

FIELD SAMPLING PLAN

**Stehekin and Newhalem Firing Ranges
North Cascades National Park Complex
Stehekin and Newhalem, Washington**

P13PD01436

**PART I – FIELD SAMPLING PLAN
PART II – QUALITY ASSURANCE PROJECT PLAN**

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1.0 INTRODUCTION

This document serves as the Sampling and Analysis Plan (SAP) for an Engineering Evaluation/ Cost Analysis (EE/CA) at the Stehekin and Newhalem Firing Ranges located in North Cascades National Park (Sites). This SAP is intended to apply to investigational activities taking place at the Sites during an EE/CA conducted by the National Park Service (NPS).

This SAP has been prepared as a guide for sampling associated with Site characterization as required to complete the EE/CA. All sampling will be conducted following this SAP.

As presented in Section 2.3 There has been one previous investigation conducted at each Site.

Figures 1.0 and 2.0 shows the geographic locations of the Sites.

The SAP is comprised of the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPP) and includes the following sections:

Section 1 - Introduction
Section 2 - Site Background

Part I: Field Sampling Plan

Section 3 - Sampling Program, Rationale and Locations
Section 4 - Field Methods and Procedures

Part II: Quality Assurance Project Plan

Section 5 - Project Management
Section 6 - Quality Control Requirements
Section 7 - Assessment and Oversight
Section 8 - Data Validation and Usability
Section 9 - Measurement and Data Acquisition
Section 10 - References

Appendix A - Standard Operating Procedures
Appendix B - Laboratory QA/QC Documentation (available upon request)

1.1 Objectives

This SAP describes the collection and analysis of soil samples to be performed in the field concurrently with investigational activities taking place at the Site during an EE/CA. The sampling effort will provide the following data to complete an EE/CA:

Site Investigation

- Near surface soil samples will be collected to determine lead concentrations in soil.
- Samples will be collected in the area surrounding the firing range. Sample locations will

- be field-fit as required to determine the extents of lead impacts.
- Soil samples will be collected outside of the firing range area to determine background lead concentrations.
- Soil samples will be collected according to Multi-Increment (MI) sample collection methodologies.

Section 3.1 further explains how data will be used.

1.2 Project Schedule

Data collection will occur during site characterization which is expected to be completed in one visit per Site. The data will be presented in the EE/CA Report.

2.0 SITE BACKGROUND

Site background information was presented Preliminary Assessment for each Site (Kleinfelder 2003a and 2003b). The study area, Site history, previous Site investigations and environmental setting are summarized below.

2.1 Study Area and Environmental Setting

The Stehekin Site is located within Lake Chelan National Recreation Area in Chelan County, Washington, in North Cascades National Park Complex. The town of Stehekin lies at the head of Lake Chelan, approximately 55 miles by boat from Chelan, Washington, and the area is accessible only by boat, plane, or hiking through extensive wilderness. The firing range is located at the end of a 300' access road diverging from the Stehekin Valley Road approximately 7.5 miles from the Stehekin Landing. The range is in a forested area on the extreme lower flank of Rainbow Mountain above McGregor Meadows. About 25 yards in length with four target boards, the shooting range is located in an old borrow pit. The target area, backed by a low soil berm or the foot of a cut bank to the north, is approximately 50' wide. Both the east and west sides of the firing range are contained by soil berms.

The Newhalem Site is located within Ross Lake National Recreation Area, in Whatcom County, Washington, in North Cascades National Park Complex. The firing range is located approximately 2,000 feet northwest of the Skagit River (at river mile 92) to the north of Washington State Route 20. The firing range is located in an opening in a densely forested area of the valley, off the dirt access road that cuts perpendicular to a power line and is closed to the public by a locked gate.

2.2 Site History

Various types of small arms were believed to have been used at both Sites.

The Stehekin range was used by valley residents for sighting in-hunting rifles. The range was also used for NPS personnel for small arms proficiency practice. The primary weapons fired at the range are rifles (Kleinfelder, 2003a).

The Newhalem range was originally used by residents of Newhalem before the formation of the North Cascades National Park Complex. The Newhalem range was used by NPS personnel and Newhalem residents. The range was also used for NPS personnel for small arms proficiency practice. The primary weapons fired at the range were rifles and handguns (Kleinfelder, 2003b).

2.3 Previous Site Investigations

There has been one previous investigation conducted at each Site as follows:

- Preliminary Assessment of Two Sites, North Cascades NPS Complex , Stehekin, Washington, prepared by Kleinfelder, 2003a.
- Preliminary Assessment of Firing Range, North Cascades NPS Complex , Newhalem, Washington, prepared by Kleinfelder, 2003b.

The results of both investigations stated that that existing data and information do not indicate a threat to human health, the surrounding environment, or local wildlife (Kleinfelder, 2003a and 2003b). No data was collected at the firing ranges.

PART I: FIELD SAMPLING PLAN

3.0 SAMPLING PROGRAM, RATIONALE AND LOCATIONS

The Field Sampling Plan (FSP) for this investigation has been developed to provide guidance for sampling during investigatory activities associated with the EE/CA.

3.1 Experimental Design and Sampling Rationale

The general objective of this sampling effort is collect sufficient data to conduct the EE/CA.

3.1.1 Sampling and Decision Units

MI sampling is based on dividing the project area into a series of Sampling and Decision Units (SU and DU respectively). A Sampling Unit (SU) also sometimes identified as a Decision Unit (DU), is the area and depth of soil (the sampled population) to be characterized by the average concentration of the MI sample. A DU may contain one or more separate SUs that are sampled using MI techniques. SUs must be restricted to actual source zones and must incorporate only areas that are similar as far as impacts as to not dilute contamination. SUs/DUs selected based on future land use scenarios may also be referred to as Exposure Units.

SUs must be delineated so that the mean analyte concentrations obtained are directly relevant to well defined project and or/risk objectives. They are the smallest volume of soil for which a concentration value will be obtained, and the basic unit about which a decision or conclusion based on an analytical result can be made.

A DU is a specific area (or volume of soil) about which a decision is to be made. In the ideal and most direct case, the DU and SU are the same volume of soil. As noted above, a DU may be

composed of a single SU, or may include multiple SUs, if the DU is very large in size. The critical concern is that the entire area of a DU is consistent as far as contamination distribution and future use/exposure scenario, as with an SU.

3.1.2 Multi Increment (MI) Sample Collection

MI combines many small increments of soil from a large number of random sampling points across a defined exposure area. It differs from typical composite sampling in two ways: the number of grabs (increments) is much greater and each MI sample represents an entire area of interest or decision unit. Theoretically, MI mitigates single sample variability that results from discrete sampling or composites with limited increments. An approximate 0.5 to 1 kg sample is sent to the laboratory for processing in its entirety to help address compositional and distributional heterogeneity. MI sample replicates are typically normally distributed. A series of equivalent (by weight and/or volume) aliquots are collected for each DU to maintain a desirable mass of about 1 kg. A minimum of thirty aliquots will be collected from each DU. Each aliquot will consist of a minimum of 20 grams. Aliquots will be collected at an approximate depth of zero to two-inches below the vegetative mat.

3.2 Sample Media and Parameters

All sampling described below is required to achieve the project objectives. The focus of sample collection activities proposed in this SAP is evaluation of the environmental Site media described in Section 3.3. Table 3.0 summarizes the sample media and parameters to be measured.

3.3 Soil Sampling Locations

Sampling locations will be field-fit as required to characterize the Site. Samples will be based on Decision Units (DUs) to be determined at the beginning of sampling events for each of the Sites. DUs are described further in Section 3.1.1. DUs will be determined for both up and down-range areas of the Sites. Background samples will be collected in the vicinity of each Site outside of any potentially impacted areas.

4.0 FIELD METHODS AND PROCEDURES

The following field methods and procedures will be used during this project (see Section 5.7 for laboratory analytical methods):

- Site Mobilization;
- Mobilization of Equipment, Supplies and Containers;
- Equipment Decontamination; and
- Field Sample Collection.
 - Soil Sampling

Referenced Standard Operating Procedures (SOPs) are included in Appendix A.

4.1 Site Mobilization

RMC will identify and provide all necessary personnel, equipment and materials for mobilization and demobilization to and from the Site to collect samples. Equipment mobilization includes ordering and purchasing equipment and supplies. A complete inventory of available equipment and supplies will be conducted prior to the start of sampling.

4.2 Equipment, Supplies and Containers

Equipment and supplies necessary for field sampling are summarized in Table 4.0. This table separates field items into the following categories: sampling, health and safety, equipment and personal decontamination and general field operations.

The primary sample containers for this project will be new, polyethylene bags.

4.3 Equipment Decontamination

All non-dedicated and non-disposable sampling equipment will be decontaminated prior to use at each SU/DU and between media types. Equipment decontamination procedures outlined in the SOP, *Standard Procedures for Sampling Equipment Decontamination* (RMC SOP 6, provided in Appendix A) will be used in this sampling program. Equipment will be decontaminated by placing the sampling equipment in a bucket filled with deionized (DI) water and non-phosphate soap and removing any visible residual material from the sampling equipment with a brush. Any residual soap or debris will be removed by pouring DI water over the equipment. Sampling equipment will then be double rinsed with DI water. Upon completion of this procedure, all equipment will be air dried and stored in a “clean” vessel or wrapped with foil until ready for use. Disposable “one-use” sampling equipment will be used whenever possible.

4.4 Field Sampling and Data Collection

Table 4.1 provides a summary of the analyses that will be conducted during the EE/CA. The sample volumes and containers and preservation requirements for these samples are specified in the QAPP (Part II). Samples for chemical analysis will be identified as follows:

- Samples collected at Stehekin will be identified with a ST identifier;
- Samples collected at Newhalem will be identified with a NE identifier;
- Background samples will be identified with a BG identifier;
- Each sample will contain a DU identifier.

The methods that will be used to collect the samples are discussed below.

4.4.1 Soil Sampling

Soil sampling will be conducted to determine concentrations of lead as required. Soil samples will be collected according to the MI sampling protocols discussed in Section 3.1.2 and the following Standard Operating Procedures presented in Appendix A:

4.4.2 Investigation-Derived Waste

Investigation-derived waste (IDW) generated during this study will be handled in accordance with OSWER Directive 9345.3-02 *Management of Investigation-Derived Wastes During Site Inspections* (EPA, 1991). Collecting only the volume of material needed to satisfy laboratory analytical requirements will minimize the generation of IDW. Any excess material will be discarded at the sample collection point.

4.5 Sample Alteration Form

Changes to sample collection methodologies, procedures, equipment or parameters will be documented on a Sample Alteration Form (Table 4.2). The Sample Alteration Form will be included when reporting applicable results.

PART II: QUALITY ASSURANCE PROJECT PLAN

5.0 PROJECT MANAGEMENT

The QAPP for the former Stehekin and Newhalem Firing Ranges EE/CA has been developed in accordance with EPA QA/R-5 guidance for preparing QAPPs (EPA, 2001). This section covers the basic area of project management, including the project organization, background and purpose, project description, quality objectives and criteria, special training, documentation and records.

5.1 Project Organization

Organization and responsibilities specific to this investigation are discussed in this section. Laboratory services will be provided by ALS Laboratories (ALS), located in Kelso, Washington. ALS is a State of Washington-approved laboratory, which will analyze the samples for lead.

For this data collection effort, key management personnel are as follows:

<u>Individual</u>	<u>Role/Responsibility</u>
Todd Leeds	Project Manager

The field management team will be determined prior to arrival at the Sites.

The NPS Project Coordinator is Kerri Cook.

The Project Manager will be responsible for the overall management and coordination of the following:

- Coordination with NPS regarding the status of the project;
- Providing oversight of the subcontractors;
- Preparing status reports;
- Supervising production and review of deliverables;
- Tracking work progress against planned budgets and schedules;
- Informing NPS of changes in the EE/CA and/or other project documents;
- Notifying NPS immediately of significant problems affecting the quality of data or the ability to meet project objectives;
- Procuring subcontractors to provide sampling and analytical support;
- Providing oversight of report preparation;
- Organizing and conducting a field planning meeting;
- Coordinating with the laboratory regarding the analytical, data validation and Quality Assurance/Quality Control (QA/QC) issues related to sample analysis;
- Reviewing analytical results and deliverables from subcontractors;
- Incorporating changes in the EE/CA and/or other project documents;
- Scheduling personnel and material resources;
- Implementing field aspects of the EE/CA, including this SAP and other project documents;
- Implementing the QC measures specified in the QAPP in this and other project documents;
- Implementing corrective actions resulting from staff observations, QA/QC surveillance and/or QA audits;
- Providing oversight of data management;
- Coordinating and overseeing the efforts of the subcontractors providing sampling and analytical support;
- Scheduling and conducting field work;
- Notifying the analytical laboratory of scheduled sample shipments and coordinating work activities;
- Gathering sampling equipment and field logbooks and confirming required sample containers and preservatives;
- Maintaining proper chain-of-custody forms and shipping of samples to the analytical laboratory during sampling events;
- Ensuring that sampling is conducted in accordance with procedures detailed in this SAP and that the quantity and location of all samples meet the requirements of the SAP; and
- Identifying problems at the field team level, resolving difficulties in consultation with the QA/QC staff, implementing and documenting corrective action procedures at the field team level and providing communication between the field team and NPS management.

The roles and responsibilities of other field team members will be to assist the Project Manager with sampling activities, sample handling and overall documentation.

5.2 Quality Assurance/Quality Control Organization

The Project Manager or designated representative, will be responsible for the Quality Assurance/Quality Control of the data that are generated during implementation of the SAP. The Project Manager will be responsible for the following:

- Reviewing and approving project specific plans;

- Directing the overall project QA/QC program;
- Maintaining QA/QC oversight of the project;
- Reviewing QA/QC sections in project reports, as applicable;
- Reviewing QA/QC procedures applicable to this SAP;
- Auditing selected activities of this project, as necessary;
- Initiating, reviewing and following up on response actions to address QA/QC problems, as necessary;
- Consulting with the Project Coordinator, as needed, on appropriate QA/QC measures and corrective actions;
- Arranging performance audits of measurement activities, as necessary; and
- Providing written reports on QA/QC activity to the Project Manager.

5.3 Background and Purpose

Site background information for the former Stehekin and Newhalem Firing Ranges is provided in Section 2.0 of this SAP. The purpose and objectives of the work assignment are discussed in Section 1.1 of this SAP. The purpose of this QAPP is to provide guidance to ensure that all environmentally related data collection procedures and measurements are scientifically sound and of known, acceptable and documented quality conducted in accordance with the requirements of the project.

5.4 Project Description

The QAPP addresses field work, data collection and laboratory analyses performed for this work assignment. Detailed project descriptions are outlined in the FSP sections above.

5.5 Data Quality Objectives (DQOs) and Criteria for Measurement

This section provides internal means for control and review so that environmentally-related measurements and data collected in this study are of known quality. The subsections below describe the DQOs (Section 5.5.1) and data measurement objectives (Section 5.5.2).

5.5.1 Data Quality Objectives

The DQO process is a series of planning steps based on the scientific method that are designed to ensure that the type, quantity and quality of environmental data used in decision-making are appropriate for the intended purpose. The EPA has issued guidelines to help data users develop site-specific DQOs (EPA, 1994b). The DQO process is intended to:

- Clarify the study objective;
- Define the most appropriate type of data to collect;
- Determine the most appropriate conditions from which to collect the data; and
- Specify acceptable levels of decision errors that will be used as the basis for establishing the quantity and quality of data needed to support the design.

The goal of the DQO process is to help ensure that data of sufficient quality are obtained to support removal response decisions, reduce overall costs of data sampling and analysis activities

and accelerate project planning and implementation. Data Quality Objectives are summarized in Table 5.0.

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified. The DQO process consists of seven steps, of which the output from each step influences the choices that will be made later in the process. These steps include:

- Step 1: State the problem;
- Step 2: Identify the decision;
- Step 3: Identify the inputs to the decision;
- Step 4: Define the study boundaries;
- Step 5: Develop a decision rule;
- Step 6: Specify tolerable limits on decision errors; and
- Step 7: Optimize the design.

During the first six steps of the process, the planning team develops decision performance criteria (DQOs) that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the DQOs. A brief discussion of these steps and their application to this project is provided below.

Step 1: State the Problem

The purpose of this step is to describe the problem to be studied so that the focus of the study will be unambiguous. The sampling specified in this SAP will be conducted to provide Site-specific data to confirm the appropriate action for the Sites..

Step 2: Identify the Decision

This step identifies what questions the study will attempt to resolve and what actions may result. The study will determine the overall extents of lead concentrations in soils and screen this data in comparison to background conditions and State of Washington Model Toxics Control Act (MTCA, 173-340 WAC) Method A regulatory cleanup levels which are summarized as follows:

- Human Health – Unrestricted use: 250 parts per million
- Human Health – Industrial use: 1000 ppm
- Ecological Health – Plants: 50 ppm
- Ecological Health – Soil Biota: 500 ppm
- Ecological Health – Wildlife: 118 ppm

Step 3: Identify the Inputs to the Decision

The purpose of this step is to identify the information that needs to be obtained and the measurements that need to be taken to resolve the decision statement. Based on the study questions, the following information is required:

- Lead concentrations in Site soils.

Step 4: Define the Boundaries of the Project

This step defines the spatial boundaries of the project. The entire project will be field-fit and performed within the area of the former Stehekin and Newhalem Firing Ranges. The DUs will be determined based on an initial assessment of the configuration of each range. The size of each decision unit will conform to recommendations of 25 to 10,000 square meters (e.g. up to 2.5 acres) as described by the United States Army Corps of Engineers (USACOE, 2009). Typical depths for each DU will be zero to two-inches below the vegetative mat.

Step 5: Develop a Decision Rule

The EE/CA decision process consists of the following steps:

- 1) Decide if the data collected is sufficient to complete the EE/CA;
- 2) Compare sample results with regulatory guidelines (see Step 2, above).

Step 7: Optimize the Design for Obtaining Data

This step identifies a resource-effective data collection design for generating data that are expected to satisfy the DQOs. The data collection design (sampling program) is described in detail in the FSP (Part 1 of this SAP).

5.5.2 Data Measurement Objectives

Based on the information provided on the DQOs, all analytical samples will be analyzed using EPA methods and other standard analytical techniques. Every reasonable attempt will be made to obtain a complete set of usable analytical data. If a measurement cannot be obtained or is unusable for any reason, the effect of the missing data will be evaluated by the Project Manager. Table 4.1 summarizes the analytical methods and data measurement objectives for analyses that will be conducted in the field investigations.

5.5.3 Quality Assurance Guidance

The field QA program has been designed in accordance with EPA's *Guidance for the Data Quality Objectives Process* (EPA, 1994b) and the EPA's *Requirements for Quality Assurance Project Plans for Environmental Data Operations* (EPA, 1997).

5.5.4 Precision, Accuracy, Representativeness, Completeness and Comparability Criteria

Precision, Accuracy, Representativeness, Completeness and Comparability (PARCC) parameters are indicators of data quality. PARCC goals are established for the Site characterization to aid in assessing data quality, as discussed in the following paragraphs:

Precision. The precision of a measurement is an expression of mutual agreement among individual measurements of the same property taken under prescribed similar conditions. Various measures of precision exist, depending upon “prescribed similar conditions.” As a rule of thumb, a program should strive to achieve a field sampling variance for triplicate DU samples of less than 50% relative standard deviation (RSD), and preferably 30% RSD, and laboratory subsampling variance should be less than 20% RSD, and preferably 10% RSD.

Accuracy. Accuracy is the degree of agreement of a measurement with an accepted reference or true value and is a measure of the bias in a system. Accuracy is quantitative and usually expressed as the percent recovery (%R) of a sample result. Ideally, it is desirable that the reported concentration equals the actual concentration present in the sample. Acceptable QC limits for %Recovery (R) are 75% to 125% for Laboratory Control Sample/ Laboratory Control Sample Duplicates (LCS/LCSDs), method-defined for surrogates and laboratory-defined for Matrix Spikes/Matrix Spikes Duplicates (MS/MSDs). Chemical analytical data will be validated for accuracy using surrogates, MS/MSDs and LCS/LCSDs, as applicable.

Representativeness. Representativeness expresses the degree to which sample data accurately and precisely represent; (a) a characteristic of a population; (b) parameter variations at a sampling point; and/or (c) an environmental condition. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling plan and the absence of cross-contamination. Good Representativeness will be achieved through: (a) careful, informed selection of sampling sites; (b) selection of testing parameters and methods that adequately define and characterize the extent of possible contamination and meet the required parameter reporting limits; (c) proper gathering and handling of samples to avoid interference and prevent contamination and loss; and (d) collection of a sufficient number of samples to allow characterization. Representativeness is a consideration that will be employed during all sample location and collection efforts and will be assessed qualitatively by reviewing field procedures and reviewing actual sampling locations versus planned locations.

Completeness. Completeness is a measure of the amount of usable data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Evaluating the PARCC parameters will assess usability. Those data that are validated and need no qualification, or are qualified as estimated data, are considered usable. Rejected data are not considered usable. Completeness will be calculated following data evaluation. For this work, a completeness goal of ninety-percent is projected for each analytical test. If this goal is not met, additional sampling may be necessary to adequately achieve project objectives.

Comparability. Consistency in the acquisition, handling and analysis of samples is necessary for comparing results. Where appropriate, the results of analyses obtained will be compared with the results obtained in previous studies. The data collected during the EE/CA is the first round of sampling at the Sites. Hence, there are no previous studies/data for comparability. For the purposes of this sampling event, comparability will be limited to consistency with the methodologies used in sample collection and

analysis such as standard EPA analytical and QC methods. Comparability is a qualitative parameter and cannot be assessed using QC samples.

5.6 Field Measurements

Field measurements will be limited to the measurement and delineation of SUs and DUs.

5.7 Laboratory Analytical Methods

Analytical methods with corresponding laboratory reporting limits (LRLs) are specified on Table 4.1. Laboratories with established protocols and quality assurance procedures that meet or exceed applicable EPA guidelines will analyze samples by following these methods. Samples will be analyzed using EPA-approved or recommended methods when available and will include all associated QA/QC procedures recommended in each method.

Samples will be submitted to ALS Laboratories, certified with the State of Washington. The laboratory will:

- Demonstrate ability to achieve the required detection limits,
- Be certified by the State of Washington; and
- Have an established internal QA/QC program.

If contradictions between the laboratory QA/QC manuals or other documents are identified, information in this SAP supersedes all other documents.

5.7.1 Soil

Soil will be analyzed for lead as specified in Table 4.1.

6.0 QUALITY CONTROL REQUIREMENTS

Triplicate samples must be collected in order to verify that an MI sample truly represents the decision unit. The collection of triplicate samples allows for the calculation of relative standard deviation (RSD). This is markedly different from the typical duplicate sample that is collected from the same material as the primary sample. A minimum of one triplicate set is required for all MI sampling projects.

Quality control will include collecting 1 triplicate sample for every 10 DUs at each Site. The triplicates will be collected at an offset of each aliquot location. The offset distance will be determined based on the size of the DU and aliquot spacing. The offset spacing will be configured to represent two additional, distinctive sample sets. This will ensure that each of the triplicate sample aliquots do not coincide with another aliquot location. The triplicates will be submitted "blind" to the sample laboratory, i.e., they will be given a separate sample identification number from the environmental sample, unidentifiable to the laboratory, as described above. Triplicates will be run for the same analytical suite as the environmental samples. Triplicate samples will be identified using the suffix T1, T2 and T3.

One sample from each Site will be randomly selected for duplicate tests. Splits of these four samples will be created in the laboratory and identified as duplicates by adding the suffix “-D”

Samples for preparation of matrix spikes and laboratory duplicates will be selected at random by the laboratory. Separate samples do not need to be collected in the field. The laboratory will perform and report all analyses under QA/QC procedures that include the results of method blanks, laboratory control samples, matrix spikes and laboratory duplicates. Additional method-specific quality control procedures such as interference check samples, serial dilution and internal standards will be used as specified for each analytical method in SW-846 (U.S. EPA 2003).

Due to the nature of the contaminants at this Site, ambient, equipment and trip blanks will not be collected.

6.1 Instrument/Equipment Testing, Inspection and Maintenance Requirements

All instruments and equipment will be regularly tested, inspected and maintained according to manufacturers’ instructions. Field equipment will be tested and inspected daily before use. Any equipment found to not be functioning properly will be repaired or replaced. Laboratory equipment will be tested, inspected and maintained in accordance with the laboratory QA/QC manual and manufacturers’ recommendations.

6.2 Instrument Calibration & Frequency

6.2.1 Field Instruments

Site personnel will follow the manufacturer's specifications to calibrate any field equipment prior to each day. These manufacturers’ specifications are included in RMC’s SOPs (Appendix A).

6.2.2 Laboratory Equipment

Procedures and schedules for the calibration of laboratory equipment are described in the appropriate SW-846 and EPA methods and in the laboratory’s Quality Assurance Plan. These procedures and schedules will be followed for all laboratory work.

6.3 Data Management

Data will be submitted to the Project Manager in both hard copy and electronic form. To avoid transcription errors, report tables will be prepared directly from the electronic submittals.

7.0 ASSESSMENT / OVERSIGHT

This section describes the number, frequency and type of assessment activities needed for this project. Assessments coordinated by the Project QA Officer will include: (1) a readiness review prior to initiating each major portion of field work; and (2) a data quality assessment (DQA).

The readiness review will be conducted with both the field staff and analytical laboratories as a technical check to determine if the staff, subcontractors, equipment and record keeping system are in place to start work in accordance with this QAPP. At the review, the QA Officer will

review the project objectives, methodologies, record keeping requirements and schedule with the field team and laboratories to make sure they are familiar with and prepared to meet project requirements. The QA Officer will make sure all systems are ready before field work is initiated.

The DQA will be conducted to determine whether the data meet the assumptions that the DQOs and data collection design were developed under and whether the total error in the data are tolerable. This assessment will include complete data verification and validation as described in Section 8.0. *Guidance for the Data Quality Assessment Process* (EPA QA/G-9) will be consulted.

The Project Manager will be responsible for implementing any necessary corrective actions. The occurrence and resolution of major quality issues identified during assessment activities will be documented in memorandum to the NPS Project Manager.

8.0 DATA VALIDATION AND USABILITY

8.1 Data Review, Validation & Verification Requirements

The data validation process evaluates whether the specific requirements for an intended use have been fulfilled and ensures that the results conform to the user's needs. The data validation process develops the QC acceptance criteria or performance criteria.

Data verification confirms that the requirements of the specified sampling and analytical methods were followed. This process involves reviewing the results of sampling and analysis to determine conformance with the QC requirements described for the project. The data verification process ensures the accuracy of data by using validated methods and protocols and is often based on comparison with reference standards.

Requirements and methods for data validation and verification are listed in Tables 8.0 and 8.1.

8.2 Validation & Verification Methods

Data will be reviewed to ensure that the requirements stated in Tables 4.1 and 8.0 were met. Data validation and verification will be conducted using the methods described in Table 8.1. Definitions for data verification and validation are as follows:

Data Verification: A consistent, systematic process that determines whether the data have been collected in accordance with the stated requirements for each activity. The verification process is independent of data validation and is conducted at various levels both internal and external to the data generator (laboratory). Data verification includes confirming that all sampling activities were conducted in accordance with the procedures described in this SAP.

Data Validation: An evaluation of the technical usability of the verified data with respect to planned objectives. Data validation is performed external to the data generator (laboratory), using a defined set of performance criteria to a body of data in the evaluation process. This may include checks on some or all of the calculations in the data set and reconstruction of some or all final reported data from initial laboratory data (e.g., chromatograms, instrument printouts). It is in the data validation process that data

qualifiers for each verified data are evaluated. It extends beyond the analytical method to protocols or QAPPs to address the overall technical usability of the generated data.

One hundred-percent of the data will be validated according to Table 8.1 requirements by the Project QA Officer or a subcontractor experienced in conducting this type of data verification. Data will be reviewed as it is received throughout the project. If problems are uncovered as a result of the validation effort, the QA Officer and Project Manager will be immediately notified. The QA Officer or Project Manager will discuss possible corrective actions with the laboratory prior to implementation. The Project Manager will immediately notify NPS of any data verification or validation issues that may affect the success of the project.

Any deviations from the analytical control limits specified in Tables 4.1 and 8.1 will be evaluated in terms of their effect on the data usability. Data usability will be assessed using the National Functional Guidelines for Data Review (Inorganic & Organic, February 1994). The completeness goal for the project is ninety-percent valid data.

The results of the data validation and verification will be summarized in a Data Review Report, to be prepared as part of the EE/CA.

8.3 Reconciliation with Data Quality Objectives

The data validation and verification results will be compared to the DQOs stated in Table 5.0 and with the PARCC parameters described in Table 8.0. This evaluation will summarize the QA/QC performance by PARCC criteria including completeness calculations expressing the percent complete of valid data compared to the total number of samples collected. The result of the data validation and verification will be summarized in the Data Review Report described above.

8.4 Reporting Limits

The reporting limits provided in Table 4.1 are the minimum levels that the laboratory will report analytical results without a qualifier when an analyte is detected. The laboratory can typically detect analytes at concentrations of up to an order of magnitude lower than the reporting limits; in this case, when a positive detection is less than the reporting limit, the value may be reported and qualified as an estimated concentration.

8.5 Holding Times

Holding times are storage times allowed between sample collection and sample extraction or analysis (depending on whether the holding time is an extraction or analytical holding time) when the designated preservation and storage techniques are employed. Sample preservation and holding time requirements for samples collected in the field investigations are summarized in Table 4.1. Holding times for soil samples for analysis of lead is 180 days with no preservative. All samples will be cooled and stored at 4 degrees Celsius (± 2 degrees Celsius) until the requested analyses are performed.

8.6 Quality Control Analyses

To provide an external check of the quality of the field procedures and laboratory analyses, one type of QC sample will be collected and analyzed. One triplicate sample for every 10 DUs at each Site will be analyzed.

One sample from each Site will be randomly selected for duplicate tests. Splits of these four samples will be created in the laboratory and identified as duplicates by adding the suffix “-D”

In addition to the external QA/QC controls, the laboratory maintains internal QA procedures. Internal QC samples will include laboratory blanks (i.e., method blanks, preparation blanks), laboratory duplicates, MS/MSDs and LCS/LCSDs, as discussed in Appendix B.

8.7 Special Training Requirements

The only special training required for this investigation is the health and safety training (29 CFR 1910.120) described in the Health and Safety Policy for the project (RMC, 2013).

9.0 MEASUREMENT AND DATA ACQUISITION

This section covers sample process design, sampling methods requirements, handling and custody, analytical methods, QC, equipment maintenance, instrument calibration, supply acceptance, non-direct measurements and data management.

