	
					Aquatic insect tissue	2006	F	F	ACZ	P	L58821	Selenium (EPA 6020)	MDL PQL	F		Aquatic insect tissue samples were not analyzed for all RI/FS target analytes. SDG L58821 does not have raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages (except 1) for review	
					Aquatic insect tissue	2007 2008	F	F	CAS	F	K0708092 K0710642 K0804253 K0806057 K0808685	Selenium (EPA 7742)	MDL MRL	F		Aquatic insect tissue samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages for review	
					Fish tissue	2006	F	F	ACZ	P	L58844 L58845 L58846 L58847 L58848	Selenium (EPA 6020)	MDL PQL	F		Fish tissue samples were not analyzed for all RI/FS target analytes. SDG L58844 does not have raw data. The remaining SDGs are complete. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages (except 1) for review	

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Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group ID	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Site-Specific Selenium Standard (Field Studies) (continued)	NewFields and Simplot	Data were used to document and evaluate the current chemical, biological and physical conditions in Hoopes Spring and locations downstream to evaluate whether or not existing selenium concentrations are adversely affecting the quality of the fish population and aquatic community. Ultimately, this field data will be used with lab data to support development of a Site-Specific Selenium Standard (SSSS) that is protective of the aquatic community.	NewFields, 2007. Final Work Plan – Field Monitoring Studies for Developing a Site-Specific Selenium Criterion, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, April 27, 2007.	NewFields and HabiTech, Inc., 2009, Final Data Report – Fall 2006 - Fall 2008 Field Monitoring Studies for Developing a Site-Specific Selenium Criterion, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, by NewFields Boulder LLC and HabiTech, Inc., Laramie, WY, May 15.	Fish tissue	2007 2008	F	F	CAS	F	K0709398 K0709437 K0709438 K0709440 K0709442 K0709444 K0708689 K0710142 K0711040 K0711043 K0711045 K0711047 K0711049 K0711052 K0711056 K0711057 K0711062 K0711481 K0711649 K0804251 K0804339 K0807359 K0808982 K0808689 K0808692 K0808697 K0808700 K0808701 K0808703 K0808706 K0808710	Selenium (EPA 7742)	MDL MRL	F	Surface water, sediment, periphyton, benthic invertebrates and fish samples were collected from up to 11 locations in fall 2006, spring and fall 2007, and spring and fall 2008. Samples were collected from reference locations in South Fork Tincup Creek, several locations along Crow Creek and Deer Creek upstream of Sage Creek, Hoopes Spring, several locations along Lower Sage Creek downstream of Hoopes Spring, and several location along Crow Creek downstream of Sage Creek. Benthic community, fish population and community, and physical habitat assessments were also conducted.	Fish tissue samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages for review	
					Fish tissue	2008	F	F	ACZ	P	L72018	Selenium (EPA 6020)	MDL	F		Aquatic insect tissue samples were not analyzed for all RI/FS target analytes. SDG L72018 does not have raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages (except 1) for review	
					Egg tissue	2007 2008	F	F	CAS	F	K0710142 K0711481 K0712111 K0807359	Selenium (EPA 7742)	MDL MRL	F		Fish egg samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages for review	
