



REMOVAL EVALUATION WORK PLAN CHURCH ROCK SITES 1 and 1E PHASE II

Prepared For:

Rio Algom Mining LLC

Prepared By:

SENES Consultants Limited

October 2010



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PHASE II**

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ACRONYMS AND ABBREVIATIONS

AOC	Administrative Order on Consent
bgs	below ground surface
Bi-214	bismuth-214
BLM	Bureau of Land Management
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	U.S. Code of Federal Regulations
CR	Church Rock
COC	constituents of concern
C-O-C	chain of custody
cpm	counts per minute
DCGL	derived concentration guideline level
DOE	Department of Energy
DQA	data quality assurance
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
HSEC	Health, Safety, Environment & Community
IDW	investigation derived waste
IRA	Interim Removal Action
RSE	Removal Site Evaluation
MARSSIM	Multi-Agency Radiation Site and Survey Investigation Manual
MDC	minimum detectable concentration
MDL	method detection limit
NaI	sodium iodide
NMED	New Mexico Environmental Division
NMMA	New Mexico Mining Act
NNEPA	Navajo Nation Environmental Protection Agency
NPDES	National Pollutant Discharge Elimination System
OSC	On Scene Coordinator
PAL	Preliminary Action Level
pCi/g	picocuries per gram
POLREP	U.S. EPA Pollution Report
PPE	personal protection equipment
QA/QC	quality assurance / quality control
QAPP	Quality Assurance Project Plan
Ra-226	radium-226
RAML	Rio Algom Mining LLC
RSE	Removal Site Evaluation
RSO	Radiation Safety Officer
RWPR	Red Water Pond Road
SOP	Standard Operating Procedure
SOW	Scope of Work
UMTRCA	Uranium Mill Tailings Radiation Control Act
UNC	United Nuclear Corporation
VOC	Volatile Organic Chemical

1.0 INTRODUCTION

This Removal Site Evaluation (RSE) Phase II Work Plan (Work Plan) describes the objectives, scope of work and methods for conducting an RSE at Church Rock Sites 1 and 1E. The Work Plans have been prepared in two phases in accordance with the provisions of the United States Environmental Protection Agency (EPA) Administrative Order on Consent (AOC) (CERCLA Docket No. 2010-13) and the associated Scope of Work (SOW) into which they have been incorporated by reference. The AOC and SOW were previously provided as Exhibits A and B respectively of the Phase I Work Plan. The Phase I Work Plan was provided on August 26, 2010 (RAML, 2010).

This document represents the Phase II Work Plan.

1.1 SITE BACKGROUND

The former Quivira Church Rock sites are located approximately 16 miles northeast of Gallup, McKinley County, New Mexico, as shown on Figure 1.1, General Location and *Site Plan*. The Church Rock 1 and 1E sites are reclaimed and closed uranium mine sites.

From the late 1960's into early 1986, Kerr-McGee Corporation conducted exploration and the development of two underground mines at Church Rock 1 and Church Rock 1E in Section 35, T17N, R16W and Section 36, T17N, R16W, respectively of McKinley County. The land on Navajo Tribal Uranium Leases 14-20-0603-9987 and 14-20-0603-9988 respectively were leased by Kerr-McGee Corporation.

Church Rock 1 was a former underground mine where ore was hoisted to surface via a shaft and temporally stockpiled prior to truck haulage to the Quivira Ambrosia Lake milling operation. Mine water was pumped to surface and discharged to a series of holding ponds where the water was treated prior to release to the receiving environment.

A number of surface structures existed during the operating years that consisted of shaft collar and head frame, ventilation raises and ore stockpile area; office, hoist house, maintenance shops and warehousing complex; mobile equipment repair shop, fuel and oil storage facilities, main electrical transformer & switch gear, explosive storage area, internal roads and water drainage to divert water from the waste areas and rock storage areas. The areal extent of the leased area of Church Rock 1 is estimated at approximately 43 acres.

Production at Church Rock 1 ceased in 1983 and Quivira Mining Company submitted an Abandonment and Reclamation Plan to BLM in January 1987. Records indicate that the mine had been placed in standby mode on January 31, 1985. The Abandonment and Reclamation Plan

was reviewed by the BLM, Navajo Tribal Government and Bureau of Indian Affairs as part of the Department of Interiors trust responsibilities and was approved by the BLM. On September 5, 1990, a “Finding of No Significant Impact” and a final Record of Decision by the U.S. Bureau of Land Management (BLM) was issued that allowed for the reclamation of Church Rock I and IE in accordance with the stipulated conditions.

According to the plan and conditional approval, mine dewatering pumps were removed from Church Rock 1 in January 1986. Additional work outlined in the plan and approval included the following. Mine equipment to include hoists, compressors, headframes, and generators were to be removed from the site. Buildings were to be removed and foundations destroyed. Sediments from the mine water ponds were excavated and placed in shaft and ventilation raises. Pond sediments and waste rock were deposited in these underground openings. Grizzlies were to be placed over all shaft openings and monitored for 1 year for subsidence and backfilled as needed. These mining openings were then capped with a 4 foot concrete cap. Final land reclamation to include reseeding to the native landscape was to be done. Mine excavation waste piles and all disturbed areas were to be covered with a minimum of 1 foot of topsoil and reseeded with a seed mixture recommended by BIA for the Church Rock area. Bore hole foundations supporting the casing wall were to remain in place, but surface ventilation fans, transformers, switches, ductwork, electrical cables, and fences were to be removed from the bore hole area.

In addition, the ponds used as settling basins for mine solids and radium treatment facility were to be drained and allowed to dry. All sludge and settled solids were to be scraped from the sides and bottoms of the ponds and the material used to backfill the mine shafts and ventilation raises.

Church Rock 1E consisted of similar structures but on a much smaller scale. The leased area for Church Rock 1E is approximately 10 acres. Requirements for this site are addressed the same manner as for Church Rock 1 in the abandonment and reclamation document. Thus, material use at Church Rock 1E was likely to be on a smaller scale than at Church Rock 1.

Historical aerial photographs of Church Rock I (circa 1979) as shown in Figure 1.2 depict the industrial infrastructure as generally presented in the northern part of the site, the waste rock site is located on the west side of the property, and the mine water sedimentation ponds to the south and the SE sector of the property. The final clarification pond, discharges to the “unnamed” arroyo which is located in the south-eastern corner of site.

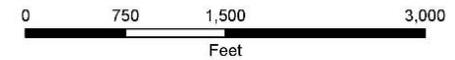
Church Rock 1E shown in Figure 1.3 is smaller but has a similar mixture of site activities. These historical photographs provide site process knowledge that is useful in the survey planning and interpretation.

Church Rock 1 and 1E Removal Site Evaluation Phase II Work Plan



Legend

 Approximate Lease Boundary & Fence Line Location



Source: Google Earth Imagery July 2005



Rio Algom Mining LLC

**General Location & Site Plan
Church Rock Sites**

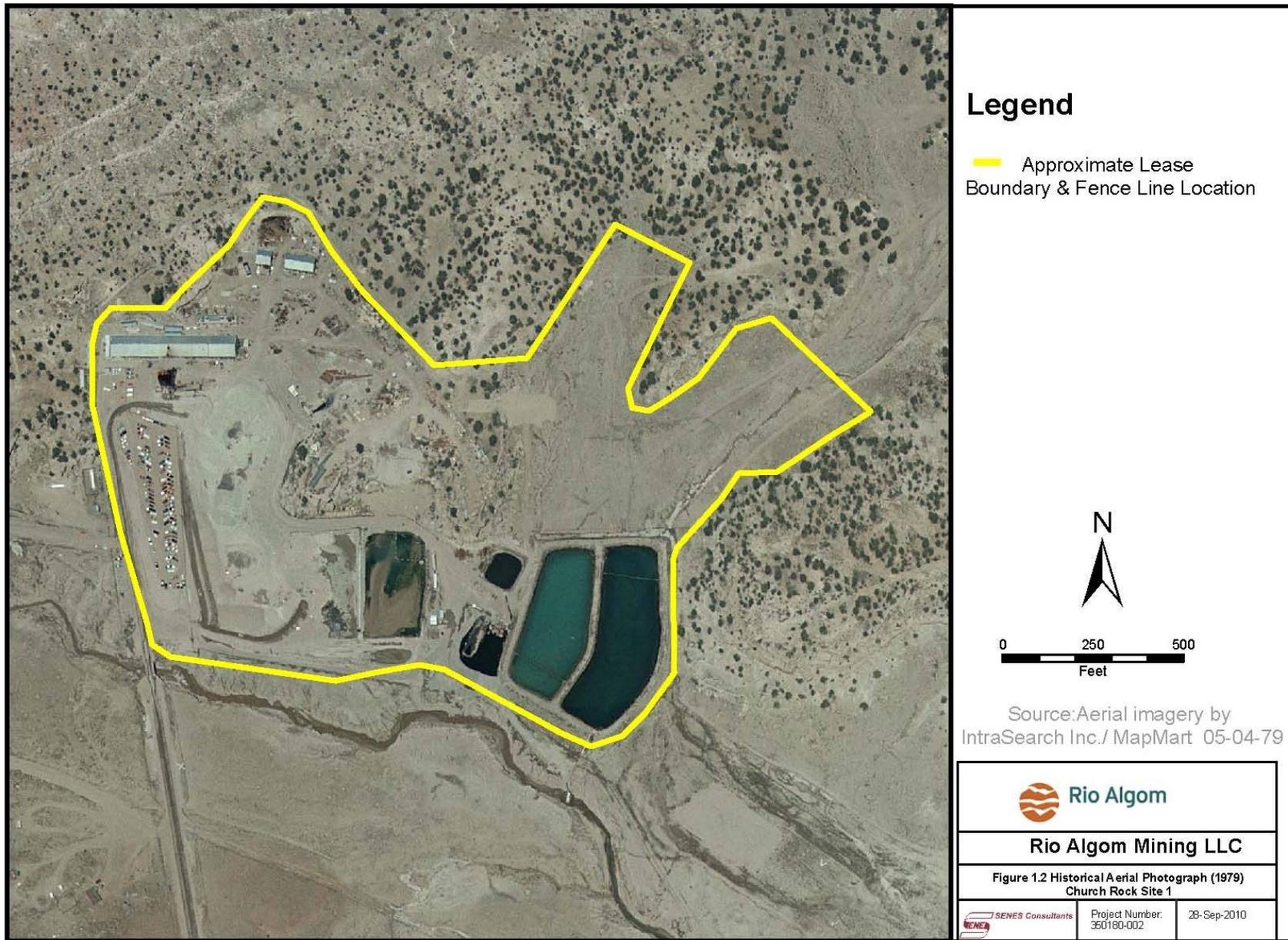


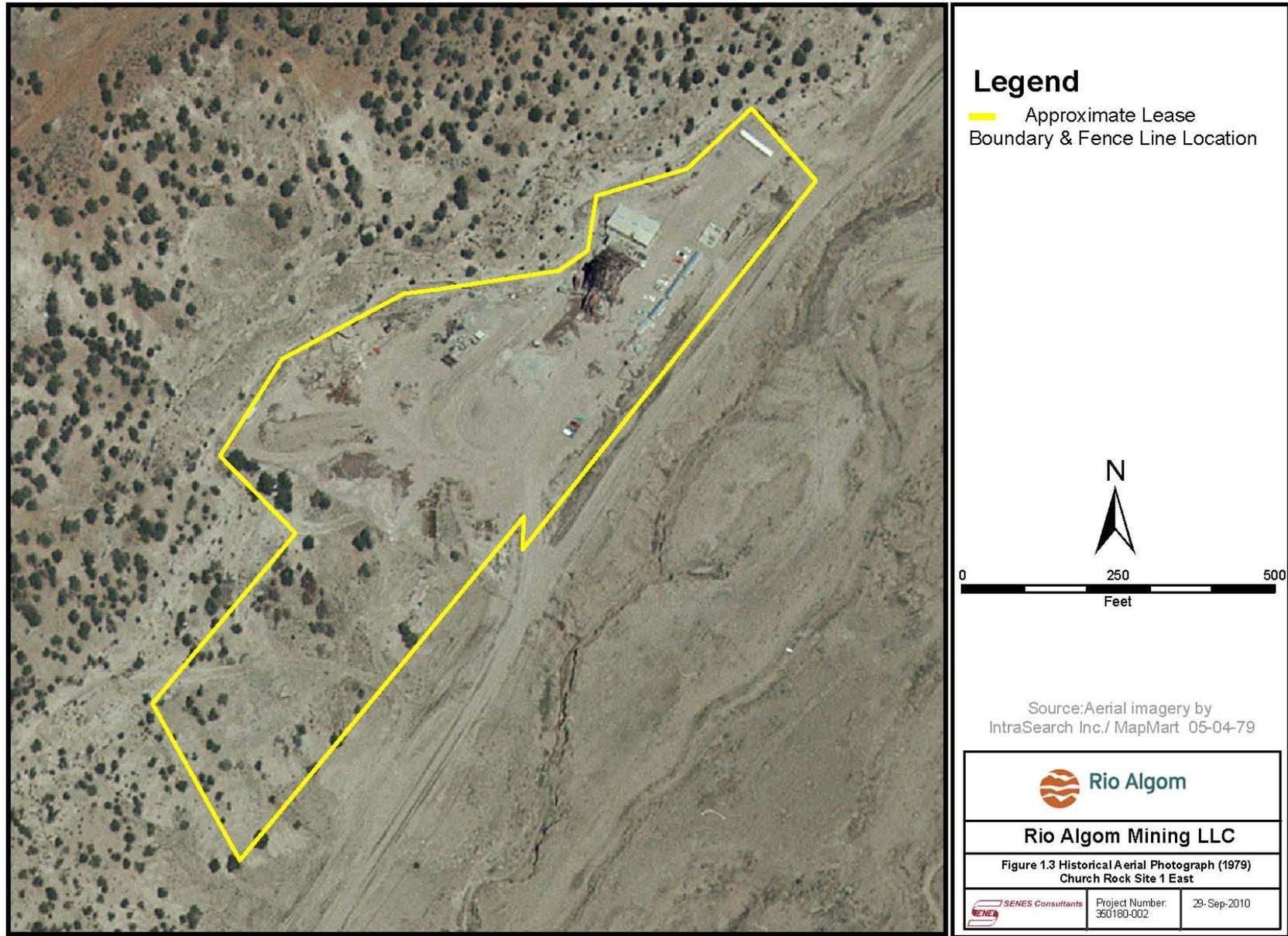
Fig. 1.1

Project Number:
350180-001

24-Aug-2010

Church Rock 1 and 1E Removal Site Evaluation Phase II Work Plan





1.1.1 Physical Setting

The Site is located in the southeastern part of the Colorado Plateau Physiographic Province. A detailed discussion of the physiography is presented in the Phase I Work Plan.

The nearest meteorological station is in Gallup. The average temperature in Gallup, 16 miles south of the Site, ranges between an average of 29 degrees Fahrenheit in January to an average of 68 degrees Fahrenheit in July. Daily extremes reach as high as 100 degrees Fahrenheit in summer and as low as -34 degrees Fahrenheit in winter. Gallup receives a total annual average precipitation of 11 inches.

Currently, areas of the Site have supported a variety of native vegetation but revegetation of some areas has had little success due to livestock grazing.

1.2 OBJECTIVES OF THE REMOVAL SITE EVALUATION

This Work Plan addresses Phase II activities of the Scope of Work for Administrative Order on Consent Interim Removal Action (IRA) (EPA, 2010). Phase II activities include characterization of the lateral and vertical extent of contamination in surface and subsurface soils at the following areas: Church Rock 1 and Church Rock 1E, along the “unnamed” arroyo in a southeasterly direction from Church Rock 1 and extending 100 feet beyond the Red Water Pond Road bridge crossing, and offsite areas (Step Outs) adjacent to the site boundary in which materials may have been carried by wind and water transport.

1.2.1 Documentation

The overriding objective of all activities is to implement the work in a safe manner that is protective of site personnel as well as nearby residents. The Field Sampling Plan previously submitted as Appendix A of the Ph I IRA Work Plan has been modified to reflect the additional activities proposed for the Phase II Work Plan. Similarly, the Quality Assurance Project Plan (QAPP) previously submitted as Appendix B in the Phase I IRA Work Plan has also been updated to reflect the range of contaminants to be sampled in Phase II.

The Health and Safety Plan and the Phase I SOP's originally provided to the EPA as Appendices C and D of the Phase I Work Plan were updated to reflect the field work proposed during Phase II and were submitted to the EPA on September 24 as part of RAML's Response to EPA's Comment Letter dated Sept. 10, 2010. The additional SOPs required to support Phase II investigations are provided in Appendix D.

1.2.2 Phase II Activities

This Phase II Work Plan addresses the program for the characterization of surface soil and subsurface areas of Church Rock (CR) Sites 1 and 1E and along the “unnamed” arroyo above and below the CR Site 1 as reflected in Figure 1.1. A detailed discussion of this planned characterization is outlined subsequent sections of this Work Plan.

Agronomic characterization will be conducted to assess the density and diversity of current vegetative cover. Parameters will be determined as described in the SOW to help with evaluation of long-term mitigation options.

2.0 PROJECT MANAGEMENT

2.1 PROJECT TEAM

The responsibilities and contact information for key project personnel as of September 30, 2010 are listed in Table 2.1 and further defined in the following sections.

Table 2.1 Site Contact Personnel

Point of Contact	Title	E-mail Address	Phone Number
Ken Black	Program Director	ken.black@bhpbilliton.com	520-247-1080 (mobile)
Scott Johnsen	Site Manager	scott.l.johnsen@bhpbilliton.com	520.419-2383 (mobile)
Doug Chambers	SENES Project Manager	dchambers@senes.ca	905-764-9380 (office)
Krista Wenzel	Health Physicist	kwenzel@senes.ca	307-315-2249 (mobile)
Bill Mckay	Field Supervisor	william.m.mckay@bhpbilliton.com	520-419-0778 (mobile)
Frank Molina	Health and Safety	frank.molina@phpbilliton.com	520-302-9753 (mobile)
Chuck Wentz	RSO	chuck.wentz@bhpbilliton.com	505-287-8851 (office)

2.1.1 Rio Algom Mining LLC (RAML) Representative

Mr. Ken Black is the Project Director for Rio Algom Mining LLC. He is responsible for overall program execution and quality, and has overall responsibility for the execution of the Work Plan activities. He will continue to take the lead on all agency communications for RAML and will be responsible for the activities of the Consultants (SENES). Mr. Black reports to the President of Rio Algom Mining LLC on this matter.

The Site Manager, Mr. Scott Johnsen, is responsible for managing all activities of the Work Plan that are associated with coordination of the field work. Mr. Johnsen will be responsible for contractor activities associated with drilling. He will also coordinate access to the Site.

RAML will appoint a health and safety representative for project execution. Mr. Chuck Wentz will act as the Project Radiation Safety Officer. Mr. Frank Molina will act as the Health and Safety Officer. William McKay is the construction field supervisor.

2.1.2 SENES Consultants Limited

The SENES Consultants Limited Project Manager, Dr. Doug Chambers, and Senior Health Physicist, Ms. Krista Wenzel, will be responsible for all activities related to chemistry, geochemistry, radiation and health physics. Dr. Chambers will have overall responsibility for coordinating the sampling and surveys, defining areas of contamination, quality of the data collected and interpretation of the data that will be presented in the investigation report, and document preparation and review.

The reporting relationships are shown in Figure 2.1. Details of signing authorities and related business confidential information are documented in RAML project files.

2.1.3 Regulatory Oversight

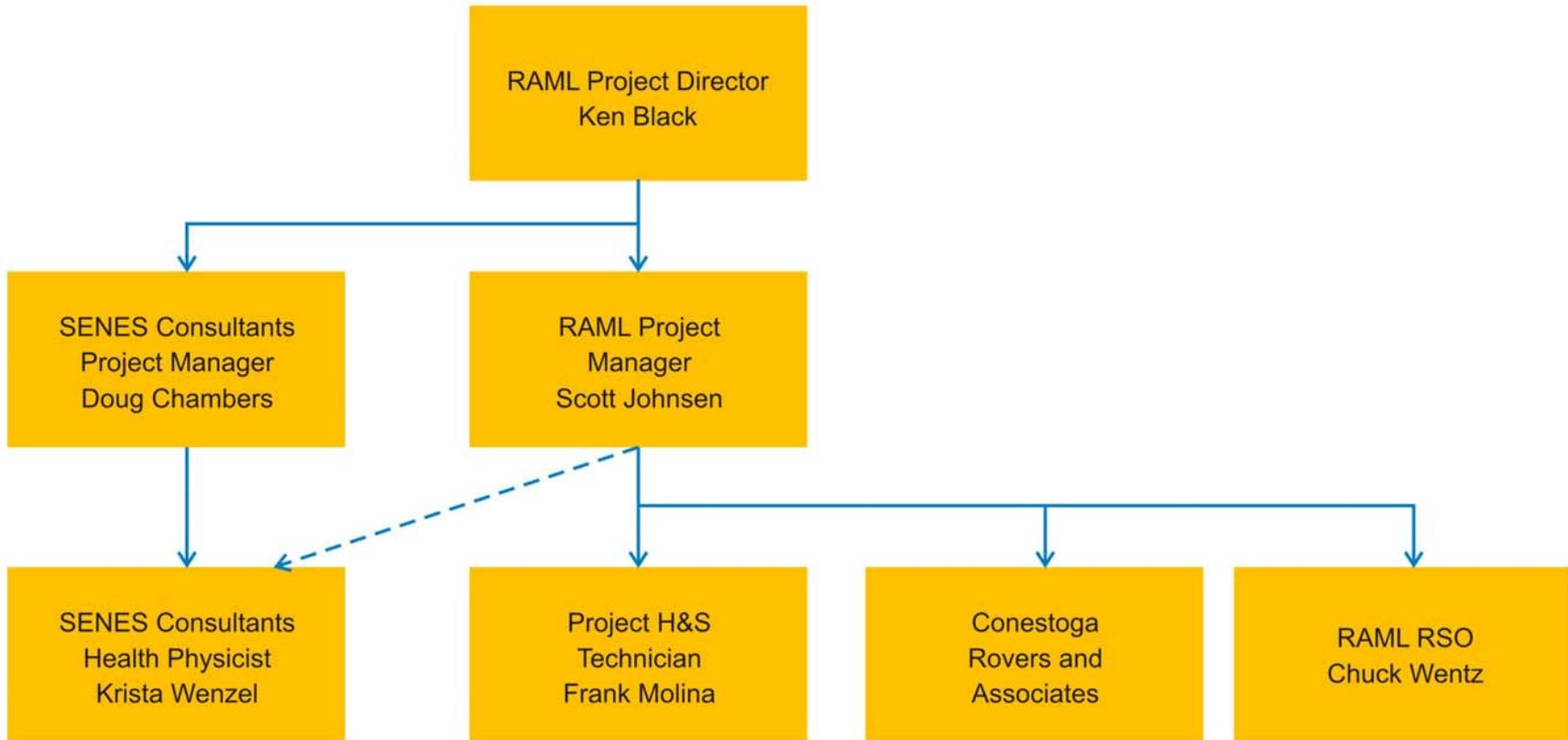
Information provided by the EPA on the regulatory oversight comprises:

- EPA Region 9 will oversee the work.
- The EPA Region 9 Remedial Project Manager (RPM) Mr. Andrew Bain is the On-Scene EPA Coordinator.

To date the specific responsibilities and authorities of the On-Scene coordinator have not been provided to RAML. All communication of approval or direction by the EPA must be provided in writing to RAML.

The role of the Navajo EPA representative(s) has not yet been defined to RAML. The Navajo EPA representative is Michele Dineyazhe.

Figure 2.1 RAML Project Team



2.2 DELIVERABLES

Within the number of working days (a day other than Saturday, Sunday and Federal Holidays) specified below, RAML will submit to EPA with a copy to NNEPA, as provided in the AOC, the following deliverables in accordance with the requirements of this Work Plan and the AOC. Unless otherwise agreed to by EPA, all submittals required by this Work Plan will be subject to 10-day EPA review and approval. Key deliverables are show in Table 2.2.

Table 2.2 Deliverables

Action/Document	Deadline
Proposed Phase I Overall Removal Action Work Plan, including: - Work Plan Outline - Construction Work Plan - Health and Safety Plan - Field Sampling Plan - Quality Assurance Project Plan	24 August 2010
Project Initiation for Phase I field activities	4 October 2010 (extended by agreement)
Completion of Phase I field activities	1 November 2010
Interim Report, including: - Phase I field activities - Sampling Report - Report using EPA pollution report (POLREP)	90 days after field work is complete monthly
Submit Phase II Overall Removal Action Work Plan, including: - Commencement of Phase II field activities - Completion of Phase II field activities	6 October 2010 (extended by agreement) 1 November 2010 1 May 2011
Comprehensive Final Report, including: - Phase I and II - Proposed post-removal site control	90 days after analytical results from the RSE are received

In addition to the hard copies and an electronic copy on a CD or DVD as specified in the AOC, an electronic copy of all deliverables created pursuant to this Work Plan should be provided electronically to the following email addresses:

Andrew Bain: Bain.Andrew@epa.gov
 Michele Dineyazhe: dineyazhe.michele@epa.gov

3.0 PHASE II WORK PLAN

3.1 BASIS OF PHASE II WORK PLAN

The Quivira Church Rock Phase II Work Plan as defined by U.S. EPA scope of work in the AOC and SOW issued August 2010, CERCLA Docket No. 2010-13 (EPA, 2010). This work consists of the site characterization of the Church Rock 1 and Church Rock 1E sites and includes the following activities:

1. Characterization of the lateral and vertical extent of contamination in surface and subsurface soils and sediments at the Church Rock 1 and Church Rock 1E, along the “unnamed” arroyo in a southeasterly direction from Church Rock 1 and extending 100 feet beyond the Red Water Pond Road bridge crossing .
2. Characterization of existing soil and vegetative cover to support agronomical assessment of the density and diversity of the vegetative cover, conduct soil analyses and to provide recommendation for cover seed mixture to be added to the vegetative plan.

The stated performance objective and specific requirements for this task is outlined in the Scope of Work for the Time-Critical Removal Action of the Administrative Order on Consent as provided in Exhibit B of the Phase I Work Plan.

For purposes of developing and executing this work plan, RAML assumes that:

- A permit is to be obtained from the New Mexico Department of Transportation for work being performed in the right-of-way (ROW) of State Highway 566;
- The Bureau of Indian Affairs have agreed to the work that will be conducted on the Red Water Pond Road;
- Safe access will be assured by EPA and Navajo EPA;
- In addition to the scope of the characterization program proposed by RAML, four additional sites are to be determined by the EPA for characterization. The locations of the four sites will be provided at least 2 weeks prior to field execution;
- EPA will consult with RAML on the four locations;
- The suite of parameters are as detailed in Section 3.2.4 of this document.

This phase of work has been divided into two main tasks and the numbering sequence follows the approach taken in Section 3.2 of the Phase I Work Plan.

Task 4a: Scope of the Characterization Studies

The SOW requires that RAML characterize surface and subsurface soils and sediments from Church Rock 1 and Church Rock 1E. The characterization work covers about 43 acres at Church Rock 1 and about 10 acres at Church Rock 1E not including Step Out areas that add approximately another 10% of the area. The tasks required include characterization of the lateral and vertical extent of contamination in surface and subsurface soils and sediments at the Church Rock Sites 1 and 1E, along the “unnamed” arroyo located immediately south of Church Rock 1 and any "Step Out" areas. This includes static and scan surveys of these areas as well as subsurface sampling to native soil in the Church Rock 1 and Church Rock 1E areas.

The scope of the sampling program will include:

- (a) waste rock areas;
- (b) former mine sedimentation ponds;
- (c) discharge point(s) into the arroyo;
- (d) mixed waste disposal areas, and;
- (e) “Step Outs” areas that are adjacent to the site boundary in which wind and water transport may carry material.

Based on historical studies and reviews, there are no known mixed waste disposal areas. Other described areas are likely to have been used at the site.

In addition, four sites are to be chosen by the EPA will be screened for a full suite of contaminants (see Section 3.2.4). In accordance with SOW requirements, the EPA will determine the locations of the four samples based on past operational history.

EPA will determine the four locations upon submittal of the Field Sampling Plan/Quality Assurance Project Plan (FSP/QAPP) work plans based on site operational history and probable usage of solvents, acids, bases and other materials. At this time, the recommended suite of parameters for analysis includes Ra-226 activity, total uranium, stable metals concentrations, volatile organic compounds, semi-volatile organic compounds and total petroleum hydrocarbons (see Section 3.2.4).

Task 4b: Characterization of Subsurface Soils

Sampling and analysis surface and sub-surface soils in the areas described will be conducted in accordance with the field sampling plan in Appendix A and the Quality Assurance Project Plan. Depth sampling techniques may incorporate auger drilling, trenching and down- the-hole drilling methods to determine the extent of waste limits and the chemical or physical characterization of the waste materials. Drilling will be employed where the native soils are too deep to intercept by other methods.

The schedule for this characterization work is dependent on the following factors:

- a. Receipt of written approval of this Work Plan by EPA, after consultation with NNEPA;
- b. Mobilization by the contractor at the Site;
- c. Subsurface drilling during the winter conditions will be weather dependent.

The contractor is to provide a schedule for completion of the characterization work. RAML expects characterization field work would be completed in less than one month from the start of the work.

Upon approval by EPA, after consultation of the Navajo Nation EPA (NNEPA), this characterization plan will be carried out by SENES Consultants. The contractor will be required to commence field work no later than 1 November 2010 (weather dependent). The specific schedule for subsurface sampling will be provided by the contractor. It is anticipated that this work will be completed by 1 May 2011.

Task 5: Characterization of the Existing Soil and Vegetative Cover

The purpose of this element of the work plan is to assess the current conditions of the vegetative cover, develop vegetative maps of the type, density and diversity of the cover material and to make recommendations to enhance the vegetative cover that will minimize erosion of soils.

Agronomic sampling to characterize existing soils and vegetation will be done along with other characterization field work. In addition to sampling, current vegetation will be inventoried and mapped. The soils will be sampled at locations based on the soil type and amount of previous disturbance, and analyzed for typical agronomic parameters important for revegetation to include: pH, texture, organic matter, available nutrients, sodium adsorption ratio (SAR), cation exchange capacity (CEC) and electrical conductivity (EC). Vegetation will be sampled for types and major species on the natural and revegetated portions of the site, and mapped at an appropriate scale.

3.2 FIELD SAMPLING PLAN

The following sections describe in detail the sampling for the above listed three tasks.

3.2.1 Sampling Plan

The field radiological stationary measurements and scans will consist of direct gamma radiation level measurements using a scintillation detector coupled with a single-channel rate meter and a

GPS. Use of GPS will facilitate development of a site survey map with radiological isopleth contours in various ranges of uncorrected raw data and Ra-226 concentrations in soil.

A static gamma radiation measurement grid based on a random origin in accordance with MARSSIM (EPA, 2000a) guidance and will have an 80 foot triangular grid. A relationship between gamma radiation and the soil sample Ra-226 concentration will be developed to predict surface soil concentrations at locations without soil samples. In addition, a roving gamma survey will be conducted of the site between these stationary points.

The sampling plan for the sites and "Step Out" areas based on an 80-foot triangular grid has been established for the two areas and this extends to adjacent "Step Out" locations just outside the areas as shown in Figure 3.1. The triangular grid is cast on a random origin in accordance with MARSSIM (EPA, 2000a) guidance documents. Static gamma radiation measurements will be collected at all these points located on the map. Locations that interfere with buried water lines, fencing or overhead power lines will be relocated in the field to the nearest offset.

Church Rock 1 and 1E Removal Site Evaluation Phase II Work Plan

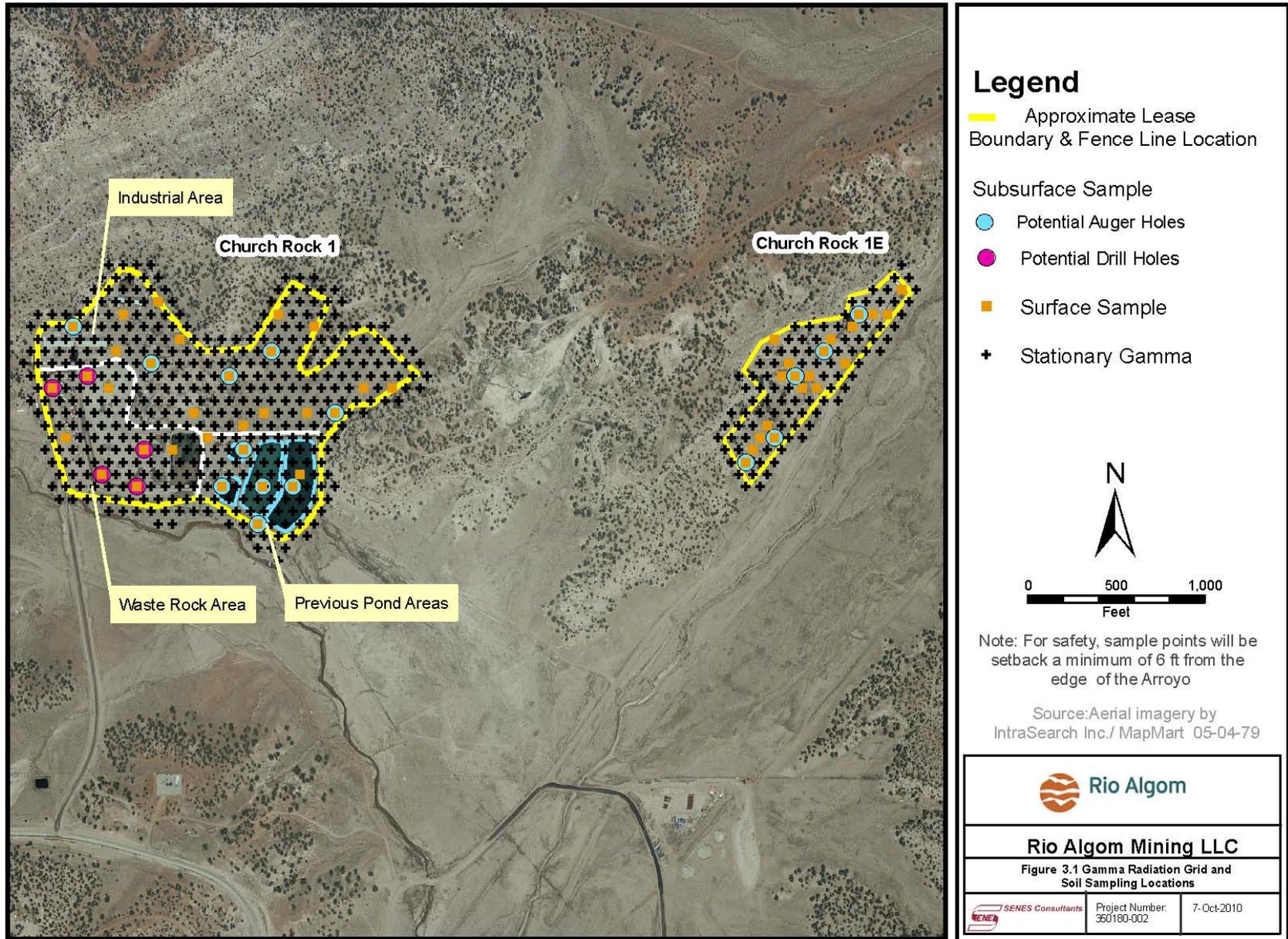


Table 3.1 provides the number of grid points considered in the project. Random soil sampling locations will be selected from these points.

Table 3.1 80 Foot Triangular Grid Points

	Church Rock 1	Church Rock 1E	Total
Boundary Area	342	76	418
Step Out	98	49	147
Total	440	125	565

Surface and subsurface soil sampling will be conducted per Phase II of the SOW. Soil samples will be collected manually as grab samples and submitted to the laboratory and analyzed for COCs as outlined in Appendix A. Sample locations will be randomly selected from the gamma radiation stationary point locations that are shown in Figure 3.1.

Surface soil sampling will be conducted at the survey areas as shown. Surface soil samples will be collected manually as grab samples at the surface (0-6 inches) as required by the SOW and submitted to the laboratory and analyzed. The surface soil samples will be collocated with the stationary gamma measurements.

Trenching, down-the-hole drilling or portable auger techniques, as appropriate, will be used to support characterization of subsurface concentrations of COCs and delineate the extent of mine waste. Drilling will be employed when it is determined the sampling depth to native soil is deeper than can be reached using trenching or power auger.

Deep subsurface soil samples, which have been defined as soil samples that are taken by the use of a drill rig, will also be collected. Depth will vary by location, surface samples will be taken at 0 to 6 inches and every 5 feet to native soil. Shallower subsurface samples will be completed using a power auger mounted on a “bobcat”.

The drill program targets may be guided by pre-mining and post-mining topographic survey data. The sampling program will be used to ascertain whether there is difference in concentrations with depth particularly for the waste rock area and the extent of the deposited materials.

Composite samples will be collected from four points determined by the EPA within the investigation area. As required by the SOW, the samples will be analyzed for Ra-226, total uranium stable metals concentration, volatile organic compounds, semi-volatile organic compounds and total petroleum hydrocarbons.

The Church Rock 1 site will be sub-divided into the following three areas; waste rock pile, pond area and industrial site. Five potential locations for subsurface sampling will be determined for each sub-area by random sampling from the surface sample locations for the waste rock and industrial areas of Church Rock 1. Five judgmental locations will be specified for the pond area

to ensure that the former ponds are measured. Thus, there are a total of 15 locations proposed on the Church Rock 1 site where subsurface investigations will occur; however, native soil may be encountered during the sub-surface investigations at the 30 to 36" soil horizon.

Five random locations will be selected from the surface soil sampling locations at the Church Rock 1E site.

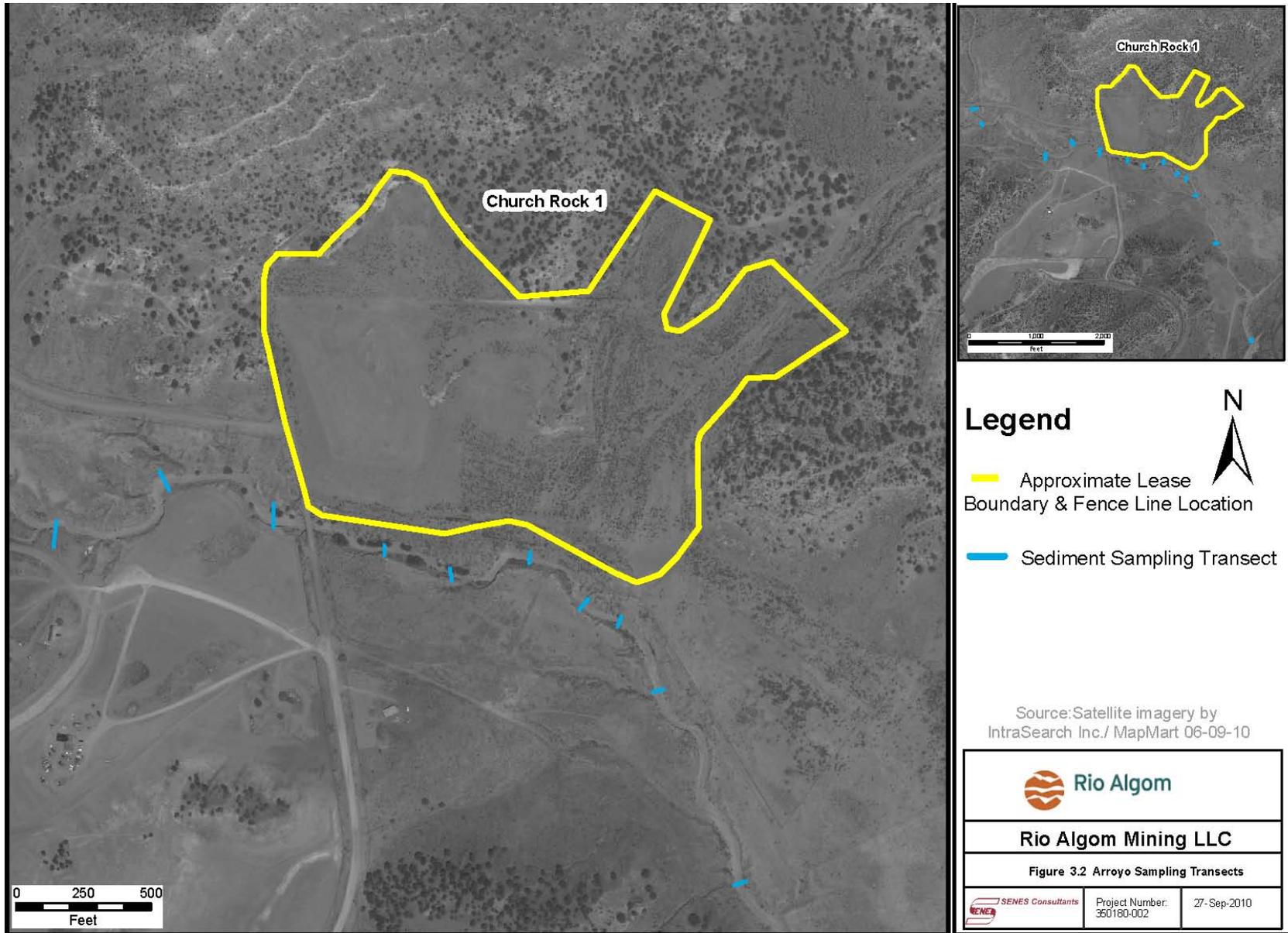
In total, there are a total of 20 drill locations for the investigation. Other than in the area of the waste rock pile, it is anticipated that sampling can be done with the use of augers. Sample splits will be collected from 10% of the locations. Split samples (replicates) will be submitted to the EPA's laboratory for quality assurance purposes.