9.1 Sample Process Design

The general goal of the field investigation is to collect sufficient data to complete the EE/CA. Sections 3.0 and 4.0 of this SAP describe the Field Sampling Plan.

9.2 Sampling Methods Requirements

Sampling equipment, containers and overall field management are described below.

9.2.1 Sampling Equipment and Preparation

Sampling equipment required for the field program for environmental sampling, health and safety monitoring, equipment and personal decontamination and general field operations are presented in Table 4.0 of this SAP.

Field preparatory activities include review of SOPs, procurement of field equipment, laboratory coordination, confirmation of Site access and a field planning meeting attended by field personnel and QA staff. Site mobilization is described in Section 4.1 of this SAP.

9.2.2 Sample Containers

Containers for the environmental samples that will be collected during the field program are specified in Table 4.1.

9.2.3 Sample Collection

Samples collected during this field program will consist of soil and water collected as a Quality Control equipment blank. All sample collection procedures are outlined in Section 4.4 and SOPs in Appendix A. The following SOPs apply to all applicable sample collection activities:

RMC SOP 1, Standard Procedures for Collection of Surface Water Samples and General Water Sample Handling

RMC SOP 2, Standard Procedures for Collection of Surface Soil Samples

RMC SOP 4, Standard Procedures for Sample Handling, Documentation and Shipping

RMC SOP 5, Standard Procedures for Sampling Equipment Decontamination

RMC SOP 9, Standard Procedures for Multi Incremental Sampling

9.3 Sample Handling and Custody Requirements

Custody and documentation for field and laboratory work are described below, followed by a discussion of corrections to documentation.

9.3.1 Field Sample Custody and Documentation

Samples analyzed through laboratories coordinated by RMC will be labeled using procedures established in the SOP, *Standard Procedures for Sample Handling, Documentation and Shipping* (RMC SOP 4). Sample labels will include the Site name, sample identification number, date and time of sample collection and required analyses. Sampler's initials will be recorded on the labels with permanent ink markers or pens at the time of sample collection.

9.3.2 Chain-of-Custody Requirements

A Chain-of-Custody Record will be completed at the time of sample collection. Field personnel will record the sample identification number, sampling date and time, sample matrix, sampler's initials and analytical requirements with permanent ink pens. Completed Chain-of-Custody Records will be reviewed for completeness prior to sample submittal. Samples will be relinquished under the Chain-of-Custody Procedures identified in the SOP, *Standard Procedures for Sample Handling, Documentation and Shipping* (RMC SOP 4).

9.3.3 Sample Packaging and Shipping

After the sample containers are sufficiently packaged, the plastic bag containing the samples will be sealed. Ice will be placed between the plastic bags and cooler.

9.3.4 Field Logbooks and Records

Documentation of field activities will be conducted in accordance with the SOP, *Standard Procedures for Sample Handling, Documentation and Shipping* (RMC SOP 4). The field sampling team will maintain a comprehensive field logbook that includes notes regarding instruments used, Site and weather conditions, GPS coordinates, vegetative community observations, sample time, sampler's name, analytical parameters, sample handling and chain of

custody. The field activities will be recorded in bound, sequentially numbered, waterproof notebooks. All entries will be made in permanent ink and will be clear, objective and legible. Where required, representative photographs will also be taken of field activities and sample locations and a description will be recorded in the logbook. The Field Operations Manager is responsible for maintenance and document control of the field logbooks.

9.3.5 Laboratory Custody Procedures and Documentation

Laboratory custody procedures are provided in each laboratory's QA Manual. Upon receipt at the laboratory, each sample shipment will be inspected to assess the condition of the shipping cooler and the individual samples. This inspection will include measuring the temperature of the cooler (to document that the temperature of the samples is within the acceptable criteria if cooling is required) and verifying sample integrity. The enclosed chain-of-custody records will be cross-referenced with all of the samples in the shipment. Laboratory personnel will then sign these chain-of-custody records and copies will be provided to field personnel. The sample custodian may continue the chain-of-custody record process by assigning a unique laboratory number to each sample on receipt. This number, if assigned, will identify the sample through all further handling. It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, data reporting and disposal.

9.3.6 Corrections To and Deviations From Documentation

For the logbooks, a single strikeout initialed and dated is required for documentation changes. The correct information should be entered in close proximity to the erroneous entry. All deviations from the guiding documents will be recorded in the logbook(s).

9.4 Analytical Methods Requirements

Samples collected during this project will be analyzed in accordance with standard EPA and/or nationally-accepted analytical procedures. The selected EPA-approved laboratories will adhere to all applicable QC requirements established by the subcontract. The methods to be used for chemical analysis and the associated holding times are shown in Table 4.1.

9.5 Quality Control Requirements

Field, laboratory and internal office QC are discussed below.

9.5.1 Field Quality Control Samples

Quality control checks will be employed during field activities to ensure the quality and integrity of sample collection. Triplicate QC samples will be collected in the field and submitted to the appropriate laboratory for analysis, as described in Section 6.0.

All triplicate samples will be collected according to standard MI sampling procedures. Triplicate samples will be prepared at a frequency of 10-percent of all DU samples obtained during the study and will be handled and analyzed in the same manner as the environmental samples.

9.5.2 Laboratory Quality Control Samples

The approved EPA contract laboratory will follow all laboratory QC checks, as defined in the analytical methods listed in Section 5.7. Quality control data are necessary to determine precision and accuracy and to demonstrate the absence of interferences and/or contamination. Each type of laboratory-based QC will be analyzed at a rate of five-percent or one per batch (a batch is a group of up to 20 samples analyzed together), whichever is more frequent. Results of the QC will be included in the QC package and QC samples may consist of laboratory blanks, laboratory duplicates, MS/MSDs and/or LCS/LCSDs (whichever are applicable) and any other method-required QC samples.

Laboratory blank samples will be analyzed to assess possible contamination so that corrective measures may be taken, if necessary. Duplicate samples are aliquots of a single sample that are split on arrival at the laboratory or upon analysis. Results obtained for two replicates that are split in a controlled laboratory environment may be used to assess laboratory precision of the analysis. MS/MSD and LCS/LCSD analyses may be used to determine both precision and accuracy.

Both normal and QC samples will be spiked with surrogate compounds, when applicable, and a percent recovery will be calculated for each surrogate.

9.5.3 Internal Quality Control Checks

Internal QC checks will be conducted throughout the project to evaluate the performance of the project team during data generation. All internal QC will be conducted in accordance with EPA CLP methods and requirements.

9.6 Equipment Maintenance Procedures

All laboratory equipment will be maintained in accordance with each laboratory's SOPs.

9.7 Instrument Calibration Procedures and Frequency

Calibration of field and laboratory instruments is addressed in the following subsections.

9.7.1 Field Equipment

Field instruments used in the field investigation consist of a GPS unit used to measure sample station coordinate. The GPS receivers require no special calibration procedure and all measurements will be conducted according to the manufacturer's suggested procedures.

9.7.2 Laboratory Equipment

Calibration of laboratory equipment will be based on written procedures approved by laboratory management. Instruments and equipment will be initially calibrated and subsequently continuously calibrated at approved intervals, as specified by either the manufacturer or more updated requirements (e.g., methodology requirements). Calibration standards used as reference

standards will be traceable to the EPA, National Institute of Standards and Technology or another nationally-recognized reference standard source.

Records of initial calibration, continuing calibration and verification, repair and replacement will be filed and maintained by the laboratory. Calibration records will be filed and maintained at the laboratory location where the work is performed and may be required to be included in data reporting packages.

9.8 Acceptance Requirements for Supplies

Prior to acceptance, all supplies and consumables will be inspected to ensure that they are in satisfactory condition and free of defects.

9.9 Non-Direct Measurement Data Acquisition Requirements

Non-direct measurement data include information from Site reconnaissance, literature searches and interviews. The acceptance criteria for such data include a review by someone other than the author. Any measurement data included in information obtained from the above-referenced sources will determine further action at the Site only to the extent that those data can be verified.

9.10 Data Reporting

Sample results and QC data will be delivered to the Project Manager as an electronic data deliverable (EDD) in addition to a hard-copy data package. Electronic copies of all project deliverables (including graphics) are maintained by project title. Electronic files are routinely backed up and archived.

10.0 REFERENCES

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United States Environmental Protection Agency (EPA). 1991. Management of Investigation-Derived Wastes During Site Inspections, Office of Emergency and Remedial Response, Washington, DC, OERR Directive 9345.3-02.

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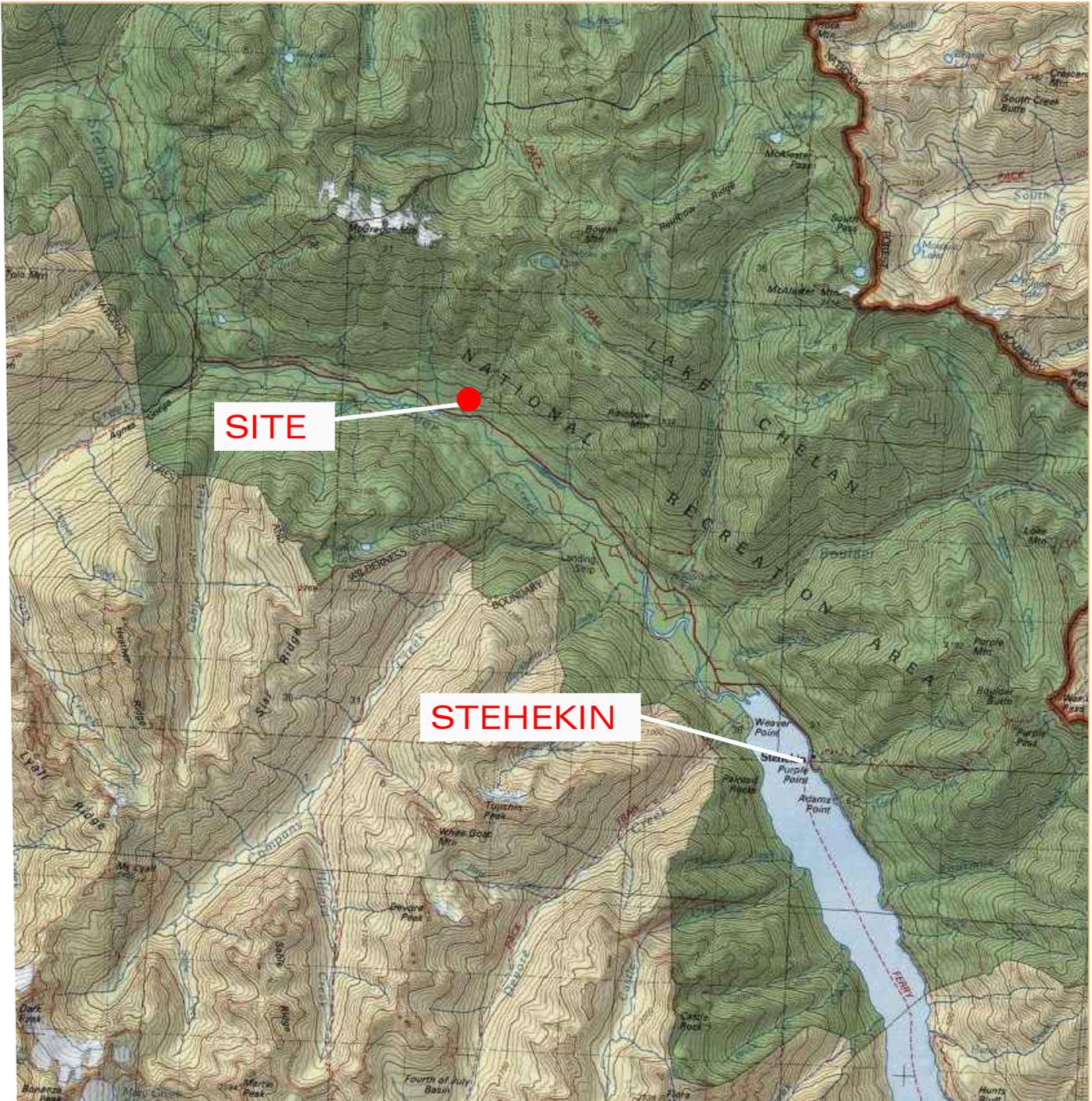
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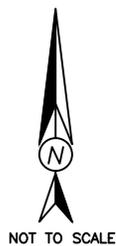
United States Environmental Protection Agency, Environmental Response Team (EPA/ERT). 1999. Standard Operating Procedures (SOPs).

U.S. EPA 2003. SW-846 On-Line (<http://www.epa.gov/epaoswer/hazwaste/test/main.htm>)

FIGURES



NOTE:
LOCATIONS NOT SURVEYED



NORTH CASCADES NP

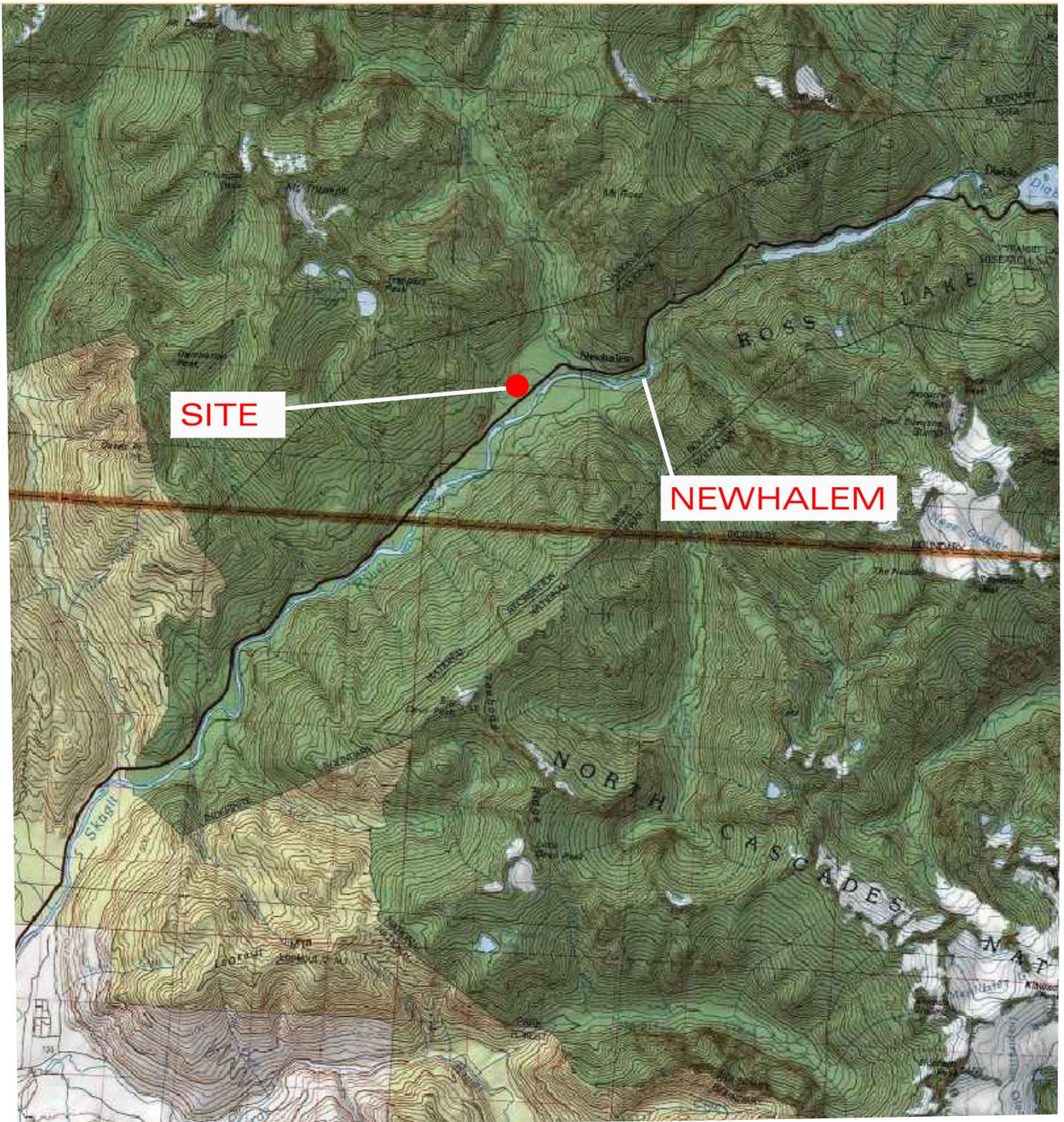
FIGURE 1
STEHEKIN FIRING RANGE
SITE MAP

RESOURCE MANAGEMENT CONSULTANTS
8138 SOUTH STATE ST.
SUITE 2A
MIDVALE, UT 84047
801-255-2626

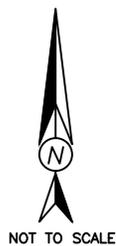


AUGUST 2013

nc site map.dwg



NOTE:
LOCATIONS NOT SURVEYED



NORTH CASCADES NP

FIGURE 2
NEWHALEM FIRING RANGE
SITE MAP

RESOURCE MANAGEMENT CONSULTANTS
8138 SOUTH STATE ST.
SUITE 2A
MIDVALE, UT 84047
801-255-2626



AUGUST 2013

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TABLES

Table 3.0
 Sampling Objectives
 Stehekin and Newhalem Firing Ranges
 Sampling and Analysis Plan

Media/Parameters	Sampling and Analysis Objectives	Data Use
Site Soils: Lead concentrations and distribution.	Determine lead concentrations.	Determine lead impacts and extents.

Table 4.0
 Recommended Field Equipment and Supplies
 Stehekin and Newhalem Firing Ranges
 Sampling and Analysis Plan

<u>Sampling</u>	<u>Health & Safety</u>	<u>Decontamination</u>	<u>General</u>
Soil core tool or soil pick	Latex gloves (or equivalent)		GPS
Steel shovel	Sunscreen	Plastic trash bags (1 box)	Wooden stakes or pin flags
Self-sealing plastic bags (gal. size)	Rain Gear	Deionized water (3 gallons)	Flagging (2 rolls)
Field logbook	Copy of HASP	Alconox	Coolers
Survey lathe, trimmed to 6"	Rain Shelter	Plastic buckets (2 5-gal)	Copy of SAP/HASP
Plastic trash bags (1 box of large - 30 count)		Scrub brushes (1)	Tape measure
Survey lathe , sample collection scoops		Sprayer (1-liter)	Ice
survey tape			Camera
Scale or volume measurement device			
Sample sieves (<2mm)			
Field Sampling Plan			

Table 4.1
Sample Collection Guide - Target Analytes and Collection Requirements
Stehekin and Newhalem Firing Ranges
Sampling and Analysis Plan

Parameters	Method	PRL ¹	Container	Volume ²	Temperature ³	Preservative	Technical Holding Times (Days)	Maximum Lab Precision (RPD%), and Accuracy (LCS % Recovery)
Pb (Total) - Soil	SW-846 6010C	1 mg/K	LDPE Jar or Bag	>500 grams	4°C +/- 2°	N/A	180	+/- 35%, 75%-125%
Soil Sample Preparation	Multi-Increment	NA	NA	NA	NA	NA	NA	NA
Soil Duplicate Sample Splits at Laboratory	One sample from each COC (Site) will be randomly selected for duplicate tests. Splits of these samples will be created in the laboratory after grinding is complete and will be identified as duplicates by adding the suffix "-D"							
Pb (Total) - Water	EPA 200.7	10 ug/L	Laboratory Supplied Bottle	TBD	4°C +/- 2°	HCl pH<2	180	+/- 20%, 85%-115%

N/A - Not Applicable

PRL - Practical Reporting Limit

1 - All units are mg/kg based upon dry weight unless otherwise noted.

Reporting limits are goals and may vary. These goals are at or near method detection limits and may be impacted by sample volume and/or sample matrix.

2 - Sample will be sieved to <2mm in laboratory

3 - Laboratory will measure the temperature of each cooler upon receipt to ensure proper temperature was maintained (4°C +/- 2°).

Table 4.2
Sample Alteration Form
Stehekin and Newhalem Firing Ranges
Sampling and Analysis Plan

Project Name and Number:

Material to be Sampled:

Measurement Parameter:

**Standard Procedure for
Field Collection and
Laboratory Analysis
(Cite Reference):**

**Reason for Change in Field
Procedure or Analysis
Variation:**

**Variation from Field or
Analytical Procedure:**

**Special Equipment,
Materials or Personnel
Required:**

Initiators Name: _____

Date: _____

Project Officer: _____

Date: _____

QA Officer: _____

Date: _____

Table 5.0
Sample Information Summary, Data Quality Objectives, Data Uses, Data Type, and QC Levels
Stehekin and Newhalem Firing Ranges
Sampling and Analysis Plan

Data Quality Objectives	Existing Data Summary	Design Rational, Data Needs and Parameters⁽¹⁾	Scheduling and Sample Selection Procedures⁽²⁾	Data Use	Analysis Type	Measurement Classification and QC Level
Determine lead concentrations in Site soils.	None	Conduct Multi-Increment Sampling to determine lead concentrations for soils in multiple Decision Units (DUs) at each Site.	Samples will be collected during characterization activities.	Determine extents of lead impacts (if present).	Analytical Laboratory soil metals analysis (Pb, dry weight).	Definitive (Laboratory)

1 - Detection limits and Methods are specified on Table 4.1.

2 - Locations will be determined in the field.

Table 8.0
Precision, Accuracy, Representativeness, Comparability and Completeness (PARCC)
Stehekin and Newhalem Firing Ranges
Sampling and Analysis Plan

Parameter	QC Program	Evaluation Criteria	Acceptance Criteria	Recommended Corrective Actions
Precision	Lab Duplicate Splits of Ground Samples.	Relative Percent Difference (RPD)	RPDs: soil samples +/- 35 percent if > 5 times LRL, or, +/- LRL if < 5 times LRL	Verify the RPD calculation. If correct, determine if matrix interference or heterogeneous samples are factors in poor RPD. If matrix effects or heterogeneous samples are not observed, reanalyze the associated investigative samples and MS/MSD. If appropriate, reextract or redigest and reanalyze the associated investigative samples and MS/MSD.
	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Relative Percent Difference (RPD)	See method-specific control limits ¹	Verify the RPD calculation. If this is correct, determine if matrix interference or heterogeneous samples are factors in poor RPD. If matrix effects or heterogeneous samples are not observed, reanalyze the method duplicate and associated investigative samples.
Accuracy	Matrix Spike (MS)	Percent Recovery	See method-specific control limits ¹	Verify the matrix spike percent recovery calculations and evaluate the LCS percent recoveries. If the calculations are correct and the LCS recoveries are acceptable, determine if matrix interference is a factor in the poor recoveries. If matrix effects not observed, reanalyze the MS and associated samples. If appropriate, reextract or redigest and reanalyze the MS and associated investigative samples.
	Matrix Spike Duplicate (MSD)	Percent Recovery	See method-specific control limits ¹	Same as above.
	Laboratory Control Samples (LCS)	Percent Recovery	See method-specific control limits ¹	Verify the percent recovery calculations. Evaluate the standard to determine if it is faulty. If it is, prepare a new standard and reanalyze the LCS and associated investigative samples. If necessary, recalibrate the instrument. Do not continue analysis until problem solved.
Representativeness	Holding Times	Representative of Environmental Conditions	Holding times met 100 percent	Evaluate whether data is critical to decision making. If so, resample and reanalyze for parameter exceeding holding time.
	Method Blanks	Qualitative Degree of Confidence	See method specific requirements ¹	Evaluate instrument, locate source of contamination, perform system blanks to confirm that system blanks meet performance criteria. Re-analyze method blank and associated samples. If method blank still above acceptance criteria, reextract or redigest the method blank and all associated samples.
	Equipment/Rinsate Blanks	Qualitative Degree of Confidence	Target analytes <1 X LRL; 5-10 X LRL for laboratory-induced contaminants.	Suggests field sampling-induced contamination may have occurred. Evaluate all associated QC samples. If all other QC samples are within prescribed acceptance limits, but equipment blank is not (e.g., positive identification of target analytes observed), contact USEPA immediately to determine if resampling and/or reanalysis is required.
	Field Triplicates	Quantitative Degree of Confidence	90 Percent of Field Duplicates Meet RSD Goals	If acceptance criteria not met, evaluate reasons for not meeting criteria (i.e., matrix interferences or heterogeneous samples) and make recommendations on whether resampling and/or reanalysis is necessary to improve degree of confidence.
Comparability	Standard Units of Measure	Qualitative Degree of Confidence	Laboratory Methods Followed	Revise analytical reports with correct units.
	Standard Analytical Methods		SOPs Followed	If SOPs not followed, evaluate whether reanalysis is necessary to obtain reliable data.
Completeness	Complete Sampling	100 Percent Valid ² Samples	90 Percent Valid ² Data	If not enough samples were collected for project needs, collect and analyze additional samples for parameters needed for key decisions.

¹Laboratory Control limits are specific to individual analytical/digestion methods and any deviation outside control limits are reported (see method-specific SOPs in Appendix A).

²Valid means that samples meet all evaluation criteria (i.e., are not rejected for any reason).

Precision is a measure of how repeatable data are and is often measured by sample duplicates.

Accuracy is a measure of how close the data are to the actual, or real value, measured by certified reference materials and matrix spikes.

Representativeness is a measure of how representative a sample is of the sample population and is achieved by accurate sampling procedures and appropriate sample homogenization.

Comparability looks at ongoing projects and how variable one set of data is relative to another. Comparability helps to measure the scientific consistency of the system to past work.

Completeness is a measure of how many data points collected are usable; 90% usable data is considered to be an acceptable value for completeness.

Table 8.1
Data Validation and Verification Requirements
Stehekin and Newhalem Firing Ranges
Sampling and Analysis Plan

Data Validation and Verification Steps	Data Validation and Verification Methods
Samples were collected according to established locations and frequencies.	→ Comparison with Sampling Plan
Sample collection and handling followed established procedures.	→ Review of field notes, field procedures and COCs
Appropriate analytical methods were used; internal laboratory calibration checks were performed according to the method-specified protocol.	→ Review of analytical methods and case narratives provided with laboratory reports. Documentation of any communications with laboratory concerning problems or corrective actions.
Required holding times and laboratory reporting limits were met.	→ Comparison with established holding times and LRLs.
Field Duplicates for QA/AC	→ Field duplicates met acceptance criteria tabulation of RSDs and comparison with PARCC parameters
Acceptance criteria (see Table 8.0) for field and laboratory QC samples (field blanks, field dups, equipment/rinsate blanks, method blanks, LCS) were met.	→ Tabulation of RPDs and spike recoveries, and direct comparison with method-specific acceptance criteria (see SOPs in Appendix A). Comparison with PARCC parameters.
Appropriate steps were taken to ensure the accuracy of data reduction, including reducing data transfer errors in the preparation of summary data tables and maps.	→ Maintain permanent file for laboratory hardcopies of analysis reports. Minimize retyping of data and error check data entered into database, tables, maps, etc.

RPD - Relative Percent Difference
LRL - Laboratory Reporting Limit

APPENDIX A
STANDARD OPERATING PROCEDURES

RMC SOP 1
STANDARD PROCEDURES FOR COLLECTION OF SURFACE WATER SAMPLES AND
GENERAL WATER SAMPLE HANDLING

1.0 Purpose

This SOP describes the procedures that will be used for collection of surface water samples. The procedures will ensure that samples are collected and handled properly and that appropriate documentation is completed.

1.0 Sampling Equipment:

- Log forms / Field notebook / Chain of Custody Forms (COC) – Documentation of sample activities, field notes and sample custody.
- Sample containers – Containers provided by laboratory for the collection, storage and transportation of samples.
- Direct reading instruments – field instruments to measure pH, conductivity and temperature.
- Disposable sampling gloves – to prevent exposure to water and the prevention of cross-contamination.
- Custody seals – seals to be placed on sample containers to maintain sample integrity.
- 0.45 um filter apparatus with inert filters – for filtering samples in preparation for the analysis of dissolved metals. For samples filtered in the field.
- Nitric acid (HNO₃, supplied by the analytical laboratory) – for sample preservation.
- Water velocity meter and tape measure – to measure stream flow (where applicable).
- Laboratory grade reagent water – for preparation of bottle blanks.
- Distilled water – for rinsing direct reading instruments.
- Custody seals – seals to be placed on sample containers to maintain sample integrity (where required).

2.0 Procedure

Sample bottles will remain sealed until the water sample is collected. At that time, the bottle lid will be removed and placed, top down, in an appropriate place. The sample bottle will be placed under the flow of water. If wading is required for sample collection, the sample must be collected upstream of wading personnel to avoid the sampling of suspended sediments. The container will be rinsed three times. After rinsing the container will be completely filled; any overflow of the sample container will be kept to a minimum. Sediment disturbance shall be kept to an absolute minimum. The sample cap will then be replaced on the sample bottle. All surface water samples will be collected in accordance with containers, volumes, preservatives, temperatures and holding times as outlined in Table 2-2 of the Sampling and Analysis Plan.

3.0 Dissolved Metals Analysis

Samples may be filtered by the analytical laboratory or in the field. Samples filtered by the laboratory will not be preserved in the field. Surface water samples collected for analysis of Dissolved (D) Metals and filtered in the field will be a minimum volume of 500 ml, collected in a poly or glass container. The samples may be field filtered. The field filtering methodology will include the following steps:

- 1: Sample shall be collected in a 1000 ml bottle.
- 2: Sample is poured into the top of the disposable plastic filter. A cartridge filter and peristaltic pump may also be used.
- 3: Vacuum pump is attached to the filter and pumped. If a cartridge filter is used the sample will be pumped through the filter using clean tubing.

4: When the bottom compartment of the filter is full, the water is to be transferred into a 500 ml sample container which shall be rinsed three times, the sample will be preserved with 2 ml of nitric acid (HNO₃), sufficient to bring the sample to pH <2.

5: The pH level in samples will be verified using pH paper before bottles are sealed. The pH level in samples will be verified using pH paper before bottles are sealed.

4.0 Total Metals Analysis

Surface water samples collected for analysis of Total (T) Metals will be a minimum volume of 500 ml or volume specified by the analytical laboratory, collected in a poly or glass container, and preserved with 2 ml of nitric acid (HNO₃), sufficient to bring the sample to pH <2. The pH level in samples will be verified using pH paper before bottles are sealed.

5.0 Cations/Anions ,Total Suspended Solids and other Analyses

Cations/Anions and Total Suspended Solids samples shall be collected in accordance with the methodologies outlined in the Procedure section of this SOP. Samples will not be preserved.