Site-Specific Selenium Standard (Lab Studies)	NewFields for Simplot	Lab study data used to document selenium concentrations in fish and egg tissue and in media that affect the growth of trout. These data were then used to develop relationships between selenium concentrations in tissue and exposure media and to define a site-specific selenium concentration for each species evaluated. Ultimately, this lab data will be used with field data (see above) to support development of a Site-Specific Selenium Standard (SSSS) that is protective of the aquatic community.	NewFields, 2008, Revised Draft Work Plan – Laboratory Toxicity Tests for Developing a Site-Specific Selenium Threshold for Trout, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, May 9.	NewFields, 2009, Draft Final Brown Trout Laboratory Reproduction Studies Conducted in Support of Development of a Site-Specific Selenium Criterion, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, June 17.	Surface water	2007 2008	F	F	SVL	F	131784	Alkalinity (SM 2320B), sulfate (EPA 300.0), hardness by calculation, selenium (SM 3114C)	MDL MRL	F	Fish and fish egg samples were collected from reference locations in Montpelier Creek and Stump Creek, several locations along Crow Creek upstream of Sage Creek, Lower Sage Creek downstream of Hoopes Spring, and several locations along Crow Creek downstream of Sage Creek. Field parameters were measured and surface water samples were collected at these same locations.	Surface water samples were not analyzed for all RI/FS target analytes. The SDG is complete. Data were validated, however, instrument calibration was not verified.	Complete lab data packages for review	
					Fish tissue	2007 2008	F	F	CAS	F	K0710142 K0711481 K0711649 K0803855 K0804217 K0807359 K0807359	Selenium (EPA 7742)	MDL MRL	F		Fish tissue samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages for review	
					Egg tissue	2007 2008	F	Yes	CAS	F	K0710142 K0711481 K0712111 K0807359	Selenium (EPA 7742)	MDL MRL	F		Fish egg samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validated in accordance with the study's Work Plan QAPP.	Complete lab data packages for review	
Geochemical Investigation of Overburden Disposal Areas - Area-wide	MFG and TetraTech for the IPWG	Characterize conditions within overburden disposal areas at various phosphate mines in SE Idaho.	Refer to Appendix A of final (2008) Tetra Tech report.	MFG, Inc., 2005. Final Report to the Idaho Phosphate Working Group – Geochemical Review, August 5, 2005. Tetra Tech. 2008. Geochemical Characterization of Phosphate Mining Overburden, Characterization of Solids and Vapor Composition in Overburden Disposal Facilities with Implications for Weathering and Selenium Release, prepared for the Idaho Phosphate Working Group, February.	Overburden-Soil and Pore gas samples	2006	F	P	Field analyses of pore gas samples; Smith Geotechnical (grain size); ACZ (soil/overburden)	P	L56202 L56203 L56644	Selenium (EPA6020), SPLP Selenium (EPA 1312 amd 6020), AGP/ANP and sulfur forms (EPA 600/2-78-054 1.3), TOC (by combustion)	MDL PQL	NP	Smoky Canyon Mine Panel A external disposal area and Panel D pit backfill disposal area	Lab data packages included in final report appendices.	Partial lab data packages available; non-CLP methods for most parameters	
Smoky Canyon Mine Reclamation Monitoring	Simplot and Newfields	Monitor selenium uptake by vegetation in reclaimed areas.	NewFields, 2006, Smoky Canyon Mine Environmental Monitoring Program Plan, prepared for J.R. Simplot Company, Smoky Canyon Mine, Afton, WY, December 21, 2006.	Letter report to M. Johnson and A. Prouty (Simplot) from R. Rawlinson (NewFields), dated December 27, 2007, Re: Selenium analysis of vegetation tissue from A Panel reclamation area. J.R. Simplot Company, 2008, Annual Environmental Monitoring Report - CY2007, Smoky Canyon Mine Environmental Monitoring Program, April 9, 2008.	Vegetation	2007	F	F	CAS	P	K0709236 K0709237 K0709238 K0709239 K0709241	Selenium (EPA 7742), total solids	MDL MRL	NP	Vegetation samples were collected within Panel A reclamation area (associated with mining in Panels B and C). Composite samples and species specific samples were collected from sampling quadrants. Samples were also collected from non-mining-disturbed areas to establish control areas.	Vegetation samples were not analyzed for all RI/FS target analytes. SDG K0709237 does not have raw data. The remaining SDGs are complete.	Complete lab data packages (except 1) for review	