The primary radionuclide of concern at the site is Ra-226, due to its decay into alpha-emitting radon progeny, which diffuse into the atmosphere and impose internal radiation exposure through inhalation, and gamma-emitting decay products from Ra-226 decay products remaining in the soil, which would pose a direct external radiation exposure. Thus determination of Ra-226 would provide the primary radiation hazard assessment associated with uranium ore and impacted soils. Nevertheless, determination of Ra-226 content would also provide estimation of other radionuclide concentrations of concern (U-natural and Th-230) in soil derived from uranium ore because all of the radionuclides should be in secular equilibrium. However, Ra-226 may not be able to accurately estimate other radionuclide concentrations in processed waste materials since Ra-226 is likely to be in partial secular equilibrium.

Background reference locations will be investigated and, upon EPA site approval, be measured for gamma radiation and Ra-226 surface soil concentrations. Background areas exist from previous surveys; however, the locations may not be closely enough associated with the sites to provide adequate reference to compare to on-site levels of radionuclides. As described in MARSSIM (EPA, 2000), a site background reference area should have similar physical, chemical, geological, radiological and biological characteristics as the survey unit being evaluated. Background reference areas are normally selected from non-impacted areas, but are not limited to natural areas undisturbed by human activities.

Arroyo Sediment

Arroyo sediment samples will be collected from transects along the streambed of Unnamed Arroyo from upstream of the confluence of the former UNC discharge with this Unnamed Arroyo to downstream above the confluence with the Pipeline Canyon Arroyo. The spacing of transects is closer along the boundary of the Church Rock 1 from the bridge to below the Church Rock 1 discharge point with a proposed seven locations in this area. The four upstream transects from the Church Rock 1 site will be above the UNC discharge point and two downstream sites prior to the confluence with the Pipeline Arroyo. The locations are shown on Figure 4.2; however, the exact locations will be determined in the field.



Along each transect, three grab samples will be collected for laboratory analysis of Ra-226. The three samples will be evenly spaced across the ephemeral streambed of the arroyo. Static surveys will be done at each sample point and a scanning gamma radiation survey will also be performed longitudinally along the axis of the streambed channel.

3.2.2 Quality Assurance Program

This section introduces the Quality Assurance Project Plan (QAPP) detailed in Appendix B of this document.

A Quality Assurance Project Plan (QAPP) was developed for the project and is presented in Appendix B. The QAPP was prepared to describe the project requirements for all field and Contract Laboratory activities and data assessment activities associated with this Work Plan. The QAPP presents in specific terms the policies, organization, functions, and quality assurance/quality control (QA/QC) requirements designed to meet the objectives for the sampling activities described in this Work Plan. Additionally, the QAPP provides guidance that establishes the analytical protocols and documentation requirements to ensure the data are collected, reviewed, and analyzed in a consistent manner. The QAPP was prepared in accordance with the document EPA Requirements for Quality Assurance Project Plans (EPA, 2001); the EPA guidance document Guidance for Quality Assurance Project Plans (EPA, 2002a) was also used.

Consistent with the QAPP, SENES will manage all data pertinent to this project by establishing data handling procedures and a centralized database management system. Appendix B provides details on the data management procedures that will be implemented during this project.

3.2.3 Data Evaluation

At the four locations determined by the EPA, soil samples will be analyzed for Ra-226, total uranium, Th-230, TPH, VOC, SVOC, and stable metals (arsenic, molybdenum, selenium, and vanadium). The Ra-226 results will be compared to the PAL in the SOW to identify the extent and depth of materials above the PAL.

Mapping and summary of surface gamma radiation levels will be developed for both gamma radiation count rate and predicted Ra-226 concentrations. Statistical relationships between gamma radiation and surface soil concentrations will be developed to support this.

3.3 HEALTH, SAFETY, ENVIRONMENT AND SECURITY MANAGEMENT

The specific HSE management plans developed to date are provided in the Phase I Work Plan and RAML's Response Letter to EPA's Comments dated September 10, 2010. The plan is a living document and will be updated and amended from time to time as field work is initiated. Internal risk assessment and risk management plan will be completed for each aspect of project execution, once the contractors are selected. These specific components of the HSEC management plans include:

- Safety and health management roles and responsibilities.
- Environmental management roles and responsibilities.
- Hazard identification including applicable Fatal Risk Control Standards (FRCS), work-place and task-specific hazard assessment procedures, and project-specific hazards.
- Risk mitigation and controls including applicable established Risk Management risks and controls, project-specific risks and controls.
- Safety targets and objectives including required frequency for tool box meetings, work site inspections, job and critical task observations.
- Site specific training including radiation safety.
- Project safety tasks, designates and schedule.
- Contractor Health, Safety and Environment Plans.

As part of the qualification process, the contractor will provide RAML with evidence of a HSE program that considers the normal hazards involved with fencing installation and repair projects the activities required in this Work Plan and that is consistent with the RAML corporate HSEC requirements. As part of the qualification process, the contractor will also provide RAML with evidence of an environmental management program that considers management, dust control and containment of waste. In addition, the contractor must be made familiar with the special nature of the Site conditions. These special conditions include the potential for incidental contact with residual materials from the Site operations as well as natural hazards such as wildlife. The Site's severe topographic relief imposes the need for experienced contractor personnel and the use of appropriate fall protection measures. The fence is readily accessible along the entire perimeter with safe access possible in all areas reviewed to date.

Prior to the initiation of work, RAML will provide the contractor employees with a health and safety and environment briefing an induction on HSEC and particularly regarding the Site background issues and current conditions. The topics will include potential exposure to radiation, management of hazardous or dangerous substances, and sharp or jagged metal debris. This briefing will identify areas at the Site to avoid. The Radiation Safety Officer (RSO) will also provide a briefing on radiation hazards, controls and monitoring.

RAML and the contractor will establish a communication system (satellite phone, cell phones or radios) so that emergency medical help can be summoned, if necessary. All work will be conducted in teams of at least two persons because of the remote location of the work.

Prior to field work being initiated RAML will review the Emergency Response Plan (ERP) as part of induction for all contractors and subcontractors.

RAML has met with the community, EPA and other interested parties on September 28 to discuss the background to planned remedial activities at the Church Rock sites.

3.4 EXECUTION AND CONTRACTING STRATEGY

3.4.1 Project Team

The responsibilities and contact information for key project personnel as described in Section 2 and listed in Table 2.1.

3.4.2 Reporting Relationships and Authority Levels

The reporting relationships are shown in Section 2. Details of signing authorities and related business confidential information are documented in RAML project files.

3.4.3 Licences, Permits and Statutory Approvals

RAML has been informed by the EPA that no licenses, permits or statutory approvals are required to execute the work described herein, since this work is defined by the EPA as a Time Critical Removal Action under an U.S. EPA Administrative Order on Consent dated August 2010 (EPA, 2010). RAML will submit a permit application to the NWDOT for any work that is conducted in the highway ROW. This permit will be in place prior to the commencement of chip sealing.

3.5 PROJECT CONTROL

3.5.1 Logistics

The project manager is responsible for all logistics. The project manager will be supported, as required, by staff from the RAML Ambrosia Lake site and by the Project Director.

All logistics will be defined by the site project manager. For logistical arrangements that directly affect the local residents, these arrangements will be defined in consultation with the Navajo

EPA representative and, if required, a local representative of the residents. RAML values the communities in which we work and will make every effort to complete the works without disturbing the local residents.

At this time, it is envisioned that:

- Contractors and site personnel will be lodged in Gallup.
- The project manager or his designate will be present in the field throughout the project execution.
- A staging area will be required where contractors can place vehicles and materials during field activities. If safe access can be provided, preferably this would be located on the former Quivira property. Advice will be sought by the Project Manager from the local representative on an appropriate staging area.

3.5.2 Contracts

Fair bidding processes will be employed by RAML for any services. RAML has contracted with an expert consultant, SENES Consultants for advice on the radiological and erosion management practices. Field drilling management will be provided by RAML staff and, if required, a third party contractor experienced in RAML requirements and practices.

3.5.3 Materials and Procurement

Fair bidding processes will be employed by RAML for all goods and services. Where possible, preference will be given to qualified local suppliers for services and materials. Procurement is the responsibility of the RAML project team, with advice from SENES Consultants on specialized matters related to radiation control.

3.5.4 Site Management

Site management is being conducted by RAML and this team will provide oversight to SENES Consulting and [Conestoga-Rovers & Associates \(CRA\)](#). However, if specialized services are required in the final work plan, other components of management may be subcontracted to the successful bidder.

3.5.5 QA/QC and Performance Monitoring

A QA/QC plan will be required of the contractor. This plan will be approved by RAML prior to execution.

3.5.6 Reporting and Closeout

The project reporting schedule is defined in Table 2.2 regarding project deliverables. During the project, the project manager will be responsible for:

- daily and weekly reporting from the contractors and consultants on progress, costs and safety performance, issues and exceptions;
- regular reporting to the Project Director; and
- preparation of information for any required reporting to the EPA (this has not yet been defined).

RAML will also define a reporting process to the local stakeholders – either a formal or informal process, as defined within our community consultation program.

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APPENDIX A

FIELD SAMPLING PLAN

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APPENDIX A: FIELD SAMPLING PLAN

A.1 SURFACE SOIL, SHALLOW SUBSURFACE & SEDIMENT SAMPLING

The sampling plan for the sites and "Step Out" areas based on an 80-foot triangular grid has been established for the two areas and this extends to adjacent "Step Out" locations just outside the areas as shown in Figure A.1. The triangular grid is cast on a random origin in accordance with MARSSIM (EPA, 2000a) guidance. Static gamma radiation measurements will be collected at all these points. Table A.1 provides the number of grid points considered in the project. Random soil sampling locations will be selected from these points.

Table A.1 80 Foot Triangular Grid Points

	Church Rock 1	Church Rock 1E	Total
Boundary Area	342	76	418
Step Out	98	49	147
Total	440	125	565

Arroyo sediment samples will be collected from transects along the Unnamed Arroyo from upstream of the UNC discharge to downstream above the confluence with the Pipeline Canyon Arroyo. The spacing of transects is closer along the boundary of the Church Rock 1 from the bridge to below the Church Rock 1 discharge point with a proposed seven locations in this area. The four upstream transects from the Church Rock 1 site include side arroyos above the UNC discharge point. There are two downstream sites prior to the confluence with the Pipeline Arroyo. The locations are shown on Figure A.2; however, the exact locations will be determined in the field.

Along each transect, three grab samples will be collected for laboratory analysis of Ra-226. The three samples will be evenly spaced across the ephemeral streambed of the arroyo. Static surveys will be done at each sample point and a scanning gamma radiation survey will also be performed longitudinally along the axis of the channel.

Surface and subsurface soil sampling will be conducted per Phase II of the SOW. These samples will be used to characterize Ra-226. Soil samples will be collected manually at surface, the 18 to 24" soil horizon and the 30-36" soil horizon at each surface soil sampling location.

Subsurface samples will be collected to characterization of concentrations below the 30-36" horizon samples from the surface soil program where native soil has not been reached by that depth. Samples will be collected every 5 feet of depth until native soil is reached. Subsurface samples will be collected using auger or drilling as required. At this time, it is anticipated that augers can be used to collect subsurface programs other than for the investigation at the waste

rock piles. The program will be used to ascertain whether there is difference in concentrations with depth particularly for the waste rock pile. The depths of material will be primarily defined using the differences in topography between current conditions and before mining activity (e.g. 1962); however, the subsurface investigation program will confirm these depths.

Soil grab samples from the subsurface program to be collected from the surface from 0 to 6 inches and from the subsurface every five feet from the ground surface to native soil. If the depth to native soil is less than five feet from the surface or from the previous sample, one soil grab sample will be collected from the mid-depth of the non-native material in addition to a native soil sample.

The surface and subsurface soil samples are co-located with stationary gamma measurements. The field radiological stationary measurements and scans will consist of direct gamma radiation level measurements using a scintillation detector coupled with a single-channel rate meter and a GPS. Use of GPS will facilitate development of a site survey map with radiological isopleth contours in various ranges of uncorrected raw data and Ra-226 concentrations in soil.

Four, 5-point composite samples will be collected from points determined by the EPA within the investigation area. As required by the SOW, the samples will be analyzed for Ra-226, total uranium stable metals concentration, volatile organic compounds, semi-volatile organic compounds and total petroleum hydrocarbons.

A.2 ANALYTICAL PROGRAM

Locations for surficial and near-surface sampling were selected from the triangular grid of gamma radiation static points. The target number is 30 sample locations from Church Rock 1 and 20 samples from Church Rock 1E and these are shown in Figure A.1. There will be 10 samples from outside the lease boundary for each Mine Area that will be selected based on Step Out investigations. These are randomly selected and result in 80 sample locations where surface, 18 to 24" and 30 to 36" soil samples will be collected resulting in a total of 240 soil samples.

Drilling will be used to collect sample locations at some of the surface locations; however, surface sample locations may reach native soil in the 30 to 36" soil horizon and therefore drilling will not be required at these locations. The Church Rock 1 site was sub-divided into the following three areas; waste rock pile, pond area and industrial site. Five (5) potential drill locations will be sub-sampled from the surface sample locations from each of these subareas. As required, judgmental locations have been assigned to pond areas not measured by the random sampling. There are a total of 15 random locations plus 2 judgmental locations proposed on Church Rock 1 where drilling will occur; however, native soil may be encountered during the

sub-surface investigations at the 30 to 36” soil horizon and drilling will not be required at those sites.

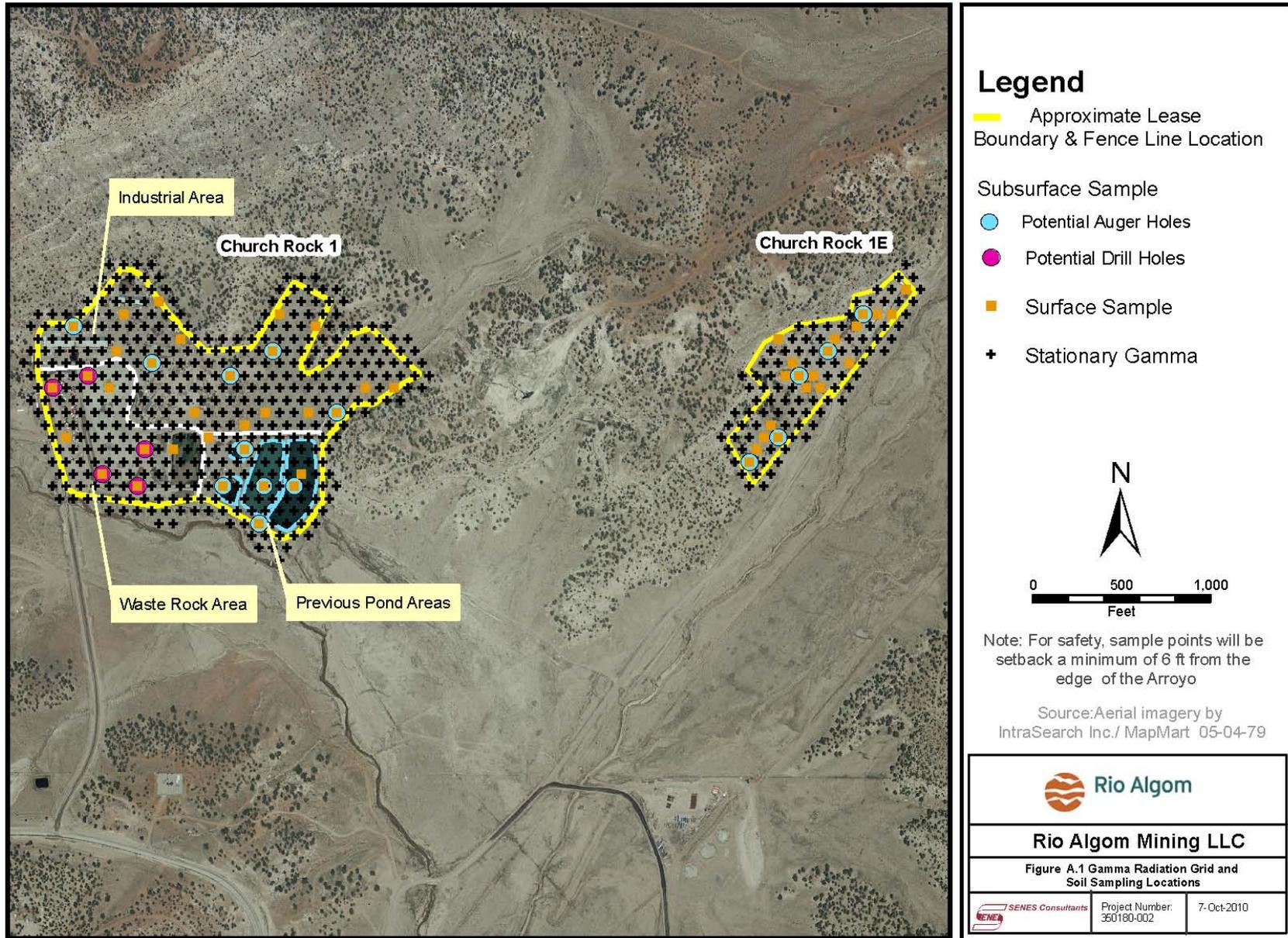
Five random locations will be selected from the surface soil sampling locations at the Church Rock 1E site. An estimate of the total number of samples is difficult since it is not clear whether native soil will already have been met from the surface program. Assuming that 12 locations have native soil deeper than 30 to 36” bgs and the average depth to native soil is 20 feet, this suggests that 60 samples may be collected from the deep drilling program. Drilling is not intended for locations outside the lease boundary. The potential grid locations are shown in Figure A.1. Should the random location be on an inaccessible area (e.g. the slope of the waste rock pile, field decisions will relocate these drilling locations to a safe position.

A total of more than 300 soil samples, plus associated QA/QC samples may be collected for laboratory analysis from within the areas. Another 60 samples (20 locations with three depths for each location) are planned for the Step Out Areas. A map of the surface sample locations is shown in Figure A.2.

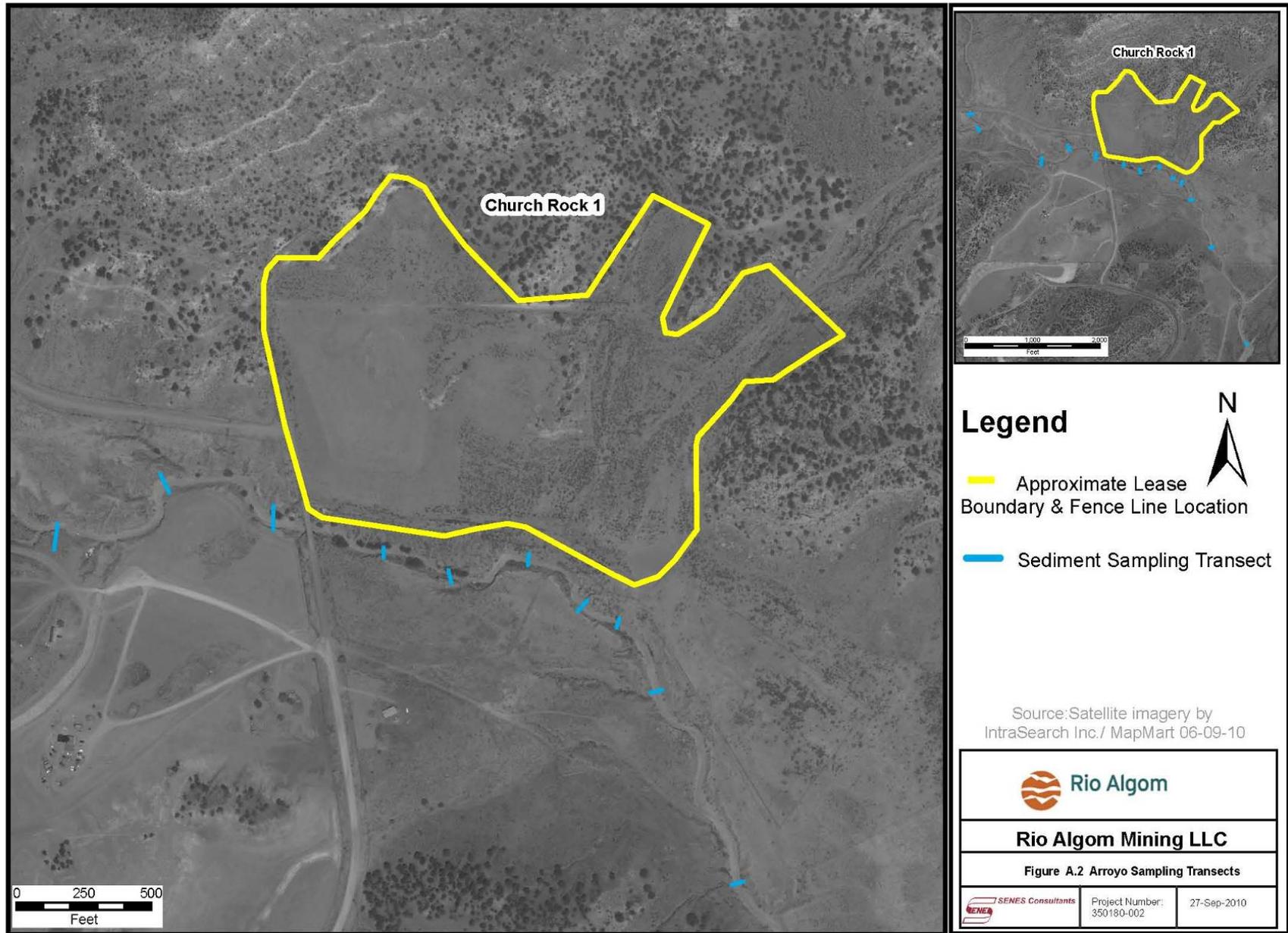
The arroyo program will have 80 samples based on 13 transects times 3 locations per transect plus related QA/QC measurements.

Background sample locations may include another 30 surface (0 to 6”) samples plus QA/QC for each background area selected.

Church Rock 1 and 1E Removal Site Evaluation Phase II Work Plan



Church Rock 1 and 1E Removal Site Evaluation Phase II Work Plan



A.3.1 Analyses

A.3.1.1 Radionuclides and Total Uranium

Ra-226 will be performed on all soil samples to characterize the type and quantity of COCs. Ra-226 will be analyzed by EPA Method 901.1 and metals by SW-846 6020 as shown in Table B.1. This table is also a summary of pertinent field sampling information (i.e., sample containers, preservative and holding times).

A.3.1.2 Stable Metals

Analysis for stable metals including arsenic, selenium, molybdenum, and vanadium will be performed on soil samples to characterize the type and quantity of COCs. They will be analyzed by SW-846 6010 as shown in Table B.1. This table is also a summary of pertinent field sampling information (i.e., sample containers, preservative and holding times).

A.3.1.3 Volatile Organic Compounds (VOC) and Semi-Volatile Organic Compounds (SVOC)

VOC and SVOC analysis will be performed on soil samples at EPA-determined locations based on operational history of the sites as shown in Figure 2.2 to characterize the type and quantity of COCs. VOC will be analyzed by EPA Method SW-846 8260B and SVOC by SW-846 8270C as shown in Table B.1. This table is also a summary of pertinent field sampling information (i.e., sample containers, preservative and holding times).

A.3.1.4 Total Petroleum Hydrocarbons (TPH)

TPH analysis will be performed on soil samples to characterize the type and quantity of COCs. TPH will be analyzed by EPA Method SW-846 8015M as shown in Table B.1. This table is also a summary of pertinent field sampling information (i.e., sample containers, preservative and holding times).

A.3.1.5 Agronomic Analysis

Agronomic analysis will be carried out for the following features:

- pH;
- Electrical conductivity;
- Saturation percentage;
- Texture;
- Rock Fragment Percentage;
- Sodium Adsorption Rate (SAR);

- Nitrate;
- Phosphorus;
- Potassium;
- Chloride;
- Sulfate;
- Organic Carbon.

Table B.2 provides further information on analytical methods and procedures.

A.3.2 Field Methods and Procedures

A3.2.1 Surface Soil, Shallow Subsurface, and Sediment Samples

Surface soil grab samples will be collected by carefully removing the top layer of soil or debris to the desired sample depth with a decontaminated spade, shovel, or equivalent. Samples collected from the area may contain large grain sizes (e.g., gravel and cobbles). An attempt will be made to select locations in the area that are free of any particularly large pieces. Shallow subsurface samples will be collected with a hand auger, shovel, or power auger, depending on soil conditions. Unless instructed otherwise, samples received by the laboratory will be analyzed "as received." Therefore, extraneous material (e.g., rocks greater than 2-inch in diameter, leaves, sticks) will be removed at the time of sample collection.

Each soil sample will be recorded on the Surface Soil Sample Log Form provided in SOP 7, Surface and Shallow Subsurface Soil Sampling. Samples will be labelled and handled following the sample preservation and chain-of-custody protocols described in this section, SOP 4, Field Documentation, and SOP 6, Sampling Handling and Shipping. Sampling equipment will be decontaminated as described in SOP 5, Equipment Decontamination. Samples for VOCs will be handled in accordance with SOP 8, Soil Sampling for VOC Analysis.

A4.2.2 Deep Subsurface Soil Samples

Deep subsurface samples (collected with the use of a drill rig) will be collected. Once the desired interval is reached, a 6-inch interval of material will be collected.

Each subsurface soil sample will be recorded on as required by SOP 9, Deep Subsurface Soil Sampling. Samples will be labelled and handled following the sample preservation and chain-of-custody protocols described in this section, SOP 4, Field Documentation, and SOP 6, Sampling Handling and Shipping. Sampling equipment will be decontaminated as described in SOP 5, Equipment Decontamination.

A.4.2.4 Radiological Field Gamma Radiation Measurements

Gamma radiation measurements are related to the amount of radioactivity in the soil are efficiently collected at a large number of points using standard methods supported by MARSSIM. The gamma radiation program will include stationary measurements at static points including soil sampling locations and with a roving survey and scan approach. A site-specific predictive relationship will be developed between the gamma radiation levels and the surface Ra-226 concentration using statistical methods.

A.4.2.4.1 Field Direct Gamma Radiation Level Correlation for Surface Soil

The radiological characterization for the surface soil consists of stationary direct gamma radiation level measurements as well as scans for additional characterization of the survey area and boundaries. The gamma radiation survey methods with the Ra-226 concentrations from soil sampling will provide the aerial extent of Ra-226 contamination in the top six-inch soil layer that will allow greater characterization of the Site compared to relying on surface soil sampling alone. Ra-226 is primarily an alpha-emitting radionuclide with a gamma radiation emission of 186 keV at about 4% intensity. Field measurement of alpha radiation from soil using radiation detection instruments is an inadequate technique due to its short range and self-absorption. The low energy and intensity of Ra-226 gamma radiation emission makes field determination of Ra-226 by gamma radiation measurement a difficult task. However, Ra-226 content in soil can be determined by measuring gamma radiation levels of its decay products Bi-214 and Pb-214. These radionuclides emit higher energy and more frequent gamma emissions that which are easily detected and quantified by a sodium iodide (NaI) scintillation detector. The field survey consisting of direct gamma radiation level measurement is consistent with the flow diagram for selection of field survey instrumentation for direct measurements presented in Figure 4.2 of the MARSSIM (EPA, 2000a).

The direct gamma radiation measurements, using a NaI scintillation detector, provide radiation levels in counts per unit time. The counts per unit time for a given radioactivity depend on the efficiency of the detector. Therefore, a site-specific correlation between direct gamma radiation levels and Ra-226 soil concentrations, as discussed in Section 6.6.2 of the MARSSIM (EPA, 2000a), may be used to convert the counts per minute (cpm) readings to the Ra-226 soil concentration in pCi/g. The conversion factor, pCi/g/cpm, is dependent upon several factors, as described below.

- Efficiency of a particular detector. The 2-inch x 2-inch NaI scintillation detector provides a high efficiency for gross gamma radiation level measurements in the field.
- The direct gamma radiation level survey for Ra-226 in soil is a surrogate for gamma measurement of Bi-214, similar to the measurement described in Section 4.3.2 of the

MARSSIM. Bi-214 is a decay product of Ra-226 through Rn-222, a gaseous form, some of which emanates from soil. This phenomenon results in activity disequilibrium between Ra-226 and Bi-214 in the soil. The fraction of Rn-222 emanation varies with different geometric characteristics of a particular soil. Therefore, a site-specific calibration is necessary.

- Other gamma-emitting naturally occurring radionuclides in soil, such as potassium-40 and Th-232 decay series, and cosmic gamma rays will be included in this gross gamma radiation level measurement. Therefore, this contribution to gross gamma count needs to be corrected. These interferences are generally constant and allow for the use of linear regression to determine the correlation with the intercept term describing the contribution from other radionuclides.

Prior to conducting the gamma radiation measurements, the operating high voltage levels of the NaI detector will be established in accordance with manufacturer instructions. The operating high voltage that will yield the lowest noise, optimum efficiency and least sensitivity to voltage fluctuations in the field will be established by determining the high voltage plateau of the detector.

The field gamma radiation correlations, static measurements, and scans for Ra-226 content in soil will be performed using a Ludlum 2221 Ratemeter/Scaler. The Ratemeter/Scaler is connected to a 2-inch by 2-inch NaI crystal scintillation detector (Ludlum 44-10), which detects gamma radiation emitted from Bi-214 and Pb-214 which are decay products of Ra-226 in the soil.

Soil samples for the correlation will be collected using the surface soil sampling SOP. A five-point composite sample at a depth of 0" to 2" and 2" to 6" will be collected from each of the gamma radiation level measurement location. One soil sample aliquot point will be from the center point directly under the detector, and the other four aliquots from four points that are 18 inches from the center points in four directions (90 degrees apart). Each soil sample aliquot will be approximately 200 grams, collected by using the hand scoop method if soil texture is loose, or a using a hand auger if soil texture is sufficiently compacted. The sampling locations will be marked with flags. The five 200-gram soil sample aliquots will be combined (total of 1000 gram) in a mixing bowl, homogenized and placed in a sample bag. Each sample bag will be marked and labeled with appropriate sample identification. Soil sampling equipment will be decontaminated between each sampling location using the SOP. All soils samples will be shipped to the radioanalytical laboratory for Ra-226 on a dry basis using EPA gamma spectroscopy method 901.1.

The selection of soil sample locations will also include background samples. Background areas will be investigated and two or more sites may be chosen as reference background areas per

MARSSIM (EPA, 2000). These areas would be chosen for population statistical tests for comparison to sampling areas to include the sites as well as the arroyo. Sample locations will be determined using an equally spaced triangular grid, cast on a random origin.

Radiation level surveys will be generally performed using a detector with lead collimator to minimize the interference. This is consistent with the technique described in Section 6.4.1.1 of MARSSIM (EPA, 2000a).

To determine the correlation between gamma radiation level counts and corresponding Ra-226 concentration in soil content (i.e. to determine a calibration factor) a linear regression analysis will be performed on the sample Ra-226 concentration in pCi/gm, and the associated gamma radiation count rate (cpm) from all the sample locations. A relationship should be developed for the paired Model 2221 rate meter and Model 44-10 detector system.

A4.2.4.2 Field Direct Gamma Radiation Level Measurements for Surface Soil

NaI scintillation detectors will be used for stationary direct radiation level measurements and scans for determining Ra-226 content in surface soils for the characterization survey. A 2-inch by 2-inch NaI detector is an appropriate detector for this type of survey (Section 6.7.2 of MARSSIM [EPA, 2000a]).

The 2-inch by 2-inch NaI detector will be connected to a single-channel rate meter, which provides necessary operating voltage to the detector. The rate meter receives signals from the detector and reports in terms of counts of radiation detected per minute. The rate meter will be setup to report gross counts, as recommended in Section 4.7.3 of the MARSSIM (EPA, 2000a). A GPS will be used to establish systematic grids. The GPS coordinates will be referenced to the New Mexico State Plane Coordinate System.

Stationary Measurements

Static surveys will be performed at specified grid nodes within survey areas or other locations, such as correlation sampling points as needed in the field. The grid nodes were determined using a 80-foot triangular grid cast on a random origin. The 80-foot triangular grid will be extended beyond the initial survey area boundary to assist with the boundary delineation evaluation. Figure A.1 shows the stationary measurement locations.

A technician will hold the detector at approximately 18 inches from the ground surface above the desired survey point to obtain a one minute integrated count. The technician will perform the static (stationary) gamma radiation survey according to the methods detailed in the SOP.

Scan Surveys

Scan radiation surveys (walkthrough surveys) will be performed by walking at a rate of about three feet per second with the detector at about 18 inches above the ground surface. Scan surveys will be performed hot spots by walking in serpentine shape along transects. The distance between transects of an area will be determined based on the static survey of the grid nodes in that survey area and will be no further than 30 feet apart within the area.

The scan radiation surveys will also be performed at survey area boundaries to delineate lateral extent of Ra-226 contamination. This scan survey will be performed by walking along the 80-foot spacing transects perpendicular to the initial perimeter of each survey area. These transects would run between the most outer 80-foot static grid node inside the initial boundary to the next 80-foot grid node outside the survey area boundary. There may be additional transects outside the area boundaries to explore the “step-out” areas.

For the scan surveys, the Ludlum 2221 with external RS-232 output connector will be coupled to a Trimble XRT Pro mapping grade GPS receiver/data logger (or similar model) to collect and store the survey data. The GPS receiver will store in the electronic data file the gamma radiation count rate and its corresponding location coordinates. This configuration can provide a gamma radiation intensity level in counts per minute (cpm) at approximately every three feet along the scan path based on a scan rate of three feet per second and reporting of count rate every second. The GPS receiver/antenna will be carried in a backpack. At the end of each survey day, the field data will be downloaded to a laptop computer for processing.

A.4.2.5 Surveying

Surveyed locations will include stationary and scan gamma measurements, surface soil samples, soil borings, excavations and other physical features, such as roads and survey area boundaries. It is anticipated that the surveying will be completed using a backpack GPS unit.

All measurements will be referenced to the State Plane Coordinate System, North American Datum 1983 and North American Vertical Datum 1988. Each sampling location will be marked with a wooden stake, a wooden lath or pin flag, and will have the corresponding sample identification number written on the marker. During surveying, the northing, easting and elevation will be stored in the GPS unit and downloaded onto a computer. In addition, the northing, easting and elevation will be recorded in a bound field notebook.

The GPS unit will be checked daily for accuracy at a control point or benchmark with a known northing and easting. The northing and easting will be recorded on a field form. Other information reported on the GPS Benchmark Elevation Form, located in Appendix B, will include date, time, weather, problems, repairs and comments.

A.4.2.6 Field Quality Control Samples

Equipment rinsate samples and field replicates will be collected for all soil sampling events. Field replicate soil samples will be collected at a rate of five percent for the primary laboratory and at a rate of 10 percent for the EPA's secondary laboratory. The field replicate soil samples will be splits of the original grab sample.

To the extent possible and practical, dedicated sampling equipment will be used. However, equipment rinsate blanks will be prepared at the Site by passing laboratory-provided reagent water of known quality through decontaminated non-dedicated sampling equipment. At the end of each day, the sampling team will take one equipment rinsate sample from each set of non-dedicated sampling equipment just before its final use.

- The field log will identify the team members, date, and sampling area. This identification procedure will associate the equipment rinsate samples with a specific team's field decontamination procedure on each day. The rinsate sample sets from the team will be submitted each day along with the field samples. Equipment rinsate samples will be collected at a frequency of one each day per analysis type. It is assumed that the non-disposable sampling equipment may include stainless steel bowls, hand trowels, shovels, split-spoon samplers, excavator bucket, and auger flights. Collection of rinsate blanks is summarized as follows: Rinsate blanks will be collected by pouring contaminant-free reagent-grade water directly over decontaminated sample collection equipment and into sample containers. The sample containers used for rinsate blanks are summarized in the QAPP location in Appendix A. Rinsate blanks will be labeled and transported to the analytical laboratory using the same procedures used for primary samples. Rinsate blanks will be analyzed for the same analytes that are specified for associated field samples.
- The laboratory will conduct the analyses of rinsate blanks in an identical fashion to the associated field samples (i.e. aqueous rinsate blank samples for soil samples will be prepared and analyzed as soil samples and reported accordingly).

Whenever rinsate blanks are sampled for VOCs and SVOCs, trip blanks will accompany the samples to the laboratory and analyzed for VOCs and SVOCs.

In addition to the rinsate samples, sample replicates (splits) of all of the surface and subsurface soil samples will be collected at a rate of 10%. The EPA will prepare an in-house split sampling plan to describe who in the EPA would verify the sampling and splitting procedures and selection. The samples will be submitted to EPA's laboratory for analysis.

A.4.2.7 Decontamination Procedures

All soil sampling equipment will be cleaned and decontaminated prior to use at each location. Additional details on decontamination procedures are located in SOP 5, Equipment Decontamination. Large equipment such as drill rigs, augers and the backhoe bucket will be decontaminated using a pressure washer, if possible. Smaller equipment such as trowels and shovels will be decontaminated as follows:

- Wash the equipment in low- or non-phosphate detergent (e.g., Alconox® or Liqui-Nox® solutions made as directed by the manufacturer);
- Rinse twice with potable water;
- Rinse once with de-ionized or distilled water; and
- Rinse water will be handled as IDW.

A.4.3 Sample Containers and Storage

After collection, samples will be properly stored to prevent degradation of the integrity of the sample prior to its analysis. As applicable, this includes analyzing the sample within prescribed holding times. Where practicable, personnel may electronically document sample handling and storage. Holding times are to be maintained from the time of sampling until the time of analysis.

All samples designated for off-site laboratory analysis will be packaged and shipped in accordance with applicable U.S. Department of Transportation regulations. Samples will be sealed in the appropriate sampling container. A chain-of-custody seal will be placed on the sample container. The samples will be packed securely in an ice chest and samples will be preserved in accordance with the specifications set forth in Table 6.2 through Table 6.4.

Samples collected for SPLP analysis will be collected in accordance with the above description of soil and sediment sampling procedures in 6.4.1. Soils collected for SPLP analysis do not require preservation or refrigeration. Once collected and placed in the sample container, it will be catalogued and properly labeled to be shipped to the laboratory accompanied with the necessary chain of custody.

A.4.4 Disposal of Investigation Derived Waste

Generation of IDW such as equipment decontamination wastewater, rinsate, soil cuttings, sample containers, and personal protective equipment (PPE) will be minimal. Soil cuttings generated from excavation will be put back into the pit once excavation is complete at each location. Any residual will be evenly spread on the ground surface on top of the pit or drill hole from which they came.

Decontamination wastewater, rinsate sample containers, and PPE will be characterized, as necessary, and disposed of in accordance with State and Federal Regulations.

A.4.5 Sample Documentation and Shipment

A.4.5.1 Field Notes

The on-site geologist/environmental scientist will use a weather-resistant, bound, survey-type field logbook with numbered, non-removable pages to record in black or blue indelible ink all field activities including soil sampling, trenching, drilling, etc. Daily information entered in the logbook will include:

- Dates and times;
- Name and location of the work activities;
- Weather conditions;
- Personnel, subcontractors and visitors on site;
- Sample locations and methods (including sampling equipment);
- Time of sample collection, and sample depths;
- Samples submitted to the laboratory for analyses;
- Sample type (e.g., soil, rinsate water, co-located, or trip blanks);
- Name of carrier transporting the sample (e.g., name of laboratory and shipping carrier);
- Photograph numbers and descriptions (if applicable);
- Description of decontamination activities;
- Schematic drawings of sample locations (if not done on field forms);
- Any deviations from the field sampling plan;
- Health & Safety meetings, including topics discussed and attendees;
- Accidents, including near misses;
- Other relevant observations as the field work progresses;
- Problems and corrective actions;
- Field equipment calibration methods;
- Investigation Derived Waste.

At the end of each field day, the project field book will be dated and signed by the field person who took notes during the day. If the entire page is not used a line will be drawn through the unused portion of the page. If pages are accidentally skipped, a line will be drawn through the entire page. All corrections will be made by drawing a line through the erroneous information and initialing the change.

If electronic record-keeping systems are employed, procedures will ensure that:

- All original entries recorded are sufficiently backed up to avoid loss;
- A system that preserves both the original record and any changes to the record, inclusive of the identification of the individual making the change, exists and will be implemented;
- An archived record of all data entries will be protected to prevent unauthorized access or amendment of the electronic data;
- Entries will be complete enough to allow for the historical reconstruction of all records;
- The review of the records will be documented.

Additional details for the project field books are located in the SOPs.

A.4.5.2 Sample Identification

All samples will be labeled in a clear, precise way for proper identification in the field and for tracking in the laboratory. The samples will have identifiable and unique numbers. Detailed sampling handling procedures are provided in the SOPs, Sample Handling and Shipping, located in Appendix C. At a minimum, the sample labels will contain the following information:

- Facility name;
- Sample number;
- Sample depth;
- Date of collection;
- Time of collection;
- Initials or name of person(s) collecting sampling;
- Analytical parameter(s);
- Method of sample preservation.

A.4.5.3 Labeling

The sample designation will be recorded on the sample label and logbook, and will comprise three parts or fields.

Samples will be numbered sequentially for each type of sample collected (i.e., surface sampling, soil boring, field gamma measurement).

- Part 1 will be designated as the survey area.
 - Q1MI, Q1SO for Quivira Mine Site 1 and step out, respectively
 - Q1EM, Q1ES for Quivira Mine Site 1E and step out, respectively
 - BKG1, BKG2, ... respectively for background areas.