6.0 Stream flow Measurement

Stream flow volumes shall be measured during surface water sampling activities. To minimize sediment disturbance during sampling, the stream flow measurements should be conducted either downstream from the sampling point or after the completion of sample collection. RMC uses an electronic flow meter. The procedure for measuring stream flows is as follows:

- 1: Measure the width of the stream and divide the width into 0.5 foot increments.
- 2: At the midpoint of each 0.5 foot increment record the total depth of the stream. The water velocity shall be measured at 0.6 of the total height of the water (e.g. if the water is one foot deep the velocity is measured at a depth of 0.4 foot from the surface or 0.6 feet from the streambed).
- 3: Turn the electronic stream meter gauge on. Set the meter to record the average velocity. Insert the stream flow gauge into the water at the midpoint of each segment with the arrow pointing in the direction of flow. Measure the velocity for approximately one minute and record the average.
- 4: Calculate the stream flow by calculating the area of each 0.5 foot wide segment by multiplying the width times depth. To obtain the flow volume for each 0.5 wide segment multiply the area of the segment by the average flow velocity for the segment. The obtain the total stream flow add the total stream flow for each segment. An Excel spreadsheet is typically used for the calculations.

Calculations:

Segment flow volume = depth of 0.5 foot segment x width x flow velocity (feet/sec.) = cubic feet/ second
Total flow volume = sum of segment flow volumes.

7.0 Equipment Blank Collection

Equipment Blanks will be collected by pouring laboratory-grade de-ionized water through or on decontaminated sampling equipment. The water will be collected as a surface water sample and analyzed for the project contaminants of concern (COCs)

8.0 Labeling

Each sample will be labeled with the following information:

- Sample identification;
- Project number/name;
- Analyses requested;
- Preservatives (if required);
- Date/time collected; and
- Samplers initials.

8.0 Documentation

Field activities shall be recorded in a hard bound field notebook. Field notes shall include all pertinent information including but not limited to:

- Date and time samples were collected;
- Physical description of sample area;
- Identification of samples collected;
- Total number of samples collected;
- Total number of samples collected from each sample location;
- Physical description of samples;
- Preservatives used for samples;
- Sample container types;
- Filtered vs. Unfiltered samples (water);
- Analysis to be performed;
- Weather conditions;
- Hand sketches of subject area(s); and
- Description and date of any photograph(s) taken.

Sample handling and Chain of Custody documentation shall be in accordance with RMC SOP 5 found in this document.

9.0 Demobilization

After Decontamination, sample equipment will be stored in the appropriate, clean containers. Any equipment that suffers damage or excessive wear while conducting sampling will be labeled and reported to the equipment manager for the necessary maintenance, repair and/or replacement.

SOP 2
STANDARD PROCEDURES FOR COLLECTION OF SURFACE AND NEAR SURFACE SOIL
SAMPLES

1.0 Purpose

This SOP describes the procedures that will be used for sampling surface soils from ground surface to a maximum of 18 inches below surface. Samples will be collected with a Decontamination shovel or hand auger/probe. Specific soil sampling locations will be determined from the project work plan.

2.0 Sampling Equipment:

- Hand Auger/Probe and/or Shovels – For the collection of soil samples below the ground surface.
- Log forms / Field notebook / Chain of Custody (COC) - Documentation of sample activities, field notes and sample custody.
- Sample containers - Containers provided by laboratory for the collection, storage and transportation of samples. Plastic bags may also be used.
- Stainless steel sample spoons – For the collection of surface soil samples and composite sample mixing. Disposal sampling implements may also be used.
- Sample location staking – For the marking and identification of sample locations. Staking should be easily visible for surveying.
- Disposable sampling gloves – to prevent exposure to soils and the prevention of cross-contamination.
- Custody seals – seals to be placed on sample containers to maintain sample integrity.
- GPS – for recording the sample location (where required).

3.0 Decontamination Equipment:

- 5 gallon buckets – For washing and the collection of rinsate.
- Alconox - Soap
- Scrub brushes – For cleaning sampling equipment.
- Distilled water – For final equipment rinse.
- Culinary tap water – for equipment rinse.
- Garbage bags – for clean equipment storage.

4.0 PROCEDURE:

All samples shall be collected using Decontaminated equipment. Decontamination procedures are detailed in RMC SOP 6.

4.1 Discrete Samples

If significant vegetation, rocks, or debris prevent collecting the surface samples then the upper 2-3 inches of soil will be scraped away from the sample location with a shovel or stainless steel spoon. The underlying soil will then be collected and placed into sample containers with a stainless steel spoon or gloved hand. Composite samples will be homogenized as described below. Coarse grained soils, gravel and rock fragments will be removed wherever possible.

4.2 Composite Samples

Composite samples will be collected (as described above) by placing sub samples into a stainless steel mixing bowl or a clean plastic bag, or by hand with new, clean sampling gloves. The sample will be

homogenized with a stainless steel spoon or gloved hand. The homogenized soil will be packaged in a laboratory-supplied sample container, labeled and placed in a cooler to maintain temperature.

4.3 Sediment Samples

Sediment samples will be collected from depths of up to 10 cm using a procedure similar to that used for discrete surface soil samples.

5.0 Sample Preparation

Soil Samples collected for human health risk assessment shall be sieved to <250 microns. The <250 micron fraction is then analyzed for metals. For ecological screening/risk assessment purposes, sieving should not occur. Sieving shall be performed by the laboratory.

6.0 Labeling

Each soil sample will be labeled with the following information:

- Sample identification;
- Project number/name;
- Analyses requested;
- Date/time collected; and
- Samplers initials.

7.0 Documentation

Field activities shall be recorded in a hard bound field notebook. Field notes shall include all pertinent information including but not limited to:

- Date and time samples were collected;
- Physical description of sample area;
- Identification of samples collected;
- Total number of samples collected per sampling event;
- Total number of samples collected from each sample location;
- Physical description of samples;
- Preservatives used for samples;
- Sample container types;
- Filtered vs. Unfiltered samples (water);
- Analysis to be performed;
- Weather conditions;
- Hand sketches of subject area(s); and
- Description and date of any photograph(s) taken.

Sample handling and Chain of Custody documentation shall be in accordance with RMC SOP 5 found in this document.

8.0 Demobilization

After Decontamination, sample equipment will be stored in the appropriate, clean containers. Any equipment that suffers damage or excessive wear while conducting sampling will be labeled and reported to the equipment manager for the necessary maintenance, repair and/or replacement.

SOP 5
STANDARD PROCEDURES FOR SAMPLE HANDLING, DOCUMENTATION, AND SHIPPING

1.0 Purpose

This section describes the handling and documentation procedures that will be used once soil and water samples are collected. The procedures will ensure that samples are handled properly and that appropriate documentation is completed.

2.0 Sample Handling

All samples will be promptly placed into a cooler to maintain a temperature of 4°C. Typically, samples selected for chemical analysis will be delivered at the end of each day to the analytical laboratory. If they are not submitted to the laboratory on the same day, they will be stored in a refrigerator in a locked storage room until they can be delivered to the laboratory.

3.0 Sample Identification and Labeling

Soil samples will be labeled in such a way as to identify the area and depth from which they were taken. Water samples will be labeled as to identify when and where they were collected from. Duplicate samples will always be labeled in the same manner such that the laboratory cannot tell they are duplicate (i.e., as a “blind duplicate”). Each sample container will be immediately labeled with the following information:

- Project name
- Project number
- Sample identification
- Date and time collected
- Analysis requested
- Filtered or unfiltered (water)
- Samplers initials
- Preservative used (water)

This information will also be recorded in the field logbook.

5.0 Custody Seals

Custody seals shall be used to prevent tampering and to maintain sample integrity. A seal shall be placed across the top of sample jars or across the seals of plastic sample bags. The seal shall be signed and dated by the sampler who collected the sampler.

6.0 Chain-of-Custody (COC)

COC documentation will begin in the field for each sample submitted to the laboratory and will also be maintained by laboratory personnel. Samples that are submitted to AEC will use the COC provided by AEC. A COC for each sampling event will be completed and will accompany each sample batch to the analytical laboratory. Sample custody means that all samples will remain in the possession or observation of the sampler at all times, or in a locked facility until delivery to the analytical laboratory. A sample COC form is provided in Appendix D. Copies of the COC forms shall be stored in a three ring binder for sample tracking.

7.0 Field Book

RMC field personnel will maintain a field logbook to record all field activities. The field logbook will be a weather-resistant bound field book. All data generated during the project and any accompanying comments will be entered directly into the logbook in indelible ink; any corrections will be made with single line-out deletions. At no time will any pages be removed from the field logbook.

Each day's field activities will be documented, including the following minimum information:

- Date of field activity;
- Time of field activity;
- RMC field personnel's initials;
- Project name;
- Project number;
- Date and time samples were collected;
- Physical description of sample area;
- Identification of samples collected;
- Total number of samples collected per sampling event;
- Total number of samples collected from each sample location;
- Physical description of samples;
- Preservatives used for samples;
- Sample container types;
- Filtered vs. Unfiltered samples (water);
- Analysis to be performed;
- Weather conditions;
- Hand sketches of subject area(s); and
- Description and date of any photograph(s) taken.

8.0 RMC Sample Logbook

RMC will maintain a sample logbook, which will track all samples collected and/or accepted by RMC. The logbook will provide a unique, six digit alphanumeric identifier that will be assigned to each sample collected. All samples collected will be assigned an identifier number, regardless of that samples' submission to a laboratory. The next available chronological number in the sample logbook will determine the identifier, and this number will be cross-referenced with a sample description number, assigned in the field.

The RMC Sample logbook will be a covered, bound journal with non-removable pages. At no time will any pages be removed from the sample logbook.

All entries into the sample logbook will be made in indelible ink; and all corrections shall consist of initialed, line-out deletion. Data contained therein will include:

- Unique identifier number;
- Date;
- Project number;
- Sample description number;
- Sampler initials; and Lab acceptance initials.

SOP 6
STANDARD PROCEDURES FOR SAMPLING EQUIPMENT DECONTAMINATION

1.0 Purpose

This SOP details the Decontamination protocols for sampling equipment. In order to reduce the risk of transferring materials from one sample site to another, and to assure that there is no cross-contamination of samples, the following procedures will be used.

2.0 Decontamination Equipment:

- 5 gallon buckets – For washing and the collection of rinsate.
- Alconox - Soap
- Scrub brushes – For cleaning sampling equipment.
- Distilled water – For final equipment rinse.
- Culinary tap water – for equipment rinse.
- Garbage bags – for clean equipment storage.

3.0 DECONTAMINATION PROCEDURES:

RMC uses the following Decontamination procedure for equipment:

3.1 Gross contaminant removal

This step involves scrubbing the equipment using an Alconox and water solution and a stiff scrub brush. The scrubbing will continue until all visible contaminants are removed from the equipment. This water will be changed as necessary. The Alconox and water solution is typically prepared and stored in a clean 5-gallon bucket.

3.2 Clean detergent wash

This step involves using a clean volume of Alconox and water solution. Equipment will be washed in this solution once all gross contaminants have been removed during Step 1. This solution will also be changed as necessary. The Alconox and water solution is typically prepared and stored in a clean 5-gallon bucket.

3.3 Clear water rinse

This step involves rinsing the equipment in clear, culinary tap water. This water will be changed as necessary to maintain its purity. The water solution is typically collected and stored in a clean 5-gallon bucket.

3.4 Distilled water rinse

Distilled water will be used as a final rinse for all Decontamination procedures. The water will be poured from a new container, or sprayed from a suitable container or the equipment will be submerged in a suitable container. Decontamination (equipment) blanks will be collected as required in the Sampling and Analysis Plan. The water solution is typically collected and stored in a clean 5-gallon bucket.

3.5 Decontamination fluid disposal

Decontamination fluids shall be disposed of on-site in the tailings impoundment area.

RMC SOP 9

STANDARD PROCEDURES FOR MULTI INCREMENTAL (MI) SAMPLING

1.0 Purpose

This SOP describes the procedures that will be used for the collection of Multi-incremental (MI) samples (also identified by the by the acronym MIS). The MI sampling process, as described in this SOP, if applied correctly, may provide a more representative view of mean contaminant concentrations than traditional sampling approaches.

The objective of environmental sampling is to quantify some property of the media sampled, such as the amount of a contaminant present in soil at a given site. Traditionally, environmental cleanup programs across the nation have relied on discrete sampling to characterize environmental media. However, the number of discrete samples often collected at a contaminated site does not lend itself to statistically valid interpretation and cannot accurately quantify contaminant concentrations due to the heterogeneity of environmental media. In other words, it is impossible to identify the true mean of a population without the census of every data point. In the case of a 3,000 cubic yard soil stockpile, for example, the entire mass would have to be analyzed to determine the true mean concentration. Since it is impossible to sample and analyze the entire population due to practical considerations and cost limitations, statistical methods are used to determine a representative concentration. (AKDEC, 2009)

1.2 Glossary of Method-Specific Terms

1.2.1 Sampling Units

A sampling Unit (SU) also sometimes identified as a Decision Unit (DU), is the area and depth of soil (the sampled population) to be characterized by the average concentration of the MI sample. A DU may contain one or more SUs that are sampled using MI techniques. SUs must be restricted to actual source zones and must incorporate only areas that are similar as far as impacts as to not dilute contamination. SUs/DUs selected based on future land use scenarios may also be referred to as Exposure Units.

SUs must be delineated so that the mean analyte concentrations obtained are directly relevant to well defined project and or/risk objectives. They are the smallest volume of soil for which a concentration value will be obtained, and the basic unit about which a decision or conclusion based on an analytical result can be made.

1.2.2 Decision Units

A DU is a specific area (or volume of soil) about which a decision is to be made. In the ideal and most direct case, the DU and SU are the same volume of soil. As noted above, a DU may be composed of a single SU, or may include multiple SUs, if the DU is very large in size. The critical concern is that the entire area of a DU is consistent as far as contamination distribution and future use/exposure scenario, as with an SU. Either all or a percent of the SUs composing

the DU may be sampled in an MI fashion, the number of SUs sampled depending on the confidence of the data that are extended from the SUs to the DU.

2.0 Equipment

The following equipment and materials may be required:

- Spray paint , pin flags, or rope to mark either grid corners or outline the sampling grid.
- Incremental sampling tools such as coring devices, stainless steel or plastic spoons or scoops. Scoops may be used but only in conjunction with scales, so that aliquots of equal mass are collected from each location.
- Clean Zip-lock bags, 5-gallon plastic containers, or other appropriate large container for placing the increments. The capacity of the container should be adequate to hold the sample volume, which typically ranges from 1-2 kilograms.
- Standard environmental sampling equipment including coolers and ice for cold storage of samples after collection.
- Field logbook and pen field documentation.
- Global Positioning System (GPS), tape measures or other survey equipment to document locations of DUs.
- Personal protective equipment as specified by the Health and Safety Plan for the project.

3.0 Procedure

Increments of soil will be collected within each cell of the SU or DU. Increments will be approximately the same weight. A scale may be used if necessary. For surface soil sampling, a coring or similar tool may be used to facilitate the rapid collection of uniform and representative increments from a consistent depth interval/horizon. This will allow for the collection of equal volumes for each increment and that an equal mass is obtained under the assumption that the density of the sampled medium is uniform across the cell of the SU. The size of the coring tool will be selected based on the volume of the increments and media to be sampled, which is in turn calculated based on number and depth of the increments and the fact that an adequate total sample mass is typically 1-2 kilograms dry weight. This mass will overcome the effects of compositional heterogeneity due to the inherent inhomogeneous nature of soil and sediment. It is not necessary to precisely determine the GPS location of every increment collected. This assumes that the SU/DU has been properly identified and the relative position of the increment location within each cell is recorded.

The SU or DU will be demarcated in the field using pin flags, spray paint or rope and recorded with a GPS. Increments will be selected as defined in the sampling plan or field-fit based on site conditions. The increments will be collected from the depth specified in the Sample and

Analysis Plan (SAP) using a coring tool or other method that ensures equal volume is collected for each increment. Unless specified in the SAP, the vegetative mat will be included in the sampled interval. If used, the stainless-steel sampler will be pushed into the soil until the sampler is full and will not penetrate further. The sampler is then removed carefully, and the soil is pushed out of the sampler. For samples collected with scoops or similar tools, the sample will be weighed to provide uniformity between samples.

The sample (increment or aliquot) will be directly placed into a large re-sealable bag, 5-gallon bucket, or similar large container. The holes left by sampling may be filled using surrounding soil.

Soil samples should not include large rocks or pebbles unless they are part of the overall soil matrix. It is not necessary to decontaminate the sampling tool between the increments within a DU or SU.

The USACOE (2007) recommends using a systematic-random sampling design when collecting individual increments to build each sample (Hewitt et al. 2005b, in USACOE, 2007). This sampling design is analogous to systematic grid sampling (USEPA 2002, in USACOE, 2007), where the starting location is chosen randomly and the remaining sampling locations are laid out in a regular pattern (Cressie 1993, in USACOE, 2007). To use this approach, the sampler begins at a point on the edge of the area to be characterized and collects an increment of surface soil after a predetermined number of steps, while walking back and forth in a systematic manner across the area of interest. Replicate or triplicate samples may be collected in a similar manner by using a different start point.

Prior to the collection of replicate samples or MI samples from another SU or DU, the sampling tool will be decontaminated according to requirements set forth in RMC SOP 6 – Standard Procedures for sampling Equipment Decontamination. The replicate samples from the same SU/DU will be collected following a different path, as shown in Figure 1. The specific relative location of the replicate increments within each SU/DU cell will be established in a random manner to eliminate potential bias. To select the relative increment location for a replicate increment in a cell, the cell may be divided in turn into sub-grids and a sub-cell may be selected by randomly generating a number on a calculator.

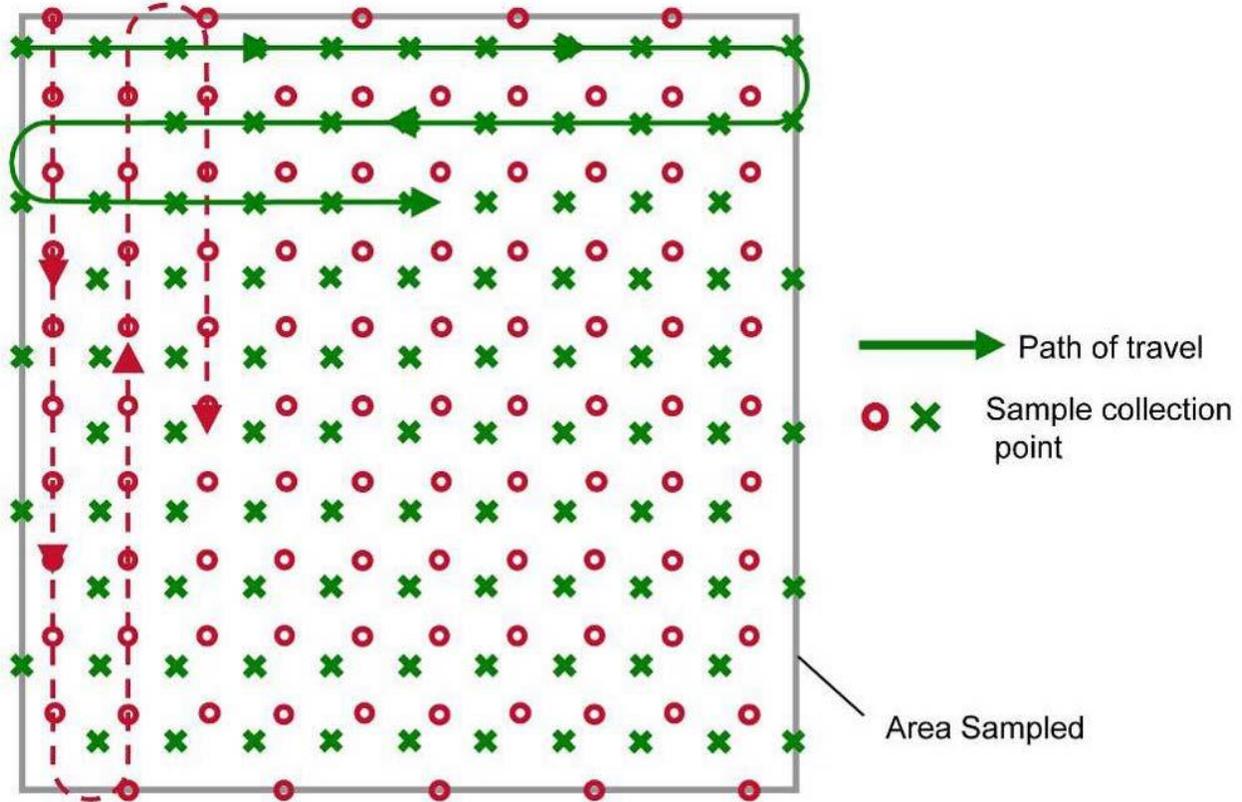


Figure 1: Systematic-random 100-increment sampling pattern used for collecting samples in grid areas (USACOE, 2007)

The large re-sealable bag containing the total sample volume will be handled in accordance with RMC SOP 5 – Standard Procedures for Sample Labeling, Handling, Documentation and Shipping.

4.0 Documentation

All Documentation will be conducted in accordance with the project SAP and RMC SOP 5 – Standard Procedures for Sample Labeling, Handling, Documentation and Shipping.

5.0 Demobilization

After completion of sampling, sample equipment will be stored in the appropriate, clean containers. Any equipment that suffers damage or excessive wear while conducting sampling will be labeled and reported to the equipment manager for the necessary maintenance, repair and/or replacement.

6.0 References and MI Method Background Documents

State of Alaska Department of Environmental Conservation (AKDEC). 2009. Draft Guidance on Multi Increment Soil Sampling. Division of Spill Prevention and Response Contaminated Sites

Program. March.

State of Hawaii Department of Health. 2009. Technical Guidance Manual for the Implementation of the Hawai'i State Contingency Plan Interim Final. Office of Hazard Evaluation and Emergency Response. .

U.S. Army Corps of Engineers (USACOE). 2009. Interim Guidance 09-02, Implementation of Incremental Sampling of Soil for the Military Munitions Response Program. 20 July.

U.S. Army Corps of Engineers (USACOE). 2007, Protocols for Collection of Surface Soil Samples at Military Training and Testing Ranges for the Characterization of Energetic Munitions Constituents.

U.S. Environmental Protection Agency. 2006. SW-846 Method 8330B, Appendix A Collecting and Processing of Representative Samples for Energetic Residues in Solid Matrices from Military Training Ranges

APPENDIX B
LABORATORY QA/QC DOCUMENTATION



DOCUMENT TITLE: SUBSAMPLING AND COMPOSITING OF SAMPLES

REFERENCED METHOD:

SOP ID: GEN-SUBS

REV. NUMBER: 5

EFFECTIVE DATE: 06/30/2013



SUBSAMPLING AND COMPOSITING OF SAMPLES

ALS-KELSO

Confidential

SOP ID: GEN-SUBS Rev. Number: 5 Effective Date: 06/30/2013

Approved By: Harvey Jacky Date: 6/13/13
 Department Supervisor - Harvey Jacky

Approved By: Suzanne LeMay Date: 6/13/13
 QA Manager - Suzanne LeMay

Approved By: Jeff Grindstaff Date: 6/13/13
 Laboratory Director - Jeff Grindstaff

Issue Date: _____ Doc Control ID#: _____ Issued To: _____

Proprietary



Standard Operating Procedure

for

SUBSAMPLING AND COMPOSITING OF SAMPLES

Confidential

1. SCOPE AND APPLICATION

- 1.1. This standard operating procedure describes procedures for obtaining subsamples used for laboratory analysis. The procedure also describes general practices for making composite samples from multiple individual samples. Procedures are given for aqueous, soil, sediment, vegetation and miscellaneous matrices. The SOP does not apply to tissue samples. Procedures for tissue samples are described in the GEN-TISP and MET-TDIG SOPs.
- 1.2. The SOP describes routine, or default, procedures for samples that do not require VOC analyses. Handling of VOC samples is described in SOP VOC-5035. Program or project-specific requirements may differ from those described in the SOP. Samples analyzed by EPA CLP procedures are specifically excluded from this procedure, and will be handled according to the applicable SOW.
- 1.3. Multi-increment samples require special handling and subsampling procedures. In addition to routine procedures, this SOP also includes instructions for handling and sampling from multi-increment samples submitted to the laboratory.
- 1.4. This procedure does not apply to situations where the entire sample (container) is used for the analysis.

2. METHOD SUMMARY

- 2.1. Obtaining a representative analytical subsample from the field sample submitted is essential to providing meaningful data. The subsample must be taken to most closely reflect the predominant composition of the sample. For aqueous and liquid samples, this is usually accomplished by shaking or inverting the sample. For soil, sediment, powders, and other solids the procedures are more involved. Procedures for subsampling are based on the information given in the references listed.
- 2.2. Some projects may employ multi-increment (MI) sampling in the field. The primary objective of MI sampling is to control the certain statistical errors associated with discrete sampling. Some studies have shown that MI sampling, using 30+ sample increments within a decision unit (a defined field sampling area) may provide a more representative view of contaminant concentrations than traditional discrete sampling approaches. References listed provide additional background on MI sampling. When this approach is taken it is important that laboratory procedures are consistent with field procedures when taking samples.



- 2.3. Unique sample matrices such as vegetation, wood and wood chips, mechanical parts and filters, etc. pose additional challenges to obtaining representative samples. For these samples the laboratory staff should consult with the Project Manager to determine the subsampling strategy. These special situations will be handled on a case-by-case basis. Service requests should list any specific sample preparation required.

3. DEFINITIONS

- 3.1. Sample – A portion of material taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity (ASTM).
- 3.2. Subsample – A portion of a sample taken for the purpose of estimating properties or composition of the whole sample (ASTM).
- 3.3. Composite sample – A mixture of multiple samples or subsamples produced to result in one sample representative of multiple field samples.
- 3.4. Representative subsample – A subsample collected in such a manner that it reflects one or more characteristics of interest (a defined by the project objectives) of the laboratory sample from which it was collected (ASTM).
- 3.5. Multilayered sample – A sample consisting of two or more clearly differentiated components (ASTM).
- 3.6. Multi-increment (MI) sample – A field sample consisting of multiple bulk containers from one decision unit (defined in a MI sampling plan) submitted to the lab for subsampling into a representative sample for analysis.

4. INTERFERENCES

- 4.1. When obtaining subsamples it is important to minimize any chances for sample contamination or cross-contamination between samples. Work should be performed in an organized and neat manner. Spilling of samples (from overfilled containers, etc) should be minimized and spills cleaned up. Equipment and laboratory tools used with samples should be cleaned between samples to prevent cross-contamination.
- 4.2. Analysis-specific interferences are described in the applicable analytical SOP.

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 personal 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 ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.



6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

- 6.1. Refer to the analytical SOP for sample collection preservation and storage of samples. Subsamples and composite samples held for later analysis should be preserved and stored in the same manner as specified for field samples.
- 6.2. Projects for MI samples may include additional instructions not found in the analytical SOP. The analyst should consult with the Project Chemist, or refer to the Project Chemist's instructions, prior to working with these samples.

7. APPARATUS AND EQUIPMENT

- 7.1. Laboratory balance capable of weighing the desired sample mass. There are various makes and models of balances available for use, with each department having balances appropriate for its use. For weighing solids and non-aqueous liquids (wastes), use a top-loader balance. Ensure that the mass (sample + container) to be placed on the pan is within the calibration-verified range of the balance.
- 7.2. Wiley laboratory mill, Model 4. Operate the Wiley mill following the manufacturer's recommendations.
- 7.3. Sieve shakers.
- 7.4. Shatter box.
- 7.5. Mechanical mixer and/or shaker.
- 7.6. Stainless steel or Glass mixing bowl.
- 7.7. Metal or disposable spoons and spatulas.
- 7.8. Aluminum foil.
- 7.9. Weighing boats, plastic or aluminum
- 7.10. Clean sample containers and lids (various sizes) as specified in the applicable test SOP.
- 7.11. Common laboratory glassware/apparatus (beakers, flasks, pipets, syringes, etc.).
- 7.12. Multi-Increment Samples
 - 7.12.1. Flat spatula
 - 7.12.2. Flat stainless steel masons trowel
 - 7.12.3. Volatile sample containers.
 - 7.12.3.1. 250–500 milliliter (ml) narrow mouth, amber bottles (recommended)
 - 7.12.3.2. 4–8 ounce (oz) amber jars with Teflon lined septum lids



7.12.4. Large stainless steel spoon or scoop

7.12.5. Large clean containers (a large stainless steel or glass bowl, Ziploc bags, or 5-gallon bucket)

7.12.6. #10 (2mm) sieve

7.12.7. Stainless steel cookie sheet or other tray

8. REAGENTS

8.1. Dichloromethane, acetone, and acetonitrile may be used during the noted procedures for cleaning and decontamination of equipment.

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.

10. PREVENTIVE MAINTENANCE

10.1. No preventive maintenance is required other than normal glassware and apparatus cleaning.

11. PROCEDURE

11.1. Aqueous samples – Subsampling

11.1.1. Examine the sample. Thoroughly mix all samples by vigorous shaking. Immediately open the container and obtain the subsample. Additional filtering of the subsample may be required by the analytical SOP.

11.1.2. If the sample is multi-layered (a water layer with a sand/sediment layer that cannot be mixed or non-aqueous liquid layer) the Project Chemist should be consulted on how to proceed with the sample. Additional analyses or sample preparations may be necessary depending on the client's data needs. Document the condition of the sample and decision made on subsampling.

11.2. Aqueous samples – Compositing

11.2.1. The customer may require compositing based on flow rates to create a flow proportional composite. The compositing instructions are included with the Form V. Equal volume compositing is assumed if there are no specific instructions provided for compositing ratios.

11.2.2. Setup the necessary glassware and/or sample container receiving the composite sample. Ensure that proper measuring glassware is used, typically a graduated



cylinder or volumetric flask for larger volumes and pipet or syringe for smaller volumes.

11.2.3. Working quickly, mix the individual samples (as described above), open the container(s) and obtain the composite aliquot. Add each aliquot to the composite container and cap between samples.

11.2.4. Once all composite aliquots are obtained, cap and mix the composite sample. Label the container appropriately. Complete all documentation necessary to describe the compositing procedure, including samples used, aliquot taken, etc.

11.3. General considerations – Non-liquid samples

11.3.1. The analyst must first understand what the sample matrix of interest is. The project information should be consulted. If the sample appears to be homogeneous (other than extraneous materials described below) particle size reduction is not necessary. Particle size reduction should be performed only when required by the project QAPP, project specifications, or client request. If particle size reduction is required, use the appropriate apparatus (Wiley mill, shatter box, etc.) to perform crushing, grinding, milling, or sieving, and document. Refer to ASTM D6323 for guidelines on performing particle size reduction.

11.3.2. Once the matrix of interest is known, examine the sample for presence of extraneous material. The default procedure is to remove these items, or not include in the representative subsample. However, the presence of these materials should be documented in lab records and the Project Manager should be consulted prior to subsampling. Some examples are given below.

11.3.2.1. Soil, solid, and sediment samples may include such material as larger rocks, sticks, leaves, pieces of metal, man-made materials, etc.