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Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group ID	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Area B Tailings Revegetation Pilot Study	TetraTech and NewFields for Simplot	Data from the Area B Tailings Revegetation Pilot Study used to determine the type, amount and depth of organic matter needed to support a plant community; evaluate the seed mixture used, and determine the plant uptake of selenium on the various treatments tested.	MFG, Inc., 2004. Draft Plan to Study Plant Uptake of Selenium from Tailings Impoundments at the Smoky Canyon Mine, prepared for J.R. Simplot Company, March 2004. NewFields, 2006, Pilot Study Work Plan, Revegetation of Tailings at the Smoky Canyon Mine, prepared for J.R. Simplot Company, March.	NewFields and Redente, E., Inc., 2005. Draft Plant Uptake of Selenium from Tailings at the Smoky Canyon Mine Tailings Impoundments, Prepared for J.R. Simplot Company, February 2005. 2008 and 2009 Annual Operations and Monitoring Reports for Tailings Impoundments (pending)	Soil	Summer 2007	F	NP	SVL	P	131122 131123	Calcium, copper, iron, magnesium, manganese, sodium, potassium, phosphorous (DTPA), selenium (SM 3114C), total organic matter (H60#24), ammonia as N (EPA 350.1), nitrate/nitrite (EPA 353.2), pH-soil (9045D); split samples analyzed for selenium (SM 3114C), TKN nitrogen (ASA M9)	MDL	NA	Soil samples were collected from each test plot before and after the soil was amended with organic matter.	Soil samples were not analyzed for all RI/FS target analytes. SDGs contain COCs, data reports and QC data but no raw data.	Partial lab data packages for review	
					Vegetation	Summer 2008 2009	F	F	CAS	F	K0806700 K0806776 K0806819 K0806821 K0906433 K0906436 K0906441 K0906445 K0906447 K0906449	Selenium (EPA 7742)	MDL	NA	Vegetation samples of each of the seeded plant species were collected from each test plot.	Vegetation samples were not analyzed for all RI/FS target analytes. All SDGs contain COC, data reports, QC data and raw data.	Complete lab data packages for review	
Pole Canyon Overburden Disposal Area Amendment and Revegetation Greenhouse Study	E. Redente (CSU) for Simplot	Laboratory study to evaluate effects of various soil/overburden amendments on selenium uptake by plants.	NewFields, 2006. Final Work Plan to Conduct a Greenhouse Study of Plant Uptake of Selenium from the Smoky Canyon Mine Pole Canyon Overburden Disposal Area, prepared for J.R. Simplot Company, September 2006.	NewFields and Redente, E., 2008. Final Greenhouse Study Report, Plant Uptake of Selenium from Overburden at the Smoky Canyon Mine, Pole Canyon Overburden Disposal Area, prepared for J.R. Simplot Company, March 2008.	Soil-Overburden	2006	NP	P	Colorado State Univ	NP	--	pH, electrical conductivity, Sodium Absorption Ratio, cation exchange capacity (CED), % Organic matter, ammonia nitrogen, nitrate nitrogen, AB-DTPA extractable P and K, Texture, total extractable selenium (refer to Soil Sample Collection for Greenhouse Studies Standard Operating Procedure, Appendix A of work plan)	NP	NP	Soil-overburden collected from surface of Pole Canyon overburden disposal area			
					Vegetation	2006 2007	NP	NP	Colorado State Univ	NP	--	Selenium (Amacher et al., 2001)	NP	NP	Greenhouse-grown vegetation in soil-overburden from Pole Canyon overburden disposal area			