- Part 2 will be a field that begins with alphabetic characters that identify the type of sample. Sample-type codes include the following:
 - ER = equipment rinsate blank
 - SS = surface soil
 - SSSa = shallow subsurface soil, 18-24 inches
 - SSSb = shallow subsurface soil, 30-36 inches
 - SBS = Subsurface soil
 - TB = trip blank
 - GM = gamma measurement
- Part 3 will be three digits that follow the alphabetic character(s) and will be sequential (e.g., "001" for the first sample location collected, "002" for the second sample location collected, "003" for the third sample collected). In the case of a soil sample at depth, Part 2 will end with depth interval, referenced to below ground surface (bgs) in parentheses. The depth will be in feet for subsurface soil and inches as required for the surface samples.

As an example, sample designation Q1EM-SS004(0-2) is the 4th surface soil sample collected from 0 to 2 inches below ground surface from the Mine Site 1. Replicate samples will be hidden from the laboratory by using a "200" identifier in the sample designation. The replicate sample designation for the example described above would be Q1EM-SS204(0-2).

A.4.5.4 Chain-of-Custody

Samples should be treated in accordance with SOP 12, Sample Handling and Shipping. Each sample and/or measurement will be properly documented to facilitate timely, accurate, and complete analysis of data. The documentation system is used to identify, track, and monitor each sample from the point of collection through final data reporting. Where practicable, this documentation system may be electronic. Chain-of-custody protocol will be implemented and followed for all samples. A sample is considered to be in a person's custody if it is: 1) in a person's physical possession, 2) in view of the person after taking possession, or 3) secured by that person so that no one can tamper with it.

Chain-of-custody forms will be used to ensure that the integrity of samples is maintained. Each form will include the following information:

- Sample number;
- Date of collection;
- Time of collection;
- Sample depth;
- Analytical parameter;

- Method of sample preservation;
- Number of sample containers;
- Shipping arrangements and airbill number, as applicable;
- Recipient laboratories;
- Signatures of parties relinquishing and receiving the sample at each transfer point.

Whenever a change of custody takes place, both parties will sign and date the chain-of-custody form, with the relinquishing person retaining a copy of the form. The party that accepts custody will inspect the custody form and all accompanying documentation to ensure that the information is complete and accurate. Any discrepancies will be noted on the chain-of-custody form.

A.4.5.5 Packaging and Shipment

All packaging will be in accordance with SOP 12, Sample Handling and Shipping. After collection, samples will be properly stored to prevent degradation of the integrity of the sample prior to its analysis. As applicable, this includes adding the appropriate chemical preservative to the sample, storing the sample in a refrigerated environment, and analyzing the sample within prescribed holding times. Where practicable, SENES may electronically document sample handling, preservation, and storage. Sample preservation and holding times are to be maintained from the time of sampling until the time of analysis.

All samples designated for off-site laboratory analysis will be packaged and shipped in accordance with applicable U.S. Department of Transportation regulations. Samples will be sealed in the appropriate sampling container. Sample containers will be placed in clean protective foam or bubble pack sleeves. The caps of all sample bottles shall be checked for tightness to prevent sample leakage during transport. Care will be taken to prevent over-tightening and breakage of bottle caps.

The samples will be packed securely in a cooler or other appropriate container, and samples will be preserved in accordance with the specification. For those samples requiring preservation at 4°C, the samples will be placed on ice in coolers in the field. Sufficient water ice (not "blue ice" or similar products) will be utilized to cool the samples during shipment. Sufficient ice shall be placed in each cooler such that: 1) some ice is still present upon arrival at the laboratory, and 2) the samples are cooled to 4 °C or below. The ice will be double wrapped in resalable plastic bags. Sufficient packing material will be placed in each ice chest to minimize the potential for sample bottles to shift and become damaged or broken during shipment. Packing material may include bubble pack or foam material. Samples should be thoroughly cooled before placing in packing material so the packing material serves to insulate the pre-cooled sample. Each cooler will contain a temperature blank consisting of a 40 millimeter vial. The drain plug on the shipping container will be closed and sealed on the inside and outside with duct tape.

Sampling personnel will inventory the sample bottles from the Site prior to shipment to ensure that all samples listed on the chain-of-custody form are present. All bottles collected from a specific sampling interval will be packed and shipped together in the same shipping container. The originals of the analysis request and chain-of-custody forms will be sealed in a waterproof plastic bag and firmly attached to the lid of the container. The cooler will be taped shut using strapping tape over the hinges and custody seals placed across the top and sides of the cooler lid. Custody seals will be used to preserve the integrity of each sample container and cooler from the time the sample is collected until it is opened by the laboratory. A custody seal will be placed over the opening of the cooler. Clear tape will be placed over the custody seals to prevent inadvertent damage during shipping. The tape should not allow the seals to be lifted off with the tape and then reattach without breaking the seal.

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN

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ATTACHMENTS

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Attachment 2 Laboratory Quality Assurance Plan (LQAP) of ALS Laboratory Group (PDF file)

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ACRONYMS AND ABBREVIATIONS

%D	percent difference
%R	percent recovery
AALA	Association of Laboratory Accreditation
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
C-O-C	chain-of-custody
CRADA	Cooperative Research and Development Agreement
°C	degrees Celsius
DOT	department of transportation
DQOs	data quality objectives
EPA	U.S. Environmental Protection Agency
ICB/CCB	initial calibration blank/continuing calibration blank
ICP	inductively coupled plasma
ICS	interference check sample
LCS	laboratory control sample
LIMS	laboratory information management system
LQMP	laboratory quality management plan
MD	matrix duplicate
MDA	minimum detectable activity
MDL	method detection limit
MS	matrix spike
MSD	matrix spike duplicate
NECR	Northeast Church Rock
NIST	National Institute of Standards and Technology
	precision, accuracy, representativeness, completeness,
PARCC	comparability
PCBs	polychlorinated biphenyls
PRGs	preliminary remediation goals
QAPP	Quality Assurance Project Plan
QA	quality assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QC	Quality Control
RCA	recommendations for corrective action
RL	reporting limit
RER	replicate error ratio
RFs	response factors RPD relative percent difference
SOP	standard operating procedure SSL soil screening level
UNC	United Nuclear Corporation
USDA	United States Department of Agriculture

APPENDIX B: QUALITY ASSURANCE PROJECT PLAN

B.1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) is a component of the Removal Site Evaluation Work Plan prepared by Rio Algom Mining LLC (RAML) specific to the Church Rock Site. This QAPP was prepared to describe the project requirements for all field and Contract Laboratory activities and data assessment activities associated with the Work Plan. This QAPP presents in specific terms the policies, organization, functions, and quality assurance/quality control (QA/QC) requirements designed to meet the objectives for the sampling activities described in the Work Plan. Additionally, this QAPP provides guidance that establishes the analytical protocols and documentation requirements to ensure the data are collected, reviewed, and analyzed in a consistent manner.

This QAPP is based on the following:

- EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5 (U.S. EPA, 2001).
- Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA/240/B-06/001. (EPA, 2006).
- EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846; U.S. EPA Third Edition, Final Update III, December 1996).
- EPA 100-400 - Series Methods for the Determination of Inorganic Substances in Environmental Samples (U.S. EPA/600R-93-100, August, 1999a).
- Prescribed Procedures for Measurement of Radioactivity in Drinking Water (U.S. EPA/600/4-80-032, August, 1980).
- Methods of Soil Analysis (American Society of Agronomy, 1982).
- United States Department of Agriculture (USDA), Handbook No. 60, (USDA, 1954).

This Quality Assurance Project Plan (QAPP) is a component of the Removal Site Evaluation Work Plan prepared for RAML specific to the Church Rock Site. The Work Plan contains a description of the Site, Site background, constituents of concern, proposed sampling activities and this QAPP, and is intended to describe the project requirements for all field, sample analysis, and data assessment activities associated with this project.

This QAPP presents in specific terms the policies, organization, functions, and quality assurance/quality Control (QA/QC) requirements to meet the project-specific objectives associated with soil sample collection and analysis. Detailed field procedures for soil sample collection and field analysis are also described in the Work Plan.

B.1.1 QAPP Objectives

The specific objective of this QAPP is to provide the guidance that will be followed for chemical analysis of soil samples to ensure that the data are of sufficient quality to support the project objectives and the data end uses. This QAPP also presents the project organization and QA/QC procedures to be followed by the Contract Laboratory for all sample analysis.

B.1.2 Document Organization

The remainder of this QAPP is organized as follows: Section B 2.0 Project Organization. This section describes the organization for this project.

- Section B 3.0 Quality Assurance Objectives for Measurement Data. This section presents the field and Contract Laboratory analytical procedures that will be followed to ensure that all measurement data collected during this project meet the project quality assurance objectives. This section also includes the procedures for instrument calibration for all anticipated analyses performed by the Contract Laboratory.
- Section B 4.0 Sampling Procedures. This section references back to the Work Plan.
- Section B 5.0 Sample Custody. This section presents the Contract Laboratory chain-of-custody (C-O-C) procedures. Field C-O-C procedures are defined in the Work Plan.
- Section B 6.0 Analytical Procedures. The analytical procedures to be used by the Contract Laboratory are presented in this section.
- Section B 7.0 Internal Quality Control Checks. The SENES and Contract Laboratory internal QC checks are presented in this section.
- Section B 8.0 Data Reduction, Reporting, Verification, and Validation. The procedures for reducing, reporting, verifying, and validating field and chemical data are defined in this section.
- Section B 9.0 Performance and Systems Audits. The SENES and Contract Laboratory procedures for performance and systems audits are presented in this section.
- Section B10.0 Preventative Maintenance Procedures. The preventative maintenance procedures that will be followed by the Contract Laboratory are detailed in this section. General procedures for field-related tasks are presented in this section; specific details will be included in the Work Plan.
- Section B 11.0.O Corrective Actions. This section defines the corrective actions that will be implemented in the event of field or Contract Laboratory non-conformances.
- Section B12.0 Quality Assurance Reports to Management. The quality assurance reporting requirements for this project are presented in this section.
 1. Attachment 1 Quality Control Procedures. This attachment includes the following information for all methods included in Table B.1:
 2. Control limits that will be used for matrix spike (MS), matrix spike duplicate (MSD), and laboratory control sample (LCS) - standard assessment.

3. Method specific calibration requirements, QC sample analysis frequency, and corrective action procedures.
4. Method specific reporting limit (RL) requirements.

The specific criteria that will be used for data assessment are as follows:

- Control Limits. The control limits for this project are based on the referenced analytical method or current industry standards.
- Calibration Requirements, QC Sample Analysis Frequency, and Corrective Action Procedures. The analytical methods listed in Section 4 were used as the source for establishing instrument calibration, QC sample analysis frequency, and corrective action requirements for this project.
- Reporting Limits. The RLs for this project will reflect the RLs established by the Contract Laboratory.

B.2.0 ORGANIZATION

At the direction of the RAML or their appointed representative, SENES will have the overall responsibility for the implementation of this project. SENES responsibilities include preparing the project plans and conducting the field activities. Descriptions of the responsibilities and authorities for the key positions as they relate to project QA and QC are provided below. In addition, the organization of the Contract Laboratory is provided in the attached ALS Quality Laboratory Assurance Plan.

B.2.1 RAML

The RAML Representative and Site Manager have the overall responsibility for the successful completion of the sampling program. They are responsible for:

- Developing scopes of work.
- Defining project objectives and schedules.
- Reviewing and analyzing overall task performance with respect to planned requirements and authorizations.
- Interfacing with the federal and state regulatory agencies. Approving all reports (deliverables) before their submission to the federal and state regulatory agencies.

B.2.2 LS Laboratory Group Staff

ALS Laboratory Group staff involved with sample preparation and analysis will consist of experienced professionals who possess the degree of specialization and technical competence to perform the required work in an effective and efficient manner.

B.2.3 ALS Laboratories Training Requirements

ALS Laboratory Group staff associated with the project will have sufficient training to safely, effectively, and efficiently perform their assigned tasks. Training records are available in the LQAP (Attachment 2).

B.3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

Data quality refers to the level of reliability associated with a particular data set or data point. The data quality associated with environmental measurement data is a function of the sampling plan rationale, the sample collection procedures, and the analytical methods and instrumentation used in making the measurements. The overall QA objective is to develop and implement procedures for field sampling, C-O-C, Contract Laboratory analysis, and data reporting that will provide data that meet task-specific objectives and that are legally defensible. Objectives are qualitative and quantitative statements that specify the field and Contract Laboratory data quality necessary to support specific decisions or regulatory actions. The objectives describe which data are needed, why the data are needed, and how the data are to be used to meet the needs of this sampling program. Objectives also establish numeric limits for the data to allow the data user (or reviewers) to determine whether the data collected are of sufficient quality for their intended use.

The objectives for this project are included in Section 3.0 of the Work Plan. The objectives were developed in accordance with the Guidance for the Objectives Process, EPA QA/G-4 (U.S. EPA, 2000). The remainder of this section defines how the data will be assessed to meet the task-specific objectives and the criteria that will be used to define acceptable limits of uncertainty.

B.3.1 Data Types

The data types required for this project are based on the task-specific objectives, the end-use of the analytical data, and the level of documentation. Both screening and definitive data will be collected. The specific type of data that will be collected for each sampling task are defined in the Work Plan. Whether data are considered screening or definitive is based on the method of sample collection, preparation, and analysis. Definitive data include data that are collected using standard sampling methodology and analytical methodology of known precision and accuracy. Screening data include data that are collected using non-standard sampling methodology or

collected using rapid, less precise methods of analysis with less rigorous sample preparation or quality control as compared to analytical methods from which definitive data are generated. For this project all data from the Contract Laboratory are considered definitive.

B.3.2 Data Quality Definition and Measurement

To determine the overall quality of definitive data, the results of QC sample analysis will be evaluated in terms of the precision, accuracy, representativeness, completeness, and comparability (PARCC) objectives established in this QAPP. The QC samples that will be used to assess the quality of both the field and Contract Laboratory data (prepared both in the laboratory and in the field) are described later in this section.

B.3.2.1 Precision

Precision is the reproducibility of measurements under a given set of conditions. For large data sets, precision is expressed as the variability of a group of measurements compared to their average value (i.e., standard deviation).

B.3.2.2 Accuracy

Accuracy is the degree of agreement of a measurement or an average of measurements with an accepted reference or "true" value, and is a measure of bias in the system. The accuracy of a measurement system is affected by errors introduced through the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analytical techniques.

Contract Laboratory Accuracy. Contract Laboratory accuracy will be assessed quantitatively through the analysis of MS/MSD samples LCS, interference check samples (metals analysis only), post digestion spikes, and response factors for calibration standards, and internal standard recoveries.

B.3.2.3 Representativeness

Representativeness is a qualitative expression of the degree to which sample data accurately and precisely represent a characteristic of a population, a sampling point, or an environmental condition. Representativeness is maximized by ensuring that, for a given task, the number and location of sampling points and the sample collection and analysis techniques are appropriate for the specific investigation, and that the sampling and analysis program provides information that reflects "true" site conditions.

Contract laboratory data will be evaluated for representativeness by assessing whether the laboratory followed the specified analytical criteria in this QAPP and their standard operating procedures (SOPs). In addition representativeness will be evaluated by assessing compliance with sample preservation and holding time criteria, and the results of method and instrument blank sample results, ICB/CCB results (metals analysis only), trip blanks, equipment rinsate blanks, source water blanks, and field replicate sample analyses.

B.3.2.4 Comparability

Comparability is a qualitative parameter that expresses the confidence with which one data set may be compared to another. Comparability is dependent on similar QA objectives and is achieved through the use of standardized methods for sample collection and analysis, the use of standardized units of measure, normalizing results to standard conditions, and the use of standard and comprehensive reporting formats as defined by this QAPP.

Contract laboratory data comparability is dependent on the use of similar sampling and analytical methodology and standard units of measure between different tasks at a specific site. For this project, chemical data will be collected using standard sampling and analyses procedures. Data comparability will also be assessed by comparing investigative sample data to QA or QC sample data.

B.3.2.5 Completeness

Completeness is the measure of the amount of valid data obtained from a measurement system relative to the amount of data scheduled for collection under correct, normal conditions. Completeness measures the effectiveness of the overall investigation in collecting the required samples, completing the required analyses, and producing valid results.

Contract laboratory data completeness is a quantitative measure of the percentage of valid data for all analytical data as determined by the precision, accuracy, and holding time criteria evaluation. Completeness will be calculated using the completeness equation by dividing the total number of valid data points by the total number of data points. The Contract Laboratory completeness goal for data collected under this QAPP is 95 percent.

If the 95 percent completeness goal is not met for field or laboratory data, the RAML Project Manager will be immediately notified. The determination regarding the need for corrective action will be based upon how critical the data are to the project objectives and will be made by the SENES and the RAML Project Managers in conjunction with federal and state regulatory agencies Project Manager.

B.3.3 Method Detection Limits, Reporting Limits, and Instrument Calibration Requirements

B.3.3.1 Method Detection Limits

The MDL is an empirically derived value that is used to estimate the lowest concentration a method can detect in a matrix-free environment. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero.

The Contract Laboratory will at a minimum perform MDL studies during initial method setup, annually, or whenever the basic chemistry of a procedure is changed. The MDLs will be method specific and include any cleanup method used. The MDLs will be established for all target analytes in an interference-free matrix using the procedures in 40 Code of Federal Regulations (CFR), Part 136, Appendix B, or an equivalent statistical approach. To ensure that the valid MDL values are determined, the laboratory will analyze an MDL check sample by spiking an interference-free matrix with all target analytes at approximately two times the calculated MDL. The MDL check sample will be taken through all the preparatory and determinative steps used to establish the calculated MDL values to verify a response is detected. If any of the target analytes are not detected, then the concentration will be increased in another MDL check sample, and the analysis repeated until the failed target analytes are detectable. The detectable target analyte concentrations will be used in lieu of the calculated MDL values to establish the lowest detected concentration for samples taken through all appropriate method* procedures. The laboratory may demonstrate continued method detection capability by analyzing the check sample on a quarterly basis, in lieu of the annual MDL study. When multiple instruments or confirmation columns are used for the same method, separate MDL studies may be replaced by the analysis of an MDL check sample on all instruments/columns. The MDL check sample will be analyzed after major instrument maintenance or changes in instrumentation or instrumental conditions to verify the current sensitivity of the method.

B.3.3.2 Reporting Limits

The RL is the lowest concentration that can be reliably achieved within limits of precision and accuracy during routine operating conditions and is based on the MDL for each analyte. The RL is established at a factor of five to ten times the MDL, but no lower than three times the MDL for any target analyte. For example RLs for the analytical methods included in this QAPP are presented in Attachment 2. The laboratory-specific RLs for each method included in this QAPP will be back checked against the project objectives to ensure that data usability goals are met. Data reporting requirements are described in Sections B7.0 and B9.0 of the QAPP.

B.3.4 Instrument Calibration

The following sub-section describes the procedures that will be used for instrument calibration by the Contract Laboratory. The procedures that will be followed for field meter or instrument calibration are detailed in the Work Plan. Analytical quality control requirements, evaluation criteria, acceptance criteria, preventative maintenance, and corrective actions are discussed later in this QAPP.

B.3.4.1 Contract Laboratory Instrument Calibration Procedures

Instrument calibration is necessary to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet the required RLs. Calibration establishes the dynamic range of an instrument, establishes response factors to be used to quantify results, and demonstrates instrument sensitivity. Criteria for calibration are specific to the instrument and the analytical method. The following paragraphs describe procedures that will be followed by the Contract Laboratory for instrument calibration.

Standard/Reagent Preparation. All instruments will be calibrated in accordance with the Contract Laboratory's SOPs. To ensure the highest quality standard, primary reference standards will be used by the Contract Laboratory and will be obtained from the National Institute of Standards and Technology (NIST), EPA Cooperative Research and Development Agreement (CRADA) vendors, American Association of Laboratory Accreditation (AALA) vendors, or other reliable commercial sources. When standards are received at the Contract Laboratory, the date received, supplier, lot number, purity, concentration, and expiration date will be recorded in a standards logbook. Vendor certifications for the standards will be retained in the files and made available upon request. Standards will be obtained in their pure form or in a stock or working standard. Dilutions will be made from the vendor standards. All records regarding standards will unambiguously trace their preparation, use in calibration, expiration dates, and quantification of sample results. All standards will be given a standard identification number, and the following information recorded in the appropriate file (standards logbook): source of standard, the initial concentration of the standard, the final concentration of the standard, the volume of the standard that was diluted, the solvent and the source and lot number of the solvent used for standard preparation, the expiration date of the standard, and the preparer's initials. All standards will be verified prior to use.

After preparation and before routine use, the identity and concentration of the standards will be verified. Verification procedures include verification of the standard's concentration by comparing its response to a standard of the same analyte prepared or obtained from a different source. Reagent purity will be assessed by analyzing an aliquot of the reagent lot using the analytical method in which it will be used; for example, every lot of laboratory grade water is

analyzed for undesirable contaminants prior to use in the laboratory. Standards will be routinely checked for signs of deterioration (e.g., discoloration, formation of precipitates, and changes in concentration), and will be discarded if deterioration is suspected or the expiration date has passed. Expiration dates will be taken from the vendor recommendation, the analytical methods, or from internal research.

Instrument Calibration. Criteria for calibration are specific to the instrument and the analytical method. Each instrument will be calibrated according to the analytical methods following manufacturer's guidelines and using standard solutions appropriate to the type of instrument and the linear range established for the method. All reported analytes will be present in both initial and continuing calibrations, which must meet the acceptance criteria specified in the analytical method. The instrument calibration will be from lowest to the highest calibration standard and the lowest calibration standard concentration will be at the RL for each target analyte.

Multipoint calibrations will contain the minimum number of calibration points specified in the method with all points used for the calibration being contiguous. If more than the minimum number of standards is analyzed for the initial calibration, all of the standards analyzed will be included in the initial calibration. The only exception is the dropping of a standard from the calibration that has been statistically determined as an outlier, providing that the requirement for the minimum number and RL standard criteria are met.

All instrument calibration information will be documented, and at a minimum include the equipment to be calibrated, the reference standards used for calibration, the calibration techniques, actions, acceptable performance tolerances, frequency of calibration, and calibration documentation format. The Contract Laboratory will maintain records of standard preparation and instrument calibration. Calibration records will include daily checks using standards prepared independently of the calibration standards, and instrument response will be evaluated against established criteria. The analysis logbook, maintained for each analytical instrument, will include at a minimum the date and time of calibration, the initials of the person performing instrument calibration, and the calibrator reference number and concentration.

B.3.5 Contract Laboratory Batch Quality Control Logic

The frequency of instrument calibration and QC sample analysis for the analytical methods are batch controlled. All sample data for this project will be associated with sample batch QC samples that were extracted or prepared concurrently with the site samples and analyzed in the same analytical batch (analyzed on the same instrument relative to the primary sample results). The identity of each preparation or analytical batch will be unambiguously reported with the analyses so that a reviewer can identify the QC samples and the associated environmental samples. The following paragraphs define sample and instrument batches.

Sample Batch. For this project, a sample batch is a group of twenty or less environmental samples of the same matrix which are extracted or prepared within the same time period (concurrently) or in limited continuous sequential time periods with the same lot of reagents. Keeping batches "open" for more than two hours will not be accepted; samples and their associated QC samples (method blank, LCS, MD, and MS/MSD) will be prepared in a continuous process. The sample batch will be analyzed sequentially on a single instrument (as practicable).

Analytical Batch. The analytical batch is a group of 20 or less environmental samples that are analyzed together within the same analytical run sequence as defined by the method calibration criteria or in continuous sequential time periods. Samples in each batch will be of similar matrix, will be treated in a similar manner, and will use the same reagents.

B.3.6 Elements of Quality Control

The quality control parameters and samples that will be used to evaluate analytical data in terms of the PARCC criteria are described in this section. These include QC samples prepared both in the field and by the Contract Laboratory. Method specific quality control procedures, frequency of QC sample analysis, acceptance criteria (control limits), and corrective action procedures are included in Attachment 2.

B.3.6.1 Field Elements of Quality Control

For field sampling, quality control samples are used to assess sample collection techniques and to assess environmental conditions during sample collection and transport. For this project, field QC samples will include temperature blanks and field replicate samples (samples that are submitted blind to the laboratory).

Temperature Blanks and Cooler Temperature: Temperature blanks will be used to evaluate the internal temperature of the cooler and assess whether the sample temperature criterion of 4°C + 2 degrees Celsius (°C) was met during sample shipment when applicable. The temperature of the blank is measured at the time the samples are received by the Contract Laboratory and recorded on the C-O-C. Temperatures that exceed the temperature criterion indicate that the samples may not have been handled or transported properly.

Trip Blanks: Trip blanks will be analyzed for VOCs to detect any potential cross-contamination of samples that may occur from sample containers, during sample transit to the laboratory, or during sample storage at the laboratory. Trip blanks will be prepared by the laboratory and consist of 40 milliliter (ml) amber glass vials filled with acidified reagent-grade water and then sealed with a cap with a Teflon™ septum. The trip blanks samples will accompany the empty

sample bottles from the laboratory to the Site. One set of trip blank samples will be placed in the sample cooler at the start of each day of sampling and remain in the cooler throughout the day. The trip blanks will then be shipped with the samples to the laboratory. Trip blanks will not be submitted with soil samples.

Equipment Rinseate Blank Samples: Equipment rinseate blank samples will be used to evaluate representativeness and will be prepared in the field (after decontamination of sampling equipment is complete) by collecting the final rinse water into the appropriate sample container. Equipment rinseate blanks will be collected on a daily basis for groundwater or surface water samples when non-dedicated equipment is used for sampling.

Field Replicate Samples: Field replicate samples are soil samples that are submitted blind to the Contract Laboratory to assess variability in the sample media and to assess sampling and analytical precision. A field replicate sample is a single grab sample that is replicated into two samples during collection. For each field replicate sample pair, one of the samples is labeled with the correct sample identification and the other is labeled with fictitious sample identification. This replicate sample pair is then submitted to the same Contract Laboratory as two separate samples. Precision will be evaluated by calculating the RPD between the field replicate sample pairs for all analytes detected at or above the RL. RPD calculations will not be performed when either one or both of the sample results for the field replicate sample pairs are reported as less than the RL.

Although the RPD will be calculated between field replicate samples, the results will not be used as a basis for qualifying data or accepting or rejecting data. The RPD and actual results will be evaluated qualitatively to assess precision of field sample collection procedures. An RPD within ± 30 percent will be used as an indication of good agreement between the parent and replicate sample results and that good field procedures were followed.

B.3.6.2 Contract Laboratory Elements of Quality Control

The Contract Laboratory will, as a minimum, analyze internal QC samples at the frequency specified by the analytical method and in this QAPP. Method-specific quality control procedures, frequency of QC sample analysis, acceptance criteria (control limits), and corrective actions are provided in Attachment 2. The following paragraphs discuss holding time and the QC samples that will be used to assess laboratory data quality.

Sample Holding Time: Sample holding time reflects the length of time that a sample or sample extract remains representative of environmental conditions. For methods that do not require sample extraction one holding time will be evaluated, the length of time from sample collection to analysis. For methods that require sample extraction prior to analysis two holding times will

be evaluated; the length of time from sample collection until sample extraction, and the length of time from sample extraction to sample analysis. These holding times will be compared to the holding times specified by the respective analytical method. The holding times for each analytical method included in this QAPP are listed in Attachment 1. Samples will not be analyzed outside of the specified method holding times without approval by the SENES Project Manager.

Method Blanks: Method blanks will be used to monitor the Contract Laboratory preparation and analytical systems for interferences and contamination from glassware, reagents, sample manipulations, and the general laboratory environment. The method blank is an analyte-free matrix (reagent grade water or laboratory grade sand) to which all reagents will be added in the same volumes or proportions as used in sample processing. Method blanks will be taken through the entire sample preparation/extraction and analytical process. Method blanks will be prepared and analyzed with each analytical or preparation batch of environmental samples up to a maximum of 20 samples of a similar matrix. No analytical data will be corrected for the presence of analytes in blanks.

Internal Standards. Internal standards are compounds that behave similarly to the target analytes during analysis and will be used to assess accuracy for gas chromatography/mass spectroscopy (GC/MS) analysis. Internal standards will be prepared and added to the initial calibration standard (ICAL), the continuing calibration verification standard (CVS), and all samples (field and QC) prior to analysis. Internal standard data will be reviewed for compliance with the analytical method acceptance criteria.

Surrogate Spikes. Surrogate spikes will be used to evaluate the accuracy of analytical instrument performance for all organic analysis. Surrogate spikes will be added to each sample for organic compound analysis, including QC samples, prior to extraction as specified in the laboratory's standard operating procedure (SOP). The percent recovery of each surrogate spike will be calculated and compared to the project acceptance criteria (Attachment 2).

Initial and Continuing Calibration Blanks. Initial and continuing calibration blank (ICB/CCB) samples are analyzed with each sample batch with method SW-846 6020 (ICP) to determine whether metals are introduced into samples during preparation by the laboratory. The same criteria that used to evaluate method are used to evaluate the ICB/CCB and associated sample data.

Laboratory Control Samples. Laboratory control samples will be used to measure laboratory accuracy in the absence of matrix interference. Laboratory control samples are prepared in the laboratory and consist of samples of a known matrix (reagent grade water or laboratory grade sand) spiked with a known quantity of specific target analytes at a level less than or equal to the

midpoint of the calibration curve for each analyte. The midpoint is defined as the median point in the curve, not the middle of the range. These samples are taken through the entire sample preparation and analytical process. LCSs will be prepared and analyzed with each analytical or preparation batch of environmental samples up to a maximum of 20 samples of a similar matrix. If more than one LCS is analyzed in an analytical batch, results from all LCSs analyzed will be reported.

Matrix Spikes and Matrix Spike Duplicates. Matrix spikes measure matrix-specific method performance and will be used to assess accuracy and precision. Unlike LCSs, MS/MSD samples will be used to assess the influence of the sample media (media interference) on sample analysis. Samples for MS/MSD analysis will be collected from each sampling location and will be media specific (e.g., sediment, sludge, and groundwater). A minimum of one MS/MSD sample pair will be analyzed with every batch of RAML samples in a sample delivery group of up to 20 field samples. Each MS/MSD sample will be spiked with the compounds specified by this QAPP prior to sample extraction or analysis at a concentration less than or equal to the midpoint of the calibration curve for each analyte. The sampled scheduled for MS/MSD analyses will be designated on the C-O-C form.

Matrix Duplicate Samples. Matrix duplicate samples are identical to field replicates, except that the duplicate sample does not have a false identification. Precision will be evaluated by calculating the RPD between the MD and parent sample pairs for all analytes detected at or above the RL. RPD calculations will not be performed when either one or both results is less than the RL.

Interference Check Sample. The interference check sample (ICS), used in inductively coupled plasma (ICP) analyses only, contains both interfering and analyte elements of known concentrations and is analyzed at the beginning and end of each run sequence. The ICS is used to verify background and interelement correction factors.

Serial Dilution. Serial dilutions are conducted for metals analysis to assess positive or negative interferences when the concentration of a metal detected in a sample is ten times greater than the instrument detection limit (after sample dilution). A five-fold dilution of the sample is analyzed and compared to the results of the original analysis. If the difference between the original and diluted sample results is greater than 10 percent, a chemical or physical interference is suspected.

Field Replicates. As discussed previously, field replicates will be used to assess both sampling and analytical precision. The purpose of submitting samples "blind" to the Contract Laboratory is to assess the consistency or precision of the laboratory's analytical system. Precision will be evaluated by calculating the RPD between the parent and field replicate samples.

As discussed previously, although the RPD will be calculated between field replicate samples, the results will not be used as a basis for qualifying data or accepting or rejecting data. The RPD and actual results will be evaluated qualitatively as additional evidence to support data comparability and quality. An RPD within + 30 will be used as an indication of good agreement between the parent and duplicate sample results and that good laboratory procedures were followed.

B.4.0 SAMPLING PROCEDURES

B.4.1 Sample Collection Procedures

The sample collection procedures are defined in Appendix A of the Work Plan.

B.5.0 SAMPLE CUSTODY AND SHIPPING

To ensure that samples are identified correctly and remain representative of the environment, the sample documentation and custody procedures outlined in this section will be used during the sampling program to maintain and document sample integrity during collection, transportation, storage, and analysis. Field sampling personnel will be responsible for ensuring that proper documentation and custody procedures are initiated at the time of sample collection, and that individual samples can be tracked from the time of sample collection until custody of the samples is transferred to the Contract Laboratory. The Contract Laboratory will be responsible for maintaining sample custody and documentation from the time the laboratory receives the samples until final sample disposition.

B.5.1 Chain-of-Custody

C-O-C procedures provide an accurate written record of the possession of each sample from the time it is collected in the field through laboratory analysis. A sample is considered in custody if one of the following applies:

- It is in an authorized person's immediate possession.
- It is in view of an authorized person after being in physical possession.
- It is in a secure area after having been in an authorized person's physical possession.
- It is in a designated secure area, restricted to authorized personnel only.

B.5.1.1 Contract Laboratory Chain-of-Custody Procedures

Upon receipt by the Contract Laboratory, the integrity of the shipping container will be checked by verifying that the custody seals are not broken. The cooler will be opened and examined for

evidence of proper cooling and the presence of temperature blanks when applicable. The individual sample containers will be checked for breakage, damage, or leakage. The contents of the shipping container will then be verified against the C-O-C. If any problems are found, they will be documented on the sample custody form(s) and the SENES Project Manager will be notified immediately. The shipping receipts will be placed with the C-O-C records and stored in the project files.

If the samples and documentation are acceptable, each sample container will be assigned a unique laboratory identification number and entered into the laboratory's sample tracking system. Sample tracking will be documented in the LIMS, or other appropriate tracking system. Other information that will be recorded includes date and time of sampling, sample description, due dates, and required analytical tests.

The Contract Laboratory will follow their SOPs for sample log-in, storage, tracking, and control (Attachment 2). Sample custody will be maintained within the laboratory's secure facility until the samples are disposed. The Contract Laboratory will be responsible for sample disposal, which will be conducted in accordance with all applicable local, state, and federal regulations. All sample disposals will be documented and the records maintained by the Contract Laboratory in the project file.

B.5.2 Sample Packaging and Shipping Procedures

All samples will be shipped in accordance with all applicable State and Federal Department of Transportation (DOT) requirements. The following paragraphs describe general sample packaging requirements.

All samples will be packaged and shipped to Fort Collins, Colorado within two business days of sample collection via a commercial carrier according to SOP 12 "Sample Handling and Shipping" and by using the following procedures:

- Sample labels will be completed and attached to sample containers.
- The samples will be placed upright in a waterproof metal or equivalent strength plastic ice chest or cooler.
- Wet ice in double Ziploc™ bags (to prevent leakage) will be placed around, among, and on top of the sample bottles when applicable. Enough ice will be used so that the samples will be chilled and maintained at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ during transport to the laboratory.
- To prevent the sample containers from shifting inside the cooler, the remaining space in cooler will be filled with inert cushioning material, such as shipping peanuts, additional bubble pack, or cardboard dividers.
- The original copy of the completed C-O-C Form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid.

- The lid will be secured by wrapping strapping tape completely around the cooler in two locations.
- "This Side Up" labels will be placed on two sides of the cooler.
- Custody seals will be placed in two locations (the front right and back left of the cooler) across the cooler closure to ensure that any tampering is detected. The date and initials of the sampler will be written on the custody seal.
- A copy of the C-O-C record and the signed air bill will be retained for the project files.
- The samples will be shipped priority to:

ALS Laboratory Group / 225 Commerce Drive / Fort Collins, CO 80524
ph: (970) 490-1511 / toll free (800) 443-1511 / fax: (970) 490-1522

B.5.3 Final Project Files Custody Procedures

The final project files will be maintained by SENES and will be under the custody of the Project Manager in a secured area. At a minimum, the project file will contain all relevant records including:

- Field logbooks
- Field data and data deliverables
- Photographs
- All original field logs
- Clean container certifications from laboratory.
- Contract Laboratory data deliverables.
- Data verification reports.
- Data assessment reports.
- Progress reports, QA reports, interim study reports, etc
- All custody documentation (tags, forms, airbills, etc.).

B.6.0 ANALYTICAL PROCEDURES

This section describes the analytical procedures that will be used for the acquisition of chemical data and includes the relevant aspects of field and Contract Laboratory procedures (sample preparation and extraction procedures, and instrumentation). Analytical quality control requirements, evaluation criteria, acceptance criteria, calibration procedures, preventative maintenance, and corrective actions are discussed in following sections.

B.6.1 Contract Laboratory Analytical Procedures

B.6.1.1 Analytical Methodology

The specific analytical methods for this project are from the following:

- EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846; U.S. EPA Third Edition, Final Update III, December 1996).
- EPA 100-400 - Series Methods for the Determination of Inorganic Substances in Environmental Samples (U.S. EPA/600R-93-100, August, 1999a).
- Prescribed Procedures for Measurement of Radioactivity in Drinking Water (U.S. EPA/600/4-80-032, August, 1980)
- Methods of Soil Analysis (American Society of Agronomy, 1982).
- United States department of Agriculture (USDA), Handbook No. 60, (USDA, 1954)

The analytical methods are briefly described in Attachment 1. All samples will be prepared and analyzed in accordance with this QAPP, the referenced analytical method, and in accordance with the Contract Laboratory's SOPs.

B.6.1.2 Data Reporting Requirements

The following criteria for reporting data will apply for all samples:

- MDLs and sample results will be reported to one decimal place more than the corresponding RL, unless the appropriate number of significant figures for the measurement dictates otherwise.
- All target compound non-detections will be reported (at a minimum) as less than the RL.
- If target analytes are detected at or above the RL, they will be reported as quantified.

Additional Reporting Requirements for Definitive Data. The Project Manager will be notified immediately regarding the failure of sample data to meet the RL to assess potential corrective action. The decision to implement corrective action will be based on whether there are any analytical alternatives or clean up steps that would improve the reporting limit and whether the elevated reporting limits will adversely affect data use. Any data that do not meet the MDLs or RLs due to sample dilution will be included in the case narrative and the supporting documentation (chromatograms) will be included in the data packages.

B.7.0 INTERNAL QUALITY CONTROL CHECKS

Internal quality control checks are used to evaluate whether field measurements and sampling procedures and laboratory analytical method performance is within acceptable limits of precision and accuracy. The following sections describe the internal QC that will be followed for both field and Contract Laboratory activities.

B.7.1 Sample Collection

The accuracy and precision of the field sampling procedures will be assessed as described in Section B3.0 of this QAPP. Sample representativeness will be assessed by the analysis of field replicate samples.

B.7.2 Contract Laboratory Analysis

The general objectives of the internal Contract Laboratory QC program are to:

- Ensure that all procedures are documented, including any changes in administrative and/or technical procedures.
- Ensure that all analytical procedures are validated and conducted according to method guidelines and laboratory SOPs.
- Monitor the performance of the laboratory using a systematic inspection program.
- Ensure that all data are properly reported and archived.

The Contract Laboratory will conduct internal quality control checks for analytical methods in accordance with their SOPs, the individual method requirements, and this QAPP. The Contract Laboratory will notify the Project Manager in writing before making significant changes resulting from corrective actions to this QAPP or analytical methodology. The SENES Project Manager and the RAML Project Managers will be notified if the data impacts the task specific objectives.

Contract Laboratory quality control consists of two distinct components, a laboratory component and a matrix component. The laboratory component measures the performance of the laboratory analytical process during sample analyses, while the matrix component measures the effects of a specific media on the method performance. The QC samples that will be used to assess the laboratory component and the media component of analysis are described Section B3.0 of this QAPP. The criteria against which the QC data will be evaluated are listed in Attachment 2. Corrective actions for instrument calibrations or QC sample data out of compliance are listed in the corrective action summary tables included in Attachment 2.

B.8.0 DATA REDUCTION, REVIEW, REPORTING, VERIFICATION, VALIDATION, AND RECORD-KEEPING

The data reduction, review, reporting, verification, and validation procedures are described in this section to ensure that; (1) complete documentation is maintained, (2) transcription and data reduction errors are minimized, (3) the data are reviewed and documented, and (4) the reported results are qualified if necessary. Laboratory data reduction and verification procedures are required to ensure the overall objectives of analysis and reporting meet method and project specifications.

B.8.1 Data Reduction

B.8.1.1 Contract Laboratory Data Reduction

The Contract Laboratory will reduce all analytical data (both screening and definitive) in accordance with the analytical methods and the guidance presented in Sections B3.0 of this QAPP. Refer to Section B3.0 of this QAPP for equations that will be used by the Contract Laboratory to assess precision and accuracy, and refer to Section B3.0 and Attachment 2 regarding instrument calibration and target analyte quantitation.

B.8.2 Data Review

B.8.2.1 Contract Laboratory Data Review

Prior to the release of data to SENES, the Contract Laboratory will perform in-house data review under the direction of the Contract Laboratory Project Manager and/or the laboratory QAO and will prepare and retain full analytical and QC documentation. In general, the Contract Laboratory data review will be conducted as described in the following paragraphs.

The bench analyst will conduct the initial data review based on established protocols specified in laboratory SOPs and analytical method and this QAPP. At a minimum, this review will include the following:

- An assessment of sample preparation procedures and documentation for accuracy and completeness.
- An assessment of sample analysis procedures and documentation for accuracy and completeness.
- Assessments of whether the appropriate SOPs were followed.
- Assessment analytical results for accuracy and completeness.
- An assessment of whether QC samples are within established control limits and method blank data are acceptable.

- An assessment of whether documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, out-of-control forms, if required, are complete, holding times are documented, etc.).