11.3.2.2. Wood or bark samples may include chunks of soil, mud, rocks, etc.

11.3.2.3. Vegetation samples may include chunks of soil, mud, rocks, sticks (not of the sample type, etc.).

11.3.2.4. Sediment samples may include rocks, twigs, vegetation, organisms, etc.

11.3.2.5. Sediment/marine projects, organisms are typically analyzed under separate sampling and analysis plans.

11.3.2.6. Mechanical parts, filters, etc., may include chunks of soil, mud, rocks, sticks, etc.

11.3.3. Examine soil samples to determine if the sample contains significant amounts of water. If the amount of water is greater than approximately 30%, treat the sample as a sediment sample.

11.3.4. Samples which are especially heterogeneous, as well as various special matrices, may require additional preparation. These will be handled on a case-by-case basis after consultation with the appropriate supervisors and Project Manager. Unique matrices for TCLP and other leaching procedures should be handled according to the applicable SOP or reference method.

11.4. Soil/solid Samples – Subsampling



11.4.1. Samples in jars

11.4.1.1. Using a spatula or other utensil made of an inert material, thoroughly mix and homogenize the sample, making sure to loosen sample from the sides of the container, and continue mixing the entire contents, breaking up soil clumps, etc., until there is no visible segregation of the sample by layer, grain size, color, etc. The sample should appear uniform in color and texture.

11.4.1.2. Once mixed, remove the desired mass of sample for the analysis and document accordingly. Recap the jar and return to storage.

11.4.2. Samples in sleeves (core samples) and large bulk containers.

11.4.2.1. Samples in sleeves are emptied into a metal or glass homogenizing container and thoroughly stirred using a spatula or other utensil. When homogenized the appropriate sample portions are placed in jars. Perform additional drying and grinding only when specified for the project. Client specifications for drying and grinding will be communicated by the Project Chemist.

11.4.2.2. When working with sleeves and resulting homogenized samples/subsamples, always double-check the sample ID on the sleeve against the sample numbers on the samples.

11.5. Soil/solid samples – Compositing

11.5.1. Thoroughly mix each individual sample as described in 11.4 above.

11.5.2. Combine equal masses from each of the individual samples into a clean stainless steel mixing bowl. The amount used will depend upon the number of analyses to be performed on the composite and/or the amount available. The analyst preparing the composite will document the mass of each individual sample used for the composite, the date and time of compositing, and any other pertinent observations.

11.5.3. The sample is thoroughly homogenized using a spatula or other utensil and returned to clean glass jars. The sample container is labeled as a composite and with the sample identification, dated, and initialed.

11.5.4. The composite sample and remaining individual samples are returned to storage.

11.6. Sediment Samples – Subsampling

11.6.1. Standard procedure calls for mixing overlying water into the sample. EPA SW-846 methods for organic extractions specify to decant and discard overlying water. However, the Puget Sound Protocols and others have options for decanting and discarding this water, decanting and performing a separate water analysis, or mixing the water into the sample. The analyst should confirm which option is to be used on the sample. For projects not within the scope of the Puget Sound Protocols or similar project plans, the overlying water should be decanted and



discarded for organics analysis. For metals and inorganics, mix the overlying water into the sample.

11.6.1.1.**Note:** If water is decanted and discarded and percent solids is to be applied or determined, a separate solids determination must be made on the decanted sample.

11.6.2. Thoroughly mix and homogenize the sample, making sure to mix the entire contents of the jar. Additional steps may be needed to homogenize the sample (break up soil clumps, etc.). The sample should be mixed so there is a uniform color and texture. See section 11.4.1.1.

11.6.2.1.**Note:** Sediment samples may contain considerable amounts of organics matter. Ensure that samples are thoroughly mixed. Document the presence of substantial organic matter, shells, etc.

11.6.3. Once mixed, remove the desired mass of sample for the analysis and document accordingly. Recap the jar and return to storage.

11.6.4. The subsample is transferred to an appropriate, labeled container. The sample container is stored in the appropriate refrigerator in sample receiving and any empty sleeve can be stored at room temperature.

11.7. Sediment Samples – Compositing

11.7.1. Thoroughly mix each individual sample as described in 11.6 above.

11.7.2. Combine equal masses from each of the individual samples into a clean stainless steel mixing bowl. The amount used will depend upon the number of analyses to be performed on the composite and/or the amount available. The analyst preparing the composite will document the mass of each individual sample used for the composite, the date and time of compositing, and any other pertinent observations.

Note: Equal masses are used unless otherwise instructed. It may be required to use the entire jar or other measure.

11.7.3. The sample is thoroughly homogenized using a spatula or other utensil and returned to clean glass jars. The sample container is labeled as a composite and with the sample identification, dated, and initialed.

11.7.4. The composite sample and remaining individual samples are returned to storage.

11.7.5. Samples should be received prepared from the field as sample increments. Although unlikely, in cases where proper preparation of increments from *large bulk samples* does not occur in the field, the following steps will be taken.

11.7.5.1. When obtaining sample increments from a large bulk container (bucket, large jar, large bag, etc.) be sure to sample from the center and remove the soil 1–2 inches deep. Using the large spoon or scoop, collect the sample increment according to the work plan. Scoop approximately 30–60 grams into a large, clean container and move on to the next sample increment



location. Be cautious of oversize material, which means more mass may need to be taken from each increment to end with the 30–50 g sub-sample after sieving (a 5 kg field sample may not be uncommon). Increments can be sieved directly into the bucket, or they can be bagged and sieved later.

11.8. Incremental sampling can be accomplished in the laboratory set up to conduct sub-sampling according to the following procedure.

11.9. Basic Procedure for Incremental Sampling Methodology (ISM)

11.9.1. After the 30–50 sample increments have been field collected into a container. (a 5 kg field sample may not be uncommon) air dry the entire sample (all received containers) in aluminum pans rinsed 3 times with DCM (dichloromethane/methylene chloride). Note, if Aluminum is a target analyte of interest then substitute the aluminum pans for glass or stainless steel. Air drying may take 2–4 days with occasional stirring.

11.9.2. The sample is considered air dried when the dried sample weight loss is less than 1 percent in a 4 hour period of air drying. However, due to high variability of laboratory samples, sample dryness should be confirmed by a senior analyst or supervisor prior to going further with the procedure.

11.9.3. Rinse all utensils and equipment with DCM three times prior to use (stainless steel tray, mortar & pestle, 2mm sieve & catch pan, trowel, ISM spatula).

11.9.4. Lightly grind the air dried sample with a mortar & pestle in order to break up dirt and clay chunks (do not size reduce rocks or vegetation) and pass sample through a 2mm sieve.

11.9.5. Weigh the remaining +2mm fraction in an appropriate sized jar and record the weight on the ISM bench sheet. Describe the +2mm fraction on the bench sheet (size of rocks, type of any vegetation, etc).

11.9.6. Weigh and record the weight of the –2mm fraction.

11.9.7. Mix the sample, dump on a DCM-rinsed stainless steel pan, and spread the sample out with a trowel, forming a rectangle no more than 1cm deep.

11.9.8. Divide the sample into a minimum of 30 equal sections (30 to 50 sections is recommended) using the trowel blade.

11.9.8.1. Collect an equal (approximate) amount of sample from each of the sections (based on the chart below) and place into an appropriate sized labeled jar (4oz for organics, 2oz for metals & TS). Scrape the modified flat spatula along the bottom of the tray and pull straight up to make sure all depths and particle sizes are represented in the collection area. Record the exact final weight of sample for each test on the ISM bench sheet and on the jar. Metals tests should be weighed on an analytical balance. All larger amounts can be done on a 2–place balance. The subsampling process must be repeated for each separate analysis to be performed on the sample. The subsampling process must be performed for each individual



QC sample as well. The entire aliquot in the jar will be analyzed (TOC is the exception).

11.9.8.2.If sample volume is sufficient, it is recommended to repeat the process into a second jar to obtain a second backup sample in the event that re-analysis is required.

11.10. Recommended Aliquot Size and QC Requirements:

Test	Air Dried Aliquot	Approximate Amount per Increment	QC Requirement
8151	40.00 g	1.33 g	MS/DMS per 20
8270	30.00 g	1.33 g	MS/DMS per 20
8270 LL	20.00g	0.67 g	MS/DMS per 20
PCB-LL	30.00 g	1.00 g	MS/DMS per 20
PEST-LL	30.00 g	1.00 g	MS/DMS per 20
PCB	15.00 g	0.50 g	MS/DMS per 20
PEST	15.00 g	0.50 g	MS/DMS per 20
PAH	10.00 g	0.50 g	MS/DMS per 20
PAH ULL	20.00g	0.67 g	MS/DMS per 20
8290/Dioxin	15.00 g	0.50 g	MS/DMS per 20
TOC	15.00 g	0.50 g	None
Total Solids	15.00 g	0.50 g	DUP per 10
200 Total Metals	1.0000 g	0.0333 g	DUP/MS per 10
6000 Total Metals	1.0000 g	0.0333 g	DUP/MS per 20
Hg	0.5000 g	0.0167 g	DUP/MS per 20

11.11. Alaska Methods AK102 and AK103 call for the extraction of from 10–30 g of sample material (soil). For MI purposes, the minimum required amount of material per analysis is 30 g.

11.12. Place the remaining –2mm sample into jars labeled as “–2mm archive.” If there are multiple jars, label them as “1 of 3”, “2 of 3”, etc. Give all jars to SMO for barcode labeling. Usually, the –2mm archive and test archive (back-up samples) jars are placed in a freezer, while the +2mm archive and test jars (with QC) are placed on the room temperature shelves.

12. Laboratory Analysis

12.1. The laboratory must analyze the entire contents of the prepared (or submitted) jar. The results may be less defensible if only a subsample or fraction of the jar contents is analyzed.



-
13. Procedure for ISM following State of Hawaii DOH Protocol
- 13.1. Samples requesting the Hawaii DOH procedure require wet and/or dry sieving depending on the test/analytes being aliquoted. Refer to a copy of the Hawaii DOH procedure and/or the Project Manager for details before beginning.
- 13.1.1. Subsample bulk MI samples to be tested for SVOCs, including TPH-D, some PAHs, and Mercury, unstable pesticides, should be subsampled without drying or sieving in order to minimize chemical loss or alteration and meet holding times for analysis.
- 13.1.1.1. If both SVOC and non-volatile PAHs are targeted contaminants of interest then include testing for both in laboratory subsamples collected from the multi-Increment sample prior to drying and sieving
- 13.1.1.2. Refer to Table 2a. of Technical Guidance Manual Notes: Decision Unit and Multi-Increment Sample Investigations , March 2011, State of Hawaii, Department of Health, Reference document number 2011-143-RB. See PMs for a copy of this document.
- 13.2. For wet ISM aliquots, organic tests (SVG/SVM) require a larger aliquot size to accommodate for the extra water content. In most cases, low-level organic tests will require a 40g wet aliquot (max weight capacity for most tests) and normal level tests will require a 20g wet aliquot (double the target dry weight).
- 13.3. Use a separate sample from the wet material and test for soil moisture in order to convert analytical results to dry-weight basis.
- 13.4. Not all samples from Hawaii require the State of Hawaii DOH procedure. See service request and/or verify with the Project Manager.
14. Procedure for ISM on 8330B Explosives
- 14.1. Samples from Ammunition Depots and anywhere except Firing Ranges (not DOD):
- 14.1.1. Follow the basic ISM procedure, except all utensils/pans need rinsed 3 times with Acetonitrile (instead of DCM). Collect a 10.00g aliquot and place in a 4oz amber jar (explosives are sunlight sensitive).
- 14.2. Samples from Firing Ranges:
- 14.2.1. Grinding: For firing ranges, the entire -2mm portion collected from the sieving procedure must be ground to a powder in the shatter box.
- 14.3. 8330B DOD samples:
- 14.3.1. Grinding: For DOD work, the entire -2mm portion collected from the sieving procedure must be ground to a powder in the shatter box prior to proceeding. Note: high-speed milling, such as in the shatter box, can elevate sample temperature due to friction. The thermal stability of the target analytes should be considered when performing this grinding procedure. Method 8330 B specifies a



2-minute (or longer) cool down period between five 60-second grinding intervals to maintain acceptable temperatures and minimize loss of volatile energetic contaminants.

14.3.2. SRM: A SRM (supplied by the Organic LC instrument lab) must be taken through the grinding and ISM procedure (already dry so doesn't need to be air dried or sieved). Shatter box 50g to 100g of the well-mixed SRM, and then make a 10g aliquot after grinding. Place the aliquot in 4oz amber jar. Archive the remaining SRM in an amber jar.

14.3.3. Grinding Blank: Matrix sand blanks (use baked sand) must be ground in the shatter box between each sample and aliquoted following the ISM procedure. The blanks can be ground in equal portions and then recombined at the end to make one sample requiring one ISM aliquot procedure. (Example: To ISM a 200g portion for use in making the final 10g aliquot, divide 200g by the number of samples needing shatter box and grind that amount of matrix sand between each sample. Recombine all ground matrix sand at the end and ISM one 10g aliquot from the 200g of ground matrix sand.) Archive the remaining matrix sand in an amber jar.

15. Analyte-Specific Considerations

15.1. Metals:

15.1.1. It has been proven that grinding can greatly improve the reproducibility for metals analyses. However, erosion of the metals surfaces used in grinding may contribute to a high bias in the samples. It is recommended that the tungsten carbide grinding mill is used when grinding soils in the shatter box thereby limiting the amount of potential bias in the prepared samples.

15.1.2. When grinding soil samples that may potentially contain ores of malleable metals (e.g lead, Copper, tin) be aware that the malleable particles may tend to smear during grinding, and may be lost from the samples to equipment surfaces. This anomaly may bias sample results low, decontamination of equipment surfaces may be difficult and could result in high bias in subsequent samples from carry over.

15.1.3. Reproducibility for Lead analyses in unground, incrementally sampled (IS) samples from small arms firing ranges may have an unacceptable large variability. The large variability for Lead may be due to single particles of Lead between one and two millimeters in diameter being present in only some of the replicate splits. If the end data is to assess risk of accidental ingestion of Lead, precision for the concentration of lead contained in larger particles may be of less interest than the Lead contained in the finer, less than 0.25 mm, fraction. Using a finer mesh sieve (0.25 mm rather than 2 mm) may improve precision and reproducibility. However, sieving unground samples through sieves finer than two millimeters is not appropriate if analyzing for high explosives or propellants. Typical mass sizes for energetic analytes are in particles sizes greater than 0.59 millimeters.

15.1.4. MI samples collected for Arsenic analyses that contain greater than 20 mg/K total Arsenic should be tested for bioaccessible Arsenic. This should be discussed with the project manager. If deemed appropriate, the entire <2mm fraction of the respective samples should be sieved to a ≤ 0.25 mm, representatively sub-sampled



and analyzed for bioaccessible Arsenic using SBRC methodology, 1–2 grams are required.

15.2. Polycyclic Aromatic Hydrocarbons (PAHs)

15.2.1. Currently there is little information in published procedures specific to the laboratory processing of IS samples for PAHs. Laboratory processing of samples per EPA Method 8330 B as described in Section 14.1 is recommended.

15.3. Perchlorate

15.3.1. Currently there is little information in published procedures specific to the laboratory processing of IS samples for Perchlorate. Laboratory processing of samples per EPA Method 8330 B as described in Section 14.1 is recommended. A 10 gram aliquot is required for all propellants and explosives. It is recommended that a 10 gram IS sample should be extracted with 100mL of DI water for Perchlorate analysis by EPA Method 314.

15.4. Vegetation samples

15.4.1. Since vegetation samples often are not amenable to standard mixing and homogenization techniques, or specific sections of the vegetation are targeted, these are handled on a case-by-case basis with instructions from the Project Chemist. The Project Chemist should obtain sample-specific instructions from the client. The Project Chemist will then communicate the specification to the lab personnel using the ALS Form V document for the project. If the client makes reference to specific procedures, methods, or technical references, the Project Chemist will make the document(s) available to the laboratory personnel.

15.5. Paperboard samples

15.5.1. In general, prepare paperboard samples as described below. Project-specific instructions may replace these.

15.5.2. Look at SR, determine the jars you will need:

15.5.2.1.

Metals = 8 oz jar.
Voa = 8 oz jar.
Dioxins = 8 oz jar.
SVG = 32 oz jar.
SVM = 32 oz jar.
PHC (8315) = 8 oz jar.
Gen Chem (not Biology) = 8 oz jar.

15.5.3. Make sample labels according to test and put on appropriate jar.

15.5.4. If FDA Ext is on the Service Request for PHC you will need a 16 oz jar per sample. Do Not Composite into one sample. Each sample is a separate sample.

15.5.5. Prepare the FDA Ext first.



- 15.5.5.1. Cut the sheet of paper into one 10" x 10" square.
- 15.5.5.2. Cut the 10" x 10" into strips at the cut lines 7 ½, 5 and 2 ½.
- 15.5.5.3. Cut the strips at the cut lines 7 ½, 5 and 2 ½. This will make 16 2" squares.
- 15.5.5.4. Put each sample into its own jar and label accordingly. i.e. (1,2 3, etc.) PHC will composite in the lab.

15.5.6. Put one sheet of paper into shredder, run the shredder back and forth to get the entire sample out. Use tongs to remove any remaining sample in bottom of shredder (make sure to turn off before you do this)

15.5.7. Shred equal amounts of each sample (1 or more sheets) to create the composite sample. Homogenize sample thoroughly and aliquot into each jar needed for analysis. Put sample storage on lid of jar.

15.5.8. Dioxins get sent out to Houston. Label lid "Out".

15.5.9. Take all composites to Sample Storage for ALS labeling and shelving.

15.5.10. Update Composites as being done....Open Starlims, double click on Ad Hoc by Test (Under Results entry), highlight samples composited and click the Update to Done button at the top of page. Do not add jars when asked. Just click the X on the right hand corner.

16. QA/QC REQUIREMENTS

16.1. Ongoing QC Samples required for each sample batch (20 or fewer samples) are described in the SOP for Sample Batches and in the determinative SOPs.

17. DATA REDUCTION AND REPORTING

17.1. All compositing and subsampling data must be recorded into the bench records by the analyst. In addition to sample volumes and masses, sample identifications, etc., this should include descriptions of unique samples or sample components.

17.2. It is the supervisor's responsibility to ensure that analytical data is reviewed and to ensure that all quality control requirements have been met.

18. TRAINING

18.1. Refer to the determinative SOPs and the SOP for Documentation of Training for standard procedures.

19. REFERENCES

19.1. *Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples*, U.S. Environmental Protection Agency, EPA/600/R-03/027, November 2003



- 19.2. *Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities*, ASTM D 6323, Annual Book of ASTM Standards, 1999.
- 19.3. Test Methods for Evaluating Solid Waste, EPA SW-846, Final Update III, December 1996.
- 19.4. *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, January, 1996.
- 19.5. Draft Guidance on Multi-Increment Soil Sampling State of Alaska, Department of Environmental Conservation, March 2007.
- 19.6. Technical Guidance manual; Hawai'i Department of Health, Office of Hazard Evaluation and Emergency response, 2009.
- 19.7. Standard operating Procedure, In Vitro Method for Determination of Lead and Arsenic Bioavailability; Solubility/Bioavailability Research Consortium, Document 8601-102.011-0601-1099-RN01.
- 19.8. Figure 1: Multi Incremental Sampling Worksheet

20. CHANGES SINCE THE LAST REVISION

- 20.1. Sec. 11.11: Added 8270 LL and SIM PAH LL. Also corrected aliquot mass for 8270 and PAH.
- 20.2. Sec. 15.2.1: Added section reference for 8330B processing.
- 20.3. Sec. 15.3: Added section reference for 8330B processing.

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

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Effective Date: November 1, 2011

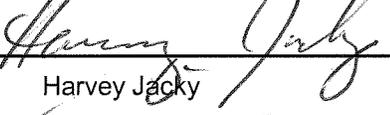
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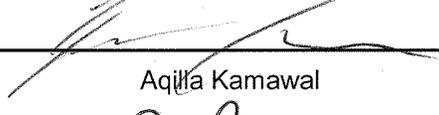
Laboratory Director/Technical Director:  11/11/11
Jeff Grindstaff Date

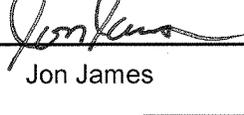
Quality Assurance Program Manager:  11/11/11
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Jon James Date

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Appendices

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2.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is a 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, air, and other material. Columbia Analytical Services operates a network of laboratory facilities located in Arizona, California, Florida, New York, Texas, and Washington.

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. Laboratory management is committed to ensuring the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set forth in the Columbia Analytical Services, Inc. Quality and Ethics Policy Statement, September 2010 (Appendix A) and in this Quality Assurance Manual (QAM). Columbia Analytical Services, Inc. is committed to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. The laboratory also strives for improvement through varying continuous improvement initiatives and projects.

Quality Management Systems are established, implemented and maintained by management. Policies and procedures are established in order to meet requirements of accreditation bodies and applicable programs, such as the Department of Defense (DOD) Environmental Laboratory Accreditation Program, as well as client's quality objectives. Systems are designed so 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. Quality Systems are applicable to all fields of testing in which the laboratory is involved.

Quality Control (QC) procedures are used to continually assess performance of the laboratory and quality systems. Columbia Analytical maintains control of analytical results by adhering to written standard operating procedures (SOPs), using analytical control parameters with all analyses, and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

This QAM is applicable to the facility listed on the title page. The information in this QAM has been organized according to requirements found in the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009), the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, USEPA, 2001; and *General Requirements for the Competence of Testing and Calibration Laboratories*, ISO/IEC 17025:2005.

3.0 PROGRAM DESCRIPTION

The purpose of the QA program at Columbia Analytical 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 Columbia Analytical:

"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."

UNCONTROLLED

3.1 Quality Management Systems

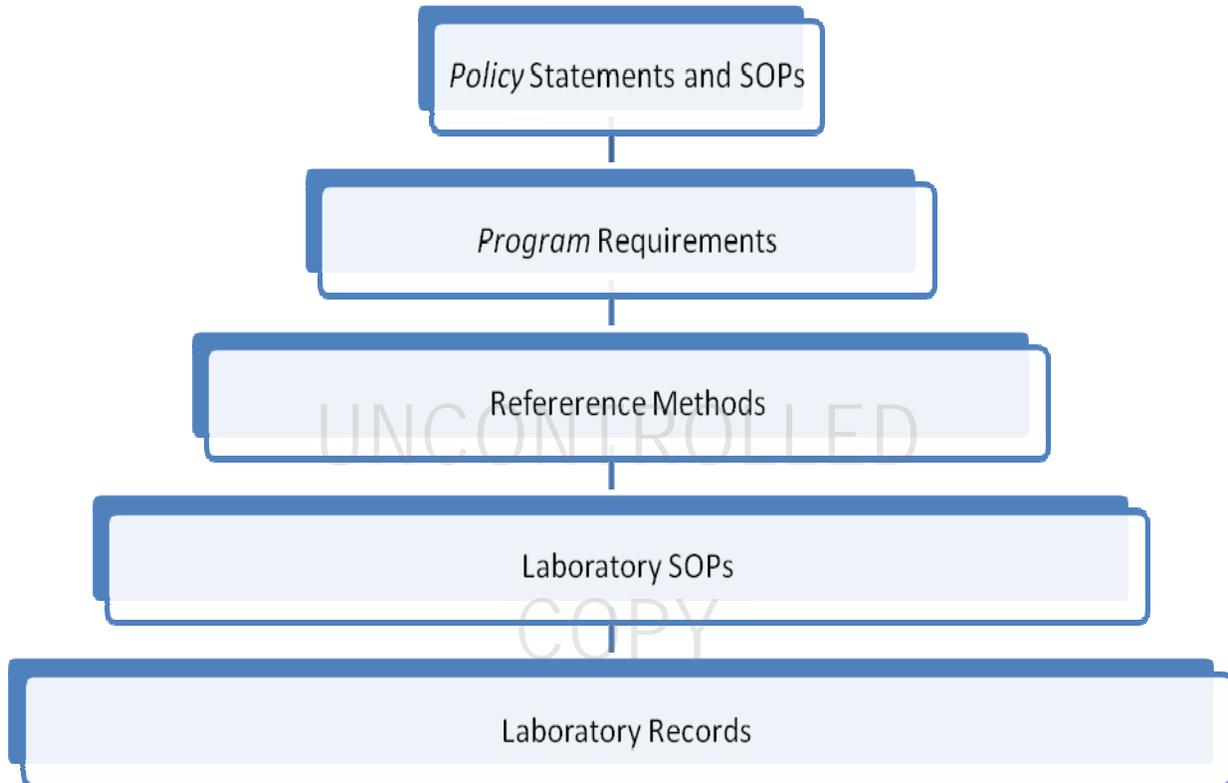
In support of this mission, the laboratory has developed a Quality Management System to ensure all products and services meet our client's needs. The system is implemented and maintained by the Quality Assurance Program Manager (QA PM) with corporate oversight by the Chief Quality Officer (CQO). These systems are based upon ISO 17025:2005 standards, upon which fundamental programs (NELAC 2003, 2009 and DoD QSM) are based. Implementation and documentation against these standards are communicated in corporate policy statements, this QAM, and SOPs. Actual procedures, actions and documentation are defined in both administrative and technical SOPs. Figure 3-1 shows the relationships of the quality systems and associated documentation. Quality systems include:

- Standard Operating Procedures
- Sample Management and Chain of Custody procedures
- Statistical Control Charting
- Standards Traceability
- Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the quality system is assessed in several ways, including:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Feedback
- Internal and External Proficiency Testing

Figure 3-1
Relationships of Quality Management Systems and Documentation



3.2 Facilities, Equipment, and Security

Columbia Analytical features over 45,000 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, Columbia Analytical 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 (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography/High Performance Liquid Chromatography Laboratory
- Gas Chromatography/Mass Spectrometry Laboratory (2)
- Semi-volatile Organics Drinking Water Laboratories (2)
- 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 Systems (2)
- 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 3-2 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.

3.3 Technical Elements of the Quality Assurance Program

The laboratory's technical procedures are based upon procedures published by various agencies or organizations (See Section 17). The Quality Assurance Program provides to the laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained by the QA department. This includes method approvals and audit administration. In addition, internal audits are performed to assess compliance with policies and 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 and SOPs in Appendix F.

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, technical training and preventive 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.

3.4 Operational Assessments and Service to the Client

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, 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 including; on-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

Columbia Analytical utilizes a number of different methods to ensure that adequate resources are available for service demands. Senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating sufficient resources. All Requests for Proposal (RFP) documents are reviewed by the Project Manager 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 Project Plans (QAPPs) if available. Status/production meetings are also conducted regularly with the laboratory and Project Managers to inform the staff of the status of incoming work, future projects, or project requirements.

When a customer requests a modification to an SOP, policy, or standard specification the Project Manager will discuss the proposed deviation with the Client Services Manager, Laboratory Director, and department manager to obtain approval for the deviation. The QA PM may also be involved. All project-specific requirements must be on-file and with the service request upon logging in the samples. The modification or deviation must be documented. A Project-Specific Communication Form, Form V, or similar, may be used to document such deviations.

The laboratory shall afford clients cooperation to clarify the client's request and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients. The laboratory maintains and documents timely communication with the client for the purposes of seeking feedback and clarifying customer requests. Feedback is used and analyzed to improve the quality of services. The *SOP for Handling Customer Feedback (ADM-FDBK)* is in place for these events.

3.5 Document Control and Records

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL)*. The requirements of the SOP apply to all laboratory logbooks (standards, maintenance, run logbooks, etc), certificates of analysis, SOPs, QAMs, quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled Columbia Analytical 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 QA PM, 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. Logbook entries are standardized following the *SOP for Making Entries into Logbooks and onto Benchsheets (ADM-DATANTRY)*. The entries made into laboratory logbooks are reviewed and approved at a regular interval (quarterly).

A records system is used which 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)*.

External documents relative to the management system are managed by the QA PM. To prevent the use of invalid and/or outdated external documents, the laboratory maintains a master list of current documents and their availability. The list is reviewed before making the documents available. External documents are not issued to personnel.

3.6 Subcontracting

Analytical services are subcontracted when the laboratory needs to balance workload or when the requested analyses are not performed by the laboratory. Subcontracting is only done with the knowledge and approval of the client and to qualified laboratories. Subcontracting to another Columbia Analytical laboratory is preferred over external-laboratory subcontracting. Further, subcontracting 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 Corporate Quality Assurance staff is responsible for maintaining a list of qualified subcontract laboratories.

3.7 Procurement

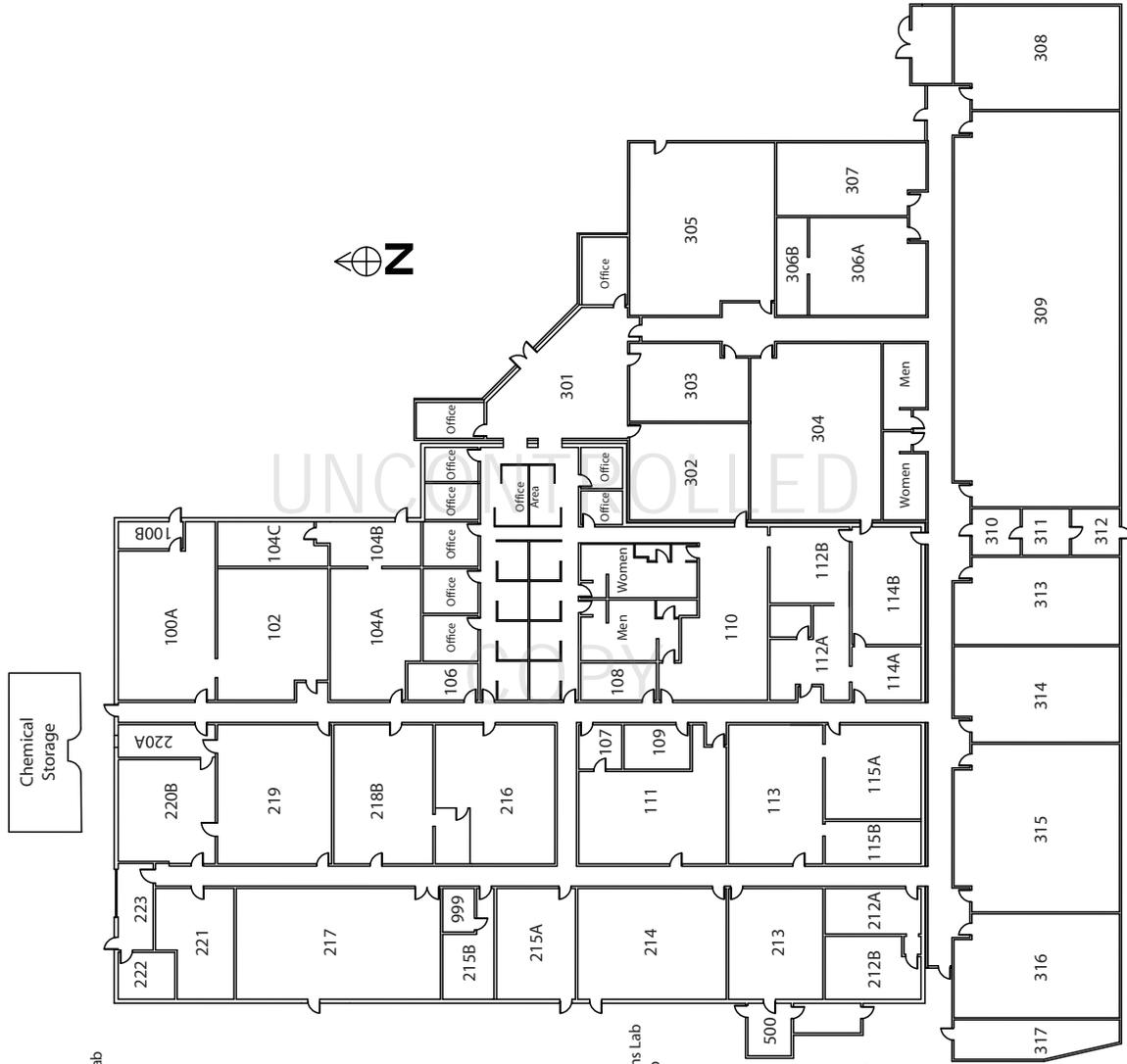
The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The corporate Policy for Standards and Reagents Expiration Dates provides default expiration requirements. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Purchasing and Approval of Vendors (ADM-PUR)*. Also, refer to section 9.4 for a discussion of reference materials.