Pole Canyon Removal Action Efectiveness Monitoring Program	NewFields, TRC and Simplot	Data from the Pole Canyon Effectiveness Monitoring Program are used to assess the overall effectiveness of the Removal Action in decreasing selenium transport to surface water and groundwater originating from the Pole Canyon Overburden Disposal Area. Monitoring stations are located within Pole Canyon and Sage Valley. Data were also collected to characterize the major-ion chemistry of groundwater discharged to South Fork Sage Creek and the hydraulic connectivity of existing groundwater monitoring wells.	NewFields, 2007, Revised Draft Effectiveness Monitoring Plan, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, September 21, 2007. NewFields, 2008, Draft Revision No. 1, Effectiveness Monitoring Plan, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, May 30, 2008.	NewFields, 2008, Annual Effectiveness Monitoring Report (2007) and Monitoring Well Installation Report, Pole Canyon Removal Action, Smoky Canyon Mine, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, May. NewFields, 2009, Annual Report (2008), Pole Canyon Removal Action, Effectiveness Monitoring, Smoky Canyon Mine, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, June.	Surface water	2007	P	P	SVL	P	122800 122877 122914 125985 127435 128019 128308 128774 129269 129344 129412 129455 129478 129479 129938 130600 131074 131272 131547 131559 131693 131875 131889 131961 131969	Selenium (SM 3500), sulfate (EPA 375.4), alkalinity (EPA 310.1), hardness (EPA 130.2), TDS (EPA 160.1) for all locations. Nitrate as N (EPA 353.2) and orthophosphate (EPA 365.2) for location LP-1 only. Calcium, magnesium, potassium, sodium (EPA 200.7), carbonate, bicarbonate, sulfate, chloride (EPA 300.0) for South Fork Sage Creek supplemental monitoring events.	MDL	P	Surface water samples were collected from Pole Canyon Creek above and below the Overburden Disposal Area, North Sage Creek above and below the Pole Canyon Creek confluence, Hoopes Spring, various locations along Sage Creek and South Fork Sage Creek.	Surface water samples were not analyzed for all RI/FS target analytes. Most SDGs contain COCs, data reports, QC data and raw data. Some COCs are missing. Data validation, when conducted, in accordance with EMP Section 4.0 (QAPP).	Different levels of completeness (full and partial) for lab data packages.	
					Groundwater	2007	P	F	SVL	P	129708 129898 129938 131838 131856 131951 131959	Selenium (SM 3500), sulfate (EPA 375.4), alkalinity (EPA 310.1) for all locations. Nitrate as N (EPA 353.2) and orthophosphate (EPA 365.2) for locations GW-15 and GW-23 only.	MDL	P	Groundwater samples were collected downgradient of the Overburden Disposal Area from alluvial and Wells Formation bedrock monitoring wells located along Pole Canyon Creek, North Fork Sage Creek, Sage Creek, South Fork Sage Creek and upgradient of Hoopes Spring.	Groundwater samples were not analyzed for all RI/FS analytes. Most SDGs contain data reports, QC data and raw data. Some COCs are missing. Data validation, when conducted, in accordance with EMP Section 4.0 (QAPP).	Different levels of completeness (full and partial) for lab data packages.	
					Surface water	2008	P	F	SVL	P	131990 132007 W801975 W802495 W802511 W802526 W802528 W802892 W802992 W803414 W804310 W805069 W8I0078 W8I0276 W8I0307 W8I0310 W8J0083 W8J0084 W8J0610 W8K0189 W8K0533 W8K0536 W8K0576 W8L0375	Selenium (SM 3500), sulfate (EPA 375.4), alkalinity (EPA 310.1), hardness (EPA 130.2), TDS (EPA 160.1) for all locations. Calcium, magnesium, potassium, sodium (EPA 200.7), carbonate, bicarbonate, sulfate, chloride (EPA 300.0) for South Fork Sage Creek supplemental monitoring locations.	MDL	P	Surface water samples were collected from the same locations as in 2007 except stations that were bypassed by the diversion pipeline. New stations replaced those stations along Pole Creek. Several discrete discharges near Hoopes Spring were also sampled beginning in 2008.	Surface water samples were not analyzed for all RI/FS target analytes. Most SDGs contain COCs, data reports, QC data and raw data. Some COCs are missing. Data validation, when conducted, in accordance with EMP Section 4.0 (QAPP).	Different levels of completeness (full and partial) for lab data packages.	