The calculations that will be used to evaluate precision and accuracy are defined in Section B3.0 of this QAPP. The acceptance criteria for calibration, precision, and accuracy assessment and the corrective action summaries are provided in Attachment 2.

When an analysis of a QC sample (blank, spike, or similar sample) indicates that the analysis of that batch of samples is not in control, the analyst will immediately bring the matter to the attention of the appropriate designated Contract Laboratory QC staff (QAO, Project Manager, Section Leader, etc.). This individual will determine whether the analysis can proceed, or if selected samples should be rerun, or specific corrective action needs to be taken before analyzing additional samples. Out-of-control analyses and information justifying accuracy or precision outside acceptance criteria will be documented. A Nonconformance Report will be prepared for all Contract Laboratory analysis out of control events that require documentation. The SENES Project Manager will be notified as soon as feasibly possible to determine the appropriate corrective action for out-of-control events resulting in unacceptable data.

After this review is complete, the analyst will sign the applicable control documentation associated with the analytical batch and forward to the appropriate reviewer. This reviewer (department manager, QAO, etc.) will be responsible for review and approval of the analytical control documentation associated with each analytical batch, as well as any corrective action explanations provided by the analyst. This individual will also be responsible for determining whether the analytical data meet quality control criteria established by the analytical methods and by this QAPP and for identifying QC problems that require further resolution. A permanent record of any corrective actions will be maintained in the Contract Laboratory files.

The Contract Laboratory Project Manager will provide the final review and approval of the analytical data that have been approved by the analyst and other designated reviewer. The Contract Laboratory Project Manager will also be responsible for reviewing all final data reports for proper format and reporting consistency prior to release of the reports to the SENES. This review will include the following as a minimum:

- Contract Laboratory name and address.
- Sample information (includes unique sample identification, sample collection date and time, date of sample receipt, and date(s) of sample preparation and analysis).
- Analytical results reported with an appropriate number of significant figures.
- Reporting limits reflecting dilutions, interferences, and corrections for dry weight as applicable.

- Method references.
- Appropriate QC results and correlations for sample batch traceability and documentation.
- Data qualifiers with appropriate references and narrative on the quality of results. Confirmation that QAPP requirements have been met.

The Contract Laboratory Project Manager and/or QAO will also be responsible for qualifying any data that may be unreliable. Data qualifications will be based on the analytical method, and this QAPP.

B.8.3 Data Reporting

B.8.3.1 Contract Laboratory Data

The Contract Laboratory will provide an electronic deliverable report in a format as specified by SENES. The Contract Laboratory will provide the electronic deliverable via electronic mail or compact disk.

B.8.4 Data Management

The individuals responsible for data management for this project include all personnel responsible for identifying, reporting, and documenting activities affecting data quality. In general, the qualifications of the individuals associated with data management activities will be commensurate with the level of expertise necessary to ensure the intended level of evaluation.

All project files will provide a traceable record for all data management activities. The Contract Laboratory will maintain a project file that includes but is not limited to the following; formulas used for data reduction, computer programs, which data transfers are electronic or manual, data review protocol, raw data files, etc. All data acquired electronically will be transferred and manipulated electronically to reduce errors inherent in manual data manipulation. Data entered, transferred or calculated by hand will be spot checked for accuracy by someone who did not perform the original entries or calculations.

The Contract Laboratory will preserve all electronic and hardcopy records sufficient to recreate each analytical event conducted pursuant to this project. The minimum records the Contract Laboratory will keep include the following:

- C-O-C forms.
- Initial and continuing calibration records including standards preparation traceable to the original material and lot number.
- Instrument tuning records (as applicable).

- Method blank results
- Spike and spike duplicate records and results
- Laboratory records.
- Raw data, including instrument printouts.
- Bench work sheets, and/or chromatograms with compound identification and quantification reports.
- Corrective action reports.
- Other method and project required QC samples and results.
- Laboratory-specific written SOPs for each analytical method.
- QA/QC function in place at the time of analysis of project samples.

Computer acquired data will also be stored on magnetic tape, disks, or other media, that can be accessed using industry-standard hardware and software for data processing, retrieval, or reporting. The laboratory will maintain all data collected for this project sampling for a minimum of seven years following submission of the data reports.

B.9.0 PERFORMANCE AND SYSTEM AUDITS

Technical systems and performance audits will be performed as independent assessments of sample collection and analysis procedures. Audit results will be used to evaluate the ability of the Contract Laboratory to:

- (1) produce data that fulfill the objectives established for this project,
- (2) comply with the QC criteria presented in this QAPP, and
- (3) identify any areas requiring corrective action.

The systems audit is a qualitative review of the overall sampling or measurement system, while the performance audit is a quantitative assessment of a measurement system, and includes both internal and external audits. SENES personnel will conduct internal audits. External audits are the responsibility of federal and state regulatory agencies. Definitive data verification and validation is also a quantitative check of the analytical process, where documentation and calculations are evaluated and verified.

B.9.1 Laboratory Performance and Systems Audits

In-house and regulatory agency audits of laboratory systems and performance will be a regular part of the laboratory's QA program. Internal audits will be conducted by the laboratory's QAO or designee, and consist of a review of the entire laboratory system and at a minimum include: examination of sample receiving, log-in, storage, and chain-of-custody documentation

procedures; sample preparation and analysis; and instrumentation procedures.

An internal audit of the laboratory may be performed by SENES, at the discretion of the RAML Representative, within six months of field investigation start up and will include a review of the following items:

- Sample custody procedures.
- Calibration procedures and documentation.
- Completeness of data forms, notebooks, and other reporting requirements.
- Data review and verification procedures.
- Data storage, filing, and record keeping procedures.
- QC procedures, tolerances, and documentation
- Operating conditions of facilities and equipment
- Documentation of training and maintenance activities.
- Systems and operations overview.
- Security of laboratory automated systems.

Electronic audits involve the examination of the electronic media used by the Contract Laboratory to collect, analyze, report, and store data. These audits are used to assess the authenticity of the data generated, and assess the implementation of good automated laboratory practices. The SENES Project Manager may perform electronic audits of the Contract Laboratory if warranted by on-site audit results.

SENES will forward audit results to appropriate management and the RAML Representative. Deficiencies and corrective action procedures will be clearly documented in the audit report.

External field audits are the responsibility of the federal and state regulatory agencies. Field audits will be conducted at any time during the field operations and will be based upon the information presented in the Work Plan and this QAPP. The audits may or may not be announced, at the discretion of the auditing agency.

B.10.0 PREVENTIVE MAINTENANCE PROCEDURES

A preventive maintenance program will be in place to promote the timely and effective completion of a measurement effort. The preventive maintenance program is designed to minimize the downtime of crucial sampling and/or analytical equipment due to unexpected component failure. In implementing this program, efforts will be focused in three primary areas: (1) establishment of maintenance responsibilities, (2) establishment of maintenance schedules for major and/or critical instrumentation and apparatus, and (3) establishment of an adequate inventory of critical spare parts and equipment.

B.10.1 Contract Laboratory Equipment

Preventive maintenance of all laboratory equipment and instruments is essential to ensure the quality of the analytical data produced. The objective of preventive maintenance is to ensure instrument operation is appropriate for both task-specific and method objectives. The Contract Laboratory has a routine preventive maintenance program to minimize the occurrence of instrument failure and other system malfunctions and will have designated individuals who perform routine scheduled maintenance for each instrument system and required support activity. The following paragraphs focus on maintenance responsibilities, maintenance schedules, record keeping, and inventory of spare parts and equipment.

Maintenance Responsibilities. Maintenance responsibilities for Contract Laboratory equipment will be assigned to designated personnel. These individuals establish maintenance procedures and schedules for each major equipment item. The instrument manufacturer service engineers will perform instrument maintenance and repair, as scheduled/needed. The analysts will perform other routine preventive maintenance tasks. Only qualified individuals will perform any maintenance activities.

Maintenance Schedules. Maintenance schedules are based on the manufacturers' recommendations and/or sample load. Maintenance activities for each instrument will be documented in a maintenance logbook, as described below.

Record Keeping. All instrument maintenance will be documented in instrument-specific bound logbooks, which are kept with the instrument. The date, initials of the individual performing the maintenance and the type of maintenance will be recorded in this logbook. Receipts from routine maintenance performed by the manufacturer's representative will be filed in the appropriate laboratory department (e.g., ion chromatograph maintenance receipts are stored in the organic section). This logbook will serve as a permanent record that documents any routine preventive maintenance performed, as well as any service performed by external individuals such as manufacturers' service representatives. In addition, all receipts from routine maintenance performed by manufacturers' representatives will be maintained in the laboratory's file. These records will be made available upon request during external audits.

Spare Parts. An adequate inventory of spare parts is maintained to minimize equipment down time. This inventory will include those parts (and supplies) which are subject to frequent failure, have limited useful lifetimes, or cannot be obtained in a timely manner.

Contingency Plan. In the event of instrument failure, every effort will be made to analyze samples by an equivalent alternate means within holding times. If the redundancy in equivalent instrumentation is insufficient to handle the affected samples, SENES will be immediately notified and the corrective action to be taken will be determined by the SENES Project Manager and RAML Project Manager (as applicable).

B.11.0 CORRECTIVE ACTIONS

B.11.1 Corrective Action Requirements

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out of control performance that may affect data quality. All proposed and implemented corrective action will be documented in the regular quality assurance reports to the appropriate project management as defined in Section 2.0 of this QAPP. The SENES Project Manager or designee will implement corrective action only after approval. If immediate corrective action is required, approvals secured by telephone from the RAML Project Manager will be documented in an additional memorandum.

For each incidence of noncompliance, a formal corrective action program will be established and implemented at the time the problem is identified. The individual who identifies the problem will be responsible for notifying the SENES Project Manager, who in turn will notify other applicable personnel. Implementation of corrective action will be confirmed in writing as described previously.

Any nonconformance with the established QC procedures specified in the Work Plan or this QAPP will be identified and corrected in accordance with the QAPP. Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings through the proper channels.

B.11.1.1 Contract Laboratory Corrective Action

Corrective actions are required whenever unreliable analytical results prevent the quality control criteria from being met, as specified by the analytical method; the Contract Laboratory's SOPs, or this QAPP. The corrective action taken depends on the analysis and the nonconformance. A summary of corrective actions that will be undertaken for problems associated with specific laboratory analyses is provided in Attachment 2 of this QAPP.

Corrective action will be undertaken if one of the following occurs:

- Blanks consistently contain target analytes above acceptance levels.
- Undesirable trends are detected in spike recoveries, spike recoveries are outside the QC limits, or RPDs between duplicate analyses are consistently outside QC limits.
- There are unusual changes in RLs.
- Deficiencies are detected during QA audits.
- Inquiries concerning data quality are received from the SENES Project Manager.

The analyst who reviews the sample preparation or extraction procedures, and performs the instrument calibration and analysis will handle corrective actions at the bench level (primarily). If the problem persists or its cause cannot be identified, the matter will be referred to the department supervisor or QA department for further investigation. Once resolved, full documentation of the corrective action procedure will be filed with the appropriate Contract Laboratory QA department. A summary of the corrective actions will be included in the data reports.

B.11.1.2 Data Verification Corrective Actions

Corrective action may be initiated during data verification or data assessment. Potential types of corrective action include resampling by the field team or reanalysis of samples by the Contract Laboratory.

Corrective actions that will be taken are dependent upon the ability to mobilize the field team, how critical the data are to the task-specific objectives, and whether the samples are still within holding time criteria. When a corrective action situation is identified by the SENES Health Physicist, the SENES Project Manager will have responsibility for authorizing the implementation of the corrective action, including resampling and documenting the corrective action and notifying the RAML Project Manager for authorization.

B.11.2 Corrective Action System

A system for issuing, tracking, and documenting completion of formal Recommendations for Corrective Action (RCA) exists for addressing significant and systematic problems. Recommendations for corrective actions are issued only by a member of the QA group, or a designee in a specific QA role. Each RCA addresses a specific problem or deficiency, usually identified during QA audits of Contract Laboratory or project operations. An RCA requires a written response from the party to whom the RCA was issued. A summary of unresolved RCAs is included in the monthly QA report to management. The report lists all RCAs that have been issued, the manager responsible for the work area, and the current status of each RCA. An RCA requires verification by the QA group that the corrective action has been implemented before the RCA is considered to be resolved. In the event there is no response to an RCA within 30 days, or if the proposed corrective action is disputed, the recommendation and/or conflict is pursued to successively higher management levels until the issue is resolved.

B.12.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Deliverables associated with this project will contain separate QA sections in which data quality information collected during specific tasks is summarized. Deliverables include reports that summarize the sampling program findings. Submission of these reports is the responsibility of the SENES Project Manager. Quality assurance sections will identify all QA samples collected and the corresponding primary samples and will report accuracy, precision, and completeness of the data as well as the results of the performance and system audits, and any corrective action needed or taken during the project.

B.13.0 DATA MANAGEMENT

Data management will be achieved using a standard relational database format. Database fields will encompass standard sample and analytical information, including:

- Sample identifications;
- Matrices;
- Analytical methods;
- Dates & times;
- Chain-of-custody information;
- Analytical results;
- Detection limits and reporting limits;
- Quality control results;
- Coordinate information.

Horizontal coordinate information will be referenced to the State Plane Coordinate System, New Mexico West, North American Datum of 1983. Vertical coordinates will be referenced to the North American Vertical Datum of 1988.

The database will serve as a central repository for data from many different project tasks. It is one foundation for making project decisions. Making sure the data are technically accurate, complete and correctly represented in the database is referred to as "data integrity." Project staff will assume that data within the database are correct and ready to use in analyses, reports, graphics, geographic information system (GIS), modeling and for other purposes. Therefore, the Database Manager will ensure that the following tasks have been applied to all data in the database:

- Data will be received from the laboratory using an electronic data deliverable (EDD) format compatible with the project database format;
- Data will be assembled and reviewed by the person compiling the data for completeness and technical accuracy;

- Data will have been validated using procedures presented in the QAPP; no draft or preliminary (i.e., unvalidated and unqualified) data will be put into the master database;
- Data will be transcribed accurately from any hard copies during data entry (100% error free transcription); and
- Data are converted and imported accurately from any electronic files (spreadsheets, ASCII files, and HDDs).

The Database Manager will also ensure that all data products (report summary tables, appendices, programs and files exported to other applications) represent the data in the database accurately.

B.14.0 ASSESSMENT AND OVERSIGHT

Program assessment and oversight will be performed by the Project Manager and/or designee and will include assessments and response actions, reports to management, as well as nonconformance and corrective action training. All personnel are responsible for ensuring that the program is implemented in accordance with this Work Plan and applicable professional standards. All personnel are also expected to stop and take appropriate action when it is determined that conditions adversely affecting the quality of the data have occurred (e.g., an instrument is not working properly). Work may be stopped to determine what further action is needed to meet the quality objectives of this study.

B.14.1 Assessments and Response Actions

Program assessment and oversight will include surveillance/audit of field sampling activities, the analytical program, and program records. Surveillance of sampling activities will focus on adherence to procedures outlined in this Work Plan and will include observation of sampling procedures and selected documentation (e.g., field logbooks).

Review of program records will include both sampling and laboratory records. Review of the laboratory data will serve as verification that the quality program as described in this Work Plan and the laboratory QAPP is being implemented, thus allowing for the collection of data that support the objectives.

B.14.2 Nonconformance and Corrective Action

All of the individuals involved in this program will follow a formalized process for documenting non-conformances. The nonconformance process consists of the following:

- Identification of the nonconformance;

- Determination of the immediate actions to be taken as a result of the nonconformance;
- Root cause analysis and identification of real root cause(s);
- Proposed action to prevent recurrence of the nonconformance and implementation of the correction; and
- Follow-up and verification of the effectiveness of the corrective action.

Any deviations from the specifications described in this Work Plan, field sampling protocols, held measurement SOPs, or laboratory quality system will be documented and addressed. A signed corrective action or field change request (see Appendix B) form will be submitted to the EPA for their approval prior to proceeding with the affected task. A prompt response from the EPA will be required to prevent delays in the execution of field activities. The form(s) will be forwarded to the RAML Project Manager and SENES Project Manager.

B.14.3 Data Validation and Usability

Data verification is used to ensure that the requirements stated in the planning documents are implemented as prescribed. Data validation is used to ensure that the results of the data collection activities support the objectives of the survey as documented in the QAPP, or permit a determination that these objectives should be modified. Data quality assessment is the scientific and statistical evaluation of data to determine if the data are of the right type, quality, and quantity to support their intended.

This plan specifies the QC checks that are to be performed during sample collection, handling, and analysis. These include calibration and analyses of check standards, blanks, spikes, and replicates, which provide indications of the quality of data being produced by specific steps of the measurement process. Data validation should document any corrective actions that were taken, which samples were affected, and the potential effect of the actions on the validity of the data. When issues are identified in the verification and validation process, the validator will make appropriate comments and/or assign data flags to alert the data user to potential limitations on the usability of the data.

B.14.4 Reconciliation with User Requirements

Data collected during the field activities will be reconciled with the requirements of the data user. There are five steps in the DQA Process:

1. Review the objectives and survey designs;
2. Conduct a preliminary data review;
3. Select the statistical test;
4. Verify the assumptions of the statistical test;
5. Draw conclusions from the data.

These five steps are presented in a linear sequence, but the DQA process is applied in an iterative fashion much like the DQO process. The strength of the DQA process is that it is designed to promote an understanding of how well the data will meet their intended use by progressing in a logical and efficient manner.

B.15.0 REFERENCES

American Society of Agronomy, 1982, Methods of Soil Analysis.

U.S. Environmental Protection Agency, 1986. EPA Test Methods for Evaluating Solid Waste. Physical/Chemical Methods (SW-846; U.S. EPA Third Edition, Final Update III, December 1996).

U.S. Environmental Protection Agency, 1999. EPA 100-400 - Series Methods for the Determination of Inorganic Substances in Environmental Samples. EPA/600R-93-100.

U.S. Environmental Protection Agency, 1980. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA/600/4-80-032.

U.S. Environmental Protection Agency, 2000. Guidance for the Data Quality Objectives Process, EPA QA/G-4. EPA/600/R-96/055.

U.S. Environmental Protection Agency, 2001. EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5.

United States department of Agriculture (USDA), 1954. Handbook No. 60.

ATTACHMENT 1 TO APPENDIX B

Analytical Procedures

Appendix B, Attachment 1

**Table B.1
Quality Control Procedures
Radionuclide and Total Metals Analyses**

Analyte	Analytical Method	Sample Container	Preservation	Holding Time	Unit of Measure	Reporting Limit	Method/ Analytical Procedure
Ra-226	EPA 901.1	Gallon ziploc bag	None	180 days	pCi/g	0.5	A homogeneous aliquot of sample is put into a standard geometry for gamma counting, and set aside for 21 day in-growth period. Samples are counted long enough to meet the required sensitivity of measurement.
Uranium	SW-846 6020A	1-8-oz glass wide-mouth jar with Teflon-lined cap	None	180 days	mg/kg	0.15	Metals in solution are analyzed using an ICP/Mass Spectrometer.
Th-230	ASTM 3972-90M	Gallon ziploc bag or 1-8 oz glass wide-mouth jar with Teflon-lined cap	None	180 days	pCi/g	0.1	A homogeneous aliquot of sample is put into a standard geometry for gamma counting. Samples are counted long enough to meet the sensitivity of measurement.
Stable Metals Arsenic	SW-846 6010	1-8-oz glass wide-mouth jar with Teflon-lined cap	None	180 days	ppb	1000.0	Metals in solution are analyzed using an ICP/Mass Spectrometer.
Stable Metals Molybdenum	SW-846 6010	1-8-oz glass wide-mouth jar with Teflon-lined cap	None	180 days	ppb	0.5	Metals in solution are analyzed using an ICP/Mass Spectrometer.
Stable Metals Selenium	SW-846 6010	1-8-oz glass wide-mouth jar with Teflon-lined cap	None	180 days	ppb	500.0	Metals in solution are analyzed using an ICP/Mass Spectrometer.

Church Rock 1 and 1E Removal Site Evaluation Phase II Work Plan

Analyte	Analytical Method	Sample Container	Preservation	Holding Time	Unit of Measure	Reporting Limit	Method/ Analytical Procedure
Stable Metals Vanadium	SW-846 6010	1-8-oz glass wide-mouth jar with Teflon-lined cap	None	180 days	ppb	1000.0	Metals in solution are analyzed using an ICP/Mass Spectrometer.
Volatile Organic Compounds	SW-846 8260B	2, 40 mL amber glass bottles with Teflon septum cap and no head space	HCl; pH < 2 Chill to 4°C	14 days	ppb	5.0	Volatile compounds are introduced onto a 30-meter capillary column in a gas chromatograph (GC), temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced with the GC. Quantification is accomplished by comparing the response of a major ion relative to an internal standard using a 5-point calibration curve.
Semi-Volatile Organic Compounds	SW-846 8270C	1 L amber glass bottle with a Teflon Cap	Chill to 4°C	7 days collection to extraction, 40 days extraction to analysis	ppb	333.3333	Semi-volatile compounds are introduced onto a 30-meter capillary column in a gas chromatograph (GC), temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced with the GC. Quantification is accomplished by comparing the response of a major ion relative to an internal standard using a 5-point calibration curve.
Petroleum Hydrocarbons	SW-846 8015M	1-4oz. glass jar-Teflon lined cap	Chill to 4°C	14 days	ppb	500	Determines the concentrations of various nonhalogenated volatile organic compounds and semivolatile organic compounds by gas chromatography.

References:

EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), (U.S. EPA Third Edition, September 1986; Final Update III, December 1996).
EPA Methods for the Determination of Inorganic Substances in Environmental Samples (EPA 100-400 Series) (EPA/600R-93/100, August 1993).

Abbreviations:

SW = Solid Waste
EPA = Environmental Protection Agency
pCi/g = picocuries/gram
mg/kg = milligrams per kilogram
ICP = inductively coupled plasma
ppb= parts per billion

Table B.2
Quality Control Procedures
Agronomic Sampling

Agronomic Analyses	Analytical Method	Method/ Analytical Procedure
pH	ASA No.9 Method 10-3.2	A saturated paste is made by mixing the soil with water in a 1:1 ratio. pH is measured with a calibrated pH probe
Electrical Conductivity	ASA No. 9 Method 10-3.3	A saturated paste is made by mixing the soil with water in a 1:1 ratio. Electrical conductivity is measured using a calibrated conductivity meter.
Saturation Percentage	USDA Handbook 60, Method 27A	A portion of the saturated pastes is collected and dried at 105°C. The loss of water weight divided by the dry weight of the soil is expressed in percent
Texture	ASA No. 9, Method 15-5	Texture is determined by mixing a weighted portion of the sample with enough water to bring the volume to 1L. After mixing density is measured using a hydrometer at 7 timed intervals as the sample settles.
Rock Fragment Percentage	ASA No. 9, Method 15-5	A weighed amount of sample is sent through a series of sieves and percentage is determined by weighting the amount of samples left on each sieve.
Sodium Adsorption Ratio (SAR)	ASA No. 9, Method 10-3.4 / SW6010B	A saturated paste is made by mixing the soil with water in a 1:1 ratio. The liquid portion is then analyzed for potassium using ICP.
Nitrate	ASA No. 9, Method 33-3.1 / EPA 353.2	Nitrate is extracted from soil using a 2M potassium chloride solution. Extract is then analyzed for nitrate by colorimetry
Phosphorus	ASA No. 9, Method 24-5.1 / EPA 365.1	Phosphorus is extracted from soil using a solution consisting of 0.03 N ammonium fluoride and 0.025 N hydrochloric acid. The extract is analyzed for phosphorus by colorimetry.
Potassium	ASA No. 9, Method 13-3.5 / SW6010B	A saturated paste is made by mixing soil with water in a 1:1 ratio. The liquid portion is then analyzed for potassium using ICP
Chloride	ASA No. 9, Method 10-2.3.2 / EPA300	Chloride is extracted from soil using distilled water. Extract is analyzed for chloride by ion chromatography.
Sulfate	ASA No. 9, Method 28-5.1	Sulfate is extracted from soil using distilled water. Extract is analyzed for sulfate by ion chromatography.
Organic Carbon	ASA No. 9, Method 29.3.5.2	Walkley-Black was developed specifically for soils and consists of a wet oxidation method using potassium dichromate, which is back-titrated with iron ⁺² This method targets organic matter in soil which is the primary source of organic carbon in soil.

ATTACHMENT 2 TO APPENDIX B

Laboratory Quality Assurance Plan (LQAP)

ALS Laboratory Group, Environmental Division



Laboratory Quality Assurance Plan (LQAP)

ALS Laboratory Group, Environmental Division
– Fort Collins, CO

Laboratory Quality Assurance Plan (LQAP)

Revision 13

March 18, 2010

ALS Laboratory Group, Environmental Division

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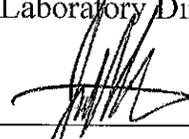
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Summary of 2010 LQAP Changes

- All additions and deletions are incorporated through tracked changes in accordance with ALS SOP 926r10.
- New text is noted in blue and deleted text is red strikethrough.
- Change sections are also designated with a bar in the left margin.

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1. INTRODUCTION

This Laboratory Quality Assurance Plan (LQAP) describes the policies, procedures and accountabilities established by the Environmental Laboratory of ALS Laboratory Group, Environmental Division (Ft Collins, CO) (ALSLG-FC) to ensure that the environmental test results reported from the analysis of air, water, soil, waste, and other matrices are reliable and of known and documented quality. This document describes the quality assurance and quality control procedures followed to generate reliable analytical data.

This LQAP is designed to be an overview of ALS operations. Detailed methodologies and practices are written in ALS Standard Operating Procedures (SOPs). Where appropriate, ALS SOPs are referenced in this document to direct the reader to more complete information. A list of current SOPs is found in Appendix H.

ALS maintains certifications pertaining to various commercial and government entities; these are listed in Appendix I. Each certification requires that the laboratory continue to perform at levels specified by the programs issuing certification. Program requirements can be rigorous; they include semiannual performance evaluations as well as annual audits of the laboratory to verify compliance.

The State of Utah has primacy in administering certification of this laboratory to perform EPA methods. Thus, the Utah State Health Department certifies ALS to perform EPA methods under Utah Rule R444-14. For that reason, reference is made to Utah Rule R444-14 in this QAPP.

ALSLG-FC is a full service environmental and radiochemistry laboratory, performing analyses for organic, inorganic, and radiological constituents in a variety of matrices. ALSLG-FC specializes in serving the Department of Energy (DOE), Department of Defense (DoD), and architect-engineering firms. ALSLG-FC routinely provides hardcopy data packages and electronic data deliverables that are easily validated by external validators.

The management team at ALS Laboratory Group, Fort Collins applies an integrated approach to quality assurance, client service, and efficient operations, that enables ALSLG-FC to produce compliant data that meet or exceed all technical and service requirements as prescribed by our clients. This Laboratory Quality Assurance Plan (LQAP) defines ALSLG-FC's quality assurance (QA) program, and communicates ALSLG-FC's goals, values and policies regarding quality, ethical conduct, data integrity, and optimized operations.

1.1 MISSION STATEMENT

To provide analytical services to help our customers make informed decisions.

1.2 VISION STATEMENT

To be recognized as a global market leader.

1.3 QUALITY POLICY

ALS is committed to producing legally defensible analytical data of known and documented quality acceptable for its intended use and in compliance with the Safe Drinking Water Act, the Clean Water Act, and the Resource Conservation and Recovery Act. This QAPP is designed to satisfy the applicable requirements of the State of Utah and other state certification programs. ALS complies with the National Environmental Laboratory Accreditation Conference (NELAC) standards.

ALS corporate management has committed its full support to provide the personnel, facilities, equipment, and procedures required by this QAPP.

ALS management is committed to improvements of the management systems through compliance with NELAC 2003 and ISO 17025:2005 ALS management is also committed to compliance with project related requirements including DOECAP QSAS and DoD QSM 4.1 Gray Boxes.

ALS management reviews its operations on an ongoing basis and seeks input from staff and clients to make improvements. See section 12.1.5 of this plan for details.

It is the policy of ALS that all employees shall be familiar with all Quality documentation.

Within this framework, ALSLG-FC performs analyses in strict accordance with promulgated methodologies, including:

- USEPA, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods;
- USEPA, Methods for Chemical Analysis of Waters and Wastes (MCAWW);
- USEPA, Methods for Determination of Metals in Environmental Samples;
- American Public Health Association (APHA), Standard Methods for the Examination of Water and Wastewater (SM);
- USEPA, Methods for Determination of Organic Compounds in Drinking Water;
- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Volume 11 – Water and Environmental Technology;

- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Volume 12 – Nuclear Energy;
- USDOE, Environmental Measurements Laboratory (EML), Procedures Manual (HASL-300);
- USEPA, Eastern Environmental Radiation Facility (EERF), Radiochemistry Procedures Manual;
- USDOE, Radiological and Environmental Sciences (RESL), Procedures Manual;
- USEPA, Prescribed Procedures for Measurement of Radioactivity in Drinking Water; and
- US, Code of Federal Regulations (40 CFR).

1.4 STATEMENT ON WASTE, ABUSE AND FRAUD

ALSLG-FC is committed to achieving our goals in the most efficient and effective manner possible, thus avoiding wasteful use of resources. This is accomplished by assuring the proper utilization of ALSLG-FC's purchased materials and equipment, and time and ability of our personnel. *Any ALS Laboratory Group, Fort Collins employee who has any suggestion or concern regarding ALSLG-FC's practices, is encouraged to discuss his/her idea or question with their Department Manager, the Quality Assurance Manager, and/or the Laboratory Director.* A means of confidentially reporting concerns anonymously is also available. Grievances and allegations of unethical conduct will be fully investigated, and appropriate actions taken.

Training regarding ALSLG-FC's Waste, Abuse and Fraud policies is provided to every new staff member, and to all employees lab-wide as an annual refresher. ALSLG-FC's policies regarding waste, abuse and fraud are included in **Appendix A**.

1.5 CODE OF ETHICS AND DATA INTEGRITY STATEMENTS

ALS Laboratory Group, Fort Collins is responsible for creating a work environment that enables all employees to perform their duties in an ethical manner. *It is ALSLG-FC's expectation that all employees exhibit professionalism and respect for clients and each other in all interactions and tasks.* ALSLG-FC requires that each employee abide by the following guidelines:

- Every ALSLG-FC employee is responsible for the propriety and consequences of his or her actions. Each employee shall conduct him or herself in a professional manner towards all clients, regulators, auditors, vendors, and other employees. Professional conduct relates to honesty, integrity, respect, and tolerance for cultural diversity.

- Every ALSLG-FC employee shall perform all assigned duties in accordance with ALSLG-FC's established quality assurance policies and quality control procedures that have been developed to ensure conformance with contractual and regulatory requirements.
- ALS Laboratory Group, Fort Collins expects all employees to use professional judgment and to document all situations thoroughly. It is the responsibility of each ALSLG-FC employee to consult the Department Manager or Quality Assurance Manager when atypical or unusual situations occur and to disclose and document the decision-making process. Every employee must disclose any instance of noncompliance. ALSLG-FC reports all noncompliance issues affecting data to the client.
- It is the responsibility of each ALSLG-FC employee to report any suspicion of unethical conduct to the Quality Assurance Manager or the Laboratory Director.

Data integrity procedures provide assurance that a highly ethical approach to testing is a key component of all laboratory planning, training and implementation of methods. The following list provides examples of improper, unethical, or illegal practices that ALS Laboratory Group, Fort Collins ***does not*** tolerate:

- Falsification of records to meet method requirements (e.g., sample records, logbooks, sample results, electronic records). This includes intentional misrepresentation of the date or time of analysis (e.g., intentionally resetting a computer system's or instrument's date and/or time to make it appear that a date/time requirement has been achieved); and unwarranted manipulation of computer software (e.g., improper background subtraction to meet ion abundance criteria for GC/MS tuning compounds).
- Improper use of manual integrations performed to meet calibration or method quality control criteria (e.g., peak shaving or peak enhancement performed solely to meet quality control requirements).
- Selective exclusion of data to meet quality control criteria (e.g., eliminating initial calibration points without technical justification).
- Misrepresentation of quality control samples (e.g., adding surrogates or tracers after sample extraction, omitting preparation steps for quality control samples; over- or under- spiking).
- Reporting results without analyses to support the results (i.e., dry-labbing).

- Notation of matrix interference as basis for exceeding acceptance limits in interference-free matrices.
- Intentional plagiarism or willful misrepresentation of another employee's work as one's own (e.g., Initial or Continuing Demonstration of Capability study (IDOC, CDOC) or Proficiency Testing (PT) study.

Strict adherence to ALSLG-FC's Code of Ethics and Data Integrity is essential to the reputation and continued health of our business. All ALSLG-FC employees are required to acknowledge their responsibility and intent to behave in an ethical manner by attesting to the requirements described above upon joining the ALSLG-FC staff, and annually thereafter. Included in **Appendix A** are the ethics documents that every employee is required to review and attest to.

1.6 REVIEW, REVISION, DISTRIBUTION AND HIERARCHY OF QA DOCUMENTS

Current copies of pertinent quality assurance guidance documents, such as ALSLG-FC's LQAP, the TNI Standards, the NELAC standards, ISO 17025:2005, the US DOE Quality Systems for Analytical Services (QSAS), the US DoD Quality Systems Manual (QSM) and others, are posted to the ALSLG-FC network so that they are accessible to every employee. Laboratory Standard Operating Procedures (SOPs) and other method references are also posted to the network for lab-wide employee access. Project-specific requirements are disseminated to the laboratory via Laboratory Information Management Systems (LIMS) program specifications (discussed further below).

ALS Laboratory Group - Fort Collins recognizes a hierarchy of guidance that provides for comprehensive definition, yet flexible coverage, thus enabling both overall program and site-specific needs to be met. An overview explaining this hierarchy is given below. **SOP 926** provides detailed guidance on the review, revision, and distribution of laboratory-generated controlled documents.

1.6.1 LABORATORY QUALITY ASSURANCE PLAN

The LQAP is an encompassing controlled-document that describes ALSLG-FC's quality assurance program and policies. All systems, policies, and procedures have been developed and implemented in accordance with applicable USEPA requirements, regulations, and guidance; the current NELAC standards; and requirements set forth in various client quality assurance documents and contractual specifications. This document has been prepared in accordance with these referenced documents, as well as others, cited in the attached **Bibliography**. The LQAP is intended to provide a 'quality requirements framework', including quality control (QC) procedures to be followed in the absence of project-specific requirements (note that project-specific requirements are communicated to laboratory staff via LIMS program specifications, which are discussed subsequently).

The Quality Assurance Manager (QAM) bears primary responsibility for ensuring that the LQAP meets industry standards. Proposed revisions to the LQAP are approved by key laboratory personnel (i.e., Laboratory Director, Quality Assurance Manager, and every Technical or Department Manager). Following approval, the QAM posts the revised LQAP to the ALSLG-FC network, revised to LQAP document in LIMS. The LIMS notifies personnel of all revised documents. It is the requirement of all employee to read and update reading records for all assigned controlled documents. Archival records of all LQAP iterations are maintained by the Quality Assurance Department.

1.6.2 STANDARD OPERATING PROCEDURES

The second kind of controlled-document in the hierarchy of quality assurance guidance are the Standard Operating Procedures (SOPs). An SOP defines the QA/QC requirements for each method and describes in detail how personnel perform procedures and evaluate data. SOPs pertaining to general practices (e.g., standards, temperature monitoring, etc.), administrative procedures (e.g., procurement of supplies and materials, etc.) and health & safety requirements (e.g., calibration and use of the hand and foot monitor), are also maintained by ALSLG-FC. It is ALSLG-FC's intent that the information contained in our SOPs are both method-compliant, and accurately reflect actual practice. *Suggestions for SOP content clarification or revision are encouraged.* SOPS are published to the network when approved.

The LIMS notifies personnel of all revised documents. It is the requirement of all employees to read and update reading records for all assigned controlled documents

This process of revision, approval and distribution is established in the ALSLG-FC SOP 926. A list of current SOPs is provided in **Appendix H**. The Quality Assurance Department manages the review, revision and controlled distribution of documents and maintains associated records.

1.6.3 LABORATORY MANAGEMENT INFORMATION SYSTEMS (LIMS) PROGRAM SPECIFICATION

The last and most specific controlled-document in this hierarchy is the LIMS program specification. The LIMS program specification is a distillation of client Quality Assurance Project Plan (QAPjP) or contractual requirements, prepared electronically by the ALSLG-FC Project Manager (PM), in collaboration with the QAM and applicable Department Managers. This custom program specification, along with the associated LIMS test code nicknames, contain directives and controls that govern testing and reporting data. The program specification is often limited in scope and addresses only those QA/QC

criteria required for a specific project. *When the client's requirements differ from those stated in the SOPs and/or LQAP, the project-specific LIMS program specification requirements supersede the others. It is the responsibility of all personnel who work with samples or data to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of the samples or data.*

2. LABORATORY ORGANIZATION AND RESPONSIBILITIES

This section provides an overview of ALS Laboratory Group, Fort Collins organization and defines key personnel, their responsibilities, and the lines of communication between these employees. An organization chart that illustrates reporting relationships is provided in **Appendix B**.

ALS policy is to perform work for clients in the most efficient manner possible, avoiding waste of resources and undue pressure on employees. It is the role of both ALS management and employees to ensure that work for clients is performed most efficiently and effectively by properly utilizing ALS purchased materials, equipment, and the time and ability of personnel.

2.1 GENERAL REQUIREMENTS FOR LABORATORY PERSONNEL

ALSLG-FC maintains sufficient personnel to perform analytical services for our clients. Each employee must have a combination of experience and education that enables him or her to demonstrate a specific knowledge of his or her job function, and a general knowledge of laboratory operations, test methods, QA/QC procedures, and records management. *All personnel are responsible for complying with the requirements that pertain to his/her assigned duties.*

2.2 KEY PERSONNEL

Education, experience and skill requirements for these positions are addressed in job descriptions. Functional responsibilities are further discussed below.

In the event of a temporary absence, key personnel must notify other key staff of their absence and reassign their duties to another employee who is qualified to perform the assigned duties. For example, a PM may assign another PM to cover his or her duties; a Department Manager may assign a senior chemist to cover his or her duties within the Department; and the Laboratory Director may assign a Manager to cover his or her duties.

2.2.1 LABORATORY DIRECTOR

The Laboratory Director (and/or designee) is responsible for:

- All laboratory operations, including: business functions such as marketing, sales and financial issues; technical functions such as sample control, preparation, analysis, data management; and quality assurance;

- Providing input and support to proposal processes, including interacting with the Sales, Technical and Quality Assurance staff, to ensure that the laboratory is capable of complying with client and regulatory requirements;
- Supervising all personnel through Management staff, who ensure that QA/QC procedures are being performed and that any nonconformances or discrepancies are documented and remedied properly and promptly;
- Ensuring that corrective actions relating to Findings from internal and external audits are completed in a timely fashion;
- Ensuring that the laboratory has the appropriate resources and facilities to perform analytical services;
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory;
- Defining the minimum level of education, experience, and skills necessary for all positions in the laboratory;
- Ensuring that only those vendors and supplies that are of adequate quality are used; and
- Directing the performance of the annual Managerial Review.

2.2.2 QUALITY ASSURANCE MANAGER

The Quality Assurance Manager reports to the Laboratory Director and is independent of daily operation and production requirements.

Therefore, the QAM is able to evaluate data objectively and perform assessments without production influence. *The QAM has authority to stop work if systems are sufficiently out of control to compromise the integrity of the data generated.*

The QAM shall have documented training and/or experience in QA/QC procedures; knowledge of quality systems as defined by NELAC; and a general knowledge of the analytical test methods for which data review is performed.

The QAM (and/or designee) is responsible for:

- Defining and implementing the quality system;
- Developing and maintaining a pro-active program for prevention and detection of improper, unethical, or illegal

practices (e.g., single- or double-blind proficiency testing studies, electronic data audits, maintaining documents that identify appropriate and inappropriate laboratory and data manipulation practices);

- Ensuring continuous improvement of laboratory procedures via training, control charts, proficiency testing studies, internal audits, and external audits;
- Coordinating the laboratory's participation in state and Federal certification programs;
- Scheduling the review and distribution and maintaining distribution records of controlled documents, including plans (e.g., LQAP, etc.) and SOPs;
- Reviewing Requests For Proposal (RFPs) to ensure compliance with required QA/QC practices;
- Facilitating external audits;
- Overseeing or conducting internal audits of the entire operation annually (technical, system, data, electronic);
- Coordinating, preparing and approving external and internal audit responses and corrective actions;
- Managing the laboratory's participation in proficiency testing (PT) studies;
- Reviewing nonconformances and approving corrective actions;
- Reviewing QC limits per established procedures;
- Ensuring that Method Detection Limit (MDL) studies are performed and documented per requirements;
- Managing the reference standards used in the calibration and/or verification of support equipment (e.g., weights, thermometers, balances);
- Revising the LQAP annually in accordance with industry standards;
- Maintaining an archival system for data records; and

- Maintaining technical and quality assurance training records, including employee demonstrations of capability (DOCs).

2.2.3 **HEALTH & SAFETY MANAGER/RADIATION SAFETY OFFICER (RSO)**

The Health & Safety Manager/Radiation Safety Officer (RSO) reports to the Laboratory Director. This Manager is responsible for establishing and monitoring adequate systems, procedures and training to ensure that the laboratory staff, facilities and operational activities conducted, function in a manner that minimizes employee risk of illness and injury, is compliant with all applicable regulations pertaining to matters of safety and health, and that limits the financial liability of the corporation as it relates to these matters. As RSO, this Manager is also responsible for discharging the duties and requirements prescribed by ALSLG-FC's Radioactive Materials License.

