Midnite Mine Superfund Site
100 Percent Design

Appendix R – Staging / Temporary Stockpiling Plan

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Prepared for:

Dawn Mining Company
PO Box 250
Ford, Washington 990413

and

Newmont USA Limited
6363 South Fiddler's Green Circle
Greenwood Village, Colorado 80111

Prepared By:

MWH Americas, Inc.
2890 E. Cottonwood Parkway, Suite 300
Salt Lake City, UT 84121

TABLE OF CONTENTS

R1.0 INTRODUCTION 1

R2.0 MATERIAL HANDLING 1

R2.1 COMMON ELEMENTS CONSIDERATIONS FOR TEMPORARY STOCKPILES 2

R2.2 EXISTING TOPSOIL STOCKPILES 2

R2.3 DEMOLITION DEBRIS FROM STRUCTURES IN CSZ 3

R2.4 PHASE 1 SOIL REMEDIATION MATERIALS 4

R2.5 CSZ GRADING MATERIALS 5

R2.6 HILLSIDE WASTE ROCK PILE MATERIAL PROCESS MATERIALS 5

R2.7 PIT 4 – PIT BOTTOM CLEANUP AND GRADING 6

R2.8 GROUNDWATER CONTROL SYSTEMS EXCAVATIONS 7

R2.9 PIT 3 – PIT BOTTOM CLEANUP AND GRADING 8

R3.0 CONSTRUCTION STORMWATER POLLUTION PREVENTION PLAN 9

R4.0 REFERENCES 10

LIST OF ACRONYMS

BODR	Basis of Design Report
CSWPPP	Construction Stormwater Pollution Prevention Plan
CSZ	Construction Support Zone
cy	cubic yard
EPA	U.S. Environmental Protection Agency
HSWRP	Hillside Waste Rock Pile
RA	Remedial Action
RD	Remedial Design
Site	Midnite Mine Superfund Site
SDTS	Structure Demolition Temporary Stockpile
STSP	Staging and Temporary Stockpiling Plan
SWMP	Stormwater Management Plan
SWRP	South Waste Rock Pile
TCSS	Temporary Clean Topsoil Stockpile
TISS	Temporary Impacted Soil Stockpile
WTP	Water Treatment Plant

R1.0 INTRODUCTION

This Staging and Temporary Stockpiling Plan (STSP) has been prepared to describe how waste rock and other excavated material will be managed to prevent contamination of previously uncontaminated areas, as well as preventing the release of contaminants to downstream areas during remedial actions (RAs) at the Midnite Mine Superfund Site (Site). This STSP is an appendix to the *Midnite Mine Superfund Site Basis of Design Report* (BODR), which presents the background and supporting information relevant to the Site and the planned RAs. The BODR also contains the engineering drawings, plans, and specifications for the Site remedial designs (RDs) that are necessary to implement the RAs. Specific locations and configurations of proposed temporary stockpiles discussed in this appendix are shown in the appropriate engineering drawings as referenced.

R2.0 MATERIAL HANDLING

This section of the STSP summarizes the material handling strategy to be used during the RA. This strategy includes minimizing the need for stockpiling of excavated material to the maximum extent practical by directly loading excavated materials into haul trucks for transport and placement in the waste containment area. Due to scheduling considerations, direct-hauling may not always be possible. Eight activities have been identified that will require temporary stockpiling of excavated materials. These eight activities/areas are:

1) Relocation of Existing Topsoil Stockpiles
2) Demolition Debris from Structures in the Construction Support Zone (CSZ)
3) Phase 1 CSZ Soil Remediation Materials
4) CSZ Grading Materials
5) Hillside Waste Rock Pile (HSWRP) Process Materials
6) Pit 4 – Bottom Cleanup and Grading
7) Groundwater Controls Systems Excavation
8) Pit 3 – Bottom Cleanup and Grading

The stockpiling activities for materials from these eight activities are primarily associated with early phases of the RA, prior to preparation of the waste containment areas for placement of

waste. If additional materials requiring stockpiling are identified as the RA construction progresses, it may become necessary to amend this STSP to include these materials and any new stockpiles areas required for their temporary storage. Any amendments to this STSP will be subject to EPA review and approval prior to implementation.

R2.1 COMMON ELEMENTS CONSIDERATIONS FOR TEMPORARY STOCKPILES

The following criteria are addressed for each of the eight stockpile areas to the extent they are applicable:

- Staging/Schedule for material stockpiling and final placement
- Estimate of material volume
- Estimated material properties, moisture conditions, contamination levels
- Stockpile area location, stockpile space (i.e. surface area) requirements
- Temporary soil cover requirements, primarily for temporary stockpiles of demolition debris
- Potential interim revegetation requirements, primarily for uncontaminated material stockpiles that may remain in place for a number of years

R2.2 EXISTING TOPSOIL STOCKPILES

Existing topsoil stockpiles located in the CSZ, which includes the construction support facilities and the proposed water treatment plant (WTP) and ponds, are shown on Drawings 2-1, 2-2, and 2-3. Key components of the Existing Topsoil Stockpiles STSP include:

- Grading the CSZ prior to construction of the new construction support facilities needed for the RA. The material in the Existing Topsoil Stockpiles will need to be relocated as part of site preparation and grading work and will be relocated as part of initial site preparation.
- Preliminary testing performed on soils in the southwest topsoil stockpile (Drawing 2-2) indicate these materials meet soil cleanup standards and may be used for clean soil cover during RA construction (MWH, 2013). If further testing verifies that these soils, and any soils in the other stockpiles shown on Drawings 2-1, 2-2, and 2-3, meet soil cleanup standards, then they will be relocated to a temporary topsoil stockpile. This

temporary clean topsoil stockpile (TCSS) will be located at the proposed WTP pond site as indicated on Drawings 2-1 and 4-1.

The WTP equalization ponds are planned for construction at the end of Phase 1 and soils stored in the TCSS will be used in initial phases of cover construction. This material will be used on Pit 4 or other areas where clean cover is required for RA construction (e.g., for areas in the footprint of the Pit 4 Overburden Pile where soil cover is needed after mine waste removal). Prior to stockpiling clean cover soils in the TCSS at the WTP pond site, soil cleanup and verification will be performed as needed so the materials placed in the temporary topsoil stockpile will not become contaminated.

If further testing indicates some of these materials do not meet soil cleanup standards, they will be temporarily stockpiled within the existing mine waste area at the Phase 1 Temporary Impacted Soil Stockpile (TISS) on the north end of the South Waste Rock Pile (SWRP). The TISS material ultimately will be consolidated in the Pit 4 waste containment area. The location of the Phase 1 TISS is also shown on Drawings 2-1 and 4-1. Materials placed in the Phase 1 TISS will be placed in Pit 4 during Phase 1 construction.

- Current estimates indicate that approximately 60,000 cubic yards (cy) of material exists in the southwest topsoil stockpile of the CSZ, approximately 13,000 cy of material exists in the southeast topsoil stockpile, and a total of approximately 25,000 cy of material exists in the northwest and northeast stockpiles.
- As discussed above, the locations of temporary stockpiles will be dependent upon whether or not the topsoil material meets soil cleanup standards (so either TCSS or TISS).
- If it is determined that some, or all, of the existing topsoil materials meet soil cleanup standards, then it is likely that the TCSS will be in place for a number of years prior to using this material in RA construction. As a result, these stockpiles of excavated topsoil will receive interim revegetative treatment to reduce the potential for erosion.

R2.3 DEMOLITION DEBRIS FROM STRUCTURES IN CSZ

Existing structures in the CSZ areas shown on Drawing 2-1 will be demolished as described in Appendix H, and the demolition debris relocated to a stockpile area as part of site preparation and access development work. Key components of the CSZ Demolition Debris STSP include:

- Demolition of these structures and relocation of the demolition debris will occur as part of initial site preparation work prior to the development of the Pit 4 waste containment area.
- Demolition debris from CSZ will be stockpiled on the north end of the SWRP in the CSZ Structure Demolition Temporary Stockpile (SDTS). The CSZ SDTS will be located adjacent to the TISS as shown on Drawings 2-1 and 4-1.
- Those portions of the demolition debris that could be transported by wind or surface water runoff will be protected by placement of a temporary 1.5-foot-thick cover of waste rock.
- Once the Pit 4 waste containment area is sufficiently developed, this demolition debris will be consolidated in an “unclassified waste zone” in the Pit 4 waste containment area (see Drawing 8-5). The volume of this demolition debris is estimated to be approximately 2,000 to 3,000 cy.

R2.4 PHASE 1 SOIL REMEDIATION MATERIALS

The Phase 1 soil cleanup operations that will occur prior to backfilling in Pit 4, and thus require temporary stockpiling, primarily include soil removal activities associated with construction of the CSZ. Specifically, this will include remediation of soils within the CSZ, Whitetail Creek Drainage cleanup area, and existing West Access Road as shown on Drawings 2-1, 2-2, 2-3, and 2-4 that do not meet the soil cleanup standard. The actual extent of the cleanup areas will be determined during the Phase 1 construction. Key components of the Phase 1 Soil Remediation Materials STSP include:

- Any contaminated materials excavated during remediation of the CSZ will be temporarily stockpiled within the Phase 1 TISS within the existing mine waste area.
- Contaminated soils from the Whitetail Creek Drainage and existing West Access Road (Drawing 2-4) also will be temporarily stockpiled within the Phase 1 TISS and ultimately will be consolidated in the Pit 4 waste containment area.
- Although the volume of material to be excavated during the Phase 1 soil cleanup is not currently known, it is expected to be relatively small (20,000 to 25,000 cy).

R2.5 CSZ GRADING MATERIALS

Preliminary grading designs for the CSZ are shown in Section 9 (Water Treatment Plant) and Section 2 (Construction Support Facilities) of the Drawings. Key components of the CSZ Grading Materials STSP include:

- Grading the CSZ as shown on Drawings 2-14 through 2-20 during initial site preparation work. It is anticipated that excess cut material will be generated during these site grading operations, and that this material, after verification sampling, likely will be relocated to the TCSS.
- These clean materials in the TCSS will be used as soil cover or in other areas requiring clean fill as part of RA construction.
- If these temporary stockpiles of cut materials remain in place for a number of years prior to using this material in RA construction, they will receive interim revegetative treatment to reduce the potential for erosion.

R2.6 HILLSIDE WASTE ROCK PILE MATERIAL PROCESS MATERIALS

HSWRP materials will be processed by screening and crushing without washing to produce drain rock for the underdrain systems in Pit 3 and Pit 4. Key components of the HWSRP processing STSP include:

- Underdrain Layer. As the underdrain layer is the first backfill layer to be placed in Pit 3 and Pit 4, both the drain rock material, and reject materials from the screening/crushing operation will need to be temporarily stockpiled prior to placement in the pits. HSWRP material processing will occur in two stages; the first stage occurring immediately prior to Pit 4 backfilling at the start of Phase 1 and the second stage immediately prior to Pit 3 backfilling at the start of Phase 2.
- Volume estimates for processed drain and reject material that will be necessary for Pit 3 and Pit 4 processing are presented in the Material Balance section of Appendix D.
- HSWRP material will be processed without washing. As a result, all processed drain and reject material will be in a relatively dry state at the time of placement in temporary stockpiles.

- The primary stockpiling area for HSWRP material will be in Area 5 of Site. This stockpile area will be expanded by relocating Protore Stockpile 7 material to the top of Protore Stockpile 6 as shown on Drawing 2-22. Once Protore Stockpile 7 is removed, the drain material stockpile footprint will be overlain with reject material from the screening operation or other quartz-monzonite-derived (lower reactivity) waste to a depth of 1 foot. This will form a "clean base" for the drain material stockpile should the underlying material be contaminated. Care will be taken during loading of stockpiled drain material for transport and placement in the pits so that processed materials are not mixed with underlying base material or penetrated and mixed with the underlying potential contaminated wastes.
- The existing safety berm along the southerly edge of Area 5 crest (at the northerly crest of the Pit 3 highwall) will be enhanced and maintained as shown on Drawing 2-23 and on Detail 19 on Drawing 2-29. Material will not be stockpiled within 15 feet of this safety berm.

R2.7 PIT 4 – PIT BOTTOM CLEANUP AND GRADING

Sediments and coarse rock that has accumulated in the bottom of Pit 4 will be removed after completion of dewatering and prior to pit-bottom grading. The volume of pit-bottom sediments is estimated to be approximately 2,400 cy (MGC, 2011). Additional coarse rock material will result from the cleanup of gravel-to-boulder-sized material that currently exists on portions of the pit floor, as well as material that will be produced during pit-wall scaling, pit-bottom grading and underdrain sump excavation. Key components of the Pit 4 Bottom Cleanup and Grading STSP include:

- Pit-bottom sediments will be dried in place, either by natural evaporation or by adding drying agents once the pit has been dewatered. As a result, the majority of these sediments will be relatively dry and will be removed with common earth-moving equipment and transported to a temporary stockpile area on the relocated Ore Stockpile 7. Measures will be taken to avoid intermingling of this potentially higher-activity waste with the lower-activity mine waste once it is removed from the pit bottom.

  The temporary stockpile area used for storage of these low-moisture sediments from Pit 4 will be located in pits excavated into the upper surface of the relocated Ore Stockpile 7 (see Drawing 2-22). These pits will be lined with non-woven geofabric to prevent

migration of Pit 4 sediments into the underlying waste rock. These relatively dry sediments will be covered with approximately 1-foot of waste rock to avoid erosion by wind or water.

- Final cleanup of the Pit 4 bottom could require wash down of the remaining fine-grained materials to a low-lying sump area in the pit bottom where, if necessary, they will be pumped into geotubes for dewatering. The geotubes will be placed on a temporary pit-bottom sediment drying pad configured as shown on Detail 7 on Drawing 4-80. This temporary pit-bottom drying pad will be located adjacent to the ramp leading into the Pit 4 as shown on Drawing 4-12. This drying pad is located approximately 35-feet above the limits of the southerly end of the drain layer, so that the geotubes can be allowed additional time to dewater during final preparation of the pit bottom, placement of the drainage layer and geomembrane, and initial placement of mine waste backfill layers. With the geotubes placed in this location, any remaining water that drains from these geotubes will flow back into the pit bottom. Impacted water that passes through the geotube fabric will be relatively free from sediment.

  Once mine waste placement reaches the elevation of the temporary drying pad used for pit-bottom sediment, the geotubes and other materials associated with the drying pad will be placed in the central portion of the Pit 4 backfill lift in an area designated for placement of unclassified waste (including high reactivity and/or high activity wastes).

- Coarse rock material removed during pit bottom cleanup, as well as coarse waste rock spoils from pit-bottom sump excavation, will be placed directly on flat areas remaining on Protore Stockpile #6, the top surface of the relocated Ore Stockpile #7, or on top of the waste rock cover placed over dry sediments, as described above, to provide additional erosion protection.

R2.8 GROUNDWATER CONTROL SYSTEMS EXCAVATIONS

The groundwater control systems are discussed in Appendix G. As part of site preparation for construction of the groundwater control systems, level working platforms will be excavated in the three stream channels down gradient of the Mine Area as shown in Section 7 of the Drawings in Volume II. Key components of the Groundwater Control Systems Excavations STSP include:

- Spoils from site preparation excavations meeting the soil cleanup criteria will be stockpiled upgradient from the excavations and used for backfilling and final surface restoration. These spoils will not be stockpiled in or immediately adjacent stream channels or other natural drainage pathways.
- Any site preparation spoils not meeting soil cleanup criteria will be hauled directly to Pit 4 during the Phase 1 backfilling operation and incorporated into the mine waste backfill. If construction of the groundwater control systems occurs before the Pit 4 waste containment area has been adequately prepared for placement of waste, contaminated site preparation spoils will be stockpiled in the Phase 1 TISS.
- Spoils from the extraction trench and barrier wall excavations will be hauled to a stockpile location on the SWRP and incorporated into the Pit 4 mine waste backfill. If any of these excavation spoils are excessively wet, they may be temporarily stockpiled in non-woven geofabric-lined pits at the TISS. The non-woven geofabric will prevent migration of the sediments into the underlying waste rock at the TISS. These spoils will be allowed to dry before placement as backfill in Pit 4. A slag-cement-bentonite method of construction is proposed for barrier walls in the groundwater control systems. The spoils will be self-hardening and will be stockpiled for a few days to weeks prior to backfilling in Pit 4.

R2.9 PIT 3 – PIT BOTTOM CLEANUP AND GRADING

Sediments and coarse rock that has accumulated in the bottom of Pit 3 will be removed after completion of dewatering and prior to pit-bottom grading. The volume of pit-bottom sediments is estimated to be approximately 3,300 cy (MGC, 2011). Additional coarse rock material will result from the cleanup of gravel-to-boulder-sized material that currently exists on portions of the pit floor and additional material that will be produced during pit-grading and underdrain sump excavation. Key components of the Pit 3 Pit Bottom Cleanup and Grading STSP include:

- Pit–bottom sediments will be dried in place, either by natural evaporation or by adding drying agents once the pit has been dewatered. As a result, the majority of these sediments will be relatively dry and will be removed and transported to a temporary stockpile area on the East Waste Rock Pile using common earth-moving equipment. Measures will be taken to avoid intermingling of this potentially higher-activity waste with the lower-activity mine waste once it is removed from the pit bottom.

As now envisioned, these low-moisture sediments from Pit 3 temporarily will be placed in pits in the Phase 2 TISS near the existing WTP site, within the footprint of the existing Protore Stockpile No. 1 on the East Waste Rock Pile (see Drawing 4-1). These pits will be lined with non-woven geofabric to prevent migration of pit sediments into the underlying waste rock. The relatively dry sediments will be covered with approximately 1-foot of waste rock to avoid erosion by wind or water.

- Final cleanup of the pit bottom could require wash down of the remaining fine-grained materials to the low-lying sump area in the Pit 3 bottom where, if necessary, they will be pumped into geotubes for dewatering. The geotubes will be on a temporary pit-bottom sediment drying pad configured as shown on Detail 7 on Drawing 4-80. This temporary pit-bottom drying pad will be located on a bench above the ramp leading into the Pit 3 as shown on Drawing 4-37. This drying pad is located more than 50-feet above the upper limits of the drain layer to allow additional time for the geotubes to dewater during final preparation of the pit bottom, placement of drainage layer and geomembrane, and placement of initial mine waste layers in Pit 3. With the geotubes placed in this location, any remaining water that drains from the geotubes will flow back into the pit bottom. This impacted water that passes through the geotube fabric will be relatively free from sediment.

  Once mine waste placement reaches the elevation of the temporary pit-bottom sediment drying pad, the geotubes and other materials associated with the drying pad will be placed in the central portion of the Pit 3 backfill lift in an area designated for placement of unclassified waste (including high-activity and/or high-reactivity waste).

- Coarse rock material removed during pit bottom cleanup, as well as coarse waste rock spoils from pit-bottom sump excavation, will be placed directly on geofabric placed on flat areas in the TISS, or on top of the waste rock cover placed over dry sediments, as described above, to provide additional erosion protection.

R3.0 CONSTRUCTION STORMWATER POLLUTION PREVENTION PLAN

A Construction Stormwater Pollution Prevention Plan (CSWPPP) will be prepared in accordance with the Master Stormwater Management Plan (SWMP) in Appendix O. The CSWPPP will detail specific procedures and physical structures that will be implemented to prevent

discharges of turbid water to the surface water at and surrounding the temporary stockpile areas.

R4.0 REFERENCES

Miller Geotechnical Consultants (MGC), 2011. *Geologic Investigations of Pits and Assessment of Pit Sediments - Design Investigation Report, Revision 2.* Prepared on behalf of Newmont USA Limited and Dawn Mining Company for the U.S. EPA Region 10. April 28.

MWH Americas, Inc. (MWH), 2013. *Geotechnical Evaluation Report, IX Treatment Facility, Midnite Mine – Revision 0.* Prepared for Newmont USA Limited and Dawn Mining Company. May.

Midnite Mine Superfund Site
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Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments

This plan is intended to satisfy the requirement for a Confirmation Sampling and Remedial Action Effectiveness Monitoring Plan as stipulated by the Consent Decree.

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Prepared for:

Dawn Mining Company
PO Box 250
Ford, Washington 990413

and

Newmont USA Limited
6363 South Fiddler's Green Circle
Greenwood Village, Colorado 80111
Prepared By:

Prepared by:

Environmental Resources Group (ERG)
8809 Washington St NE, Suite 150
Albuquerque, New Mexico, 87113

TABLE OF CONTENTS

S.1.0 INTRODUCTION 1

S.2.0 ANALYTICAL APPROACHES 8
S.2.1 Field Gamma Surveys 8
S.2.1.1 Methodology 8
S.2.1.2 Instrumentation 9
S.2.1.3 Gamma/Ra-226 Correlation 10
S.2.2 Onsite Gamma Spectroscopy 13
S.2.2.1 Methodology 14
S.2.2.2 Instrumentation 14
S.2.2.3 System Calibration 14
S.2.3 Onsite X-ray Florescence (XRF) Analysis 16
Methodology 17
S.2.3.1 Instrumentation 18
S.2.3.2 Calibration 18
S.2.4 Offsite Laboratory Analysis 19
S.2.4.1 Offsite Laboratory Methods 20
S.2.4.2 Special Sample Handling/Analysis Protocols 21

S.3.0 REMEDIAL SUPPORT SURVEY PLAN 22
S.3.1 Gamma Scanning 22
S.3.1.1 Gamma Scanning for Excavation Support 22
S.3.1.2 Gamma Surveys for Characterization of Class 2 Areas and Borrow Materials 24
S.3.2 In-situ XRF Surveys 24
S.3.2.1 In-situ XRF Surveys for Excavation Support 24
S.3.2.2 In-situ XRF Surveys for Characterization of Class 2 Areas 25
S.3.3 Onsite Sample Analysis 26
S.3.3.1 Onsite Sample Analysis for Excavation Support 26
S.3.3.2 Onsite Sample Analysis for Characterization of Class 2 Areas 27
S.3.4 Decision Criteria for Remedial Support Surveys 28
S.3.4.1 Surface Materials 28
S.3.4.2 Sediments 29

S.4.0 FINAL STATUS SURVEY PLAN 30
S.4.1 Final Status Survey Units 31
S.4.2 Final Status Surveys for Surface Materials 36
S.4.2.1 Final Status Gamma Survey 36
S.4.2.2 Final Status Surface Material Sampling 38
S.4.2.3 Determination of Compliance for Surface Materials 39
S.4.3 Final Status Surveys for Sediments 44
S.4.3.1 XRF Surveys for Mine Drainages 44
S.4.3.2 Gamma Surveys for Mine Drainages 45
S.4.3.3 Sediment Sampling 45
S.4.3.4 Determination of Compliance for Sediments 46

S.5.0 DATA QUALITY ASSURANCE / QUALITY CONTROL (QA/QC) 51

S.6.0 REFERENCES 54

LIST OF FIGURES

Figure S-1 – Photo overlooking Pit 4 from near the northern boundary of the mined area. 1
Figure S-2 – Mined area features (EPA, 2006a). 2
Figure S-3 – Equivalent uranium (eU) concentrations (pCi/g) in surface materials based on aerial gamma survey measurements (EPA, 2011). 2
Figure S-4 – Generalized mine waste removal, analytical assessment and decision diagram for remediation of surface materials (note: CL = cleanup level). 6
Figure S-5 – Generalized mine waste removal, analytical assessment and decision diagram for remediation of sediments (note: CL = cleanup level). 7
Figure S-6 – Example backpack scanning system configuration. 10
Figure S-7 – Correlation plot soil sampling and gamma scanning design. 12
Figure S-8 – Essential elements of a NaI-based gamma spectroscopy counting system for soil samples. 15
Figure S-9 – Example onsite soils lab setup including sample processing and gamma counting stations in a temporary, portable onsite trailer. 15
Figure S-10 – Class 1 zones delineated by remedial schedule. 34
Figure S-11 – Class 1 and Class 2 survey units. 35
Figure S-12 – In-situ XRF survey transects and laterally alternating direct soil sampling locations for onsite intrusive XRF analysis and offsite laboratory analysis. 44

LIST OF TABLES

Table S-1 – Cleanup Levels for Midnite Mine Surface Material (from ROD Table 8-3) 3
Table S-2 – Cleanup Levels for Midnite Mine Sediments (from ROD Table 8-4) 3
Table S-3 – Approximate Percentages of Samples to be Analyzed in the Onsite Soils Lab and Offsite at a Commercial Laboratory 19
Table S-4 – Analytical Schedule for Samples to be Analyzed Offsite at a Commercial Laboratory 20
Table S-5 – Analytical Parameters, Cleanup Levels, Analysis Methods and Detection Limits for Surface Materials 20
Table S-6 – Analytical Parameters, Cleanup Levels, Analysis Methods and Detection Limits for Sediments 21
Table S-7 – Calculated U-238 and U-234 Concentrations in Impacted Sediments if Natural (total) Uranium is Present at the ROD Cleanup Level for Uranium 49

LIST OF ATTACHMENTS

Attachment S-1 – Technical Basis

Attachment S-2 – Quality Assurance Project Plan for Remedial Action Analytical Support and Verification (QAPP)

Attachment S-3 – Determination of Bedrock During Remedial Excavation

LIST OF ACRONYMS

CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
cm	centimeter
COC	Constituents of Concern
DOE	US Department of Energy
DQO	Data Quality Objective
EPA	US Environmental Protection Agency
eU	equivalent uranium
g	gram
GIS	geographical information systems
GPS	global positioning system
HPGe	high-purity germanium
kg	kilogram
kV	kilovolt
MAA	Mining Affected Areas
MARSSIM	Multi-agency Radiation Site Survey and Investigation Manual
MCA	multi-channel analyzer
MDC	minimum detectable concentration
mg	milligram
NaI	sodium iodide
NRC	Nuclear Regulatory Commission
NPL	National Priorities List
NUREG	U.S. Nuclear Regulatory Commission regulation
pCi	picocuries
QA/QC	Quality Assurance / Quality Control
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
RI/FS	Remedial Investigation/Feasibility Study
ROD	Record of Decision
ROI	regions of interest
SMI	Shepherd Miller, Inc.
Site	Midnite Mine Superfund Site
SOP	Standard Operating Procedure
UPL	upper prediction limit
µR/h	microroentgen per hour
URS	URS Corporation
VSP	Visual Sample Plan

WAAS	Wide Area Augmentation System
XRF	X-ray fluorescence

S.1.0 INTRODUCTION

The Midnite Mine (Site), an inactive open pit uranium mine, is located in the southern reaches of the Selkirk Mountains on the Spokane Indian Reservation approximately 45 miles northwest of Spokane, Washington. The mine operated between 1954 and 1981. The topography across the mined area has significant vertical relief (Figure S-1), ranging in elevation from 2,400 to 3,570 feet. The Midnite Mine and impacted areas were added to the U.S. Environmental Protection Agency's (EPA) National Priorities List (NPL) in 2000 (EPA, 2006a). The Site is subject to Superfund cleanup under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA).

A map of Site features is shown in Figure S-2. In 2011, an aerial radiometric survey was flown at an altitude of 500 feet over the Site to measure the spatial distribution of terrestrial gamma radiation, and to estimate a corresponding distribution of uranium concentrations in terrestrial materials residing at the ground surface (Figure S-3) (EPA, 2011). A detailed ground-based radiological survey was conducted in 1999 (SMI, 1999a and 1999b) and this survey included measurements of gamma exposure rates along with estimated concentrations of uranium, radium-226 (Ra-226) and thorium-230 (Th-230) in surface materials across the site and adjacent undisturbed areas.

The EPA's Record of Decision (ROD) for the Midnite Mine (EPA, 2006a) requires consolidation of above-ground mine waste deposits and impacted soils/sediments into Pits 3 and 4. The current estimate of the total volume of materials to be consolidated into Pits 3 and 4 is approximately 18,750,000 cubic yards (Miller Geotechnical, 2011). The cleanup levels specified in the ROD for surface materials and sediments[1] are shown in Tables S-1 and S-2.

Figure S-1 – Photo overlooking Pit 4 from near the northern boundary of the mined area.

[1] These cleanup levels are generally based on upper 95% tolerance limits on "background" samples from nearby, non-impacted reference areas believed to have environmental characteristics similar to background conditions in the mined areas prior to mining disturbance (URS, 2005).

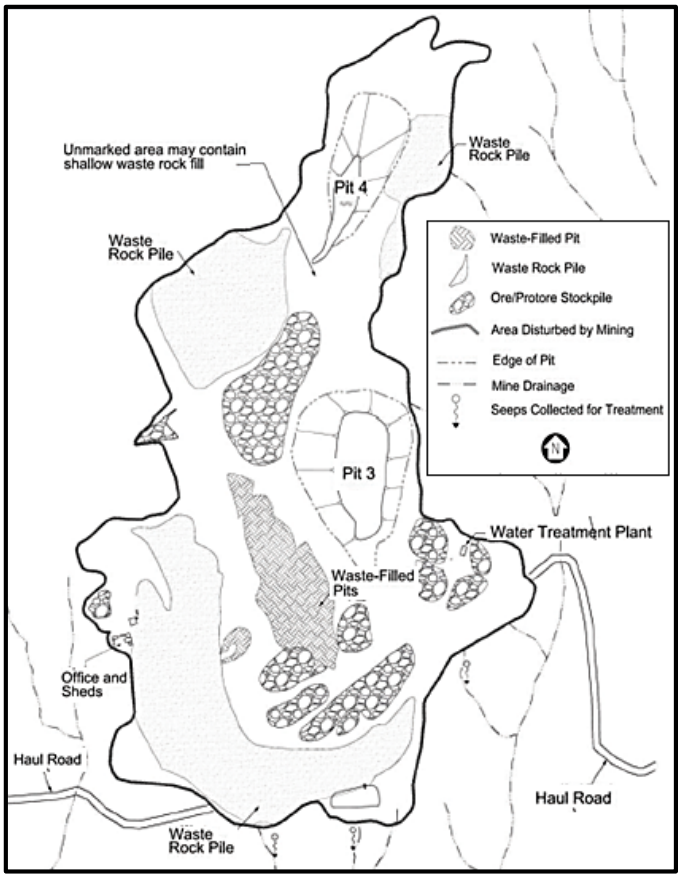

Figure S-2 – Mined area features (EPA, 2006a).

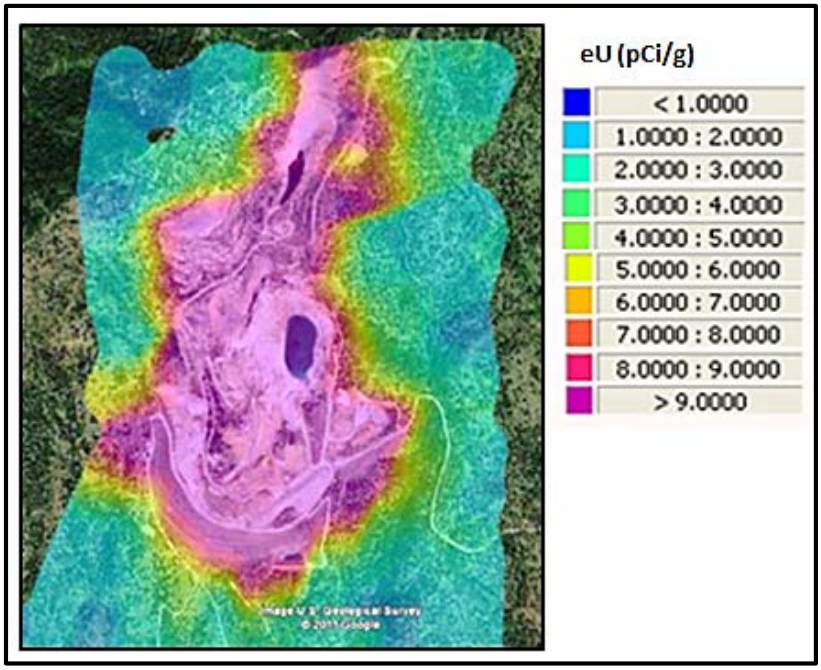

Figure S-3 – Equivalent uranium (eU) concentrations (pCi/g) in surface materials based on aerial gamma survey measurements (EPA, 2011).

Table S-1 – Cleanup Levels for Midnite Mine Surface Material (from ROD Table 8-3)

Contaminant of Concern	Cleanup Level
Uranium (total)[2]	43 mg/kg
Lead-210	7.5 pCi/g*
Radium-226	4.7 pCi/g

*Note: Table 8-3 lists the units for Lead-210 as pCi/kg. It is assumed this is a typographical error and correct units should be pCi/g as stated in Table 8-4.

Table S-2 – Cleanup Levels for Midnite Mine Sediments (from ROD Table 8-4)

Contaminant of Concern	Cleanup Level
Lead-210	20 pCi/g
Uranium-238	31 pCi/g
Uranium-234	41 pCi/g
Radium-226	13 pCi/g
Chromium	43.4 mg/kg
Manganese	1,179 mg/kg
Selenium	1.7 mg/kg
Uranium (total)	93.2 mg/kg
Vanadium	41 mg/kg

A 2010 investigation of mine waste deposits, underlying soils, local access roads, and local drainage sediments (Miller Geotechnical, 2011), indicates that of the Constituents of Concern (COC) specified in the ROD for surface materials (Table S-1 above), one or more of these COCs were found to exceed respective cleanup criteria in soils underlying mine waste deposits and/or along access roads. With respect to sediments, exceedances of cleanup criteria (Table S-2 above) along local drainage channels were limited to one or more of the following COCs: uranium (total and/or isotopic), radium-226 (Ra-226), vanadium and manganese (Miller Geotechnical, 2011).