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision

Table 3. Post-Site Investigation Data (2004-2009)

Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group ID	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Pole Canyon Removal Action Efectiveness Monitoring Program (continued)	NewFields, TRC and Simplot	Data from the Pole Canyon Effectiveness Monitoring Program are used to assess the overall effectiveness of the Removal Action in decreasing selenium transport to surface water and groundwater originating from the Pole Canyon Overburden Disposal Area. Monitoring stations are located within Pole Canyon and Sage Valley. Data were also collected to characterize the major-ion chemistry of groundwater discharged to South Fork Sage Creek and the hydraulic connectivity of existing groundwater monitoring wells.	NewFields, 2008, Draft Revision No. 1, Effectiveness Monitoring Plan, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, May 30, 2008.	NewFields, 2009, Annual Report (2008), Pole Canyon Removal Action, Effectiveness Monitoring, Smoky Canyon Mine, prepared for J.R. Simplot Company, Smoky Canyon Mine, Pocatello, ID, June.	Groundwater	2008	F	F	SVL	F	W803502 W803648 W804381 W810146 W810147 W8J0084 W8K0495 W8L0375	Selenium (SM 3500), sulfate (EPA 300.0), alkalinity (SM 2320B), TDS (EPA 160.1) for all locations. Calcium, magnesium, potassium, sodium (EPA 200.7), sulfate, chloride (EPA 300.0), carbonate, bicarbonate (SM 2320B) for locations GW-16, GW-18, GW-23, GW-24, GW-25 and MC-MW-1 only.	MDL	F	Groundwater samples were collected from the same monitoring well locations as in 2007. Samples were also collected from one new shallow alluvial monitoring well downgradient of the Pole Canyon Overburden Disposal Area.	Groundwater samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data. Data validation conducted for a subset of data packages in accordance with SI QAPP.	Complete lab data packages for review	
				Pending	Surface water	2009	P	P	SVL	F	W9A0296 W9A0476 W9B0447 W9D0072 W9D0587 W9F0035 W9F0139 W9G0065 W9G0791 W9G0805 W9I0250 W9I0311 W9I0739 W9I0785 W9J0052 W9J0695 W9K0535 W9K0582 W9K0583	Selenium (SM 3500), sulfate (EPA 375.4), alkalinity (EPA 310.1), hardness (EPA 130.2), TDS (EPA 160.1) for all locations. Calcium, magnesium, potassium, sodium (EPA 200.7), carbonate, bicarbonate, sulfate, chloride (EPA 300.0) for South Fork Sage Creek supplemental monitoring locations.	MDL	P	Surface water samples were collected from the same locations as in 2007 except stations that were bypassed by the diversion pipeline. New stations replaced those stations along Pole Creek. Several discrete discharges near Hoopes Spring were also sampled beginning in 2008.	Surface water samples were not analyzed for all RI/FS target analytes. Most SDGs contain COCs, data reports, QC data and raw data. Some COCs are missing. Data validation, when conducted, in accordance with EMP Section 4.0 (QAPP).	Complete lab data packages for review	
				Pending	Groundwater	2009	F	P	SVL	F	W9C0446 W9D0009 W9F0035 W9F0082 W9F0139 W9F0188 W9J0152 W9J0313 W9J0471 W9J0695 W9K0579	Selenium (SM 3500), sulfate (EPA 300.0), alkalinity (SM 2320B), TDS (EPA 160.1) for all locations. Calcium, magnesium, potassium, sodium (EPA 200.7), sulfate, chloride (EPA 300.0), carbonate, bicarbonate (SM 2320B) for locations GW-16, GW-18, GW-23, GW-24, GW-25 and MC-MW-1 only.	MDL	P	Groundwater samples were collected from the same monitoring well locations as in 2007. Samples were also collected from one new shallow alluvial monitoring well downgradient of the Pole Canyon Overburden Disposal Area.	Groundwater samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data.	Complete lab data packages for review	
Panels F and G Consent Order Water Quality Monitoring Program	NewFields and Simplot	Panels F and G Consent Order Water Quality monitoring data are used to characterize background surface water and groundwater quality, identify changes in water quality relative to pre-mining conditions, and demonstrate compliance with applicable surface water and groundwater quality standards.	NewFields, 2008, Final Panels F and G IDEQ Consent Order Water Quality Monitoring Plan, prepared for J.R. Simplot Company, Pocatello, ID, January 8, 2008.	Pending, for inclusion in 2009 Annual Environmental Monitoring Report	Surface water	2009	F	NP	SVL	F	W9F0177 W9J0694	Manganese, zinc (EPA 200.7); cadmium, chromium (III) (VI), nickel (EPA 200.8); dissolved selenium (EPA 270.3 or 200.8); total recoverable selenium (EPA 3005/270.3 or 200.8); hardness (EPA 130.2); sulfate (EPA 300.0 or 375.4); TDS (EPA 160.1); TSS (EPA 160.2)	MDL	NP	Surface water samples are collected from surface seeps in overburden areas, surface runoff in overburden areas, South Fork Sage Creek, South Fork Sage Creek springs, Books Spring, Deer Creek and Crow Creek.	Surface water samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data.	Complete lab data packages for review	
			NewFields, 2009, Comprehensive Environmental Monitoring Program Plan, Draft Revision No. 1, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, April 1, 2009.	Pending, for inclusion in 2009 Annual Environmental Monitoring Report	Groundwater	2009	F	NP	SVL	F	W9F0177 W9G0244 W9H0619 W9J0614	Aluminum, barium, beryllium, chromium, manganese, zinc (EPA 200.7); antimony, arsenic, cadmium, copper, lead, thallium, zinc (EPA 200.8); mercury (EPA 245.1), selenium total and dissolved (270.3/200.8/SM 3114C), nitrate + nitrite as N (EPA 353.2); fluoride (EPA 340.2), sulfate (EPA 300.0 or 375.3), TDS (EPA 160.1), TSS (EPA 160.2)	MDL	NP	Groundwater samples are collected within and downgradient of the active mineral extraction area.	Groundwater samples were not analyzed for all RI/FS target analytes. All SDGs contain COCs, data reports, QC data and raw data.	Complete lab data packages for review	