Key responsibilities of the Health & Safety Manager/RSO (and/or designee) include:

- Ensuring that all employees have sufficient training to perform their job without unnecessary risk of illness or injury, providing health and safety, including radiation safety, training for new employees, and maintaining health and safety-related training records;
- Providing procedural guidance in the form of the Chemical Hygiene Plan (CHP), Radiation Protection Plan (RPP), Respiratory Protection Plan (ResPP), Emergency and Contingency Plan (ECP) and Health and Safety SOPs, and ensuring that these guidances are reviewed by laboratory staff;
- Ensuring that the laboratory facilities are maintained and operated in a safe manner, including:
 - (a) Performing routine safety inspections of all operational areas;
 - (b) Performing routine radiation surveys and managing the radiation dosimetry program; and
 - (c) Performing personal monitoring, as indicated, for chemical and other exposures.
- Maintaining the laboratory's Colorado Radioactive Materials License and ensuring compliance with the terms of the license. Included in this responsibility are:

- (a) Procuring and managing radioactive sources and standards;
- (b) Maintaining the laboratory's radioactive materials inventory, which also includes directing prescreen analyses that provide initial characterization of potential sample radioactivity;
- (c) Overseeing permitted low level radioactive materials releases to the sanitary sewer; and
- (d) Ensuring that radioactive materials waste are transported in accordance with all Federal and state regulations, and are transferred only to facilities that possess a radioactive materials license.

2.2.4 FACILITIES/WASTE COMPLIANCE MANAGER

The Facilities/Waste Compliance Manager, reports to the Laboratory Director. This Manager is responsible for day-to-day management of the building and serves as the primary point of contact for all matters related to waste collection and disposal.

The Facilities/Waste Compliance Manager (and/or designee) is responsible for:

- Coordinating heating, ventilation, and air-conditioning (HVAC) systems operation and maintenance;
- Maintaining the uninterruptible power supply (UPS) and coordinating maintenance and repairs to the electrical system;
- Maintaining the in-house vacuum system;
- Coordinating repairs to the building (e.g., doors, locks, windows, cabinetry);
- Maintaining the building's security and fire alarm system;
- Interfacing with fire inspectors; and responding to security and fire alarms on a 24-hour basis;
- Implementing waste reduction procedures;
- Managing the accumulation of radioactive waste in the laboratory;

- Developing and maintaining Satellite Accumulation Areas (SAAs) and 90-Day Storage Areas;
- Overseeing all waste disposal operations performed by ALSLG-FC, including (1) ensuring compliance with Federal, state, and local regulations for waste handling and disposal in accordance with RCRA, TSCA, and radioactive waste disposal regulations; (2) managing hazardous waste shipments to Temporary Storage and Disposal Facilities (TSDFs); (3) managing sanitary sewer releases; and (4) managing sample archives and the return of samples and sample residues to clients;
- Training personnel on proper techniques for sample handling and waste disposal, according to standards implemented by Federal, state, and local authorities and maintaining associated training records; and
- Supervising the Sample Receiving Department.

2.2.5 INFORMATION SYSTEMS MANAGER

The Information Systems (IS) Manager reports to the Laboratory Director. This Manager is responsible for administering the network, maintaining data recovery systems, and for managing personal computing (PC) equipment and peripherals, thus supporting instrumentation and LIMS. The IS Manager (and/or designee) is responsible for:

- Managing and maintaining the laboratory computer system. This function includes determining and purchasing appropriate hardware and verifying that its function meets intended objectives, establishing network server structure, and developing and implementing proper maintenance and backup procedures;
- Procuring, configuring and maintaining all printers and copiers;
- Serving as a technical resource on computer-related issues;
- Documenting related operating procedures through SOPs, manuals or other proprietary documentation;
- Supervising recovery of all systems in the event of a disaster;

- Along with the Laboratory Information Systems Manager, analyzing information flow in the laboratory and suggesting the most effective hardware, applications software, and/or programming changes as solutions to meet long-term customer requirements; also, implementing those changes in data acquisition and management by purchasing hardware or software, where software is not developed internally; and
- Maintaining and implementing existing and future communications systems, including all internet and telephone systems.

2.2.6 LABORATORY INFORMATION MANAGEMENT SYSTEMS MANAGER

The Laboratory Information Management Systems (LIMS) Manager reports to the Laboratory Director, and bears the primary responsibility for the LIMS, which serves the needs of the technical, business, and management functions of the laboratory.

Key responsibilities of the LIMS Manager (and/or designee) include:

- Designing and developing information systems that relate to data capture and reporting;
- Maintaining and supporting applications that access LIMS and maintaining and supporting database back-end applications used for LIMS;
- Documenting changes and procedures through SOPs, manuals or other proprietary documentation;
- Developing software, as needed, using the appropriate tools, and per industry standard methodologies and validations;
- Overseeing and assisting with the implementation, testing and verification of upgrades made to instrument software;
- Coordinating all efforts to automate and improve electronic systems and processes throughout the laboratory;
- Developing interfaces necessary to achieve the requirements for client-specified electronic data deliverables (EDDs), and managing all deliverable formats provided to clients (hardcopy, electronic); and

- Providing training, as applicable, for all LIMS-related applications.

2.2.7 PROJECT MANAGER

Project Managers report to the Laboratory Director. *The Project Manager serves as the primary point of contact between clients and ALSLG-FC.* Each PM (and/or designee) is responsible for:

- Managing and coordinating the laboratory's performance after contract award, by defining technical and service requirements for personnel via LIMS, and interacting with clients and laboratory personnel to ensure that technical criteria and client service needs are met, including monitoring holding times (if appropriate) and deliverable deadlines, for all project sample analyses;
- Reviewing and approving any nonconformances reported by the laboratory and notifying the client, if appropriate, and communicating with clients pro-actively to ensure that all client service and technical concerns are resolved promptly;
- Reviewing all final reports for completeness, compliance with project requirements, clerical accuracy, and reasonableness;
- Generating, as directed by prompts provided in ALSLG-FC's proprietary EDD generator, and transmitting EDDs to their clients as required; and
- Communicating to the Laboratory Director any potential need for new or improved capabilities based on clients' feedback.

2.2.8 TECHNICAL OR DEPARTMENT MANAGER

Technical and Department Managers report to the Laboratory Director. These Managers exercise day-to-day supervision of laboratory personnel, procedures, and reporting of results. They maintain technical expertise in their area of specialization (e.g., organics, inorganics, radiochemistry).

Technical Managers and Department Managers (and/or their designee) are responsible for:

- Providing technical education and training to personnel, certifying that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited, and providing documentation of

employee capability and training to the QA Department, and ensuring that training and documentation are up to date;

- Assigning job tasks and prioritizing analyses;
- Developing and implementing a preventive maintenance program for instrumentation in their laboratory, and ensuring that all equipment is maintained, serviced, and properly calibrated;
- Monitoring QA/QC standards of performance, including ensuring that corrective actions are developed, documented, and implemented for all external and internal audit Findings, PT study failures, and other corrective actions;
- Monitoring the validity of the analyses performed and data generated in the laboratory to ensure the production of compliant data, including, contributing to and/or overseeing data review processes;
- Ensure that SOPs are compliant with promulgated methodologies and reflect current practice;
- Maintaining current, compliant MDL studies for all methods, matrices, analytes, columns, and instruments;
- Coordinating and approving the purchase of reagents, standards, glassware, and equipment that meet requirements;
- Providing input to the Laboratory Director regarding methodologies, personnel resources, software, and instrumentation; and assisting in the evaluation and/or development of new methods and technologies that improve ALSLG-FC's ability to meet clients' needs;
- Reviewing RFPs and assisting in the preparation and submission of proposals; and
- Interacting with the Quality Assurance, Information Systems, and Health and Safety Departments to ensure that the laboratory is capable of complying with client and regulatory requirements.

2.3 GENERAL TECHNICAL PERSONNEL

A chemist (analyst) or technician reports to a Technical or Department Manager. This employee performs work in accordance with ALSLG-FC's controlled

documents (e.g., SOPs, LQAP, etc.) and project-specific requirements as defined by the applicable LIMS specification. *ALSLG-FC believes that quality begins at the bench.* Accordingly, these employees are key contributors to ALSLG-FC's success.

A chemist or technician is responsible for:

- Demonstrating proficiency in the analyses for which they are responsible *before* analyzing samples (e.g., performing acceptable Initial Demonstration of Capability, IDOC studies), and documenting this demonstration of proficiency;
- Performing analyses, recording all data accurately, directly, and promptly, and interpreting and reviewing data according to established procedures;
- Read and understand all assigned SOPs and plan documents;
- Complying with all QA/QC requirements that pertain to their job function;
- Complying with all health, safety, and waste disposal requirements, as applicable;
- Maintaining and repairing instrumentation;
- Demonstrating good house-keeping practices;
- Disclosing all instances of nonconformances promptly and in writing using the NCR process (**SOP 928**); and
- Participating in training sessions.

3. **QUALITY ASSURANCE INDICATORS AND OTHER MEASUREMENT PARAMETERS**

ALS Laboratory Group, Fort Collins' objective is the development and implementation of policies and procedures that provide results of known, documented, and appropriate quality. This LQAP defines general policies for the analysis, documentation, evaluation, validation, and reporting of data. Specific, detailed procedures for chain-of-custody, calibration of instruments, analysis, reporting, quality control, audits, preventative maintenance, and corrective actions, are provided in SOPs as listed in **Appendix H**.

In order to produce data of known, documented, and appropriate quality, ALSLG-FC:

- maintains an effective quality assurance program that measures and verifies laboratory performance;

- provides for a Quality Assurance Department that is independent of the operational groups and that has stop-work authority, and that has the responsibility and authority to audit the laboratory and develop and enforce corrective actions;
- evaluates technical and service requirements of all analytical services requests before accepting samples from a client/project. This evaluation includes a review of facilities, instrumentation, staffing, turnaround times, and any project-specific quality control or reporting requirements;
- provides sufficient flexibility to allow controlled changes in routine methodology in order to achieve client-specific data requirements as prescribed in client documents and contracts;
- documents initial demonstration of capability (IDOC) and continuing demonstration of capability (CDOC) for all methods according to Appendix C of the NELAC standards;
- performs all analyses according to promulgated methods or methods developed and validated by ALSLG-FC and documented in SOPs;
- recognizes as soon as possible and discloses and corrects any factors that adversely affect data quality; and
- maintains complete records of sample submittal, raw data, laboratory performance, and completed analyses to support reported data.

3.1 DATA QUALITY INDICATORS

Data Quality Indicators (DQIs) are qualitative and quantitative statements developed by data users that specify the quality of data from field and laboratory data collection activities in order to support specific decisions or regulatory actions. The DQIs describe *what* data are needed, *why* the data are needed, and *how* the data will be used to address the problem being investigated. DQIs also establish qualitative and quantitative goals that allow the data user to determine whether the data are of sufficient quality for the intended application.

The principal DQIs are **precision**, **accuracy** (bias), **representativeness**, **completeness**, and **comparability** (i.e., the PARCC parameters). The following sections define and describe the application of these parameters. The QA/QC protocols used for the majority of analyses are adopted from SW-846 and 40 CFR methodologies, the USEPA Organics and Inorganics CLP SOWs, and various radiochemistry guidances, which contain detailed descriptions of the quality control measures routinely employed.

3.1.1 PRECISION

Precision is an expression of the reproducibility or degree of mutual agreement among independent measurements as the result of repeated

application of the same process under similar conditions. Precision refers to the distribution of a set of reported values about the mean, or the closeness of agreement between individual test results obtained under prescribed conditions. Precision reflects random error and may be affected by systematic error. Precision characterizes the natural variation of the matrix and the contamination that may vary within that matrix. For chemical parameters that do not allow homogenization prior to analysis (e.g., volatile organics analysis), one must review precision values carefully.

Analytical precision is a measurement of the variability associated with duplicate or replicate analyses of the same sample in the laboratory. Analytical precision is determined by the analysis of matrix spike/matrix spike duplicates (MS/MSD), laboratory control sample pairs (LCS/LCSD), or by unspiked duplicate samples (DUPs). Total precision is a measurement of the variability associated with the entire sampling and analysis process, and is determined by analysis of duplicate or replicate *field* samples, thus incorporating the variability introduced by both the field and laboratory operations.

Precision is independent of bias or accuracy, and reflects only the degree to which the measurements agree *with one another*, not the degree to which they agree with the true or accepted value of the parameter measured. Precision for stable chemistry analyses is typically expressed as relative percent difference (RPD), as defined below:

$$RPD(\%) = \frac{X_1 - X_2}{(X_1 + X_2) / 2} (100)$$

where:

RPD = Relative Percent Difference

X₁, X₂ = analyte value of sample 1 and sample 2

Precision, for radiochemical analyses, is typically measured in terms of Duplicate Error Ratio (DER), calculated as follows:

$$DER = \frac{|S - D|}{2 * \sqrt{\sigma^2_S + \sigma^2_D}}$$

where:

DER = Duplicate Error Ratio

S, D = analyte values of (S)ample and (D)uplicate

σ = One Sigma error value associated with sample result

RPDs or DERs are compared to the control limits established for the analysis method, or other quality control criteria as prescribed in the applicable LIMS program specification. Precision objectives vary per analytical method. Sample homogeneity/non-homogeneity is an important factor that influences the precision of duplicate sample results.

3.1.2 ACCURACY

Accuracy is an expression of agreement between the measured and known or accepted reference values. Accuracy is the measure of the closeness of an observed value to the “true” value (e.g., theoretical or reference value or population mean). Accuracy is influenced by random error and systematic error (bias) that occur during sampling and analytical procedures; therefore, accuracy reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ significantly from the known concentration of the spike or standard.

Accuracy is typically measured by determining the percent recovery of known target analytes (i.e., a surrogate or matrix spike) that are spiked into a field sample or reagent water or simulated solid matrix (laboratory control sample). Surrogate recovery is reported and is used to assess method performance for each sample analyzed for volatile and semivolatile organic compounds. For organic and inorganic parameters, the stated accuracy objectives apply to spiking levels at or near the midpoint of the calibration curve. For radiochemical analyses, the spiking levels for the control spikes may vary from five to fifty times the method reporting limit.

Percent recovery is calculated as:

$$R(\%) = \frac{(C_1 - C_2)(100)}{C_3}$$

where:

R% = Spike amount recovered

C₁ = Concentration of analyte in spiked sample

C₂ = Concentration of analyte in unspiked sample

C₃ = Concentration of spike added

Acceptance limits are usually based upon established laboratory performance for similar samples. Other quality control criteria may be prescribed in the applicable LIMS program specification. Recoveries outside the established limits may indicate some assignable cause other than normal measurement error, and the need for corrective action. This corrective action may include reanalysis of the quality control

sample, recalibration of the instrument, reanalysis of the affected samples in the batch, re-preparation of samples in the batch, or flagging and qualifying the data as suspect if the problem cannot be resolved. For contaminated samples, recovery of matrix spikes may depend on homogeneity, matrix interference, and dilution requirements for quantitation.

Both accuracy and precision are calculated for each batch and the associated sample results must be interpreted by considering these specific measures. The quality assurance objectives for precision and accuracy are to achieve the quality control acceptance criteria specified in the appropriate analytical procedure.

For organic analyses, precision and accuracy are determined by using matrix spike and matrix spike duplicate samples and/or surrogate spike compounds and laboratory control samples. For inorganic analyses, precision and accuracy are determined by using duplicate samples or matrix spike duplicate samples (precision) and matrix spike and laboratory control samples (accuracy). For radiological analyses, precision and accuracy are determined from the results of duplicate samples or matrix spike duplicate samples (precision), laboratory control sample duplicates (precision) and laboratory control samples (accuracy).

Samples identified as field blanks cannot be used for duplicate or matrix spike sample analyses.

QC limits for accuracy and precision may be developed from intra-laboratory historical data or adopted from prescribed limits required by the client. If quality control acceptance criteria do not exist for a given method, then the laboratory may establish advisory control limits derived from a minimum of four data points. Until verified by a statistically significant data population, the control limits will be considered as advisory limits only, and the laboratory will not automatically initiate reanalysis if these limits are not achieved. See Section 9.3 for further discussion of control limits and control charts.

Bias describes the systematic error of a measurement process that causes errors in one direction from the true value. Sources of bias include incomplete homogenization before subsampling and incomplete extraction of target analytes. Calibration drift, which is the nonrandom change in a measurement system over time, is another example of systematic error, and is detectable by the periodic measurement of calibration check standards. *Bias is **not** equivalent to accuracy.*

3.1.3 REPRESENTATIVENESS

Representativeness is a qualitative element. It expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary.

Sample handling protocols (e.g., holding times, storage, preservation and transportation) have been developed to preserve the representativeness of the samples. Proper documentation establishes that quality control protocols have been followed, and sample identification and integrity are ensured. *ALSLG-FC makes every attempt to ensure that the aliquots taken for analysis are homogenous*

and representative of the samples received.

3.1.4 COMPARABILITY

Comparability is a qualitative expression of the confidence with which one data set can be compared to another. Comparability is achieved by:

- following established, standardized, and approved sample collection techniques and analytical methods;
- achieving holding times;
- reporting results in common units;
- using consistent detection levels; and
- reporting data according to consistent rules.

See Chapter 10 of this LQAP for further discussion of standard units typically used to report various analytical parameters.

3.1.5 COMPLETENESS

Completeness is an expression of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Completeness is the percentage of measurements that are judged to be usable (i.e., that meet project-specific requirements). Completeness goals are defined in the site sampling and analysis plan, QAPjP or contract, and vary with the size and complexity of the project. Completeness goals of 80-95% are traditionally accepted as realistic. ALSLG-FC's objective is 100% completeness for samples unaffected by matrix interferences.

It is recognized that some samples are highly contaminated with target and/or non-target compounds, which necessitate cleanups, multiple analyses, and/or extensive dilutions. In these instances, the internal QC results for a sample help to demonstrate the impact upon recoveries and detection limits due to these atypical situations.

Factors that adversely affect completeness include:

- receipt of samples in which chain-of-custody or sample integrity is compromised in some manner (e.g., broken containers, improperly preserved);
- receipt of insufficient volume to perform initial analyses or repeat analysis if initial efforts do not meet QC acceptance criteria;

- receipt of samples for which more than 50% of the holding time has expired; and
- receipt of samples that contain high levels of contamination that can cause persistent effects on instrumentation designed for trace-level analyses.

The equation used to calculate completeness is:

$$C\% = \frac{S}{R} (100)$$

where:

C = completeness

S = number of successful analyses

R = number of requested analyses

The USEPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test as a result of random error, assuming the confidence interval is established at 95% (preamble to 40 CFR Part 136, Vol. 49, No. 209, October 26, 1984). As the number of compounds measured increases in a given sample, the probability for realizing statistical error also increases. The number of compounds present in various methods (e.g., GC/MS Methods SW8260B and SW8270C, ICAP Method SW6010B and Gamma Spectroscopy Method EPA 901.1), increases the probability that one or more analytes will not meet acceptance criteria, to significantly more than the 5% per analyte frequency. The number of target analytes included in these methods can be used to show that a minimum of four to seven target analytes will exceed the control limits established for these methods as a result of the statistical probability for random error. *Establishing quality control criteria that are not consistent with the measurement of the quality objectives for which they are intended is discouraged.*

3.2 TRACEABILITY

Traceability is the extent to which results can be substantiated by hard-copy documentation, electronic or computer-generated data calculations, computer software, and data generation. Traceability documentation exists in two forms: (1) that which links final numerical results to authoritative measurement standards, and (2) that which explicitly describes the history of each sample from collection to analysis. Measurement traceability is further discussed in Chapter 7 of this LQAP.

3.3 SENSITIVITY

The term sensitivity is used in a broad sense to describe the various limits that enable a laboratory to meet project-specific data quality objectives (DQOs). These limit types include: instrument detection limit (IDL), method detection limit (MDL), method quantitation limit (MQL) or method reporting limit (RL), contract-required detection limit (CRDL), and contract-required quantitation limit (CRQL).

3.3.1 IDL AND MDL

The IDL is a minimum value that addresses the detection capability of the instrument *only*, hence IDL studies are performed on a per analyte per instrument basis. IDL studies are particularly important to metals analyses. These IDL studies must be conducted on a quarterly basis, per method requirements, or whenever there is a significant change in instrument components or reagents.

The MDL is a minimum value that addresses the detection capability for the sample preparation procedures and the instrument. Hence, ALSLG-FC performs MDL studies for each preparatory and determinative method combination, matrix, instrument, and analytical column. MDL studies are performed with a frequency that's prescribed by the method, at minimum, annually. Some Wet Chemistry methods require MDL studies to be performed every 6 months. MDL studies are also required for method validation, and whenever the basic chemistry of a procedure changes.

MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. MDLs are determined from replicate analysis (minimum of seven) of a sample in a given matrix containing the analyte(s). 40 CFR Part 136 Appendix B defines the MDL is defined as:

$$\text{MDL} = t(n-1, 1-\alpha, = 0.99) \times \sigma$$

where:

σ = Standard deviation of the replicate analyses

$t(n-1, 1-\alpha, = 0.99)$ = Student's t-value appropriate to a 99% confidence level

An MDL check sample, at a concentration about half that spiked for the MDL study and approximately twice the calculated MDL, is also analyzed with the MDL study (immediately following), to demonstrate that the MDL is valid. Performance criteria is that the MDL check is acceptable if it yields a confident positive detection (i.e., all analytes in the check sample can be identified by method-specified criteria). If MDL check sample results do not support the determined MDL,

appropriate corrective actions must be taken (e.g., repeat MDL check sample analysis, repeat MDL study, raise MDL).

An MDL study is not performed for radiological analyses, or any components for which spiking solutions are not available or relevant (e.g., pH, ignitability, etc.). Reporting limits for these kinds of parameters, where applicable, are established based on the laboratory's knowledge of extraction efficiency, instrument sensitivity, and experience with the procedure. **SOP 329** provides additional information about MDL studies.

Although the QA Department provides oversight, each Department Manager is responsible for ensuring that all IDL and MDL studies are conducted and documented as needed.

Results calculated between the MDL and the MQL (RL), see section following, contain a significant amount of error (approximately $\pm 100\%$). Therefore, values reported between the MDL and MQL (RL) are qualified as estimated – 'J' flagged for organic parameters, 'B' flagged for inorganic parameters. Also, IDL and MDL values are based on an interference-free matrix, and cannot evaluate the effects of sample matrix on the calculated IDL or MDL. Therefore, established IDLs and MDLs may not be achievable in environmental matrices.

3.3.2 MQL, RL

ALSLG-FC defines MQL (RL) as the analyte concentration at or above which the laboratory's precision and accuracy requirements can be routinely demonstrated and achieved. The statistical error associated with this region of a calibration curve is significantly smaller than that associated with the region near the MDL. The MQL (RL) values for most analytes reported by ALSLG-FC are numbers that are approximately 3 to 5 times the values of the MDL for those analytes. It is ALSLG-FC's policy to analyze a calibration standard at or below the MQL (RL) when performing an initial calibration.

The MQL or RL is the lowest level that can be reliably measured by a laboratory with defined limits of precision and accuracy. The USEPA CLP SOW uses the terms CRDL and CRQL to describe *contractually-required* levels of reporting. These reporting terms do not describe instrument sensitivity.

3.4 MINIMUM DETECTABLE CONCENTRATION

The minimum detectable concentration (MDC) is used for radiochemical procedures and is defined as the concentration at which there is a 95% confidence that an analyte signal will be distinguishable from an analyte-free sample.

The general formula for calculating the MDC is based on calculations derived by Curie (Curie, L.A., "Limits for Qualitative Detection and Quantitative Determination," Analytical Chemistry 40(3); pp. 586-693; 1968) and is calculated as follows:

$$MDC = \frac{(4.65 \times \sigma_b) + 2.71}{T * K}$$

where:

MDC = Minimum Detectable Concentration

σ_b = Standard deviation of the measurement background

T = Sample count time

K = Factor for incorporating efficiency, abundance, aliquot yield, ingrowth and decay, and activity conversion factors

3.5 TOTAL PROPAGATED UNCERTAINTY

Total propagated uncertainty (TPU), is a summation of the various uncertainties present in a measurement process, and is an integral part of every reported radiochemical value. TPU, reported as \pm TPU, is the expressed estimated measure of the total uncertainty inherent in that reported radiochemical result.

The components of the TPU are classified as either random or systematic. Random uncertainties, also called counting uncertainties (CU), derive from the statistically random (normally distributed) nature of radioactive decay, and are estimated as the square root of the total number of counts acquired during analysis. In cases where the chemical yield is determined by the analysis of a radioactive tracer, the yield uncertainty (YU) is also a random uncertainty, and is estimated as the square root of the total number of tracer counts acquired. CU and YU are calculated in activity units to afford comparability to the sample result.

Systematic uncertainties are attributable to actual errors in the measurement of a physical quantity. For example, if a balance has an accuracy of $\pm 0.1\%$, the results of those gravimetric measurements are not normally distributed, but rather are assumed to be biased by that amount. Estimates of systematic uncertainties in laboratory processes are somewhat subjective, but should be supported by empirical data whenever possible. Systematic uncertainties associated with the preparation of a sample are called preparation uncertainties (PU), and are defined based on the number of volumetric and gravimetric measurements, quantitative transfers, etc. Systematic uncertainties associated with the analysis, called instrument uncertainties (IU), include biases associated with sample positioning, standard values, calibration coefficients, etc. PU and IU are typically provided as a percentage of the final result. To afford comparability to sample results, PU and IU are expressed in activity units by multiplying the percentage by the sample activity (A).

All contributions to TPU are considered to be independent of each other, and the individual contributions are combined as the square root of the sum of the squares

(see equation below). The final TPU result is expressed in activity units, such as pCi/g or pCi/L.

$$TPU = \sqrt{CU^2 + YU^2 + (A * PU)^2 + (A * IU)^2}$$

TPU is expressed as a value at a specific confidence interval. The default convention at ALSLG-FC is to provide the TPU at the 2-sigma confidence interval. This asserts approximately a 96% confidence level that the actual sample value is within the reported uncertainty range of the calculated result. **SOP 708** provides more information about the calculation and use of TPU.

3.6 QUALITY ASSURANCE PROJECT PLAN (QAPjP) EXCEPTIONS

As a result of the unknown nature of environmental samples prior to analysis, ALS Laboratory Group, Fort Collins has minimal control over analytical and quality control complications that result from sample matrix conditions. These conditions may include highly concentrated samples that contain target compounds of interest and/or non-target components; high organic content (both natural and synthetic); and extremes in pH, viscosity, solubility, etc. Each of these conditions may require a different approach.

Analysis for some samples may be achieved through the use of reduced aliquot sizes. Some sample matrices may require the laboratory to use cleanup and/or dilution techniques in order to analyze the sample by the desired protocol. Unfortunately, reduction of analysis aliquot or diluting a sample necessitates raising reporting limits (RLs) or MDCs, and often adversely impacts the calculation of surrogate, tracer, and matrix spike compound recoveries.

ALSLG-FC has the responsibility to identify matrix interferences that preclude the generation of ‘compliant’ data. This determination may be made by demonstrating reproducibility (i.e., reanalysis of the affected sample) to show that the quality control measurement failure resulted from sample matrix conditions beyond the laboratory’s control and not as a result of analytical error. For example, if the surrogate or tracer recoveries are outside of control limits, then samples may be re-extracted and/or reanalyzed. Repeated non-compliant results indicate that sample matrix probably prevented the laboratory from reporting results deemed compliant.

Analytical projects containing particularly “dirty” samples (i.e., highly contaminated with target compounds and/or matrix co-extractives) will often fail to meet pre-established completeness goals (set forth in the QAPjP), when prior site history does not reveal the matrix constituents issues. Although the laboratory performs all analytical testing and cleanup procedures by the prescribed protocols, the results obtained may not meet validation criteria as a result of elevated reporting limits or the frequency at which surrogate, internal,

tracer, or matrix spike recoveries fail to meet acceptance limits. In cases where the laboratory is unable to meet quality control criteria as a result of sample matrix complications, results that are qualified by data validation guidelines may still be useful to the end user of the data.

ALSLG-FC is committed to adhering to the method requirements and quality control procedures prescribed by our clients. ALSLG-FC strives to produce compliant data, however, uncertainties associated with environmental samples may preclude the laboratory's ability to generate fully compliant data. ALSLG-FC will not assume responsibility for conditions beyond our reasonable control, that directly impact the "validity" versus the usability of the associated analytical data generated by the laboratory.

4. SAMPLE CONTAINERS, PRESERVATION, HANDLING, HOLDING TIMES

Defining the magnitude and nature of an environmental problem, and developing an appropriate solution, requires the collection of representative samples for laboratory analysis and data evaluation. The objective of field sampling is to remove a small portion of an environment that is representative of the entire body. *Analytical methods have been standardized, but the results of analyses are only as good as the sampling protocol and the sample preservation and handling methods.* Defining sampling procedures and the quality elements applicable to environmental testing is beyond the scope of this document, and beyond the responsibility of the laboratory.

Although the laboratory is not responsible for sample collection, it is responsible for maintaining the integrity of the sample after receipt. After the sample has been collected, the constituents of the sample must remain as close as possible to the field condition (i.e., degradation must be prevented). The length of time that these constituents will remain stable is related to their character and the preservation method used. Preservation is accomplished by the addition of chemical preservatives and/or storage at a controlled temperature, and by the strict observation of prescribed maximum holding time allowances. **Appendix C** lists sample container types, preservation requirements, and holding times.

4.1 FIELD SUPPORT

Unless not required by the client, sample kits are prepared at the laboratory to provide the client with all of the sample containers, preservatives and documentation needed for the analyses needed for a project. ALSLG-FC provides shipping containers, custody documents, custody seals, clean sample bottles, labels, applicable high-purity chemical preservatives for water samples, trip blanks, and, upon request, "blue ice" packs to support field-sampling events. Hard-sided, insulated, "picnic" coolers are typically used to transport samples from the field to the laboratory. These coolers meet or exceed all protocol requirements (i.e., USDOT, USEPA, ASTM) for shipping. ALSLG-FC **SOP 205** provides further information on sample kits.

4.2 SAMPLE CONTAINERS

ALS Laboratory Group, Fort Collins provides certified clean (I-Chem 300™,

Eagle Pitcher Level 1, or equivalent) sample bottles for sample collection. Used sample bottles are never used by the laboratory. The Sample Receiving Department maintains certificates of cleanliness that are provided by the vendor for all sample bottles. These certificates are provided to the client upon request. Containers are stored in clean areas, away from laboratory processes, to prevent exposure to fuels, solvents, and other contaminants.

4.3 SAMPLE PRESERVATION AND HOLDING TIMES

ALSLG-FC provides the required chemical preservatives for water samples and, upon request, “blue ice” packs, for thermal preservation during transport. Typically, high quality reagent grade chemical preservatives (i.e., acids, solutions, etc.) are added to individual sample bottles, as appropriate per method and US Department of Transportation (DOT) requirements. Only trace metals grade nitric acid is used for preservation of metals or radiochemical samples, as applicable. It is the responsibility of those collecting the samples to properly use these materials (e.g., don’t rinse or overfill container such that the preservative is washed out), and to ensure that chemical preservation requirements are met, and proper preservation techniques (chilling) are performed. Holding times begin with the collection of samples and continue until analysis is complete. See **Appendix C** for a summary of container, preservation and holding time requirements specific to various analyses and matrices.

4.4 SAMPLE RECEIPT SCHEDULE

ALSLG-FC receives samples six days of the week, Monday through Saturday. ALSLG-FC requests that clients ship samples for delivery within one day of collection, and give advance notice to the laboratory regarding shipment of RUSH samples or samples with short hold time requirements. Shipping containers received at the laboratory on holidays or after business hours are placed in a walk-in refrigerator and opened on the next business day, unless other arrangements are made in advance.

4.5 CHAIN-OF-CUSTODY

Chain-of-custody (COC) documentation begins with field sampling and continues through laboratory analysis and disposal. A chain-of-custody record that identifies all individuals who handle the sample is used to establish an intact, continuous record of the physical possession, storage, and disposal of collected samples, including their aliquots, extracts or digestates. The chain-of-custody record is initiated in the field by field personnel who complete a COC form listing all samples. This form contains the following information and remains with the samples during transport:

- client project name and project location;
- field sample number/identification;
- date and time of sample collection;

- matrix;
- container type and number of containers for each sample;
- preservative;
- analysis requested;
- sampler's remarks and signature;
- signature of person relinquishing samples and date and time relinquished;
- custody seal number (if applicable); and
- designation of matrix spike/matrix spike duplicate (MS/MSD) samples (optional).

Note that contingent upon the sample matrix and analysis to be performed, additional sample volume may need to be submitted to accommodate MS/MSD analyses.

All transfers of samples, except directly between commercial couriers, must be recorded on the chain-of-custody form via the "relinquished" and "received by" sections. All information, except signatures, should be clearly printed.

The USEPA National Enforcement Investigations Center (NEIC) defines evidence of custody as:

- in one's actual possession, or
- in one's view, after being in one's physical possession, or
- having been in one's possession and then locked or sealed to prevent tampering, or
- kept in a secure area, restricted to authorized personnel only.

To ensure that sample custody objectives of traceability are achieved for every project, the chain-of-custody initiated in the field, is continued and maintained internally throughout the laboratory per the requirements specified in **SOP 318**. Internal chain-of-custody begins with sample acceptance and login (**SOP 202**), is maintained as samples are distributed for use throughout the laboratory (further discussed in LQAP Section 4.10), and concludes with final sample disposition (i.e., return to the client or disposal). ALSLG-FC applies a unique barcode to each sample bottle received, and maintains several scanners and PCs throughout the laboratory to document and assist with sample, aliquot, extract and digestate movement throughout the facility. This electronic process is accomplished through LIMS, which retains records of all sample and fraction transactions made.

4.6 **SAMPLE ACCEPTANCE POLICY**

ALS Laboratory Group, Fort Collins' sample acceptance policy requires that a

sample meet the following conditions:

- The sample shall be completely documented (sample identification, location, date and time of collection, collector's name, preservation type, sample type, any special remarks concerning the sample);
- The sample shall be identified by a unique identifier using durable labels completed in indelible ink;
- The sample shall be collected in adequate volume;
- The sample shall be collected in an appropriate container;
- The sample shall be delivered to the laboratory with at least one-half the holding time remaining;
- The sample shall not exceed allowed radioactivity levels; and
- The sample shall not show signs of contamination, breakage, or leakage.

Sample receipt discrepancies are documented by Sample Receiving Department personnel on the Condition of Sample Upon Receipt, Form 201 (**Appendix D**), which is forwarded to the Project Manager as part of the workorder folder. Where samples do not meet the criteria stated above, the Project Manager requests information from the client before proceeding. If the client can provide the information and, in cases of compromised sample integrity, directs the laboratory to proceed, then data acquired from the sample(s) analysis is reported and the problems noted during sample receipt are disclosed in the narrative of the final data report.

In support of the protection of employee health and of ALSLG-FC's radioactive materials license, ALSLG-FC observes prescreening protocols that designate or determine samples with radioactive content. Detailed procedures for conducting radiological survey of incoming sample packages are given in **SOP 008**, further details regarding prescreening protocols are given in **SOP 703**.

4.7 SAMPLE RECEIPT PROTOCOLS

Upon receipt of the field samples at the laboratory, personnel ensure that sample bottles are maintained according to storage requirements, and in a manner that does not contaminate the samples (see section 4.9 for further details).

Ascension numbers that increment serially each month of the year are applied as workorder number assignments. Following sample arrival and initial screen for USDOT compliance and removable radioactivity, sample receiving personnel inspect the sample and record any discrepancies using Form 201 (**Appendix D**). The following information is documented:

- client and project name, as applicable;
- presence/absence and condition of (i.e., intact, broken) custody seals on

the shipping containers;

- presence/absence of chain-of-custody and completeness;
- sample condition (intact, broken, leaking);
- presence/absence of removable sample tags;
- agreement/non-agreement between the sample labels, tags, chain-of-custody, and any other client documentation;
- receipt of adequate sample volume;
- sample temperature, where applicable;
- presence/absence of headspace in VOA and ²²²Radon vials; and
- chemical preservation, where applicable.

Sample temperature is verified upon receipt by measuring the temperature of the temperature blank (if available) or by measuring the temperature of a representative samples(s) with an infrared (IR) temperature device. See **SOP 210** for instructions and procedures related to IR temperature guns. Samples that require thermal preservation are considered acceptable if the temperature upon arrival is between just above freezing to 6°C. Samples that require thermal preservation but are hand-delivered to the laboratory immediately after collection, may not meet the temperature requirement. If the hand-delivered sample is packed in ice, then Sample Receiving personnel record its temperature and note that the chilling process was initiated.

4.8 SAMPLE LOGIN POLICIES AND PROCEDURES

After completing sample receipt procedures, the following sample information and analytical requests are entered into LIMS under the unique workorder number assigned:

- client name, contact, address, phone number;
- ALSLG-FC Project Manager;
- date and time of sample receipt;
- unique laboratory identifier for each sample;
- sample description, including date/time of collection;
- analyses requested (LIMS calculates holding times for each analysis);
- program specification or other special instructions, if applicable; and
- due date.

In general, a group of received samples is assigned one workorder number in LIMS. Each sample container is assigned a unique ALSLG-FC identifier

(barcode) that is placed on each container. This unique identification includes all samples, subsamples, and subsequent extracts and/or digestates.

See **SOPs 201 and 202** for additional information about sample login and distribution.

4.9 **SAMPLE STORAGE**

Samples requiring thermal preservation are stored in designated refrigerated storage areas that are maintained just above freezing to 6°C, centered at 4±2°C. Freezer storage areas are maintained at freezing to -20°C, centered at -15±5°C. The temperature of refrigeration units is monitored continuously using electronic min/max thermometers and recorded each business day, near to the beginning of the work shift. If the temperature exceeds the prescribed range, then corrective action is taken and documented immediately, and the client notified, if appropriate; see **SOP 326** for further details. Directives for corrective action pertaining to catastrophic failure of cooling units (as well as laboratory ovens, etc.) are included in ALSLG-FC's Emergency and Contingency Plan (ECP).

Samples are stored away from all standards, reagents, food and other sources of contamination. Samples are stored in such a manner as to prevent cross-contamination. For example, pure product or potentially contaminated samples are tagged as "hazardous" and stored within a secured area, separate from other samples. ALSLG-FC provides designated sample storage areas according to the following parameter groups: metals, inorganics (WetChem), semivolatiles, organics, volatile organics, fuels, and radiochemical analyses.

Samples having suspected radioactive activity and scheduled also for stable chemical analyses are refrigerated. Samples to receive tritium analyses are refrigerated. Samples designated for radiochemistry analyses *only*, with the exception of tritium, are segregated and maintained at ambient temperature.

To effectively monitor the storage and potential contamination of volatile organic samples, ALSLG-FC observes a refrigerator blank program (detailed in **SOPs 511, 512**).

To provide for the safe containment of sample material that could be released as a result of sample container failure, all samples are stored in secondary containment bins. These secondary containment bins are of a sturdy and inert nature, and are sufficient in size to fully contain the sample(s) in the event of a spill, leak or breakage. The bin(s) may be uniquely identified (labeled) to assist in locating samples via the chain-of-custody system. The bins are thoroughly cleaned between uses.

4.10 **SAMPLE ACCESS**

It is ALSLG-FC's policy that neither samples nor data may be released to unauthorized personnel. In order to ensure that this policy is maintained, the

laboratory facilities are maintained under controlled access and are restricted to authorized personnel only (see **SOP 132** for further details pertaining to building security).

As discussed previously in this section, ALSLG-FC personnel follow strict sample handling and internal chain-of-custody procedures to ensure the integrity of all data generated. Limited access electronic controls in LIMS further protect the validity of the data results. Samples are scanned and transacted in LIMS when they are removed from a storage area for preparation or analysis. The sample ID, analyst, date, time, and location are recorded with each transaction. Likewise, the samples are scanned and transacted in LIMS upon their return to the storage unit. Barcode scanning and LIMS transaction is also observed for the return of sample remainders to the client, and for disposal (see LQAP Section 4.13). ALSLG-FC **SOP 318** contains internal chain-of-custody details; procedures for sample return to the client are described in **SOP 027**.

4.11 SAMPLE HOMOGENIZATION AND SUBSAMPLING

Obtaining a representative aliquot of sample for testing is critical to the representativeness of the analytical results obtained. Proper subsampling techniques, particularly for solid matrices, are a component of each bench employee's technical instruction. Sample homogenization procedures prior to radiochemical analysis are prescribed in **SOP 721**. Representative subsampling procedures for stable chemistry analyses, may be discussed in individual preparatory SOPs, and additional guidance, "Subsampling Soils and Sediments", is also posted to the ALSLG-FC network for ready reference. Client-specified procedures for homogenization or aliquotting may also be defined in the applicable LIMS program specification.

4.12 SUBCONTRACTING ANALYTICAL SERVICES

ALS Laboratory Group, Fort Collins strives to identify the need to subcontract specific analytical procedures during the bid response process. Analyses may also need to be subcontracted, however, in cases of emergency where the ability to meet sample holding time criteria is endangered. In these instances, ALSLG-FC compiles a list of qualified subcontract laboratories that are suitable to perform the needed analyses, then submits the list to the client for selection and approval. If NELAC certified analyses are to be subcontracted, the subcontract laboratory must also hold NELAC certification for the analyses that are to be conducted. The same concept regarding subcontract laboratory qualifications may apply for other program samples (e.g., DOD laboratory approval status is required for the analyses to be conducted in the case of DOD samples that must be subcontracted for analysis). Note that for subcontracted DOD sample analyses, the subcontract laboratory must receive project-specific approval from the DOD client before any samples are analyzed.