The purpose of this Analytical Support and Verification Plan (Plan) is to detail the analytical approaches and methods that will be used to support remedial action for surface materials and sediments at the Midnite Mine Superfund Site (Site), and to provide comprehensive plans for how those approaches will be applied in terms of guiding excavations and verifying compliance with ROD cleanup levels. This Plan pertains only to remediation or potential remediation of surface materials and sediments in impacted or potentially impacted areas, and has been

[2] Throughout this document, “uranium” refers the natural (total) form unless an isotopic form (e.g. U-238) is specified.

designed to meet respective data quality objectives (DQOs) for the project. Appendix S does not apply to the Blue Creek contingency cleanup action.

The Plan includes four basic analytical approaches that will be used to determine the necessary horizontal and vertical extent of remediation of surface materials and sediments (remedial support surveys), and to demonstrate compliance with respective ROD cleanup levels (final status surveys). These analytical approaches include:

1. Field gamma surveys and gamma/soil Ra-226 correlations.
2. Onsite analysis of Ra-226 concentrations using sodium iodide (NaI)-based gamma spectroscopy.
3. Onsite analysis of the concentrations of metals based on X-ray Florescence (XRF) measurements.
4. Offsite analysis of all ROD cleanup parameters for surface materials and sediments at an approved commercial laboratory.

The technical bases for these approaches, along with the rationale for the statistical methods to be used for evaluating compliance, are provided in Attachment S-1. Selection was based on the DQO process for remediation of surface materials and sediments. The seven steps of the DQO process are provided in the Quality Assurance Project Plan for Remedial Action Analytical Support and Verification (QAPP) (Attachment S-2). Each of the four analytical approaches above will be used to accomplish two basic analytical objectives (remedial support surveys and final status surveys), but the manner in which they will be applied differs for each objective.

Generalized flowchart overviews of the Plan for remediation of surface materials and sediments, including mine waste removal[3], analytical assessment and decision criteria, are depicted in Figures S-4 and S-5. These generalized diagrams are based on the DQOs for remediation of surface materials and sediments. Comprehensive details and additional technical considerations, supporting information and requirements are provided in this Plan along with its supporting attachments (Attachments S-1 – S-3). To facilitate simplicity in citation throughout this document, the supporting attachments, respective appendices and associated content that are referenced where applicable in this Plan are outlined as follows:

[3] All above ground mine waste will be removed and placed within the waste containment area. After the above grade mine waste has been removed, remedial support surveys and ultimately final cleanup verification surveys will be conducted in accordance with the procedures outlined in Appendix S.

- Attachment S-1 – Technical Basis
- Attachment S-2 – Quality Assurance Project Plan (QAPP)
    - o Appendix 1 – Standard Operating Procedures (SOPs)
        - AS-SOP 1 – Decontamination for Field Sampling
        - AS-SOP 2 – Surface Material and Sediment Sampling
        - AS-SOP 3 – Sampling Processing
        - AS-SOP 4 – Onsite Gamma Spectroscopy
        - AS-SOP 5 – Field-portable XRF Procedures
        - AS-SOP 6 – Gamma Surveys
    - o Appendix 2 – Corrective Action Report Form
    - o Appendix 3 – Approved Laboratory Quality Assurance Plans
    - o Appendix 4 – Laboratory Certification
- Attachment S-3 – Determination of Bedrock during Remedial Excavation

This document is organized to first provide basic details of the analytical approaches that will be employed for the project (Section S2.0), and to then provide specific detailed information on how the approaches will be applied for remedial support surveys (Section S3.0) and final status surveys (Section S4.0). Data quality assurance/quality control (QA/QC) provisions are summarized in Section S5.0, and the DQOs and details of a comprehensive QA/QC program for implementation of this Plan are provided in the QAPP.

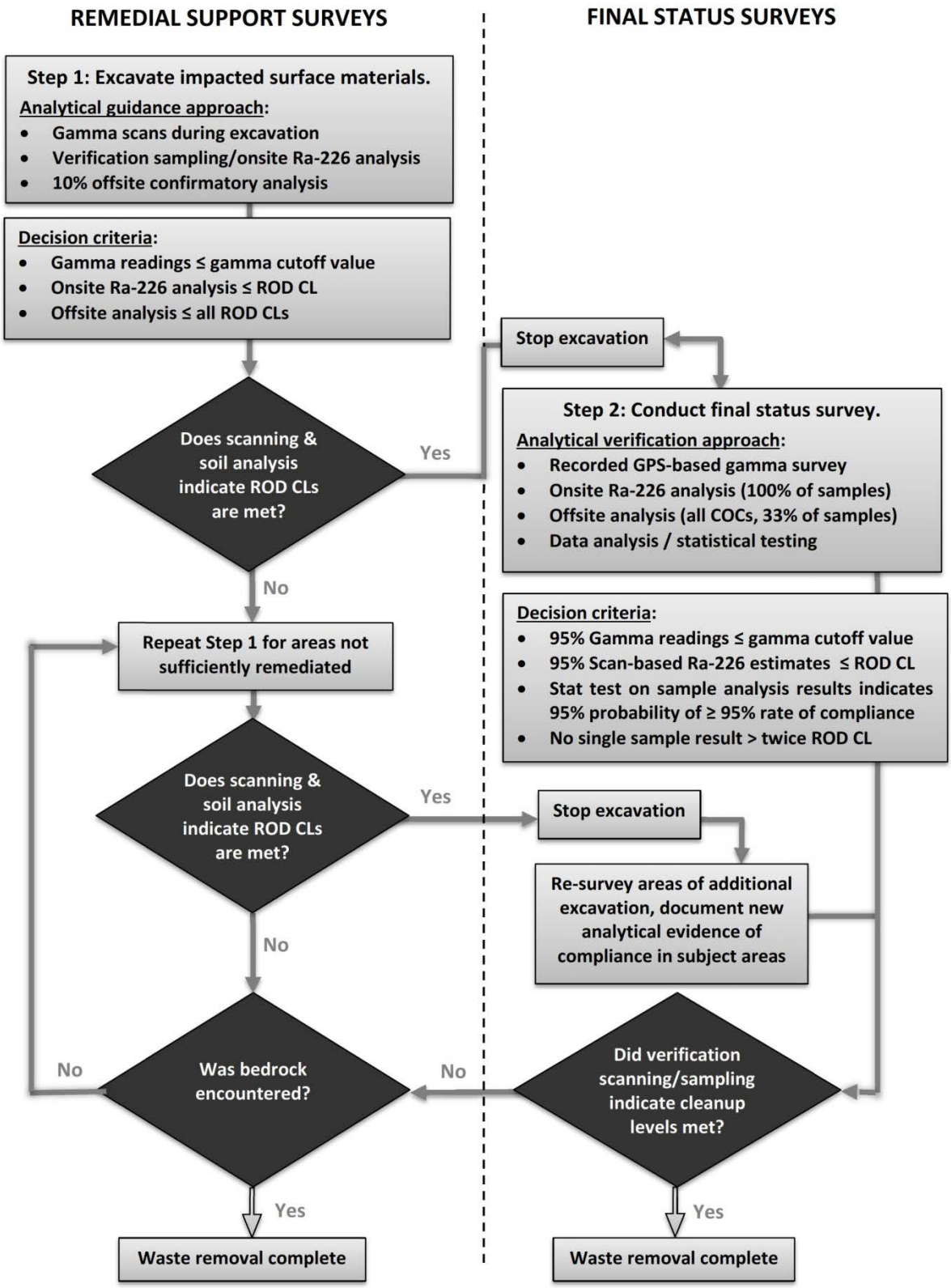

Figure S-4 – Generalized mine waste removal, analytical assessment and decision diagram for remediation of surface materials (note: CL = cleanup level).

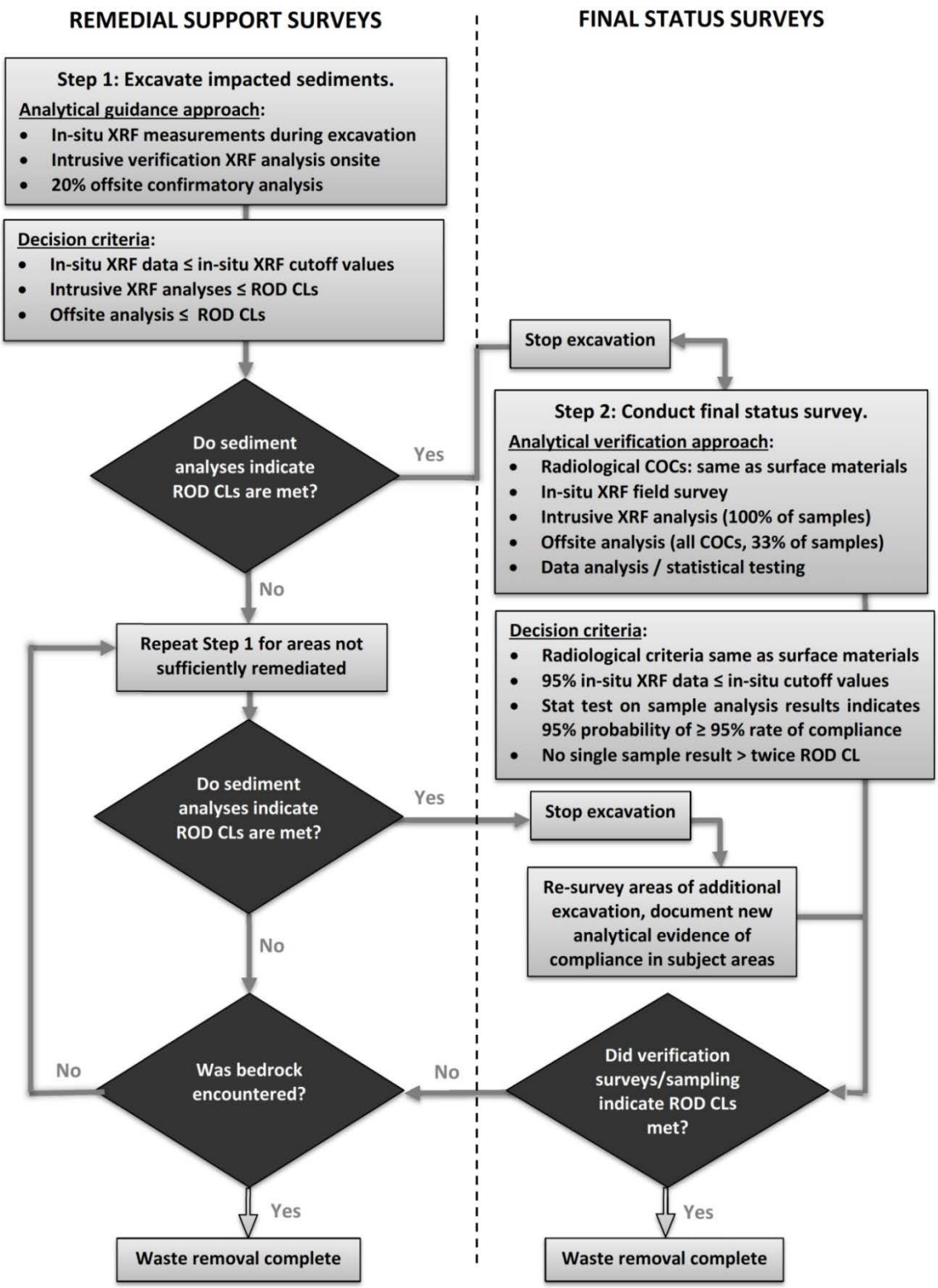

Figure S-5 – Generalized mine waste removal, analytical assessment and decision diagram for remediation of sediments (note: CL = cleanup level).

S.2.0 ANALYTICAL APPROACHES

This section describes common elements of the analytical approaches that will be used for both remedial support and final status surveys. Application of these approaches varies depending on the analytical objective (remedial support or final status surveys) and on the remedial area of focus (surface materials or sediments). Information concerning the different applications and remedial areas of focus are included in this Section where appropriate to aid with clarity. Full details of how these analytical approaches will be applied for each analytical objective and remedial area of focus are provided in Sections S3.0 and S4.0.

S.2.1 Field Gamma Surveys

Gamma surveys will be conducted in the field to indirectly evaluate Ra-226 concentrations in surface materials and sediments residing at or near the ground surface. The statistical relationship between gamma readings and Ra-226 concentrations at the ground surface will be the basis for probabilistic gamma cutoff values (screening levels) that will be used to screen the ground surface for compliance with the ROD cleanup levels for Ra-226 in surface materials (4.7 pCi/g) and sediments (13 pCi/g).

Initial gamma cutoff values for surface materials and sediments have been established (20 µR/hr and 33 µR/hr respectively) based on previous Site correlation data (Attachment S-1). These values may be revised over time as additional correlation data are developed throughout the cleanup. The technical basis for the use of gamma scanning, gamma/Ra-226 correlations, and gamma cutoff values at the Site (Attachment S-1) was developed as part of the DQO process. Corresponding elements of the Plan are summarized in the DQO statements provided in the QAPP. A Standard Operating Procedure (SOP) for gamma surveys is provided in AS-SOP 6.

S.2.1.1 Methodology

While the approaches, DQOs and data evaluation criteria described above are applicable to all gamma surveys at the Site, the exact gamma scanning methodology to be employed depends on whether the scanning is conducted for purposes of remedial support surveys, or for final status surveys. Gamma scanning for remedial support will generally not be recorded, while all final status gamma surveys will be officially recorded to provide a permanent final record of gamma radiation conditions across remediated areas. Major commonalities of the methods for either objective include the exclusive use of a specified gamma detector/rate meter Model

pairing (Section S2.1.2), and that a detector scan height of 1 meter above the ground surface will be used to evaluate respective readings against the gamma cutoff value.

For remedial support surveys, a properly trained field technician will manually monitor and guide the vertical and horizontal extent of excavations required based on comparisons of readings in the excavated areas against the gamma cutoff value. For this routine remedial support scanning, the only instrumentation that will generally be needed is a properly calibrated gamma detector/rate meter pairing as specified in Section S2.1.2.

Recorded gamma surveys will utilize the same modern technologies/methods that have become standard health physics practice for radiological characterization and remedial applications across the U.S. and abroad (e.g. Adsleya et al., 2004; Johnson et al., 2006; Meyer et al., 2005; Vitkus et al., 2007; Whicker et al., 2008). This includes use of GPS-based gamma scanning systems with automated electronic data acquisition software and associated methods for data collection, mapping, quantitative and spatial analysis, and data interpretation. The 2010 Mine Waste Investigations study at the Site included gamma surveys of haul roads and mine drainage channels using these same technologies and methods (Miller Geotechnical, 2011).

Additional specific details of how these gamma survey methods will be used to meet remedial support and final status survey objectives are provided in Sections S3.0 and S4.0 respectively. Generalized procedures for gamma surveys are provided in AS-SOP 6.

S.2.1.2 Instrumentation

A specific technical requirement for all gamma survey measurements conducted for the purposes of this Plan (whether for remedial support or final status surveys) is that a properly calibrated Ludlum Model 44-10 gamma detector (NaI-based scintillometer with 2” x 2” NaI crystal) coupled with Ludlum Model 2350 rate meter must be used for consistency with the instrument pairings used for previous gamma surveys at the Site (e.g. SMI, 1999a; Miller Geotechnical, 2011). The validity of the initial gamma/Ra-226 correlation and associated gamma cutoff values, as well as comparisons of gamma survey data with earlier study data, is dependent on this consistency.

For recorded surveys, each scanning system will be mounted on a backpack with the detector positioned at about one meter above the ground surface (Figure S-6). Maintaining consistency in this detector height may require backpack mounting adjustments for field technicians of different personal height. The GPS receiver will be mounted at the top of the backpack with a clear view of the sky. GPS receivers will be Wide Area Augmentation System (WAAS)-enabled

to help ensure adequate spatial accuracy (typically within ± 3-5 meters of the true location on the Earth's surface[4]). Each scanning system will be coupled to a field computer with appropriate data acquisition software. The mounting system configuration may be modified to suit site conditions, but detector height will be maintained as specified and the functionality of the basic system will not change.

Figure S-6 – Example backpack scanning system configuration.

S.2.1.3 Gamma/Ra-226 Correlation

Based on the analysis provided in Attachment S-1, initial gamma cutoff values to evaluate excavated areas for compliance with cleanup levels for surface materials and sediments will be 20 µR/hr and 33 µR/hr respectively. Early in the cleanup sequence, additional gamma/soil Ra-226 correlation data will be collected to verify the validity of the initial correlation and respective gamma cutoff value, and to update these assessment criteria as warranted. It should be noted that recent gamma surveying to in the Whitetail Creek area indicated that cutoff values may be impacted by underlying geologic conditions. If this is the case, it is possible that different cutoff values will be used for different areas depending on the underlying geology. Necessary

[4] Based on extensive experience with these scan systems, once GPS readings are initiated for a given scan run, the precision of subsequent GPS measurements relative to the initial location fix is generally expected to be on the order of ± 1 meter in terms of true distance and direction. Normal GPS accuracy and precision can be adversely affected in forested areas or in narrow canyons.

conditions for collecting additional correlation data include a lack of significant gamma shine (see Attachment S-1) and relatively uniform soil Ra-226 concentrations across areas to be sampled and scanned. Such conditions become more likely as excavations proceed and materials with higher gamma activity are removed from a given area resulting in lower Ra-226 concentrations in surface materials (closer to the 4.7 pCi/g cleanup criterion). The protocols for collection of additional correlation data are as follows:

1. Review the initial correlation data provided in Figure 1 of Attachment S-1 and select an appropriate location in an excavated area for a new correlation plot in which ambient gamma readings have fallen to a relatively consistent level somewhere in the range between 10-60 µR/hr, and where this level is spatially uniform well beyond the boundaries of the proposed correlation plot. Where possible, levels selected for new correlation data should attempt to fill in data gaps in the initial correlation data set (i.e. attempt to target areas with uniform gamma levels near 15, 28, 45 and 60 µR/hr), though the greatest emphasis should be placed on plots expected to have Ra-226 concentrations near the cleanup criterion (i.e. below 40 µR/hr, see Figures 1 and 2 in Attachment S-1).
2. Establish a 100 m^2 plot in the selected area and scan to obtain the average gamma exposure rate across the plot (100% scan coverage).
3. Obtain 9 sub-samples (about 40 grams each) of surface soils across the plot (to a depth of 15 cm), and composite the 9 sub-samples into a single sample (about 350 grams). The procedures for soil sampling are provided in AS-SOP 1 and AS-SOP 2. The scanning/soil sampling design for correlation plots is shown in Figure S-7.
4. Deliver correlation samples to the onsite soils lab for processing in accordance with AS-SOP 3.
5. Perform Ra-226 analysis using NaI-based gamma spectroscopy as described in Section 2.2 and in accordance with the respective procedures indicated in AS-SOP 4. Keep the samples canned/sealed after counting in accordance with AS-SOP 3.
6. For the fraction of samples to be sent to the offsite laboratory for subsequent analysis (10% or more as directed by the Field Supervisor and/or Field Program Director), send the sealed/canned correlation samples to the approved offsite laboratory per the specifications of Section 2.4.2, along with Section B.3.2 of the QAPP.
7. Add each new gamma/soil Ra-226 correlation plot data pairing to the initial correlation data set and evaluate for consistency and update the gamma cutoff value as warranted.

Disregard any obvious outliers that may result from gamma shine or "hot particles"[5], or that are otherwise clearly non-representative relative to the majority of the correlation data.

The regression equation for the gamma/Ra-226 correlation may be periodically updated throughout the cleanup as more correlation data are generated. The regression equation in use at the time recorded gamma survey data are collected will be applied accordingly (i.e. scan data collected and evaluated early in the cleanup will not subsequently be re-evaluated based on any later revisions to the gamma/Ra-226 correlation and respective gamma cutoff value). Any revision of the regression equation based on updated correlation data (and respective revision to the gamma cutoff value) will be assigned an effective date of use moving forward and this date will be documented. Because all recorded gamma scan data include a date stamp, the proper regression equation and gamma cutoff value can be used to evaluate scan data collected at any given period of the cleanup sequence. The technical basis for this protocol is detailed in Attachment S-1 (Section 3.1).

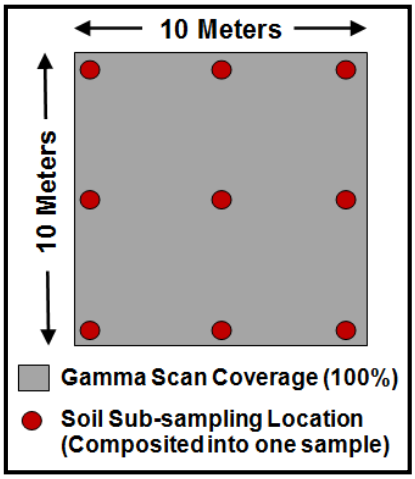

Figure S-7 – Correlation plot soil sampling and gamma scanning design.

As indicated in Section 3.1 of Attachment S-1, the cleanup level for Ra-226 in surface materials (4.7 pCi/g) is almost exclusively limiting relative to cleanup levels for other COCs in surface materials (uranium and Pb-210), meaning that in virtually all cases, remediation to meet the Ra-

[5] In a context of correlation plot soil sampling, the term "hot particles" refers to a circumstance in which one or more small, ore-grade mineralized rocks (e.g. less than 0.5-inch diameter) with anomalously high Ra-226 concentrations relative to the bulk soil sample, skew the analytical results for the soil sample in a manner that is not representative of the vast majority of soils across the correlation plot. A few tiny ore rocks cannot be detected in-situ in the field above ambient background readings with gamma scanning, but if by random chance one or more of them happen to be contained in a composite soil sample from the plot, they can easily be detected by gamma spectroscopy analysis in a lead-shielded counting well.

226 cleanup level will simultaneously ensure that cleanup criteria for other COCs are also achieved. In a small percentage of cases, remediation to the Ra-226 cleanup level may not result in compliance with the uranium cleanup level (43 mg/kg). Based on data obtained in the Mine Waste Investigation (Miller Geotechnical, 2011), this situation might occur in about 5 percent of sampled locations, very close to a conceptually analogous limit of 5 percent on Type I decision errors ($\alpha = 0.05$). To provide additional assurance that a 95 percent rate of compliance will be met, XRF screening for uranium concentrations will be conducted for soil samples (see Section S.2.2.3) collected in areas with a higher likelihood of significant radiological disequilibrium between uranium and Ra-226 (e.g. as suggested by data provided in the Mine Waste Investigations report).

Each day that a given detector/rate meter pairing will be used for gamma survey screening, instrument quality control measurements will be performed to ensure that the system is functioning properly and to quantify instrument precision and natural temporal variability in ambient gamma radiation due to fluctuations in atmospheric or other conditions (e.g. changes in barometric pressure, soil moisture, indoor radon decay products, etc.). Data Quality Assurance / Quality Control (QA/QC) procedures for both remedial support surveys and final status surveys are provided in the QAPP.

S.2.2 Onsite Gamma Spectroscopy

In addition to evaluating remedial progress and ultimate remedial effectiveness based on gamma surveys across applicable land areas, gamma emissions from individual soil samples will be used to directly estimate soil Ra-226 concentrations in an onsite soils lab (see Attachment S-1, Section 3.5). The primary utility of this onsite analytical capability includes the following:

1. Provides near real-time verification that the gamma-cutoff value for remedial support surveys accurately predicts compliance with the soil Ra-226 cleanup criterion throughout the cleanup.
2. Allows additional correlation sampling/measurements to be performed during the cleanup in order to update correlation data and respective statistical relationship, and refine the gamma cutoff value as needed.
3. Directly evaluate soil Ra-226 concentrations in areas where gamma shine is suspected of negatively influencing the accuracy of the gamma cutoff value.

4. Provide supplemental soil Ra-226 analysis data as part of final status surveys (Section A.3.2.3).

S.2.2.1 Methodology

The analytical methodology for sample Ra-226 analysis in an onsite soils lab will be generally based on that which was used for 2005 cleanup activities at the Dawn Mill Site (Whicker et al., 2006). The methodology relies on NaI-based gamma spectroscopy analysis of samples placed in a lead-shielded counting well (along with the NaI detector) during counting to help block ambient background radiation that is not associated with the sample from reaching the NaI detector. The method can yield highly sensitive measurements of gamma emissions from a relatively small sample of soil (e.g. 150-200 grams). Method detection limits for soil Ra-226 concentrations have previously been calculated at about 0.75 pCi/g (Whicker et al., 2006), well below the 4.7 pCi/g cleanup criterion for the Midnite Mine.

All samples will be dried, homogenized and weighed prior to onsite Ra-226 analysis, with 150-200 gram aliquots placed in special soil counting cans that will be sealed prior to counting (Figure S-9). Procedures for sample processing in the onsite soils lab are provided in standard operating procedure AS-SOP 3. Further information and details of the onsite Na-based gamma spectroscopy methodology are provided in standard operating procedure AS-SOP 4 and in Attachment S-1 (Section 3.5).

S.2.2.2 Instrumentation

The essential elements of the system will include a 3×3 inch NaI detector, coupled to a PC-based multi-channel analyzer (MCA) (Figure S-8). The counting well will consist of lead rings and plates, arranged such that both sample and detector are adequately shielded from background radiation during counting. The system to be used at the Midnite Mine will be identical or functionally equivalent to that used at the Dawn Mill Site in 2005.

S.2.2.3 System Calibration

The key to the effectiveness of the onsite gamma spectroscopy approach is system calibration against site-specific soil Ra-226 calibration standards. These calibration standards will be developed based on samples collected onsite early in the cleanup. These samples will be sieved, dried, weighed, canned and evaluated by onsite spectroscopy analysis to establish three energy regions of interest (ROIs) as detailed in Section 3.5 of Attachment S-1. Once ROIs are established the calibration standards will be sealed and counted the same day (day zero counts). Day zero counts in each ROI will be summed for use in a single overall calibration

algorithm (described below). The canned/sealed calibration standards will then be sent to a qualified commercial laboratory for Ra-226 analysis by gamma spectroscopy after full radon ingrowth using a high-purity germanium (HPGe) detector (EPA Method 901.1, modified for soil samples).

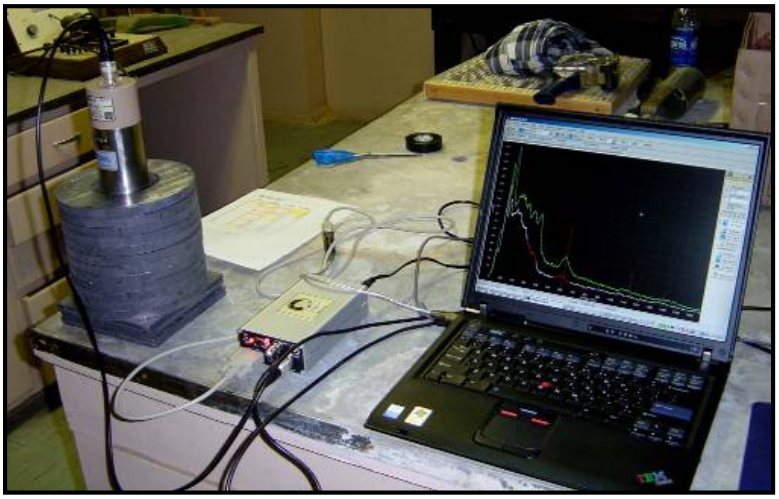

Figure S-8 – Essential elements of a NaI-based gamma spectroscopy counting system for soil samples.

Figure S-9 – Example onsite soils lab setup including sample processing and gamma counting stations in a temporary, portable onsite trailer.

Regression analysis will be performed on paired results from the onsite lab and official HPGe results from the commercial laboratory in order to determine one or more statistical regression equations for use in an overall calibration algorithm. The calibration algorithm for use in analyzing field samples may involve two separate regressions, one for a higher range of field

sample values (e.g. 10 to 50+ pCi/g) and another for a low range of field sample values that are near the Ra-226 cleanup level (e.g. up to about 10 pCi/g). The low end calibration relationship may not be linear as the "signal to noise" ratio becomes more limiting at very low Ra-226 concentrations. In general, a linear regression on all calibration data should have a statistical coefficient of determination (R^2) value well above 0.95, but in the low range of values, where the "signal to noise" ratio is likely to be lower, an R^2 value of 0.75 can prove to be effective. What matters most for the calibration algorithm is not the statistical R^2 value on the relationship, but a demonstrated ability to produce data with levels of accuracy and precision similar to that of HPGe analysis at the offsite lab (e.g. Whicker et al., 2006). Throughout this Plan and its associated attachments, use of the term "calibration algorithm" in association with gamma spectroscopy for Ra-226 analysis in the onsite soils lab refers to the above definition.

The calibration algorithm will be used to estimate "full-ingrowth" Ra-226 concentrations in field samples based on combined day zero counts from the three ROIs (see Attachment S-1, Section 3.5). Throughout the cleanup, at least 10 percent of soil samples analyzed in the onsite lab will also be sent to a commercial laboratory for Ra-226 analysis (after full radon ingrowth)[6]. These results will be used to evaluate the performance of the calibration, evaluate the accuracy of onsite results (with T-tests, Wilcoxon Rank Sum and ANOVA tests), and to update the calibration algorithm as appropriate. Data QA/QC procedures for onsite gamma spectroscopy measurements are provided in AS-SOP 4 and quality control criteria are specified in the QAPP.

S.2.3 Onsite X-ray Florescence (XRF) Analysis

Onsite XRF analysis approaches based on EPA Method 6200 will be used to help evaluate COCs other than Ra-226. This approach is primarily applicable for estimating the concentrations of metals in sediments, but may also be used to evaluate uranium concentrations in surface materials. The basic methodology is analogous to calibrating field gamma survey readings against soil Ra-226 analysis results from a commercial laboratory (i.e. gamma/Ra-226 correlations), as well as calibrating onsite Ra-226 analysis by gamma spectroscopy against paired analysis results from an offsite commercial laboratory.

[6] This minimum percentage refers to samples collected for remedial support purposes, and these samples will also be analyzed for uranium and Pb-210 at the offsite laboratory. For final status surveys, 33% of all samples analyzed in the onsite soils will subsequently be sent to the offsite laboratory for analysis of all COCs.

Methodology

Site-specific XRF Method Calibrations

At the initiation of the project, Site-specific method calibrations of both field in-situ XRF measurements and intrusive XRF analysis (in the onsite soils lab) versus analytical results from the offsite laboratory will be developed based on linear regression analysis of respectively paired data. The XRF methodology will follow EPA Method 6200 (Attachment A to AS-SOP 5). Specific XRF procedures for this project are provided in AS-SOP 5.

To develop these onsite method calibrations, in-situ XRF measurements will be conducted at the sediment (or soil) surface (in-situ XRF), and samples will be collected at 20 percent of the field in-situ measurement locations for both onsite intrusive XRF analysis and offsite laboratory analysis. The samples will be collected systematically at every fifth in-situ location. Additional samples and measurements may be made at the discretion of the field team based upon variability in concentrations at the site, with the intent to collect samples from locations representing the full range of the in-situ XRF metals measurements, specifically for manganese (which appears to be the limiting COC for sediments), and for uranium (which will be the focus of XRF measurements for surface materials).

Samples will be collected following the procedures in AS-SOP 1 and AS-SOP 2 for sample collection, and subsequently processed in the onsite soils lab following the procedures in AS-SOP 3. The samples will be specifically prepared and analyzed with the intrusive XRF method following the procedures provided in AS-SOP 5, which includes drying, grinding, and sieving through a 60-mesh sieve. The sieved sample will then be homogenized and placed in a soil cup for intrusive XRF analysis. The soil cup will then be sent to an analytical laboratory for analysis of applicable metals as indicated in Table S-2.

The results from the offsite laboratory analysis will be paired with corresponding intrusive XRF analysis results, as well as with the in-situ XRF analysis results, and then evaluated with least squares regression analysis. The correlation coefficient (r) for the results should be 0.7 or greater for the XRF data to be considered screening level data, or 0.9 or greater for definitive level data. Where a respectively significant statistical calibration (regression) curve can be effectively established for each metal concentration, the results of future in-situ XRF field measurements or intrusive XRF analyses in the onsite soils lab will be adjusted using the corresponding method calibration (regression) equation to estimate the metal concentration at the location or for the sample in question.

Should XRF correlation coefficients be smaller than can be considered acceptable, traditional soil sampling and offsite analysis at a commercial laboratory will be used to determine compliance with clean-up criteria for metal parameters in accordance with the DQOs established for this Plan (see QAPP).