Receipt procedures include technical review of the purchase order/request to verify that what was received is identical to the item ordered. The laboratory checks new lots of reagents for unacceptable levels of contamination prior to use in sample preservation, sample preparation, and sample analysis by following the *SOP for Checking New Lots of Chemicals for Contamination (ADM-CTMN)*.

3.8 Review of Requests, Tenders and Contracts (Procedures for Accepting New Work)

Requests for new work are reviewed prior to signing any contracts or otherwise agreeing to perform the work. The specific methods to be used are agreed upon between the laboratory and the client. A capability review is performed to determine if the laboratory has or needs to obtain certification to perform the work, to determine if the laboratory has the resources (personnel, equipment, materials, capacity, skills, expertise) to perform the work, and if the laboratory is able to meet the client's required reporting and QC limits. The results of this review are communicated to the client and any potential conflict, deficiency, lack of appropriate accreditation status, or concerns of the ability to complete the client's work are resolved. Any differences between the request or tender and the contract shall be resolved before any work commences. The client should be notified at this time if work is expected to be subcontracted. Each contract shall be acceptable both to the laboratory and the client. Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work. If a contract needs to be amended after work has commenced, the contract review process is repeated and any amendments are communicated to all affected personnel. Changes in accreditation status affecting ongoing projects must be reported to the client.

**Figure 3-2
Columbia Analytical Services-Kelso Laboratory Floor Plan**



- | | |
|---------------|---|
| Room # | Semivolatile Organics GC Instrument Lab |
| 100A | Electrical Room |
| 100B | Semivolatile Organics GC Office |
| 102 | Organics LC Labs and Offices |
| 104 A - C | Deionized Water-System Room |
| 106 | Oven Room |
| 107 | Zero Headspace Extraction Lab |
| 108 | Storeroom |
| 109 | Lunch Room |
| 110 | General Chemistry Lab |
| 111 | Microbiology Lab |
| 112A | Copy Center |
| 112B | ICP-MS Lab |
| 113 | Grain Size |
| 114A | Total Solids/Oven Room |
| 114B | ICP Lab |
| 115A | Metals Reporting |
| 115B | Mercury and Flame AA Lab |
| 212A | Low Level Mercury Lab |
| 212B | General Chemistry Lab |
| 213 | General Chemistry Lab |
| 214 | Drinking Water Lab |
| 215A | Information Technology |
| 215B | Data Review & Storage |
| 216 | Sample Storage |
| 217 | Volatiles Organics Lab |
| 218 | Semivolatile Organics GC/MS Lab |
| 219 | Semivolatile Organics GC/MS Office |
| 220B | Semivolatile Organics GC/MS Extractions Lab |
| 221 | Drinking Water Sample Preparation Lab |
| 222 | Purchasing Office |
| 223 | General Receiving |
| 301 | Reception Lobby |
| 302 | Lunch Room |
| 303 | Conference Room |
| 304 | Sample Storage |
| 305 | Sample Receiving |
| 306A | Pharmaceutical Metals |
| 306B | Pharmaceutical Microbiology |
| 307 | Pharmaceutical Instrument Lab |
| 308 | Pharmaceutical General Chemistry Lab |
| 309 | Organics Sample Preparation Lab |
| 310 | Communication Room |
| 311 | Electrical Room |
| 312 | Fire Room/Water-System |
| 313 | Glass Wash Room |
| 314 | Clean Room |
| 315 | Metals Digestion Room |
| 316 | Tissue Preparation |
| 317 | Mechanical |
| 500 | Janitorial Room |
| 999 | Computer Room |

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4.0 PROFESSIONAL CONDUCT, DATA INTEGRITY, AND ETHICS

One of the most important aspects of the success of CAS is the emphasis placed on the integrity of the data provided and the services rendered. This success is reliant on both the professional conduct of all employees within CAS as well as established laboratory practices. All personnel involved with environmental testing and calibration activities must familiarize themselves with the quality documentation and implement the policies and procedures in their work.

4.1 Professional Conduct

To promote quality, CAS requires certain standards of conduct and ethical performance among employees. The following examples of documented 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.
- It is the responsibility of all Columbia Analytical employees to safeguard sensitive company information, client data, records, and information; and matters of national security concern should they arise. 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 sign a confidentiality agreement upon hire to protect the company and client's confidentiality and proprietary rights.

4.2 Prevention and Detection of Improper, Unethical or Illegal Actions

It is the intention of CAS to proactively prevent and/or detect any improper, unethical or illegal action conducted within the laboratory. This is performed by the implementation of a program designed for not only the detection but also prevention. Prevention consists of educating all laboratory personnel in their roles and duties as employees, company policies, inappropriate practices, and their corresponding implications as described here.

In addition to education, appropriate and inappropriate practices are included in SOPs such as manual integration, data review and specific method procedures. Electronic and hardcopy data audits are performed regularly, including periodic audits of chromatographic electronic data. Requirements are described in the Policy for Internal Quality Assurance Audits and details are listed in laboratory administrative SOPs. All aspects of this program are documented and retained on file according to the company policy on record retention.

The CAS Employee Handbook also contains information on the CAS ethics and data integrity program, including mechanisms for reporting and seeking advice on ethical decisions.

4.3 Laboratory Data Integrity and Ethics Training

Each employee receives in-depth (approximately 6-8 hour) core Data Integrity/Ethics Training. New employees are given a QA and Ethics orientation within the first month of hire, followed by the core training within 1 year of hire. On an ongoing basis, all employees receive semi-annual ethics refresher training. Topics covered are documented in writing and all training is documented. It is the responsibility of the QA PM to ensure that the training is conducted as described.

Key topics covered are the organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues and record keeping. Training includes discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation.

Trainees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, or civil/criminal prosecution.

The training session includes many concepts and topics, numerous examples of improper actions (defined by DoD as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional), legal and liability implications (company and personal), causes, prevention, awareness, and reporting mechanisms.

4.4 Management and Employee Commitment

Columbia Analytical 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 Columbia Analytical Employee Handbook. This includes:

- CAS Open Door Policy (CAS Employee Handbook) – Employees are encouraged to bring any work related problems or concerns to the attention of local management or their Human Resources representative. However, depending on the extent or sensitivity of the concern, employees are encouraged to directly contact any member of upper management.

- CAS Corporate Ethics Point Program – An anonymous and confidential reporting system available to all employees that is used to communicate misconduct and other concerns. The program shall help minimize negative morale, promote a positive work place, and encourage reporting suspected misconduct without retribution. Associated upper management is notified and the investigations are documented.
- Use of flexible work hours. Within reason and as approved by supervisors, employees are allowed flexible work hours in order to help ease schedule pressures which could impact decision-making and work quality.
- Operational and project scheduling assessments are continually made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads. Procedures for subcontracting work are established, and within the Columbia Analytical laboratory network additional capacity is typically available for subcontracting, if necessary.
- Gifts and Favors (CAS Employee Handbook) – To avoid possible conflict of interest implications, employees do not receive unusual gifts or favors to, nor accept such gifts or favors from, persons outside the Company who are, or may be, in any way concerned with the projects on which the Company is professionally engaged.

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All employees are required to sign and adhere to the requirements set forth in the Columbia Analytical *Confidentiality and Conflicts of Interest Employee Agreement* and the Columbia Analytical *Commitment to Excellence in Data Quality* (see Appendix A).

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5.0 ORGANIZATION AND RESPONSIBILITIES

The Columbia Analytical/Kelso staff, consisting of approximately 150 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. All employees share the responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 5-1 lists the Columbia Analytical 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 (Location) 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 **Quality Assurance Program Manager (QA PM)** has the authority and responsibility for implementing, maintaining, and improving the quality system. This includes coordination of QA activities within the laboratory, ensuring that all personnel understand their contributions to the quality system, ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system, evaluating the effectiveness of training; and monitor trends and continually improve the quality system. Audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews can all be used to support quality system implementation. The QA PM is responsible for ensuring compliance with NELAC standards (and ISO, DoD QSM, etc. as applicable). The QA PM works with laboratory staff to establish effective quality control and assessment plans and has the authority to stop work in response to quality problems. The QA PM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and ensuring the annual review of technical SOPs; maintaining QA records such as metrological records, archived logbooks, PT results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; and performing internal QA audits.

The QA PM reports directly to the Laboratory Director and also reports indirectly to the Chief Quality Officer. It is important to note that when evaluating data, the QA PM 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 Columbia Analytical laboratories. The CQO is responsible for oversight of QA PMs regulatory compliance efforts (NELAC, ISO, DOD, etc). The CQO performs annual internal audits at each laboratory; maintains a database of laboratory certification/accreditation programs; approves company-wide SOPs; maintains a database of approved subcontract laboratories; provides assistance to the laboratory QA staff and laboratory managers; prepares a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA PM, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Metals Department Manager (for the Laboratory Director) and the CQO or Laboratory Director (for the QA PM).
- In the event that work is stopped in response to quality problems, only the Laboratory Director or Quality Assurance Program Manager has the authority to resume work.
- 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 Columbia Analytical's EH&S Director.
- The **Client Services Manager** is responsible for the Client Services Department defined for the laboratory (i.e. Project Managers, electronic deliverables, etc.) 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. Sample management handles all 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 Manager** is a scientist assigned to each client to act as a technical liaison between the client and the laboratory. The Project Manager is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the Columbia Analytical laboratory and administrative staff to ensure that client-specific needs are understood and that the services Columbia Analytical 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 QC program meeting department needs. Each **Department Manager and Supervisor** has the responsibility to ensure that QC functions are carried out as planned, and to guarantee the production of high quality data. Managers and bench-level supervisors monitor the day-to-day operations to ensure that productivity and data quality objectives are met. A 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 is 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.

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**Table 5-1
Summary of Technical Experience and Qualifications**

Personnel	Years of Experience	Project Role
Jeff Grindstaff, B.S.	22	Laboratory Director
Julie Gish, M.S.	19	Quality Assurance Program Manager
Lynda Huckestein, B.S.	22	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	21	Metals Department Manager
Harvey Jacky, B.S.	22	General Chemistry Department Manager
Aqilla Kamawal, B.A.	11	Semi-Volatile Organics Department Manager
Jon James, B.A.	20	HPLC, GC/MS Organics Department Manager
Christina Kerksieck, B.S.	3	Microbiology Technical Manager
Elieen Arnold, B.A.	29	Environmental Health and Safety Officer
Mike Sullivan, B.S.	11	Information Technology
Jeff Christian, B.S.	32	Chief Operations Officer
Lee Wolf, B.S.	26	Chief Quality Officer/Quality Assurance Director

6.0 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 computing equipment.

6.1 Software Quality Assurance Plan

Columbia Analytical 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 this plan are policies for software validation and control.

6.2 IT Support

The local Columbia Analytical 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, Columbia Analytical corporate IT provides support for network-wide systems. Columbia Analytical also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

6.3 Information Management Systems

Columbia Analytical has various systems in place to address specific data management needs. The Columbia Analytical Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request. Included on the service request is a summary of client information, sample identification, required analyses, work instructions, deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003[®] domain). 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. The server is also used for 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.

6.4 Backup and Security

Columbia Analytical 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- Columbia Analytical 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. Columbia Analytical 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.

7.0 SAMPLE MANAGEMENT

7.1 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. Columbia Analytical 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

Columbia Analytical 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, IV for hazardous waste samples; 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; EPA 40CFR parts 136 and 141; 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 7-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to 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.

Columbia Analytical routinely provides sample containers with appropriate preservatives for our clients. Containers are purchased as precleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample 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 7-1 shows the chain-of-custody form routinely used at Columbia Analytical 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 Columbia Analytical. 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. Columbia Analytical keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. Columbia Analytical also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When Columbia Analytical 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 (SMO-GEN) for maintaining the samples' chain of custody, packaging and shipment. Dry ice gel ice is the only temperature preservative used by Columbia Analytical, unless otherwise specified by the client or receiving laboratory.

7.2 Sample Receipt and Handling

Standard Operating Procedures (SMO-GEN) 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.

Once samples are delivered to the Columbia Analytical sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 7-2 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 Manager and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample container 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 Manager for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. Columbia Analytical has five walk-in cold storage units which house the majority of sample containers received at the laboratory. In addition, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. Columbia Analytical also has nine sub-zero freezers capable of storing samples at -10 to -30° C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored real time with an electronic temperature monitoring system.

Columbia Analytical adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor 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. To document holding time compliance, 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 holding time compliance can be demonstrated. 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.

7.3 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 7-1 is a copy of the chain-of-custody form routinely used at Columbia Analytical.

Facility security and access is important in maintaining the integrity of samples received at Columbia Analytical-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 Columbia Analytical facility is equipped with an alarm system and Columbia Analytical 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)*.

7.4 Project Setup

The analytical method(s) 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 Manager will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Manager ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the service request. To communicate and specify project-specific requirements, a Tier V form (Figure 7-3) is used and accompanies the service request form.

**Table 7-1
Sample Preservation and Holding Times**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert (SM 9223)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ^d	6-24 hours ^e
Coliform, Fecal and Total (SM 9221, 9222D)	W, S, DW	P,G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ^d	6-24 hours ^e
Fecal Streptococci (SM 9230B)	W	P,G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ^d	6-24 hours ^e
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days ^{EPA}
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days ^{EPA}
Ammonia (SM 4500NH ₃)	W, DW	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Biochemical Oxygen Demand (SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W, S	P,G	Cool, 4°C	28 days
Chlorine, Total Residual (SM 4500 Cl F)	W,S	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500CN E,G)	W, S,DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Inorganic Tests				
Cyanide, Weak Acid Dissociable (SM 4500CN I)	W,S	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron (CAS SOP)	W, D	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0, SM 4500 F-C)	W,S	P,G	Cool, 4°C	28 days
Fluoride (EPA 9056)	W,S	P,G	Cool, 4°C	Analyze immediately
Formaldehyde (ASTM D6303)	W	G Amber	Cool, 4°C	48 hours
Hardness (SM 2340C)	W, DW	P,G	HNO ₃ to pH<2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrocellulose	S	G	Cool, 4°C	28 days
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, S	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	48 hours
Nitrate (EPA 9056)	W,S	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, S	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	48 hours
Nitrite (EPA 9056)	W,S	P,G	Cool, 4°C	Analyze immediately
Orthophosphate (SM 4500 P-E)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Inorganic Tests				
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Phenolics, Total (EPA 420.1,9056)	W, S	G Amber	Cool, 4°C, H ₂ SO ₄ to pH<4	28 days
Perchlorate (EPA 314.0)	W, DW,S	P,G	Protect from temp. extremes	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Residue, Total (SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS) (SM2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500SiO ₂ C)	W	P Only	Cool, 4°C	28 days
Specific Conductance(SM 2510 B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W, S	P,G	Cool, 4°C	28 days
Sulfide (SM 4500S ₂ D)	W	P,G	Cool, 4°C, Add Zinc Acetate,plus Sodium Hydroxide to pH>9	7 days
Sulfide (SM 4500S ₂ F)	W	P,G	Cool, 4°C, Add Zinc Acetate,plus Sodium Hydroxide to pH>9	7 days
Sulfide (9030/934)	W, S	P,G	Cool, 4°C, Add Zinc Acetate,plus Sodium Hydroxide to pH>9	7 days
Sulfides, Acid Voaltile	S	G	Cool, 4°C	14 days
Sulfite (SM 4500SO ₃ B)	W	P,G	None Required	24 hours
Surfactants (MBAS) (SM 5540 C)	W	P,G	Cool, 4°C	48 hours

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Inorganic Tests				
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H ₂ SO ₄ or HCL to pH<2	28 days
Organic Carbon, Total (9060 & SM 5310 C)	W	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Organic Carbon, Total (ASTM-D4129)	S	P,G	Cool, 4°C	28 days
Organic Halogens, Total (EPA 9020)	W	G, Teflon-Lined Cap	Cool, 4°C, H ₂ SO ₄ to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO ₃ to pH<2	6 months

COPY

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Metals				
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4°C	24 hours
Metals (200.7, 200.8, 200.9, 6010, 6020)	W,DW	P,G	HNO ₃ to pH<2	6 months
Metals (200.7, 200.8, 200.9, 6010, 6020)	S	G, Teflon-Lined cap	Cool, 4°C	6 months
Mercury (EPA 245.1, 7470, 7471)	W, DW	P,G	HNO ₃ to pH<2	28 days
Mercury (7471)	S	P,G	Cool, 4°C	28 days
1631E	W	F	Cool, 4°C, HCl or H ₂ SO ₄ to pH<2	90 days
1631E	S	F	Freeze < -15°C	1 Yr
Methyl Mercury 1630	W,S,T	F	HCL to pH<2	6 months
Arsenic Species 1632	W	G	HCL to pH<2, Cool < 4°C	28 days

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Gasoline Range Organics (8015, NWTPH-Gx)	W	G, Teflon-Lined, Septum Cap	Cool, 4°C, HCl to pH<2, No headspace	14 days
Gasoline Range Organics (8015, NWTPH-Gx)	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
Purgeable Halocarbons (624, 8021, 8260)	W	G, Teflon-Lined, Septum Cap	No Residual Chlorine Present: HCl to pH<2, Cool, 4°C, No Headspace	14 days
Purgeable Halocarbons (624, 8021, 8260)	W	G, Teflon-Lined, Septum Cap	Residual Chlorine Present: 10% Na ₂ S ₂ O ₃ , HCl to pH<2, Cool, 4°C	14 days
Purgeable Halocarbons (8021, 8260)	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
Purgeable Halocarbons (8021, 8260)	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4C	48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Halocarbons (8021, 8260)	S	Method 5035	Sodium Bisulfate Cool, 4°C	48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	W	G, Teflon-Lined, Septum Cap, No Headspace	No Residual Chlorine Present: HCl to pH<2, Cool, 4°C, No Headspace	14 days
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	W	G, Teflon-Lined, Septum Cap, No Headspace	Residual Chlorine Present: 10% Na ₂ S ₂ O ₃ , HCl to pH<2, Cool 4°C	14 days

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4C	48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	S	Method 5035	Sodium Bisulfate Cool, 4°C	48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile (624, 8260)	W	G, Teflon-Lined, Septum Cap	Adjust pH to 4-5, Cool, 4°C, No headspace	7 days
EDB and DBCP (EPA 8260)	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg Na ₂ S ₂ O ₃ , No Headspace	28 days
Vinyl chloride, styrene, 2-chloroethyl vinyl ether (8260)	W	G, Teflon-Lined, Septum Cap	Cool, 4°C, Minimize Headspace	7 days

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Nonyl Phenols	W	G, Teflon-Lined Cap	H ₂ SO ₄ to pH<2, Cool, 4°C	28 days
Organotins (CAS SOP)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 ^f days until extraction; 40 days after extraction
Otto Fuel	W	G, Teflon-Lined Cap	Cool, 4°C	7 ^f days until extraction; 40 days after extraction
Resin and Fatty Acids (NCASI 85.02)	W	G, Teflon-Lined Cap	NaOH to pH >10, Cool, 4°C ^g	30 days until extraction; 30 days after extraction
Methanol in Process Liquid NCASI 94.03	L	G, Teflon-Lined Cap	Cool, 4°C	30 days
HAPS – Condensates NCASI 99.01		G, Teflon-Lined Cap	Cool, 4°C	14/30 days
HAPS – Impinger/Canisters NCASI 99.02			Cool, 4°C	21 days
Perfluorinated Compounds HPLC/MS/MS	W	P	Cool, 4°C	14 days until extraction; 40 days after extraction
PBDE/PBB – ROHS GC/MS	W,S,T	G	Cool, 4°C	40 days after extraction
Pharma Personal Care Products 1694	W	Amber G, Teflon-Lined Cap	Cool, < 6°C	7 ^f days until extraction; 30 days after extraction
Nitroaromatics and Nitramines 8330B	W,S	G, Teflon-Lined Cap	Cool, 4°C	S 14, W 7 days until extraction; 40 days after extraction
Nitroaromatics/Nitoramines HPLC/MS/MS	W,S,T	G	Cool, 4°C Tissues < -10 C	S 14, W 7 days until extraction; 40 days after extraction
Organic acids HPLC/MS/MS	W	G, Teflon-Lined, Septum Cap	H ₂ SO ₄ to pH<2, Cool, 4°C	14 days

Table 7-1 (continued)
Sample Preservation and Holding Times^a

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 ^f days until extraction;40 days after extraction
Alcohols and Glycols (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7 ^f days until extraction;40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7 ^f days until extraction;40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7 ^f days until extraction;40 days after extraction
Chlorinated Herbicides (EPA 8151)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7 ^f days until extraction;40 days after extraction
Chlorinated Phenolics (EPA 1653)	W	G, Teflon-Lined Cap	H2SO4 to pH<2, Cool, 4°Cg	30 days until extraction; 30 days after extraction
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Darkg	7 ^f days until extraction;40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081, 8082, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 ^f days until extraction;40 days after extraction
Organophosphorus Pesticides (EPA 8141, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Darkg	7 ^f days until extraction;40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7 ^f days until extraction;40 days after extraction

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined, Septum cap	Ascorbic Acid, HCl to pH _≤ 2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	DW	G, Teflon-Lined, Septum cap	Cool, 4°C, 3 mg Na ₂ S ₂ O ₃ , No Headspace	14 days
Carbamates, Carbamoyloximes (EPA 531.1)	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH<3; 80 mg/L Na ₂ S ₂ O ₃ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/40mL NaS; Cool, <6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH _≤ 2; Cool 4°C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L Na ₂ S ₂ O ₃ if Res.Cl. Cool 4°C	7 days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate (EPA 547)	DW	G, Amber, Teflon-Lined Cap	100 mg/L Na ₂ S ₂ O ₃ , Cool, 4°C	14 days
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH ₄ Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH _≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Nitrosoamines (EPA 521)	DW	G, Amber, Teflon-Lined Cap	Dechlorinate at collection ^g	14 days until extraction; 28 days after extraction
Selected Pesticides and Flame Retardants (EPA 527)	DW	G, Amber, Teflon-Lined Cap	See Method, Cool, 4°C	14 days until extraction; 28 days after extraction

**Table 7-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark ^g	14 days until TCLP ext'n;
			TCLP extract: Cool, 4°C, Store in Dark ^g	7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C	14 days until TCLP ext'n;
			TCLP extract: Cool, 4°C	7 days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C	14 days until TCLP ext'n;
			TCLP extract: Cool, 4°C	7 days until extraction; 40 days after extraction
Mercury (EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C	28 days until extraction
			TCLP extract: HNO ₃ to pH<2	28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C	180 days until extraction;
			TCLP extract: HNO ₃ to pH<2	14 days until TCLP ext'n;
Volatile Organics (EPA 1311/8260)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Minimize Headspace	14 days until TCLP ext'n;
			Extract: Cool 4°C, HCL to pH,2, No Headspace	14 days after extraction

- a For EPA SW-846 methods the method number is listed generically, without specific revision suffixes.
- b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
- c P = Polyethylene; G = Glass, F- Fluoropolymer
- 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 7-2

**Columbia Analytical Services, Inc.
Cooler Receipt and Preservation Form**

PC _____

Client / Project: _____ Service Request *KII* _____

Received: _____ Opened: _____ By: _____ Unloaded: _____ By: _____

1. Samples were received via? *Mail Fed Ex UPS DHL PDX Courier Hand Delivered*
2. Samples were received in: (circle) *Cooler Box Envelope Other* _____ *NA*
3. Were custody seals on coolers? *NA Y N* If yes, how many and where? _____
If present, were custody seals intact? *Y N* If present, were they signed and dated? *Y N*

Cooler Temp °C	Temp Blank °C	Thermometer ID	Cooler/COC ID	NA	Tracking Number	NA	Filed

7. Packing material used. *Inserts Baggies Bubble Wrap Gel Packs Wet Ice Sleeves Other* _____
8. Were custody papers properly filled out (ink, signed, etc.)? *NA Y N*
9. Did all bottles arrive in good condition (unbroken)? *Indicate in the table below.* *NA Y N*
10. Were all sample labels complete (i.e analysis, preservation, etc.)? *NA Y N*
11. Did all sample labels and tags agree with custody papers? *Indicate major discrepancies in the table on page 2.* *NA Y N*
12. Were appropriate bottles/containers and volumes received for the tests indicated? *NA Y N*
13. Were the pH-preserved bottles (*see SMO GEN SOP*) received at the appropriate pH? *Indicate in the table below* *NA Y N*
14. Were VOA vials received without headspace? *Indicate in the table below.* *NA Y N*
15. Was C12/Res negative? *NA Y N*

Sample ID on Bottle	Sample ID on COC	Identified by:

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broke	pH	Reagent	Volume added	Reagent Lot Number	Initials	Time

Notes, Discrepancies, & Resolutions: _____

8.0 ANALYTICAL PROCEDURES

Columbia Analytical employs methods and analytical procedures from a variety of external 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, EPA 40CFR parts 136 and 141, 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 17.0. 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 Columbia Analytical is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix G. Further details are described below.

8.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

Columbia Analytical maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements as described in the *SOP for Preparation of Standard Operating Procedures*. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Program Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA PM 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 *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

8.2 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 Manager 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 Manager 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.

8.3 Modified Procedures

Columbia Analytical 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.

8.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that Columbia Analytical 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
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 between the start of processing of the first and last sample of the batch.
- 7) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.
- 8) (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.
- 9) 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).
- 10) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.
- 11) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.
- 12) 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.

8.5 Specialized Procedures

Columbia Analytical 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 trace-level Mercury and Methylmercury analyses, reductive precipitation metals analysis, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs).

8.6 Sample Cleanup

Columbia Analytical commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods (3620, 3630, 3640, 3660, and 3665) 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.

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9.0 CALIBRATION PROCEDURES

All equipment and instruments used at Columbia Analytical 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. 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. Records are maintained to provide traceability of reference materials.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standards. All analytical measurements generated at Columbia Analytical are performed using materials where possible 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. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective; is taken out of service until it is repaired. When an instrument is taken out of service, an *Out of Service* sign is placed by the laboratory on the instrument. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

9.1 Temperature Control Devices

Temperatures are monitored and recorded each day for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators/freezers. Temperatures are recorded in either laboratory logbook or through Check Point® Wireless Monitoring System. During weekends and holidays a min/max thermometer may be used.

Laboratory records contain the recorded temperature, 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 is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is

recertified by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standard on an annual basis.

9.2 Analytical Balances

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 bracketing 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 procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC. The weights are recertified using NIST traceable standards by an accredited metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by an accredited metrology organization.

9.3 Water Purification Systems

Columbia Analytical uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Following a written SOP, the status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily in a bound record book. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked on a daily basis at a point downstream of the purification system at a tap in the laboratory.

9.4 Source and Preparation of Standards and Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors where possible have fulfilled the requirements for 9001 certification and/or are ISO 17025 accredited. Columbia Analytical Service 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 Reagent Login and Tracking* (SOP ADM-RTL). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

9.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards whose concentrations fall within the instruments linear range. 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.).

9.6 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.

9.7 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.

9.8 GC/MS Systems

All GC/MS instruments are calibrated at multiple 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. 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).

9.9 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)*.

9.10 LC/MS Systems

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL)*.

9.11 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 Columbia Analytical include total phenolics, phosphates, surfactants and tannin-lignin.

9.12 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 Columbia Analytical acceptance limits are used to evaluate the calibration curve prior to sample analysis.

9.13 Discrete Auto-Analyzer (automated absorbance analysis)

A minimum of five standards and a blank are used to calibrate the instrument. Initial calibration points cannot be “dropped” from the resulting calibration curve. Method specific acceptance limits are used to evaluate the calibration curve prior to sample analysis.

9.14 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be “dropped” from the resulting calibration curve. A correlation coefficient of ≥ 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 drinking water inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

9.15 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, *Environmental Resource Associates* QC samples (or equivalent) and duplicates.

9.16 Ion-selective electrode

The method-prescribed numbers of standards 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.

9.17 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP Checking Volumetric Labware (ADM-VOLWARE)*. 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.

9.18 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.

10.0 QUALITY CONTROL

A primary focus of Columbia Analytical's QA Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. Columbia Analytical 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 test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

10.1 Quality Control Objectives

10.1.1 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the 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 sample material may be obtained from an outside source or may be prepared in the laboratory. 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 may be used to meet this requirement, provided acceptance criteria is met.

10.1.2 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).

Columbia Analytical 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.

10.1.3 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.

10.1.4 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 reviewed each year and may be updated if 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 QA PM. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. 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. Procedures for establishing control limits are found in the *SOP for Control Limits* (ADM-CTRL_LIM).

10.1.5 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. Columbia Analytical 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* (GEN-SUBS) and the *SOP for Tissue Sample Preparation* (MET-TISP). Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

10.1.6 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 objective is to generate data of known quality with the highest level of comparability, completeness, and usability. 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 Columbia Analytical or project-specified data qualifiers.

10.2 Method Detection Limits, Method Reporting Limits, and Limits of Detection/Quantitation

Method Detection Limits (MDL) for methods performed at Columbia Analytical/(Location) is determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation (ADM-MDL)*, which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD 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. limit of quantitation- LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

10.3 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.