ALSLG-FC's Project Manager must receive permission from the client, in writing, before the subcontract laboratory can be procured and samples forwarded

to the laboratory. At a minimum, the specific terms of the subcontract laboratory agreement must include:

- analytical method required (e.g., SW-846, 40 CFR, etc.);
- number and type of samples expected;
- project-specific quality control requirements;
- deliverables required (hardcopy, electronic);
- laboratory certifications required;
- price per analysis; and
- turnaround time requirements.

See **SOP 103** for guidance on evaluating a subcontract laboratory's qualifications. Detailed procedures pertaining to submitting samples to a subcontract laboratory are provided in **SOP 103**.

4.13 SAMPLE DISPOSAL

After completion of sample analysis and submission of the project report, unused portions of samples are retained by the laboratory for a minimum of 90 days from date of invoice. Samples are disposed or returned to the client according to the nature of the samples and the client's specifications. ALSLG-FC documents and retains all conditions of disposal and correspondence between all parties concerning the final disposition of the sample.

Samples, digestates, leachates, extracts, and process waste that are characterized as hazardous, radioactive, or mixed waste are disposed in accordance with Federal and state laws and regulations. ALSLG-FC maintains records to demonstrate that all disposal efforts were conducted in compliance with these laws and regulations. This documentation includes the unique sample identity, date of disposal, nature of disposal (e.g., sample depleted, sample disposed in hazardous waste facility, sample disposed in mixed waste facility, sample returned to client); and name of the individual responsible for disposal.

5. LABORATORY FACILITIES

Appendix E contains a diagram of the ALS Laboratory Group, Fort Collins laboratory facility. ALSLG-FC maintains constant and consistent test conditions throughout the facility (e.g., temperature, air purification, lighting). All entrances and exits are wired to a laboratory-wide security system that is monitored continuously. Access to the laboratory area from the front offices is restricted by means of keypad locks requiring numeric security code entry. Visitors must sign in at the front desk and must be escorted at all times (some vendors are allowed access without continuous escort, in order to facilitate repairs or deliveries). Further details pertaining to building security are provided in **SOP 132**.

The following sections highlight areas of the laboratory that are involved with sample

receipt, handling, preparation, and analysis of samples.

5.1 SAMPLE RECEIPT AREAS

ALSLG-FC's sample receiving area consists of a large dedicated room of more than 500 ft². It contains two fume hoods and radiation survey equipment to safely handle incoming radioactive and mixed waste samples. There is an outside access door to facilitate sample delivery and shipping of sample kits. Adjacent to the sample receiving area is the bottle storage room and the radioactivity prescreening lab.

5.2 SAMPLE STORAGE AREAS

ALSLG-FC's sample receiving area has a walk-in cooler and a freezer that are used for temporary storage of samples that require thermal preservation. In addition, there are several designated sample storage locations throughout the laboratory that are used to store samples scheduled for specific analyses (see section 4.9 for further details).

5.3 SAMPLE PREPARATION AREAS

The laboratory has nine sample preparation/extraction/digestion areas. These areas are divided as follows: six radiochemistry preparation laboratories; two organics extraction laboratories; and one metals digestion laboratory. The total floor space of these six laboratories is approximately 4500 ft².

Laboratory preparation procedures are segregated as much as possible to minimize the potential for contamination, maximize processing efficiency, and maintain analytical integrity. Rigorous cleaning of glassware (**SOPs 334** and **720**) and apparatus ensures that cross-contamination is minimized. Each laboratory area has a dedicated or locally shared HVAC system that continuously exchanges the laboratory air with filtered and conditioned outside air. There are 34 laboratory hoods in the six sample preparation areas, and each sample preparation area has at least one hood that is capable of maintaining an average face velocity of 100 feet per minute.

5.4 STANDARDS PREPARATION AREAS

A dedicated radiochemical standards preparations room, and an organics standards preparation area are maintained. Metals and inorganic standards are stored independently from sample storage areas and are prepared in their respective laboratory areas.

5.5 ANALYTICAL LABORATORIES

The ALSLG-FC facility houses a volatile organics analysis (VOAs) laboratory that is on an upper level of the building, away from all other laboratory operations. The ALSLG-FC facility also houses one general chemistry (WetChem) laboratory, two radiochemical counting rooms, a total organic carbon (TOC) laboratory area, two gas chromatograph (GC)/high performance liquid

chromatography (HPLC) labs, a semivolatile organic compounds (SVOCs) laboratory, and a metals laboratory that contains separate inductively coupled plasma (ICP), mercury, and inductively coupled plasma/mass spectrometry (ICP/MS) rooms.

5.6 OTHER LABORATORY AREAS

Other areas of the ALSLG-FC facility include a tank room for compressed gasses, several waste management areas, telephone and computer storage rooms, staff offices, Reporting Group and Reports Management data processing rooms, and various scanning/reproduction and supply storage areas.

5.7 DEIONIZED WATER SYSTEM

Within the laboratory, there are two main deionized (DI) water distribution systems available for glassware cleaning, bulk reagent preparation, and general use. One system is located in the janitor's area and serves the radiochemistry side of the facility (ASTM Type II water generated). The other system is located adjacent to the metals laboratory area and serves the stable chemistry side of the facility (ASTM Type I water generated). These DI water systems are capable of continuously delivering water that meets the requirements specified for the ASTM water type, and are monitored and documented each business day to ensure that the water meets these criteria. ALSLG-FC also maintains a third treated water system that is used to support washing of stable chemistry laboratory glassware.

DI water is defined as municipal tap water that has been treated by passing it through a particulate filter, activated carbon unit, cation exchange resin, anion exchange resin, mixed bed resin, and a final "polishing" cartridge. This water contains no detectable heavy metals or inorganic compounds of interest, and is free of organic compounds of analytical interest above ALSLG-FC's routine reporting limits. Additionally, a benchtop Millipore Synergy 185™ unit is available for laboratory use should further finishing be desired.

SOP 319 provides detailed information pertaining to ALSLG-FC's DI water systems, including discussions of independent monthly testing to verify that electronic readouts of water quality are accurate, maintenance by a vendor contractor, and corrective measures to be taken should water quality degrade to below acceptable limits.

6. ANALYTICAL PROCEDURES

ALS Laboratory Group, Fort Collins is capable of analyzing various matrices, including surface and groundwater, drinking water, soil, sediment, vegetation, tissue, filter and aqueous and solid wastes. ALSLG-FC does not routinely perform analyses on air (non-particulate), however, analysis of these matrices may be available through our sister laboratories. Analyses are performed using promulgated methodologies as requested by the client and their regulators, and as required by ALSLG-FC's certifying authorities. *New iterations of established methodologies are evaluated on an ongoing basis and implemented as client needs dictate.* Analytical procedures are conducted in strict adherence with SOPs

that describe the preparation, analysis, review and reporting of samples. In some cases, these SOPs may also describe proprietary methods developed by ALSLG-FC and used per the client's request. A list of ALSLG-FC's analytical capabilities is presented in **Appendix C**. A list of ALSLG-FC's SOPs is provided in **Appendix H**. References for analytical procedures used are presented in the attached **Bibliography**. ALSLG-FC also, upon request, develops and validates procedures that are more applicable to a specific client objective.

6.1 ANALYTICAL METHODS

Selection of the appropriate method is dependent upon data usage and regulatory requirements. ALSLG-FC may modify existing methods in order to:

- achieve project-specific objectives;
- incorporate modifications or improvements in analytical technology;
- address unusual matrices not covered in available methods; and
- provide analytical capabilities for an analyte for which there are no promulgated methodologies.

ALSLG-FC discloses method modifications to our clients by providing the appropriate SOP for review.

6.2 METHOD COMPLIANCE

Compliance is the proper execution of recognized, documented procedures that are either approved or required. Strict adherence to these procedures is necessary to provide data acceptable to a regulatory body of competent jurisdiction in a specific regulatory context.

Compliance is, however, separate from, but not inconsistent with, technical scientific quality. ALSLG-FC understands that the expectations of our clients commonly include the assumption that data and reports will satisfy a regulatory purpose and will be found acceptable and compliant with regulatory requirements.

6.2.1 UNDERSTANDING THE REGULATORY FRAMEWORK

Compliance is not likely to be achieved in the absence of an understanding of the regulatory framework. Upon receipt of a statement of work (SOW), ALSLG-FC attempts to ascertain, prior to accepting samples:

- what regulatory jurisdiction pertains to a project (USEPA, State Department of Health, etc.)
- within the regulatory jurisdiction, what body of regulations has primacy (RCRA, SDWA, CWA, etc.); and

- within this context, what QA/QC protocols are required (DOE, DoD -- AFCEE, NFESC, etc.).

ALSLG-FC works with our clients to achieve a mutual understanding of all requirements and makes the following commitments:

- ALSLG-FC will proactively attempt to identify and understand the regulatory context of client's needs.
- ALSLG-FC will strive to be expert in understanding and executing the regulatory requirements for compliance.
- ALSLG-FC will ensure that we have the capabilities, resources and facilities to perform the requested analyses.
- ALSLG-FC will identify and disclose to clients instances of non-compliance in a forthright and timely fashion.

6.2.2 RESOLVING COMPLIANCE CONTRADICTIONS

Multiple regulatory jurisdictions may overlap for a specific project, which may cause uncertainty or contradictions to arise. Similarly, methods and protocols may be prescribed in a scope of work or QAPjP that either will not achieve stated or implied DQOs, or that conflict with the regulatory requirements. ALSLG-FC will attempt to detect these inconsistencies and contradictions and will disclose them to clients in a timely fashion. ALSLG-FC voluntarily accepts a responsibility to provide information to our clients; however, the primary responsibility for resolving inconsistencies with regulators remains with the client.

6.2.3 DISCLOSURE OF NON-COMPLIANCE

As previously stated, it is ALSLG-FC's policy to disclose in a forthright manner any detected non-compliance that may affect the usability of data produced by ALSLG-FC. It is not within our expertise to predict the manner in which a specific regulator or regulatory body will interpret the rules governing analysis; therefore, ALSLG-FC is unable to guarantee compliance. It is ALSLG-FC's policy that our responsibility begins with a bona-fide and competent attempt to evaluate potential compliance issues, and ends with disclosure of any findings that may enable our clients to make an informed decision.

Procedures for documenting non-compliances and applying corrective actions are given in **SOP 928**. A copy of ALSLG-FC's Nonconformance Report (NCR) is provided in **Appendix F**.

6.3 NON-STANDARD METHOD VALIDATION

When a non-promulgated method (i.e., methods other than EPA, ASTM, etc.) is required for specific projects or analytes of interest, or when the laboratory develops a procedure, the laboratory must establish the validity of the method prior to extracting or analyzing a client's samples. *Validity is established by meeting criteria for precision and accuracy.* Method development and validation must include the following:

- Initial Demonstration of Capability (IDOC) for each analyst performing the method;
- MDL studies or MDC determination, as applicable, for every analyte, matrix, instrument, and column (if applicable);
- validated extraction and analytical criteria; and
- SOP generation and approval per established processes.

7. MEASUREMENT TRACEABILITY AND CALIBRATION

ALS Laboratory Group, Fort Collins follows a well-defined calibration routine for all instruments and equipment. Calibration may be performed by laboratory personnel using certified reference materials traceable to NIST or equivalent certified materials, or by external calibration agencies or equipment manufacturers. The discussion in this section of the LQAP is general in nature because the requirements for calibration are instrument or equipment and method specific. Details of calibration procedures and requirements can be found in ALSLG-FC's standard operating procedures (SOPs), analytical methods and operations manuals.

A list of all major instrumentation available at ALSLG-FC is provided in **Appendix G**. The Quality Assurance Department maintains this list.

7.1 TRACEABILITY OF CALIBRATION

ALSLG-FC's program of calibration and/or verification and validation of equipment must ensure that, wherever possible, measurements performed by the laboratory are traceable to national standards of measurement. ALSLG-FC requests and maintains calibration certificates (e.g., weights, thermometers, balances) that demonstrate traceability to national standards of measurement. If traceability to national standards of measurement is not available or applicable, then ALSLG-FC provides evidence of correlation of results (e.g., verifying an in-line resistivity meter by reading the system's output with a conductivity meter; participating in a PT studies).

7.2 REFERENCE STANDARDS OF MEASUREMENT

ALSLG-FC uses reference standards of measurement (such as Class S weights or NIST-traceable thermometers) for calibration verification purposes only (i.e., these reference standards are not available to laboratory staff for general use). Reference standards of measurement are calibrated or verified by a qualified

vendor that must provide, where possible, traceability to a national standard of measurement. Thermometer Masters are independently recertified annually, weight masters are independently recertified every five years. Certificates of vendor calibration/verification for the reference standards recertifications are maintained by the Quality Assurance Department.

The certified reference standards are then used to annually verify other measurement devices (e.g., laboratory thermometers, laboratory weight sets) in-house. The in-house verification efforts are managed by the Quality Assurance Department. All items so verified are tagged with a sticker indicating the unique identity of the device, the date of verification and the initials of the technician who performed the verification, and the date the verification is valid through. Procedures for the in-house verification of thermometers are given in **SOP 923**. Procedures for the verification of weight sets are given in **SOP 901**.

7.3 TRACEABILITY OF STANDARDS, SOLVENTS AND REAGENTS

ALSLG-FC purchases the highest quality standards, solvents, and reagents appropriate to the analytical methodologies employed. The vendor must supply a Certificate of Analysis, Certificate of Purity, or equivalent. These certificates are maintained by the Department who uses the materials.

With the exception of extraction solvents, each Department documents the date of receipt, date opened and an expiration date for all standards and reagents by labeling the original container, or certificate and/or by entering this information into ALSLG-FC's Standards and Reagents database. Because of the quantity of solvents consumed in a short time frame, solvents are labeled only with the date received.

Each Department is responsible for the preparation, documentation, storage and disposal of its chemicals. Standards preparation information is documented by entry in a ALSLG-FC's Standards and Reagents database. The following information, needed to maintain traceability of the standard, is recorded for each standard:

- date of receipt of reference standard;
- unique internal identification number and traceability to purchased stock or neat compounds, as applicable (i.e., vendor/lot numbers; unique ALSLG-FC identifier);
- date of preparation;
- name of preparer;
- amount of reference material used;
- volume/identity of reagents and solvents used;
- final volume;

- concentration;
- expiration date of the stock and prepared standards.

See **SOP 300** for additional information about standards preparation, storage, and expiration. Verification (re-verification) of radiochemical standards is also addressed in SOP 300.

7.4 GENERAL REQUIREMENTS FOR CALIBRATION

Each calibration is dated and documented to ensure that it is traceable to the method, instrument, date of analysis, analyte, concentration, and response. Sufficient information must be documented to permit reconstruction of the calibration. Acceptance criteria for calibrations must comply with method requirements.

7.5 INSTRUMENT CALIBRATION

This section defines the essential elements of initial instrument calibration (ICAL) and continuing instrument calibration verification (CCV). These procedures ensure that the data will be of known, documented, and appropriate quality for a given application. *Samples yielding concentrations that exceed the upper limit of the calibration curve shall be diluted and reanalyzed, if possible, to bring the results within the calibrated range. Results of samples outside the known calibration range, above or below, must be reported as qualified values and discussed in the case narrative.*

Initial instrument calibration is used for quantitation and continuing instrument calibration verification is used to confirm the validity of the initial calibration. The following items are required of both initial and continuing instrument calibrations:

- The details of the instrument calibration procedures, including evaluation and acceptance criteria, and corrective measures to be taken in the event that these acceptance criteria are not met, must be included or referenced in the test method SOP.
- Sufficient raw data records must be retained to allow reconstruction of the instrument calibration (e.g., calibration date, test method, instrument, date of analysis, name of analyst, concentration of standard(s), response, response factor).

Additional essential elements of initial as well as continuing instrument calibrations are discussed below.

7.5.1 INITIAL INSTRUMENT CALIBRATION

The following items are essential elements of initial instrument calibration:

- Samples must be quantitated from the ICAL, unless the reference method states otherwise.
- The initial calibration range must consist of at least the minimum number of calibration points specified by the reference method. If the reference method does not specify the number of calibration standards, then the minimum number is two, not including blanks or a zero standard. Exception: multi-component analytes, such as chlordane, toxaphene or Aroclors, may be analyzed using a one-point calibration, per SW-846 guidance, if so requested by the client.
- The lowest calibration standard must be above the detection limit (MDL) and at or below the RL (i.e., the method reporting limit must be within the calibrated range of the method).
- Calibration standards must include concentrations at or below the regulatory limits, if these limits are known to the laboratory.
- Criteria for the acceptance of an initial instrument calibration must be established (e.g., RSD, correlation coefficient, etc.).
- If ICAL results are outside acceptance criteria, then corrective action must be performed, and the instrument recalibrated before analyzing samples.
- Exclusion of initial calibration points without technical justification is not allowed (poor injection or power failure are valid reasons to exclude a calibration point).
- All reported target analytes and surrogates must be included in the initial calibration.
- The ICAL must be verified (see section 7.5.3) before samples can be analyzed.

7.5.2 CONTINUING INSTRUMENT CALIBRATION

A continuing calibration verification (CCV) standard must be analyzed with the frequency prescribed in the reference method, or as dictated by the applicable LIMS program specification (typically within every 12hr time period). For example:

- When an ICAL is not performed on the day of analysis, then validity of the initial calibration must be verified with an

acceptable CCV prior to sample analysis.

- A CCV must be repeated at the beginning and end of each analytical sequence. (For GC/MS methods that use an internal standard, only one CCV must be analyzed before each analytical sequence). Some methods additionally prescribe that a CCV must be analyzed after every 10 (or 20) samples analyzed.

The following items are essential elements of continuing instrument calibration:

- With the exception of multi-component analytes, all reported target analytes must be included in the continuing instrument calibration standard.
- Criteria for the acceptance of a CCV must be established (e.g., %D, %Drift, from the initial calibration).
- If the CCV results exceed acceptance criteria, then corrective actions must be performed. If routine corrective action procedures do not produce a second consecutive CCV within acceptance criteria, then a new calibration must be performed and successfully verified.

Additional aspects of calibration verification are discussed below.

7.5.3 CALIBRATION VERIFICATIONS

All ICALs must be verified with a *second source* standard obtained from a different manufacturer/vendor and traceable to a national standard, when available. If a different manufacturer/vendor is not available, the laboratory must request a different lot number of the standard.

In most cases, a second-source initial calibration verification (ICV) standard is analyzed immediately after the ICAL and before any samples are analyzed. However, analysis of an ICV is not required, if the continuing calibration verification (CCV) standard is from a second source.

The concentrations of the calibration verification standards must be varied within the established calibration range. At least one of the standards must fall below the middle of the calibration range. ALSLG-FC usually accomplishes this criterion by analyzing the ICV at a different and lower concentration than the CCV. Acceptance criteria for an ICV are usually the same as those for a CCV.

Sample data associated with an unacceptable calibration verification standard may be reported as qualified data in the following cases:

- When the acceptance criteria for the CCV is exceeded high (i.e., high bias), and only non-detects were determined for the affected analyte(s) in associated samples, then those non-detects may be reported.
- When the acceptance criteria for the CCV is exceeded low (i.e., low bias), then these sample results may be reported if they exceed a maximum regulatory limit.
- When the acceptance criteria for the CCV are exceeded (high or low), and the effect on the system from previous sample analysis is substantiated (e.g., by reanalysis or sample response characteristics on a different detector), then the sample results may be reported.

Other levels of concentrations and frequencies of analysis for calibration checks (ICVs, CCVs) may be required by specific client programs. These requirements, which supercede method, SOP or LQAP requirements otherwise stated, are communicated to the laboratory staff via LIMS program specifications.

8. PREVENTIVE MAINTENANCE AND REPAIR OF EQUIPMENT

ALSLG-FC maintains an organized maintenance program that is broader than the particular instruments or devices a specific employee may operate or is familiar with. The objective of ALSLG-FC's equipment maintenance program is to provide a structure of care that prevents quality control failures and minimizes lost productivity that results from equipment malfunction or failure. Within this program are provisions for corrective actions, maintaining spare parts, and a contingency plan in the event of catastrophic failure (e.g., loss of power for a significant period of time).

See Appendix G for a comprehensive list of ALSLG-FC's equipment.

ALSLG-FC's maintenance program is based on equipment manufacturer's recommendations, operator training guidance, and other considerations (e.g., sample throughput). The established maintenance program applies to all laboratory primary instrumentation, as well as support equipment (see Section 8.6 for a definition of what constitutes support equipment). Provisions for documenting all routine and non-routine instrument equipment maintenance and repairs is also established within the maintenance program.

Responsibilities for applying ALSLG-FC's maintenance program rests with the Department that utilizes the equipment, the Quality Assurance Department bears responsibility for

certain support equipment such as balances, ovens, refrigerators, freezers, and temperature measurement devices. Only authorized personnel are permitted to perform maintenance.

Culturally, ALSLG-FC makes a distinction between ‘operational’ and ‘routine’ maintenance, that external parties generally do not. ALSLG-FC considers the normal/typical things that operators do to keep the equipment functioning properly (e.g., septum replacement, reagent refill, etc.), as ‘operational’ maintenance, and does not generally view these tasks as routine maintenance events that require specific documentation in a dedicated maintenance log. ALSLG-FC’s view is that the fact that the equipment is performing properly and yielding acceptable QC results, evidences that these maintenance tasks were performed as needed. ALSLG-FC’s maintenance system does, however, provide for attestations that this maintenance was performed, where applicable. In contrast, ALSLG-FC defines routine maintenance as those things done in-house only periodically (i.e., that are beyond what is performed as usual ‘operational’ maintenance), that are short of vendor repair (e.g., annual GFPC drawer evaluation).

Documentation requirements are discussed further in Section 8.4 below.

Note that ALSLG-FC does not consider ‘priming’, or analysis of solvent blanks, which generally get recorded in the instrument run log, as maintenance.

8.1 MAINTENANCE SCHEDULES

In general, ALSLG-FC performs maintenance as needed (including preventive considerations). Certain aspects of routine maintenance are considered to be ‘operational’, and are performed each time the instrument is run. Other maintenance is performed ‘periodically’ (e.g., roughly monthly, contingent upon sample throughput). Each instrument operator is responsible for the performance of their own instrument, and may perform maintenance duties at their discretion. For these reasons, ALSLG-FC’s culture is not one of ‘scheduled’ maintenance, in the traditional (calendar) sense. Consequently, although the Department Manager provides oversight, it is not necessary or practicable to create formal maintenance schedules, or to have maintenance performance synchronized within the Department.

ALSLG-FC maintains service contracts for most major analytical equipment, including gas and high-performance liquid chromatographs, mass spectrometers, liquid scintillation counters, and cold vapor atomic absorption and inductively coupled plasma spectrophotometers. Preventive maintenance is included in most of these service contracts. Service contracts that include preventive maintenance are also retained for ALSLG-FC balances and the DI water system.

8.2 SETTINGS

ALSLG-FC’s equipment list (Appendix G) depicts the following information: a) the identity and type (i.e., manufacturer and model number) of equipment (including its configuration) and its software; b) the equipment’s serial number or

other unique identification; c) the current location; d) the date received and date placed in service (if available); and e) the condition when it was received (e.g., new, used, reconditioned).

While it is true that some settings (e.g., detector wavelength) may be stipulated in reference methods, most instrument settings are not specifically prescribed, as they are instead, dictated by acceptable outcome (e.g., peak resolution, etc.). In a similar vein, ALSLG-FC provides typical instrument settings in the associated determinative SOP, but actual settings may vary contingent upon instrument performance and contributing factors, such as ambient conditions and operator subjectivity.

For the most part (i.e., not applicable to some types of equipment), instrument configuration and settings information is captured electronically by the instrument's 'method' files. Typically there is an 'acquisition' method file and a 'quantitation' method file that together, control the manner in which the data are obtained and subsequently calculated. These instrument files are archived via established laboratory electronic backup protocols (Form 159 – IS / LIMS Policy Statement), and are retrievable, thus providing for the reconstruction of data. The utilization of proper settings is evidenced by analytical data and QC results that meet performance criteria.

8.3 TRENDS

The dominant focus of trending contained in pertinent guidance documents relates to the generation of acceptable 'at on-set' and 'continuing' method QC checks. Concurrent with these requirements, ALSLG-FC's culture for trending observation labwide consists of ensuring that acceptable instrument checks are generated, and that the system is not producing any artifacts at levels of concern, prior to analyzing sample sets.

The expertise of the operator is a major component in effective equipment operation. Experienced operators develop an intuitive sense as to how their instrument is performing. Generally this sense is not based on a specific indicator, as there may be many contributing factors to that particular indicator, but rather on an accumulation of cues (similar to those factors that would be considered during the troubleshooting process). Because this type of expertise does not lend itself well to documentation, ALSLG-FC emphasizes cross-training to ensure consistent data generation, and the retention of 'corporate knowledge'.

8.4 EQUIPMENT DOCUMENTATION REQUIREMENTS

Analysts are responsible for maintaining calibration/verification and maintenance records of all instruments and equipment involved in the creation of the analytical data they generate. Considerations of maintenance, settings and trends, and their documentation, vary widely contingent upon the type of equipment, how automated it is, and the degree of sample throughput. Documentation can be accomplished by various means, electronically and via hardcopy. For example,

ICP, ICP/MS and CVAA routine maintenance is entered into the instrument's PC and printed out in the raw data header, while service contract maintenance and repair are documented in hardcover logbooks. Labwide, dedicated hardcover maintenance logbooks are assigned to each piece of major ALSLG-FC instrumentation, however, the manner in which equipment documentation is recorded, is at the discretion of the Department Manager. It is not ALSLG-FC's intent to unify or centralize maintenance information.

Although the manner of record keeping varies, in order to provide a clear and complete history of repairs and maintenance associated with the instrument, each entry may, but not limited to, include the following elements:

- the date of the maintenance or repair;
- the reason for the maintenance or repair (e.g., was this action taken to correct a problem or was this action routine instrument maintenance);
- a full description of the maintenance or repair conducted;
- the name of the analyst or vendor who performed the maintenance or repair;
- reference that it was verified that the equipment is operating properly before being placed back in service (SOP 317), and where this information can be found; and
- the initials of the analyst making the entry and date of entry.

Where applicable, the identity of the reference material used as an instrument check must also be recorded, and where applicable, a statement as to the calibration's expiration must also be made.

Details regarding equipment documentation are also provided in SOP 303. Note that maintenance logs are included in monthly logbook review.

Table 8.1 (Maintenance Snapshot) following provides a brief summary of laboratory equipment, an overview of associated maintenance performed, and comments regarding how associated maintenance documentation is accomplished.

8.5 CORRECTIVE ACTIONS, SPARE PARTS, CONTINGENCY PLAN

8.5.1 CORRECTIVE ACTIONS

Corrective measures for failed QC checks are given in the associated determinative SOP. General procedures for removing equipment from service and placing new or repaired equipment into service, are provided in SOP 317. Detail regarding corrective measures and repair for support equipment failures (e.g., ovens, cooling units, pipets, DI

water system), are discussed in SOPs 320, 326, 321 and 319, respectively. Actions to be taken in the event of catastrophic failure are discussed in Section 8.5.3 below.

ALSLG-FC maintains service contracts (preventive maintenance, repair) for most major analytical equipment. Some equipment (particularly some support equipment) does not lend itself to repair and would likely be replaced instead, per requirements given in SOP 127.

8.5.2 SPARE PARTS

An adequate inventory of spare parts is required to minimize equipment downtime. This inventory should include those parts and supplies that:

- are subject to frequent failure;
- have limited useful lifetimes, or
- cannot be obtained in a timely manner should failure occur.

Department Managers are responsible for maintaining an adequate inventory of necessary spare parts for all major instruments and equipment items. Examples of spare parts maintained for major instrumentation include: septa, inserts, columns, tube fittings, filaments, source parts, and traps.

8.5.3 CONTINGENCY PLAN

In the event of a catastrophic instrument failure, ALSLG-FC will make every effort to analyze samples within holding times by alternate means. If the redundancy in instrumentation is insufficient to handle the affected samples, then the Department Manager will notify the Project Manager immediately. In turn, the PM will notify the client to discuss options that will ensure successful completion of the project.

ALSLG-FC will also take appropriate mitigating steps and notify the client should significant power, cooling unit, etc. failures occur that create circumstances which could adversely impact the client's sample results. An automated system is in place to notify the IS Manager and Laboratory Director should a power outage of significant duration occur. However, any employee who notes an outage or unit failure is responsible for contacting the Department Manager or Laboratory Director, who will in turn direct the necessary actions. The specific course of action taken is dependent upon the nature and extent of the failure. General procedures to be followed in the event of catastrophic failure are provided as an appendix to ALSLG-FC's Emergency and Contingency Plan (ECP).

8.6 SUPPORT EQUIPMENT

ALS Laboratory Group, Fort Collins defines support equipment as all those devices which are not the primary determinative instrument defined by the analytical method, which support laboratory operations. Support equipment includes balances, ovens, refrigerators, freezers, water baths, temperature measurement devices, and mechanical (e.g., Eppendorf™ pipets. Per ALSLG-FC's definition, support equipment also includes: desiccators; centrifuges; vortex mixers; sonicators; homogenizers (including ball mills, riffle splitters and shatter boxes); pressure filters; vacuum pumps; zero headspace (ZHE) extractors; tumbling devices; platform shakers; water baths; chillers; heating blocks, mantles, hot and stir plates; evaporators; muffle furnaces; kilns and cleanup apparatus.

Additionally, ALSLG-FC's deionized (DI) water systems (SOP 319) and health physics equipment (Appendix G) and are also considered to be support equipment.

Requirements pertaining to glassware are given in SOPs 334 and 720. Procedures for maintaining computers and other electronic devices (e.g., printers, backup devices, etc.) are developed, implemented and maintained by the IS Department (Form 159, et. al.)

Support equipment must be calibrated or verified, typically annually, within the applied range of use. NIST-traceable references must be used when available, and the results of the calibration/verification must be documented and within the specifications required of the application for which the equipment is intended.

All support equipment must be maintained in proper working order, and records must be retained to document the equipment's performance, maintenance, and repair. *Each business day, near to the beginning of the work shift, the proper functioning and calibration of the following equipment must be verified: balances, ovens, refrigerators, freezers.* Water bath temperatures must be verified each day of use. Additional monitoring must also be performed and documented, if so prescribed by a test method (e.g., recording the temperature of a water bath during digestion).

Per **SOP 321**, the volumes dispensed from mechanical pipets are verified prior to each use, as these volumes are critical measurements. Because automatic dispensing devices used to deliver solvents or reagents (e.g., for sample preservation and extractions) are not used to deliver critical volumes, these devices are exempt from daily verification.

Where necessary, in-house verifications are performed to document the capability of graduated laboratory glassware (e.g., records are on file in the Quality Assurance Department that document the capacity of the cyanide Midi-Dist sample tube glassware).

Certificates of Accuracy are acquired from the manufacturer and are retained on file within each Department for glass microliter syringes.

The following SOPs provide additional information about calibration and verification of support equipment:

- **SOP 305** -- balance calibration and verification
- **SOP 320** -- monitoring and recording of oven temperatures
- **SOP 326** -- monitoring refrigerator and freezer temperatures.

9. **QUALITY CONTROL PROCEDURES**

ALS Laboratory Group, Fort Collins' quality control program provides a systematic process that enables the laboratory to evaluate and control the validity of analytical results, by measuring and monitoring accuracy and precision by method and matrix; by developing control limits and using these limits to detect errors or out-of-control events; and by requiring corrective actions to prevent or minimize the recurrence of these events. ALSLG-FC observes QC procedures to ensure that sample data meet laboratory and client quality objectives.

The purpose of preparing and analyzing QC samples is to demonstrate accuracy and precision of the sample data and efficacy of the method for the target analytes being investigated. Acceptance criteria may be dictated by reference methods or by project requirements. All assessments of QC data are performed after all rounding and significant figure truncations have been performed.

For all analyses performed by ALSLG-FC, the QC concepts and samples described in the following sections are mandatory. Determinative SOPs contain a Table that summarizes the types and frequency of QC samples, acceptance criteria, and corrective actions required. Observation of maximum holding time allowance is discussed in LQAP Chapter 4.

9.1 **DEFINITION OF BATCH**

9.1.1 **PREPARATION BATCH**

A preparation batch consists of as many as 20 field samples of the same or similar matrix, that are prepared together by the same analyst(s) within a limited or continuous time period, following the same method, and using the same kind of equipment and same lots of reagents. Each batch must contain the appropriate number and kind of method control

Table 8.1 Maintenance Snapshot

<p>Extractions - GPC (Gel Permeation Cleanup) Apparatus Column flow 5.0 (+/-0.1)mL/min (stipulated in SOP) SOP 641. Maintenance discussed sufficiently in SOP (adequate MeCl2 supply, proper flow rate; no leaks, all connections tight). Maintaining hardcover log.</p>	<p>Extractions - Ignitability Apparatus SOP 629. Operation and checks discussed sufficiently in SOP (Procedures). If not functioning properly, most likely replace. No maintenance log required.</p>
<p>Extractions Support - Kilns Desired setting given in SOP. Unit either works or doesn't. Vendor repair possible contingent upon problem, most likely replaced. No maintenance log required.</p>	<p>Extractions Support - Recirculating Chillers Setting = sufficient water flow, stated in SOP. Either works or doesn't. Vendor repair possible contingent upon problem, most likely replaced. No maintenance log required.</p>
<p>Extractions Support - Lunar Lander Pressure Filter SOP 608, 609, 666. No special considerations, either works or doesn't. Most likely replace rather than repair. No maintenance log required.</p>	<p>Extractions Support - Rotary Tumbler SOPs 603, 608, 609, 666, 668. Desired setting given in SOP. Device either works or doesn't. Vendor repair contingent upon problem (replaced). No maintenance log required.</p>
<p>Extractions Support - Mixer, Homogenizer No special considerations, either works or doesn't. Most likely replace rather than repair. No maintenance log required.</p>	<p>Extractions Support - Sonicators (handheld) (bath, hand-held) - SOPs 665, 673. Temperature requirement and recording directives given in SOPs, as applicable. Specific setting does not need documented (aside from temperature which is evidenced by acceptable reading, setting not critical). Maintenance log not required, unit either works or doesn't (replaced).</p>
<p>Extractions Support - Nitrogen Evaporator SOPs 637, 665. Desired setting given in SOP. Unit either works or doesn't. Vendor repair not likely, replaced. No maintenance log required.</p>	<p>Extractions Support - Steam Generator & Evaporator SOPs 607, 672. Desired setting given in SOP (i.e., valve open & flowing). Unit either works or doesn't. Vendor repair not likely, replaced. No maintenance log required.</p>
<p>Extractions Support - RapidVap Concentrator Various SOPs. Unit either works or doesn't. Vendor repair not likely, replaced. No maintenance log required.</p>	<p>Extractions Support - Zero Headspace Extractors SOPs 608, 669. Detailed operation/maintenance contained in SOP. Benchsheet attestation.</p>
<p>Metals - ICP Analyzer, Autosampler, includes ICP/MS SOPs 807, 827, 834. Maintenance tasks are discussed sufficiently in the SOPs (check gas pressures and supply; for ICP/MS, verify that cooling water for instrument is flowing; check filters on rear of instrument and vacuum monthly; check pump tubing (replace when necessary); check drainage bottle (empty if necessary). Routine maintenance is documented via run data headers, which are files maintained on the instrument PC. Settings are included in calibration report printout. Hardcover maintenance logs are being kept for repair documentation.</p>	<p>Metals - Mercury Analyzer (CVAA) SOP 812; does not presently contain maintenance text in Procedures Section, SOP will be revised accordingly per established publication schedule (make sure lamp life is adequate; clean window, replace Nafion cartridge as necessary; check adequate reagent supply; replace tubing, tighten fittings as necessary). Routine maintenance is documented via run data header, which is an e-file maintained on the instrument PC. Settings are included in calibration report printout. Hardcover maintenance log is being kept for repair documentation.</p>
<p>Organics - Gas Chromatograph, (Autosampler), Purge & Trap (includes sample heaters); all detectors (including MS) GC SOPs: 402, 406, 407, 408, 424, 425, 434, 438, 444 GC/MS SOPs: 506, 525. ECDs (SOP 016 for leak/wipe tests), FPD (detector considerations), FIDs (periodic cleaning, jet replacement - indicated by instrument performance), PIDs (adequate lamp, clean window), others?. Instrument injection port and seal maintenance (liner cleaning/replacement, septum replacement, etc.); front portion of column clipped off, splitter cleaned, where applicable; check adequate gas flow and reserves (columns & detectors); replace traps (including Vocab) as needed; use of column and trap bake cycles; tubing clean; fittings tight. Autosampler maintenance essentially means operate with adequate flushes. Maintaining maintenance logs/headers.</p>	<p>Organics - TOC Analyzer, Autosampler SOP 670. Routine maintenance discussed as Section 8.1 (check adequate gas pressure and supply; make sure 8-port valve connections are tight; check adequate reagent supply; check halogen scrubber and gas/liquid separator; check mist trap and drain if necessary). Maintaining hardcover log.</p>

Table 8.1 Maintenance Snapshot

<p>Organics - HPLC & Components, includes LC/MS-MS SOPs 404, 408, 439, 446, 447, 336 Maintenance discussed sufficiently in SOPs (check that eluent supply and pressure are adequate; replace column frits/guard columns/columns as necessary; all fittings tight; empty and refill HPLC water supply; replace detector lamp as necessary). Maintaining hardcover log.</p>	
<p>RAD - Alpha Spectrometer (towers & octetes) Operationally discussed sufficiently in SOP. Certain practices, like detector segregation (U/Th vs Am/Pu) could be considered preventive maintenance; also, use of the 'thin film' method for Polonium. Daily vacuum pressure check; weekly cleaning. Periodic maintenance provided for in established instrument training modules. Detectors are placed 'off-line' rather than repaired (either cleaned or changed out). Some repair can be done in-house (replace octet tubing, check connections), otherwise via vendor. Hardcover maintenance log is being kept. Detectors are tracked so that trend information can be ferreted out if needed.</p>	<p>RAD - Liquid Scintillation Counters SOP 704. Operationally discussed sufficiently in SOP. LSC operation is more about being optically clean. Instrument is checked annually (alignment, etc.) under service contract. In-house repair is really not applicable. Hardcover maintenance log is being kept.</p>
<p>RAD - Gamma Spectrometers SOP 713. Operationally discussed sufficiently in SOP. Check N2 tank and fill as necessary; hose and fittings checks; clean detectors, shields weekly. Periodic maintenance provided for in established instrument training modules. Some repair can be done in-house, otherwise maintenance is provided under contract. Hardcover log being maintained.</p>	<p>RAD - Scaler w/ Lucas Cell Counter Operationally discussed sufficiently in SOP, although a lot of expertise is involved and that expertise/knowledge is primarily obtained by demonstration, not via an operator's manual (each use, make sure cell and tubing are clean and that fittings are tight). Cells are good for 20 runs, then CCV checked. If not acceptable, run CCV again. If still not acceptable, take cell out-of-service per SOP 317. Photomultiplier is cleaned monthly. Plateau is verified annually. Periodic maintenance provided for in established instrument training modules. Hardcover log is being maintained.</p>
<p>RAD - Gas Flow Proportional Counter (Counting Room, PreScreen) SOP 724. Operationally discussed sufficiently in SOP (daily tank pressure check and flow to instrument). Periodic puck maintenance; planchet holder and slides cleaning also performed; provided for in established instrument training modules. Detectors are put 'off-line' rather than repaired. A drawer evaluation is conducted annually. Paragon is considering putting the GFPCs under a maintenance contract. A hardcover log is being maintained.</p>	
<p>RAD Support - Ball Mill SOP 336. Operation/maintenance defined sufficiently in SOP. Device either works or doesn't. Since performance doesn't vary, no maintenance log required.</p>	<p>RAD Support - Riffle Splitter SOP 336. Operation/maintenance defined sufficiently in SOP. Device either works or doesn't. Since performance doesn't vary, no maintenance log required.</p>
<p>RAD Support - Platform Shakers SOP 336. Operation/maintenance defined sufficiently in SOP. Device either works or doesn't. Since performance doesn't vary, no maintenance log required.</p>	<p>RAD Support - Shatterbox SOP 336. Operation/maintenance defined sufficiently in SOP. Device either works or doesn't. Since performance doesn't vary, no maintenance log required.</p>
<p>Support - Balances SOP 305. Vendor serviced (cleaned, certified) annually (records maintained by QA Dept.). Daily use checks (e.g., level, pan/chamber clean, etc.) and calibration verification defined in SOP (performed by Department). Logbooks (Form 301) that contain acceptance limits and corrective action directives are kept & subject to monthly review.</p>	<p>Support - Ovens & Muffle Furnaces SOP 320. Assigned verified thermometers (muffle furnaces rough-checked with thermistor). Daily performance check defined in SOP, performed by Department. Logbooks (Form 312) that contain acceptance limits and corrective action directives kept & subject to monthly review. Problems usually thermometer-related (managed by QA Dept.), when necessary, appliance vendor contacted for servicing (records maintained by Facilities or QA). Departmental staff make minor setting adjustments when necessary (documentation of setting not required, proper setting evidenced by acceptable readings). 'Return to Service' actions defined in SOP, recorded in logbook.</p>

Table 8.1 Maintenance Snapshot

Support - Centrifuges Desired setting given in SOP, but performance not critical. Unit either works or doesn't (vendor repaired if possible, replaced).	Support - Thermometers (glass, electronic, IR) - SOP 923. Annual verification and records managed by QA Dept. Directives to notify QA if malfunction given in SOPs 320, 326, 210. Corrective measures, return to service discussed in SOP 923.
Support - Cooling Units (Refrigerators, Freezers) SOP 326. Assigned verified thermometers. Continuous electronic monitoring discussed in SOP. Logbooks (Form 347) that contain acceptance limits and corrective action directives kept Departmentally & subject to monthly review. Problems usually thermometer-related (managed by QA Dept.), or defrost needed (managed by Dept.). When necessary, appliance vendor contacted for servicing (records maintained by Facilities or QA). Departmental staff make minor setting adjustments when necessary (documentation of setting not required, proper setting evidenced by acceptable readings). 'Return to Service' actions defined in SOP, recorded in logbook	Support - Vacuum Pumps (instrument, independent) - Organic, Inorganic, RAD. Those affiliated with instruments are included in instrument maintenance practices/documentation. Potential oil change for stand-alones. Managed by Facilities. Not capital equipment. No maintenance log required.
Support - Dessicators Tight seals on unit, for indicating Drierite™, replace when pink (can be 'recharged'). SOP directives are sufficient.	Support - Vortex Mixers No real setting, technique driven (training). Device either works or doesn't. Vendor repair not likely, replaced. No maintenance log required.
Support - Heating Mantles / Hot Plates / Stir Plates Desired setting given in SOP, not critical, so long as monitored temperature can be sustained. Unit either works or doesn't. Vendor repair unlikely, replaced. No maintenance log required.	Support - Water/Sonic Baths Complete unit, or device on top of hot plates. Temperature requirement and recording directives given in SOPs. Specific setting does not need documented (evidenced by acceptable temperature). Should not leak. Maintenance log not required, unit either works or doesn't (vendor repaired if possible, replaced).
Support - Millipore Water System SOP 319. Monitoring (Departmental), acceptance criteria, corrective actions discussed in SOP. Context with larger resin units also discussed. Logbooks (Form 712) subject to monthly review maintained. Independent monthly check also discussed. If needed, unit repair may be attempted in-house, most likely unit would be replaced (discussed in SOP); SOP 127 requirements. Documentation of setting not required, proper setting evidenced by acceptable readings. 'Return to Service' criteria = acceptable readings must be maintained.	
WetChem / Metals - pH Meter / ISEs / Conductivity Meter SOP 1126, 1128, several others. Care & feeding of probes is adequately addressed as Procedures in SOPs. Probe/instrument manuals are listed in the SOPs Reference Section.	WetChem - Ion Chromatograph, Autosampler SOPs 1113, 1125. Maintenance considerations need to be added to the beginning of SOP Section 9 (check that eluent supply and pressure are adequate; replace column frits/guard columns/columns as necessary; check that gas supply and pressure are adequate; all fittings tight; empty waste container as necessary). Consider adding detector maintenance detail as well. Settings are included in calibration report printout. Instrument is not networked, still subject to IS backup protocols. Hardcover repair log is being maintained.
WetChem - Cyanide Distillation Apparatus SOP 1110. Necessary detail already in SOP.	WetChem - UV Spectrophotometer Various SOPs. Performance mostly involves optically clean and matched cuvettes. Sufficient operational maintenance detail is given in the SOPs. Hardcover maintenance log kept.
WetChem - Flow Injection Analyzer SOPs 1127, 1129, 1123. Sufficient operational detail in Sections 11-14 of SOP (adequate reagent supply; clean, unclogged tubing; fittings tight). Software methods stored in instrument PC, subject to IS back-up protocols (no printout). Hardcover repair log is maintained.	

samples (e.g., MB, LCS) and matrix-specific QC samples (e.g., MS/MSD, DUP). Cleanup procedures may be included as part of the preparation batch. All field and QC samples in the batch should be subjected to the same preparation and cleanup procedures.