Operational XRF Field Measurements and Sample Analysis

Like gamma cutoff values for evaluation of compliance Ra-226 cleanup levels based on gamma survey data, in-situ XRF cutoff values will be developed based on the correlation between in-situ measurements and analytical results from the commercial laboratory. These XRF cutoff values will be used to evaluate compliance with ROD cleanup levels for metals (particularly manganese) based on in-situ XRF survey data and to respectively guide excavation decisions. XRF cutoff values will be analyte specific, and will be based on 95 percent upper prediction limits (UPLs) on respective regression relationships. This conservative approach will provide a high degree of confidence that XRF surveys with in-situ measurements will result in compliance with ROD cleanup levels for metals.

In addition, all in-situ XRF surveys will include random and biased sampling at about 5 percent of measurement locations for confirmatory analysis in the onsite soils lab with the intrusive XRF methodology. Both XRF methods will be performed according to the specifications of Method 6200 and procedures found in AS-SOP 5. Provided that XRF analysis with the intrusive method is demonstrated to produce definitive level data, the concept of a cutoff value will not be employed for evaluation of intrusive XRF analysis results. Further details of the operational application of in-situ XRF surveys and intrusive XRF sample analysis in the onsite soils lab for remedial support and final status surveys are provided in Sections S3.0 and S4.0.

S.2.3.1 Instrumentation

Instrumentation for onsite XRF analysis will involve a field-portable, ThermoFisher Scientific Niton XL3t GOLDD+ (or equivalent model) XRF Analyzer. The Niton XL3t instrument uses a 50 kV X-Ray tube source for the analysis of inorganic metal concentrations. Operation will follow the ThermoFisher Scientific Inc. User's Guide for the instrument (Attachment B to AS-SOP 5).

S.2.3.2 Calibration

The XRF instrument will be calibrated daily prior to use for both the in-situ analysis and the intrusive analysis methods, according to procedures in the manufacture's user guide (Attachment B to AS-SOP 5) using calibration checks with certified reference materials and field

blanks as described in EPA Method 6200. QC samples will also be analyzed prior to sample analysis, as described in AS-SOP 5.

S.2.4 Offsite Laboratory Analysis

As previously described, all samples collected will be analyzed onsite for Ra-226 by gamma spectroscopy. The percentage of samples to be analyzed onsite by intrusive XRF depends on the sample matrix category (surface materials or sediments). The percentage of these samples to be sent offsite to a commercial laboratory depends on the analytical objective (remedial support or final status survey) and on sample matrix category (surface materials or sediments). A summary of this analysis schedule is shown in Table S-3. The analyses to be performed by the offsite laboratory also depends on the analytical objective for which the sample was collected (remedial support or final status survey). The offsite laboratory analysis schedule is shown in Table S-4. Further details of these protocols are provided where appropriate in Sections S3.0 and S4.0.

Table S-3 – Approximate Percentages of Samples to be Analyzed in the Onsite Soils Lab and Offsite at a Commercial Laboratory

Onsite Analysis:	**Gamma Spectroscopy**	**Intrusive XRF**
Surface Materials	100%	Judgmental
Sediment	100%	100%
Remedial Support Samples	**% Sent Offsite***	**% Sent Offsite***
Surface Materials	10%	5%
Sediment	5%	10%
Final Status Survey Samples	**% Sent Offsite***	**% Sent Offsite***
Surface Materials	33%	33%
Sediment	33%	33%

*For initial calibration of onsite analysis methods, up to 100% of samples may be sent offsite.

Table S-4 – Analytical Schedule for Samples to be Analyzed Offsite at a Commercial Laboratory

Analytical Objective	Offsite Laboratory Analysis Parameters	
Remedial Support Samples	**Radiological**[1, 3]	**Metals**[2, 3]
Surface Materials	Ra-226, Pb-210, U	U
Sediment	Ra-226, Pb-210, U	U, Cr, Mn, Se, V
Final Status Survey Samples	**Radiological**[1, 3]	**Metals**[2, 3]
Surface Materials	Ra-226, Pb-210, U	U
Sediment	Ra-226, Pb-210, U-238, U-234, U	U, Cr, Mn, Se, V

[1]Radiological parameters will be analyzed from the canned/sealed sample aliquot previously analyzed onsite by gamma spectroscopy (see Section 2.4.2 and the QAPP).

[2]Metals will be analyzed from XRF cup sample aliquot previously analyzed by XRF onsite (see Section 2.4.2 and the QAPP).

[3]U refers to the natural (total) form of uranium (U-nat), which is both radioactive and a metal. Where both a canned sample and paired XRF aliquot of the same sample are submitted, U-nat will be analyzed for each sample fraction (see Section 2.4.2 and the QAPP).

S.2.4.1 Offsite Laboratory Methods

The proposed analytical methods to be employed by the offsite laboratory for each ROD parameter, along with respective ROD cleanup levels and method detection limits are shown in Tables S-5 and S-6. Further details on laboratory methods are provided in the QAPP.

Table S-5 – Analytical Parameters, Cleanup Levels, Analysis Methods and Detection Limits for Surface Materials

ROD Parameter	Cleanup Level	Proposed Method	Method Detection Limit
Uranium (total)	43 mg/kg	EPA 3050/EPA 6020	0.05 mg/kg
Lead-210	7.5 pCi/g	EPA 3050/EPA 909.0M	0.1 pCi/g
Radium-226	4.7 pCi/g	EPA 901.1 (soil, full Rn ingrowth)	0.4 pCi/g

Table S-6 – Analytical Parameters, Cleanup Levels, Analysis Methods and Detection Limits for Sediments

ROD Parameter	Cleanup Level	Proposed Method	Method Detection Limit
Lead-210	20 pCi/g	EPA 3050/EPA 909.0M	0.1 pCi/g
Uranium-238	31 pCi/g	EPA 3050/EPA 908.0	0.4 pCi/g
Uranium-234	41 pCi/g	EPA 3050/EPA 908.0	0.4 pCi/g
Radium-226	13 pCi/g	EPA 901.1 (soil, full Rn ingrowth)	0.4 pCi/g
Chromium	43.4 mg/kg	EPA 3050/EPA 6020	0.1 mg/kg
Manganese	1,179 mg/kg	EPA 3050/EPA 6020	0.5 mg/kg
Selenium	1.7 mg/kg	EPA 3050/EPA 6020	0.05 mg/kg
Uranium (total)	93.2 mg/kg	EPA 3050/EPA 6020	0.05 mg/kg
Vanadium	41 mg/kg	EPA 3050/EPA 6020	0.1 mg/kg

S.2.4.2 Special Sample Handling/Analysis Protocols

As detailed in the QAPP, samples sent offsite to the commercial laboratory will require special handling and analysis procedures. This requirement is primarily due to the fact that onsite analysis of Ra-226 will be calibrated to predict full radon ingrowth concentrations based on analysis the same day the samples are collected, prepared and sealed (day zero counting). These samples will be shipped in the original counting can and must remain sealed for at least 21 days before counting with a high-purity germanium (HPGe) detector (Method 901.1) at the offsite lab. Only then can the sample be unsealed and processed for analysis of the other radiological parameters as indicated in Table S-4.

Similarly, samples analyzed onsite by XRF that are scheduled for offsite analysis will be sent to the offsite lab in the special XRF soil cup. The laboratory will process the contents of the soil cup as normal and analyze for all metals as indicated in Table S-4. The XRF cup aliquot will be labeled with the same sample ID number as its paired canned sample fraction, but with a qualifying “(Aliquot B)” designation. Uranium (total) will thus be analyzed for two separate aliquots of the same sample (the canned gamma spectroscopy aliquot and the XRF cup aliquot). This could provide information on differences in concentrations that may be related to differences in sample processing and respective particle size fractions (XRF samples will be sieved through a very fine 60-mesh sieve while gamma spectroscopy samples will represent a much coarser particle size distribution). The procedures indicated in the QAPP for notifying the offsite laboratory of these special protocols will be followed (see QAPP, Section B.3.2).

S.3.0 REMEDIAL SUPPORT SURVEY PLAN

This section provides plans for how the analytical approaches described in Section S2.0 will be applied to support remedial excavation of surface materials and sediments at the Midnite Mine. The primary objective of remedial support surveys is to provide real-time, or near real-time analytical estimates of the levels of COCs present at the ground surface as remedial excavations progress across the Site, and based on these data, to determine when the horizontal and vertical extent of remedial excavation is sufficient to support a decision that the area in question is ready for final status surveys (Section S4.0).

A secondary objective of remedial support surveys is to generate additional radiological characterization data in areas that were not physically disturbed by historic mining activities, but are potentially impacted due to migration of contaminants from wind and/or water transport processes (i.e. potentially impacted Site margins or Class 2 “halo” areas). The purpose is to better define the boundaries of Class 2 survey areas as defined in Section S.4.1. Class 1 and Class 2 areas have been initially estimated based on previous radiological surveys of the Site (EPA, 2006a; EPA, 2011; SMI, 1999a and 1999b) and are depicted in Figures S-10 and S-11 of Section S.4.1. This additional characterization effort will primarily involve gamma scanning and limited sampling for onsite Ra-226 and XRF analysis, and will be conducted in the early stages of the cleanup sequence (e.g. while large amounts of materials already known to exceed cleanup levels are being removed and transported to Pits 3 and 4).

S.3.1 Gamma Scanning

S.3.1.1 Gamma Scanning for Excavation Support

For planning purposes, it is initially assumed that all portions of Class 1 areas will require remediation. Once above-grade mine materials have been excavated down to the original ground surface, gamma shine will be reduced and at this point in the remedial process, recorded, screening-level gamma scans will be conducted across each survey unit to characterize the general spatial distribution of residually contaminated soils. Ground coverage of these recorded gamma screening surveys will be on the order of 10% or less (e.g. 40-50 meter transect spacing), and scan speeds may exceed typical walking speeds (ATVs may be used for this initial scanning). This screening-level information will be used to plan initial soil excavation areas and sequences within each survey unit. This information may also be useful for identifying areas that may not require further remediation, but this must be confirmed via

interim remedial support gamma scanning and soil sampling and ultimately, via final status gamma surveys and soil sampling.

Once excavation of soils below the original ground surface has commenced within a given survey unit, gamma scanning to support remedial excavations will generally not be recorded. A properly trained field technician will manually monitor and guide the vertical and horizontal extent of excavation required based on comparisons of readings in the excavated areas against the gamma cutoff value. For routine remedial support scanning, the only instrumentation needed is a gamma detector. Remedial support scanning will be performed at 1 meter above the ground and will cover 100 percent of the area that is actively being excavated (a maximum distance between adjacent scan paths or static measurement locations of about 3 meters[7]).

Additional scanning near the ground surface will also be conducted as needed to better resolve small potential hot spots and/or slightly buried sources of elevated gamma emissions. Scan speeds for remedial support surveys will be typical walking speeds or less (2-3 mph or slower, depending on terrain and spatial consistency of readings). If readings temporarily increase above the cutoff value while scanning, the surveyor will repeat scan measurements at slower speeds (and possibly lower detector height) in the vicinity of the area in question to verify elevated readings before directing further excavation to remove the apparent hot spot. In areas where significant gamma shine from adjacent areas is possible or apparent, tungsten shields may be used to help reduce the confounding effects of gamma shine. Details of issues and protocols related to gamma shine are provided in Section 3.4 of Attachment S-1.

The Field Supervisor and/or Field Program Director will likely require interim GPS-based scans that are recorded for data review and assessment of remedial progress. For example, unofficial, screening-level gamma scans would help to determine whether a given area in question is ready for final status surveys. Unofficial screening surveys need not cover 100 percent of the area in question or be surveyed at walking speeds as these data will not be used for formal evaluation of compliance with the gamma cutoff value (i.e. they will not be included as part of final status surveys). It is the responsibility of the Field Supervisor to determine the

[7] Based on previous observations and experience in the field, at a detector height of one meter, lateral detector response to significantly elevated planar (non-point) gamma sources at the ground surface is estimated to be about 2 meters, giving each detector an estimated "field of view" of about 4 meters diameter at the ground surface. This does not imply a system detector can register increased gamma readings from a small point source 2 meters away, but does indicate that scattered photons from larger elevated source areas (e.g. 10 m^2) are likely to be detected at that distance. Based on the Scan MDC calculations provided in Attachment S-1, a distance of 3 meters between scan paths or individual measurements is conservatively expected to provide 100% coverage.

amount of respective evidence necessary to make a correct decision as to whether the area in question is ready for final status surveys.

With respect to remedial support surveys for sediments in mine drainages, gamma scanning may be used as an initial screening method for guiding excavations with respect to Ra-226 concentrations (based on the gamma cutoff value for sediments), but a combination of in-situ XRF survey measurements (Section S.3.2) and direct sampling for intrusive XRF analysis (Section S.3.3) will be the primary means for guiding excavations and generating sufficient analytical data to support a decision that the area in question is ready for final status surveys.

S.3.1.2 Gamma Surveys for Characterization of Class 2 Areas and Borrow Materials

With respect to the secondary remedial support objective of additional radiological characterization in outlying margins of the Site ("halo" areas) early in the cleanup sequence, gamma scanning will be officially recorded with GPS-based systems as described in Section S.2.1. Target gamma survey coverage in these halo areas will be on the order of 50 percent, but actual coverage is expected to vary considerably as these areas are often forested and rugged terrain may be encountered (under these circumstances, accessibility and/or safety can become highly limiting in terms of coverage that can feasibly be achieved). It is the responsibility of the Field Program Director and Field Supervisor to ensure that sufficient analytical data is collected around these "halo" areas to support a change in the currently estimated boundaries of Class 1 and Class 2 survey areas. All evidence and proposed changes will be submitted to EPA for review and a decision regarding the change.

In borrow material areas, gamma survey measurements will be used to screen soils to be used for final cover. Gamma screening will also be conducted for materials previously stockpiled onsite that are intended for use as final cover. Borrow material gamma screening surveys will be conducted on a weekly basis during periods of active excavation. Four random grab samples of borrow materials will also be collected on a weekly basis and analyzed in the onsite lab to verify that the material meets the soil cleanup standards. Assuming a maximum borrow material excavation rate of 20,000 cubic yards per week, this is equivalent to a minimum volume-based sampling frequency of 1 sample per 5,000 cubic yards.

S.3.2 In-situ XRF Surveys

S.3.2.1 In-situ XRF Surveys for Excavation Support

Every in-situ XRF survey measurement to support remedial excavations need not be recorded, though logbook notes on typical readings and any anonymously high values in areas that

generally appear to be in compliance should be documented. In-situ XRF surveys pertain primarily to remediation of sediments, though limited in-situ XRF surveys for uranium (only) will be conducted to support remedial excavation of surface materials (see Attachment S-1, Section 3.5). In-situ XRF surveys used to support remedial excavation will include random and biased sampling at about 5 percent of in-situ measurement locations for confirmatory analysis in the onsite soils lab with the intrusive XRF methodology. Both XRF methods will be performed according to the specifications of Method 6200 and procedures provided in AS-SOP 5.

A properly trained field technician will manually monitor and guide the vertical and horizontal extent of excavation required based on comparisons of in-situ XRF readings in the excavated areas against the XRF cutoff values (see Section S.2.3.1). In general, if the reading for manganese in sediments along mine drainages is in compliance with manganese cutoff value, other sediment COCs are expected to be in compliance as well (see Attachment S-1), though this should periodically be verified by assessing measurement values for other COC metals.

The spatial density of in-situ measurements needed to effectively support remedial excavations will depend on conditions and the degree of variability in analytical results. An initial target density of 5 measurements across an area on the order of 100 m^2 will be evaluated for potential effectiveness in this regard (based on comparisons against random confirmation sampling and intrusive XRF analysis), but this target density may be adjusted as appropriate based on evaluations early in the cleanup as well as real-time assessments of area conditions and measurement variability). If XRF survey results across a sizeable area are relatively uniform, a reduced measurement density may be warranted while if results are highly variable, more measurements will likely be required. In wet conditions, soil moisture can interfere with in-situ measurement accuracy and samples should be taken, processed in the lab (including drying), and analyzed with the intrusive XRF analysis method.

It is the responsibility of the Field Program Director and Field Supervisor to determine the amount of XRF measurement and sample analysis data necessary to make a correct decision as to whether the area in question is ready for final status surveys.

S.3.2.2 In-situ XRF Surveys for Characterization of Class 2 Areas

With respect to the secondary remedial support objective of additional radiological characterization in outlying “halo” areas early in the cleanup sequence, in-situ XFR surveys will not be conducted, but intrusive XRF analysis will be performed on all samples collected for analysis of Ra-226 with gamma spectroscopy (see Section S.3.3.2).

S.3.3 Onsite Sample Analysis

In order to monitor and confirm the reliability and accuracy of the gamma cutoff value and associated gamma/Ra-226 correlation, and to also directly compare analytical results with ROD cleanup levels, both random and biased soil samples will be collected in areas that have been excavated until gamma scan readings are at or below the gamma cutoff value, as well as in outlying “halo” areas that are potentially impacted but may or may not be subject to remedial excavation (Class 2 areas). All samples will be collected and processed onsite in accordance with the procedures indicated in AS-SOP 1, AS-SOP 2 and AS-SOP 3. These samples will primarily be analyzed in the onsite soils lab (via gamma spectroscopy and/or intrusive XRF analysis), but some percentage will also be analyzed at the offsite commercial laboratory.

S.3.3.1 Onsite Sample Analysis for Excavation Support

The number of samples needed to support remedial excavations cannot be specified in advance as this will depend on analytical results. If, for example, 5 samples are taken in a given area believed to be in compliance with the gamma cutoff value and the average Ra-226 result from the onsite lab is close to or above the cleanup level, more samples would be required as this would be a potential indication that the gamma cutoff value and gamma/Ra-226 correlation are not representative of the area in question. Possible reasons for this would include gamma shine from adjacent areas or a high degree of small-scale spatial heterogeneity in soil concentrations. Conversely, if the average Ra-226 result is about half of the cleanup level and no individual samples exceed the cleanup level then additional sampling is unlikely to be necessary.

In the event that samples must be sent offsite for rapid turn-around Ra-226 analysis in order to support remedial excavations (e.g. if the onsite laboratory is temporarily not operational due to equipment problems or scheduling conflicts), an approach has been developed to accomplish this without needing to wait for full radon ingrowth to occur. This approach is detailed in Section 3.6 of Attachment S-1. For all samples to be sent offsite, regardless of the analytical objective (remedial support or final status surveys) or sample matrix type (surface materials or sediments), it is mandatory that offsite analysis of Ra-226 is performed using gamma spectroscopy (Method 901.1). In the case that rapid turnaround Ra-226 analysis by the offsite lab is required, the offsite laboratory will use Method 901.1 with the alternate protocol that samples will be counted the same day they are sealed (without waiting 21 days for full ingrowth of radon). The offsite lab will be informed in advance of this protocol (along with specific instructions attached to the chain-of-custody/analytical request form), and will be instructed to immediately report results to the Field Program Director or Field Supervisor for subsequent

calculation of full-ingrowth Ra-226 estimates using Equation 1 in Attachment S-1. Further protocols for this circumstance are provided in in Section 3.6 of Attachment S-1.

It will be the responsibility of the Field Supervisor, in collaboration with the Field Program Director, to determine whether the number of samples collected for remedial excavation support purposes is sufficient to support a decision that the area in question is ready for final status surveys. Although a number of factors can affect this decision (e.g. combined gamma scan and soil sampling evidence, along with QA/QC information related to respective results), basic decision criteria for samples collected as a part of remedial support are that virtually 100 percent of individual sample results, whether analyzed onsite or offsite, should fall below ROD cleanup levels. In general it is expected that rapid turn-around analytical results from the onsite soils lab (gamma spectroscopy and/or intrusive XRF analysis) will be sufficient to make such decisions, provided that previous results have demonstrated acceptable agreement with confirmatory results from the offsite laboratory, and that QA/QC data for onsite analytical systems are within specified control limits (see QAPP).

Based on the analysis provided in Attachment S-1, guiding remedial excavation of surface materials based on gamma screening techniques and onsite soils lab measurements to ensure compliance with the Ra-226 cleanup level is expected to simultaneously address uranium and Pb-210 cleanup levels for surface materials in about 95 percent of all cases. However, XRF analysis for uranium concentrations will be used where appropriate to provide additional assurance that the rate of compliance with the cleanup criterion for uranium will be at least 95 percent. For remedial support surveys, such screening will primarily involve in-situ XRF measurements and limited collection of samples for intrusive XRF analysis in areas with a higher likelihood of significant radiological disequilibrium between uranium and Ra-226 (e.g. as suggested by data provided in the Mine Waste Investigations report). In addition to this biased screening for uranium, some limited random XRF screening and sample analysis will also be conducted for surface materials.

S.3.3.2 Onsite Sample Analysis for Characterization of Class 2 Areas

With respect to the secondary remedial support objective of additional radiological characterization in outlying margins of the Site (“halo” areas) early in the cleanup sequence, the number of samples to be collected is again dependent on analytical results as well as consistency of initial samples with gamma scan-based estimates of Ra-226. If, for example, 5 samples in a given area are taken and the average Ra-226 result from the onsite lab (on day zero) is close to the average Ra-226 concentration that is predicted for the same area based on

gamma survey data and the gamma/Ra-226 correlation, then additional sampling would be unwarranted. It is the responsibility of the Field Program Director and Field Supervisor to ensure that sufficient analytical data is collected around these “halo” areas to support a change in the currently estimated boundaries of Class 1 and Class 2 survey areas. All evidence and proposed changes will be submitted to EPA for review and a decision regarding the change.

S.3.4 Decision Criteria for Remedial Support Surveys

S.3.4.1 Surface Materials

The surface material removal decision diagram presented in Section 1.0 (Figure S-4) is generalized and applies to both remedial support and final status surveys. Key decision criteria for remedial support surveys are summarized on the left side of the diagram (Figure S-4). It is the responsibility of the Field Supervisor and Field Program Director to determine when a given area is ready for final status surveys. A complete listing of decision criteria guidelines for evaluation of when a given area is ready for final status surveys based on remedial support survey data is as follows:

<u>Compliance Evaluation Criteria</u>

1. Nearly 100 percent of gamma readings across the area in question (e.g. > 98 percent) should be less than the gamma cutoff value in use at the time of the evaluation. If as much as 2 percent or more of the area exceeds this criterion, either more remediation is necessary (e.g. hot spots are still present), or gamma shine may be present (in which case additional soil samples should be collected and analyzed onsite for Ra-226).
2. Nearly 100 percent of analytical Ra-226 results for surface material samples analyzed either onsite or offsite by gamma spectroscopy (e.g. > 98 percent) should be less than or equal to the ROD cleanup level for Ra-226.
3. Nearly 100 percent of in-situ and/or intrusive XRF measurements for uranium (e.g. > 98 percent) should indicate compliance with the uranium cleanup level in areas suspected of radiological disequilibrium.
4. Nearly 100 percent of any offsite sample analysis results for uranium and Pb-210 (e.g. > 98 percent) should be less than or equal to respective ROD cleanup levels.
5. Exposed bedrock will not be evaluated in a context of compliance with ROD cleanup levels, but gamma scans must be conducted to document gamma readings above the exposed bedrock.

Data QA/QC Criteria (see QAPP for further details)

1. Daily QC measurements for field gamma measurement instruments and associated scanning systems must fall within established control limits (see QAPP and AS-SOP 6) for the corresponding period of use in the field.
2. Daily QC measurements for the XRF instrument and associated measurement methods must fall within established control limits (see QAPP and AS-SOP 5) for the corresponding period of use in the field.
3. Daily QC measurements for the onsite gamma spectroscopy system (measurements of 2 site-specific soil Ra-226 calibration standards) must fall within established control limits (see QAPP and AS-SOP 4) for the corresponding period of onsite use.
4. At least 90 percent of onsite analysis results for Ra-226 in field samples based on onsite gamma spectroscopy should fall within the limits of 90 percent prediction intervals on the current method calibration algorithm (expressed in units of activity concentration). This will limit the probability of under-estimation of equivalent full ingrowth results at the offsite lab (a Type I estimation error) to less than 5 percent.
5. At least 90 percent of XRF data should fall within 90 percent prediction intervals on method calibration (regression) curves. This will limit the probability of under-estimation of equivalent results at the offsite lab (a Type I estimation error) to less than 5 percent.

S.3.4.2 Sediments

The sediment removal decision diagram presented in Section S.1.0 (Figure S-5) is generalized and applies to both remedial support and final status surveys. Major decision criteria for remedial support surveys are summarized on the left side of the diagram (Figure S-5). It is the responsibility of the Field Supervisor and Field Program Director to determine when a given area is ready for final status surveys. A complete listing of decision criteria guidelines for evaluation of when a given area is ready for final status surveys based on remedial support survey data is as follows:

Compliance Evaluation Criteria

1. Nearly 100 percent of in-situ XRF screening measurements for manganese across the area in question (e.g. > 98 percent) should be less than the respective in-situ XRF cutoff value. If as much as 2 percent or more of readings exceeds this criterion, more remediation is likely necessary (identifiable hot spots may still present). Random spot

checks for readings of other metal COC parameters should be performed to verify that they also meet respective in-situ XRF cutoff values.

2. Nearly 100 percent of analytical results for sediment samples (e.g. > 98 percent) analyzed either onsite by intrusive XRF measurements, or offsite by specified laboratory methods, should be less than or equal to the ROD cleanup levels for sediments.
3. Analytical screening for Ra-226 in sediments should follow the same decision criteria guidelines indicated in Section 3.4.1 for surface materials (with the exception that both the ROD cleanup level for Ra-226 and associated gamma cutoff value differ for sediments).
4. Exposed bedrock will not be evaluated in a context of compliance with ROD cleanup levels, but gamma scans must be conducted to document gamma readings above the exposed bedrock.

Data QA/QC Criteria (see QAPP for further details)

1. Daily QC measurements for the XRF instrument and associated measurement methods must fall within established control limits (see QAPP and AS-SOP 5) for the corresponding period of use in the field.
2. At least 90 percent of XRF data should fall within 90 percent prediction intervals on method calibration (regression) curves. This will limit the probability of under-estimation of equivalent results at the offsite lab (a Type I estimation error) to less than 5 percent.

S.4.0 FINAL STATUS SURVEY PLAN

This section provides plans for how the analytical approaches described in Section S.2.0 will be applied when conducting final status surveys across defined survey units. These survey units have been initially estimated (Section S.4.1.1) and will be further defined as appropriate based remedial support survey data collected in “halo” areas during remedial activities, and on any future adjustments to the remedial schedule or excavation sequencing. The objective of final status surveys is to qualitatively, quantitatively and statistically demonstrate that remedial excavations have resulted in compliance with cleanup levels for surface materials and sediments (Tables S-1 and S-2). Final status surveys will generally follow the scanning, sampling and data analysis approaches described in MARSSIM, the Multi agency Radiation Site Survey and Investigation

Manual (NRC, 2000)[8], though some differences are necessary due to project circumstances (see Attachment S-1 for further information).

S.4.1 Final Status Survey Units

For reasons detailed in Attachment S-1, MARSSIM-based Class 1 areas will be divided into 15-25 acre survey units for surface materials, and for sediments, along distinct mine drainage segments based on geographic and remedial schedule considerations. Class 2 survey units for surface materials will encompass the "halo" of potentially impacted areas surrounding Class 1 areas, and will generally be delineated into four basic survey units based on compass direction quadrants (NE, SE, SW, and NW). For reasons detailed later in this Section, Class 2 survey units are unlikely to be practical or meaningful along narrow Class 1 impacted haul roads or mine drainages. The prospectively estimated survey units provided in this Section are based on the following factors and considerations:

1. MARSSIM-based categorization as Class 1, 2 or 3 areas as follows:

    - Class 1: Areas likely to be impacted and that prior to remediation are generally expected to have sampling results in excess of the cleanup levels specified in the ROD.
    - Class 2: Areas that may be slightly impacted, but are generally not expected to have sampling results in excess of ROD cleanup levels.
    - Class 3: Adjacent areas with no current or historic evidence of impacts.

    The initial classifications provided in this section are based on known areas of mine disturbance and mining affected areas (MAA) as identified/depicted in the ROD (EPA, 2006a) and the Mine Waste Investigations Report (Miller Geotechnical, 2011), along with potentially impacted areas based on previous gamma survey data (EPA, 2011; SMI, 1999a and 1999b).

2. The remediation schedule, with Class 1 areas divided into "zones" in accordance with expected remedial completion dates for various portions of the site. Class 1 access

[8] MARSSIM is a unified technical approach and guidance document developed by the U.S. Nuclear Regulatory Commission (NRC), U.S. Department of Energy (DOE), and the U.S. Environmental Protection Agency (EPA) for assessing remedial performance at radiologically impacted sites. This guidance has gained wide regulatory acceptance and use at radiologically impacted sites in the U.S. and abroad.

roads and mine drainages located outside the boundaries of Class 1 zones are characterized in terms of lineal feet rather than areal extent.

3. Limiting the maximum size of survey units within a Class 1 zone to a reasonable areal extent (e.g. 15-25 acres) depending on the overall size of the Class 1 zone. For access roads and mine drainages, survey units will involve segment lengths (in lineal feet) and adjacent margins.
4. Relatively uniform sizing and spacing of survey unit subdivisions within each Class 1 zone.
5. The number of survey units that can be reasonably expected to be surveyed per year. In general, completion dates for most Class 1 zones (including site access roads and mine drainages) are currently estimated to occur in a four-year period between 2019 and 2023.
6. Practical survey unit boundaries in terms of the actual spatial sequence of remedial progression that will occur over time within each Class 1 zone (this sequence cannot be precisely known in advance, but can be estimated for planning purposes).
7. Class 2 areas involve a margin or “halo” around the perimeter of Class 1 areas, with outer limits estimated based on previous gamma survey data (EPA, 2011; SMI, 1999a and 1999b). These limits will be adjusted if necessary when more data become available as part of further characterization surveys to be conducted early in the cleanup sequence. The Class 2 halo surrounding the site will be divided into four basic survey units based roughly on compass-oriented quadrants (NE, SE, SW, and NW).
8. Class 3 areas are assumed to represent unimpacted background conditions and will not be formally surveyed, but some confirmation sampling/scanning in these areas will take place when refining the outer limits of Class 2 areas early in the cleanup sequence.

Based on the above considerations, in particular on the remedial schedule, the site has been preliminarily delineated into separate Class 1 zones (Figure S-10). The ID label prefixes in Figure S-10 represent remediation phase numbers and corresponding sequential zone numbers. Respective areal dimensions (acres) or lineal feet (for access roads and mine drainages) along with estimated remedial completion dates are also annotated. These zones are further divided (where applicable) into proposed Class 1 final status survey units in Figure S-11, which also includes proposed Class 2 survey units. The survey unit ID labels in Figure S-11 include suffix numbers to represent subdivided survey units within a given Class 1 zone or

Class 2 area. Class 3 areas are not labeled, but are assumed to include locations beyond the perimeter of Class 2 (and in some cases, Class 1) areas.

For Class 1 access roads and mine drainages, Class 3 areas are likely to border these features very closely as the potential for lateral contamination transport, and thus adjacent gradation of related impacts, is likely to be spatially limited in most cases [i.e. the immediate margins of these narrow features are likely to go from impacted to not impacted within comparatively short distances (e.g. 10 meters or less)]. This general circumstance is evident from gamma survey data collected along roads and mine drainages (Miller Geotechnical, 2011). As a result, it would likely be difficult to accurately or meaningfully implement Class 2 survey units along the margins of these narrow Class 1 features.

Regarding factor number (6) above, the field construction contractor will have its own particular strategies for accomplishing the work in the most efficient manner possible. For this reason, proposed delineation of individual survey unit boundaries are only prospective estimates and are subject to modification. Moreover, the overall estimated limits of Class 1 areas may change as more Site data is generated during the cleanup.