10.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ 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.

10.3.2 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.

10.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of 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.

10.3.4 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.

10.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in for calibration. Once it is determined that there is no defect or error in 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.

10.3.6 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.

10.3.7 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.

10.3.8 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.

$$\text{Recovery (\%)} = (M/T) \times 100$$

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

10.3.9 Laboratory Control Samples

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.

$$\text{Recovery (\%)} = (M/T) \times 100$$

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

10.3.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

10.3.11 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:

$$\text{Recovery (\%)} = (S - A) \times 100 \div T$$

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.

10.3.12 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:

$$\text{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

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.

10.3.13 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.

10.3.14 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.

10.3.15 Control Charting

The generation of control charts is routinely performed at Columbia Analytical. 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).

10.3.16 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at Columbia Analytical undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at Columbia Analytical; 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.

11.0 DATA PROCESSING, VALIDATION, AND REPORTING

Columbia Analytical reports the analytical data produced in its laboratories to the client via the certified analytical report. 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.

11.1 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 using 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 (ADM-DREV)*.

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 (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 (ADM-INT)*.

11.2 Confirmation Analysis

11.2.1 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, 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.

11.2.2 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.

11.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and 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, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. 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 NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM 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.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL ($< \frac{1}{2}$ 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) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are 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 as 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. Efforts are made to meet the project MRL's including alternative analysis.
- **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. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP for *Confirmation Procedure for GC and HPLC Analysis (SOC-CONF)*. If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including additional cleanup.
- **Surrogate Results (Organic)** – The percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, 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** – The LCS 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. Samples associated with the ‘out of control’ LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 are used to determine if the LCS is effective in validating the analytical system and the associated samples.
- **Matrix Spike Results** – The MS percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results are 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.

11.4 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 analyst or chemist. Prior to release of the report to the client, the Project Manager 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 scanned and archived by service request number. Columbia Analytical 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 (ADM-RG)* addresses the flagging and qualification of data. The Columbia Analytical-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 Manager to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Manager 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 Manager accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the client.

11.5 Documentation

Columbia Analytical 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 (ADM-ARACH)*.

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.

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.

11.6 Deliverables

In order to meet individual project needs, Columbia Analytical provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 11-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) the following specialized deliverables:

- DoD QSM – Army Corp of Engineers, Air Force Center for Environmental Excellence, Navy
- Drinking water - State specific formats

When requested by the client or relevant to the validity of reported results, the estimation of measurement uncertainty will be provided to a client or regulatory agency. How the uncertainty will be reported may be dictated by the client's reporting specifications. Procedures for determining and reporting uncertainty are given in the *SOP for Estimation of Uncertainty of Measurements*.

When requested, Columbia Analytical provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. Columbia Analytical is capable of generating EDDs with many different formats and specifications. 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.

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**Table 11-1
Descriptions of Columbia Analytical Standard Data Deliverables**

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Chain of custody documents and sample/cooler receipt documentation
3. Sample analytical results
4. Method blank results
5. Surrogate recovery results and acceptance criteria for applicable organic methods
6. Dates of sample preparation and analysis for all tests
7. Case narrative - **optional**

Tier II. 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. Laboratory Control Sample result(s) with calculated recovery and including associated acceptance criteria
4. Case narrative - **optional**

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative - **required**
2. Summary forms for all associated QC and Calibration parameters, with associated control criteria/acceptance limits

Note: Other summary forms specified in QAPPs or project/program protocols, or those related to specialized analyses such as HRGC/MS will be included.

Tier IV. Full Data Validation Package.

1. All raw data associated with the sample analysis, including but not limited to:
 - a. Preparation and analysis bench sheets and instrument printouts,
 - b. For organics analyses, all applicable chromatograms, spectral, confirmation, and manual integration raw data. For GC/MS this includes tuning results, mass spectra of all positive hits, and the results and spectra of TIC compounds when requested.
 - c. QC data,
 - d. Calibration data (initial, verification, continuing, etc),
 - e. Calibration blanks or instrument blanks (as appropriate to method).
2. If a project QAPP or program protocol applies, the report will be presented as required by the QAPP.

12.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of Columbia Analytical/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.

12.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of Columbia Analytical/Kelso are conducted regularly by various regulatory agencies and clients. Appendix G lists the certification and accreditation programs in which Columbia Analytical/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of Columbia Analytical/Kelso are conducted regularly under the direction of the Quality Assurance Program Manager. The internal audit procedures are described in the *SOP for Internal Audits (ADM-IAUD)*. 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.
- Technical/method audits – minimum of 3 per quarter
- Hardcopy report audits – minimum of 2 per quarter.
- Chromatographic electronic data audits – 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 a defined timeframe. 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 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, 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.

12.2 Performance Audits

Columbia Analytical/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. General procedures for these analyses are described in the SOP for *Proficiency Sample Testing Analysis (ADM-PTS)*. Columbia Analytical 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 QA PM, Laboratory Director, the laboratory staff, and the Chief Quality Officer. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformance (NCAR) procedures.

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13.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at Columbia Analytical (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 Columbia Analytical contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at Columbia Analytical before it may be 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).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at Columbia Analytical. 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 E for a list of preventive maintenance activities and frequency for each instrument.

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14.0 CORRECTIVE AND PREVENTIVE ACTION

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form (See Figure 14-1). The procedure and responsibilities for addressing nonconforming work is defined in the SOP ADM-CA *Corrective Action*. Nonconformances are reported to the client using various means (voice, email, narrative, etc). When a nonconformance occurs that casts doubt on the validity of the test results or additional client instructions are needed, the Project Manager notifies the client the same business day that the nonconformance is confirmed and reported. The QA PM 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 QA PM. The QA PM 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 Manager is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

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, prompts corrective action. 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 QA PM may examine and pursue alternative solutions. In addition, the appropriate Project Manager is notified in order to ascertain if the client needs to be notified.

Part of the corrective action process involves determining the root cause. Identifying the root cause of a nonconformance can be difficult, but important for implementing effective corrective action. Root cause principles are used to determine assignable causes, which leads to corrective action taken to prevent recurrence. Various preventive action processes are used for eliminating a potential problem or averting a problem before it occurs. This is explained in the *SOP for Preventive Action (ADM-PA)*.

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the Project Manager) is responsible for documenting the complaint. If the Project Manager is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA PM for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback (ADM-FDBK)*.

Figure 14-1

Nonconformity and Corrective Action Report

NCAR No: *Assigned by QA*

PROCEDURE (SOP or METHOD): _____	EVENT DATE: _____
EVENT: <input type="checkbox"/> Missed Holding Time <input type="checkbox"/> QC Failure <input type="checkbox"/> Lab Error (spilled sample, spiking error, etc.) <input type="checkbox"/> Method Blank Contamination <input type="checkbox"/> Login Error <input type="checkbox"/> Project Management Error <input type="checkbox"/> Equipment Failure <input type="checkbox"/> Unacceptable PT Sample Result <input type="checkbox"/> SOP Deviation <input type="checkbox"/> Other (describe): _____	
INCLUDE NUMBER OF SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED	
DETAILED DESCRIPTION	
ORIGINATOR: _____ DATE: _____	
PROJECT MANAGER(S): _____ NOTIFIED BY: _____ DATE: _____	

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ROOT CAUSE OF NON-CONFORMITY (POTENTIAL CAUSES COULD BE TRAINING, COMMUNICATION, SPECIFICATIONS, EQUIPMENT, KNOWLEDGE)

What is the cause of the error or finding:
--

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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 <u>and</u> prevent its reoccurrence. Include Project Manager Instructions here.
Is the data to be flagged in the Analytical Report with an appropriate qualifier? <input type="checkbox"/> No <input type="checkbox"/> Yes

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____ Date: _____ Comments:
QA PM Verification and Approval of Corrective Action _____ Date: _____ Comments:
Project Manager Verification and Approval of Corrective Action _____ Date: _____ Comments:
Customer Notified by <input type="checkbox"/> Telephone <input type="checkbox"/> Fax <input type="checkbox"/> E-mail <input type="checkbox"/> Narrative <input type="checkbox"/> Not notified (Attach record or cite reference where record is located.)

15.0 QUALITY ASSURANCE REPORTS AND MANAGEMENT REVIEW

Quality assurance requires an active, ongoing commitment by Columbia Analytical personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Nonconformance and Corrective Action Report (NCAR) (see Section 14.0) 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 addressed.

It is the responsibility of each laboratory unit to provide the Project Manager 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 Manager, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written by the Project Manager to explain any unusual problems with a specific analysis or sample, etc.

The QA PM provides overview support to the Project Managers 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 regularly communicates with the Laboratory Director to review the various QA/QC activities, priorities, and status of program implementation; including 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.

An annual management review of the quality and testing systems is performed as described in the *SOP for Managerial Reviews of the Laboratory's Quality Systems and Testing Activities* (ADM-MGMTRVW). This is done to identify any necessary changes or improvements to the quality system or quality assurance policies. This review is documented in a Managerial Review of the Laboratory's Quality Systems and Testing Activities and sent to senior management.

16.0 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 Columbia Analytical are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at Columbia Analytical 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 Columbia Analytical. 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). Quality Systems training begins with Quality Assurance orientation for new employees and reading the Quality Assurance Manual. During the employees first year, the employee attends Core Ethics training and learns about Columbia Analytical Services quality systems. Each employee participates in annual Ethics Refresher training, which is part of the Columbia Analytical Improper Practices Prevention Program.

Columbia Analytical 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 corporate, company-wide training and development program, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained in the QA department. Training requirements and its documentation are described in the *SOP for Documentation of Training*. (ADM-TRANDOC). A training plan is developed whenever an employee starts a new procedure to new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

16.1 Initial Demonstration of Capability (IDOC)

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.
- 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 16-1. 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.

16.2 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 sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- 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.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- 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.

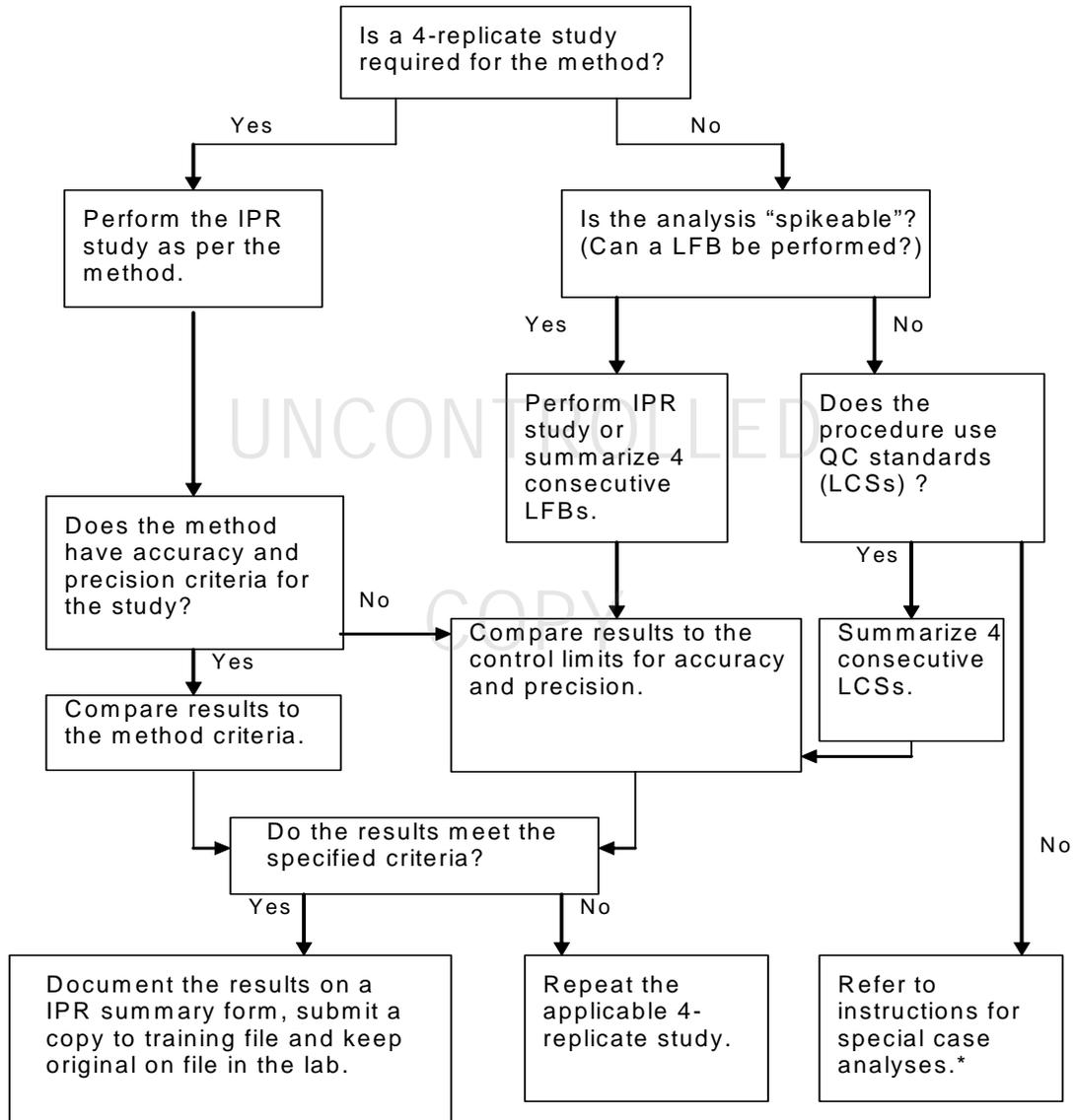
16.3 Documentation of Training

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 Columbia Analytical resumes. QA maintains a database to record the various technical skills and training acquired while employed by Columbia Analytical. 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* (ADM-TRANDOC).

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**Figure 16-1
Initial Demonstration of Capability Requirements**



* Refer to the SOP for Documentation of Training for details.

17.0 REFERENCES FOR QUALITY SYSTEMS, EXTERNAL DOCUMENTS, MANUALS, STANDARDS, AND ANALYTICAL PROCEDURES

The analytical methods used at Columbia Analytical 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 Columbia Analytical are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- TNI Standard – Environmental Laboratory Sector, Volume 1, *Management and Technical Requirements for Laboratories Performing Environmental Analysis*, EL-V1-2009.
- Quality Standards. American National Standard *General requirements for the competence of testing and calibration laboratories*, ANSI/ISO/IEC 17025:2005(E)
- DoD Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/2010
- *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).
- *Procedure Manual for the Environmental Laboratory Accreditation Program*, Washington Department of Ecology, 10-03-048, September 2010.
- *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), Final Update IV (February 2007), and updates posted online at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>. See Chapters 1, 2, 3, and 4.
- *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*, 20th Edition (1998) and SM On-Line. 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.
- *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).
- *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).

APPENDIX A

Approved Signatories

QA Program Documents

Corporate Policies

Administrative Corporate SOP List

COPY

APPROVED SIGNATORIES FOR ANALYTICAL REPORTS

Columbia Analytical Services, Kelso, WA

ARNOLD, EILEEN
BAILEY, JOSH
CHAN, JIM
CORONADO, JEFFREY
DEGNER, CARL
DOMENIGHINI, LISA
GRINDSTAFF, JEFF
HADERLY, DOUGLAS
HARRIS, LISA
HOLMES, HOWARD
HUCKESTEIN, LYNDA
JACKY, HARVEY
JAMES, JON
KAMAWAL, AQUILLA
KENNEDY, LES
LEAF, CHRIS
MIHAI-LAZAR, CARMEN
MOORE, RACHEL
MURRY, SHANE
PORTWOOD, LOREN
REASONER, KAREN
SALATA, GREGORY
SENKBEIL, RANI
SHELDON, BRIAN
WALLACE, ED

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QA Program Files

Program	Location
Quality Assurance Manual	Q:\QA Manual\QAM.rXX.DOC
Software Quality Assurance Plan	Corp IT
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Q:\MDL Tracking\MDL_LIST.r1.XLS
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	QAM App A
Personnel resumes/qualifications	HR dept
Personnel Job Descriptions	HR Department
CAS/KELSO DATA QUALITY OBJECTIVES	CAS Kelso DQO 20XX.rX.xls
Master Logbook of Laboratory Logbooks	QA Masterlog-001
Standard Operating Procedure Database	Q:\ENVIRONMENTAL\1 SOP & Policy Statements\1_ Kelso SOP.xls

Corporate Policies

POLICY TITLE	POLICY DATE	DATE APPROVED	DATE EFFECTIVE
CAS Quality and Ethics Policy Statement	September 2010	9/28/10	9/28/10
Policy for Data Review and Validation	September 2010	9/9/10	9/10/10
Policy for Internal Quality Assurance Audits	May 2009	5/5/09	7/1/09
Policy for Standards and Reagents Expiration Dates	September 2009	9/15/09	9/28/09
Policy for Use of Accreditation Organization's Name, Symbols, and Logos	September 2009	9/21/09	10/1/09
Policy for Conducting Research, Method Development, and Method Investigations	December 2009	12/15/09	12/17/09 Replaced by SOP 7/1/11

Corporate SOPs

SOP TITLE	SOP Code	Rev	SOP Date	Date of Last Review
SOP for Checking New Lots of Chemicals for Contamination	ADM-CTMN	5	5/2/11	5/4/11
SOP for Control Limits	ADM-CTRL_LIM	7	12/14/09	12/22/10
SOP for Corrective Action	ADM-CA	6	9/15/09	9/22/10
SOP for Data Recall	ADM-DATARECALL	0	9/21/07	11/22/10
SOP for Document Control	ADM-DOC_CTRL	8	9/15/09	9/22/10
SOP for Document Management Policy Implementation	ADM-DOC_MGMT	0	6/16/11	6/30/11
SOP for Documentation of Training	ADM-TRANDOC	12	4/28/11	5/15/11
SOP for Estimation of Uncertainty of Measurements	ADM-UNCERT	6	9/23/10	9/29/10
SOP for Handling Customer Feedback	ADM-FDBK	5	12/14/09	12/22/10
SOP for Making Entries into Logbooks and onto Analytical Records	ADM-DATANTRY	9	9/27/10	9/29/10
SOP for Managerial Review of the Laboratory's Quality Systems and Testing Activities	ADM-MGMTRVW	4	5/2/11	5/5/11
SOP for Manual Integration of Chromatographic Peaks	ADM-INT	4	10/5/10	10/9/10
SOP for Method Development, Investigation, and Transfer	ADM_MDEV	0	6/16/11	6/16/11
SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	ADM-MDL	9	9/8/09	9/21/10
SOP for Preparation of Electronic-data for Organic Analyses for Electronic-data Audits	ADM-E_DATA	3	8/29/07	11/22/10
SOP for Preparation of SOPs	ADM-SOP	10	12/20/10	12/22/10

SOP TITLE	SOP Code	Rev	SOP Date	Date of Last Review
SOP for Preventive Action	ADM-PA	1	12/14/09	12/22/10
SOP for Proficiency Testing Sample Analysis	ADM-PTS	3	9/22/10	9/29/10
SOP for Purchasing and Approval of Vendors	ADM-PUR	4	10/15/09	10/5/10
SOP for Qualification of Subcontract Laboratories	ADM_SUBLAB	5	9/15/09	9/22/10
SOP for Significant Figures	ADM-SIGFIG	8	1/28/09	1/13/10
SOP for Tape Backup and Tape Archiving	ADM-TAPEBU	0	10/3/11	10/15/11

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Forms

FORM	FILE NAME	DATE
Audit Finding Response Form	Audit Finding Response Form	8/12/2010
CASED Employee Development Plan Template	CAS EDP Template_033011_form only.doc	3/30/11
Complaint Report	Complaint Report_r121509	12/15/09
Critical Job Function Authorization Statement	Critical Job Function Authorization Statement_r071206	7/12/06
Data Re-submittal Request Form	Data Resubmittal Request Form_r112107	11/21/07
Demonstration of Capability Certification Statement (no table version)	DOC Certification Statement_r071206-	7/12/06
Extraction Solvent Critical Consumables Evaluation	Extraction Solvent Critical Consumables Evaluation_r050311.doc	5/3/11
Laboratory Training Certification	LAB-TRNG_r092109	9/21/09

FORM	FILE NAME	DATE
Metals Critical Consumables Evaluation	Metals Critical Consumables Evaluation_r050311.doc	5/3/11
Method Detection Limit Study Calculation Spreadsheet	MDL_FORMR4_r030510	3/5/10
New Vendor Evaluation	Vendor Evaluation Form_r101509	10/15/09
Nonconformity and Corrective Action Report	NCAR09_r092109	9/21/09
Preventive Action Report	PA Report_r072108	7/21/08
Procedure Change Form	Procedure Change Form_r121610	12/16/10
Reagent/Consumable Critical Consumables Evaluation	Reagent Critical Consumables Evaluation_r050311.doc	5/3/11
Standard Operating Procedure Change Form	SOP Change Form_r092109	9/21/09
LOD Verification	H:\group\QA\QA_Forms\LOD Verification071610.xls	07/16/10
LOQ Verification	H:\group\QA\QA_Forms\LOQ Verification022410.xls	02/24/10
Various Training Plans	H:\group\QA\QA_Forms\Training Plans\	NA

APPENDIX B

ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

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**JEFFERY A.
GRINDSTAFF
1991 TO
PRESENT**



Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

LABORATORY DIRECTOR, KELSO LABORATORY – 2010 to Present

Responsibilities

Responsible for all phases of laboratory operations at the Kelso (WA) facility, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory

Documentation of Demonstration of Capabilities is available for review.

Experience

Technical Manager III, Pharmaceutical, GC/MS VOA And Semi-VOA Laboratories, Columbia Analytical Services, Inc., Kelso, Washington – 1997-2010 Primary responsibilities include leadership of the Pharmaceutical, GC/MS VOA and Semi-VOA staff, management of method development, training, data review, tracking department workload, scheduling analyses. Responsible for ensuring data quality and timeliness. Also responsible for project management and coordination for pharmaceutical clients.

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, instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants.

Education

Sampling and Testing of Raw Materials, PTI International, 2004.

Leadership Training, Richard Rogers Group, 1996

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**

Mr. Grindstaff has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.

Affiliations

American Chemical Society. 1989

Current Position	TECHNICAL MANAGER I, KELSO LAB QUALITY ASSURANCE MANAGER – 2008 to Present
Responsibilities	Responsible for the overall implementation of the laboratory QA program. Oversees implementation of Quality management systems including: Quality Assurance Manual, Certifications, SOP Control, Proficiency Testing (PT), Non-Conformity, Preventative Actions, Internal Auditing, Control Charting, Documentation of Training, and Metrology. Conducts employee QA training including orientations, sop, and ethics. Maintains state, agency and program certifications/accreditations. Acts as primary point of contact during laboratory audits coordinates audit responses and corrective actions.
Experience	<p>Scientist IV, Semi-Volatile Mass Spectrometry Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 2002-2008. Primary responsibilities were analysis, interpretation and report generation for semivolatile organics by GC/MS. Analyses included EPA 625, 8270, SIM, and other miscellaneous methodology.</p> <p>Technical Manager I, Semi-Volatile GC Organics Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1999-2002. Primary responsibilities include supervision and oversight of semi-volatile GC department. This includes initiating new methods, staff training, workload management, and instrument maintenance/troubleshooting. Duties include departmental compliance with CAS QA and Safety policies. Responsible for analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, EPA 8141A, Organotins, and CLP Pesticides.</p> <p>Scientist III, Semi-Volatile Organics Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1996-1999. Primary responsibilities were analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, and CLP-Pesticides. Secondary responsibilities include organics semi-volatile sample preparation.</p> <p>Scientist, Volatile Organics Sample Preparation, Employer's Overload, Longview, Washington – assigned to the Columbia Analytical Services, Inc., Kelso, Washington facility, 1996. Primary duties included the preparation of water, soil, sediment and tissue samples using EPA Methods 3510, 3520, 3540, 3550, and 3545. Other duties were the further clean up of extracts using EPA Methods 3620 (Florsil), 3610 (Alumina), 3630 (Silica gel), 3650 (Acid/Base Partitioning), and 3660 (Sulfur).</p> <p>Organics Chemist and GC/MS Chemist, Coffey Laboratories, Portland, Oregon, 1990-1996. Primary responsibilities included sample preparation and analysis for EPA FID, ECD, and HPLC using various EPA SW-846 and 500-series methods, as well as other methodology. Later, moved to GC/MS position which included sample preparation, analysis, and associated instrument maintenance for EPA Methods 625, 8027, and 525 BNA's. Also responsible for data review and approval of data packages.</p> <p>QC Manager/QC Supervisor and Product Manager, Corn Products, Frito-Lay, Inc., Vancouver, Washington, 1982-1990. Manager of the QC department overseeing three supervisors and approximately 30 technicians. Responsible for department cost, accuracy, timeliness of data and safety performance. Later, responsible for production oversight of brand name snacks. Responsible for cost, quality and safety performance over three shifts. Managed four supervisors directly and approximately 60 employees indirectly.</p> <p>Food Technologist, QA Department, Kraft, Inc., Buena Park, California, 1978-1981. Responsible for audits, formulations, finished product evaluation, batch reviews and technical support.</p>
Education	<p>MS, Food Science, Minor in Industrial Engineering, Oregon State Univ. Corvallis, Oregon, 1978.</p> <p>BS, Food Science, Minor in Business Administration, Utah State University, Logan, Utah, 1975</p>
Affiliations	ASQ-American Society of Quality, ISO/IEC 17025:2005 Expanded Internal Auditor Course
Achievements	<p>Quality Improvement Team Leader, Coffey Laboratories, Portland, Oregon. 1991</p> <p>Methods Improvement Program, Coffey Laboratories, Portland, Oregon. Seminars on Development and Implementation 1990.</p> <p>Statistical Process Control and Total Quality Management, Frito-Lay, Vancouver, Washington. Routine Training Classes 1986-1988.</p>

JEFFREY A. CORONADO
1989 TO PRESENT



Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	TECHNICAL MANAGER IV, INORGANICS DEPARTMENT MANAGER – 2001 to Present
Responsibilities	Oversee the operation of the Metals Group. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, 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.

HARVEY L. JACKY
1999 TO PRESENT



Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	TECHNICAL MANAGER II – 2008 to Present
Responsibilities	<p>Oversee the operation of the General Chemistry and Microbiology groups. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation.</p> <p>Documentation of Demonstration of Capabilities is available for review.</p>
Experience	<p>Project Manager III, <i>Columbia Analytical Services, Inc., Kelso, WA</i>, 1999-2008. 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. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies.</p> <p>Director of Project Management, <i>Coffey Laboratories, Portland, Oregon</i>, 1997-1999. Responsible for technical project management. Communicated with clients to determine needs and expectations. Monitored laboratory production and ensured the timely completion of analytical projects. Technical consultant for clients regarding environmental compliance. Supervised and managed other members of the project management team. Served as a member of the senior management team for oversight of general operations, strategic planning, finances, and policy.</p> <p>Project Manager/Chemist, <i>Coffey Laboratories, Portland, Oregon</i>, 1997-1999. Served as primary liaison between Coffey Laboratories and major clients. Ensured that work was completed in a timely manner and done to client specifications. Served as technical consultant regarding environmental chemistry, soil remediation, and waste water industrial compliance. Clients included the Oregon Department of Transportation, Hazmat Unit, Portland, Oregon; Raythion Demilitarization Co., Umatilla, Oregon; Hydroblast - Wastewater Evaporator Systems, Vancouver, Washington; and Union Pacific Railroad, Northwest Region, Klamath Falls, Oregon.</p> <p>Technical Sales Representative, <i>Coffey Laboratories, Portland, Oregon</i>, 1995-1997. Responsible for marketing and sales, including actively prospecting for new potential clients. Additional responsibilities included procurement and preparation of all major project bids; ensuring that client expectations were met; and maintaining customer satisfaction. Served as consultant regarding industrial compliance issues, environmental remediation projects, and hazardous waste management.</p> <p>Senior Chemist/Laboratory Chemical Hygiene Officer, <i>Coffey Laboratories, Portland, Oregon</i>, 1988-1995. Performed analytical tests including Anions by Ion Chromatography (EPA 300.0), PAHs by HPLC (EPA 8310), Cyanides (EPA 335), and other inorganic, wet chemistry, and organic analytical tests on a wide variety of sample matrices. Responsible for the initial quality assurance review of work performed, supervised and managed personnel. Developed and implemented Laboratory Chemical Hygiene Plan. Directed personnel in regards to safety issues and hazardous waste management. Served as consultant and teacher regarding analytical methodology, environmental compliance, and industrial hygiene.</p>
Education	<p>40-Hour Hazmat Certification, <i>PBS Environmental</i>, 1996.</p> <p>Industrial Emergency Response, <i>SFSP Seminar</i>, 1991</p> <p>BS, Zoology, <i>Oregon State University, Corvallis, Oregon</i>, 1988.</p> <p>BS, General Science, <i>Oregon State University, Corvallis, Oregon</i>, 1988.</p> <p>COURSEWORK, General Studies, <i>Linfield College, McMinnville, Oregon</i>, 1981-1982.</p>
Publications/ Presentations	<p><i>Biochemical and Physical Factors Involved in the Application and Measurement of a Soil Bioremediation System</i>. Biogeochemistry, Portland State University, 1996</p>
Affiliations	American Chemical Society, Member since 1988

Current Position | **SCIENTIST II** – 2011 to Present

Responsibilities | Experience in pharmaceuticals, food microbiology and environmental samples. Experience in validation/qualification of all laboratory equipment (IQ/OQ/PQ). Development of new methods, SOP, validation protocols and report writing. Experience in design and operation of custom Microbiology testing (e.g. MIC Test (Minimum Inhibitory Concentration Test). Subject Matter Expert (SME for CAS environmental and pharmaceutical microbiology laboratories.

Technical Manager Environmental-Review and approval of method development and investigations. Final approval of SOP. Review and final approval of analyst training records. Assist in PT corrective actions.

Experience

Scientist I, *Columbia Analytical Services, Kelso, WA.*, 2008-2011. Microbiologist performing routine and non-routine microbiology testing of pharmaceutical raw materials, excipients and drug products in accordance with applicable methods (USP, BAM, AOAC). Method development and validation as required. Subject Matter Expert (SME) for CAS environmental and pharmaceutical microbiology laboratories.

Analyst III, *Columbia Analytical Services, Kelso, WA.*, 2008-2011. Responsible with analysis of BOD, CBOD, Total Coliform, Fecal Coliform, E. Coli, Heterotrophic Plate Count/Total Plate Count, Colilert/Quantitray, Bacteria Swab, Enterococcus, Enterolert, Dissolved Oxygen, Yeast and mold, Aerobic Plate, Sheen Screen, IRB, SRB, SCYM-Bart.