9.1.2 ANALYSIS BATCH

The analysis batch (or sequence) consists of samples that are analyzed together within the same or continuous time period, on the same instrument, and processed using the same calibration. Each analysis sequence must contain the appropriate number and kind of standards and samples as defined by the method. If samples from a preparation batch are analyzed in multiple analysis batches, extended method control and matrix-specific QC samples need not be analyzed with every analysis batch.

Where no sample pre-treatment (such as extraction or digestion) is required prior to analysis (e.g., analysis of volatile organic compounds, anions analysis by ion chromatography, etc.), the preparation batch and analysis sequence are equivalent.

9.2 PREPARATION BATCH QC SAMPLES AND STANDARDS – DEFINITION AND USE

The results of quality control samples provide an estimate of accuracy and precision for the preparation and analysis steps of sample handling. The following sections describe the QC information provided by each of these analytical measurements.

9.2.1 METHOD BLANK

A method blank (MB) consists of an aliquot of well-characterized, controlled, or certified matrix (e.g., reagent water, Ottawa sand, solid reference material, boiling chips) that is processed through the entire sample preparation, cleanup, and analysis procedure. For radiochemical analyses, a suitable blank solid matrix has not been identified; therefore, reagent water is routinely used for the blank for most solid matrices. The volume or weight of the blank must be approximately equal to the sample volume or weight processed for sample analyses.

The purpose of the MB is to demonstrate that interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware, are known and minimized. A method blank should not contain target analytes at or above the reporting limit, unless otherwise permitted in the method. Other maximum blank contamination control criteria may apply, as indicated in the associated LIMS program specification.

While some methods may require background correction, sample results are typically not corrected for blank contamination.

9.2.2 **LABORATORY CONTROL SAMPLE**

A Laboratory Control Sample (LCS) consists of an aliquot of well-characterized, controlled, certified matrix (e.g., reagent water, sand, solid reference material, Teflon™ chips) that is spiked with analytes of interest and processed through the sample preparation, cleanup, and analysis procedure.

The purpose of the LCS is to provide an estimate of bias based on recovery of the compounds from the clean, controlled matrix, and to demonstrate that the laboratory is performing the method within accepted guidelines without potential non-matrix interferences.

Where sample pretreatment is not required, such as with ion chromatography or gamma spectroscopy analysis, or the analysis of volatile organic compounds, the ICV standard or other appropriate control standard may be employed as the LCS.

An LCS for methods with extensive lists of analytes that may interfere with one another may include a limited number of analytes, but the analytes included must be representative of as many analytes as is practical.

Other client-specific QC requirements may be prescribed in the applicable LIMS program specification. The requirements set forth in the LIMS program specification supercede those stated in the method, SOP or LQAP.

9.2.3 **MATRIX SPIKE/MATRIX SPIKE DUPLICATE**

A matrix spike (MS) or matrix spike duplicate (MSD) is a field sample to which known concentrations of target analytes are added before the sample is processed. The purpose of MS/MSD samples is to assess the performance of the method for a particular matrix and to provide information about the sample's homogeneity. Results of the MS/MSD samples are evaluated in relation to the method QC samples to determine the effect of the matrix in regards to accuracy and precision. Sample results are not corrected for MS/MSD excursions.

To generate MS/MSD pairs for any analysis, there must be an adequate volume/weight of field sample available. Inadequate sample volumes preclude the possibility of generating this pair of QC samples. ALSLG-FC asks clients to designate the sample to be used for MS/MSD analysis to ensure that adequate sample volumes are collected.

For some analyses, changing the composition of the sample in any way invalidates the analysis to be performed (e.g., hardness, alkalinity, pH). Therefore, an MS/MSD pair cannot be generated for these analyses. Normally, duplicate sample aliquots are analyzed in order to generate an estimate of the method's precision.

Other client-specific quality control requirements may be prescribed in the applicable LIMS program specification. The requirements set forth in the LIMS program specification supercede those stated in the method, SOP or LQAP.

9.2.4 SAMPLE DUPLICATE

A sample duplicate (DUP) is a second representative portion of sample that is carried through the preparation, cleanup and analysis process. Results for the duplicate sample are compared to the initial sample analysis results as a means of evaluating precision. For organic analyses, the MS/MSDs fulfill this function. The degree of sample homogeneity directly impacts the integrity of the sample duplicate analysis.

Precision criteria for sample duplicate analyses are those prescribed in the reference method and/or SOP, unless otherwise superceded by client-specific requirements contained in the applicable LIMS program specification.

9.2.5 SURROGATES

Surrogates are organic compounds that are similar to the target analytes, but are unlikely to be present in actual field samples. They are introduced into all field and QC samples in a batch prior to sample preparation, and provide an estimate of bias based on recovery of similar compounds, for a given extraction technique and analysis method combination. Sample results are not corrected for surrogate recoveries.

Acceptance criteria for surrogates are those prescribed in the reference method and/or SOP, unless otherwise superceded by client-specific requirements contained in the applicable LIMS program specification.

9.2.6 CHEMICAL YIELD MONITORS OR ISOTOPIC TRACERS

Chemical yield monitors are used in radiochemical analyses and provide information similar to the surrogate spikes discussed above. The primary difference between a chemical yield monitor and a surrogate is that sample results are corrected for chemical yield recoveries and not corrected for surrogate recoveries. A chemical yield monitor is a substance that has similar chemical characteristics as the parameter being measured. It is introduced into all field and QC samples in a batch during the preparation procedure. Chemical yield

monitors provide information regarding the performance of a method on a sample-by-sample basis.

Chemical yield monitors are evaluated against established laboratory control limits. These ALSLG-FC default control limits may be superceded by other quality control criteria specified in the applicable LIMS program specification.

9.3 CONTROL CHARTS

Control charts are a tool that can assist the laboratory in evaluating process control and trends. Control charts are used as a visual queue giving warning before a measurement system drifts into an out-of-control situation. Information such as radiochemical calibration parameters, results of daily efficiency checks, etc. can be documented in control charts. Accuracy control charts, discussed further below, that contain method LCS (and surrogate, as applicable) performance information, are managed through LIMS. Although the QAM is responsible to annually review LCS information, and determine if a significant change to a method or process has occurred. The QAM then notifies technical management if the mean and standard deviation of LCS data show significant change (>10%). QC limits can be updated after review by technical personnel as appropriate. LCS information is accessible to *all* bench personnel **for their consideration**, through LIMS.

Further discussions of control charts and control limits and other considerations such as outlier rejection and trend evaluation follow below.

9.3.1 ACCURACY CONTROL CHARTS

Accuracy (recovery) for a batch can be evaluated by plotting the individual percent recovery points for analytes on a control chart and comparing the values against the current control limits. If the spike recovery values for the current analytical batch meets the acceptance criteria for that method, then the data point (and batch) are accepted. **If not, and re-preparation/analysis is possible, the batch is generally reprocessed. At minimum, the failure(s) is considered a non conformance and is narrated in the laboratory data package. See the QC Table of each determinative SOP for further details as to the appropriate corrective actions to be taken for controlled failures.**

Accuracy control charts are generally maintained for each method that utilizes an LCS. For methods that cannot use LCS samples (e.g., pH, flashpoint, conductivity), other tools, **such as periodic participation in 3rd party Performance Test sample analysis**, are used to assess method control.

If fewer than 20 data points for a method, matrix, and analyte combination are acquired, then control charts yield scant information.

9.3.2 CONTROL LIMITS

Control limits for each controlled analyte are calculated, and can be updated, using ALSLG-FC's LIMS. The recovery values from all data processed within a specified date range, are used to calculate the control limits and compile the control chart. **Standard outlier tests, based on the population number evaluated (e.g., Dixon $n \leq 20$; Grubbs $n = 3-147$; etc.), per their restrictions/requirements, may be applied.**

The upper and lower control limits of the control chart are designated as the value equal to the average recovery plus or minus three times the standard deviation (i.e., 99% confidence interval).

The upper and lower warning limits for the control chart are designated as the value equal to the average recovery plus or minus two times the standard deviation (i.e., 95% confidence interval).

The average recovery, standard deviation, minimum value, maximum value, and population are displayed on each control chart.

Control limits are updated as needed (e.g., acquisition of a sufficient number of data points to establish meaningful control limits for a newly implemented method; if deemed appropriate as a result of a corrective action investigation; etc.). The frequency with which control limits are updated may vary for different methods. Generally, intra-laboratory historical control limits are not updated more than once per year.

9.3.3 OUTLIER REJECTION

For the generation of control charts, and other quality control data that monitor the laboratory's performance, it is essential to prevent spurious or erroneous data from being incorporated. It may be necessary to reject data as an outlier to prevent an adverse effect on the values being calculated. **Only established statistical approaches may be used, such as application of the Grubbs, Dixon, etc., tests, to identify and handle outliers. Any data point meeting established outlier criteria is justified to be rejected, however, the analyst has the discretion to reaccept the data point where it is technically sound to do so.** In every case, the cause of the outlier rejection must be clearly understood before any data point is **manually** rejected.

For the purposes of statistically determining whether a data point is an outlier or not, ALSLG-FC may use the procedures discussed in the Dixon Rank Sum Test, the Grubbs Test, **or other established appropriate statistical treatment.** If a data point is determined to be

an outlier, it **generally** will not be incorporated into the dataset when updating QC limits.

See SOP 329 for further details regarding the processing of MDL studies and evaluation of outliers.

9.3.4 TREND EVALUATION

Trend analysis techniques can be applied to control charts as a preventive tool to help indicate conditions that could cause an analysis to become out of control. In evaluating control charts, a trend is recognized if one or more of the following situations exist:

- A series of seven successive points occur on the same side of the mean;
- A series of five successive points occur going in the same direction;
- Two consecutive points occur between the warning and control limits;
- A single value occurs outside of control limits.

Actions may be employed for trends identified. Items which might be considered but not limited to include:

- Has there been a change in instrumentation or personnel?
- Has instrument maintenance been properly performed?
- What conditions have changed since the trend began?
- Have standard or spike solutions changed?

9.4 SECOND COLUMN OR SECOND DETECTOR CONFIRMATION

Second column or detector confirmation is performed for several GC and HPLC methods. Whenever two dissimilar chromatography columns or two detectors of a different nature are available for a given method, the laboratory performs second column or second detector confirmation analysis to confirm the identity of target analytes in field samples. When second column analysis is performed for any chromatography technique, the following policies apply:

- Every attempt will be made to calibrate the second (confirmatory) column in the same manner as the quantitative (primary) column. The same initial and continuing calibration standards will be analyzed on the confirmation column in the same manner as the quantitation column. The purpose of this dual calibration requirement is to allow the

possibility of reporting quantitative results from the confirmation column if interferences on the primary column prevent accurate target analyte quantitation.

- For chromatographic techniques, the determination of target analytes in a sample depends solely on peak retention times observed in both primary and secondary column chromatograms. If target analyte peaks are present at the proper retention times in both confirmation and quantitation column chromatograms at levels above the MDL, then ALSLG-FC considers this analyte to be confirmed.
- In general, ALSLG-FC reports the higher value of the two columns per SW8000C guidance (e.g., 8011, 8081, 8082, 8141, 8151, 8021). It is also ALSLG-FC's policy to report the higher value of the two columns for other EPA methods (e.g., 608, 615).

If no interferences are present, and an analyte's value from either the primary or secondary column is greater than the reporting limit but between the MDL and the reporting limit on the other column, then ALSLG-FC reports the higher value that is greater than the reporting limit for that analyte.

- ALSLG-FC customarily reports the value from the primary column for methods SW8330 and SW8332. Co-elutions or interferences are frequently observed on the secondary column for these HPLC methods.
- Other reporting rules may apply as dictated in the applicable LIMS program specification. The rules of the LIMS program specification supercede standard ALSLG-FC policy.

9.5 **MANUAL RE-INTEGRATION POLICIES AND PROCEDURES**

Many data collection systems allow the analyst to reprocess data, thereby allowing for the manual re-integration of analyte peaks. ALSLG-FC makes every attempt to optimize peak integration parameters; however, manual reprocessing of data must be performed to correct a data system's integration error (e.g., incorrect or missed peak assignment, over- or under-integration of area). Manual re-integrations may not be performed solely to meet initial or continuing calibration criteria or any QC criteria (e.g., tuning, or surrogate or spiking compound recovery).

Whenever a manual integration is performed, the analyst performing this process must include a hardcopy of the original and re-integrated peak in the final data report. In addition, the analyst must initial and date the re-integrated page and document the reason for re-integration on the printout. The re-integration must be documented in the case narrative.

Further details regarding manual integration procedures are given in **SOP 939**.

10. DATA REDUCTION, VALIDATION AND REPORTING

Data transfer and reduction are essential functions in summarizing information to support conclusions. It is essential that these processes are performed accurately and are followed by multiple reviews before data are submitted to the client. All analytical data generated by ALSLG-FC are extensively reviewed for accuracy and completeness. The data validation process consists of data generation, reduction, and multiple levels of review, as described below.

10.1 DOCUMENTATION OF RAW DATA

Where possible, raw data are captured and processed electronically using verified software programs (see **SOPs 709 and 1400** for further information regarding software verification).

To facilitate manual documentation of raw data (where suitable LIMS benchsheet interfaces do not yet exist), ALSLG-FC creates custom logbooks comprised of forms or benchsheets that are tailored to contain the information required to adequately document the process being performed, and the associated data. The Quality Assurance Department controls these forms and benchsheets, and issues bound and paginated logbooks to the laboratory as needed via controlled distribution.

As applicable, hardcover, bound laboratory notebooks (most frequently used for instrument maintenance logs or Project Manager notebooks) are also issued via controlled distribution to laboratory staff as needed.

The manually recorded raw data are entered into the laboratory logbook directly, promptly, and legibly in indelible ink. All raw data entries must, at a minimum, contain the following information:

- the initials of the individual who performed the process;
- the date the process was performed;
- the methodology used; and
- the identity of all samples or standard solutions that were employed in carrying out the process.

Raw data must be maintained as part of the laboratory's records. Raw data not only includes instrument outputs, but sample preparation, standard materials documentation, and equipment maintenance information as well. Raw data may be archived electronically or as hardcopy.

10.2 CORRECTION OF ERRORS IN DOCUMENTS

During the course of processing and reviewing sample preparations and analysis results, it may be necessary to correct documentation errors. Detailed requirements for the correction of manual documentation errors are prescribed in

SOP 303; the correction of electronic information is governed by LIMS controls and audit trails. In summary, manual entries may not be obliterated by erasure, use of correction fluid, or other means. In order to maintain the integrity of the documentation generated by the laboratory, changes to hardcopy documentation must be made in the following manner:

- A single line must be struck through the error so that the original text remains legible;
- As applicable, a corrected entry must be made adjacent to the error; and
- The person making the change must initial and date the corrective entry.

If not clearly evident, the reason for the data change must be indicated.

10.3 DATA REDUCTION

ALS Laboratory Group, Fort Collins analysts perform data reduction. This process consists of interpreting instrument results and verifying calculated concentrations in samples from the raw data. The complexity of the data reduction is dependent on the specific analytical method and the number of discrete operations involved in obtaining a measurement (e.g., digestions, dilutions, cleanups, concentrations). The analyst calculates the final reportable values from raw data or enters all necessary raw data into the LIMS so that the LIMS can calculate the final reportable values.

Data are reduced according to protocols described in SOPs and method-specific review checklists. Computer software used for data reduction is validated before use and verified regularly by manual calculations. All information used in calculation is recorded in order to facilitate reconstruction of the final results (e.g., raw data, calibration files, tuning records, results of standard additions, interference check results, sample response, and blank or background-correction protocols). Information about the preparation of the samples is maintained in order to facilitate reconstruction of the final results (e.g., weight or volume, percent moisture for solids, extract volume, dilution factor).

Copies of all raw data and the calculations used to generate the final results, as recorded in hardbound laboratory notebooks, spreadsheets, electronic data files and LIMS record files, are retained in the project file to allow reconstruction of the data reduction process.

10.4 REPORTING OF SAMPLE RESULTS

Sample results are reported either on an “as-received” basis, or in units of dry-weight measure. The number of significant figures reported is consistent with the limits of uncertainty inherent to the analytical method. In most cases, results are reported to no more than two or three significant figures. Analytical problems, and/or any modifications of referenced methods are noted in the data package case narrative.

Standard units appropriate to the analytical method are used to report all sample results. Measurements for radiochemical analyses are reported in units of activity such as:

- picocuries per liter (pCi/L), aqueous; or picocuries per gram (pCi/g), solid matrix samples.
- disintegrations per minute per liter (dpm/L) or disintegrations per minute per gram (dpm/g).
- Becquerels per liter (Bq/L) or Becquerels per gram (Bq/g).

It should be noted that one (1) Curie is equal to 2.22×10^{12} dpm; and is also equal to 3.7×10^{10} Bq.

Standard units for inorganic and organic analyses are units of mass per volume (aqueous samples), or mass per weight (solid matrix samples). For example, Wet Chemistry parameters such as hardness, total organic carbon (TOC), etc., are typically reported in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg). Metals results for liquid samples may be reported as mg/L or as micrograms per liter ($\mu\text{g/L}$). Some methods have specific reporting units mandated by their analysis technique. For example, pH is reported as pH units, and specific conductance is reported as milli-Siemens (mmho/cm) or micro-Siemens ($\mu\text{mho/cm}$).

10.5 DATA REVIEW

ALSLG-FC employs multiple levels of data review. All data generated and reduced follow review protocols specified in laboratory SOPs (such as **SOPs 052** and **715**), and method-specific checklists. The preparatory technician and analyst who generates the analytical data perform a **Level 1** review of the data for correctness and completeness. This data review verifies that:

- the appropriate SOPs have been followed;
- any special sample preparation or analytical requirements that were communicated to the laboratory via the LIMS program specification have been met;
- all sample preparation information is correct and complete;
- all analysis information is correct and complete;
- QC samples meet criteria for frequency, accuracy and precision;
- all calculations, conversions, and data transfers are accurate;

- all documentation is present and complete, including benchsheets and/or run logs, any applicable NCRs, and documentation and presentation of manual integrations per SOP 939, as applicable.

Procedures for handling unacceptable data are discussed subsequently (LQAP Section 10.6).

Following completion of the Level 1 Review, the analyst then forwards the data to the Department Manager or another qualified reviewer whose function is to provide an independent **Level 2** review of the data. In addition to the elements evaluated in the Level 1 review described above, the Level 2 reviewer verifies that:

- the calibration data are scientifically sound, appropriate to the method, and completely documented;
- qualitative identification of target analytes is correct;
- quantitative results are correct.

The Level 2 reviewer selects a sample and verifies it to the benchsheet. If no errors are found, then the review is considered complete. If any problems are discovered, then additional samples are verified to the benchsheet with the process continuing until no additional errors are found or until the data package has been reviewed in its entirety. The Level 2 review is documented by recording the date and initials of the reviewer on the checklist employed. This sign-off signifies that the data are approved for release and a final report is prepared.

Once the final report is prepared, an additional overall technical review is performed before it is routed to the Project Manager for a **Level 3** review. The intent of this review is to verify that the report is complete and that the data meet the overall objectives of the project.

Each step of the review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the analysis and/or review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data produced are consistently of known, documented, and appropriate quality.

10.6 PROCEDURES FOR HANDLING UNACCEPTABLE DATA

All QC information is recorded in the same format, with the same units, as that of the associated sample results. It is the analyst's responsibility to evaluate QC data against applicable prescribed limits. When an analysis of a QC sample (e.g., MB, LCS, CCV, etc.), indicates that the associated samples do not meet requirements, the analyst must immediately notify the Department Manager. The Department Manager then consults with the PM (and QAM, as applicable) to determine whether or not the affected samples must be re-prepped and/or re-analyzed, and/or

if specific corrective action needs to be taken before additional analysis may proceed. A Nonconformance Report (NCR) as discussed in Chapter 11 of this LQAP, is initiated per **SOP 928**, as applicable. If the non-compliant data cannot be corrected, then the affected results must be flagged as discussed below, and the discrepancy disclosed in the data package case narrative. The completed NCR Form is included in the data report.

10.7 DATA REPORTING

Data reports contain final sample results, the methods of analysis used and limits of detection, and QC data. The extent of supportive data included (e.g., benchsheets, run logs, calibration data, instrument raw data printouts, etc.), is contingent upon the type of report contracted by the client.

Results of subcontracted data are clearly indicated as subcontract laboratory results when incorporated into the final data package report.

10.7.1 FACSIMILE OR IMAGED REPORTS

For projects that require rapid turnaround of sample analysis results, the laboratory may provide a facsimile or imaged e-mail attachment to the client, followed by the full data report at a later date. If the analysis results provided by facsimile or imaged e-mail attachment have undergone the same review processes followed for final data packages, then this forwarded report indicates that the sample analysis results are final. However, if the accelerated turnaround time requirements preclude a full review/validation of the sample data, then the report is marked as “PRELIMINARY” to indicate that results may change as the review process is completed.

10.7.2 HARDCOPY DATA PACKAGES

The format and content of a data report is dependent upon project specifications, and it is beyond the scope of this document to describe project-specific report requirements. In the absence of client-specified data package deliverables, the following sections describe the items that must be included in all data reports.

10.7.2.1 COVER LETTER

Items contained in the cover letter include:

- the client’s name and address;
- ALSLG-FC’s name and address, name of contact and telephone number;
- a tabular presentation of field/client sample ID, ALSLG-FC Sample ID, date received, matrix, and date collected. This item is typically presented as an attachment, the Sample Cross Reference Table;

- a list of each analysis performed and total number of pages for each analytical report;
- identification of all test data provided by a subcontract laboratory;
- a discussion of previously submitted or partial reports that pertain to the samples discussed in the current report; and
- the signature of ALSLG-FC's Project Manager or designee.

10.7.2.2 REPORT FORMAT

Analysis reports are presented in tabular format, and consistent significant figures and units of measurement are used. The following information is included in each report:

- laboratory name, client name, project name and/or number;
- client/field sample ID and ALSLG-FC sample ID;
- date of sample receipt, date and time of sample collection, and date/time of sample preparation and/or analysis;
- sample matrix;
- reporting units and identification of whether the sample results are reported on an "as-received" or dry weight basis;
- method reference for the parameter analyzed and method reporting limits;
- identification of numerical results with values below the method reporting limit;
- case narrative that identifies test methods, describes any deviation from the method or contractual requirements, additions or exceptions to the SOP, and discloses any conditions that may affect the quality of the results;
- identification of sample results that did not meet sample acceptance criteria;
- footnotes or qualifiers referenced to specific data (as applicable) and explanations or keys to flags and abbreviations used;

- surrogate and tracer recoveries, where applicable;
- where applicable, a statement of the estimated uncertainty of the test result; and
- a signature and title, or equivalent electronic identification, of the personnel who accepts responsibility for the content of the report, and the date of issue.

If a report is reissued, the amendments must clearly state that the report is reissued. The cover letter and case narrative must describe why the report has been reissued and which sample results have been reissued.

10.7.2.3 QC REPORTS

Each final report includes QC reports that summarize results from the associated LCS, MB, and matrix QC samples. Additional QC samples may be prepared and reported to comply with project-specific requirements.

10.7.2.4 DATA QUALIFIERS – FLAGGING CODES

Whenever the data quality objectives of the LQAP are not met, the associated sample results must be flagged with the appropriate flagging codes. These codes are applied only in the event that the laboratory cannot generate (through reanalysis) fully compliant data. If sample values are reported outside the calibration range of the method or unreliable interferences exist in the sample, then descriptive codes are applied to the result.

Data qualifiers are added by the laboratory prior to reporting the analysis results. The laboratory appends data qualifiers to each environmental field sample based on an evaluation of all available QC information (e.g., MS/MSD samples, laboratory blanks, LCSs, calibration verification standards, etc.). Analytical batch comments are added to the narrative section of each data report to explain any nonconformance or other issues.

Other flagging practices may be observed if so dictated by the applicable LIMS program specification.

10.7.3 ELECTRONIC DATA DELIVERABLES (EDDS)

The electronic data deliverables generated by the laboratory are project-specific and are produced in a format specified by the client.

Information presented in corresponding fields of the hardcopy report and EDD are identical as both are generated from LIMS. Before submitting the EDD file, the Project Manager or designee verifies that the EDD is complete and meets the client's format requirements. All EDDs are submitted to the client on computer disks or are transmitted electronically.

10.8 RECORDS AND DATA STORAGE

Records provide the direct evidence and support for the necessary technical interpretations, judgments, and discussion concerning laboratory results. These records, particularly those that are anticipated to be used as evidentiary data, provide the historical evidence needed for later review and evaluation. Records must be legible, identifiable, and retrievable. They must be protected against damage, deterioration, fire, theft, vermin, and loss. Though only 5-year retention

is required by NELAC, ALSLG-FC retains all records for a minimum of seven (7) years, or as otherwise specified per the client's contract.

Laboratory records include the following kinds of documentation:

- personnel qualifications, experience, and training;
- correspondence between ALSLG-FC and clients;
- quality assurance records (e.g., retired SOPs and LQAPs, PT study results, internal and external audit reports and responses);
- contents of laboratory logbooks;
- equipment maintenance records;
- traceability of standards, solvents and reagents;
- instrument checks and calibrations;
- raw data;
- final data reports; and
- sample management records (e.g., sample login, field and internal chain-of-custody, storage, disposal).

10.8.1 ELECTRONIC RECORDS

ALSLG-FC employs a multi-level system that addresses both the frequent backup of sample results (in LIMS) and the periodic backup of raw data (from both networked and non-networked instruments). Additionally, the software that ALSLG-FC uses for these backups, contains a disaster recovery module that allows for the complete recovery of the backup database, in its entirety. In short, ALSLG-FC's LIMS is backed up hourly, and, along with all network servers, is additionally backed up to tape each business day. As indicated in the IS and LIMS Policy Statement (**Appendix A**), instrument backups are performed approximately monthly. Contingent upon the volume of analysis, the frequency of backup might vary.

Backup of the instrument computers is done centrally by the IS Manager if the instrument computer is on the network. It is the responsibility of the operator/user to coordinate a convenient time for both the IS Manager and the user for non-network instrument backup. The instruments that are not on the network are backed up using portable devices. These devices, as well as media, are checked out from the IS Manager, then are returned to the IS Manager for safe storage.

An electronic archive for maintaining final project reports was implemented in 2001. Upon completion of a workorder, all data reports are scanned to create image files that are catalogued and saved

to a dedicated server that is backed up daily as described above. The scanned images remain available on the network for review should any questions regarding the data arise. Retention of hardcopy data reports prior to 2001 is discussed below.

10.8.2 HARDCOPY RECORDS

Prior to electronic compilation and storage, ALSLG-FC created paper copies of project reports. These hardcopy data archives are retained off-site by a records storage contractor. The QAM maintains a database inventory of all records that are stored at the contractor's facility. The contractor is responsible for the maintenance and protection of these records. Access to the records is limited to only designated individuals. If any records need to be retrieved from the storage site, the requestor must fill out an archive request form (Form 136) and submit it to the Quality Assurance Department. E-mail requests directed to the QAM are also acceptable. The QA Department then requests the records from the contractor, who retrieves the records and delivers them to the laboratory on the next business day.

Hardcopy originals of records that have been imaged and verified may be destroyed confidentially (i.e., shredded). Detailed procedures for archiving records and submitting archive requests are provided in **SOP 069**.

As of this writing, no provisions have been made to permanently destroy any records generated by ALSLG-FC. Should ALSLG-FC permanently destroy any records, written notification will be provided to all clients affected.

In the event that the laboratory changes ownership, the responsibility for the retention of records in accordance with the guidelines established in this LQAP, is conferred to the new owner. Should ALSLG-FC go out of business, ALSLG-FC will inform our clients in writing of this business decision, and will transfer records at the client's request.

10.9 CLIENT INQUIRIES/COMPLAINTS

The focal point of contact with the client is the ALSLG-FC Project Manager. If a complaint or any circumstance raises doubt concerning ALSLG-FC's compliance with its policies or procedures, or with the requirement of a method or quality system, it is the Project Manager who initiates investigation and follows through to resolution. The QAM, Department Managers, and Laboratory Director are made aware of, and involved in, the resolution process as needed. Documentation of the complaint and its resolution are maintained as part of the project records. Where resubmission of data is required and/or implementation of preventive measures is necessary, an NCR Form (**Appendix F**) is used and processed (**SOP**

928), through the QAM. ALSLG-FC will respond to all complaints in a timely fashion.

10.10 CONFIDENTIALITY

All laboratory results and associated raw data are confidential and may not be released to or discussed with any party other than the client who requested the analytical services. Access to laboratory records and LIMS is limited to laboratory personnel, on a restricted basis, based on need (i.e., job function). Records are available for an accrediting authority's on-site review, and records specific to the client (as well as quality system records) are available to the client for client audits. ALSLG-FC expects that auditors will honor our clients' and ALSLG-FC's confidentiality requirements, and will not discuss any results, documents, or records viewed during the course of an audit.

Confidentiality is included as a component of ALSLG-FC's ethics training, which is provided to each person as they join the ALSLG-FC staff, and annually, as a refresher training, thereafter.

11. CORRECTIVE ACTIONS

Corrective action is necessary when any measurement system fails to meet the requirements of this LQAP, the appropriate SOP or project-specific instructions, or whenever an error is detected. Items that may need corrective action range from a minor problem such as an analyst failing to initial a form, to a major problem such as a chemist preparing a sample using the wrong reference method.

Corrective actions fall into two general categories: short-term and long-term. Short-term corrective actions are those that can be applied immediately. Examples include: having an analyst initial a form where the initial was missed, or correcting an error in a logbook entry per procedures described in SOP 303. Long-term corrective actions are those that require a clarification of practice or a change in policy in order to effectively resolve the problem. Corrective actions must be completed by the date designated by the QA Department (i.e., within 21 calendar days or less, unless otherwise provided for). Associated SOPs may need to be revised and republished for long-term corrective actions, laboratory staff must be re-trained in accordance with the updated procedures.

11.1 RESPONSIBILITIES FOR CORRECTIVE ACTION INITIATION

The type of corrective action taken is coordinated by the Department, Quality Assurance and applicable Project Managers. A controlled Nonconformance Report (**Appendix F**) is used to document the corrective action. *Any individual who notes a problem or deviation is responsible for initiating the NCR in a timely manner.*

It is the responsibility all personnel who work with samples to note any discrepancies or nonconformances that occur with sample handling. It is the responsibility of the chemists who prepare samples for analysis to document any problems that are noted during sample preparation. It is the analyst's responsibility to monitor the proper functioning of the analytical system prior to,

during and following sample analysis. To accomplish this, various DQIs as discussed in Chapter 3 of this LQAP are monitored and evaluated against laboratory established or project-specific QA/QC requirements. If the evaluation reveals that any of the QC acceptance criteria are not met, then the analyst must immediately correct the problem. When an acceptable resolution cannot be achieved and/or data quality is negatively impacted, the analyst must notify the Department and Project Managers and must initiate an NCR (**SOP 928**) immediately. Per the guidance contained in SOP 928, the laboratory shall notify all affected clients of potential data quality issues in a timely manner, and corrective actions taken to resolve the issue shall be completed in a reasonable timeframe, with documentation submitted to the client.

11.2 **ALS LABORATORY GROUP, FORT COLLINS CORRECTIVE ACTION PROCESS**

Non-conformances are reported (documented) electronically through a LIMS interface that is available to all staff. The individual who discovered the problem or deviation is responsible for initiating the next sequential NCR in LIMS. Note that in addition to documenting laboratory sample or test issues, NCRs are also used to address client inquiries, and to investigate Performance Test (PT) sample failures.

Documented on the NCR are the initials of the initiator and descriptions of the method, workorder(s) and samples affected; the type, content and extent of the problem noted; the probable cause and the root of the problem (if known); measures taken to prevent recurrence; the specific corrective actions taken and their outcome; and the final disposition/resolution of the data.

As described in **SOP 928**, the processing of the NCR flows from the initiator, to their immediate Supervisor and/or Department Manager and the relevant Project Manager(s), and finally to the Quality Assurance Manager. In this manner, a consensus is achieved as to what specific corrective actions are to be taken. The Project Manager, at his or her discretion, may or may not contact the client to discuss options based on the nature of the nonconformance. Whether or not the client is contacted is noted on the NCR, if the client is contacted, the Project Manager documents who was contacted and when. The Project, Department and Quality Assurance Managers electronically sign and date the NCR, documenting their final approval and verification of the disposition of the data. The LIMS provides for delegation of signature authority as needed to cover key staff outages.

The LIMS, which is subject to ALSLG-FC's frequent backup protocols, maintains an archive of all NCRs generated. In this manner, NCRs are retained as part of the laboratory's electronic records. Also, contingent upon the level of data deliverable specified by the client, a copy of the associated NCR report is included in the analytical data package. Corrective actions that require follow-up, including those initiated by internal or external auditors, are catalogued in a separate LIMS Table that tracks audit findings. This LIMS Audit Findings Table

is managed by the QA Department but is available to all staff on a read-only basis.

12. AUDITS

12.1 INTERNAL AUDITS

Periodic evaluations conducted by the Quality Assurance Department and the analysis of Proficiency Test (PT) samples are two types of internal audits used to assess and document the performance of laboratory staff and processes. Audit documentation constitutes a permanent record of the conformance of ALSLG-FC's measurement systems to quality system requirements.

Internal audits include both technical and systems audits, and are performed periodically per an annual schedule developed and maintained by the Quality Assurance Department. Considerations taken into account in developing the internal audit schedule include, but are not limited to, requests made by the Laboratory Director; the scheduled occurrence of external audits; as needed to support a specific project's requirements; to verify the continued effectiveness of corrective actions previously taken; or in response to an identified need to evaluate compliance in any area of laboratory operations. The intention of the internal audit schedule is to provide for the evaluation of each laboratory area or system at least once annually, thereby providing an overview of laboratory operations. Form 168 or other audit questionnaire may be used as a guide to conduct and document internal audits. Each year, the internal audits conducted are compiled into the annual Quality Systems Audit (QSA), which is discussed subsequently (LQAP Section 12.1.3).

All internal audits are conducted by QA staff or designees who, by experience, are deemed to be knowledgeable in the area assessed. The assigned auditor identifies the scope, time frame and expected duration of the audit, and communicates this information to the applicable Department Manager. The auditor reviews relevant information such as regulations, contract requirements, published procedures, SOPs, etc., prior to the audit. The criteria set forth in these applicable guidances establish the basis of the audit. These reference materials may also be used as auditor's aids.

The audit is conducted in an efficient and professional manner. Findings, Observations and comments are communicated to the Department Manager.

Short-term corrective actions may be taken at the time an item is noted, or an appropriate long-term corrective action plan may be developed. An audit is considered to be closed-out when deficiencies have been satisfactorily corrected.

An audit report summarizing the Determinations made and the corrective actions taken or planned is compiled; the original auditor's notes are customarily included as an attachment of the audit report. The outcome of the audit is communicated to the Laboratory Director. Internal audit corrective actions requiring follow up are

tracked in a LIMS Table that is available for viewing to all laboratory personnel. The QAM oversees satisfactory completion of corrective measures taken. Internal audit records are maintained by the Quality Assurance Department.

See **SOP 937** for additional information pertaining to internal audit procedures.

12.1.1 INTERNAL TECHNICAL AUDITS

Departmental functions that may be reviewed during a technical audit may include, but are not limited to:

- Adherence to SOPs and compliance with promulgated method requirements during sample preparation and analysis;
- Maintenance of internal chain-of-custody;
- Proper preparation, storage, use and documentation of standards;
- Performance and documentation of instrument maintenance;
- Performance and documentation of data review;
- Evaluation of documentation practices pertaining to benchsheet and logbook entries, Nonconformance Report (NCR) generation and analyst demonstration of capability.

12.1.2 INTERNAL SYSTEM AUDITS

Examples of elements that may be reviewed as a system audit may include, but are not limited to:

- An assessment of the SOP process, including procedures for submitting and approving revisions, update and distribution of SOPs, tracking of employee SOP assignments and sign-offs, SOP electronic file management, and archiving of older SOP iterations and records.
- LIMS data capture and reporting processes.
- Sample handling, storage and disposal practices, including maintenance of sample storage areas, sample tracking and internal chain-of-custody documentation, duration of retention, and disposal designation and documentation.
- Use of ALSLG-FC's Standards and Reagents database.
- Performance and documentation of laboratory logbook review.

12.1.3 ANNUAL QUALITY SYSTEMS AUDIT

A lab-wide review of conformance to ALSLG-FC's quality system is conducted annually by the QA Manager or designee(s) as required by Section 5.5.3.1 of the NELAC Standard. The annual Quality Systems Audit (QSA) shall be managed, conducted and reported according to the audit procedures described above. Inputs to the QSA may include, but are not limited to, summaries of the following: Nonconformance Reports (NCRs), Proficiency Testing (PT) study results, deficiencies noted during data review, internal audit Determinations, and Determinations made via external audits.

12.1.4 PROFICIENCY TESTING STUDIES

ALS Laboratory Group, Fort Collins participates in agency studies and/or contracts approved vendors to provide PT samples in accordance with a schedule developed and maintained by the Quality Assurance Department. Participation in PT studies enables ALSLG-FC to demonstrate capability for continued accreditation, competency in a newly developed method, or the effectiveness of corrective actions taken.

ALSLG-FC participates in the following inter-laboratory proficiency testing studies:

- Water Supply (WS) -- twice annually
- Water Pollution (WP) -- twice annually
- Soil/Hazardous Waste and UST -- twice annually
- Radiochemistry -- twice annually
- US Department of Energy (USDOE) Mixed Analyte Performance Evaluation Program (MAPEP) -- twice annually

These PT studies support various regulatory programs (SDWA, CWA, RCRA) and require that the laboratory perform analyses per various methodologies (e.g., EPA 600 series, MCAWW, ASTM, SW-846), matrices and analytes. Analyte lists include: volatile organics, semivolatile organics, organochlorine pesticides, polychlorinated biphenyls, organophosphorous pesticides, phenoxyacid herbicides, high explosives, petroleum hydrocarbons, metals, minerals, nutrients and radionuclides. The analyses of PT samples are conducted in-house, in the manner prescribed by the provider, and within the turnaround time stipulated. The PT samples are distributed to the laboratory and are processed by qualified analysts who routinely perform the analytical method.