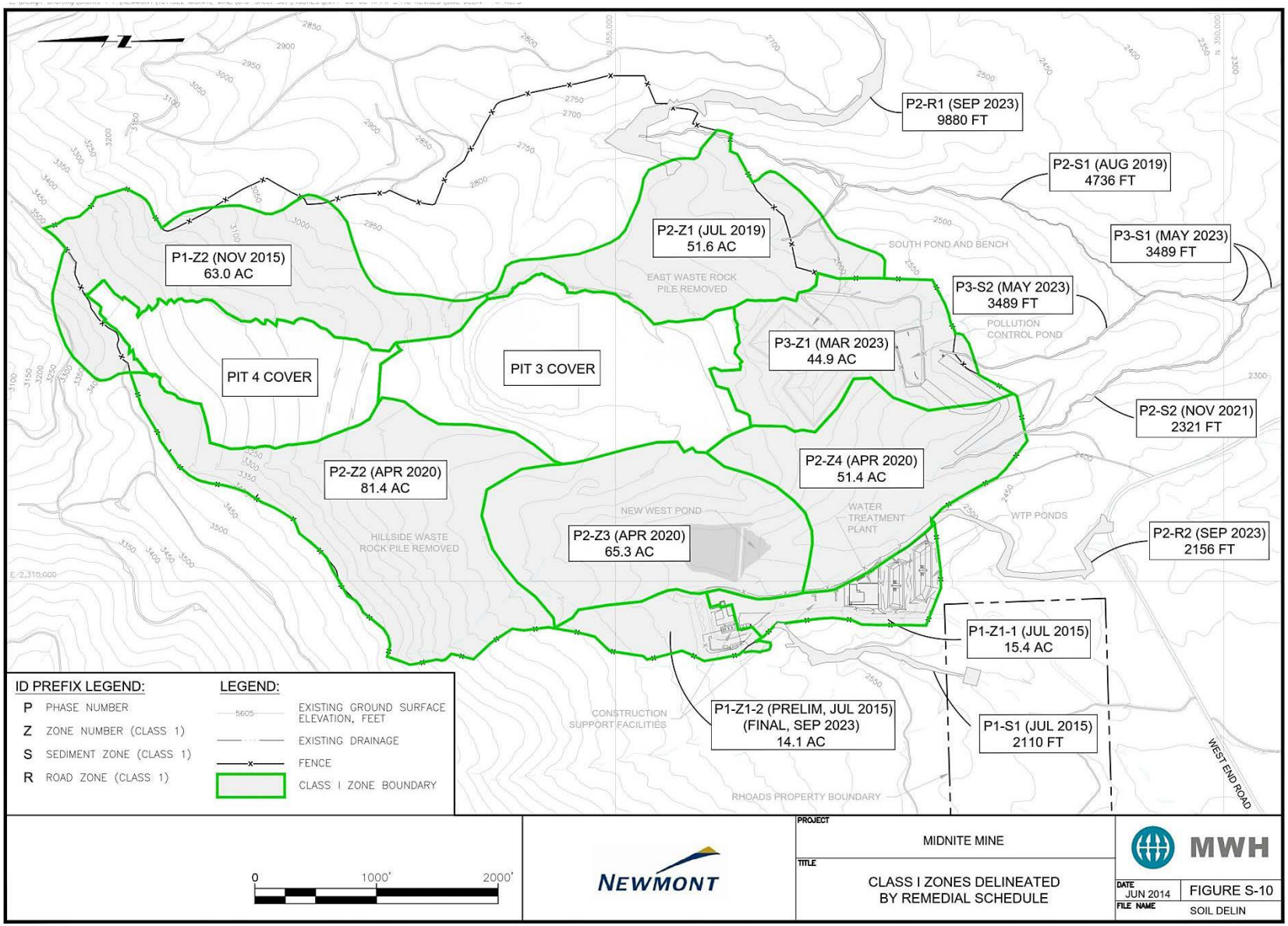

Figure S-10 – Class 1 zones delineated by remedial schedule.

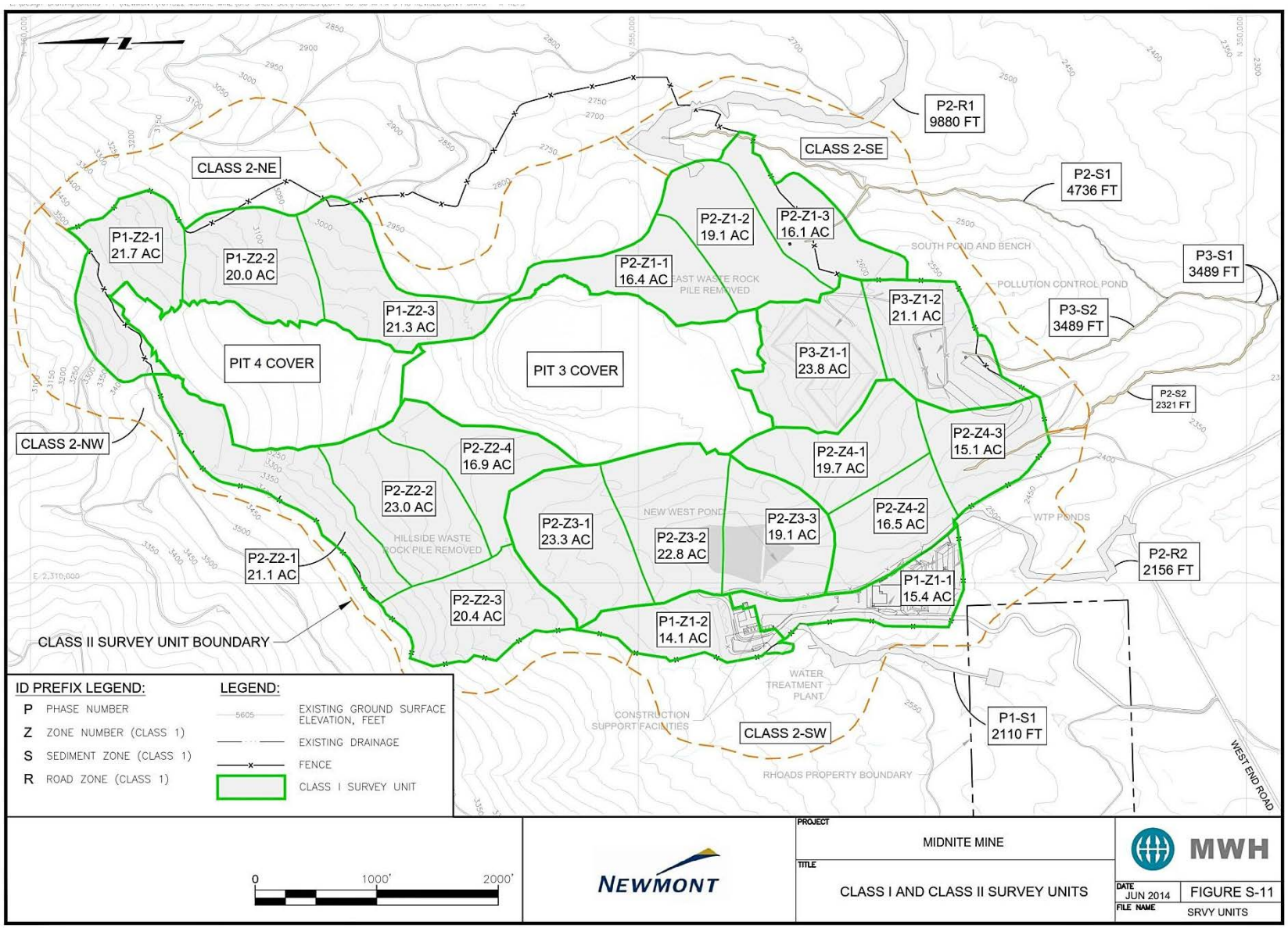

Figure S-11 – Class 1 and Class 2 survey units.

S.4.2 Final Status Surveys for Surface Materials

S.4.2.1 Final Status Gamma Survey

Final status gamma surveys will be conducted and evaluated in advance of any final status soil sampling. Respective data will be recorded to provide a permanent record of gamma readings across the final status survey unit. Results will be mapped and quantitatively/spatially assessed with respect to the current gamma cutoff value. This protocol will provide further assurance (in addition to previous remedial support surveys) that the survey unit is also ready for final status sampling of surface materials or sediments. In addition, final status gamma scan data will be converted to estimates of soil Ra-226 concentrations using the current gamma/Ra-226 regression equation, and results will be mapped for direct comparison against the Ra-226 cleanup level across the entire survey unit.

If the final status gamma scan indicates that further remediation should be conducted prior to direct sampling of surface materials or sediments, additional spot remediation will be conducted as needed and final status gamma scanning will be repeated in these areas. This process is essentially a sub-component of the overall process reflected in Figure S-4 (Section S.1.0). Once all final status gamma scan data indicate that the survey unit is sufficiently remediated to justify final status sampling of surface materials or sediments, direct final status sampling will commence in accordance with the provisions of Section S.4.2.2.

Recorded final status gamma surveys will be conducted in accordance with the methodology, instruments and functional system specifications described in Section S.2.1 and procedures indicated in AS-SOP 6. Detector shielding will not be used for final status surveys as gamma shine is generally not expected to be a significant issue in areas that are ready for final status surveys. However, there could be circumstances where gamma shine could be present near the boundary of a survey unit (e.g. if an adjacent survey unit has yet to be remediated) or near areas where naturally mineralized bedrock prevented further excavation. In such cases, gamma survey-based evidence of compliance between soil sampling locations cannot be relied upon and additional soil sampling across such areas will be required to verify compliance with ROD cleanup levels. The Field Program Director will be notified of such circumstances and will determine the appropriate number and locations of additional soil samples to be collected. Judgemental sampling will be conducted to address these or other circumstances based on gamma survey data. All related information will be documented in final status survey reports.

In the event that excavation in portions a given survey unit has proceeded to bedrock, the area of bedrock will be documented with a separate final status gamma survey over the exposed area of bedrock, and the data will be saved as a separate gamma scan file and a designation of "bedrock" will be included in the scan file name. Attachment S-3 describes the process to determine when bedrock has been encountered. Surface material sampling will not be possible or applicable in such areas, but this will not reduce the required number of final status samples to be collected across the survey unit as a whole (i.e. the sampling density will simply be increased in all areas where bedrock was not encountered).

Target gamma scan coverage for final status surveys will be 100 percent for all Class 1 survey units, meaning a distance between adjacent scan tracks of about 3 meters[9], though practical considerations such as safety and terrain will influence actual distances that can reasonably be maintained between adjacent scan tracks. For Class 2 areas, target coverage will be on the order of 50 percent, though these areas are largely forested and some unsafe or otherwise inaccessible terrain may be encountered. As a result, scan coverage in Class 2 areas may vary considerably (e.g. 10-70 percent). Scanning speeds for all final status surveys will be conducted at typical walking speeds (2-3 mph depending on terrain).

In the event that an upset of traffic or stormwater/erosion control measures may have caused an area to become re-contaminated after the final status survey has been conducted, the area in question will be re-surveyed with gamma scanning at 100 percent coverage and the data will be evaluated against the gamma cutoff value. If evidence of re-contamination exists, the area will again be remediated under the provisions of this Plan, and a second survey of the re-remediated portion of the survey unit will be conducted to verify compliance using protocols indicated for identified hot spots (gamma scanning and at least two soil samples as indicated in Section S.4.2.3.1). In the event that a temporary access road must be constructed across a previously remediated survey unit in order to facilitate overall remedial construction activities, re-surveys will also be conducted upon final reclamation and closure of the temporary road (using the same re-survey protocols as indicated above). All re-surveys will be documented and results will be included in final status survey reports. Native grass seed will be spread across final status survey areas, and following any necessary additional excavation and re-surveys, to help

[9] Based on scan MDC calculations provided in Attachment S-1, a distance of 3 meters between scan paths or individual measurements is conservatively expected to provide 100% coverage. See also footnote 7 in Section S.3.1.1.

mitigate the potential for interim growth of invasive weed species prior to implementation of the final revegetation plan as detailed in Appendix D.

Once the final status gamma scan data have been collected and reviewed by the Field Program Director in terms of data completeness and QA/QC (see QAPP), standard GIS software (e.g. ArcGIS) will be used for official mapping, interpolation with kriging methods, and quantitative/spatial analysis for inclusion in final status survey reports. The data mapping, kriging and spatial analysis will be performed by a qualified GIS analyst, while the Field Program Director (a qualified environmental health physicist) will develop the formal analysis of the resulting data and maps for inclusion in final status survey reports.

S.4.2.2 Final Status Surface Material Sampling

Once the final status gamma survey indicates that the survey unit is ready for direct sampling, a minimum of 75 samples will be collected. Samples will be collected along a random start square or triangular sampling grid pattern generated using the Virtual Sample Plan (VSP) software package (VSP, 2012). At each sampling location, a discrete grab sample will be collected to a depth of 6 inches (15 cm) in accordance with standard operating procedures AS-SOP 1, AS-SOP 2 and AS-SOP 3. A minimum of 350 grams of surface material (soil, sand and/or gravel) will be collected at each location in accordance with the procedures indicated in AS-SOP 1 and AS-SOP 2. To the extent possible, any rocks larger than about 1 cm in diameter in the bulk sample will be discarded at the sampling location. A field duplicate sample will be collected at a minimum of 5 percent of sampling locations (at least 4 field duplicates for every 75 samples – see QAPP).

All surface material samples will be processed and analyzed in the onsite soils lab for Ra-226 according to the methodology described in Section S.2.2.2 and procedures provided in AS-SOP 3 and AS-SOP 4. Of the 75+ samples from each survey unit, at least 33 percent (25+ samples), plus at least one field duplicate (a minimum of 26+ samples total), will subsequently be sent to an approved commercial laboratory for analysis of all parameters indicated in Table S-5 (Section S.2.4). Selection of the 33 percent of final status samples from each survey unit to be sent offsite for analysis will be performed with a random number generator (e.g. available in Microsoft Excel® or from specialized programs available on the internet). Quality Assurance / Quality Control requirements and SOPs for surface material and sediment sampling, onsite processing and onsite/offsite laboratory analysis, are provided in the QAPP.

S.4.2.3 Determination of Compliance for Surface Materials

Once all final status survey data for a given survey unit have been obtained, data quality and completeness evaluations will be conducted, followed by qualitative, quantitative and statistical data assessments for compliance with ROD cleanup levels. This section describes the methods, considerations and protocols that will be used to evaluate and determine compliance with ROD cleanup levels for surface materials.

S.4.2.3.1 Data Analysis

Once qualified as useable data in accordance with the QAPP, final status survey results will be mapped and spatially, qualitatively, quantitatively and statistically evaluated to assess compliance with ROD cleanup levels. Gamma survey based evidence of compliance will be evaluated in two ways. First, final status gamma survey data will be directly evaluated against the current gamma cutoff value as expected to limit actual Type I error rates on this analytical measure to 5 percent or less (see Attachment S-1, Section 3.1). At least 95 percent of gamma readings across the survey unit must be less than or equal to the gamma cutoff value in use at the time of the final status survey[10].

Secondly, the regression equation for the gamma/Ra-226 correlation in use at the time of the final status survey will be used to convert raw gamma scan data into estimates of soil Ra-226 concentrations across the entire survey unit. Descriptive statistics of these results will be generated, and respective data will be mapped. At least 95 percent of these gamma-based estimates of Ra-226 concentrations must be equal to or less than the respective ROD cleanup level (4.7 pCi/g). This, combined with the mapped data, will provide another form of assurance that the survey unit is in compliance with the Ra-226 cleanup level, based on nearly perfect knowledge of the entire population of gamma exposure rates across the survey unit. Based on all available data to date, demonstration of compliance with the Ra-226 cleanup level for surface

[10] As indicated in Section S.2.1.3 and in Attachment S-1 (Section 3.1), any refinement of the gamma/Ra-226 correlation and associated gamma cutoff value over time will not affect Type I error rates on previous decisions made during the cleanup. Final status gamma scan data collected early in the cleanup will not subsequently be re-evaluated based on later revisions to the gamma/Ra-226 correlation and respective gamma cutoff value. Any revision of the regression equation based on updated correlation data (and respective revision to the gamma cutoff value) will be assigned an effective date of use moving forward and this date will be documented. Because all recorded gamma scan data include a date stamp, the proper regression equation and gamma cutoff value will be used to evaluate final status scan data collected at any given period of the cleanup sequence. The technical basis for this protocol is detailed in Attachment S-1 (Section 3.1).

materials will ensure with an approximate probability of 95 percent or higher that uranium and Pb-210 are also in compliance (see Attachment S-1, Section 3.3).

There is a possibility that in some areas, excavations may necessarily terminate at native bedrock containing naturally occurring background levels of radionuclides in excess of ROD cleanup levels. The EPA's policy with respect to background at CERCLA sites is that cleanup levels are not set below natural background levels, and the CERCLA program does not remediate to levels below that of natural background (EPA, 2002; EPA, 2006a). For this reason, areas excavated to bedrock will not be evaluated in a context of compliance with ROD cleanup levels, but will be scanned to document final status gamma readings over such areas (see Section S.4.2.1). Determination of when bedrock has been encountered during excavation is defined in Attachment S-3. Any areas excavated to bedrock will be delineated and identified accordingly in final status survey maps.

With respect to statistical analysis of direct sampling results, the cleanup levels for the Midnite Mine are not based on above background levels of dose or risk but are instead functionally equivalent to background threshold values. For this reason, conventional MARSSIM statistical testing for compliance based on sampling results is not applicable (see Attachment S-1, Section 6.1). Instead, soil analysis results will be statistically evaluated with one-sample proportion testing using ProUCL v4.0 software (EPA, 2007) to determine whether or not direct sampling data support a statistical determination (at the 95 percent confidence level) that at least 95 percent of the survey unit meets the Ra-226 cleanup level. The statistical testing will limit both the potential exceedance rate and Type I decision error rate to 5 percent ($\alpha = 0.05$).

Per MARSSIM guidance, the null hypothesis for the statistical testing will be that the survey unit does not meet a 95 percent rate of compliance with ROD cleanup levels. This protocol assigns the potential decision error of greatest concern to the Type I error rate. A Type I error would occur if a remediated area is incorrectly determined to meet a 95 percent rate of compliance with applicable cleanup levels. The consequences of this type of error would include the potential for human health risks greater than those associated with the cleanup level (i.e. greater than the upper range of background). The consequences of a Type II error are of lesser concern than the consequences of a Type I error. A Type II error would occur if a remediated area is incorrectly determined to have a rate of compliance below 95 percent. The consequences of a Type II error would include more cleanup than is necessary to meet ROD cleanup levels and unwarranted remediation costs.

The calculated number of samples needed to limit Type I error rate to 5 percent for one-sample proportion hypothesis testing is 52 samples (Attachment S-1, Section 6.1). Given that there is a practical need for relatively large survey units (15-25 acres) due to logistical considerations such as remedial schedule and the number of survey units that can reasonably be remediated and surveyed in a given year (Section S.4.1), the overall analytical approach to the cleanup has been designed to ensure that both Type I and Type II errors are analytically controlled (as opposed to statistically controlled) as follows:

- Remedial support surveys in advance of final status surveys will be analytically intensive (100 percent coverage gamma scanning, XRF surveys, and random/biased confirmatory sampling with onsite/offsite analysis). These measures are expected to preemptively limit the potential for actual decision error rates (as opposed to statistical estimates) for either type of decision error to well below 5 percent.
- The proposed number of final status survey samples to be collected and analyzed in each survey unit (75 samples) is approximately 33 percent greater than what is statistically needed to limit Type I error rates to 5 percent, and final status gamma surveys will provide 100 percent coverage of the survey unit (see related discussion in Section 3.0 of Attachment S-1).

The statistical hypothesis testing will be based on Ra-226 results for all final status samples collected in each survey unit (at least 75 samples), with 66% of the results (e.g. from 50 samples) obtained in the onsite soils lab, and 33 percent of the results (e.g. from 25 samples) obtained from the commercial lab. The 33 percent of samples analyzed offsite will have also been analyzed onsite (all samples will be analyzed onsite for Ra-226), but only the offsite results will be used for the statistical testing as double counting paired onsite/offsite results as though they were independent sampling results would violate basic statistical testing assumptions. Selection of the 33 percent of samples from each survey unit to be sent offsite will be performed with a random number generator. Paired analytical results for the 33 percent of samples analyzed both onsite and offsite will be statistically evaluated in terms of agreement (e.g. parametric T-tests, non-parametric Wilcoxon Rank Sum tests, ANOVA tests). In the event that onsite lab results are determined to be unacceptable (e.g. a clear high bias is apparent for onsite lab results), remaining onsite analysis samples for the survey unit in question will be submitted to the offsite lab for analysis.

In the event that the survey unit passes the one-sample proportion testing for a 95 percent rate of compliance, but one or more individual sample results exceed the ROD cleanup level for Ra-

226, respective results cannot also exceed a secondary cleanup level of twice the ROD cleanup level (9.4 pCi/g) and still be considered to represent an acceptable remedial outcome. Any individual sample result that exceeds the secondary cleanup level will automatically trigger a secondary investigation to determine if a "hot spot" may be present. This secondary investigation will include a repeat of 100 percent coverage gamma scanning of the area in question, and collection of at least two additional soil samples in close proximity to the location of the original sample. If a hot spot above the ROD cleanup level is confirmed, it will be remediated to the ROD cleanup level, re-scanned and re-sampled to document successful remediation. If a hot spot is not confirmed, secondary investigation results will replace the original result that incorrectly suggested a potential hot spot.

The surface material removal decision diagram presented in Section 1.0 (Figure S-4) is generalized and applies to both remedial support and final status surveys. Respective decision criteria, including major elements of those detailed in this Section, are summarized in Figure S-4. A more complete/detailed summary listing of decision criteria for evaluation of compliance with ROD cleanup levels based on final status survey data for surface materials in a given survey unit is as follows:

<u>Compliance Evaluation Criteria</u>

1. At least 95 percent of gamma readings across the survey unit must be less than or equal to the gamma cutoff value in use at the time of the final status survey.
2. At least 95 percent of predicted Ra-226 concentrations in surface materials based on gamma survey data and the gamma/Ra-226 correlation (i.e. the respective regression equation in use at the time of the final status gamma survey) must be less than or equal to the ROD cleanup level (4.7 pCi/g).
3. One-sample proportion testing of direct sampling results for Ra-226 indicates (at the 95% confidence level) that at least 95 percent of the survey unit meets the Ra-226 cleanup level.
4. At least 95 percent of the offsite analysis results for uranium and Pb-210 must be less than or equal to respective ROD cleanup levels.
5. No single individual surface material sampling result for any analytical parameter can exceed a secondary cleanup level of twice the respective ROD cleanup level. If this occurs, a secondary investigation will be triggered to determine if a hot spot above the ROD cleanup level exists. If so, it will be remediated to ROD cleanup levels, re-

surveyed, and results documented. If not, the secondary investigation sampling data will be documented and will replace the original sample result.

6. Exposed bedrock will not be evaluated in a context of compliance with ROD cleanup levels, but gamma scans must be conducted to document gamma readings above the exposed bedrock.

Data QA/QC Criteria (see QAPP for further details)

1. Daily QC measurements for field gamma measurement instruments and associated scanning systems must fall within established control limits (see QAPP and AS-SOP 6) for the corresponding period of use in the field.
2. Daily QC measurements for the XRF instrument and associated measurement methods must fall within established control limits (see QAPP and AS-SOP 5) for the corresponding period of use in the field.
3. Daily QC measurements for the onsite gamma spectroscopy system (measurements of 2 site-specific soil Ra-226 calibration standards) must fall within established control limits (see QAPP and AS-SOP 4) for the corresponding period of onsite use.
4. At least 90 percent of onsite analysis results for Ra-226 in field samples based on onsite gamma spectroscopy should fall within the limits of 90 percent prediction intervals on the current method calibration algorithm (expressed in units of activity concentration). This will limit the probability of under-estimation of equivalent full ingrowth results at the offsite lab (a Type I estimation error) to less than 5 percent.
5. At least 90 percent of XRF data should fall within 90 percent prediction intervals on method calibration (regression) curves. This will limit the probability of under-estimation of equivalent results at the offsite lab (a Type I estimation error) to less than 5 percent.

S.4.2.3.2 Reporting

For each survey unit, a separate final status survey report will be prepared by the Field Program Director and submitted to the QAM and Supervising Contractor for review. The report will include summary data tables, mapped data (e.g. color-coded gamma survey results along with sampling locations and respectively annotated soil Ra-226 concentrations), along with a narrative regarding all qualitative, spatial and statistical analysis results in a context of the protocols, information and decision criteria detailed in the previous section. Upon review/approval of the Field Program Director and QAM, the report will be submitted to EPA for review and comment. Once EPA comments are addressed and the report is approved by the

EPA, the survey unit will be considered to be in compliance with ROD cleanup levels and to be acceptably remediated.

S.4.3 Final Status Surveys for Sediments

S.4.3.1 XRF Surveys for Mine Drainages

Final status surveys for sediment along mine drainages will first involve in-situ XRF surveys for metals along five transects that are parallel to the segment of drainage channel representing each sediment survey unit (Figure S-12). The middle transect will fall in the thalweg (center) of the channel, with two transects on either side. On each side of the thalweg transect, one transect will represent the approximate lateral extent of excavation, and the other will represent margins about 10 meters beyond the lateral extent of excavation (Figure S-12). The density of in-situ XRF measurements (Method 6200) may depend on the exact technology used (e.g. manual data collection or integrated XRF/GPS systems with automated data logging), but will include a measurement spacing of no less than 50 meters along each transect.

Random and biased confirmation sampling will be conducted at about 5 percent of in-situ XRF survey sediment measurement locations (1 out of every 20 locations) for confirmatory analysis in the onsite soils lab with the intrusive XRF methodology. Both XRF methods will be performed according to the specifications of Method 6200 and procedures found in AS-SOP 5. Ten percent (10%) of the XRF soil cups analyzed onsite with the intrusive XRF method will be sent offsite for confirmatory analysis of all applicable metals (Table S-4).

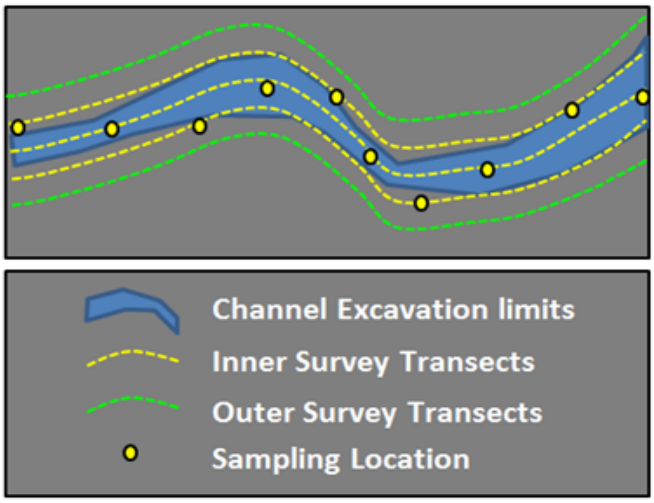

Figure S-12 – In-situ XRF survey transects and laterally alternating direct soil sampling locations for onsite intrusive XRF analysis and offsite laboratory analysis.

S.4.3.2 Gamma Surveys for Mine Drainages

Final status gamma surveys will be conducted along mine drainages to document final gamma readings and to indirectly evaluate compliance with the Ra-226 cleanup level for sediments between direct sampling locations (see Section S.4.1 and Attachment S-1). The methods and protocols indicated in Section S.2.1 and procedures found in AS-SOP 6 for recorded GPS-based scanning will be followed, and applicable data review and final status decision criteria as indicated in Section S.4.2 for surface materials will be applied. A preliminary gamma cutoff value for sediments has been established (Attachment S-1), though as for surface materials this value could change slightly based on future data.

S.4.3.3 Sediment Sampling

Final status sediment sampling will not be conducted until after in-situ XRF and gamma surveys along mine drainage channels have been performed and evaluated (Sections S.4.3.1 and S.4.3.2). Once a survey unit has been determined ready for direct sampling of sediments, samples will be collected (to a depth of 6 inches) along the three innermost transects as described in Section S.4.3.1 and in a laterally alternating grid sampling pattern (Figure S-12). At minimum, a total of 75 final status sediment samples will be collected within each drainage channel survey unit. A field duplicate sample will be collected at a minimum of 5 percent of sampling locations (at least 4 field duplicates for every 75 samples – see QAPP).

Of the minimum 75 samples from each mine drainage survey unit to be collected and analyzed in the onsite soils lab, at least 33 percent (25+ samples), plus at least one field duplicate (a minimum of 26+ samples total), will subsequently be sent to an approved commercial laboratory for analysis of all parameters indicated in Table S-5 (Section S.2.4). Selection of the 33 percent of final status samples from each survey unit to be sent offsite for analysis will be performed with a random number generator. Paired analytical results for the 33 percent of samples analyzed both onsite and offsite will be statistically compared with parametric T-tests and non-parametric Wilcoxon Rank Sum tests to evaluate agreement between onsite and offsite analytical results. Quality Assurance / Quality Control requirements and SOPs for surface material and sediment sampling, onsite processing and onsite/offsite laboratory analysis, are provided in the QAPP.

Separate from the above minimum number of final status sediment samples (75+ samples) to be collected and analyzed for final status statistical hypothesis testing, the confirmatory samples

from the in-situ XRF survey that are collected at 5 percent of in-situ XRF survey locations and are analyzed onsite (via intrusive XRF), along with the fraction of these samples to be subsequently analyzed for applicable metals at the offsite (10 percent), will also be evaluated. These samples will be analyzed only for applicable metals (not for radiological parameters). The objective is not for final status hypothesis testing, but for confirmation of in-situ XRF survey results. These sample results will be evaluated only in this context for final status survey data assessments.

At each mine drainage sampling location, a minimum of 350 grams of sediment and/or soil will be collected in accordance with the procedures indicated in AS-SOP 1 and AS-SOP 2. To the extent possible, any rocks larger than about 1 cm in diameter in the bulk sample will be discarded at the sampling location. All sediment samples will be processed and analyzed in the onsite soils lab for Ra-226 and metals according to the methodologies described in Section S.2.2 (gamma spectroscopy) and Section S.2.3 (intrusive XRF analysis), and in accordance with the procedures provided in AS-SOP 3, AS-SOP 4 and AS-SOP 5.

S.4.3.4 Determination of Compliance for Sediments

Once all final status survey data for a given survey unit have been obtained, data quality and completeness evaluations will be conducted, followed by qualitative, quantitative and statistical data assessments for compliance with ROD cleanup levels. This section describes the methods and protocols that will be used to evaluate and determine compliance with ROD cleanup levels for sediments.

S.4.3.4.1 Data Analysis

Once qualified as useable data in accordance with the QAPP, final status survey results will be mapped and spatially, qualitatively, quantitatively and statistically evaluated to assess compliance with ROD cleanup levels. Final status determination of compliance with the cleanup levels for sediments will be evaluated in several ways. First, at least 95 percent of the in-situ XRF survey data for each COC within the survey unit should be less than or equal to respective in-situ XRF cutoff values (Section S.2.3.1). Secondly, at least 95 percent of the confirmatory sampling and intrusive XRF analysis results for the in-situ XRF survey must fall within 95 percent prediction intervals on the in-situ XRF method calibration (regression) curve (this will confirm that the in-situ XRF survey data are reliable in terms of being categorized as screening level data).

With respect to final status gamma surveys along mine drainage survey units, at least 95 percent of gamma readings along the mine drainage survey unit must be less than or equal to the gamma cutoff value for sediments that is in use at the time of the final status survey. In addition, the regression equation for the gamma/Ra-226 correlation in use at the time of the final status survey will be used to convert raw gamma scan data into estimates of soil Ra-226 concentrations along the entire mine drainage survey unit. Descriptive statistics of the results will be generated, and respective data will be mapped. At least 95 percent of these gamma-based estimates of Ra-226 concentrations must be equal to or less than the respective ROD cleanup level (13 pCi/g). This, combined with the mapped data, will provide another form of assurance that the survey unit is in compliance with the Ra-226 cleanup level, based on nearly perfect knowledge of the entire population of gamma exposure rates across the survey unit.

With respect to statistical hypothesis testing for the 75+ final status sediment sampling results generated for this purpose (Section S.4.3.3), the discussion of statistical testing considerations and protocols indicated in Section S.4.2.3.1 for surface materials also applies to hypothesis testing for sediments. One-sample proportion tests will be used to statistically determine whether sediment sampling results indicate (at the 95 percent confidence level) that a 95 percent rate of compliance has been achieved along the mine drainage survey unit for each ROD sediment parameter indicated in Table S-2.

The statistical hypothesis testing will be based on analytical results for final status samples collected in each survey unit (at least 75 samples). For duplicate analytical results generated for the same samples via both onsite and offsite laboratory analyses (which for sediments will represent 33 percent of samples for all parameters except Pb-210, U-234 and U-238), only the offsite laboratory results will be combined with the remaining 66 percent of sample results obtained onsite to represent the final status data set to be used for the statistical testing. Double counting of duplicate onsite/offsite results for the same samples as though they were independent sampling results will not be allowed as this would violate basic statistical testing assumptions. Selection of the 33 percent of samples from each survey unit to be sent offsite will be performed with a random number generator.

In the case of sediment samples, analysis of Pb-210, U-234 and U-238 can only be analyzed at the offsite laboratory (sample digestion and alpha spectroscopy is required for these analytes). This means that there will only be 25+ sampling results for these three radiological sediment parameters. Given this circumstance, at least 95 percent of results for these parameters must be at or below their respective cleanup levels, and statistical testing for other parameters to

evaluate compliance will be used as a surrogate to infer statistical compliance for these parameters. The validity of this protocol is based on several factors. First, as indicated in the Mine Waste Investigation study (Miller Geotechnical, 2011), manganese is expected to be the limiting cleanup parameter in most cases along mine drainages. If the statistical testing indicates that at least 95 percent of the mine drainage survey unit has manganese concentrations at or below the corresponding ROD cleanup level, then in general the concentrations of Pb-210, U-238 and U-234 can also be expected to meet this criterion.

Secondly, if the statistical testing indicates that at least 95 percent of the mine drainage survey unit has uranium concentrations at or below the ROD cleanup level for uranium, U-238 and U-234 can also be expected to meet this criterion for their respective cleanup levels. This expectation is supported by a 2002 study of radiological equilibrium in various environmental media at the Site (URS, 2002). In that study, a consistent and statistically significant linear relationship ($R^2 = 0.94$) between U-238 and its U-234 decay product was found to exist in impacted mine sediments at the Site, with a slight degree of disequilibrium present relative to normal relative isotopic activity concentrations found in natural (total) uranium. Based on this information, estimates of isotopic uranium activity concentrations that can be expected for impacted sediments were calculated for a hypothetical circumstance where the measured natural (total) uranium concentration is exactly equivalent to the uranium cleanup level (Table S-12). The results indicate that even with the observed degree of disequilibrium, if the sediments are in compliance with the uranium cleanup level, U-238 and U-234 will also be in compliance with their corresponding cleanup levels. This would also be true if U-238 and U-234 were in perfect secular equilibrium.