Scientist, *Roche Molecular Systems, Alameda, California*, 2000-2007. Produced master cell banks for new controls. Test and certify controls for manufacturing. Prepared DNA Panels for projects. Extensive mammalian cell culture experience with excellent sterile technique. Lyophilization, RNA transcription, and Bacteriophage Production, DNA extraction/purification from all cell types. Responsible for equipment calibration, validation, and preventative maintenance. cGMP experience. Experience in writing IR's (Investigation Reports), SOP's (Standard Operating Procedures), and satisfying CAPA's (Corrective Action Preventative Action). Responsible for cryostorage inventory/management. Maintained documentation updated database and produced "Certificates of Analysis". Responsible for lab purchasing, lab and instrument maintenance. Point person for cell repository ordering. Prepared and participated in internal/external audits.

Lab Assistant, *Center for Biomedical Laboratory Science, San Francisco University, San Francisco, Californian*, 1999-2000. Performed research in Dr. Lily Chen's lab using the following techniques: transformation of bacteria and yeast, plasmid isolation from bacteria and yeast, agarose gel electrophoresis, restriction digestion and PCR.

Internship Assistant, *Center For Biomedical Laboratory Science, San Francisco University, San Francisco, California*, 1998-1999. Assisted with various laboratory preparations and organized Med-Tech Administrative Program.

Education

BS, Microbiology, *San Francisco State University, San Francisco, CA*, 2000.

Current Position	TECHNICAL MANAGER II, Semivolatile Organics Department Manager – 2009 to Present
Responsibilities	Oversee the operation of the Semivolatile Organics Department. Responsible for the quality and timeliness of analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. Documentation of Demonstration of Capabilities is available for review.
Experience	TECHNICAL MANAGER I, GC SEMI-VOA, Columbia Analytical Services, Inc., Kelso, Washington, 2007 to 2009. Responsible for supervision of GC Semi-VOA staff, interfacing with Project Management Team, working with Extractions group, method development, training, data review, tracking department workload, scheduling analyses, and operation, maintenance and troubleshooting GC instrumentation. Also responsible for department adherence to strict QA/QC policies of the organization. SCIENTIST III, GC SEMI-VOA, Columbia Analytical Services, Inc., Kelso, Washington, 2002 to 2007. Responsible for operation, maintenance, and troubleshooting of GC/ECD and GC/FPD instrumentation. Performed instrumental analysis and all stages of data review for tests performed in SVG. Also involved in problem-solving with Extractions group, training, and workload coordination. Chemist II, Pesticide Laboratory, Oregon Department of Agriculture, Portland, Oregon, 2000-2002. Responsible for non-routine sample extraction and analysis of phenoxy herbicides, chlorinated acids, organochlorines, organophosphates, organonitrogens, sulfonyl ureas, carbamates, and other unclassified pesticides using a wide array of GC and LC instrumentation, including ECD, ELCD, FPD, AED, MS, and fluorescence detection. Also responsible for instrument maintenance, method development, data review, training, and assisting in workload coordination. Chemist I, Pesticide Laboratory, Oregon Department of Agriculture, Portland, Oregon, 1999-2000. Performed sample extraction and analysis by GC and LC using FDA and EPA methodologies. Research Technologist, Shriners Hospital, Portland, Oregon, 1995-1999. Worked with extracellular matrix proteins independently and under the supervision of/as assistant to post-doctoral associates. Protein isolation, purification, and characterization using the following techniques: cell culture, liquid chromatography (reverse-phase, ion-exchange, affinity), differential centrifugation, immunoprecipitation, SDS-PAGE, immunoblotting, and ELISA assay. Research Assistant/Thesis Student, Reed College, Portland, Oregon, 1994-1995. Reviewed literature, devised and conducted synthetic organic experiments, and analyzed results using NMR and IR instrumentation.
Education	BA, Chemistry, Reed College, Portland, Oregon, 1996.

Current Position	Technical Manager II, VOA/MS, Semivolatile GC/MS and HPLC Department Manager, 2009-Present
Responsibilities	Oversee the operation of the Volatiles GC/MS, Semivolatile GC/MS and HPLC laboratories. Responsibilities include organizing and prioritizing workload, training and development of staff, working with PCs on client specific project requirements, departmental budgets, workload coordination, method development efforts and resource allocation. Responsible for the quality and timeliness of analytical reports. Other responsibilities include ensuring compliance with CAS QA protocols and assisting staff with troubleshooting equipment and procedural problems. Documentation of Demonstration of Capabilities is available for review.
Experience	Technical Manager I, VOA and PHC/HPLC Laboratories, Columbia Analytical Services, Inc., Kelso, Washington, 2004-2009. Oversee daily operation of the Volatiles GC/MS and PHC/HPLC laboratories. Responsibilities include organizing and prioritizing workload, initiating process improvements, training and development of staff and working with PCs on client specific project requirements. Responsible for analytical duties as listed below for Scientist IV. Other responsibilities include ensuring compliance with CAS QA protocols and assisting staff with troubleshooting equipment and procedural problems. Scientist IV, VOA Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1999-2004. Perform sample analysis and data review for EPA methods 524.2, 624 and 8260. Duties also include Project Management. Scientist III/Project Chemist, Supervisor Pesticides GC Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1998-1999. Primary responsibilities included workload scheduling, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Scientist III, Semi-volatile Gas Chromatography Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1996-1998. Primary responsibilities included analysis of samples using GC and HPLC techniques, report generation, data review, preparation of analytical standards, maintenance of instrumentation, Client Services and some Project Management. Routine duties included analysis of soil and water samples for pesticides, PCBs, CLP Pesticides, Explosives and PAHs using EPA methods. Scientist II, Semi-volatile Gas Chromatography Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1994-1996. Primary responsibilities included analysis of samples using GC and HPLC techniques, report generation, data review, preparation of analytical standards, maintenance of instrumentation and client service/project management duties. Laboratory Analyst III, Semi-volatile Gas Chromatography Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1994. Primary responsibilities included analysis of samples by GC/ECD, GC/FID, GC/FPD, GC/NPD and HPLC techniques. Standard analytical methods performed were EPA method 515.1, 504, 8150, 8011, 8150M (for chlorinated phenols), 8310, and 8015. Laboratory Analyst II, Organic Extractions Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1991-1992. Responsibilities included extraction of soil and water samples for various SVOCs, and TCLP extraction of SVOC and VOC compounds using TCLP equipment. Other duties included performing cleanup procedures, validation studies, MDL studies, and the training of employees in advanced extraction procedures and techniques.
Education	Introduction to LC Methods Development & Troubleshooting, Hewlett-Packard, Tacoma, Washington, 1995. HPLC Maintenance Seminar, Waters, Portland, Oregon, 1994. GC/HPLC Maintenance Seminar, Hewlett-Packard, Olympia, Washington, 1993. Gas Chromatography Seminar, Curtis Matheson Scientific, Kelso, Washington, 1992. HPLC Seminar, Hewlett-Packard, Kelso, Washington, 1991. BA, Chemistry/Biology, The Evergreen State College, Olympia, Washington, 1991. AA, Arts and Sciences, Lower Columbia College, Longview, Washington, 1990.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFETY 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.

LYNDA A. HUCKESTEIN
1989 TO PRESENT



Columbia Analytical Services, Inc., 1317 South 13th Ave., 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 of 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 D. CHRISTIAN
1989 TO PRESENT



Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

DIRECTOR OF OPERATIONS – 2010 to Present

Responsibilities

Responsible for oversight of operating units of Columbia Analytical Services, inc. with all Laboratory Directors reporting to the COO. Primary responsibilities include establishment of consistent quality, technical, and client service enhancements across the company, as well as the financial performance of the individual operating units. In addition, a significant role is to represent operations as a member of the Senior Management Team (SMT) consisting of the Chief Executive Officer, Chief Financial Officer, Chief Quality Officer, and the Director of Information Technology.

Experience

Vice President/Laboratory Director, Kelso Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1993-2010. 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.

Coursework, Pacific Lutheran University, Tacoma, Washington. 1988-1989.

Coursework, Tacoma Community College, Tacoma, Washington. 1970-1971, 1988-1989.

CERTIFICATION, Chemistry, L.H. Bates Technical, Tacoma, Washington, 1976-1978.

Coursework, Central Washington University, Ellensburg, Washington. 1969-1970.

Numerous Training/Educational Activities via Conferences, Professional Seminars, and Factory Training, 1989-2010.

**Publications/
Presentations**

Mr. Christian has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.

Current Position

DIRECTOR OF QUALITY ASSURANCE – 2008 to Present

Responsibilities

Directing the overall corporate-wide quality systems and ethics programs for all CAS facilities. Responsible for ensuring that CAS quality systems and data integrity standards are implemented at all facilities. Act as liaison with government entities involving quality, technical and operational issues. Provide QA input and policy as needed for operations, development initiatives, special projects, planning, and information technology implementation. Provide assistance to QA Program Managers.

Experience

Technical Manager IV, Quality Assurance Program Manager, *Columbia Analytical Services, Inc., Kelso, Washington* – 2002 to 2008. As part of the management team, responsibilities included the overall management and implementation of the laboratory QA program. This included maintaining accreditations and certifications, and maintaining all necessary documents (QA Manual, SOPs, and QA records). Acted as primary point of contact during laboratory audits and provided audit responses and corrective actions. Coordinated performance audits (PE/PT testing) and conducted internal audits.

Scientist IV, Quality Assurance Program 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 of projects for clients, identifying client needs, and preparation of data reports.

Semivolatile Organics Department Manager, *Columbia Analytical Services*, 1988-1994. Responsibilities included overall management of the department. Supervised GC/MS analyses, data review, reporting and related QA/QC functions. Responsible for supervision of staff, training, and scheduling. Beginning in 1992, responsibilities included being a Project Chemist for organics EPA-SAS and other clients. This involved scheduling projects for clients, identifying client requirements, 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 semivolatiles by EPA protocol, including Methods 8240, 8270 and CLP. Coordinated extraction and GC-GC/MS areas to manage sample/data flow through the lab. Also performed HPLC analysis and pesticide analysis by GC using EPA Methods.

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.

Education

Pharmaceutical Laboratory Control Systems, *Univ. of Wisconsin Short Course, Las Vegas*, 2004

Test Method Validation in Pharmaceutical Development and Production, *Univ. of Wisconsin Short Course, Las Vegas*, 2004

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, *Eastern Washington University, Cheney, Washington*, 1985.

**Publications/
Presentations**

Selected Ion Monitoring: Issues for Method Development, Panel Discussion, Association of Official Analytical Chemists, (AOAC) Pacific Northwest Regional Meeting, 1995.

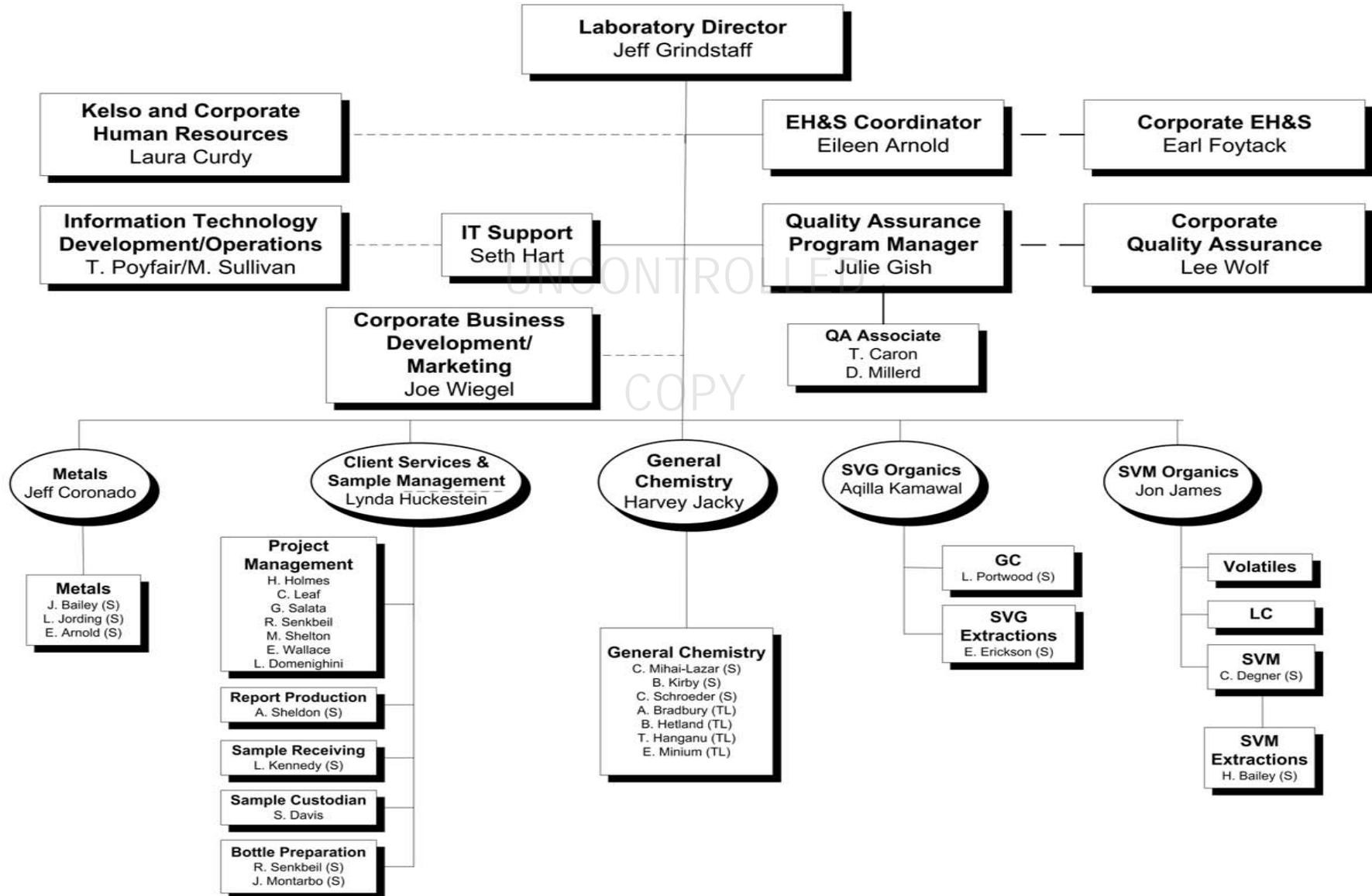
Method Enhancement Techniques for Achieving Low level Detection of Butyl Tin in Marine Sediments and Tissues, Association of Official Analytical Chemists (AOAC) Pacific Northwest Regional Meeting, 1994.

The Determination of Low-Level Concentrations of Polynuclear Aromatic Hydrocarbons (PAHs) in Soil and Water Using Gas Chromatography/Mass Spectroscopy Selected Ion Monitoring (GC/MS SIM), HazMat West, Long Beach, California, 1992.

Affiliations

American Chemical Society. American Society for Quality.

**Environmental and General Testing Division
Kelso, Washington
Laboratory Organization**



APPENDIX C
MAJOR ANALYTICAL EQUIPMENT

UNCONTROLLED

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GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balances (16): Precisa, Mettler, OHOUS, Adams models	1990-2011	LM	13
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autotitrator – Thermo Orion 500	2007	LM	3
Calorimeters (2): Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	13
Colony Counter - Quebec Darkfield	1988	LM	2
Conductivity Meters (2): YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5): COD (4)	1987, 1989	LM	4
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	4
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	5
Drying Ovens (12): Shel-Lab and VWR models	1990-2010	LM	13
Air Drying Cabinets	2011	LM	NA
Flash Point Testers (2): ERDCO Setaflash Tester	1991	LM	3
Petroleum Systems Services	2005	LM	3
Flow-Injection Analyzers (2): Bran-Leubbe	2002	LM	2
Lachat 8500	2007	LM	2
Ion Chromatographs (4) Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Dionex ICS-2000 with Chromchem Data System	2006	LM	3
Dionex ICS-1600 with Chromchem Data System	2009	LM	
Ion Selective Electrode Meters (5) Fisher Scientific Accumet Model 50	1997	LM	4
Fisher Scientific Accumet Model 25	1993	LM	4
Fisher Scientific Accumet Model 20	2000	LM	4
Orion Model 920A	1990	LM	4
Corning pH/ion Meter Model 135	1992	LM	4
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	13

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
pH Meters (2): Fisher Scientific Accumet Model 20	1993	LM	5
Fisher Scientific Accumet Model AR25	2005	LM	5
Shatter Box (2): GP 1000	1989	LM	5
SPEX 8530	2011		
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	5
Total Organic Carbon (TOC) Analyzers (2) Coulemetrics Model 5012	1997	LM	3
Teledyne Tekmar Fusion 1	2009	LM	3
Total Organic Halogen (TOX) Analyzers (2): Mitsubishi TOX-100	2001	LM	2
Turbidimeter - Hach Model 2100N	1996	LM	5
UV-Visible Spectrophotometers (3): Hitachi 100-40 Single Beam	1986	LM	4
Beckman-Coulter DU520	2005	LM	4
Perkin Elmer Lambda 25	2008	LM	4
Abrazix	2011	LM	2
Discrete Autoanalyzer –Westco SmartChem AD20-1	2011	LM	2
Vacuum Pumps (3): Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991		
Chem Star 1402N-01	2011		
Water Baths/Incubators (6): Various Fisher Scientific and VWR Models	1986 - 2009	LM	13

METALS LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance (8) Mettler AE 200 analytical balance Various Mettler, Sartorius, and Ohaus models	1988-2010	MM	12
Atomic Absorption Spectrophotometers (5): Varian SpectrAA Zeeman/220 AA (2) CETAC Mercury Analyzer M-6000A Perkin Elmer AAnalyst 200 Flame AA CETAC Mercury Analyzer M-6100	2000 2000 2005 2010	LM LM MM MM	2 2 2 2
Buck AA Spectrophotometer Model 205	2008	LM	2
Atomic Fluorescence Spectrophotometer Brooks-Rand Model III (2) Leeman Mercury Analyzer (1)	1996, 2005 2006	LM LM	3 2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (1) - Labconco	2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (2) Thermo Jarrell Ash, Model IRIS Thermo Scientific Model iCAP 6500	2000 2007	MM MM	3 3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS): VG Excell Thermo X-Series Nexion Model 300D	2001 2006 2011	MM MM MM	3 2 2
Muffle Furnace (2) - Thermolyne Furnatrol - 53600	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
Analytical Balance (4) Mettler PM480, BB300 ,AG204 OHaus EP613	1999 - 2011	MM	12
Centrifuge – Beckman J-6B	1988	LM	12
Drying Ovens (2) Fisher Model 655G VWR Model 1305U	1991 1999	LM LM	12 12
Evaporators/concentrators Organomation N-Evap (8) Organomation S-Evap (8) Zymark Turbovap (2)	1990-2010 1990-2010 1998-2000	LM LM LM	12 12 12
Extractor Heaters: Lab-Line Multi-Unit Models for Continuous Liquid-Liquid and Soxhlet Extractions (102)	1987-2007	LM	8
Solids Extractors: Sonic Bath VWR (2) Sonic Horn (5) Soxhtherm Gerhardt (2) OI Analytical (6)	1991 -1994 1994 2000 2008	LM LM LM	6 6 6
Extractors, TCLP (10): Millipore TCLP Zero Headspace Extractors (5) TCLP Extractor - Tumbler (12 position)	1987-1992 1989	LM LM	2 2
Gel Permeation Chromatography (GPC) (6) ABC single column (4) J2 Scientific AccuPrep (2)	1998, 1999, 2007 2005, 2010	LM LM	4 4
Muffle Furnace - 4	1994-2006	LM	4
Solid Phase Extractors (18) – Horizon SPE-Dex 4790	2003, 2006,2008	LM	4

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Chromatography Data Systems (12) HP Enviroquant (8) Thruput Target (4) Varian Saturn (1)	1994-2003 1998-2000 2003	LM LM LM	7 4 2
Gas Chromatographs (17): Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (2) Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (6) Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors Agilent 7890A Dual ECD Detectors Agilent 7683B autosampler (2) Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (4)	1990 – 1995 1991 2001, 2005, 2007,2011 2003 2010 1995 2001, 2005	LM LM LM LM LM LM LM LM	6 3 6 3 6 3 6
Varian Ion trap GC/MS: Varian 3800 GC w/CP8400 autosampler Varian Saturn 2100T mass spectrometer	2003 2006 2003	LM LM LM	2 2 2
Thremo Ion Trap ITQ-90C GC/MS w/TriPlus autosampler	2008	LM	2

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance - Mettler AB 104-S	2000	MM	6
Enviroquant Chromatography Data Systems (9)	1994-2003	LM	6
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	6
Semivolatiles GC/MS Systems (11):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	6
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	6
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler	1994	LM	6
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	LM	6
Agilent 6890/5973 with ATAS PTV and 7683 Autosampler	2004	LM	6
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler	2007	LM	6
Agilent 7890A/5975C with Agilent 7693 Autosampler (2)	2010	LM	6
Semivolatiles GC/MS/MS – Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	2

HPLC LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance - Mettler BB240	1994	MM	4
Drying Oven - Fisher Model 630F	1991	LM	4
Evaporator – Turbo Vap	2009	LM	4
Centrifuge Marathon 21K	1996	LM	4
HP Enviroquant Chromatography Data Systems 4	1994-2002	LM	3
High-Performance Liquid Chromatographs (3):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	2
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	2
Agilent 1260 Infinity with Diode Array UV Detector	2011	LM	2
High-Performance LC/MS (3)			
Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS and Autosampler	2005	MM	2
API 5000 LC/MS/MS and SIL-20AC Autosampler	2008	MM	2
AB Sciex 5500 and Shimadzo DGU 20A5	2011	MM	2
Agilent 1100 HPLC -UV/Fluorescence detectors- Pickering PCX-5200 Post-column derivitization unit	2003	LM	2

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VOLATILE ORGANICS LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	5
Drying Ovens (2):			
Boekel 107801	1989	LM	5
VWR 1305 U	1991	LM	5
Sonic Water Bath - Branson Model 2200	1989	LM	5
Volatile GC/MS Systems (9):			
Agilent 5890/5970	1989	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 5890/5971	1991	LM	5
Tekmar 3000 Purge and Trap Concentrator	2001	LM	5
Dynatech ARCHON 5100 Autosampler	1995	LM	5
Agilent 5890/5972A	1993	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 6890/5973	2001	LM	5
Tekmar 3100 Purge and Trap Concentrator	2001	LM	5
Varian Archon Autosampler	2001	LM	5
Agilent 6890/5973	2005	LM	5
Tekmar Velocity Purge and Trap Concentrator	2005	LM	5
Tekmar Aquatech Autosampler	2005	LM	5
Agilent 6890/5973 (2)	2007	LM	5
Tekmar 3000 Purge and Trap Concentrator	2007	LM	5
Varian Archon 5100 Autosampler	2007	LM	5
Agilent 7980A/5975C (2)	2010, 2011	LM	5
Teledyne Tekmar-Automx	2010,2011	LM	5
Hewlett-Packard 5890 Series II with PID/PID/FID	1991	LM	2
EST-ENCON Purge and Trap Concentrator	1991		
Dynatech Archon 5100 Autosampler	1992		

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 & 11g DBMS running on Redhat Advanced Server 4.0 (Linux) platform connected/linked via both fiber and MPLS circuits.	1994-2007	LM	NA
1 - Network Server Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 SP2 Advanced Server, 1 for Applications running Windows 2003 Advanced Server SP2. Data acquisition capacity at 195 GB with redundant tape and disk arrays.	2004-2008	LM	NA
Approximately 80+ HP, Dell, Kyocera Laserjet printers (various types including models III, 4, 5, 8150, 4000, 4041, 4050, 4200 4250, 8150, 1720dn, W5300, 1300D, M4000)	1991 - 2010	LM	NA
Approximately 280 + Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2010	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97, Office 2007.	1996 - 2010	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 - Brother 4750e; Brother 2920; Brother 1860	1991 - 2010	LM	NA
Copiers/Scanners: Konica BizHub 420 (1), BizHub 600 (1), BizHub 920 (2), BizHub Pro 1050 (3), BizHub Pro 1051 (1). All are accessible via LAN for network scanning.	2000 - 2010	LM	NA
Dot Matrix Panasonic KX-P1150	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, CASLIMS 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

DATA QUALIFIERS AND ACRONYMS

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Inorganic Data Qualifiers

- * The result is an outlier. See case narrative.
- # The control limit criteria are not applicable. See case narrative.
- B The analyte was found in the associated method blank at a level that is significant relative to the sample result as defined by the DOD or NELAC standards.
- E The result is an estimate amount because the value exceeded the instrument calibration range.
- J The result is an estimated value that was detected outside the quantitation range.
- U The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM definition: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.*
- i The MRL/MDL or LOQ/LOD is elevated due to matrix interference.
- X See case narrative.
- Q See case narrative. One or more quality control criteria was outside the limits.

UNCONTROLLED

Metals Data Qualifiers

- # The control limit criteria are not applicable. See case narrative.
- J The result is an estimated value that was detected outside the quantitation range.
- E The percent difference for the serial dilution was greater than 10%, indicating a possible matrix interference in the sample.
- M The duplicate injection precision was not met.
- N The Matrix Spike sample recovery is not within control limits. See case narrative.
- S The reported value was determined by the Method of Standard Additions (MSA).
- U The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM 4.1 definition: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.*
- W The post-digestion spike for furnace AA analysis is out of control limits, while sample absorbance is less than 50% of spike absorbance.
- i The MRL/MDL or LOQ/LOD is elevated due to matrix interference.
- X See case narrative.
- + The correlation coefficient for the MSA is less than 0.995.
- Q See case narrative. One or more quality control criteria were outside the limits.

Organic Data Qualifiers

- * The result is an outlier. See case narrative.
- # The control limit criterion is not applicable. See case narrative.
- A A tentatively identified compound, a suspected aldol-condensation product.
- B The analyte was found in the associated method blank at a level that is significant relative to the sample result as defined by the DOD or NELAC standards.
- C The analyte was qualitatively confirmed using GC/MS techniques, pattern recognition, or by comparing to historical data.
- D The reported result is from a dilution.
- E The result is an estimate amount because the value exceeded the instrument calibration range.
- J The result is an estimated value that was detected outside the quantitation range.
- N The result is presumptive. The analyte was tentatively identified, but a confirmation analysis was not performed.
- P The GC or HPLC confirmation criteria were exceeded. The relative percent difference is greater than 40% between the two analytical results.
- U The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM 4.1 definition: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.*
- i The MRL/MDL or LOQ/LOD is elevated due to a chromatographic interference.
- X See case narrative.
- Q See case narrative. One or more quality control criteria was outside the limits.

Additional Petroleum Hydrocarbon Specific Qualifiers

- F The chromatographic fingerprint of the sample matches the elution pattern of the calibration standard.
- L The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter molecular weight constituents than the calibration standard.
- H The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of heavier molecular weight constituents than the calibration standard.
- O The chromatographic fingerprint of the sample resembles an oil, but does not match the calibration standard.
- Y The chromatographic fingerprint of the sample resembles a petroleum product eluting in approximately the correct carbon range, but the elution pattern does not match the calibration standard.
- Z The chromatographic fingerprint does not resemble a petroleum product.