PT study results are evaluated by the Quality Assurance Department and the applicable Department Manager as they become available. The NCR and corrective action process as described in Chapter 11 of this LQAP, is used to address any deficiencies that are noted. An archive of PT study reports, maintained by the QA Department, is posted to the network for lab-wide access.

12.1.5 ANNUAL MANAGERIAL REVIEW

A lab-wide Managerial Review is performed annually as required by Section 5.5.3.2 of the NELAC Standard. The Managerial Review assesses operational effectiveness in terms of meeting ALSLG-FC's business goals. It is a tool used to document and facilitate the consideration and introduction of needed operational changes and improvements.

The Managerial Review is performed by a designee under the direction of the Laboratory Director. The general techniques of scoping, assessment interview, reporting and follow-up as described in the internal audit procedures discussed above and outlined in SOP 937, are used to conduct the annual Managerial Review. The contents of the annual Managerial Review are considered to be confidential. A confidential footer must, therefore, appear as a component of the annual Managerial Review report.

Inputs to the Managerial Review may include, but are not limited to the following: a snapshot summary of product generated (i.e., number of samples analyzed and the types of analyses performed), various business assessment reports (e.g., TAT, on-time delivery), output from the annual QSA (i.e., problem areas identified), interview of laboratory staff, and presentation of items discussed during strategic planning sessions and/or Manager's meetings.

12.2 EXTERNAL AUDITS

External audits may be performed by a state or Federal agency or a client as part of an ongoing certification process. Items evaluated by external assessors may include, but are not limited to, reviews of the following: analytical capabilities and procedures; COC procedures; document control; quality systems; and QC procedures. Blind PT samples may be submitted to the laboratory as a form of external audit.

See **Appendix I** for a list of ALSLG-FC's state and Federal certifications. Should

ALSLG-FC drop or lose an accreditation, the PMs must notify all clients that may be affected in a timely manner.

13. PERSONNEL TRAINING

The selection of well-qualified personnel is a factor that contributes to ALSLG-FC's success. Therefore, qualifications of personnel are based upon education and experience. In order to maintain qualified staff, provide personnel advancement within the laboratory, and to provide for personnel's ongoing awareness of potential hazards and protective measures, ALSLG-FC follows a formal documented program of orientation and training. Records of Health & Safety and waste training are maintained by the Health & Safety Manager/RSO and Facilities/Waste Compliance Manager. Technical training records are forwarded to the Quality Assurance Department for retention.

13.1 ORIENTATION

Before working in the laboratory, new employees receive a four-part orientation as described below:

- Human resources -- involves matters of immediate personal concern, such as benefits and company policies
- Quality assurance -- addresses topics related to ethical conduct, good laboratory practices and ongoing documentation of employee capability demonstrations. Required readings (SOPs, LQAP) are assigned at this time.
- Health & safety -- provides for a review of ALSLG-FC's various safety program documents (Chemical Hygiene Plan, CHP; Radiation Protection Plan, RPP; Emergency and Contingency Plan, ECP; Respiratory Protection Plan, ResPP; Waste Management Plan, WMP); as well as other safety and security training.
- Department functional orientation -- focuses on the new employee's basic understanding of their role within the Department and the overall role of Operations within the structure of ALSLG-FC. The Departmental training expands upon the employee's scientific background and work experience to provide the employee with a level of competence that enables the individual to successfully function within the defined responsibilities of his/her position.

Temporary employees receive the same orientation as regular staff, with the exception of the human resources orientation.

SOP 143 details information regarding quality assurance orientation and training for new employees.

13.2 TECHNICAL TRAINING

Chemists (analysts) and technicians are qualified to perform specific analytical procedures and methods. The qualification process, at a minimum, consists of background/theory training, on-the-job training, and demonstration of proficiency. Additional training may include further individualized instruction, programmed learning, conferences and seminars, and specialized training by instrument manufacturers.

Department Managers are responsible for providing documentation of analytical training and proficiency for each employee in their group(s) to the Quality Assurance Department for retention.

13.2.1 INITIAL DEMONSTRATION OF CAPABILITY (IDOC)

New analysts and technicians are trained by Department Managers according to the following guidelines:

- The new employee reads the SOP(s) pertinent to the analytical method being learned, and receives background/theory instruction, as applicable.
- The new employee observes the procedure in which the analytical method and required process documentation is demonstrated by trained personnel. Job requirements are outlined and quality control measurements are defined. For most methods, the trainee performs an Initial Demonstration of Capability (IDOC) by preparing and/or analyzing four (4) blank spike samples under the supervision of the Technical or Department Manager, or an analyst proficient in that method.
- The results of the new employee's preparation and/or analysis are evaluated and problems and corrective actions are discussed. If the blank spike recovery and precision data meet quality control criteria for that method, the employee is deemed to have demonstrated proficiency and is allowed to work on client samples. If the values generated are outside acceptance limits, then training continues until the trainee can consistently meet the acceptance criteria for the method.
- After the certification process has been successfully completed, the Department Manager forwards the documentation to the Quality Assurance Department for retention.

13.2.2 CONTINUING DEMONSTRATION OF CAPABILITY (CDOC)

ALSLG-FC's personnel are required to demonstrate their proficiency upon hire and with each batch of samples. Results from the laboratory control sample (LCS) spike performed by the chemist (analyst) or technician is evaluated ongoing and significant problems are dealt with immediately through the peer review process, non conformance system,

and training. This LCS data is available to review upon request. Alternately, MDL studies and reports from PT sample analysis may also be used to demonstrate an employee's capability.

13.2.2.1 METHOD DETECTION LIMIT (MDL) STUDIES

Most of the analytical methods employed at ALSLG-FC require the periodic generation of MDL data. The generation of acceptable MDL values requires a thorough understanding of the total analytical process and is a rigorous test of the proficiency of the analytical staff that performs the analysis. An analyst's or technician's performance in an MDL study that generates values that are consistent with past performance may be used to demonstrate initial and/or continuing proficiency in a method. This MDL information may be used in lieu of other demonstrations of proficiency, except where a regulatory promulgated method explicitly requires specific procedures to be followed for the initial demonstration of proficiency.

13.2.2.2 PROFICIENCY TEST (PT) SAMPLES

As discussed in Chapter 12 of this LQAP, ALSLG-FC participates in several proficiency testing programs. These programs typically submit single-blind standards to the laboratory and return a performance summary after results have been evaluated by the sponsoring agency or qualified vendor. Successful participation in these PT study programs by personnel is a rigorous demonstration of the staff's ability to perform routine analytical procedures. Records of successful participation in these programs may be used to demonstrate that an employee has been adequately trained in the methods that he/she performs. This IDOC/CDOC information may be used in lieu of other demonstrations of proficiency, except where a regulatory promulgated method explicitly requires specific procedures to be followed for the initial demonstration of capability.

13.3 TRAINING RECORDS

Technical and quality assurance training records are maintained by the Quality Assurance Department. Health & Safety training records are managed and retained by the Health & Safety Manager/RSO. Waste management training records are managed and maintained by the Facilities/Waste Compliance Manager. Employee training record files may contain, but are not limited to, the following:

- signed annual Ethics training documents

- resume or personnel qualifications form
- transcript or diploma
- QA training and signature/initial on file
- documentation of annual assigned SOP readings
- documentation of annual LQAP reading
- IDOC documentation
- PT study results
- MDL study results
- off-site training certificate

14. GLOSSARY, ACRONYMS AND SYMBOLS

14.1 GLOSSARY

<u>TERM</u>	<u>DEFINITION</u>
Acceptance Criteria:	Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQ)
Accreditation:	The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)
Accrediting Authority, Primary:	The agency or department designated at the Territory, State, or Federal level as the recognized authority with responsibility and accountability for granting NELAC accreditation for a specified field of testing. (NELAC) [1.5.2.3]
Accuracy:	The degree of agreement between a observed value and the accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations. (QAMS)
Aliquot:	A discrete, measured, representative portion of a sample taken for analysis. (EPA QAD)
Ambient:	Usual or natural surrounding conditions, e.g. ambient temperature – the natural, uninfluenced temperature of the surroundings. (NIRP Glossary)
Analyte:	The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family

TERM

DEFINITION

	and that are analyzed together. (DoD QSM)
Audit:	A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)
Background:	Ambient signal response recorded by measuring instruments that is independent of radioactivity contributed by the radionuclides being measured in the sample. (DOE QSM)
Batch:	Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to twenty environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates, or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)
Bias:	The deviation of a single measured value of a random variable from a corresponding expected value, or a fixed mean deviation from the expected value that remains constant over replicated measurements within the statistical precision of the measurement (Synonyms: deterministic error, fixed error, systematic error). (DOE QSM)
Blank:	A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, or analysis. The blank is subjected to the same analytical and measurement process as the associated samples. Blanks include: <u>Equipment blank</u> : a sample of analyte free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC) <u>Field blank</u> : a blank prepared in the field by filling a clean container with pure deionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER) <u>Trip blank</u> : Contaminant free water, or appropriate matrix, which accompanies bottles and samples during shipment to assess the potential for sample contamination during shipment. Trip blanks are not opened in the field, and are required for Volatile Organic Analysis only. (NIRP)

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Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Method blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all the steps of the analytical procedures. (NELAC)

Reagent blank: a sample consisting of reagent(s), without the target analyte(s) or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Blind Sample: A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample, but not the composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. See Initial Calibration. (NELAC)

Calibration, Continuing: The process of analyzing standards periodically to verify the maintenance of calibration of the analytical system.

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration, Initial: The process of analyzing standards, prepared at specified concentrations, to define the quantitative response, linearity and dynamic range of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a continuing calibration do not conform to the requirements of the method in use or at a frequency specified in the method. See Calibration.

Calibration, Initial Check/Verification (ICV): Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution which is different from the stock used to prepare calibration standards. (NIRP Glossary)

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Carrier:	Carriers are typically non-radioactive (e.g. natural strontium, barium, yttrium) elements. They follow similar chemical reactions as the analyte during processing and are added to samples to determine the overall chemical yield for the analytical preparation steps. The yield of the carrier is typically determined gravimetrically or by ICP and is used to correct radiochemical results for acceptable losses occurring during the preparation process. (DOE QSM)
Chain-of-Custody (COC) Form:	Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers, the mode of collection, preservation, and requested samples. (NELAC)
Confidential Business Information (CBI):	Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain information identified as such in full confidentiality. (NELAC)
Confirmation:	Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second column calibration, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures. (NELAC)
Conformance:	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)
Control Chart:	A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.
Control Limit:	A range within which specified measurement results must fall to signify compliance. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that nonconforming data be investigated and flagged.
Corrective Action:	The action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence. (ISO 8402)
Counting Efficiency:	The ratio of the net count rate of a radionuclide standard source to

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	its corresponding known activity. (DOE QSM)
Counting Uncertainty (Poissonian):	A statistical estimate of uncertainty in a radiochemical measurement due to the random nature of decay. Every radiochemical result is reported with an associated counting uncertainty, usually at the 95% confidence interval.
Data Quality Indicators:	The qualitative or quantitative statements that specify the quality of data required to support decision for any process requiring chemical or physical analysis.
Data Reduction:	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)
Daughter:	A nuclide formed by radioactive decay of a parent radionuclide.
Deficiency:	An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)
Demonstration of Capability (DOC):	A procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
Detection Limit, Analyte:	The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)
Detection Limit, Instrument (IDL):	The concentration of an analyte that produces an output signal twice the root mean square of the background noise, or the parameter determined by multiplying by three the standard deviation obtained of three to five times the desired IDL on three nonconsecutive days with seven consecutive measurements per day. IDL is only required for the metals and analysis. (DOE QSM)
Detection Limit, Method (MDL):	The Method Detection Limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. It may be determined using replicate spike samples prepared by the lab and taken through all steps of the method. The detection limit is calculated using the appropriate student's t-parameter times the standard deviation of a series of spiked samples. (Ref. 40 CFR Part 136, Appx. B)
Digestion:	A process in which a sample is treated (usually in conjunction with heat) to convert the sample into a more easily measured form. (DoD QSM)

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Dilution Factor:	The factor by which the dilution level of the sample differs from that of a predefined method blank. The method blank is prepared within the prescribed parameters of the method, and has a dilution factor of one. The dilution factor does not include a dryness factor. (DOE QSM)
Document Control:	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
Dry Weight:	The weight of a sample based on percent solids. The weight after drying in an oven at $105\pm 5^{\circ}\text{C}$.
Duplicate, Replicate Analysis:	<p>The analyses or measurements of the variable of interest performed identically on two sub samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation, or storage internal to the laboratory. (EPA-QAD)</p> <p>The measurements of the variable of interest performed identically on two or more sub-samples of the same samples within a short time interval. (NELAC)</p>
Duplicate (Replicate) Error Ratio (DER/RER):	A measure of precision used to assess agreement between radiochemical duplicates (replicates) that compares the discrepancy between two measurements to the associated uncertainties.
Duplicate, Replicate Sample:	<p>A second aliquot of the same sample that is treated the same as the original sample in order to determine the precision of the method.</p> <p>A second, separate sample collected at the same time, from the same place, for the same analysis, as the original sample in order to determine overall precision.</p>
Eluent:	A solvent used to carry the components of a mixture through a stationary phase. (DoD QSM)
Elution:	A process in which solutes are washed through a stationary phase by the movement of a mobile phase. (DoD QSM)
Energy Calibration:	The correlation of the multi-channel analyzer (MCA) channel number to decay energy, obtained from the location of peaks from known radioactive standards. (DOE QSM)
False Negative:	An analyte incorrectly reported as absent from the sample, resulting in

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	potential risks from their presence. (DoD QSM)
False Positive:	An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern. (DoD QSM)
Finding:	An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)
Half Life ($T_{1/2}$):	The time required for 50% of a radioactive isotope to decay. (DOE QSM)
Holding Time (Maximum Allowable):	The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR Part 136)
Homogeneity:	The degree to which a property or substance is evenly distributed throughout a material.
Interference, Spectral:	Occurs when particulate matter from the atomization scatters the incident radiation from the source or when the absorption or emission of an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible. (DoD QSM)
Interference, Chemical:	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte. (DoD QSM)
Internal Standards:	A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (NELAC)
Isomer:	Generally, any two chemicals with the same chemical formula but with a different structure. (DoD QSM)
Isotope:	A variation of an element that has the same atomic number of protons but a different weight because of the number of neutrons. Various isotopes of the same elements may have different radioactive behaviors, some are highly unstable. (NIRP Glossary)
Lot:	A quantity of bulk material of similar composition processed or manufactured at the same time.
Matrix:	The substrate of a test sample. Field of Accreditation Matrix: these matrix definitions shall be used when accrediting a laboratory: <u>Drinking Water:</u> any aqueous sample that has been designated a

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potable or potential potable water source.

Non-Potable Water: any aqueous sample excluded from the definition of Drinking Water matrix. Includes surface water, groundwater, effluents, water treatment chemicals, and TCLP or other extracts.

Solid and Chemical Materials: includes soils, sediments, sludges, products, and by-products of an industrial process that results in a matrix not previously defined.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device. (NELAC)

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Minimum Detectable Activity (MDA, Lower Limit of Detection):

The minimum detectable activity is the smallest amount (activity or mass) of an analyte in a sample that will be detected with a probability beta of nondetection (Type II error) while accepting the probability alpha of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). For the purposes of this standard, the alpha and beta probabilities are both set at 0.05 unless otherwise specified. (ANSI N 13.30 and ANSI N42.23)

Minimum Detectable Concentration (MDC):

The Minimum Detectable Activity expressed in concentration units.

National Voluntary Laboratory Accreditation Program (NVLAP):

A program administered by NIST that is used by providers of proficiency testing to gain accreditation for all compounds/matrices for which NVLAP accreditation is available, and for which the provider intends to provide NELAP PT samples. (NELAC)

Negative Control:

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Nonconformance:

An indication or judgment that a product or service has not met the requirements of the relevant specifications, contract or regulation, also the state of failing to meet the requirements. (DoD QSM)

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Performance Based Measurement System (PBMS):	A set of processes wherein the data quality needs, mandates, or limitations of a program or project are specified and serve as criteria for selecting measurement processes which will meet those needs in a cost effective manner. (NELAC)
Positive Control:	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)
Precision:	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms. (NELAC)
Proficiency Test Sample:	A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)
Qualitative:	Analysis without regard to quantity or specific numeric values. (NIRP Glossary)
Quality Assurance:	An integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)
Quality Control (QC):	The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of the users. (QAMS)
Quality Control Sample:	An uncontaminated matrix spiked with known amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)
	<u>Laboratory Control Sample (LCS)</u> : (However named, also Laboratory Fortified Blank, Blank Spike, or QC Check Sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias, or to assess the performance of all or a portion of the measurement system. (NELAC)
	<u>Laboratory Duplicate (DUP)</u> : Aliquots of a sample taken from the same container under laboratory conditions and processed and

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	analyzed independently. (NELAC)
	<u>Matrix Spike (spiked sample or fortified sample)</u> : A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)
Quantitation Limits, Practical (PQL):	Levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported at a specified degree of confidence. (NELAC) The value at which an instrument can accurately measure an analyte at a specific concentration (i.e. a specific numeric concentration can be quantified). These points are established by the upper and lower limits of the calibration range. (DoD clarification)
	The lowest concentration where the 95% confidence interval is within 20% of the true concentration of the sample. The percent uncertainty at the 95% confidence level shall not exceed 20% of the results for concentrations greater than the practical quantitation limit. (DOE QSM)
Quantitative:	Analysis with regard to quantities or specific numeric values. (NIRP Glossary)
Radioactive Decay:	The process by which a spontaneous change in nuclear state takes place. This process is accompanied by the emission of energy and subatomic particles. (DOE QSM)
Radiation Yield:	The amount of radiation of the type being measured that is produced per each disintegration, which occurs. For gamma spectrometry, this is commonly called gamma abundance. (DOE QSM)
Raw Data:	Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm, or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g. tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)
Reagent Water:	Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the

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	analytical method. (NELAC)
Region of Interest (ROI):	In radiochemical analysis, the Multi-channel Analyzer region defining the isotope of interest displayed in terms of energy or channels. (DOE QSM)
Relative Percent Difference (RPD):	A measure of precision between two duplicate (replicate) results expressed as the percent difference between the results relative to the average of the results.
Reliability Check (Daily):	A periodic check of the Continuing Calibration of an instrument used for radiochemical measurements.
Reporting Limit:	The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the statistically determined MDL, but may be higher based on any of the above considerations. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.
Retention Time:	The time between sample injection and the appearance of a solute peak at the detector. (DoD QSM)
Rounding Rules:	If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded to 11.44. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded to 11.45. If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded to 11.44, while 11.425 is rounded to 11.42. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.
Sample:	A single container or series of containers identified by a unique number comprised of material drawn from a single location or a composite of locations during a fixed period representative of that location (s) and time period(s) for the purpose of analytical testing or physical evaluation. (DOE QSM)
Selectivity:	(Analytical chemistry) The capability of a test method or instrument to respond to a target substance in the presence of non-target substances. (EPA-QAD)

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Sensitivity:	Capability of method or instrument to discriminate between measurement responses representing different levels (e.g. concentrations) of a variable of interest. (NELAC)
Signal-to-Noise Ratio:	The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in amplitude. (DoD QSM)
Split Sample:	A portion or subsample of a total sample obtained in such a manner that is not believed to differ significantly from other portions of the same sample.
Standard Operating Procedure (SOP):	A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing routine and repetitive tasks. (QAMS)
Reference Material:	<p>A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)</p> <p>A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 – 2.2)</p>
Standard (Spike) Addition:	In radiochemistry, the addition of a known quantity of a radiotracer to a sample and to a split or splits of a sample. Both the sample and split(s) are then processed through the method and the difference in response between the samples used to correct for overall bias resulting measurement bias and from losses during preparation. This method of internal calibration is used in radiochemical determinations where isotopic differentiation between target analyte and tracer is not possible.
Statistical Minimum Significant Difference (SMSD):	The minimum difference between the control and a test concentration that is statistically significant, a measure of test sensitivity or power. The power of a test depends in part on the number of replicates per concentration, the significance level selected, and the type of statistical analysis. If the viability remains constant, the sensitivity of

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	the test increases as the number of replicates is increased. (NELAC)
Surrogate:	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes. (QAMS)
Target Analytes:	Identified on a list of project-specific analytes for which laboratory analysis is required.
Tolerance Chart:	A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/-10% of a mean) based on the precision level judged to be acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radio bioassay laboratories). (ANSI)
Total Propagated Uncertainty (TPU):	An estimate or approximation of the total error associated with a measured value by propagation of individual (preparation, determination) uncertainties.
Traceability:	The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)
Tracer:	A traceable internal standard, usually a unique isotope of the element being determined, added to each sample in known amount which enables quantitation of analytes of interest independent of external means of calibration.
Tracer Chemical Recovery:	The percent yield of the recovered radioisotope after the sample/tracer aliquot has undergone preparation and instrument analysis. (DOE QSM)
Tune:	An injected standard required by the method as a check on instrument performance for mass spectrometry. (DoD QSM)
Validation:	Confirmation by examination and provision of evidence that specified requirements have been met. (EPA-QAD)
Verification:	Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

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	The result of verification leads to a decision either to restore in service, to perform adjustment, to repair or downgrade, or declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.
Warning Limits:	The limits (typically 2 standard deviations either side of the mean) shown on a control chart within which most results are expected to lie (within a 95% probability) while the system remains in a state of statistical control.

14.2 ACRONYMS

<u>TERM</u>	<u>DEFINITION</u>
AA	Atomic Absorption
AFCEE	Air Force Center for Environmental Excellence
ANSI/ASQ	American National Standards Institute/American Society for Quality
APHIS	USDA Animal and Plant Health Inspection Service
API	American Petroleum Institute
ARAR	Applicable or Relevant and Appropriate Requirement
ASCII	American Standard Code Information Interchange
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BNA	Base-Neutral and Acid Extractable Organic Compounds
BS	Blank Spike
BTEX	Benzene, Toluene, Ethylbenzene, Xylene
°C	Degrees Celsius
CAS	Chemical Abstract Service
CCC	Calibration Check Compound
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification

<u>TERM</u>	<u>DEFINITION</u>
CDPHE	Colorado State Department of Public Health and the Environment
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CF	Calibration Factor
CFR	Code of Federal Regulation
CLLE, CLE	Continuous Liquid-Liquid Extractor
CLP	Contract Laboratory Program
COC	Chain of Custody
CVAA	Cold Vapor Atomic Absorption Spectroscopy.
CWA	Clean Water Act
D	Drift or Difference
DBCP	1,2-Dibromo-3-chloropropane
DCM	Dichloromethane
DENIX	Defense Environmental Management Information Exchange
DER	Duplicate Error Ratio
DFTPP	Decafluorotriphenylphosphine
DI	Deionized
DOC	Demonstration of Capability
DoD	Department of Defense
DOE	Department of Energy
DOT	Department of Transportation
DPM	Disintegrations per Minute
DQI	Data Quality Indicator
DRO	Diesel Range Organics
ECD	Electron Capture Detector
EDB	Ethylene Dibromide
EDD	Electronic Data Deliverable
EERF	Eastern Environmental Radiation Facility

<u>TERM</u>	<u>DEFINITION</u>
EMSL	Environmental Monitoring Systems Laboratory
EPA	Environmental Protection Agency
FID	Flame Ionization Detector
FPD	Flame Photometric Detector
GALP	Good Automated Lab Practice
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption
GFPC	Gas Flow Proportional Counting
GPC	Gel Permeation Chromatography
GRO	Gasoline range organics
HECD	(Hall) Electrolytic Conductivity Detector
HEM	Hexane Extractable Material
HDPE	High-Density Polyethylene
HPGe	High Purity Germanium Gamma Spectrometer
HPLC	High-Performance Liquid Chromatography
IC	Ion Chromatography
ICAP-AES	Inductively Coupled Argon Plasma -Atomic Emission Spectroscopy
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Standard
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IPC	Instrument Performance Check
IPN	Incoming Project Notice
IRPIMS	Installation Restoration Program Information Management System

<u>TERM</u>	<u>DEFINITION</u>
IS	Internal Standard
ISO/IEC	International Standards Organization/International Electrotechnical Commission
KD	Kuderna Danish
LCS	Laboratory Control Sample
LD	Laboratory Duplicate
LFB	Laboratory Fortified Blank
LFM	Laboratory Fortified Matrix
LIMS	Laboratory Information Management System
LLRW	Low Level Radioactive Waste
LQAP	Laboratory Quality Assurance Plan
LRB	Laboratory Reagent Blank
LSC	Liquid Scintillation Counting
LUFT	Leaking Underground Fuel Tank
LUST	Leaking Underground Storage Tank
MAPEP	Mixed Analyte Performance Evaluation Program
MCAWW	Methods for Chemical Analysis of Waters and Wastes
MDA	Minimum Detectable Activity
MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MEK	Methyl Ethyl Ketone (2-Butanone)
MIBK	Methyl Isobutyl Ketone
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
MTBE	Methyl tert-butyl ether
N/A	Not applicable

<u>TERM</u>	<u>DEFINITION</u>
NIST	National Institute of Standards
NCR	Nonconformance Report
ND	Non Detect
NEIC	National Enforcement and Investigations Center
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NEPA	National Environmental Policy Act
NFESC	Naval Facilities Engineering Service Center
NIRP	Navy Installation Restoration Program
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
NVLAP	National Voluntary Laboratory Accreditation Program
OSHA	Occupational Safety and Health Administration
PAH	Polynuclear Aromatic Hydrocarbon
PARCC	Precision, Accuracy, Representativeness, Completeness, Comparability
PBMS	Performance Based Measurement System
PCB	Polychlorinated biphenyl
PCDD	Polychlorinated dibenzo-p-dioxin
PCDF	Polychlorinated dibenzofuran
PEG	Polyethylene Glycol
PEL	Permissible Exposure Limit
PETN	Pentaerthrite tetranitrate
PID	Photoionization Detector
PM	Project Manager
PNA	Polynuclear Aromatic Hydrocarbon
PQL	Practical Quantitation Limit
psi	pounds per square inch

<u>TERM</u>	<u>DEFINITION</u>
PT	Proficiency Testing
PTFE	Polytetrafluoroethylene
QA	Quality Assurance
QAPjP	Quality assurance project plan
QASS	Quality Assurance Summary Sheet
QC	Quality Control
QIP	Quench Indicating Parameter
r^2	Correlation Coefficient
RCRA	Resource Conservation and Recovery Act
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine
RFP	Request for Proposal
RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study
RL	Reporting Limit
ROI	Region of Interest
RPD	Relative Percent Difference
RPM	Revolutions Per Minute
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
RT	Retention Time
RTW	Retention Time Window
SARA	Superfund Amendments and Reauthorization Act
SDWA	Safe Drinking Water Act
SMSD	Statistical Minimum Significant Difference
SOP	Standard Operating Procedure
SOW	Statement of Work

<u>TERM</u>	<u>DEFINITION</u>
SPCC	System Performance Check Compound
SPLP, SLP	Synthetic Precipitation Leaching Procedure
SVOC	Semivolatile Organic Compound
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
TCMX	Tetrachlorometaxylene
TCL	Target Compound List
TDS	Total Dissolved Solids
TIC	Tentatively Identified Compound
TLV	Threshold Limit Value
TOC	Total Organic Carbon
TPH	Total petroleum hydrocarbon
TPU	Total Propagated Uncertainty
TRPH	Total Recoverable Petroleum Hydrocarbons
TSCA	Toxic Substances Control Act
TSDF	Treatment, Storage, and Disposal Facility
TSS	Total Suspended Solids
TVPH	Total Volatile Petroleum Hydrocarbons
USACE	United States Army Corp of Engineers
USDA	United States Department of Agriculture
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UST	Underground Storage Tank
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
WET	Waste Extraction Test
ZHE	Zero Headspace Extraction

14.3 SYMBOLS

<u>LENGTH</u>	<u>DEFINITION</u>	<u>SYNONYM</u>
um	micrometer	10 ⁻⁶ meter
mm	millimeter	10 ⁻³ meter
cm	centimeter	0.01 meter
dm	decimeter	0.1 meter
m	meter	

<u>WEIGHT</u>	<u>DEFINITION</u>	<u>SYNONYM</u>
pg	picogram	10 ⁻¹² gram
ng	nanogram	10 ⁻⁹ gram
ug	microgram	10 ⁻⁶ gram
mg	milligram	10 ⁻³ gram
g	gram	
kg	kilogram	10 ³ gram

<u>VOLUME</u>	<u>DEFINITION</u>	<u>SYNONYM</u>
uL	microliter	10 ⁻⁶ Liter
mL	milliliter	10 ⁻³ Liter
dL	deciliter	0.1 Liter
L	Liter	

<u>CONCENTRATION</u>	<u>DEFINITION</u>
ng/uL	nanograms per microliter
ug/L	micrograms per liter
ug/kg	microgram per kilogram
ug/g	microgram per gram
ug/mL	microgram per milliliter
mg/kg	milligram per kilogram
mg/L	milligram per liter
ug/m ³	microgram per cubic meter
ppb	part per billion
ppm	part per million

<u>TIME</u>	<u>DEFINITION</u>	<u>SYNONYM</u>
s or sec	second	1/60 minute
m or min	minute	60 seconds, 1/60 h
h	hour	60 minutes

<u>TEMPERATURE</u>	<u>DEFINITION</u>
°C	Degrees Celsius
°F	Degrees Fahrenheit
° K	Degrees Kelvin

<u>ACTIVITY</u>	<u>DEFINITION</u>	<u>SYNONYM</u>
Bq	Bequerels	Disintegration/s
Ci	Curie	3.7 x 10 ¹⁰ Bq
dpm	Disintegrations per minute	

<u>ELECTRICAL</u>	<u>DEFINITION</u>
V	Volt
A	Ampere
EV	Electron Volt
F	Farad
Ω	Ohm
S or mho	Siemens
W	Watt

<u>PREFIXES</u>	<u>NUMERIC AMOUNT</u>
tera	10 ¹²
giga	10 ⁹
mega	10 ⁶
kilo	10 ³
hecto	10 ²
deca	10
deci	0.1
centi	10 ⁻²
milli	10 ⁻³

micro	10^{-6}
nano	10^{-9}
pico	10^{-12}
femto	10^{-15}

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APPENDIX C

STANDARD OPERATING PROCEDURES

The SOPs required for Phase I activities were previously provided to the EPA as indicated below

SOP 1	Environmental Particulate Air Sampling for Radionuclides	SOP1 to SOP7 provided in letter of September 24, 2010 to EPA
SOP 2	Gamma Ray Intensity to Ra-226 Soil Concentration Correlation	
SOP 3	Field Gamma Radiation Surveys	
SOP 4	Field Documentation	
SOP 5	Equipment Decontamination	
SOP 6	Sample Handling and Shipping	
SOP 7	Surface and Shallow Subsurface Soil Sampling	

Two additional SOPs required for Phase II activities are provided in this appendix.

SOP-8 SOIL SAMPLING FOR SEMI-VOLATILE AND VOLATILE ORGANIC
COMPOUND ANALYSIS

SOP-9 DEEP SUBSURFACE SOIL SAMPLING



SOP-8:

**SOIL SAMPLING FOR
SEMI-VOLATILE AND VOLATILE ORGANIC COMPOUND ANALYSIS**

1.0 SCOPE

1.1 PURPOSE

This standard operating procedure (SOP) describes methods and equipment that shall be used for collecting environmental surface soil, subsurface soil, and sediment samples for volatile organic compound (VOC) analysis. This SOP, prepared in accordance with EPA Publication SW-846, *Test Methods for Evaluating Solid Waste - Physical/Chemical Methods* (EPA, 1996), defines sample collection procedures for screening and definitive sampling levels, using a soil sampler, methanol, and sodium bisulfate preservation methods according to SW-846 Method 5035A (EPA, 1996).

1.2 APPLICABILITY

This document focuses on methods and equipment that are specific to sampling surface soil and subsurface soil for VOC analysis. It is not intended to provide an all-inclusive discussion of soil sample collection methods. The standard procedures for collecting soil samples are described in SOP 7, *Surface and Shallow Subsurface Soil Sampling*, and SOP 9, *Deep Subsurface Soil Sampling*.

2.0 EQUIPMENT AND MATERIALS

The following equipment is used employed in the process of sampling for VOCs in soil samples. Decontamination equipment, field documentation requirements, sampling forms, and sampling equipment are discussed in detail in the following SOPs: SOP-4, *Field Documentation*; SOP-5, *Equipment Decontamination*; and SOP-6, *Sample Handling and Shipping*.

- Field Balance
- EPA-approved VOC sampling kit (e.g., cut plastic syringe, EnCore sample, Purge-and-Trap sampler, Terra Core™)
- 40 mL Teflon cap glass vials
- Bagged ice for sample cooling (do not use dry ice)

3.0 SURFACE AND SUBSURFACE SOIL AND SEDIMENT SAMPLING PROCEDURES

3.1 BACKGROUND

Soil and sediment samples will be collected at the surface and subsurface. Techniques in SOP-7 and SOP-9 apply to sampling by hand and by using a drill rig, respectively, and should be followed prior to techniques provided in this SOP.

3.2 SAMPLE PRESERVATION

Methanol or Sodium Bisulfate Preservation is used with VOC sampling. Refer to SW-846 Method 5035A (EPA, 1996) for full details on sample preservation. A sodium bisulfate preservative solution is used for the collection of soil samples in which the suspected VOC concentration is in the range of 0.5 to 200 micrograms per kilogram ($\mu\text{g}/\text{kg}$). For soil samples in which the VOC concentration is suspected to be greater than 200 $\mu\text{g}/\text{kg}$, either a bulk sample may be collected and the laboratory will add a water miscible solvent or the sample is collected in a vial that contains a water-miscible organic solvent (methanol).

3.3 SAMPLING TECHNIQUES

All soil samples for VOC should be collected using the following techniques:

1. Obtain a tared 40 ml glass VOC vial containing the appropriate preservative and a magnetic stirring rod. With the plunger seated in the handle, push the VOC sampler into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 grams of soil.
2. Wipe all soil or debris from the outside of the sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.
3. Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the tared 40 ml VOC vial containing the appropriate preservative, and extrude the sample by pushing the plunger down. Quickly place the lid back on the tared 40ml VOC vial. Note: When capping the 40 mL VOC vial, be sure to remove any soil or debris from the threads of the vial.
4. Because the soil vial cannot be opened without compromising the integrity of the sample, at least one additional vial of sample may be collected for dry weight determination. This additional replicate must not contain preservative, since an aliquot will be used for dry weight determination.
5. All samples for VOC analysis shall be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice. For further details on shipping and handling refer to SOP-12.

4.0 DECONTAMINATION

All non-disposable equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-5. Personnel shall don appropriate personal protective equipment as specified in the project-specific health and safety plan. Note that when handling the vials that contain methanol, methanol

resistant gloves shall be worn. Investigation-derived waste generated in the sampling process shall be managed in accordance with the Work Plan.

5.0 REFERENCES

U.S. Environmental Protection Agency. 1996. SW-846 Method 5035A Revision 0, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples.



SOP-9:
DEEP SUBSURFACE SOIL SAMPLING

1.0 SCOPE

1.1 PURPOSE

This standard operating procedure (SOP) describes methods and equipment used for collecting environmental subsurface soil samples for chemical, radiological, and geotechnical analyses. For the purposes of this SOP, deep subsurface samples shall be those that are collected using a standard drill rig outfitted with hollow-stem augers.

1.2 APPLICABILITY

This SOP defines sample collection procedures for collecting deep subsurface soil samples using a drill rig. This document is not intended to provide an all-inclusive discussion of sample collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be described in the project-specific sampling plan.

2.0 EQUIPMENT AND MATERIALS

2.1 SAMPLING EQUIPMENT AND MATERIALS

The following equipment may be employed in the collection of deep subsurface soil samples. Decontamination equipment, field documentation requirements, sampling forms, and sampling equipment are discussed in detail in the following SOPs: SOP-4, *Field Documentation*; SOP-5, *Equipment Decontamination*; and SOP-6, *Sample Handling and Shipping*.

Drilling and sampling operations are typically conducted with the use of a mobile power auger drill equipped to advance holes through overburden using hollow-stem and continuous flight augers. Soil samples are generally recovered on a continuous or discontinuous basis, with the use of a 2 inch (51 mm) diameter, 6 inch (150 mm), 2 ft (600 mm) or 2.5 ft (750 mm) long, split-spoon sampler, over the full depth of the boreholes. The split spoon sampling is carried out in conjunction with the Standard Penetration Test used to provide 'N' values for the determination of relative density in cohesionless soils and consistency in cohesive soils.

Individual soil samples are examined upon recovery by the field geologist for purposes of describing and recording texture, colour, odour and moisture content. Borehole logs are prepared on the basis of sample and drilling process observations in the field describing the encountered strata and visual or olfactory evidence of subsurface contamination, if present.

Following field logging, samples are placed into labelled, sterile, plastic bags for shipment to the laboratory for analysis. Additional samples may be retained for detailed inspection off-site as necessary.

2.2 GPS AND MAPPING SOFTWARE

A Trimble® Pathfinder ProXRT global positioning system (GPS) with differential correction and a Ranger datalogger is used to log the borehole locations.

2.3 GAMMA RADIATION METER

The calibrated exposure meter ("µR" meter or tissue equivalent ion chamber) is used to measure real-time radiation levels to ensure regulatory limits are not exceeded.

2.4 VARIOUS MISCELLANEOUS ITEMS

Survey locations are found using a map of survey areas with marked grid nodes identified by northing and easting coordinates. An ink pen and field notebooks are used to record readings, general weather conditions, and other notes. The chain of custody form from the laboratory serves as the field sampling form. Safety gear requirements are found in the project Health and Safety Plan. Minimum personal protection equipment (PPE) will include hard hat, steel toed shoes, safety vest, gloves and safety glasses.

3.0 DEEP SOIL SAMPLING PROTOCOL

3.1 BACKGROUND

Subsurface soil samples are to be collected starting at five-feet below surface and extending down to the native soil surface. This can be accomplished by continuous sampling or by augering down to the required depth, collecting a sample using a standard split spoon tube and then augering to the next five foot interval. If native soil is discovered at an interval less than 5 foot below surface or below the previous sample, a sample will be collected at approximately halfway between the native soil and the last sample. A sample of native soil will also be collected. The sample method is determined based on the characteristics of the site, the soil matrix, and/or regulatory requirements.

3.2 SAMPLING PROGRAM OBJECTIVES

The objective of the deep subsurface soil sampling is to identify the extent of potential radioactive contaminants extending from the surface to the native soil surface at five-foot intervals. Sampling objectives are typically diverse and dependent on the nature of the project objectives.

Details pertaining to sample locations, number of samples, and type of analyses required, shall be presented in project-specific work plans. An approved, contracted laboratory shall be

contacted prior to sampling to provide minimum sample sizes required to meet detection limit requirements for the requested analysis.

4.0 DEEP SOIL SAMPLING PROCEDURES

All soil sample locations shall be recorded with coordinates using a Trimble® Pathfinder ProXRT GPS system. The logbook should also include the location, date, and time of each sample taken, sampling personnel present, and any unusual conditions.

Soil samples shall be obtained using split spoon samplers. Core samples can be collected using continuous core sampling or by augering to the required depth (5-foot intervals) and then using the split spoon sampler. Continuous samples to a specific depth can be taken using an auger, core sampler, or split spoon sampler since each incremental depth of soil removed must be included in the sample.

Split-spoon samplers consist of a hollow tube consisting of two halves split lengthwise that are held together by a circular connector head at the top and a drive shoe at the bottom. This procedure is used for samples taken at each new sub-surface soil layer – i.e. after the new depth is reached using an auger. The procedures outlined below shall be followed when collecting soil samples using this method.

- Attach a stainless steel cap to the sampler.
- Attach the sampler and cap assembly to a hammer.
- For the collection of relatively undisturbed soil samples, install a liner in the sampler.
- Once the desired sample depth is reached, retrieve sampler to the surface and detach the sampler from the hammer.
- Fill sample bags using a decontaminated stainless steel bowl and spoon or spatula, as necessary. Twigs, rocks, leaves and other undesirable debris should not be included if they are not considered part of the sample. The outside of sampling containers shall be kept free from dust and other materials to the extent possible to prevent cross contamination when opened at the laboratory.
- If continuous sampling is required to a specific depth, material will be removed incrementally and soil from specific intervals will be placed in a large stainless steel bowl and then into sample bags. Care must be taken to remove roughly equal amounts of soil from each depth.
- If a composite sample is needed, place samples into a stainless steel bowl for homogenization. Prior to homogenization, remove twigs, rocks, leaves and other undesirable debris if they are not considered part of the sample.
- Split spoons, augers and other tools must be wiped clean of visible dirt and decontaminated between sample intervals.

5.0 DECONTAMINATION

Equipment used in the sampling process shall be decontaminated prior to and after field use and between sample locations/depth intervals. Decontamination procedures are presented in SOP 5, *Equipment Decontamination*. Personnel shall don appropriate personal protective equipment as specified in the SOP and in the project-specific work plan. Any investigation-derived waste generated during the sampling process shall be managed in accordance with procedures outlined in the work plan.