In the event that any given sample result for any COC exceeds a ROD cleanup level by more than twice its respective ROD cleanup level, a secondary investigation will be conducted. If a hot spot above ROD cleanup levels is confirmed, it will be remediated to ROD cleanup levels and re-sampled to document compliance. If not confirmed, the secondary investigation results will replace the original result that incorrectly suggested a potential hot spot. Any areas excavated to naturally mineralized bedrock will be characterized with gamma surveys to document approximate exposure rates, but compliance with ROD cleanup levels will not be evaluated.

Table S-7 – Calculated U-238 and U-234 Concentrations in Impacted Sediments if Natural (total) Uranium is Present at the ROD Cleanup Level for Uranium

		ROD cleanup level (pCi/g)
ROD cleanup level for U (mg/kg)	92.3	-
ROD cleanup level for U expressed in terms of activity concentration (pCi/g)	62.5	62.5
Equivalent U-238 activity concentration calculated based on disequilibrium in impacted sediments (pCi/g)*	27.4	31
Equivalent U-234 activity concentration calculated based on disequilibrium in impacted sediments (pCi/g)*	33.8	41

*Calculated estimate based on data provided in URS, 2002.

As previously indicated, in the case that excavation encounters bedrock before cleanup levels are achieved, a gamma survey will be conducted over the bedrock to document gamma readings, but such areas will not be evaluated with respect to ROD cleanup levels. The sediment removal decision diagram presented in Section S.1.0 (Figure S-5) is generalized and applies to both remedial support and final status surveys. Respective decision criteria, including major elements of those detailed in this Section, are summarized in Figure S-5. A more complete/detailed summary listing of decision criteria for evaluation of compliance with ROD cleanup levels based on final status survey data for sediments in a given survey unit is as follows:

Compliance Evaluation Criteria

1. At least 95 percent of the in-situ XRF survey data for each ROD cleanup level parameter within the survey unit should be less than or equal to respective in-situ XRF cutoff values.
2. At least 95 percent of gamma survey data collected along the mine drainage survey unit must be less than or equal to the gamma cutoff value for sediments that is in use at the time of the final status survey.
3. At least 95 percent of gamma-survey based estimates of Ra-226 concentrations must be equal to or less than the respective ROD cleanup level (13 pCi/g).
4. One-sample proportion testing of direct sampling results for each COC indicates (at the 95 percent confidence level) that at least 95 percent of the survey unit meets the corresponding cleanup level.

5. Statistical testing for manganese and uranium will serve as surrogates for statistically evaluating compliance with cleanup levels for Pb-210, U-238 and U-234, but at least 95 percent of the offsite analysis results for Pb-210, U-238 and U-234 must be less than or equal to respective ROD cleanup levels.
6. No single individual sediment sampling result for any analytical parameter can exceed a secondary cleanup level of twice the respective ROD cleanup level. If this occurs, a secondary investigation will be triggered to determine if a hot spot above ROD cleanup levels exists. If so, it will be remediated to ROD cleanup levels, re-surveyed, and results documented. If not, the secondary investigation sampling data will be documented and will replace the original sample result.
7. Exposed bedrock will not be evaluated in a context of compliance with ROD cleanup levels, but gamma scans must be conducted to document gamma readings above the exposed bedrock.

<u>Data QA/QC Criteria (see QAPP for further details)</u>

1. Daily QC measurements for the XRF instrument and associated measurement methods must fall within established control limits (see QAPP and AS-SOP 5) for the corresponding period of use in the field.
2. At least 90 percent of the confirmatory sampling/intrusive XRF analysis results for the in-situ XRF field survey must fall within 90 percent prediction intervals on the in-situ XRF method calibration (regression) curve. This will limit the probability of under-estimation of equivalent results at the offsite lab (a Type I estimation error) to less than 5 percent.
3. Daily QC measurements for field gamma measurement instruments and associated scanning systems must fall within established control limits (see QAPP and AS-SOP 6) for the corresponding period of use in the field.
4. Daily QC measurements for the onsite gamma spectroscopy system (measurements of 2 site-specific soil Ra-226 calibration standards) must fall within established control limits (see QAPP and AS-SOP 4) for the corresponding period of onsite use.
5. At least 90 percent of onsite analysis results for Ra-226 in field samples based on onsite gamma spectroscopy should fall within the limits of 90 percent prediction intervals on the current method calibration algorithm (expressed in units of activity

concentration). This will limit the probability of under-estimation of equivalent full-ingrowth results at the offsite lab (a Type I estimation error) to less than 5 percent.

S.4.3.4.2 Reporting

For each mine drainage survey unit, a separate final status survey report will be prepared by the Field Program Director and submitted to the QAM and Supervising Contractor for review. The report will include summary data tables, mapped data (e.g. color-coded gamma survey results along with sampling locations and respectively annotated soil Ra-226 concentrations), along with a narrative regarding all qualitative, spatial and statistical analysis results in a context of the protocols, information and decision criteria detailed in the previous section. Upon review/approval of the Field Program Director and QAM, the report will be submitted to EPA for review and comment. Once EPA comments are addressed and the report is approved by the EPA, the survey unit will be considered to be in compliance with ROD cleanup levels and to be acceptably remediated.

S.5.0 DATA QUALITY ASSURANCE / QUALITY CONTROL (QA/QC)

All gamma surveys, surface material/sediment sampling, and onsite/offsite laboratory measurements used to guide remedial excavations and to conduct final status surveys will subject to the data QA/QC program as detailed in the QAPP (Attachment S-2). The purpose of this program to ensure that the analytical data to be generated will be of sufficient quality to reliably support correct decisions regarding compliance with ROD cleanup levels. To meet this objective, it is necessary that the analytical uncertainties introduced by variability in instruments, laboratory methods, and survey techniques is minimized and can be qualitatively and quantitatively assessed in terms of data accuracy and precision.

In general, quality assurance (QA) includes qualitative aspects of program planning and operational management that are necessary to ensure an appropriate overall analytical design and proper implementation of planned methods and procedures. Quality control (QC) includes quantitative measures to monitor analytical method performance and to allow respective estimation of data uncertainty (accuracy and precision). A generalized summary of major elements of QA/QC program and requirements is as follows:

<u>Program QA Summary</u>:

- All gamma surveys, soil/sediment sampling, and supporting measurements used to guide remedial excavations and to conduct final status surveys will be subject to the data

QA/QC program outlined in the QAPP, and an organizational structure has been designed for the program to assure proper implementation.

- Standard operating procedures (SOPs) provided in the QAPP will be followed for gamma surveys, soil/sediment sampling, onsite gamma spectroscopy and XRF analyses, along with other protocols described in the QAPP such as sample processing and offsite laboratory submittals, data management and data QA/QC review. The program's organizational functions include oversight, data reviews and audits to assure these program elements are properly followed throughout the duration of the project.

- All staff involved in the oversight, management and implementation of remedial support and final status surveys will be qualified by education, training and experience to perform these functions. The respective organizational structure and corresponding responsibilities have been defined in the QAPP. The professional qualifications of staff involved will be maintained in the project records and can be included in an appendix to applicable portions of final reports.

- The DQO process was used to develop the analytical approaches necessary to produce environmental data of the type, quantity and quality necessary to reliably support correct decisions regarding the extent of remedial excavations required, and to subsequently determine compliance with ROD cleanup levels. DQO statements are provided in the QAPP.

- The radioanalytical approaches and methodologies to be used are proven to be effective for remediation of radiologically impacted sites, including at the nearby Dawn Millsite and at many other sites across the U.S. These approaches/methods are consistent with relevant regulatory guidance (e.g. MARSSIM) and their effectiveness is supported by published results in peer-reviewed scientific journals.

- Detailed field notes will be kept in field logbooks to document daily activities and any relevant observations regarding environmental or equipment related conditions that could affect data.

- Any offsite laboratory used to analyze soil or sediment samples will be fully qualified and appropriately accredited for analysis of the constituents specified in the ROD in terms of cleanup levels for surface materials and sediments.

- Strict chain-of-custody protocols will be followed for sampling shipping and offsite laboratory analyses. Copies of chain-of-custody forms will be kept on file.
- All data generated onsite will be reviewed daily for general consistency with expectations based on QA/QC measurements and other Site data, and all final status survey data will be verified/validated by qualified staff, including independent review by the Project Quality Assurance Manager, prior to quantitative, spatial and statistical data analysis and inclusion of results in final reports.

Project QC Summary:

- Calibration of gamma detectors and XRF instruments will be performed as specified in the QAPP and calibration data will be assessed to provide quantitative information related to instrument accuracy. Calibration certificates (as applicable) will be kept on file and will be included in appendices to applicable portions of final reports.
- Daily QC measurements will be performed in the field for all instruments and analysis systems to ensure proper instrument/system performance and this data will be used to help quantify measurement precision and data reproducibility.
- Sample analysis in the onsite soils lab (using both gamma spectroscopy and XRF) will include replicate sample analyses and collection/analysis of field duplicates to help quantify analytical precision, total propagated data uncertainty, and natural variability due to small-scale spatial heterogeneity in soil concentrations at sampling locations. A specified percentage of samples analyzed onsite will be sent to the offsite lab for confirmatory analysis and for additional analysis where required by the Plan (specifics are provided in Sections S.3.2.3 and S.3.2.6 and in the QAPP).
- The commercial laboratory used for analysis of soil/sediment samples will perform extensive QC measurements for each batch of sample results (e.g. duplicate sample analyses, sample spikes, method blanks, etc.) in order to provide quantitative indications of accuracy and precision. These requirements are extensively detailed in the QAPP.

The QAPP was developed to provide highly detailed plans regarding the above generalized summary information, and to the greatest extent possible, to follow EPA guidance regarding format and content. EPA guidance does not address all of the analytical approaches to be used on this project, but the QAPP was developed for general consistency with the technical intent of that guidance.

S.6.0 REFERENCES

Adsleya, I., M.M. Daviesa, R. Murleya, I. Pearmana, and M. Scireab, 2004. 3D GPS mapping of land contaminated with gamma-ray emitting radionuclides. Applied Radiation and Isotopes 60, 579–582.

Johnson, J.A., H.R. Meyer, and M. Vidyasagar, 2006. Characterization of Surface Soils at a Former Uranium Mill. Operational Radiation Safety. Supplement to Health Physics, Vol. 90, February.

Meyer, R., M. Shields, S. Green, and J. Johnson, 2005. A GPS-based system for radium/uranium contamination gamma scanning. Uranium Mining and Hydrogeology IV. Broder J. Merkel, Andrea Hasche-Berger (Editors). Uranium in the Environment, conference proceedings, Freiberg, September.

Miller Geotechnical Consultants, Inc. (Miller Geotechnical). 2011. Mine Waste Investigations. February 11.

Shepherd Miller Inc. (SMI). 1999a. Technical Memorandum R-D. (Radiological Characterization Site Maps for the Midnite Mine). Submitted to Bureau of Land Management, Spokane District, 1103 N. Fancher, Spokane, WA 99212. June 23. SMI# 01-252 Task 6.

Shepherd Miller Inc. (SMI). 1999b. Shepherd Miller, Inc. (SMI). 1999b. Midnite Mine Data Transmittal Report R-4 (On-Site Soils Radiological Survey). Submitted to Bureau of Land Management, Spokane District, 1103 N. Fancher, Spokane, WA 99212. June 9.

URS Corporation (URS). 2002. Radioactive Equilibrium Evaluation for Midnite Mine RI/FS. August.

URS Corporation (URS). 2005. Final Remedial Investigation Report for Midnite Mine, Stevens County, Washington. September 30.

U.S. Environmental Protection Agency (EPA). 2002. Role of Background in the CERCLA Cleanup Program. OSWER 9285.6-07P.

U.S. Environmental Protection Agency (EPA). 2006a. Midnite Mine Superfund Site, Spokane Indian Reservation, Washington, Record of Decision. Office of Environmental Cleanup. EPA Region 10. September.

U.S. Environmental Protection Agency (EPA). 2007. ProUCL Version 4.0 Technical Guide. (*A technical manual for EPA statistical software*). EPA/600/R-07/041. April.

U.S. Environmental Protection Agency (EPA). 2011. Spokane Tribe of Indians Airborne Radiological Surveys. September.

U.S. Nuclear Regulatory Commission (NRC), Office of Nuclear Regulatory Research, 2000. Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM). NUREG-1575. August.

Vitkus, T.J., and E.N. Bailey, 2007. Advantages and Lessons Learned Using GPS-Enabled Gamma Scanning for the Characterization Survey of a Large Thorium/Uranium Site. Health Physics Society Mid-Year Meeting, Knoxville, Tennessee, January 24.

VSP Development Team (VSP). 2012. Visual Sample Plan: A Tool for Design and Analysis of Environmental Sampling. Version 6.2d. Pacific Northwest National Laboratory. Richland, WA. http://vsp.pnnl.gov.

Whicker, R., P. Cartier, J. Cain, K. Milmine, and M. Griffin, 2008. Radiological Site Characterizations: Gamma Surveys, Gamma/Ra-226 Correlations and Related Spatial Analysis Techniques. Operational Radiation Safety, Health Physics, Vol. 95 (Supplement 5): S180-S189; November.

Whicker, R., M. Whicker, J. Johnson, and H. Meyer, 2006. Mobile Soils Lab: On-site Radiological Analysis Supporting Remedial Activities. Health Physics. Operational Radiation Safety. 91(2) Supplement 1:S24-S31, August.

Attachment S-1

Technical Basis

Midnite Mine Superfund Site
100 Percent Design

Attachment S-1 – Technical Basis

Note: This attachment (Attachment S-1) provides the Technical Basis for the preliminary Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (Appendix S). Appendix S and its supporting attachments are intended to satisfy the requirement for a Preliminary Confirmation Sampling and Remedial Action Effectiveness Monitoring Plan as stipulated by the Consent Decree.

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Prepared for:

Dawn Mining Company
PO Box 250
Ford, Washington 990413

and

Newmont USA Limited
6363 South Fiddler's Green Circle
Greenwood Village, Colorado 80111
Prepared By:

Prepared by:

Environmental Resources Group (ERG)
8809 Washington St NE, Suite 150
Albuquerque, New Mexico, 87113

TABLE OF CONTENTS

1.0 INTRODUCTION 1

2.0 APPLICATION OF THE DATA QUALITY OBJECTIVES (DQO) PROCESS 1

3.0 ANALYTICAL APPROACHES FOR SURFACE MATERIALS 2

3.1 GAMMA SURVEYS AND GAMMA/RADIUM-226 CORRELATIONS 4
3.2 GAMMA SCANNING SENSITIVITY 7
3.3 COCS OTHER THAN RADIUM-226 9
3.4 GAMMA SHINE EFFECTS 11
3.5 ONSITE SOILS LABORATORY 12
3.6 RAPID TURNAROUND ANALYSIS AT AN OFFSITE LABORATORY 17

4.0 ANALYTICAL APPROACH FOR SEDIMENTS 19

5.0 CONSIDERATIONS REGARDING NATURALLY MINERALIZED BACKGROUND 22

6.0 FINAL STATUS SURVEY CONSIDERATIONS 23

6.1 STATISTICAL APPROACH 23
6.2 CONSIDERATIONS REGARDING SURVEY UNIT SIZE AND NUMBER OF SAMPLES 25

7.0 REFERENCES 28

LIST OF FIGURES

Figure 1 - Left: Site-specific statistical relationship between gamma exposure rate readings and soil Ra-226 concentrations, along with 95% upper prediction limits, based on correlation data provided in the Mine Waste Investigations report (Miller Geotechnical, 2011). Right: The same graphical data, zoomed in at the lower end of the scale to allow visualization of two probabilistic gamma cutoff levels (one for the 95% UPL and one for a 75% UPL). 5

Figure 2 – Left: Separate correlations for lower and upper range correlation data from the Mine Waste Investigations study (Miller Geotechnical, 2011). Right: The same correlations, zoomed in at the lower end of the scale with a 95% UPL on low range data showing that a gamma cutoff value based on the low range data provides a 95% statistical probability of compliance at about 32 μR/hr, and that a gamma cutoff value of 20 μR/hr provides a 95% probability that Ra-226 concentrations will be less than about 3.7 pCi/g (well below the cleanup criterion for Ra-226). 6

Figure 3 – Left: Relationship between uranium and Ra-226 concentrations in 98 soil samples collected at depth underneath mine waste rock deposits (Miller Geotechnical, 2011). The pink line represents a theoretical line of equilibrium between uranium and Ra-226 concentrations across the range of observed values. Right: Frequency histogram and corresponding table of percentiles for observed uranium/Ra-226 ratios. 10

Figure 4 – Left: Relationship between Ra-226 and Pb-210 concentrations in 98 soil samples collected at depth underneath mine waste rock deposits (Miller Geotechnical, 2011). Right: Frequency histogram for observed Ra-226/Pb-210 ratios. 11

Figure 5 – Left: Relationship between full radon ingrowth estimates of Ra-226 based on NaI gamma spectroscopy measurements in an onsite soils lab, and corresponding results by HPGe analysis from a commercial laboratory during the 2005 Dawn Mill Site cleanup. Right: Relationship between day zero estimates of Ra-226 based on NaI gamma

spectroscopy measurements in the onsite soils lab (with ingrowth correction factor applied), and corresponding full-ingrowth results by HPGe analysis from a commercial laboratory. Both graphs are adapted from Whicker et al. (2006). 13
Figure 6 – ROIs for Pb-214 and Bi-214. 14
Figure 7 – Left: Linear regression curves for 87 paired HPGe/NaI results for Ra-226, where the NaI results were based on each of the 3 individual ROIs, the mean of the 3 ROIs, and on a single calibration using the sum of all counts in the 3 ROIs. On average, using the sum all counts in the 3 ROIs provides the most accurate results relative to HPGe measurements across the range of values examined. Right: Comparison of the mean result (± 1 standard deviation) for each of the NaI-based estimation methods versus the mean result for HPGe analysis at an offsite commercial laboratory. The "Summed ROIs" method produces distributional characteristics (mean, median and range) that most closely match HPGe results. 14
Figure 8 – Example calibration curve based on the sum of day zero NaI counts for three separate ROIs, versus full-ingrowth activity results by HPGe analysis at a commercial laboratory. 15
Figure 9 – Left: Relationship between Ra-226 and Pb-210 concentrations in 98 soil samples collected at depth underneath mine waste rock deposits as part of the Mine Waste Investigation study (Miller Geotechnical, 2011). Right: Relationship between Ra-226 and Pb-210 concentrations in 64 soil samples collected in non-background areas as part of RI/FS activities (URS, 2002). 16
Figure 10 – Soil Ra-226 measurement data versus elapsed Rn-222 ingrowth time for sealed samples. 17
Figure 11 – Determination of an initial gamma cutoff value providing a 95% probability of compliance with the 13 pCi/g cleanup level for Ra-226 in sediments (33 μR/hr) based on data from the Mine Waste Investigation study (left) and the White Tail Creek study (right). 21

LIST OF TABLES

Table 1 – Calculated Scan MDC values for Ra-226 in surface soils using various hypothetical scan methodologies for several small "hot spot" dimensions. 8

1.0 INTRODUCTION

This attachment to Appendix S (Attachment S-1) provides the technical basis and scientific/regulatory rationale for the analytical and statistical approaches and methods specified in the Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (Appendix S). The majority of information provided in this attachment is related to the fact that radiological parameters are a major aspect of Site impacts and cleanup criteria at the Midnite Mine (Site), and that surface materials (mine waste rock and impacted soils) represent a vast majority of areas requiring remediation and volumes of material to be removed and consolidated into Pits 3 and 4. Although the U.S. Environmental Protection Agency (EPA) has developed considerable guidance in terms of determining cleanup levels for radiological parameters at CERCLA sites, specific guidance on methodologies for evaluating compliance appears to be generally focused on direct soil sampling and analysis (e.g. EPA, 2006a), perhaps with deference as applicable to the Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM) (NRC, 2000).

Included in this attachment are technical discussions and relevant rationale pertaining to the Data Quality Objectives (DQO) process and resulting specific analytical approaches and protocols reflected in Appendix S. In addition to consideration of radiological aspects of the remedial action project, other important issues such as statistical considerations, non-radiological parameters in sediments and mineralized bedrock are discussed. Finally, this attachment also provides certain information intended to address many related EPA comments on the initial draft version of Appendix S as provided in the 60% Basis of Design report, as well as subsequent comments stemming from several technical meetings on Appendix S issues.

2.0 APPLICATION OF THE DATA QUALITY OBJECTIVES (DQO) PROCESS

In 2000, the U.S. Nuclear Regulatory Commission (NRC), U.S. Department of Energy (DOE), and the EPA released MARSSIM as a unified intra-agency technical approach and guidance document for assessing remedial performance at radiologically impacted sites (NRC, 2000). MARSSIM has gained broad regulatory acceptance in the U.S. for addressing a wide variety of radiological impacts and site-specific circumstances. Because the majority of impacts at the Midnite Mine are largely radiological in nature, a MARSSIM-based approach is an appropriate methodology for evaluating and demonstrating compliance with ROD cleanup levels. The first step described in MARSSIM is to apply a Data Quality Objectives (DQO) Process in the

planning phases of the project in order to ensure that the design of radiological surveys will produce data of sufficient quality and quantity to support decisions as to whether or not the remediation has achieved specified remedial objectives and quantitative remedial criteria.

Also in 2000, the EPA released two separate non-mandatory guidance documents detailing the DQO Process, one in a more broadly applicable context (e.g. for non-radiological types of contamination such as metals, chemicals, etc.) (EPA, 2000a), and another tailored for hazardous waste sites under programs such as CERCLA (EPA, 2000b). The DQO Process guidance for hazardous waste sites indicates the following relevant information that can be applied in a context of analytical support/verification planning for remedial actions at the Midnite Mine:

- The objective of this guidance is to "…help site managers plan to collect data of the right type, quality, and quantity to support defensible site decisions."
- While focused on Remedial Investigations (RIs) conducted as part of the CERCLA process, this guidance can be beneficial for other applications, such as planning analytical approaches to meet the requirements of remedial actions. For other applications, the DQO Process may need to be adapted to suit the specific problem, including use of "…a more liberal interpretation of the quantitative steps."

General EPA guidance on the DQO Process was updated in 2006 (EPA, 2006b), additionally describing it as follows:

- "…flexible and iterative, and applies to both decision-making (e.g. compliance/non-compliance with a standard) and estimation (e.g. ascertaining the mean concentration level of a contaminant)."

The DQO Process is helpful for planning for both remediation support surveys as well as final status surveys at the Midnite Mine, and was utilized in evaluating the analytical approaches discussed in this Technical Basis document and in developing respective plans as detailed Appendix S. The general language for the seven steps of the DQO Process as indicated in the 2000 guidance (EPA, 2000b) for hazardous waste and CERCLA sites was adopted for planning purposes. The DQO statements for the Remedial Support and Verification Plan (Appendix S) are provided in the Quality Assurance Project Plan (QAPP) (Attachment S-2 to Appendix S).

3.0 ANALYTICAL APPROACHES FOR SURFACE MATERIALS

Because constituents of concern (COCs) for surface materials are radioactive elements, the methods available for their detection and measurement in surface materials are not limited to

direct soil sampling. Under MARSSIM, radiological scanning of materials in-situ in the field plays an important role in demonstrating compliance. However, since MARSSIM was published in 2000, advanced GPS-based gamma survey technologies and associated characterization techniques have become a mature, proven, and widely used scientific methodology. These new methods have greatly increased the speed, efficiency, and accuracy of characterizing the quantitative and spatial distribution of radiological contamination, and have essentially eliminated the human surveyor error component of earlier scanning techniques (effectively improving scan sensitivity).

Although cleanup levels for surface materials at the Midnite Mine are based solely on radiological COCs, direct soil sampling is inherently limited in terms of spatial coverage. Demonstrating compliance based primarily on soil sampling, even with great emphasis on the accuracy and precision of individual sampling results (e.g. lab certifications, detection limits, analytical QA/QC, etc.), must still rely on statistical tests to infer something about population characteristics based on limited sampling data. The spatial uncertainties associated with a soil sampling approach far exceed analytical uncertainties associated with individual sampling results, no matter how accurate and precise those individual sampling results may be.

In the case of the Midnite Mine, radium-226 (Ra-226) is a COC of major importance in terms of radiological health considerations. Gamma emissions from Ra-226 and its short-lived gamma-emitting decay products in surface materials can be readily detected in-situ with gamma surveys. Gamma scans can essentially define the entire population of radioactive emissions associated with Ra-226 in terrestrial materials, and gamma/Ra-226 correlations can be used to estimate Ra-226 concentrations in a probabilistic manner based on gamma survey data. Though such estimates have greater uncertainty versus direct soil sampling at a given location, they are far more certain in terms of evaluating overall compliance across a survey unit versus statistical tests based on limited soil sampling data. This principle is changing the way health physicists evaluate radiological data, and represents a scientifically supported shift of emphasis in terms of the basis for decision making versus the traditional focus on individual sampling or measurement results for radiological parameters (Lively, 2013).

With respect to COCs other than Ra-226 for surface materials, there is evidence of radiological disequilibrium between uranium, Ra-226 and Pb-210 (Miller Geotechnical, 2011). However, there is also evidence of consistent trends in the relative directions of this disequilibrium, and corresponding cleanup levels specified in the ROD are set at levels that can accommodate a considerable degree of radiological disequilibrium. As discussed in detail in Section 3.3, study

of existing data indicates that cleaning up surface materials based primarily on Ra-226 concentrations will ensure with a probability on the order of 95 percent or higher that uranium and Pb-210 will also be successfully remediated to ROD cleanup levels.

3.1 Gamma Surveys and Gamma/Radium-226 Correlations

Radiological parameters are a major aspect of Site impacts and cleanup criteria at the Midnite Mine. One of these parameters, Ra-226, is a decay product of the U-238 decay series and is thus generally associated with uranium mine wastes, impacted soils and sediments at the Site. Radium-226 and its short-lived decay products have a strong gamma radiation signature that is readily detected in-situ in the field at or near the surface of excavated areas using hand-held radiation detection instruments. As a result, gamma survey screening is a widely used, and generally preferred, methodology for assessing Ra-226 levels in materials exposed at the ground surface due to remedial excavations. In addition to providing immediate, real-time assessment capability, gamma survey screening allows 100 percent spatial coverage of the land areas being evaluated.

Quantitative assessment with gamma survey screening techniques requires site-specific knowledge of the statistical relationship between measured terrestrial gamma radiation and the concentrations of Ra-226 in terrestrial materials residing at the ground surface. A well established and widely used analytical approach for field estimation of Ra-226 concentrations in exposed surface soils involves gamma/soil Ra-226 correlations (NRC, 2003; Johnson et al., 2006; Meyer et al., 2005; Whicker et al., 2006 and 2008). If a correlation is statistically significant, a predictive gamma cutoff value, developed based on probabilistic statistical principles, can be used to guide remedial excavations such that when an area has been excavated until all gamma readings are below the cutoff value, there is an acceptable statistical probability (e.g. 95 percent) of compliance with the soil Ra-226 cleanup criterion. This gamma cutoff value can also be used after remediation to help demonstrate compliance as part of final status surveys.

Based on evaluation of previous studies where gamma/soil Ra-226 correlations have been developed at the Midnite Mine Site, correlation data generated during the Mine Waste Investigations study (Miller Geotechnical, 2011) provide a statistical basis for an initial gamma cutoff value of 20 μR/hr. The data and technical rationale that support this initial gamma cutoff value are as follows:

- Gamma/soil Ra-226 correlation data were collected in the vicinity of impacted access roads as part of the Mine Waste Investigations study (Miller Geotechnical, 2011). Fourteen correlation plots were selected to cover a representative range of gamma exposure rate values (between about 20 and 160 µR/hr) and gamma measurements and soil samples were collected across each 100 m^2 plot. The resulting average gamma exposure rates and soil Ra-226 concentrations from these correlation plots were paired and analyzed by linear regression. The resulting statistical relationship (Figure 1) is highly significant (R^2 = 0.95).

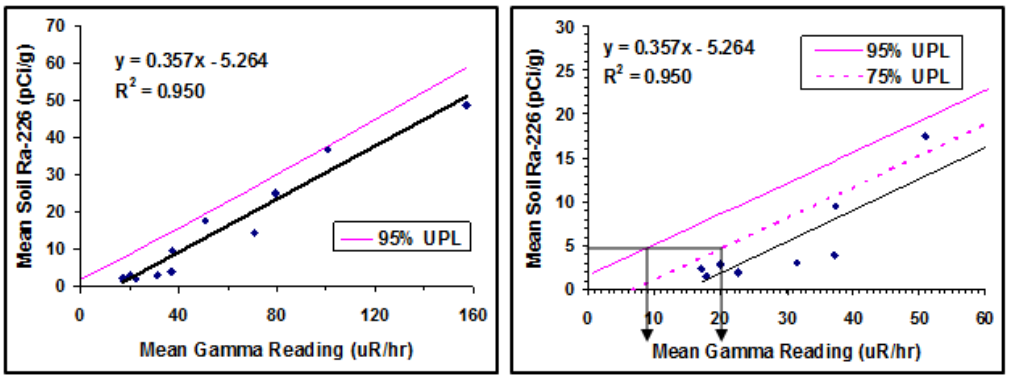


Figure 1 - Left: Site-specific statistical relationship between gamma exposure rate readings and soil Ra-226 concentrations, along with 95% upper prediction limits, based on correlation data provided in the Mine Waste Investigations report (Miller Geotechnical, 2011). Right: The same graphical data, zoomed in at the lower end of the scale to allow visualization of two probabilistic gamma cutoff levels (one for the 95% UPL and one for a 75% UPL).

- In general, the circumstances and locations under which these correlation data were generated, along with the high R^2 value on the regression and relatively low amount of data scatter about the regression line, are favorable in terms of applicability for remedial assessment.
- Regression statistics for these data indicate that when gamma exposure rates are 20 µR/hr, the statistical probability of compliance with the 4.7 pCi/g cleanup level for Ra-226 is about 75 percent. On average, soil Ra-226 concentrations will be about 1.8 pCi/g, which is close to the 1.1 pCi/g national average for natural background soils as reported by Myrick et al. (1983). The 95 percent upper prediction limit (UPL) for this regression indicates that the cleanup level for Ra-226 in surface materials corresponds to a gamma cutoff level of about 9 µR/hr (Figure 1), a value that would not be achievable as it falls below the documented range of background gamma radiation in vicinity of the Site (≈ 11-20 µR/hr; URS, 2005 and SMI, 1999). This statistical circumstance is believed to be an

artifact of an apparent slight non-linearity in the true relationship between ambient gamma radiation and soil Ra-226 concentrations at the lowest end of the correlation[1] (Figure 2). If real, this non-linearity is expected to result in an actual rate of compliance approaching 100 percent at a gamma cutoff value of 20 µR/hr. A gamma cutoff value of 20 µR/hr is consistent with the upper range of background basis for the cleanup level for Ra-226 (EPA, 2006c; URS, 2005). In summary, a 20 µR/hr gamma cutoff value is expected to err on the side of more remediation than is necessary to meet the Ra-226 cleanup level, and is also likely to be achievable.

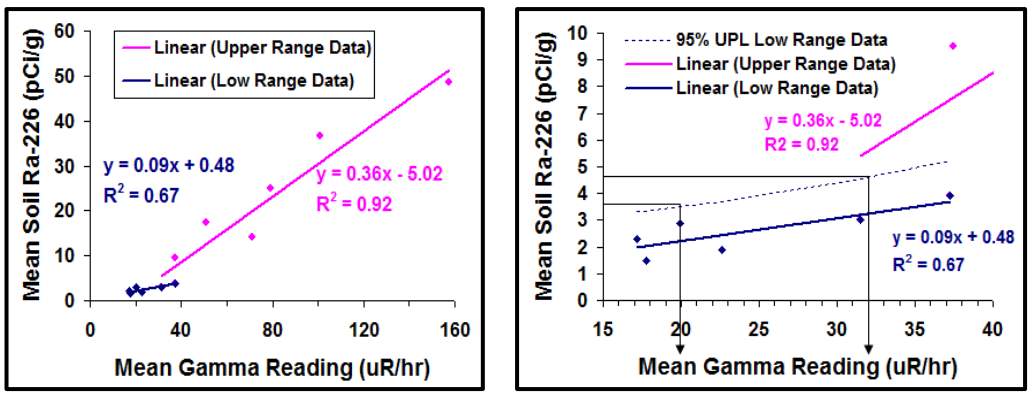


Figure 2 – Left: Separate correlations for lower and upper range correlation data from the Mine Waste Investigations study (Miller Geotechnical, 2011). Right: The same correlations, zoomed in at the lower end of the scale with a 95% UPL on low range data showing that a gamma cutoff value based on the low range data provides a 95% statistical probability of compliance at about 32 µR/hr, and that a gamma cutoff value of 20 µR/hr provides a 95% probability that Ra-226 concentrations will be less than about 3.7 pCi/g (well below the cleanup criterion for Ra-226).