Acronyms

ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service registry Number
CFC	Chlorofluorocarbon
CFU	Colony-Forming Unit
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOE	Department of Ecology
DOH	Department of Health
EPA	U. S. Environmental Protection Agency
ELAP	Environmental Laboratory Accreditation Program
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LUFT	Leaking Underground Fuel Tank
LOD	Limit of Detection
LOQ	Limit of Quantitation
M	Modified
MCL	Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL	Method Detection Limit
MPN	Most Probable Number
MRL	Method Reporting Limit
NA	Not Applicable
NC	Not Calculated
NCASI	National Council of the Paper Industry for Air and Stream Improvement
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
PQL	Practical Quantitation Limit
RCRA	Resource Conservation and Recovery Act
SIM	Selected Ion Monitoring
TPH	Total Petroleum Hydrocarbons

APPENDIX E
PREVENTIVE MAINTENANCE PROCEDURES
UNCONTROLLED

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Instrument	Activity	Maint ^a	Frequency
Refrigerators and Coolers	Record temperatures	LM	Daily
	Clean coils	LM	Annually
	Check coolant	LM	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	LM	Every month or as needed
Fume Hoods	Face velocity measured	LM	Quarterly
	Sash operation	LM	As needed
	Change filters	LM	Annually
	Inspect fan belts	LM	Annually
Ovens	Clean	LM	As needed or if temperature outside lim.
	Record temperatures	LM	Daily, when in use
Incubators	Record temperatures	LM	Daily, morning and evening
Water Baths	Record temperatures	LM	Daily, morning and evening
	Wash with disinfectant solution	LM	When water is murky, dirty, or when growth appears
Autoclave	Check sterility	LM	Every month
	Check temperature	LM	Every month
	Clean	LM	When mold or growth appears
	Calibrate thermometer	VM	Once a year
Analytical Balances	Check alignment	LM	Before every use
	Verify calibration	LM	Daily
	Clean pans and compartment	LM	After every use
	Certified calibration	VM	Once a year
Dissolved Oxygen Meter	Change membrane	LM	When fluctuations occur
pH probes	Condition probe	LM	When fluctuations occur
Fluoride ISE	Store in storage solution	LM	Between uses
Ammonia ISE	Store in storage solution	LM	Between uses
UV-visible Spectrophotometer	Wavelength check	VM	Twice a year

Instrument	Activity	Maint ^a	Frequency
Total Organic Carbon Analyzers	Check IR zero	LM	Weekly
	Check digestion/condensation vessels	LM	Each use
	Clean digestion chamber	LM	Every 2000 hours, or as needed
	Clean permeation tube	LM	Every 2000 hours, or as needed
	Clean six-port valves	LM	Every 200 - 2000 hours, or as needed
	Clean sample pump	LM	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	LM	Every 200 - 2000 hours, or as needed
	Clean IR cell	LM	Every 2000 - 4000 hours, or as needed
Total Organic Carbon Analyzers	Change cell electrolyte	LM	Daily
	Change electrode fluids	LM	Daily
	Change pyrolysis tube	LM	As needed
	Change inlet and outlet tubes	LM	As needed
	Change electrodes	LM	As needed
Flow Injection Analyzer	Check valve flares	LM	Each use
	Check valve ports	LM	Each use
	Check pump tubing	LM	Each use
	Check light counts	LM	Each use
	Check flow cell flares	LM	Quarterly
	Change bulb	LM	As needed
	Check manifold tubing	LM	Each use
	Check T's and connectors	LM	Each use
Discrete Auto Analyzer	Clean probe, wash reservoirs	LM	Every 2 weeks
	Replace peristaltic pump tubing	LM	Every 3 months
	Replace hydraulic circuit tubing	LM	Once/year

Instrument	Activity	Maint ^a	Frequency
Ion Chromatographs	Change column	LM	Every six months or as needed
	Change valve port face & hex nut	LM	Every six months or as needed
	Clean valve slider	LM	Every six months or as needed
	Change tubing	LM	Annually or as needed
	Eluent pump	LM	Annually
Atomic Absorption Spectrophotometers - FAA and CVAA	Check gases	LM	Daily
	Clean burner head	LM	Daily
	Check aspiration tubing	LM	Daily
Atomic Absorption Spectrophotometers - GFAA	Clean optics	LM	Every three months
	Empty waste container	LM	Weekly
	Check gases	LM	Daily
	Check argon dewar	LM	Daily
	Change graphite tube	LM	Daily, as needed
	Clean furnace windows	LM	Monthly
ICP - AES	Check argon dewar	LM	Daily
	Replace peristaltic pump tubing	LM	Daily
	Empty waste container	LM	Weekly
	Clean nebulizer, spray chamber, and torch	LM	Every two weeks
	Replace water filter	LM	Quarterly
	Replace vacuum air filters	LM	Monthly
ICP - MS	Check argon dewar	LM	Daily
	Check water level in chiller	LM	Daily
	Complete instrument log	LM	Daily
	Replace peristaltic pump tubing	LM	Daily
	Clean sample and skimmer cones	LM	As needed
	Clean RF contact strip	LM	As needed
	Inspect nebulizer, spray chamber, and torch	LM	Clean as needed
	Clean lens stack/extraction lens	LM	As needed
	Check rotary pump oil	LM	Monthly
	Change rotary pump oil	LM	Every six months

Instrument	Activity	Maint ^a	Frequency
Gel-Permeation Chromatographs	Clean and repack column	LM	As needed
	Backflush valves	LM	As needed
HPCL Chromatographs	Backflush guard column	LM	As needed
	Backflush column	LM	As needed
	Change guard column	LM	As needed when back pressure too high
	Change column	LM	Annually or as needed
	Change in-line filters	LM	As needed
	Leak check	LM	After column maintenance
	Change pump seals	LM	As needed
	Change pump diaphragm	LM	Annually
	Clean flow cell	LM	As needed
	Fluorescence detector check	LM	Daily
	Diode array absorbance check	LM	Daily
HPLC MS/MS	Clean ion transfer tube	LM	Daily or noticeable decrease in signal
	Clean inlet assembly	LM	Monthly or as needed
	Forepump	LM	Blast weekly; change oil every 3 months
Gas Chromatographs, Semivolatiles	Check gas supplies	LM	Daily, replace if pressure reaches 50psi
	Change in-line filters	LM	Quarterly or after 30 tanks of gas
	Change septum	LM	Daily
	Change injection port liner	LM	Weekly or as needed
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Check system for gas leaks	LM	After changing columns and after any power failure
	Clean FID	LM	Weekly or as needed
	Clean ECD	LM	Quarterly or as needed
	Leak test ECD	LM	Annually

Instrument	Activity	Maint ^a	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	LM	Daily, replace if pressure reaches 50psi
	Change in-line filters	LM	Annually or as needed
	Change septum	LM	Daily, when in use
	Change injection port liner	LM	Weekly or as needed
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Clean source	LM	As needed when tuning problems
	Change pump oil	LM	As specified by service specifications
Purge and Trap Concentrators	Change trap	LM	Every four months or as needed
	Change transfer lines	LM	Every six months or as needed
	Clean purge vessel	LM	Daily
Gas Chromatographs, Volatiles	Check gas supplies	LM	Daily, replace when pressure reaches 50 psi
	Change in-line filters	LM	Quarterly or after 30 tanks of gas
	Change septum	LM	Daily
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Check system for gas leaks	LM	After changing columns and after any power failure
	Clean PID lamp	LM	As needed
	Clean FID	LM	As needed
	Change ion exchange resin	LM	Every 60 days
	Replace nickel tubing	LM	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	LM	Daily, replace when pressure reaches 50 psi
	Change in-line filters	LM	Annually or as needed
	Change septum	LM	Daily
	Clip first foot of capillary column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Clean source	LM	As needed when tuning problems
	Change pump oil		As specified by service specifications

APPENDIX E
PREVENTIVE MAINTENANCE PROCEDURES
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Instrument	Activity	Maint ^a	Frequency
Refrigerators and Coolers	Record temperatures	LM	Daily
	Clean coils	LM	Annually
	Check coolant	LM	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	LM	Every month or as needed
Fume Hoods	Face velocity measured	LM	Quarterly
	Sash operation	LM	As needed
	Change filters	LM	Annually
	Inspect fan belts	LM	Annually
Ovens	Clean	LM	As needed or if temperature outside lim.
	Record temperatures	LM	Daily, when in use
Incubators	Record temperatures	LM	Daily, morning and evening
Water Baths	Record temperatures	LM	Daily, morning and evening
	Wash with disinfectant solution	LM	When water is murky, dirty, or when growth appears
Autoclave	Check sterility	LM	Every month
	Check temperature	LM	Every month
	Clean	LM	When mold or growth appears
	Calibrate thermometer	VM	Once a year
Analytical Balances	Check alignment	LM	Before every use
	Verify calibration	LM	Daily
	Clean pans and compartment	LM	After every use
	Certified calibration	VM	Once a year
Dissolved Oxygen Meter	Change membrane	LM	When fluctuations occur
pH probes	Condition probe	LM	When fluctuations occur
Fluoride ISE	Store in storage solution	LM	Between uses
Ammonia ISE	Store in storage solution	LM	Between uses
UV-visible Spectrophotometer	Wavelength check	VM	Twice a year

Instrument	Activity	Maint ^a	Frequency
Total Organic Carbon Analyzers	Check IR zero	LM	Weekly
	Check digestion/condensation vessels	LM	Each use
	Clean digestion chamber	LM	Every 2000 hours, or as needed
	Clean permeation tube	LM	Every 2000 hours, or as needed
	Clean six-port valves	LM	Every 200 - 2000 hours, or as needed
	Clean sample pump	LM	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	LM	Every 200 - 2000 hours, or as needed
	Clean IR cell	LM	Every 2000 - 4000 hours, or as needed
Total Organic Carbon Analyzers	Change cell electrolyte	LM	Daily
	Change electrode fluids	LM	Daily
	Change pyrolysis tube	LM	As needed
	Change inlet and outlet tubes	LM	As needed
	Change electrodes	LM	As needed
Flow Injection Analyzer	Check valve flares	LM	Each use
	Check valve ports	LM	Each use
	Check pump tubing	LM	Each use
	Check light counts	LM	Each use
	Check flow cell flares	LM	Quarterly
	Change bulb	LM	As needed
	Check manifold tubing	LM	Each use
	Check T's and connectors	LM	Each use
Discrete Auto Analyzer	Clean probe, wash reservoirs	LM	Every 2 weeks
	Replace peristaltic pump tubing	LM	Every 3 months
	Replace hydraulic circuit tubing	LM	Once/year

Instrument	Activity	Maint ^a	Frequency
Ion Chromatographs	Change column	LM	Every six months or as needed
	Change valve port face & hex nut	LM	Every six months or as needed
	Clean valve slider	LM	Every six months or as needed
	Change tubing	LM	Annually or as needed
	Eluent pump	LM	Annually
Atomic Absorption Spectrophotometers - FAA and CVAA	Check gases	LM	Daily
	Clean burner head	LM	Daily
	Check aspiration tubing	LM	Daily
Atomic Absorption Spectrophotometers - GFAA	Clean optics	LM	Every three months
	Empty waste container	LM	Weekly
	Check gases	LM	Daily
	Check argon dewar	LM	Daily
	Change graphite tube	LM	Daily, as needed
	Clean furnace windows	LM	Monthly
ICP - AES	Check argon dewar	LM	Daily
	Replace peristaltic pump tubing	LM	Daily
	Empty waste container	LM	Weekly
	Clean nebulizer, spray chamber, and torch	LM	Every two weeks
	Replace water filter	LM	Quarterly
	Replace vacuum air filters	LM	Monthly
ICP - MS	Check argon dewar	LM	Daily
	Check water level in chiller	LM	Daily
	Complete instrument log	LM	Daily
	Replace peristaltic pump tubing	LM	Daily
	Clean sample and skimmer cones	LM	As needed
	Clean RF contact strip	LM	As needed
	Inspect nebulizer, spray chamber, and torch	LM	Clean as needed
	Clean lens stack/extraction lens	LM	As needed
	Check rotary pump oil	LM	Monthly
	Change rotary pump oil	LM	Every six months

Instrument	Activity	Maint ^a	Frequency
Gel-Permeation Chromatographs	Clean and repack column	LM	As needed
	Backflush valves	LM	As needed
HPCL Chromatographs	Backflush guard column	LM	As needed
	Backflush column	LM	As needed
	Change guard column	LM	As needed when back pressure too high
	Change column	LM	Annually or as needed
	Change in-line filters	LM	As needed
	Leak check	LM	After column maintenance
	Change pump seals	LM	As needed
	Change pump diaphragm	LM	Annually
	Clean flow cell	LM	As needed
	Fluorescence detector check	LM	Daily
	Diode array absorbance check	LM	Daily
HPLC MS/MS	Clean ion transfer tube	LM	Daily or noticeable decrease in signal
	Clean inlet assembly	LM	Monthly or as needed
	Forepump	LM	Blast weekly; change oil every 3 months
Gas Chromatographs, Semivolatiles	Check gas supplies	LM	Daily, replace if pressure reaches 50psi
	Change in-line filters	LM	Quarterly or after 30 tanks of gas
	Change septum	LM	Daily
	Change injection port liner	LM	Weekly or as needed
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Check system for gas leaks	LM	After changing columns and after any power failure
	Clean FID	LM	Weekly or as needed
	Clean ECD	LM	Quarterly or as needed
	Leak test ECD	LM	Annually

Instrument	Activity	Maint ^a	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	LM	Daily, replace if pressure reaches 50psi
	Change in-line filters	LM	Annually or as needed
	Change septum	LM	Daily, when in use
	Change injection port liner	LM	Weekly or as needed
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Clean source	LM	As needed when tuning problems
	Change pump oil	LM	As specified by service specifications
Purge and Trap Concentrators	Change trap	LM	Every four months or as needed
	Change transfer lines	LM	Every six months or as needed
	Clean purge vessel	LM	Daily
Gas Chromatographs, Volatiles	Check gas supplies	LM	Daily, replace when pressure reaches 50 psi
	Change in-line filters	LM	Quarterly or after 30 tanks of gas
	Change septum	LM	Daily
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Check system for gas leaks	LM	After changing columns and after any power failure
	Clean PID lamp	LM	As needed
	Clean FID	LM	As needed
	Change ion exchange resin	LM	Every 60 days
	Replace nickel tubing	LM	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	LM	Daily, replace when pressure reaches 50 psi
	Change in-line filters	LM	Annually or as needed
	Change septum	LM	Daily
	Clip first foot of capillary column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Clean source	LM	As needed when tuning problems
	Change pump oil		As specified by service specifications

APPENDIX F

LABORATORY STANDARD OPERATING PROCEDURES

COPY

Administrative SOP Kelso

SOP Title	FILE NAME
CHECKING VOLUMETRIC LABWARE	ADM-VOLWARE
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
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	ADM-DOD
ELECTRONIC DATA BACKUP AND ARCHIVING	ADM-EBACKUP
INTERNAL QUALITY ASSURANCE AUDITS	ADM-IAUD
LABORATORY BALANCE MONITORING AND CALIBRATION	ADM-BAL
LABORATORY DATA REVIEW PROCESS	ADM-DREV
PROJECT MANAGEMENT	ADM-PCM
REAGENT LOGIN AND TRACKING	ADM-RLT
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC
SAMPLE BATCHES	ADM-BATCH
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

Technical SOP - Kelso

<u>COLIFORM, FECAL</u>	BIO-9221FC
COLIFORM, TOTAL	BIO-9221TC
<u>COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)</u>	BIO-9222D
<u>COLILERT® , COLILERT-18®, & COLISURE®</u>	BIO-9223
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B
<u>ENTEROLERT</u>	BIO-ENT
HEPTEROTROPHIC PLATE COUNT	BIO-HPC
<u>MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL</u>	BIO-QAQC
<u>SHEEN SCREEN/OIL DEGRADING MICROORGANISMS</u>	BIO-SHEEN
<u>SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION</u>	EXT-3510
<u>CONTINUOUS LIQUID - LIQUID EXTRACTION</u>	EXT-3520
<u>SOLID PHASE EXTRACTION</u>	EXT-3535
<u>SOXHLET EXTRACTION</u>	EXT-3540
<u>AUTOMATED SOXHLET EXTRACTION</u>	EXT-3541
<u>ULTRASONIC EXTRACTION</u>	EXT-3550
<u>WASTE DILUTION EXTRACTION</u>	EXT-3580
<u>SILICA GEL CLEANUP</u>	EXT-3630
<u>GEL PERMEATION CHROMATOGRAPHY</u>	EXT-3640A
<u>REMOVAL OF SULFUR USING COPPER</u>	EXT-3660
<u>REMOVAL OF SULFUR USING MERCURY</u>	EXT-3660M
<u>SULFURIC ACID CLEANUP</u>	EXT-3665
<u>CARBON CLEANUP</u>	EXT-CARCU
<u>DIAZOMETHANE PREPARATION</u>	EXT-DIAZ
<u>DMD SYNTHESIS</u>	EXT-DMD

Technical SOP - Kelso

<u>FACILITY AND LABORATORY CLEANING</u>	FAC-CLEAN
<u>OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS</u>	FAC-WATER
<u>FLASHPOINT DETERMINATION - SETAFLASH</u>	GEN-1020
<u>COLOR</u>	GEN-110.2
<u>TOTAL SOLIDS</u>	GEN-160.3
<u>SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES</u>	GEN-160.4
<u>SETTEABLE SOLIDS</u>	GEN-160.5
<u>HALIDES, ADSORBABLE ORGANIC (AOX)</u>	GEN-1650
<u>GRAVIMETRIC DETERMINATION OF HEAXANE EXTRACTABLE MATERIAL (1664)</u>	GEN-1664
<u>ALKALINITY TOTAL</u>	GEN-2320
<u>HARDNESS, TOTAL</u>	GEN-2340
<u>DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY</u>	GEN-300.1
<u>ACIDITY</u>	GEN-305.2
<u>PERCHLORATE BY ION CHROMATOGRAPHY</u>	GEN-314.0
<u>CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)</u>	GEN-325.3
<u>CHLORINE, TOTAL/FREE RESIDUAL</u>	GEN-330.4
<u>TOTAL RESIDUAL CHLORINE - METHOD 330.5</u>	GEN-330.5
<u>AMMONIA BY FLOW INJECTION ANALYSIS</u>	GEN-350.1
<u>AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE</u>	GEN-350.3
<u>NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS</u>	GEN-353.2
<u>PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE</u>	GEN-365.3
<u>PHENOLICS, TOTAL</u>	GEN-420.1
<u>ORTHOPHOSPHATE DETERMINATION USING COLORIMETRIC PROCEDURE</u>	GEN-450-PE

Technical SOP - Kelso (CONT.)

<u>DISSOLVED SILICA</u>	GEN-4500 SIO ₂ C
<u>GRAVIMETRIC SULFATE</u>	GEN-4500 SO4 C
<u>NITRITE BY COLORIMETRIC PROCEDURE</u>	GEN-4500NO2 B
<u>SULFIDE, METHYLENE BLUE</u>	GEN-4500S2D
<u>SULFIDE, TITRIMETRIC (IODINE)</u>	GEN-4500S2F
<u>TRIAZINES AS ATRAZINE by QUANTITATIVE IMMUNOASSAY</u>	GEN-4670
<u>HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION</u>	GEN-5050
<u>BIOCHEMICAL OXYGEN DEMAND</u>	GEN-5210B
<u>HALIDES, ADSORBABLE ORGANIC (AOX) - SM 5320B</u>	GEN-5320B
<u>DETERMINATION OF METHYLENE BLUE ACTIVE SUBSTANCES (MBAS)</u>	GEN-5540C
<u>TANNIN AND LIGNIN</u>	GEN-5550
<u>HALIDES, TOTAL ORGANIC (TOX)</u>	GEN-9020
<u>HALIDES, EXTRACTABLE ORGANIC (EOX)</u>	GEN-9020M
<u>TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION</u>	GEN-9030
<u>TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY</u>	GEN-9076
<u>CARBON, TOTAL ORGANIC IN SOIL</u>	GEN-ASTM
<u>AUTOFLUFF</u>	GEN-AUTOFLU
<u>SULFIDES, ACIDS VOLATILE</u>	GEN-AVS
<u>HEAT OF COMBUSTION</u>	GEN-BTU
<u>CHLOROPHYLL-a BY COLORIMETRY</u>	GEN-CHLOR
<u>TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION</u>	GEN-CN

Technical SOP - Kelso (CONT.)

<u>CYANIDE, WEAK ACID DISSOCIABLE</u>	GEN-CNWAD
<u>CHEMICAL OXYGEN DEMAND</u>	GEN-COD
<u>CONDUCTIVITY IN WATER AND WASTES</u>	GEN-COND
<u>CORROSIVITY TOWARDS STEEL</u>	GEN-CORR
<u>HEXAVALENT CHROMIUM - COLORIMETRIC</u>	GEN-CR6
<u>STANDARD TEST METHODS FOR DETERMINING SEDIMENT CONCENTRATION IN WATER SAMPLES</u>	GEN-D3977
<u>CARBONATE (CO₃) BY EVOLUTION AND COLUMETRIC TITRATION</u>	GEN-D513-82M
<u>SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT</u>	GEN-DIS.S2
<u>BULK DENSITY OF SOLID WASTE FRACTIONS</u>	GEN-E1109
<u>FDA EXTRACTABLES</u>	GEN-FDAEX
<u>FERROUS IRON IN WATER</u>	GEN-FeII
<u>FLUORIDE BY ION SELECTIVE ELECTRODE</u>	GEN-FISE
<u>FORMALDEHYDE COLORIMETRIC DETERMINATION</u>	GEN-FORM
<u>HYDROGEN HALIDES BY ION CHROMATOGRAPHY (METHOD 26)</u>	GEN-HA26
<u>HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE</u>	GEN-HYD
<u>TOTAL SULFUR FOR ION CHROMATOGRAPHY</u>	GEN-ICS
<u>ION CHROMATOGRAPHY</u>	GEN-IONC
<u>COLOR, NCASI</u>	GEN-NCAS
<u>NITROCELLULOSE IN SOIL</u>	GEN-NCEL
<u>OXYGEN CONSUMPTION RATE</u>	GEN-O2RATE
<u>CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)</u>	GEN-OSU
<u>Ph IN SOIL AND SOLIDS</u>	GEN-Phs
<u>Ph IN WATER</u>	GEN-Phw

Technical SOP - Kelso (CONT.)

<u>PARTICLE SIZE DETERMINATION - ASTM PROCEDURE</u>	GEN-PSASTM
<u>PARTICLE SIZE DETERMINATION</u>	GEN-PSP
<u>SULFIDES, REACTIVE</u>	GEN-RS
<u>TOTAL SULFIDE BY PSEP</u>	GEN-S2PS
<u>SULFITE</u>	GEN-SO3
<u>SPECIFIC GRAVITY</u>	GEN-SPGRAV
<u>SUBSAMPLING AND COMPOSITING OF SAMPLES</u>	GEN-SUBS
<u>SOLIDS, TOTAL DISSOLVED (TDS)</u>	GEN-TDS
<u>THIOCYANATE</u>	GEN-THIOCN
<u>NITROGEN, TOTAL AND SOLUBLE KJELDAHL</u>	GEN-TKN
<u>TOTAL NITROGEN AND TOTAL PHOSPHORUS BY ALKALINE PERSULFATE DIGESTION NCASI METHOD TNTP-W10900</u>	GEN-TNTP
<u>TOTAL ORGANIC CARBON IN WATER</u>	GEN-TOC
<u>SOLIDS, TOTAL SUSPENDED (TSS)</u>	GEN-TSS
<u>TURBIDITY MEASUREMENT</u>	GEN-TURB
<u>ULTIMATE BOD</u>	GEN-UBOD
<u>GLASSWASHING FOR INORGANIC ANALYSES</u>	GEN-WASH
<u>PHARMACEUTICALS, PERSONAL CARE PRODUCTS AND ENDOCRINE DISRUPTING COMPOUNDS HPLC/TANDEM MASS SPECTROMETRY (HPLC/MS/MS)</u>	LCP-1694
<u>DETERMINATION OF TRIAZINE PESTICIDES AND THEIR DEGRADATES IN WATER BY LIQUID CHROMATOGRAPHY ELECTROSPRAY IONIZATION TANDEM MASS SPECTROMETRY</u>	LCP-536
<u>ALDEHYDES BY HPLC</u>	LCP-8315
<u>Quantitative Determination of Carbamate Pesticides by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)</u>	LCP-8321
<u>NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY(HPLC)</u>	LCP-8330B
<u>Acrylamide by High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/ms/ms)..</u>	LCP-ACRYL
<u>QUANTITATIVE DETERMINATION OF AFLATOXINS By High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/ms/ms)</u>	LCP-AFLA

Technical SOP - Kelso (CONT.)

<u>Diocetyl sulfosuccinate by High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/ms/ms)..</u>	LCP-DOS
<u>QUANTITATION OF NITROAROMATICS AND NITRAMINES IN WATER, SOIL, AND TISSUE BY LIQUID CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY (LC-MS/MS)</u>	LCP-LCMS4
<u>NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY</u>	LCP-NITG
<u>QUANTITATION OF NITROPHENOLS IN SOILS BY LIQUID CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY (LC-MS/MS)</u>	LCP-NITRO
<u>ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC</u>	LCP-OALC
<u>QUANTITATIVE DETERMINATION OF OPTICAL BRIGHTENER 220 By High Performance Liquid Chromatography (HPLC)</u>	LCP-OPBr
<u>PERFLUORINATED COMPOUNDS BY HPLC/MS/MS</u>	LCP-PFC
<u>METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY</u>	MET-1630S
<u>METHYL MERCURY IN TISSUE BY ATOMIC FLUORESCENCE SPECTROMETRY</u>	MET-1630T
<u>METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY</u>	MET-1630W
<u>MERCURY IN WATER BY OXIDATION, PURGE&TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY</u>	MET-1631
<u>DETERMINATION OF ARSENIC SPECIES BY HYDRIDE GENERATION CRYOGENIC TRAPPING GAS CHROMATOGRAPY ATOMIC ABSORPTION SPECTROPOTOMETRY</u>	MET-1632
<u>MERCURY IN WATER</u>	MET-245.1
<u>METALS DIGESTION</u>	MET-3010A
<u>METALS DIGESTION</u>	MET-3020A
<u>METALS DIGESTION</u>	MET-3050
<u>CLOSED VESSEL OIL DIGESTION</u>	MET-3051M
<u>CLOSED VESSEL DIGESTION OF SILICEOUS AND ORGANICALLY BASED MATRICIES</u>	MET-3052M
<u>DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)</u>	MET-6020
<u>ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION</u>	MET-7062
<u>METALS DIGESTION FOR HEXAVALENT CHROMIUM</u>	MET-7195
<u>MERCURY IN LIQUID WASTE</u>	MET-7470A
<u>MERCURY IN SOLID OR SEMISOLID WASTE</u>	MET-7471

Technical SOP - Kelso (CONT.)

<u>SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION</u>	MET-7742
<u>SAMPLE PREPARATION OF AQUEOUS SAMPLES BY "CLEAN" TECHNIQUES</u>	MET-ACT
<u>BIOACCESSIBILITY OF METALS IN SOIL AND SOLID WASTE</u>	MET-BIOACC
<u>METALS DIGESTION</u>	MET-DIG
<u>SAMPLE FILTRATION FOR METALS ANALYSIS</u>	MET-FILT
<u>METALS LABORATORY GLASSWARE CLEANING</u>	MET-GC
<u>DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)</u>	MET-GFAA
<u>DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES</u>	MET-ICP
<u>DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)</u>	MET-ICP.MS
<u>TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS</u>	MET-RPMS
<u>METALS AND SEMIVOLATILES SPLP EXTRACTION (EPA METHOD 1312)</u>	MET-SPLP
<u>WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS</u>	MET-STLC
<u>METALS AND SEMIVOLATILES TCLP EXTRACTION (EPA METHOD 1311)</u>	MET-TCLP
<u>SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS</u>	MET-TDIG
<u>TISSUE SAMPLE PREPARATION</u>	MET-TISP
<u>ANALYSIS OF WATER AND SOLID SAMPLES FOR ALIPHATIC HYDROCARBONS</u>	PET-ALIPHAT
<u>GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY</u>	PET-GRO
<u>ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS</u>	PET-SVF
<u>ANALYSIS OF WATER AND SOLIDS SAMPLES FOR TOTAL PETROLEUM HYDROCARBONS</u>	PET-TPH
<u>ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS</u>	PHC-WIDRO

Technical SOP - Kelso (CONT.)

<u>ORGANOCHLORINE PESTICIDES AND PCBs (METHOD 608)</u>	SOC-608
<u>GLYCOLS</u>	SOC-8015M
<u>ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE</u>	SOC-8081
<u>PCBS AS AROCLORS</u>	SOC-8082Ar
<u>CONGENER-SPECIFIC DETERMINATION OF PCBs BY GC/ECD</u>	SOC-8082Co
<u>DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES</u>	SOC-8141
<u>CHLORINATED HERBICIDES</u>	SOC-8151
<u>CHLORINATED PHENOLS METHOD 8151 MODIFIED</u>	SOC-8151M
<u>SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS</u>	SOC-8270C
<u>SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - METHOD 8270D</u>	SOC-8270D
<u>SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE</u>	SOC-8270L
<u>POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC</u>	SOC-8310
<u>NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY</u>	SOC-8330
<u>RESIN AND FATTY ACIDS BY GC/MS - NCASI METHOD 85.02 MODIFIED</u>	SOC-85.02
<u>METHANOL IN PROCESS LIQUIDS AND STATIONARY SOURCE EMISSIONS</u>	SOC-9403
<u>HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES</u>	SOC-9901
<u>HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES</u>	SOC-9902
<u>ALCOHOLS</u>	SOC-ALC
<u>BUTYLINS</u>	SOC-BUTYL
<u>CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES</u>	SOC-CAL
<u>CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES</u>	SOC-CONF
<u>CPSC PHTHALATES BY GC/MS SELECTIVE ION MONITORING</u>	SOC-CPSC

Technical SOP - Kelso (CONT.)

<u>DIMP</u>	SOC-DIMP
<u>TOTAL OLEANOLIC ACID SAPONINS IN WATER BY ACID HYDROLYSIS AND HPLC/MS/MS</u>	SOC-LCMS3
<u>MONOCHLOROACETIC ACID BY GC-ECD</u>	SOC-MCA
<u>NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES</u>	SOC-NONYL
<u>ORGANOPHOSPHOROUS PESTICIDES BY GC/MS/MS</u>	SOC-OPPMS2
<u>DETERMINATION OF OTTO FUEL II IN WATER</u>	SOC-OTTO
<u>PICRIC ACID AND PICRAMIC ACID BY HPLC</u>	SOC-PICRIC
<u>POLYBROMINATED DIPHENYL ETHERS (PBDEs) AND POLYBROMINATED BIPHENYLS (PBBs) BY GC/MS</u>	SOC-ROHS
<u>SEMI-VOLATILE ORGANICS SCREENING</u>	SOC-SCR
<u>1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC</u>	SVD-504
<u>ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER</u>	SVD-508_1
<u>CHLORINATED HERBICIDES IN DRINKING WATER</u>	SVD-515.4
<u>N-NITROSAMINES BY GC/MS/MS</u>	SVD-521
<u>SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)</u>	SVD-525
<u>SELECTED PESTICIDES AND FLAME RETARDANTS IN DRINKING WATER BY GC/MS (EPA METHOD 527)</u>	SVD-527
<u>CARBAMATES AND CARBAMOYLOXIMES IN WATER BY POST-COLUMN DERIVITIZATION HPLC</u>	SVD-531 -1
<u>GLYPHOSATE IN DRINKING WATER BY HPLC</u>	SVD-547
<u>ENDOTHALL IN DRINKING WATER BY GC/MS</u>	SVD-548
<u>DIQUAT AND PARAQUAT BY HPLC</u>	SVD-549
<u>HALOACETIC ACIDS IN DRINKING WATER</u>	SVD-552

Technical SOP - Kelso (CONT.)

<u>CHLORINATED PHENOLICS BY IN-SITU ACETYLATION AND GC/MS</u>	SVM-1653A
<u>SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS</u>	SVM-625
<u>POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM</u>	SVM-8270P
<u>SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING</u>	SVM-8270S
<u>CHLORINATED PESTICIDES BY GC/MS/MS, EPA METHOD 1699 MODIFIED</u>	SVM-PESTMS2
<u>PURGE AND TRAP FOR AQUEOUS SAMPLES</u>	VOC-5030
<u>PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM</u>	VOC-5035
<u>VOLATILE ORGANIC COMPOUNDS BY GC/MS</u>	VOC-524.2
<u>VOLATILE ORGANIC COMPOUNDS BY GC/MS</u>	VOC-624
<u>AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 8021</u>	VOC-8021BTEX
<u>VOLATILE ORGANIC COMPOUNDS BY GC/MS</u>	VOC-8260
<u>VOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING</u>	VOC-8260S
<u>VOA STORAGE BLANKS</u>	VOC-BLAN
<u>SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES</u>	VOC-BVOC
<u>ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)</u>	VOC-ZHE

APPENDIX G

List of Laboratory Certifications and Accreditations

UNCONTROLLED

COPY

Federal and National Programs

- The TNI (The NELAC Institute) National Environmental Laboratory Accreditation Program (NELAP) Accredited Drinking Water, Non-Potable Water, Solid & Hazardous Waste, and Biological Tissue Laboratory
- ANSI-ASQ National Accreditation Board/ACCLASS ISO 17025:2005
- DoD- ELAP Environmental Laboratory Accreditation Program
- U.S. EPA Region 8
Approved Drinking Water Laboratory

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 Florida, Department of Health
Accredited Environmental Laboratory No. E87412
- State of Georgia, Department of Natural Resources
Certified Drinking Water Laboratory
- 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. LA080001
- 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 and Local Programs (continued)

- 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
- 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 South Carolina, Department of Health and Environmental Control
Certified Environmental Laboratory, Lab I.D. 61002
- State of Washington, Department of Ecology
Accreditation Program Lab I.D. C1203
- State of Wisconsin, Department of Natural Resources
Accredited Environmental Laboratory, Lab I.D. 998386840

A complete listing of and certifications and accreditations can be found at:

<http://www.caslab.com/Certifications/>