Smoky Canyon RI/FS Preliminary Data Usability Screening Summary (data review is ongoing and table entries remain subject to revision

Table 3. Post-Site Investigation Data (2004-2009)

Project Name	Data Collected By	Data Collection Objective/ Intended Data Use	Sampling and Analysis Plan/Field Sampling Plan/Work Plans	Data Reports	Sample Matrix	Sample Date	COCs	Copies of Field Records	Analytical Laboratory	Laboratory Reports	Sample Delivery Group ID	Analytes/Methods	Detection Limits	Data Validation Reports	Description of Spatial Coverage	Comments	Data Validation or Review Status	Data Usability Level
Panels F and G Aquatic Resources Monitoring Program	Newfields for Simplot	Data collected to document and evaluate potential impacts to aquatic resources and fisheries due to mining in Panels F and G. Specific objectives included characterization of fish populations and other communities in streams of the Crow Creek drainage basin, habitat assessment, and determining concentrations of selenium in aquatic tissue.	NewFields, 2009, Comprehensive Environmental Monitoring Program Plan, Draft Revision No. 1, Smoky Canyon Mine, Caribou County, Idaho, prepared for J.R. Simplot Company, April 1, 2009.	Pending, for inclusion in Annual Environmental Monitoring Report	Surface water sediment	2009	F	F	SVL	F	W9I0596 W9I0601	Selenium (SM 3114C), percent solids (CLP SOW90, Part F, D98)	MDL	NP	Surface water and sediment samples were collected from the same locations as the aquatic and fish tissue samples (see below).	Surface water and sediment samples were not analyzed for all RI/Fs target analytes. All SDGs contain COCs, data reports, QC data and raw data. Validation to be conducted in accordance with 2009 CEMPP QAPP.	Complete lab data packages for review	
					Fish tissue	2009	F	F	ACZ	F	L78924 L78925 L78926 L79021 L79022 L79089 L79090 L79124 L79125	Selenium (EPA 3050), percent solids (CLP SOW90, Part F, D98)	MDL	NP	Fish samples were collected from Crow Creek (3 sites), Beaver Dam Creek (3 sites), Sage Creek (1 site), South Fork Sage Creek (1 site), Deer Creek (2 sites), and North fork Deer Creek (2 sites).	Fish tissue samples were not analyzed for all RI/FS target analytes. SDGs contain COCs, data reports, QC data and raw data.	Complete lab data packages for review	
					Fish tissue	2009	F	F	CAS	F	K0908877 K0908878 K0908880 K0908882 K0908884 K0908885 K0908886 K0908887 K0908888 K0908889	Selenium (EPA 7742), percent solids (CLP SOW90, Part F, D98)	MDL	NP	Fish samples were collected from Crow Creek (3 sites), Beaver Dam Creek (3 sites), Sage Creek (1 site), South Fork Sage Creek (1 site), Deer Creek (2 sites), and North fork Deer Creek (2 sites).	Fish tissue samples were not analyzed for all RI/FS target analytes. SDGs contain COCs, data reports, QC data and raw data. Validation to be conducted in accordance with 2009 CEMPP QAPP.	Complete lab data packages for review	
					Aquatic insect tissue Periphyton	2009	F	F	CAS	F	K0908881	Selenium (EPA 7742), percent solids (CLP SOW90, Part F, D98)	MDL	NP	Aquatic insect and periphyton samples were collected from the same locations as fish samples (see above).	Aquatic samples were not analyzed for all RI/FS target analytes. SDGs contain COCs, data reports, QC data and raw data. Validation to be conducted in accordance with 2009 CEMPP QAPP.	Complete lab data packages for review	
Smoky Canyon RI/FS Treatability Study - GE ABMet Biological Treatment System	NewFields and GE for Simplot	Data from this treatability test will be used to evaluate the effectiveness of the GE ABMET® treatment system in removing selenium and other COPCs from surface water and will support development and evaulation of remedial alternatives in the FS.	NewFields, 2009, Smoky Canyon Mine, Revised Pilot Study Work Plan and Sampling Analysis Plan, Biological Selenium Removal Treatment Technology - Seep DS-7, prepared for J.R. Simplot Company, Pocatello, ID, September 17, 2009.	Pilot study report is pending, for submittal to USFS and support agencies	Surface water	2009	F	NP	IAS ASC SVL	F	W9I0672 W9I0750 W9J0153 W9J0470 W9J0525 W9K0061 W9K0337	Cadmium, manganese, selenium (EPA 6020), selenite, selenate (IC-ICP-MS), organic selenium species (HPLC-ICP-DRC-MS), nitrate as N (EPA 352.1), alkalinity (SM 2320), ammonia as N (SM 4500 NH3 G), chloride (EPA 300.0), hardness (SM 2340B), nitrate/nitrite as N (EPA 353.2), phosphorous (SM 4500 PE), sulfate (EPA 300.0/375.4), TDS (SM 2540C), TOC (SM 5310B), TSS (SM 2540 D)	MDL	F	Surface water samples were collected from the influent, an intermediate point between bioreactor one and two, and the effluent. Baseline surface water quality samples were collected from the Upper Sage Creek and Lower Sage Creek stations prior to the start of treatment system operation.	Surface water samples were not analyzed for all RI/FS target analytes. Data quality review conducted in accordance with pilot study work plan.	Complete lab data packages for review	
Smoky Canyon RI/FS Treatability Study - Liberty ZVI Treatment System	NewFields and Liberty Hydrologic for Simplot	Data from this treatability study will be used to evaluate the effectiveness of the Liverty ZVI Treatment System in removing selenium from surface water and will support development and evaluation of remedial alternatives in the FS.	NewFields, 2009, Revised Smoky Canyon Mine Pilot Study Work Plan and Sampling and Analysis Plan, Zero-Valent Iron Treatment Technology-South Fork Sage Creek Springs, prepared for J.R. Simplot Company, Pocatello, ID, September 17, 2009.	Pending completion of pilot study.	Surface water	2009	F	NP	IAS ASC SVL	F	W9I0311 W9J0151 W9J0150 W9J0207 W9J0310 W9J0469 W9J0495 W9J0526 W9J0624 W9J0660 W9K0110 W9K0574 W9L0235	Alkalinity as CaCO3 (SM 2320), iron (EPA 6010B), manganese (EPA 6020), selenium (EPA 6020/SM 3114C), selenate/selenite (IC-ICP-MS), sulfate (EPA 300.0/375.3), TDS (SM 2540C0, TSS (SM 2540D)	MDL	NP	Surface water samples were collected from the combined flow of LSS-SP-N3 and LSS-SP-N4 directed to the treatment system, the discharge of each treatment unit (8 locations), the oxidation/settling pond, and the discharge from the oxidation/settling pond. Baseline water quality samples were collected from the Upper and Lower South Fork Sage Creek and two South Fork Sage Creek springs stations prior to the start of treatment system operation.	Surface water samples were not analyzed for all RI/FS target analytes. Data quality review to be conducted in accordance with pilot study work plan.	Complete lab data packages for review	

F = Full
P = Partial
NP = Not present/Not provided
NA = Not applicable

INTRODUCTION

This checklist will be used to evaluate the usability of existing, site-specific data for Remedial Investigation (RI) and Feasibility Study (FS) data uses. Completion of this checklist ultimately results in assignment of one of four RI/FS site-characterization Data Usability Levels (Levels 0, 1, 2, 3).

The Data Usability Level defines the acceptability of data for specific data uses, as explained below.