The initial gamma cutoff value (20 µR/hr) does not necessarily represent a final value. This value may be revised as appropriate during the cleanup based on additional correlation gamma measurements, direct soil sampling, and Ra-226 analysis in an onsite soils lab (see Section 3.5). As higher activity materials are removed from remedial areas, variability in gamma readings and Ra-226 concentrations is expected to be reduced, resulting in reduced total variance about the regression line and reduced uncertainty in the gamma cutoff at the Ra-226 cleanup criterion based on the additional correlation data.

[1] Reasons for nonlinearities in correlation data near the low end of the scale (at very low background levels) are believed to be related to a threshold effect in the relationship between energy-dependent NaI gamma detector readings and the ratio of terrestrial to cosmic sources of gamma radiation. Cosmic sources of gamma radiation (which are generally uniform at a given site) along with scattered low-energy photons from both cosmic and terrestrial sources can dominate detector response until primary photons associated with Ra-226 and its decay products become concentrated enough to have significant correlative impact on readings (Whicker et al., 2008).

Obtaining significantly more correlation data, particularly in the range of values near the cleanup criterion, and revising the regression and associated UPL accordingly, is expected to increase the statistical probability of compliance associated with an achievable gamma cutoff value (e.g. 95 percent in the range of 20 µR/hr). Although the gamma cutoff value may be revised over time, any changes are not expected to be large (e.g. < 1 µR/hr), and the respective probability of compliance will always account for the amount of uncertainty present in the data available at any given time. In other words, revisions to the cutoff value that could occur later in the cleanup sequence will not affect Type I error rates on decisions that are made early in the cleanup based on concurrent cutoff values.

3.2 Gamma Scanning Sensitivity

With respect to the analytical sensitivity of this gamma screening approach, MARSSIM indicates that a theoretical minimum detectable Ra-226 concentration that can be detected in surface soils using a NaI detector with a 2" x 2" NaI crystal (like the Ludlum Model 44-10 detectors that will be used for this project) is 2.8 pCi/g (NRC, 2000). This value is based on a number of assumptions, including a contaminated soil area of 0.25 m^2 to a depth of 15 cm, and gamma scanning with the detector positioned at 10 cm above the ground surface. This scanning sensitivity concept is known as the Scan MDC (minimum detectable radionuclide concentration with a given scan system and technique).

Under the above theoretical conditions, scanning with a Ludlum 44-10 detector can easily detect soil Ra-226 concentrations equivalent to the cleanup criterion (4.7 pCi/g). However, for a site as large as the Midnite Mine, a 10-cm scanning height is not practical, and from a perspective of radiological doses or health risks, an area as small as 0.25 m^2 is not realistically significant, particularly when the soil concentrations being evaluated for compliance are close to the cleanup criterion for Ra-226 (which itself is based on the upper range of background levels). SENES has calculated Scan MDC values for Ra-226 in surface soils using 2x2" NaI detectors at 1 meter above the ground with Micro-Shield modeling and the methodology provided in NUREG 1507 and NUREG 1575 (MARSSIM). Results (Table 1) indicate that the proposed scanning method can easily detect elevated soil Ra-226 concentrations below the 4.7 pCi/g cleanup level for a hot spot of 3 meters diameter, but probably not for hot spot smaller than 2 meters diameter.

The authors of MARSSIM recognized that theoretical calculations of Scan MDCs are dependent on a number of factors (some qualitative and subjective), and empirical measurements in the field are a more definitive way to quantify Scan MDCs (NRC, 2000). Modern GPS-based

gamma scanning technologies largely eliminate uncertainties associated with real-time human surveyor error (e.g. making hundreds of real-time subjective decisions per hour regarding the "significance" of potential changes in count rate based on audible "clicks"). With modern gamma scanning technologies, the data are permanently recorded and can thus be retroactively studied in a quantitative/spatial context that is not possible with audible monitoring and instantaneous decision making in the field. In effect, this advance in gamma survey methods will increase scan sensitivity (i.e. effectively lowering scan MDCs).

Table 1 – Calculated Scan MDC values for Ra-226 in surface soils using various hypothetical scan methodologies for several small "hot spot" dimensions.

Hot Spot Diameter[1] (m)	2x2 NaI Detector Height (m)	Scan Speed (m/s)[2]	Calculated Scan MDC for Ra-226 (pCi/g)[3,4]	Calculation Source and Methodology
0.5	0.25	0.5	2.8	MARSSIM (NUREG 1507/1575 method)
1.5	1.0	1.0	6.2	SENES (NUREG 1507/1575 method)
3.0	1.0	1.0	2.1	SENES (NUREG 1507/1575 method)

[1]Hot spot depth = 15 cm

[2]1 m/s ≈ 2 mph (typical walkover scan speed)

[3]Based on 10,000 cpm background, performance factor d' = 1.38, and observation interval = 1 second

[4]Assumes U-238 and all decay progeny in equilibrium

Based on direct evidence from numerous past radiological survey projects conducted by SENES personnel, areas as small as about 4 m^2 having concentrations of Ra-226 in surface soils that are elevated above surrounding baseline levels by as little as 2-3 pCi/g can be reliably detected based on retrospective assessment of data recorded with Ludlum 44-10 detectors while scanning at three feet above the ground surface[2]. Such real-world data are consistent with the calculated scan MDC values provided in Table 1.[3] Moreover, setting the gamma cutoff value based on upper prediction limits for the correlation helps take into account radiologically elevated areas that are too small and/or too low-level to be readily detected with gamma scanning. Again, based on actual site-specific data using this gamma screening approach, the

[2] Several example gamma survey / soil sampling data sets from other sites that demonstrate scan sensitivities in this range were presented to the EPA in a Technical Meeting held at Wellpinit, WA on April 24, 2013.

[3] While this consistency appears to suggest that retrospective evaluation of recorded scan data would not lower scan MDCs, the values in Table 1 were calculated with a 60 percent tolerance for "false positives" due to human surveyor error. If that tolerance had been set at a level commensurate with that attainable with retrospective study of recorded scan data (e.g. 5 percent), calculated scan MDC values would have been significantly higher.

average soil Ra-226 concentration in areas remediated to a gamma cutoff level of 20 µR/hr is expected to be about 1.8 pCi/g, well below the 4.7 pCi/g cleanup criterion for Ra-226.

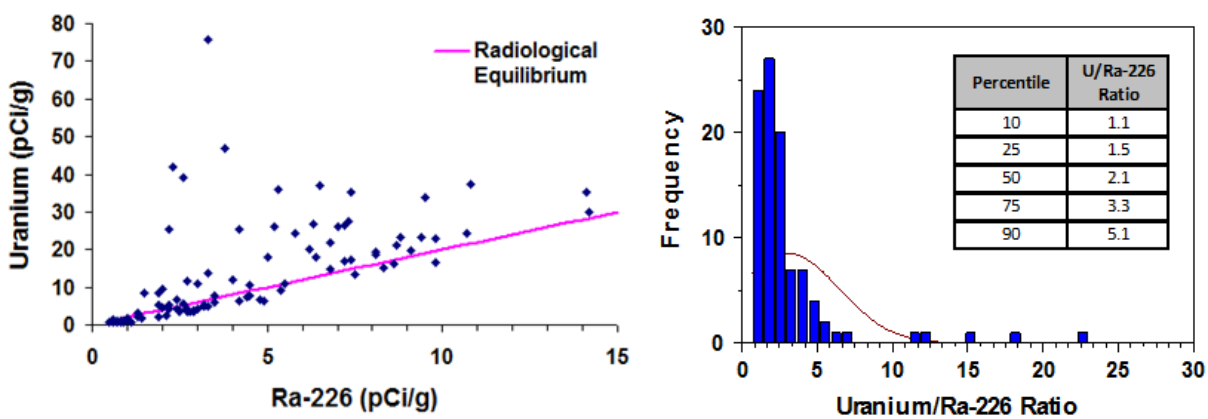
The same gamma screening approach was used during 2005 cleanup activities at the nearby Dawn Mill Site in Ford, Washington. In that case, site-specific correlation data resulted in a gamma cutoff value of 30 µR/hr based on a 95 percent probability of compliance with a soil Ra-226 cleanup criterion of 6 pCi/g. Based on this gamma cutoff value, the horizontal and vertical extent of excavations was guided with a Ludlum 44-10 detector at a scan height of 3 feet above the ground surface. The remedial outcome revealed that in approximately 95 percent of locations predicted to meet the soil Ra-226 criterion based on the gamma cutoff value, direct soil sampling confirmed that this criterion was achieved. Similar results have been demonstrated at other radiologically impacted sites. In summary, gamma screening based on statistical correlations and probabilistic gamma cutoff values is effective, accurate and reliable and the sensitivity of the gamma survey screening approach that will be used at the Midnite Mine is more than sufficient to correctly predict compliance with the cleanup criterion for Ra-226 concentrations in surface materials in the vast majority of locations screened with this approach.

3.3 COCs other than Radium-226

To address radionuclides other than Ra-226, site-specific knowledge of the quantitative/ statistical relationship between Ra-226 and each radionuclide of interest is required. A 2002 study by URS Corporation evaluated surface materials from the Midnite Mine with respect to the degree of radiological equilibrium present between uranium isotopes and their decay products (URS, 2002). The findings of this study indicate an approximate state of radiological equilibrium exists between uranium, Ra-226 and Pb-210 for surface materials at the Site, though the samples evaluated were collected at the ground surface, not at depth below ore, proto-ore, or mine rock deposits.

Data from the Mine Waste Investigation study (Miller Geotechnical, 2011) indicates that the original surface soils which now reside at depth underneath deposits of ore, proto-ore, or mine rock, have considerable radiological disequilibrium between uranium and Ra-226 (Figure 3). Because the degree of disequilibrium between Ra-226 and Pb-210 in these soils is relatively uniform (Figure 4), it can also be shown that considerable disequilibrium between uranium and Pb-210 exists. These soils will become important in terms of cleanup criteria once the bulk of the mine waste deposits are removed. A technical evaluation of whether or not gamma survey screening for Ra-226 can also be used to screen for uranium and Pb-210 is as follows:

- Based on observed relationships between uranium and Ra-226 in soils underneath the waste rock deposits (Figure 3), gamma survey screening to meet the cleanup level for Ra-226 is expected to result in compliance with the cleanup level for uranium in the vast majority of remediated locations. Cleanup levels for uranium and Ra-226 (29.1 and 4.7 pCi/g respectively) are not specified in the ROD at levels that represent radiological equilibrium. The uranium/Ra-226 ratio represented by respective soil cleanup criteria in the ROD is 6.19. Of the 98 soil samples collected underneath the waste rock deposits in the Mine Waste Investigation (Miller Geotechnical, 2011), 6 samples (about 6 percent) had uranium/Ra-226 ratios in excess of 6.19, but only 4 of these 6 samples had uranium values above 29.1 pCi/g while also having Ra-226 values below 4.7 pCi/g (Figure 3). Assuming that remediation of the Site for Ra-226 compliance (based on gamma screening) would fail to achieve compliance with the uranium criterion at these 4 locations, the overall performance of the cleanup would be very close to a 95 percent rate of compliance. This is functionally analogous to limiting the Type I error rate to α = 0.05, a statistical bound commonly set by regulatory agencies in terms of limiting decision errors on compliance with cleanup criteria.



Percentile	U/Ra-226 Ratio
10	1.1
25	1.5
50	2.1
75	3.3
90	5.1

Figure 3 – Left: Relationship between uranium and Ra-226 concentrations in 98 soil samples collected at depth underneath mine waste rock deposits (Miller Geotechnical, 2011). The pink line represents a theoretical line of equilibrium between uranium and Ra-226 concentrations across the range of observed values. Right: Frequency histogram and corresponding table of percentiles for observed uranium/Ra-226 ratios.

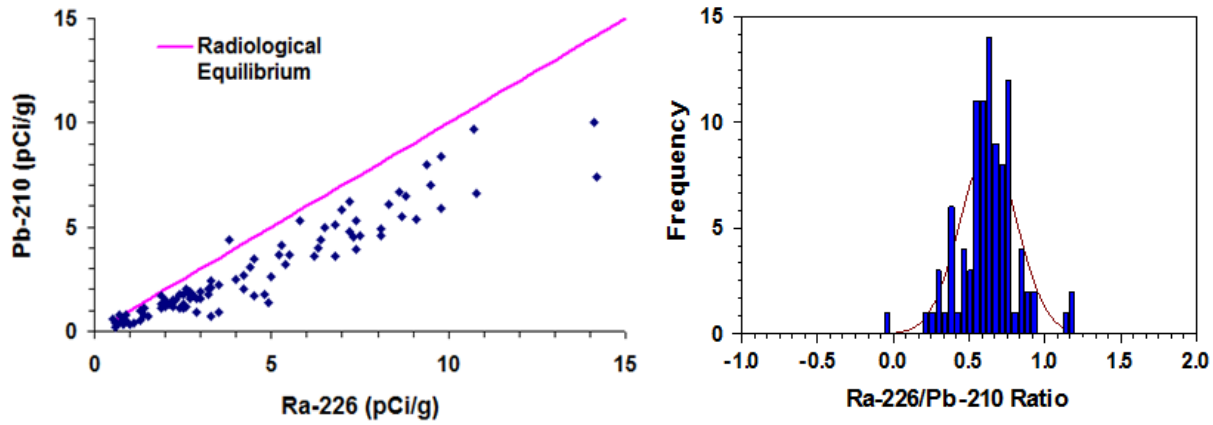


Figure 4 – Left: Relationship between Ra-226 and Pb-210 concentrations in 98 soil samples collected at depth underneath mine waste rock deposits (Miller Geotechnical, 2011). Right: Frequency histogram for observed Ra-226/Pb-210 ratios.

- Gamma survey screening to meet the cleanup criterion for Ra-226 will also simultaneously ensure that the cleanup criterion for Pb-210 is achieved. This is because Pb-210 concentrations are almost universally lower than Ra-226 concentrations (Figure 4), yet the soil cleanup criterion for Pb-210 (7.5 pCi/g) is greater than the cleanup criterion for Ra-226 (4.7 pCi/g). The Ra-226/Pb-210 ratio for the cleanup criteria specified in the ROD is 1.60, yet Ra-226/Pb-210 ratios for all soils sampled underneath the waste rock deposits are well below 1.60. These data, as well as data provided in a special Site study concerning radiological equilibrium (URS, 2002), indicate with nearly 100% certainty that if the Ra-226 cleanup level is achieved, the Pb-210 cleanup level will also be achieved (see Section 3.5 and Figure 9).

3.4 Gamma Shine Effects

In some cases, gamma survey screening for compliance with the gamma cutoff value can be negatively affected by a radiological phenomenon known as “gamma shine”. This occurs when scattered gamma photons from higher activity source materials in adjacent areas (e.g. from nearby stockpiles of mine rock) reach the detector and erroneously indicate higher Ra-226 concentrations than are actually present in surface soils directly below the detector. Gamma shine can result in more remediation than is necessary to meet cleanup criteria. One way to reduce gamma shine is to use lead or tungsten shielding around the detector, with a collimated window (opening) at the bottom to help limit photons that can reach the detector to those originating at the ground surface directly below the detector.

Use of shielding to guide excavations in a truly quantitative and probabilistic manner would require separate gamma/Ra-226 correlation studies to determine a shielded gamma cutoff

value. However, determination of an average shielded reading in various areas that meet the unshielded gamma cutoff can serve as a qualitative goal for excavations in areas where significant gamma shine is thought to exist. It should be noted that shielding cannot be expected to eliminate gamma shine, and use of any type of qualitative goal or formal cutoff value (shielded or unshielded) in areas of suspected gamma shine should be supplemented with direct soil sampling and Ra-226 analysis in the onsite soils lab.

3.5 Onsite Soils Laboratory

An alternate and more definitive method to assess soil Ra-226 concentrations in the field, for example in areas where gamma shine is suspected, is to collect individual soil samples and analyze them directly in an onsite soils lab (e.g. Whicker et al., 2006). Using a shielded gamma counting well, gamma emissions from individual soil samples (e.g. 200 grams) can be accurately measured and corresponding soil Ra-226 concentrations can be estimated directly using a multi-channel analyzer to quantify counts in specific energy regions of interest (gamma spectroscopy). The approach relies on system calibrations against soil Ra-226 reference material standards. With proper system set-up, calibration, and data analysis techniques, an onsite soils lab can provide accurate estimates of soil Ra-226 concentrations in soil samples in near real-time (e.g. within several hours after collection).

During the 2005 remediation of the nearby Dawn Mill Site, onsite soils lab estimates of Ra-226 concentrations (using NaI-based gamma spectroscopy) agreed well with corresponding estimates from a qualified commercial radiochemistry laboratory using EPA Method 901.1 [gamma spectroscopy with a high-purity germanium (HPGe) detector] (Figure 5). The degree of accuracy and precision attained with NaI measurements in the onsite soils lab for this remediation effort was nearly the same as that reported for HPGe measurements by the commercial laboratory (Whicker et al., 2006). Note from the NaI:HPGe ratios provided in Figure 5 that on average, day zero estimates from the onsite soils lab (with a full radon-222 ingrowth[4] correction factor applied) slightly overestimated Ra-226 concentrations relative to results from the commercial laboratory, a circumstance which was conservative (i.e. it tended to result in slightly lower concentrations in remediated areas).

[4] Gamma spectroscopy with either NaI or HPGe detectors relies on short-lived gamma-emitting decay products of Rn-222 (namely Bi-214 and/or Pb-214). For this reason, soil analysis by gamma spectroscopy requires that soil samples be sealed in special counting canisters for at least 21 days to allow Rn-222 and its decay products to build into a state of secular equilibrium with their long-lived Ra-226 parent. Site- and material-specific correction factors for such ingrowth can be empirically derived and applied to allow full-ingrowth estimates of Ra-226 on the same day samples are collected (Whicker et al., 2006).

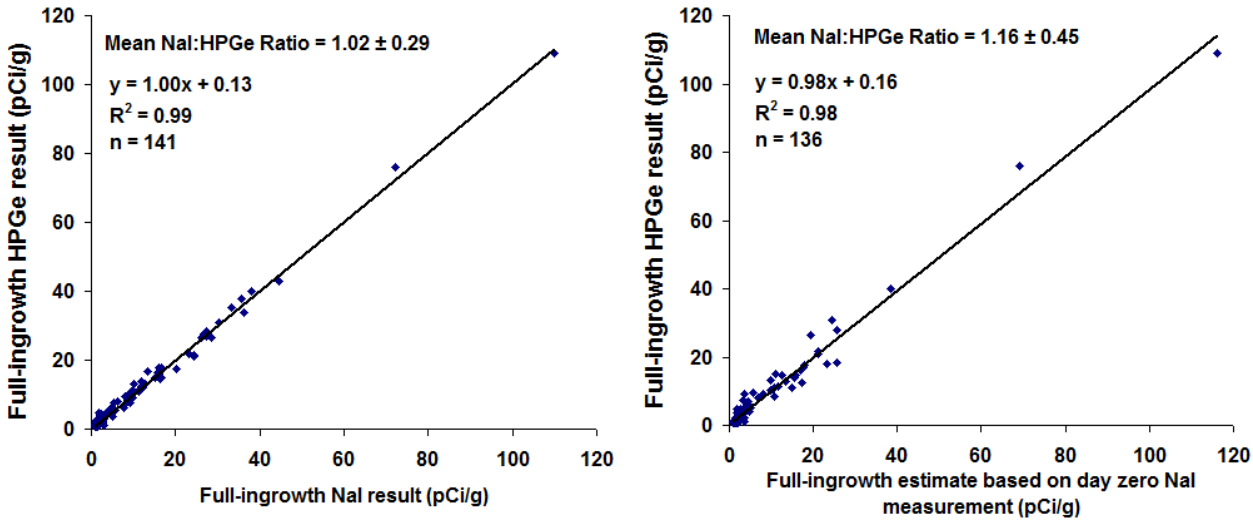


Figure 5 – Left: Relationship between full radon ingrowth estimates of Ra-226 based on NaI gamma spectroscopy measurements in an onsite soils lab, and corresponding results by HPGe analysis from a commercial laboratory during the 2005 Dawn Mill Site cleanup. Right: Relationship between day zero estimates of Ra-226 based on NaI gamma spectroscopy measurements in the onsite soils lab (with ingrowth correction factor applied), and corresponding full-ingrowth results by HPGe analysis from a commercial laboratory. Both graphs are adapted from Whicker et al. (2006).

The NaI-based estimates in Figure 5 were based on counts in three spectral regions of interest (ROIs) associated with two short-lived decay products of both Ra-226 and Rn-222 (Pb-214 and Bi-214) (Figure 6). In this case, raw calibration curves for each ROI were developed based on site-specific soil Ra-226 calibration standards from which full radon ingrowth results were obtained for both NaI counting in the onsite lab, as well as HPGe counting at the offsite lab. For each ROI, an estimate of Ra-226 concentration was obtained and the results were averaged to provide a final overall estimate.

The purpose of using multiple ROIs and averaging was to utilize all pertinent count data, help minimize the potential influence of spectral interferences (e.g. non-representative Compton scatter within one or more ROIs), and to produce the most accurate estimates possible of Ra-226. In response to EPA comments on this issue, and for the purposes of proposing a specific analytical approach for the Midnite Mine cleanup, an analysis of 87 paired offsite HPGe results and onsite soils lab estimates from the Dawn Mill Site cleanup was conducted for values across a range of greatest interest (< 11 pCi/g) to evaluate whether a specific individual ROI might produce more accurate results (Figure 7). The results indicate that on average, use of either the mean of the three ROIs, or use of a single calibration based on the sum of all counts measured in each of the three individual ROIs, can be expected to provide the most accurate results relative to HPGe measurements in an offsite commercial laboratory (Figure 7).

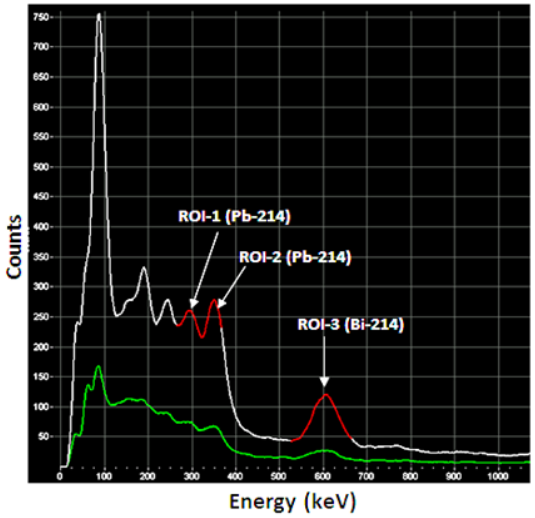


Figure 6 – ROIs for Pb-214 and Bi-214.

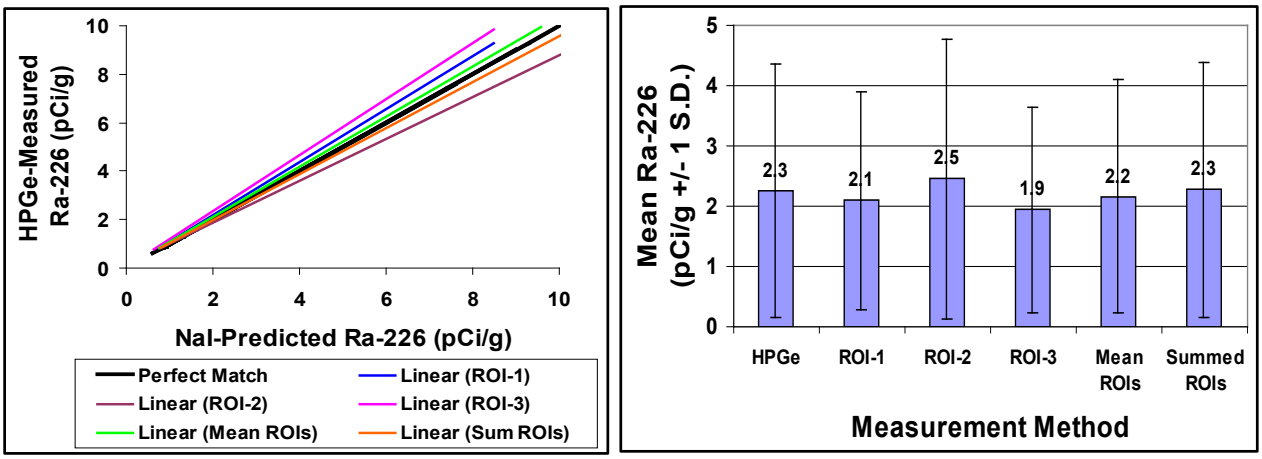


Figure 7 – <u>Left</u>: Linear regression curves for 87 paired HPGe/NaI results for Ra-226, where the NaI results were based on each of the 3 individual ROIs, the mean of the 3 ROIs, and on a single calibration using the sum of all counts in the 3 ROIs. On average, using the sum all counts in the 3 ROIs provides the most accurate results relative to HPGe measurements across the range of values examined. <u>Right</u>: Comparison of the mean result (± 1 standard deviation) for each of the NaI-based estimation methods versus the mean result for HPGe analysis at an offsite commercial laboratory. The "Summed ROIs" method produces distributional characteristics (mean, median and range) that most closely match HPGe results.

Because the calibration curves for the Dawn Millsite cleanup were based on site-specific soil Ra-226 calibration standards as measured by the offsite lab after full radon ingrowth, when unknown field samples were counted on day zero (the day field samples were collected, before any radon ingrowth) the resulting raw Ra-226 estimate would underpredict the true Ra-226 concentration. It was thus necessary to apply an empirically derived correction to account for

full radon ingrowth (Whicker et al., 2006). That correction was based on a linear regression between day zero counts and counts after full radon ingrowth.

For the Midnite Mine cleanup, a more direct system calibration will be performed to potentially reduce total propagated estimation uncertainty, simplify analytical and data management requirements, and to respectively maximize the number of samples that can be analyzed per day throughout the cleanup. This will involve a calibration between the sum of all day zero counts (e.g. number of counts in 20 minutes) within each of the three individual ROIs encompassing the Pb-214 and Bi-214 energy peaks, against full ingrowth Ra-226 activity results (pCi) from the commercial laboratory (Figure 8).

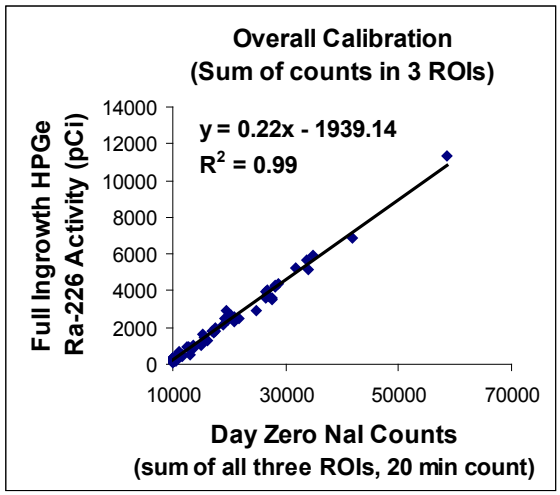


Figure 8 – Example calibration curve based on the sum of day zero NaI counts for three separate ROIs, versus full-ingrowth activity results by HPGe analysis at a commercial laboratory.

Use of a single calibration curve based on the sum of all counts within each of the three individual ROIs (Figure 8) appears to be the most accurate analytical approach overall (Figure 7), takes advantage of all relevant count data (maximizes counting statistics), and helps minimize the influence of any potential spectral interferences that may be present in the sample. Theoretically, this approach will also result in lower detection limits. More generally, the method takes advantage of the greater counting efficiency afforded by NaI-based systems (i.e. much shorter count times), yet also the greater spectral resolution offered by HPGe-based systems (i.e. better accuracy, particularly if spectral interferences are present).

This onsite gamma spectroscopy calibration approach will be site-specific for use at the Midnite Mine and will require collection, preparation and analysis of a set of site-specific soil Ra-226 calibration standards early in the cleanup. Procedures for developing the initial system

calibration, which include the possibility of using two regression curves (one for low range values and one for high range values) in an overall calibration algorithm, are provided in Section S.2.2.3 of Appendix S. Once the initial system calibration algorithm has been established, field samples can begin being analyzed for Ra-226. From this point forward, a fraction of all field samples (e.g. 10 percent) will be forwarded to the commercial lab for offsite analysis. The data resulting from samples analyzed both onsite and offsite throughout the multi-year cleanup will be used both for verification of onsite analytical performance (e.g. with T-tests, Wilcoxon Rank Sum tests, and ANOVA tests) and to refine the calibration algorithm as appropriate.

In addition to direct analysis of soil Ra-226 concentrations in the onsite soils lab, concentrations of Pb-210 can be indirectly estimated based on a predictive regression equation that models the average relationship observed between Ra-226 and Pb-210 for surface materials in non-background areas (Figure 9). Note in Figure 9 the statistical consistency in slope and intercept between the linear regression equations calculated for Ra-226 and Pb-210 data from both the Mine Waste Investigation study (Miller Geotechnical, 2011) and from an earlier study associated with Remedial Investigation/Feasibility Study (RI/FS) activities (URS, 2002). The data from both of these studies provides strong evidence that cleaning up surface materials to the Ra-226 criterion will ensure with nearly 100% certainty that the Pb-210 criterion is also achieved.

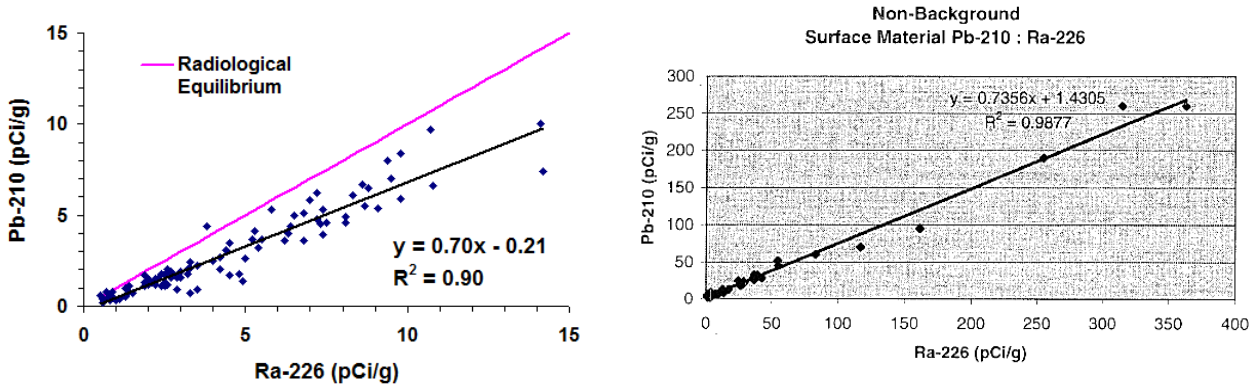


Figure 9 – Left: Relationship between Ra-226 and Pb-210 concentrations in 98 soil samples collected at depth underneath mine waste rock deposits as part of the Mine Waste Investigation study (Miller Geotechnical, 2011). Right: Relationship between Ra-226 and Pb-210 concentrations in 64 soil samples collected in non-background areas as part of RI/FS activities (URS, 2002).

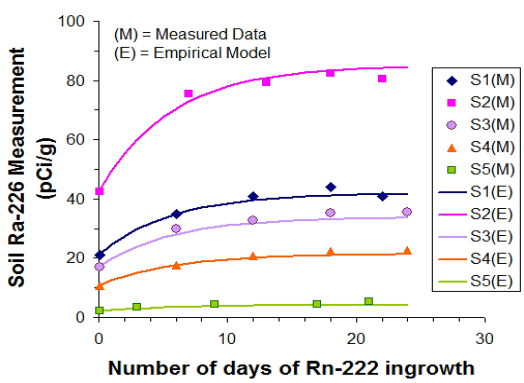
With respect to uranium, estimates based on gamma spectroscopy measurements of Ra-226 and the relationship between Ra-226 and uranium are not possible due to the high amount of variability in the relationship between uranium and Ra-226 concentrations (Figure 3). However, onsite X-ray fluorescence (XRF) measurements (see Section 4.0 and Appendix S, Sections

S.2.3.1 and S.3.2.1) can be used to estimate uranium concentrations and provide evidence of compliance with the uranium cleanup level.

3.6 Rapid Turnaround Analysis at an Offsite Laboratory

Onsite analysis of Ra-226 concentrations in soil samples (Section 3.5) will play an important role in verifying the reliability of the gamma cutoff value and the accuracy of the gamma/Ra-226 correlation with respect to guiding excavation of surface materials. However, there may be circumstances in which rapid turnaround analysis at an offsite commercial laboratory are needed (e.g. if there are onsite lab instrumentation problems and gamma shine is suspected in an area under evaluation). In this or similar circumstances, the offsite lab will be asked to count samples the same day they are processed and sealed at the laboratory, and to provide corresponding estimates of Ra-226 without any radon ingrowth. Upon receipt of these rapid turnaround results, an ingrowth correction will be applied. The equation that will be used for this ingrowth correction is described below.

During the 2005 soil remediation work at the nearby Dawn Millsite, representative samples from various locations were dried, homogenized and analyzed for Ra-226 concentrations in the onsite soils lab at various times between 0 and 24 days after sealing the samples (Figure 10). An empirical model of Ra-226 concentration measurements over time based on the ingrowth of Rn-222 (Equation 1) was fitted to the data. This model is plotted in Figure 10 for comparison against the measurements taken in the onsite soils lab. For these samples, there is good agreement between measured data and empirical modeling based on the buildup of Rn-222 and its decay products.



Equation 1: $$C_t = C_0 + C_0\left(1 - e^{-\lambda t}\right)$$

Where:

C_t = Ra-226 Conc. measure at time = t (days)

C_0 = Initial Ra-226 Conc. measure at t = 0 days[5]

λ = decay constant for Rn-222 (0.18 d^{-1})

t = time (days)

Figure 10 – Soil Ra-226 measurement data versus elapsed Rn-222 ingrowth time for sealed samples.

[5] This measure represents the amount of radon decay products present in the sample at t = 0, which will not be in approximate equilibrium with the actual amount of Ra-226 in the sample until about 21 days after sealing the sample.

Note in Figure 10 that the numerical increase in onsite soils lab measurements of Ra-226 over time is proportional to the initial measured Ra-226 concentration in the soil sample. Regardless of the starting concentration measurement (C_0), Equation 1 can be used to predict a full-ingrowth onsite soils lab measurement of soil Ra-226 after 21 days. Twenty one days is the widely accepted ingrowth period necessary for Rn-222 and its short-lived decay products to attain approximate secular equilibrium with their long-lived Ra-226 precursor in sealed soil samples.

As previously indicated, two short-lived radon decay products (Bi-214 and Pb-214) were used for the estimation of soil Ra-226 by gamma spectroscopy. Equation 1 is based on an assumption that these decay products, which have half-lives on the order of 20-25 minutes, are in equilibrium with Rn-222 (half-life = 3.8 days) at any given time (t), and that gamma spectroscopy measurements will thus reflect the concentration of Rn-222 in the sample. However, after a sample is dried and homogenized, Rn-222 will only be in partial equilibrium with its long-lived Ra-226 parent for the following reasons:

1) Given the long half-life of Ra-226 (approximately 1,600 years), the average rate of production of radon gas that remains encapsulated within individual soil grains and that cannot escape to the atmosphere is essentially constant. Gamma emissions from the decay products of this “trapped” radon gas provide a consistent but only partial base measure of the Ra-226 content in the soil sample (this is what is represented by the term C_0 as measured at t = 0 days).
2) The remainder of the Ra-226 content in the soil sample will be located at or close to the surface of individual soil grains and as these atoms gradually decay into Rn-222, the alpha recoil energy and trajectory of many of the new Rn-222 atoms is sufficient to transport them to near-surface pore spaces or directly beyond the surface of the grain where they will escape to the atmosphere (Sokoda et al., 2010). This process is known as radon emanation.

Once the sample is sealed, emanation losses to the atmosphere are prevented and the liberated radon will build into approximate equilibrium with the remaining Ra-226 present in the bulk sample (this is what is modeled by the buildup term on the right side of Equation 1). Equation 1 is similar to a model developed for the same basic measurement technique in Sokoda et al. (2010). Both models follow the general form of the well-known theoretical equation describing the ingrowth of a short-lived radioactive decay product from a long-lived parent radionuclide (e.g. Turner, 1995). Differences are that the variable “C_0” in the first term

on the right side of Equation 1 is not accompanied by a function to model losses of radon initially present due to decay, and a posteriori estimate of the final equilibrium concentration (representing the parent Ra-226 activity) in the second term has been replaced with "C_0" as for this application, the final concentration is not known in advance for day zero counting. Despite these differences, the empirical "fit" to the Dawn Mill Site data in Figure 10 for either modeling approach is nearly identical.

Although Equation 1 may work well for most sites/soils, it is likely to be somewhat site-specific (radon emanation rates for other sites/soils may differ). Soils are by nature more weathered than rock, have a greater specific surface area, and depending on their mineralogy, tend to have some degree of Ra-226 enrichment near the surfaces of soil grains. As a result, soils tend to have significantly greater radon emanation rates versus rock (Sokoda et al., 2010). For these reasons, the use of Equation 1 for ingrowth corrections will require verification via a second counting at the commercial laboratory after full radon ingrowth. This verification process is not expected to significantly alter cleanup decisions that are made based on rapid TAT results from the commercial lab as any differences are expected to be relatively minor, particularly at concentrations near the cleanup level.

4.0 ANALYTICAL APPROACH FOR SEDIMENTS

Regarding cleanup criteria for mine drainage sediments, which include non-radiological COCs, gamma screening for Ra-226 compliance cannot be relied upon to guide remedial efforts with respect to COCs other than Ra-226[6]. For metals, X-Ray fluorescence (XRF) technology can be utilized to provide near real-time estimates of respective concentrations in surface soils and sediments. XRF technology and methods have become widely used for direct field estimation of metal concentrations in soil, and have been demonstrated to produce reasonably accurate results provided established methodologies are properly employed. The standard EPA method for field analysis of the concentrations of metals in soil using XRF technology is Method 6200. This method relies on site-specific calibrations of two basic field measurement techniques (in-situ or intrusive) against confirmatory analyses by an analytical laboratory.[7]

[6] Based on this and previous studies (URS, 2002 and 2005), the relationship between Ra-226 and uranium and Pb-210 concentrations in sediments is generally not in equilibrium, and the disequilibrium is not quantitatively/directionally consistent enough for Ra-226 to be considered a highly limiting remedial parameter.

[7] Though the specific methodologies and procedures differ, the basic concept of calibrating field measurements against results from a qualified commercial laboratory is common to all of the field analysis methods that will be used at the Midnite Mine, including onsite XRF measurements for metals,

Method 6200 will be used for to support remediation of sediments with respect to non-radiological COCs (metals). XRF technology is based on exciting atoms in soil samples with x-rays and measuring resulting light spectrum emissions. The in-situ methodology involves direct measurement of soils or sediments residing in-situ at the ground surface. The intrusive methodology involves collection of samples, followed by sample processing (e.g. homogenization, drying and sieving or crushing), packing samples in special measurement containers covered with a thin Mylar film, and performing measurements in a controlled environment (e.g. an onsite laboratory) with a consistent measurement geometry and technique. The intrusive methodology is more precise and reduces measurement error, most of which is attributable to heterogeneous physical characteristics of the material being measured (e.g. high variability in particle sizes, moisture content and metal concentrations).

The Mine Waste Investigations report concluded that manganese will be the limiting COC for guiding remedial excavation of sediments in mine drainages (Miller Geotechnical, 2011). EPA Method 6200 lists manganese as one of the analytes that can be determined by this method. As a result, in-situ measurements of manganese can be used to screen the surfaces exposed by excavation of sediments and evaluate whether additional excavation is required to meet the cleanup level for manganese. If compliance for manganese is achieved, compliance with cleanup levels for other COCs in sediments can generally be expected. Confirmation of this expectation can be verified by use of the intrusive methodology, where representative samples from areas deemed likely to be in compliance (based on in-situ XRF screening) are collected, processed and analyzed in the onsite laboratory with carefully controlled sample preparation and XRF measurement of each metal indicated as a COC for sediments in the ROD.

Although uranium is not listed in Method 6200, XRF can be used to estimate uranium concentrations provided that the XRF instrument is programed to include uranium in its light emissions inventory for metals. Uranium analysis with XRF measurements can provide additional assurance of compliance with cleanup levels for surface materials as well as sediments. With respect to cleanup criteria for isotopic uranium (U-238 and U-234) in sediments, these radionuclides have been shown to be in approximate equilibrium in both surface materials and sediments at the Site (URS, 2002) and thus, respective concentrations could be calculated based on natural (total) uranium results using their normal relative contributions to the total amount of radioactivity in natural uranium (approximately 49 percent

Ra-226 analysis by gamma spectroscopy, and gamma scanning based on a cutoff value developed through gamma/Ra-226 correlations.

each). For impacted sediments, there is evidence of slight radiological disequilibrium between U-234 and U-238 (URS, 2002), but this disequilibrium is quite consistent and cleaning up to meet the uranium cleanup level for sediments will ensure compliance with U-234 and U-238 cleanup levels (see Section S.4.3.4.1 of Appendix S). The plans and details for XRF analysis methods to support remedial excavations and final status evaluations of compliance are provided in Appendix S.

Gamma survey screening for Ra-226 in sediments, though unlikely to be sufficient to determine the final extent of sediment excavations, can be used to provide supplementary indications of remedial progress on a real-time basis. The methodology would be the same as for surface materials (Section 3.1), but the gamma cutoff value will be different as the Ra-226 cleanup level for sediments is different (13 pCi/g). Correlation data from the Mine Waste Investigation study (Miller Geotechnical, 2011) indicate an initial gamma cutoff value for sediments of 33 µR/hr (Figure 11). This value is corroborated by radiological survey data subsequently collected along the White Tail Creek Drainage (WME, 2014). Although the data collected along this drainage were generated with somewhat different methodologies (discrete gamma measurements and sediment samples to a depth of 2 inches), the indicated gamma cutoff value with a 95 percent probability of compliance is identical to that generated from the Mine Waste Investigation data (Figure 11), despite apparent differences in geology in this area. Sediment samples can also be analyzed in the onsite soils lab to estimate Ra-226 concentrations on a near real-time basis (Section 3.5).

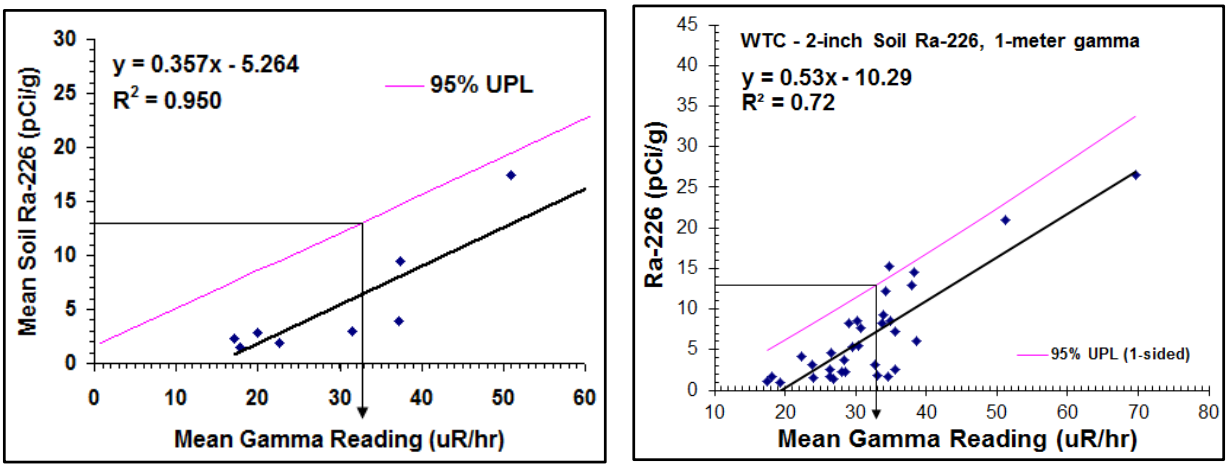


Figure 11 – Determination of an initial gamma cutoff value providing a 95% probability of compliance with the 13 pCi/g cleanup level for Ra-226 in sediments (33 µR/hr) based on data from the Mine Waste Investigation study (left) and the White Tail Creek study (right).

5.0 CONSIDERATIONS REGARDING NATURALLY MINERALIZED BACKGROUND

The reference areas used to define "background" conditions at the ground surface across the mined area prior to mining included four nearby non-impacted areas believed to have similar geologic, geochemical and hydrologic conditions relative to the mined area prior to mining (URS, 2005). The cleanup criteria defined for surface materials are based on an upper range (upper 95 percent tolerance limits) of the levels measured for respective COCs for soils in these background reference areas (EPA, 2006c). This is because for most COCs, cleanup criteria based on CERCLA standards for human health or ecological risks are below existing background levels as measured in the reference areas.

Sampling of surface materials in background reference areas to define background concentrations for COCs is appropriate when the reference areas are adequately representative of pre-mining conditions and impacts are related to deposition of materials on an otherwise undisturbed ground surface. In the case of the Midnite Mine, the original ground surface across a large portion of the Site was removed by open pit mining to reach ore bodies ranging from 16 to 300 feet below the ground surface (URS, 2005). These ore bodies occurred at the contact between granitic rock intrusions into older "meta-sedimentary" rocks of the Tongo Formation (URS, 2005). The meta-sedimentary rocks were the primary host for the mined ore bodies.

The geologic surfaces left exposed at the final extent of mining, and potentially, for adjacent bedrock formations below the soil surface in areas that were never mined but were used to stockpile ore, proto-ore and mine waste rock, are expected to contain varying amounts of natural uranium mineralization that in some locations could have uranium, Ra-226 and Pb-210 concentrations greater than the cleanup criteria established for these radionuclides in surface materials. This expectation is supported by comparing the visual nature of exposed (but otherwise undisturbed) geologic strata along the pit walls surrounding Pit 4 (Appendix S, Figure S-1) with elevated uranium concentrations in corresponding locations based on aerial gamma survey data (Appendix S, Figure S-3). The pit walls are identified in the ROD as having exposed uranium-bearing rock faces (EPA, 2006c) and those geologic strata are likely to extend underground beyond the physical extent of the excavated pits.

The spatial distribution of pre-existing, mineralized geologic materials and associated soils that are situated adjacent to the excavated pits but were never physically disturbed by mining is unknown. This circumstance could lead to a situation in which removal of mine wastes and

mine-impacted soils results in increasing concentrations as excavations approach undisturbed mineralized bedrock. Because it would be difficult or impossible to distinguish between mine-impacted soils and soils that were naturally mineralized prior to mining, excavations will necessarily continue until cleanup levels are achieved or bedrock is encountered.

The EPA's policy with respect to background at CERCLA sites is that cleanup levels are not set at concentrations below natural background levels, and the CERCLA program does not remediate to concentrations below natural or anthropogenic background levels (EPA, 2002; EPA, 2006c). Some remediation of pre-existing background soils that were not impacted by mining but naturally exceed ROD cleanup levels is likely to be unavoidable under these circumstances. A formal definition of when bedrock has been encountered and remedial excavation efforts will be terminated is provided in Attachment S-3.

6.0 FINAL STATUS SURVEY CONSIDERATIONS

6.1 Statistical Approach

Because radiological parameters are a major aspect of Site impacts and cleanup criteria at the Midnite Mine, it is reasonable to consider a MARSSIM-based approach to final status surveys. As previously indicated, this guidance was developed jointly by the EPA, NRC and DOE in order to attain respective consensus on a unified approach for the design of final status surveys at radiologically impacted sites. However, MARSSIM is primarily based on demonstrating compliance with dose-based or risk-based remedial criteria. When the COCs occur naturally in background soils (as in this case), remedial criteria under MARSSIM are defined based on excess levels of dose/risk above that which is attributable to natural background levels. In the case of the Midnite Mine, cleanup criteria for surface materials are not based on excess (above background) risk. This is because the risks due to background levels as determined for representative references areas (URS, 2005) already exceed acceptable levels as defined for CERCLA sites (lifetime cancer risks in the range of 10^{-4} to 10^{-6}). EPA guidance/policy "…does not recommend that cleanup levels be established at levels below background, even if the background level exceeds an ARAR or risk-based concentration" (EPA, 2006c). Given these circumstances, a conventional MARSSIM statistical testing approach [evaluation of an above-background cleanup level using Wilcoxon Rank Sum (WRS) testing] is not applicable.

An alternative approach for designing final status surveys would be to demonstrate that the levels of COCs after remediation are "indistinguishable from background." This approach is described in both MARSSIM and NUREG-1505 (NRC, 2000; NRC, 1998) and involves

statistical tests for differences in mean or median values between the survey unit and the background reference area. However, the cleanup criteria are not based on mean or median values for background, but on 95 percent UTLs. A non-parametric WRS tests could be used for this scenario if the distributional characteristics of concentrations in the survey unit and background reference area were similar (e.g. lognormal with similar variances), but it would require collection of new background reference area samples as only a small number background reference area samples were used to determine ROD cleanup levels (e.g. about 15 samples), they were not surface samples, and the sampling techniques and/or analytical methods previously used may not be comparable. In effect, "background" as indicated in the ROD would have to be redefined.

Another approach would be to follow MARSSIM statistical analysis methods, but utilize an unconventional interpretation of the cleanup criteria with respect to background. For example, if uranium, Ra-226 and Pb-210 were treated as though they are not present in background surface materials, then comparisons against background levels are no longer relevant or necessary under MARSSIM protocols. Under this scenario, a one-sample non-parametric sign test could be used to directly compare final status survey data against the numeric cleanup level for each COC. One-sample sign tests in MARSSIM would treat each cleanup criterion as a Derived Concentration Guideline Level (DCGL) based on dose/risk due only to residual contamination from Site operations because the COCs are assumed not to exist in background. Application of the unity rule[8] (NRC, 2000) would ensure that the total dose/risk from the final concentrations of all three COCs present in surface materials across the Site (after remediation) will not exceed the total dose/risk that is currently present in the background reference areas. The problem with this approach is that the sign test would compare the median value for final status survey data against the DCGL, which in effect, would demonstrate that median doses/risks from the survey unit will not exceed doses/risks from the 95 percent UTL for background levels in the reference areas. In other words, the percentage of the survey unit that could exceed ROD cleanup levels could approach 50 percent and still pass the statistical test.

The cleanup criteria for the Midnite Mine are essentially background threshold values against which the acceptability of the remedial outcome across the Site must be statistically demonstrated. An appropriate statistical approach for this circumstance is a one-sample proportion hypothesis test for compliance against a specified threshold value as described in the

[8] For multiple radionuclides, MARSSIM applies a "unity rule" where the sum of fractions of the median radionuclide concentration relative to its specified cleanup level for each radionuclide must be less than or equal to unity.

technical manual for ProUCL v4.0, a statistical software package developed by the EPA (EPA, 2007). The same one-sample proportion test is recommended for this circumstance in the statistical/sampling design package called Visual Sample Plan[9] (VSP, 2012). In this test, the proportion (equivalently expressed as a percentage) of the true population of COC values across the survey unit needed to meet the cleanup criteria and be considered an acceptable remedial outcome is specified (e.g. 95 percent), and the final status sampling data are statistically evaluated to determine if there is an acceptable probability of having achieved this outcome based on the final status sampling data.

A one-sample proportion hypothesis testing approach will be used for final status surveys at the Site. The performance/acceptance criteria will include a 95 percent rate of compliance with the cleanup levels, with the Type I error rate for the statistical testing limited to 5 percent ($\alpha = 0.05$). In accordance with MARSSIM principles, high-density gamma scanning across each survey unit will provide indirect evidence of compliance with the cleanup criteria at locations situated in between direct sampling locations, and will be relied upon to identify any residual “hot spots” not detected by direct sampling for potential further remedial action. In cases where remedial excavations may have uncovered naturally mineralized bedrock, these locations will not be evaluated in terms of compliance with ROD cleanup levels, but will be scanned as part of final status surveys to document final status gamma readings.

With respect to non-radiological COCs in sediments, the same one-sample proportion testing approach will be used. Evaluation of potential “hot spots” and compliance between sediment sampling locations will require transect-based in-situ XRF surveys (see Appendix S, Section S.4.3.1), though gamma scanning can be used to evaluate Ra-226 concentrations (Section 4.0).

Details of how this overall statistical approach for demonstrating compliance will be implemented, along with related evaluation and decision making protocols for both surface material and sediments, are provided in Sections S.4.2 and S.4.3 of Appendix S.

6.2 Considerations Regarding Survey Unit Size and Number of Samples

The number of samples in each survey unit that are needed to satisfy the specified statistical testing criteria (95 percent statistical confidence that 95 percent of true population values are in compliance with the cleanup criteria) will be determined using ProUCL or VSP software.

[9] Developed at Pacific Northwest National Laboratory with support from DOE, EPA, DOD, the Department of Homeland Security (DHS), the Centers for Disease Control (CDC), and the United Kingdom, VSP is a software tool that facilitates development of a defensible sampling plan based on statistical sampling theory and the statistical analysis of sample results to support confident decision making.

Example calculations in VSP indicate that the minimum number of samples needed in each survey unit would be 52 samples. This calculation assumes a null hypothesis that the status of the survey unit after cleanup is unacceptable[10] (> 5 percent of the survey unit exceeds the cleanup criteria), employs conventional values for Type I and Type II error rates ($\alpha = 0.05$ and $\beta = 0.10$ respectively), and assumes that the true percentage of values above the cleanup level in the survey unit after the cleanup [i.e. the lower bound on the gray region (LBGR) relative to a specified exceedance proportion limit of 0.05] is zero, meaning that the width of the gray region is 5 percent.

Under this null hypothesis, the gray region represents a range of possible percentage values across the survey unit where the consequences of deciding that a clean survey unit is dirty (i.e. requiring unnecessary remediation) are considered relatively minor (VSP, 2012). The width of the gray region (as determined by the LBGR) is related to the Type II error rate (β) and the amount of risk the Site operator is willing to accept that a clean survey unit will fail the statistical testing due to an insufficient number of samples (EPA, 2000b; NRC, 2000; ORAU, 2006). The value specified for α controls the probability of Type I errors (in this case potential health risk consequences) and requires a certain minimum number of samples to ensure the validity of the statistical test with respect to α (Khamis, 1988). In the example calculation above, 52 samples are needed to limit the Type I error rate to $\alpha = 0.05$, but the specified Type II error rate (β) is not actually controlled because the LBGR was set at zero (β is always set at the LBGR). To also control the probability of Type II errors (in this case unnecessary remediation), additional samples would be required depending on the values selected for β and the LBGR (Khamis, 1988). If the LBGR in the above example is assumed to be considerably larger than zero, the number of samples required increases significantly. Assuming that the true percentage of values in excess of the cleanup criteria after remediation (i.e. the LBGL) is 2.5 percent (equivalent to a default assumption of 50% of the action level as indicated in MARSSIM), the minimum number of samples required increases to 500 samples per survey unit.

The consequences of Type I errors (e.g. potential health risks slightly greater than that attributable to natural background conditions) can be controlled at $\alpha = 0.05$ with a reasonable number of samples, but the number of additional samples needed to also control the consequences of Type II errors (unnecessary remediation) must be weighed against what is

[10] For most final status survey applications, MASSIM recommends a null hypothesis that the survey unit does not meet the cleanup criteria. This ensures that the Type I error rate (α) is unaffected should there be an insufficient number of samples to also limit the probability of Type II errors to the value specified for β (ORAU, 2006; Khamis, 1988).

reasonably achievable in terms of final status survey data. EPA guidance on DQOs for hazardous waste sites (EPA, 2000b) suggests that it may be appropriate to set the width of the gray region to be relatively wide for cleanup evaluations because this "…will usually yield conclusive evidence of a successful remediation."

It is important to recognize that verification of compliance with cleanup criteria is not limited to direct soil/sediment sampling data. Gamma surveys will provide close to 100 percent spatial ground coverage across each survey unit, essentially defining the entire population of gamma exposure rates and indirectly, allowing statistically based estimates of the entire population of Ra-226 concentrations in surface materials. Compared to direct sampling and analysis, there is greater uncertainty in such estimates at a given location, but overall these estimates will provide a far more spatially comprehensive understanding of concentrations relative to the cleanup level across the entire survey unit.

Moreover, comparison of gamma readings against the gamma cutoff value (based on the UPL for the gamma/Ra-226 regression), is expected to account for this uncertainty and result in at least a 95 percent probability of compliance. Again, demonstration of compliance with the Ra-226 criterion is expected to ensure (with a statistical probability of about 95%) that other radiological COCs are also in compliance. Gamma surveys will be a crucial aspect of demonstrating compliance, and use of gamma surveys for guiding excavations (Remedial Support surveys) will help to minimize unnecessary remediation. In effect, gamma surveys and the gamma/Ra-226 correlation can be expected to provide the most effective means possible for controlling both Type I and Type II decision errors regarding compliance with cleanup levels for surface materials at this Site.

7.0 REFERENCES

Johnson, J.A., H.R. Meyer, and M. Vidyasagar, 2006. Characterization of Surface Soils at a Former Uranium Mill. Operational Radiation Safety. Supplement to Health Physics, Vol. 90, February.

Khamis, H.J., 1988. Statistics Refresher II. Choice of Sample Size. Journal of Diagnostic Medical Sonography. 4:176-183, July/August.

Lively, J. W., 2013. The Art & Power of Data Imaging. Proceedings of the ASME 2013 15th International Conference on Environmental Remediation and Radioactive Waste Management. September 8-12, 2013, Brussels, Belgium. ICEM2013-96256.

Meyer, R., M. Shields, S. Green, and J. Johnson, 2005. A GPS-based system for radium/uranium contamination gamma scanning. Uranium Mining and Hydrogeology IV. Broder J. Merkel, Andrea Hasche-Berger (Editors). Uranium in the Environment, conference proceedings, Freiberg, September.

Miller Geotechnical Consultants, Inc. (Miller Geotechnical), 2011. Mine Waste Investigations. February 11.

Myrick, T.E., B.A. Berven, and F.F. Haywood, 1983. Determination of Concentrations of Selected Radionuclides in Surface Soil in the U.S. Health Physics, Vol. 45, No. 3. September 1.

Oak Ridge Associated Universities (ORAU), 2006. Multiagency Radiation Survey and Site Investigation Manual: Implementing the MARSSIM Approach for Design and Conduct of Radiological Surveys. Notebook 2. Professional Training Programs, Radiological Safety, Assessments, and Training, ORAU. February 6-10, 2006. Teaching Staff: Eric Abelquist (CHP), Paul W. Frame (PhD, CHP), Tim Vitkus (CHP).

Shepherd Miller Inc. (SMI), 1999. Midnite Mine Data Transmittal Report R-2. (Background radiation investigation at the Midnite Mine). Submitted to Bureau of Land Management, Spokane District, 1103 N. Fancher, Spokane, WA 99212. April 29.

Sakoda, A., Y. Nishiyama, K. Hanamoto, Y. Ishimori, Y. Yamamoto, T. Kataoka, A. Kawabe, and K. Yamaoka, 2010. Differences of natural radioactivity and radon emanation fraction among constituent minerals of rock or soil. Applied Radiation and Isotopes, 68 (2010) 1180–1184.

Turner, J.E., 1995. Atoms, Radiation and Radiation Protection. Second Edition. John Wiley & Sons, Inc. New York.

URS Corporation (URS), 2002. Radioactive Equilibrium Evaluation for Midnite Mine RI/FS. August.

URS Corporation (URS), 2005. Final Remedial Investigation Report for Midnite Mine, Stevens County, Washington. September 30.

U.S. Environmental Protection Agency (EPA), 2000a. Guidance for the Data Quality Objectives Process. EPA QA/G-4. August.

U.S. Environmental Protection Agency (EPA), 2000b. Data Quality Objectives Process for Hazardous Waste Site Investigations. EPA QA/G-4HW. January.

U.S. Environmental Protection Agency (EPA), 2006a. Inventory of Radiological Methodologies For Sites Contaminated With Radioactive Materials. U.S. Environmental Protection Agency, Office of Air and Radiation. Office of EPA 402-R-06-007. October.

U.S. Environmental Protection Agency (EPA), 2006b. Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA QA/G-4. February.

U.S. Environmental Protection Agency (EPA), 2006c. Midnite Mine Superfund Site, Spokane Indian Reservation, Washington, Record of Decision. Office of Environmental Cleanup. EPA Region 10. September.

U.S. Environmental Protection Agency (EPA), 2007. ProUCL Version 4.0 Technical Guide. (*A technical manual for EPA statistical software*). EPA/600/R-07/041. April.

U.S. Nuclear Regulatory Commission (NRC), 1998. A Nonparametric Statistical Methodology for the Design and Analysis of Final Status Decommissioning Surveys. Interim Draft Report for Use and Comment. June.

U.S. Nuclear Regulatory Commission (NRC), 2000. Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM), Revision 1. NUREG 1575. Washington, D.C.

U.S. Nuclear Regulatory Commission (NRC), 2003. Standard Review Plan for the Review of a Reclamation Plan for Mill Tailings Sites Under Title II of the Uranium Mill Radiation Control Act of 1978. NUREG-1620. Final Report. U.S. Nuclear Regulatory Commission, Office of Nuclear Material Safety and Safeguards. Washington, D.C.

VSP Development Team (VSP), 2012. Visual Sample Plan: A Tool for Design and Analysis of Environmental Sampling. Version 6.2d. Pacific Northwest National Laboratory. Richland, WA. http://vsp.pnnl.gov.

Whicker, R., P. Cartier, J. Cain, K. Milmine, and M. Griffin, 2008. Radiological Site Characterizations: Gamma Surveys, Gamma/Ra-226 Correlations and Related Spatial Analysis Techniques. Operational Radiation Safety, Health Physics, Vol. 95 (Supplement 5): S180-S189; November.

Whicker, R., M. Whicker, J. Johnson, and H. Meyer, 2006. Mobile Soils Lab: On-site Radiological Analysis Supporting Remedial Activities. Health Physics. Operational Radiation Safety. 91(2) Supplement 1:S24-S31, August.

Worthington Miller Environmental (WME). 2014. White Tail Creek Sediment Evaluation – Phase 2. Data Transmittal Report. Rev 0. Midnite Mine, Wellpinit, Washington. February 20.

Attachment S-2

Quality Assurance Project Plan for Remedial Action Analytical Support and Verification (QAPP)

Midnite Mine Superfund Site
100 Percent Design

Attachment S-2 – Quality Assurance Project Plan for Remedial Action Analytical Support and Verification

Note: This attachment provides the Quality Assurance Protection Plan (QAPP) for the Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (Appendix S). Appendix S and its supporting attachments are intended to satisfy the requirement for a Confirmation Sampling and Remedial Action Effectiveness Monitoring Plan as stipulated by the Consent Decree.

Revised October 2015

Prepared for:

Dawn Mining Company
PO Box 250
Ford, Washington 990413

and

Newmont USA Limited
6363 South Fiddler's Green Circle
Greenwood Village, Colorado 80111
Prepared By:

Prepared by:

Worthington Miller Environmental, LLC
1027 W. Horsetooth Rd, Suite 200
Fort Collins, Colorado 80526

Environmental Resource Group (ERG)
8809 Washington St. NE, Suite 105
Albuquerque, New Mexico, 87113

Approval Sheet

Issued by:

Project Quality Assurance Manager: 10-26-15

Signature / Date

Jill Richards, Worthington Miller Environmental, LLC

Prepared for:

Supervising Contractor: 10/26/15

Signature / Date

Louis Miller, Worthington Miller Environmental, LLC

Alternate Project Coordinator: 10/26/2015

Signature / Date

Bill Lyle, Newmont Mining Cooperation

EPA Remedial Project Manager: 10/29/15

Signature / Date

Karen Keeley, US EPA

EPA Quality Assurance Manager Designee: Raymond Wu 10/29/15

Signature / Date

Distribution List

QAPP Recipient Name	Organization
Karen Keeley	U.S. Environmental Protection Agency
EPA Quality Assurance Manager	U.S. Environmental Protection Agency
Randy Connolly	Spokane Tribe of Indians
Bill Lyle	Newmont Mining Cooperation.
Louis Miller	Worthington Miller Environmental, LLC
Jill Richards	Worthington Miller Environmental, LLC
Laboratory Project Managers: Tessa Parke (Energy) Kathy Sattler (Anatek) Scott Habermehl (ACZ)	Energy Laboratories Inc., Anatek Labs Inc. ACZ Laboratories Inc.