Level 0	Data are not usable for the RI/FS, which includes baseline risk assessment.
Level 1	Data are considered screening-level results. They are usable for scoping the RI/FS, but they are not usable for characterizing nature and extent of contamination, fate and transport evaluation, or baseline risk assessment.
Level 2	Data are considered usable for characterizing historical site conditions, description of site physical characteristics, historical nature and extent of contamination, fate and transport evaluation (e.g., evaluation of concentration trends over time), and engineering evaluations, but not baseline risk assessment. Level 2 data are useable for RI/FS scoping activities, including development of a preliminary conceptual site model and identification of specific data needs for the risk assessment.
Level 3	Data are considered usable for all site characterization (including characterization of existing site conditions) and engineering purposes. Level 3 data can be used as supporting information for the baseline human health risk assessment but not exposure calculations. Level 3 may be fully usable for risk assessment (Level 4 data uses) if they can be validated in accordance with the project's specifications for this data use.

A fifth data usability level has also been established. **Level 4** data meet all project specifications for data used as primary lines of evidence for baseline risk assessment. Level 3 may be fully usable for risk assessment (Level 4 data uses) if they either have been or can be validated in accordance with specifications in the RI/FS QAPP. The Level 4 data use is determined through confirmation that data validation either has been already been performed, or upon completion of data validation, in accordance with project specifications.

INSTRUCTIONS

Existing, site-specific data will be subject to review to determine usability for the Smoky Canyon Mine RI/FS. A separate review checklist may be completed for each different sample matrix in the same data set. Separate reviews may also be completed for different types of data, such as field measurements and laboratory analyses.

Fill out the information below and enter the Data Usability Level determined through this review.

Data Set ID#:

Matrix:

Parameters:

Name/Date:

DATA USABILITY LEVEL:

Appropriate Data Uses (refer to table on p. 11):

Answer the numbered questions with YES or NO and provide any requested information on this checklist and the Data Usability Screening Summary Table (template is attached).

After answering each question, follow the instructions to either assign a Data Usability Level or continue with the review. It is not necessary to complete the checklist once a Data Usability Level has been assigned.

DATA USABILITY REVIEW

1. Are there dates, locations, and measurement units associated with each data point?

   YES **NO**

   If the answer is NO, then the data are **Level 0** and not usable for the RI/FS.

   If the answer is YES, continue with review.

2. Are laboratory analysis procedures described or referenced?

   YES **NO**

3. Is there enough information available regarding sample collection to determine that the samples were collected according to standard protocols?

   YES **NO**

   If the answer to either question 2 or question 3 is NO, then the data are **Level 1** and usable as screening-type information for RI/FS scoping. Data are not usable for characterizing existing site conditions.

   If the answers to both question 2 and question 3 are YES, then continue with review and use the Data Usability Screening Summary Table to record the detailed information called for in the remaining questions.

4. Who collected the data?
   Answer here and enter information on Data Usability Screening Summary Table.

5. What uses were the data originally intended for? Why were data collected?
   Answer here and enter information on Data Usability Screening Summary Table.

6. What is/are the sampled media type(s) and when were the samples collected? Answer here and enter information on Data Usability Screening Summary Table

7. Are there planning documents or reports that provide site description, sampling design with sample locations, analytical methods and detection limit, results on per-sample basis, qualified for analytical limitations, sample quantitation limits and detection limits for non-detects, field conditions for media and environment, preliminary reports, meteorological data and field reports?

   YES **NO**

   List any planning documents, work plans, sampling and analysis plans, final reports, etc., developed for the data set here and on the Data Usability Screening Summary Table.

   If the answer is NO because planning documents and/or final reports are not available, attempt to obtain them before proceeding with this review.

   If the answer is NO because planning documents and/or final reports are not available and cannot be obtained, these data are **Level 2** data.

   If the answer is YES, enter information on Data Usability Screening Summary Table and continue with data review.

8. Are the COCs or other field sampling/custody records available to document the sample locations and the date of sampling?

   YES **YES (partial, or not provided)** **NO**

   If the answer is NO, the data are **Level 2** data.

   If the answer is YES, enter information in Data Usability Screening Summary Table and continue with the review.

   If the answer is YES (partial, or not provided) explain here and on the Data Usability Screening Summary Table and then continue with review.

9. Are SOPs that describe and specify the procedures to be followed during sampling and analysis available (i.e., procedures clearly described or documented)?

   The existence of SOPs for each process or activity involved in the data collection is preferred for data used to characterize site conditions.

   YES **NO**

   If the answer is NO, there is a lower probability that data collection was properly and consistently implemented. Continue with review.

   If the answer is YES, there is a higher probability that data collection was properly and consistently implemented. Continue with review.

10. Are field records available that document the procedures followed and the conditions of the procedures?

    Field records, such as field logs and instrument calibration records, may be useful to data users as back-up documentation, but they are not minimum requirements for all site characterization data uses.

    YES **YES (partial, or not provided)** **NO**

    If the answer is NO, the data are **Level 2** data.

    If the answer is YES, enter information in Data Usability Screening Summary Table and continue with the review.

    If the answer is YES (partial, or not provided) explain here and on the Data Usability Screening Summary Table and then continue with review.

11. What laboratory analyzed the samples and what parameters were analyzed and reported?
    Answer here and enter information on Data Usability Screening Summary Table.

12. Were routine/standardized/federally documented methods used for analyses of chemicals of potential concern (refer to attached table of COPCs)?

    List laboratory analysis methods used here and on the Data Usability Screening Summary Table.

    YES **NO**

    If the answer is NO, the data may not be comparable to other data/results (from separate sources) for the same parameters, and the data are **Level 2**.

    If the answer is YES, continue with review.

13. Were EPA CLP methods used for analyses of COPCs?

    YES **NO**

    If the answer is NO, identify the non-CLP methods listed under question 12 and enter information on Data Usability Screening Summary Table; continue with review.

    If the answer is YES, enter information on Data Usability Screening Table; continue with review.

14. Are the data reported to the Method Detection Limits (MDLs)? Are the data reported to a quantitation limit (e.g., PQL, RL, etc.)?

    YES **NO**

    If the answer is NO to both questions, the detection and/or quantitation limits for "non-detect" results cannot be confirmed and the data are **Level 2**.

    If YES to either question, list the type of limit reported to (i.e. reporting limit, practical quantitation limit) and whether the MDLs are provided, and enter information on Data Usability Screening Summary Table; continue with review.

15. Does the laboratory data package contain sufficient QA/QC information to perform the following review:

    a. Evaluation of data completeness
    b. Verification of instrument calibration
    c. Measurement of laboratory precision using duplicates; measurement of laboratory accuracy using spikes
    d. Examination of laboratory blanks for contamination
    e. Assessment of adherence to method specifications and QC limits
    f. Evaluation of method performance in the sample matrix.

    YES **YES (partial)** **NO** **NOT KNOWN**

    If the answer is YES, enter information on the Data Usability Screening Summary Table; continue to question 16.

    If the answer is YES (partial), explain here and enter information on the Data Usability Screening Summary Table; continue to question 16.

    If the answer is NO the data are **Level 2**.

    If the answer is NOT KNOWN; continue to question 16.

16. If the data packages contain the QA/QC information for the items listed above (a through f), have the data packages been reviewed for those items according to a specified plan or known procedure (i.e., described in final data report)?

    YES **YES (partial)** **NO**

    If the answer to question 15 is NOT KNOWN and the answer to question 16 is NO, then the quality of the data cannot be confirmed and the data are **Level 2**.

    If the answer to question 15 is YES or YES (partial) and the answer to 16 is YES (partial) or NO, the data package contains sufficient QA/QC information that would allow review to confirm data quality, but the data have either not been reviewed or additional review may be needed. Note here and on the Data Usability Screening Summary Table the extent of data quality review performed (if any), and identify any elements from question 15 that have not been reviewed.

    Data are **Level 3a – data quality not reviewed**. Additional data quality review may be necessary for site characterization data uses.

    If the answer is YES to both questions 15 and 16, note the type of data quality review and/or data validation performed here and on the Data Usability Screening Summary Table; continue to question 17.

17. Have data been validated in accordance with a specified validation protocol?

    YES **NO**

    If the answer is NO, the data are **Level 3b – reviewed but not validated**.

    If the answer is YES, the data are **Level 3c – reviewed and validated**. Describe the data validation protocol used here and enter on the Data Usability Screening Summary Table.

Level 3 data may be usable for quantitative baseline risk assessment (i.e., primary lines of evidence for exposure calculations) if, or when, they have been validated in accordance with the project-specified validation protocols and they meet the project's data quality objectives for risk assessment data uses.

Enter final data usability level determination and identify appropriate data uses on the Data Usability Screening Summary Table.

Smoky Canyon Mine RI/FS Data Usability Levels and Appropriate Data Uses

Data Usability Level	**Appropriate Data Uses**
Level 0	Not usable for RI/FS
Level 1	RI/FS scoping
Level 2	Development of preliminary conceptual site model; evaluation of temporal or spatial trends; identification of site characterization and risk assessment data needs.
Level 3a (data quality not reviewed)	Limited to Level 2 data uses until data quality review completed; available for Level 3b data uses after data quality review completed.
Level 3b (reviewed but not validated)	Characterization of nature and extent of contamination, source characterization, identification of transport pathways. Usable for baseline risk assessment upon completion of project-specified validation.
Level 3c (reviewed and validated)	Same as Level 3b.
Level 4 = Level 3 data that have been validated in accordance with project validation protocols and meet project DQOs for risk assessment data uses.	Same as Level 3b and also baseline risk assessment (primary lines of evidence).