Table of Contents

A. Project Management and Data Quality Objectives 1
A.1 Project Organization 1
A.2 Problem Definition/Background 6
A.2.1 Purpose 6
A.2.2 Problem Statement 6
A.2.3 Background 6
A.3 Project Description and Schedule 6
A.3.1 Description of Work to be Performed 6
A.3.2 Schedule of Activities 7
A.4 Data Quality Objectives (DQOs) 7
A.4.1 Measurement Performance Criteria 10
A.5 Special Training Requirements/Certification 15
A.6 Documentation and Records 16
A.6.1 Field Operation Records 16
A.6.2 Laboratory Records 16
B. Measurement Data Acquisition 18
B.1 Field Surveys and Sampling Design 18
B.2 Onsite Analytical Methods Requirements 18
B.2.1 Onsite Sampling Handling and Custody 18
B.3 Offsite Analytical Methods Requirements 22
B.3.1 Offsite Sample Handling and Custody 23
B.3.2 Special Sample Handling/Analysis Protocols for the Offsite Laboratory 23
B.4 Quality Control Requirements 30
B.4.1 Field QC Procedures 30
B.4.2 Laboratory QC Procedures 34
B.5 Instrument/Equipment Testing, Inspection and Maintenance Requirements 36
B.5.1 Field Instruments and Equipment 36
B.5.2 Laboratory Instruments 36
B.5.3 Acceptance Requirements for Supplies and Consumables 36
B.6 Instrument Calibration and Frequency 36
B.6.1 Field Calibration Procedures 36
B.6.2 Laboratory Calibration Procedures 37
B.7 Data Acquisition Requirements 37
B.8 Data Management 38
C. Assessment/Oversight 39
C.1 Assessment and Response Actions 39
C.1.1 Field Measurement Data 39
C.1.2 Laboratory Data 39
C.1.3 Performance and System Audits 39

C.1.4 Corrective Actions 40
C.2 Quality Assurance Reports to Management 41
C.2.1 Data Validation Reports....42
D. Data Validation and Usability....42
D.1 Data Review, Validation and Verification Requirements....42
D.2 Validation and Verification Methods 42
D.2.1 Validation/Verification of Field and Onsite Analysis Data....42
D.2.2 Validation/Verification of Offsite Laboratory Data....43
E. References....45

List of Tables

Table 1: Data Quality Objectives (DQO) Process Summary....8
Table 2: Analytical Acceptance/Performance Criteria....13
Table 3: Sample Containers, Preservation, and Holding Time Requirements for Solid Samples 20
Table 4: Soil and Sediment Laboratory Parameters, Analysis Methods, and Method Detection Limits 26
Table 5: Analytical Method QC Specifications....27
Table 5: Analytical Method QC Specifications (Cont.) 28
Table 5: Analytical Method QC Specifications (Cont.) 29

List of Figures

Figure 1: Project Organizational Chart 5
Figure 3: Example frequency histograms for several series of QC measurements from different NaI detector sets used for two separate gamma survey projects. Each series was taken indoors under controlled measurement geometries. The red lines represent theoretical normal distributions. 30
Figure 4: Example instrument background quality control chart for multiple NaI detectors. 31
Figure 5: Example field strip control chart for multiple NaI detectors. 32
Figure 6: Example QC chart for soils lab measurements....33

List of Appendices

Appendix 1 Standard Operating Procedures
Appendix 2 Corrective Action Report Form
Appendix 3 Approved Laboratory Quality Assurance Plans
Appendix 4 Laboratory Certification

List of Acronyms

COC	chain of custody
DQOs	data quality objectives
EPA	United States Environmental Protection Agency
HASP	Health and Safety Plan
LCS	laboratory control sample
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols
MDL	method detection limit
mg/kg	milligrams per kilogram
MS	matrix spike
MSD	matrix spike duplicate
NELAP	National Environmental Laboratory Accreditation Program
pCi/L	picocuries per liter
pCi/g	picocuries per gram
QA	quality assurance
QAM	quality assurance manager
QAPP	quality assurance project plan
QC	quality control
QSM	quality service manual
RER	replicate error ratio
RL	reporting limit
ROD	Record of Decision
RPD	relative percent difference
SOP	standard operating procedure
SOW	statement of work
µR/hr	micro-roentgen per hour

A. Project Management and Data Quality Objectives

A.1 Project Organization

The following individuals who will be involved and the tasks for which they are responsible are discussed below. An organizational chart for the project is provided in Figure 1.

EPA Remedial Project Manager (Karen Keeley, EPA)

The EPA Remedial Project Manager (RPM) provides overall EPA project oversight, is the primary decision maker for the project for EPA, and is the primary user of the data collected in accordance with the QAPP. The RPM is responsible for ensuring an EPA approved QAPP using the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) guidance is in place before beginning the collection or use of environmental information. The RPM reviews and provides comments on QAPPs; ensures that QAPPs are reviewed by the Region 10 RQAM; concurs on acceptability of the QAPP; reviews and concurs on subsequent revisions in terms of program-specific requirements; and reviews reports and ensures plans are implemented according to EPA-approved documents.

EPA Quality Assurance Manager

The EPA Quality Assurance Manager (RQAM) will be responsible for final review and approval of the QAPP and subsequent revisions for compliance with the current version of R-5, "EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations." The RQAM will provide QA technical assistance to the EPA RPM and will conduct QA audits of the project. At this time, no RQAM audits are planned; however, the EPA RPM can request an audit by the RQAM at any time during the project. If an audit is conducted, the RQAM will report audit results to the EPA RPM.

Alternate Project Coordinator/Site Manager (Bill Lyle, Newmont Mining Cooperation)

The Alternative Project Coordinator/Site Manager will be responsible for overall management and direction of the project, including:

- Management of the Water Treatment Plant
- Primary responsibility for the completion of the project activities
- Establish policies and procedures to address the needs of the project as a whole
- Overall control of planning, scheduling, and cost
- Submittal of all project reports and documents
- Primary contact for communications with the EPA

Supervising Contractor (Louis Miller, Worthington Miller Environmental, LLC)

The Supervising Contractor will be responsible for coordinating the Site activities between the Site Manager, project staff and other contractors, and the regulatory agencies. The Supervising Contractor will:

- Coordinate and schedule day-to-day activities necessary to complete project tasks, such that the objectives of each task are met

- Orient the project team concerning project requirements and special considerations
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product
- Review the work performed on each task to help ensure its quality, responsiveness and timeliness
- Review and analyze overall task performance with respect to planned requirements and authorizations
- Develop technical reports and other project documents
- Represent the project team at meetings, if necessary
- Ensure that the Health and Safety Plan (HASP) (Tetra Tech 2009), and Quality Assurance Project Plan(s) (QAPP), and any necessary corrective actions are implemented to the best of his ability.

Field Program Director

The Field Program Director will be responsible for the directing the Analytical Support and Verification Plan (Plan) (Appendix S). Responsibilities of the Field Program Director may be shared by more than one individual, but salient qualifications will include a qualified Health Physicist and Environmental Scientist with specialized expertise and experience regarding onsite analytical programs described in Appendix S. Duties of the Field Program Director(s) will include:

- Planning the setup and implementation of the onsite analytical program as detailed in Appendix S, including procurement of all necessary equipment and instruments.
- Setting up onsite analytical facilities, systems, equipment, and instruments and developing onsite system calibrations and QC procedures.
- Ensuring that field personnel are qualified and properly trained to implement the Analytical Support and Verification Plan (Appendix S).
- Monitoring and evaluating onsite analytical operations and data during the remedial action (RA), identifying and resolving technical issues, assessing changing data needs and directing appropriate responses.
- Developing interim (remedial support) progress updates and final status survey reports.
- Reports directly to the Supervising Contractor, providing the principal point of contact and control for matters concerning analytical results of both remedial support and final status surveys.

Field Supervisor

The Field Supervisor will be responsible for all aspects of fieldwork performed in accordance with the Analytical Support and Verification Plan (Appendix S). The Field Supervisor will have a background in Health Physics and applicable environmental sciences and must be qualified by education, experience and training to implement the analytical field program at the direction of the Field Program Director. Duties of the Field Supervisor will include:

- Ensuring that all field activities, including measurements, data collection, and field recording activities are performed in accordance with the Analytical Support and

Verification Plan (Appendix S), the direction of the Field Program Director, and with this QAPP.

- Managing and ensuring proper implementation of field surveys and effective and efficient operation of the onsite soils lab (including both analytical and health and safety aspects of onsite soils lab functions). This includes overseeing field surveys, sample collection and onsite sample processing, onsite sample analysis, sample management, storage and shipping, and ensuring proper function of field equipment.
- Real-time data review and assessment, and respective collaboration with the Field Program Director regarding additional data needs.
- Identification of potential analytical problems along with respective troubleshooting and taking corrective action as needed to resolve any equipment malfunctions or systemic procedural sources of potentially unreliable data.
- Ensuring that field personnel are properly task trained, equipped, and familiar with Standard Operating Procedures (SOPs) and the HASP (Tetra Tech, 2009) and the Radiation Protection Plan (RPP) (SENES, 2013).
- Ensuring that appropriate personal protective equipment will be worn and disposed of according to the HASP.
- Reports directly to the Field Program Director, providing the principal point of contact and control for matters concerning implementation of the Analytical Support and Verification Plan (Appendix S).

Field Technician

Field Technician will be responsible for the proper implementation of fieldwork in accordance with the Analytical Support and Verification Plan (Appendix S) and the direction of the Field Supervisor and Field Program Director. Duties of Field Technicians include:

- Ensuring that all field activities, including measurements, data collection, and field recording activities are performed in accordance with the direction of the Field Supervisor, with Appendix S, and with the QAPP.
- Ensuring that appropriate personal protective equipment is worn and disposed of according to the HASP.
- Reports directly to the Field Supervisor.

Site Safety Officer

The Site Safety Officer will be responsible for health and safety at the site. Duties of the Safety Officer include:

- Ensuring that personnel have the proper site health and safety training and are familiar with the HASP.
- Ensuring that appropriate personal protective equipment will be worn and disposed of according to the HASP.

Quality Assurance Manager (Jill Richards, Worthington Miller Environmental, LLC)

The Project Quality Assurance Manager (QAM) ensures that the project's QA program is conforming to the project requirements. Duties will include:

- Coordination of the receipt of data from the offsite analytical laboratory.
- Ensuring that all data is properly reviewed, verified and validated, including collaboration with the Field Program Director with respect to data generated onsite.
- Evaluation of the data and any concerns that may arise with laboratory, and communicates with the Field Program Director regarding laboratory data reports or data validation concerns.
- Performing QA audits on various phases of the project's operations as necessary and providing QA technical assistance to project staff.
- Notifying the Field Program Director of particular circumstances that may adversely affect the quality of data and ensure implementation of corrective actions needed to resolve nonconformance's noted during assessments.

The QAM will not actively participate in the collection of samples, thereby establishing independence from the data generating team.

Analytical (Offsite) Laboratory Project Manager

The Analytical Offsite Laboratory Project Managers include the following persons: Tessa Parke (Energy Laboratories), Kathy Sattler (Anatek Labs), and Scott Habermehl (ACZ Laboratories). The Laboratory Project Managers will work directly with the Laboratory QA Officers and will be responsible for the following:

- Reviews and approves the offsite laboratory and related analytical-specific sections of the Project QAPP
- Reviews in-house chain-of-custody (COC)
- Coordinating laboratory analyses
- Reviewing/approving appropriate laboratory QA procedures
- Overseeing laboratory QA and QA/QC documentation
- Ensuring all resources of the laboratory are available to meet project schedules
- Determining whether to implement laboratory corrective actions, if required
- Overseeing laboratory data review
- Ensuring all QA/QC objectives, policies, and procedures are followed according to the laboratory QAP
- Overseeing production and final review of analytical reports

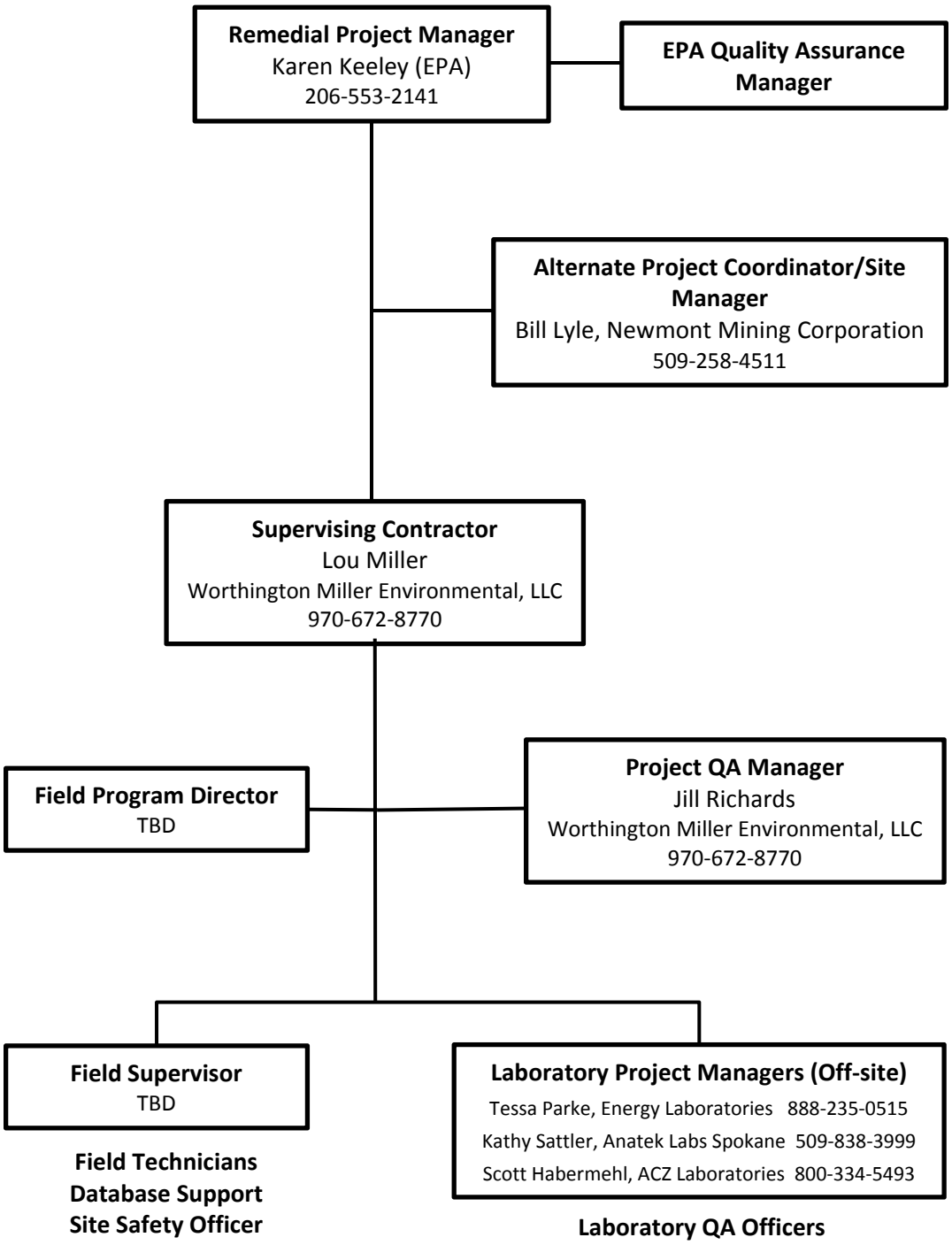


Figure 1: Project Organizational Chart

A.2 Problem Definition/Background

A.2.1 Purpose

This Quality Assurance Project Plan (QAPP) for the Analytical Support and Verification Plan (Plan) (Appendix S) was developed to assure that the data collected will meet appropriate data quality objectives (DQOs) and to describe the policies and procedures for data collection and evaluation activities associated with the Plan. The purpose of the Plan is to propose analytical approaches and methods that will be used to support remedial excavations and final status surveys to demonstrate compliance with ROD cleanup levels for surface materials and sediments at the Midnite Mine Site.

A.2.2 Problem Statement

The necessary information to be obtained includes:

- Remedial support survey data to guide remedial excavation.
- Additional characterization of potentially impacted areas.
- Final status survey data in defined survey units.
- Determination of compliance with ROD cleanup levels across the mine area and all potentially impacted areas in the vicinity of the site.

A.2.3 Background

The Midnite Mine Superfund Site is located on the Spokane Indian Reservation in eastern Washington, approximately 45 miles northwest of Spokane. Active mining occurred at the Site for over 23 years, starting in 1954. The Site includes an inactive open pit uranium mine and area and media impacted by mine-related contamination, including heavy metals and radionuclides. Long term monitoring has been ongoing at the Site in affected areas and media including groundwater, surface water and sediment.

A.3 Project Description and Schedule

A.3.1 Description of Work to be Performed

Survey data is to be collected for remedial support during remedial excavations as well as to determine compliance with ROD cleanup levels during final status surveys. The survey activities are described in detail in the Plan (Appendix S, Sections S.3.0 and S.4.0). This includes gamma scanning and collection of soil/sediment samples for onsite gamma spectroscopy, onsite XRF measurements (both in-situ and intrusive methods), and offsite laboratory analysis. Final status surveys will be conducted in Class 1 and Class 2 areas identified based on historical information and the remedial support survey data collected during the remedial activities. Class 1 and Class 2 areas have been initially estimated based on previous radiological surveys of the Site. Class 1 areas are areas likely to be impacted and that prior to remediation are generally expected to have sampling results in excess of the cleanup levels specified in the ROD. Class 2 areas encompass the “halo” of potentially impacted areas surrounding Class 1 areas; they represent areas that may be slightly impacted, but are generally not expected to have sampling results in excess of ROD cleanup levels.

A.3.2 Schedule of Activities

Remedial support surveys (gamma scans, gamma spectroscopy, XRF measurements, soil and sediment sampling) of excavated areas will occur concurrently with remedial excavation activities. Final status surveys will be conducted in Class 1 and Class 2 areas identified based on historical information and the remedial support survey data collected during the remedial activities. Final status soil or sediment sampling will not take place until after the final status gamma scans have first been performed and evaluated. This work is anticipated to occur periodically over a period of approximately 10 years.

A.4 Data Quality Objectives (DQOs)

Remedial support and final status survey DQOs for remediation of surface materials and sediments are provided in Table 1. Generalized flowchart overviews of respective remedial Plans, including mine waste removal, analytical assessment and decision criteria are depicted in Figures S-4 and S-5 of Appendix S. The generalized Plan diagrams in Appendix S, as well as the detailed specifications of the Plan as indicated throughout Appendix S, are based on the DQOs indicated in Table 1 of this QAPP.

Table 1: Data Quality Objectives (DQO) Process Summary

Step 1: Problem Statement	Step 2: Identify the Decision	Step 3: Identify Inputs to the Decision	Step 4: Define the Boundaries of the Study	Step 5: Develop a Decision Rule	Step 6: Specify Limits on Decision Errors	Step 7: Develop the Plan for Obtaining Data
REMEDIAL SUPPORT SURVEYS FOR SURFACE MATERIALS The ROD requires surface materials that exceed the cleanup levels to be excavated and consolidated in the waste containment area. During remedial action, real-time, or near real-time, data are needed to determine when excavation of surface materials is no longer required and the area is ready for final status surveys.	Principal Study Question: Do surface materials in the study area have constituents in excess of ROD cleanup levels? Possible Outcomes: 1) The data indicate that surface materials exceed cleanup levels and excavation should continue. 2) The data indicate that surface materials meet cleanup levels and final status surveys should proceed. 3) Excavation has progressed to bedrock and no further excavation can proceed. Decision Statement: The decision to be made is whether the study area is ready for final status surveys, or if further excavation and/or additional survey data are required.	ROD cleanup levels for surface materials include only radiological parameters (Table S-1, Appendix S). Compliance with ROD cleanup levels for surface materials can accommodate considerable radiological disequilibrium in these parameters. Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that Ra-226 will largely govern the final extent of excavation needed to meet surface material cleanup levels. Analytical inputs to remedial support decisions will include gamma scanning, gamma cutoff values based on gamma/Ra-226 correlations, analysis of samples for Ra-226 in an onsite soils lab using NaI-based gamma spectroscopy, and XRF measurements to screen for uranium in limited areas with potential for unusually high radiological disequilibrium between Ra-226 and uranium. Confirmatory sample analyses at an offsite laboratory will be performed on about 10% of samples.	On a macro scale, the initial study area for remedial support surveys will include all areas in the vicinity of the Site with known to exceed the cleanup criteria for surface materials (about 307 acres), along with a "halo" or margin surrounding these areas that has some potential for impacts. The study area does not include the footprint of mine pits that will serve as final repositories for contaminated materials. The initial study area is subject to change based on remedial support survey data. On a micro scale, the study area will include areas of active excavations, which will advance vertically and horizontally until remedial support survey data provide sufficient evidence to support a decision that respective locations are ready for final status surveys.	Decision criteria guidelines for evaluation of remedial support survey data for surface materials include the following: • Nearly 100% of gamma readings* ≤ gamma cutoff value. • Nearly 100% of onsite Ra-226 sample analysis results* ≤ cleanup level. • Nearly 100% of XRF results* for uranium ≤ cleanup level. • Nearly 100% of offsite analysis results* ≤ cleanup levels. A more detailed listing is provided in Section S.3.4.1 of Appendix S. If these guideline criteria are not met, then further excavation is likely necessary in the subject area. Otherwise, the area can be considered ready for final status surveys. *(e.g. > 98%)	The limit on decision errors for determination of whether an area in question is ready for final status surveys is 5% or less. The decision criteria guidelines for surface materials (Section S.3.4.1of Appendix S) are conservative and are expected to limit respective decision errors to well below 5%.	Detailed plans for obtaining remedial support survey data are provided in Sections S.2.0 and S.3.0 of Appendix S.
REMEDIAL SUPPORT SURVEYS FOR SEDIMENTS The ROD requires mine drainage sediments that exceed the cleanup levels to be excavated and contained with other waste materials in the waste containment area. During remedial action, real-time, or near real-time, data are needed to determine when excavation of sediments is no longer required and the area is ready for final status surveys.	Principal Study Question: Do sediments in the study area have constituents in excess of ROD cleanup levels? Possible Outcomes: 1) The data indicate that sediments exceed cleanup levels and excavation should continue. 2) The data indicate that sediments meet cleanup levels and final status surveys should proceed. 3) Excavation has progressed to bedrock and no further excavation can proceed. Decision Statement: The decision to be made is whether the study area is ready for final status surveys, or if further excavation and/or additional survey data are required.	ROD cleanup levels for sediments include both Radiological and non-radiological parameters (Table S-2, Appendix S). Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that non-radiological parameters (metals), particularly manganese, exceed cleanup levels to the greatest extent in sediments. Analytical inputs to remedial support decisions will include in-situ XRF screening of excavated surfaces along with sampling and intrusive XRF analysis as the primary methods to guide excavations and determine when areas are ready for final status surveys. Confirmatory sample analyses at an offsite laboratory will be performed on about 10% of samples. Gamma scans and Ra-226 analysis in the onsite soils lab will be used to provide additional information for decision making.	On a macro scale, the initial study area for remedial support surveys in mine drainages will include all areas in the vicinity of the drainages with the potential to exceed the cleanup levels for sediments. Based on existing data, about 2 lineal miles of mine drainage channels are potentially impacted. The initial study area is subject to change based on remedial support survey data. On a micro scale, the study area will include areas of active excavations, which will advance vertically and horizontally until remedial support survey data provide sufficient evidence to support a decision that respective locations are ready for final status surveys.	Decision criteria guidelines for evaluation of remedial support survey data for sediments include the following: • Nearly 100% of in-situ XRF Mn readings* ≤ Mn cutoff value. • Nearly 100% of onsite/offsite lab analysis results* ≤ cleanup levels. • Nearly 100% of gamma readings* ≤ gamma cutoff value. • Nearly 100% of onsite Ra-226 analysis results* ≤ cleanup level. A more detailed listing is provided in Section S.3.4.2 of Appendix S. If these guideline criteria are not met, then further excavation is likely necessary in the subject area. Otherwise, the area can be considered ready for final status surveys. *(e.g. > 98%)	The limit on decision errors for determination of whether an area in question is ready for final status surveys is 5% or less. The decision criteria guidelines for sediments (Section S.3.4.2 of Appendix S) are conservative and are expected to limit respective decision errors to well below 5%.	Detailed plans for obtaining remedial support survey data are provided in Sections S.2.0 and S.3.0 of Appendix S.

Step 1: Problem Statement	Step 2: Identify the Decision	Step 3: Identify Inputs to the Decision	Step 4: Define the Boundaries of the Study	Step 5: Develop a Decision Rule	Step 6: Specify Limits on Decision Errors	Step 7: Develop the Plan for Obtaining Data
FINAL STATUS SURVEYS FOR SURFACE MATERIALS The ROD requires surface materials that exceed the cleanup levels to be excavated and consolidated in the waste containment area. Once remedial support survey data provides strong evidence of compliance with ROD cleanup levels, final status surveys must provide data of sufficient quantity/quality to formally verify compliance in terms of specified acceptance parameters and decision criteria.	Principal Study Question: Do surface materials in the survey unit have constituents in excess of ROD cleanup levels? Possible Outcomes: 1) The data indicate that the survey unit exceeds ROD cleanup levels and further excavation is required. 2) The data indicate that survey unit is in statistical compliance with ROD cleanup levels, but one or more samples exceed a secondary cleanup level of twice the ROD cleanup level (further action is required). 3) The survey unit is in statistical compliance with ROD cleanup levels, and no samples exceed a secondary cleanup level of twice the ROD cleanup level (recommendation of no further action is appropriate) Decision Statement: The decision to be made is whether the survey unit meets ROD cleanup levels and associated acceptance criteria, or if additional survey data and further excavation are required.	ROD cleanup levels for surface materials include only radiological parameters (Table S-1, Appendix S). Compliance with ROD cleanup levels for surface materials can accommodate considerable radiological disequilibrium in these parameters. Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that Ra-226 exceeds cleanup levels to the greatest extent in surface materials. Analytical inputs to final status survey decisions will include recorded gamma surveys, assessment of respective data against gamma cutoff values based on gamma/Ra-226 correlations, indirect estimation of Ra-226 concentrations based on gamma scan data, analysis of all samples for Ra-226 in an onsite soils lab using NaI-based gamma spectroscopy, and analysis of 33% of samples for all surface material COCs in an offsite laboratory.	The study area for final status surveys for surface material will include defined Class 1 or Class 2 survey units. Class 1 and Class 2 areas and respective survey units have been prospectively estimated in Section S.3.2.1 of Appendix S. These areas will be refined as needed based on remedial support data and on the actual sequencing of remedial excavations. Adjacent locations are defined as unimpacted Class 3 background areas and will not be part of final status surveys. The footprint of mine pit repositories for contaminated materials is not included in the study area. Any locations excavated to bedrock will not be evaluated in terms of ROD cleanup levels, but gamma surveys will be conducted to document final status gamma readings.	Decision criteria for evaluation of final status survey data for surface material survey units include the following: • At least 95% of gamma readings ≤ gamma cutoff value. • At least 95% of gamma-based Ra-226 estimates≤ cleanup level. • Statistical test of sample Ra-226 analysis results indicates (with 95% confidence) at least 95% of the survey unit meets the cleanup level. • No single sample result > twice cleanup level. A more detailed listing is provided in Section S.4.2.3 of Appendix S. If one or more of these decision criteria are not met, then additional survey data will be required and further excavation may be necessary. Otherwise, a decision of no further action is appropriate.	The decision criteria for surface materials (Section S.4.2.3 of Appendix S) are each designed to limit respective decision errors to 5%. When combined with more restrictive decision criteria for previous remedial support surveys, cumulative analytical evidence and decision criteria are expected to limit the overall probability of both Type I and Type II errors on decisions regarding compliance with ROD cleanup levels to less than 5% (i.e. $\alpha = \beta < 0.05$).	The plans for obtaining final status survey data for surface materials are detailed in Sections S.2.0 and S.4.0 of Appendix S.
FINAL STATUS SURVEYS FOR SEDIMENTS The ROD requires sediments that exceed the cleanup levels to be excavated and consolidated in the waste containment area. Once remedial support survey data provides strong evidence of compliance with ROD cleanup levels, final status surveys must provide data of sufficient quantity/quality to formally verify compliance in terms of specified acceptance parameters and decision criteria.	Principal Study Question: Do sediments in the mine drainage survey unit have constituents in excess of ROD cleanup levels? Possible Outcomes: 1) The data indicate that the survey unit exceeds ROD cleanup levels and further excavation is required. 2) The data indicate that survey unit is in statistical compliance with ROD cleanup levels, but one or more samples exceed a secondary cleanup level of twice the ROD cleanup level (further action is required). 3) The survey unit is in statistical compliance with ROD cleanup levels, and no samples exceed a secondary cleanup level of twice the ROD cleanup level (recommendation of no further action is appropriate) Decision Statement: The decision to be made is whether the survey unit meets ROD cleanup levels and associated acceptance criteria, or if additional survey data and further excavation are required.	ROD cleanup levels for sediments include both Radiological and non-radiological parameters (Table S-2, Appendix S). Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that non-radiological parameters (metals), particularly manganese, exceed cleanup levels to the greatest extent in sediments. Analytical inputs to final status survey decisions will include recorded in-situ XRF surveys along mine drainage survey units, direct sampling along survey grids with onsite intrusive XRF analysis, analysis of 33% of samples for all surface material COCs in an offsite laboratory, recorded gamma scans and sample Ra-226 analysis in the onsite soils lab.	The study area for final status surveys for sediments along mine drainages will include defined Class 1 survey units. These survey units have been prospectively estimated in Section S.3.2.1 of Appendix S. Class 2 areas are unlikely to be realistically distinguishable from Class 1 areas along these narrow areas of potential impacts. Adjacent locations are defined as unimpacted Class 3 background areas and will not be part of final status surveys. Mine drainage survey units will be refined as needed based on remedial support data. Any locations excavated to bedrock will not be evaluated in terms of ROD cleanup levels, but gamma surveys will be conducted to document final status gamma readings.	Decision criteria for evaluation of final status survey data for mine drainage survey units include the following: • At least 95% of in-situ XRF readings for ROD parameters ≤ cutoff values. • At least 95% of gamma readings ≤ gamma cutoff value. • At least 95% of gamma-based Ra-226 estimates≤ cleanup level. • Statistical test of sample analysis results indicates (with 95% confidence) at least 95% of the survey unit meets the cleanup level for each ROD parameter. • No single sample result > twice cleanup level. A more detailed listing is provided in Section S.4.2.4 of Appendix S. If one or more of these decision criteria are not met, then additional survey data will be required and further excavation may be necessary. Otherwise, a decision of no further action is appropriate.	The decision criteria for sediments (Section S.4.2.4 of Appendix S) are each designed to limit respective decision errors to 5%. When combined with more restrictive decision criteria for previous remedial support surveys, cumulative analytical evidence and decision criteria are expected to limit the overall probability of both Type I and Type II errors on decisions regarding compliance with ROD cleanup levels to less than 5% (i.e. $\alpha = \beta < 0.05$).	The plans for obtaining final status survey data for surface materials are detailed in Sections S.2.0 and S.4.0 of Appendix S.

A.4.1 Measurement Performance Criteria

Measurement performance criteria are established for each field and laboratory measurement parameter. Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for accuracy, precision and completeness. Quality control acceptance criteria for accuracy, precision and completeness of data to meet the data objectives of the project are shown in Table 2. Definitions for accuracy, precision, completeness, representativeness, and comparability are provided below. The level of quality control effort is described in Section B.5. Project required method detection limits (MDLs) for the chemical analysis are included in Tables 5-6.

Precision

Precision is a measure of the degree to which two or more measurements are in agreement. Determining the agreement among replicate measurements of the same sample assesses the precision of the analytical method; combined precision of sampling and analysis methods is assessed from the agreement between measurements of field duplicate samples.

Field Precision Objectives

For field gamma survey measurements, precision for a given instrument over time, and precision between instruments, will be evaluated based on daily quality control measurements (see AS-SOP 6, Appendix 1). Any radiation detection instrument to be used in the field must have daily QC measurements that remain within ± 3 standard deviations of the mean of all measurements of both background and a Cs-137 check source. For field in-situ XRF measurements, precision will be evaluated on a daily basis (at least once per day) by replicate measurements of an in-situ XRF sample per Method 6200 (7 replicates) and calculation of the relative standard deviation (RSD). The RSD should be < 20% for metals (see AS-SOP 5, Appendix 1 for details on RSD calculation).

Precision of sampling and analysis methods will be assessed through the collection of field duplicate samples. Field duplicates are collected to measure the sampling and analytical variability or imprecision associated with the sample results. The relative percent difference (RPD) in the results for each analyte will be computed for each field duplicate pair using the equation provided in Section B.5.2.4. Since there are no U.S. EPA criteria for evaluation of field duplicate sample comparability, the goal for precision of field duplicate results is ± 50% RPD for solid (soil and sediment) samples. However, if one or both samples in a field duplicate pair have a concentration less than 10x the laboratory reporting limit (RL), the field precision goal will be ± 5 x the RL. It is noted here that natural variation in solid samples will affect how closely these goals are met; that is, if variation is high, then these goals may be unrealistic. Consequently, RPD results from field duplicates of solid samples will not be used as a basis of invalidating any analytical data.

Laboratory Precision Objectives

Precision of the analytical method will be assessed through duplicate analyses of laboratory QC and field samples. The relative percent difference (RPD) in the results for each analyte will be

computed for each analytical duplicate pair using the equation provided in Section B.5.2.4. Data for duplicate analysis will be evaluated only if both of the samples in the duplicate pair have a concentration greater than the laboratory RL. The limit for precision of laboratory analytical duplicates and MS/MSD is 35% RPD for solid samples >5x the RL. Precision for radiochemical analyses will also be assessed by the Replicate Error Ratio (RER) using the equation provided in Section 8.2. The laboratory RER goal is < 2.0.

For onsite laboratory sample analysis using the intrusive XRF method, precision will be evaluated on a daily basis (at least once per day) by replicate measurements of a field sample prepared for intrusive XRF analysis. Per Method 6200, 7 replicate measurements will be performed on the same sample and RSD will be calculated. The RSD should be < 20% for metals (see AS-SOP 5, Appendix 1 for details on RSD calculation).

Where appropriate, laboratory precision goals for each method and each sample type are included in Table 2. The frequency at which offsite laboratory duplicates should be analyzed is to be at a minimum rate of one duplicate per 20 samples, provided there is sufficient sample.

Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. For gamma-based estimates of Ra-226 along with the gamma cutoff value, accuracy will be evaluated by confirmatory sampling and analysis in the onsite soils lab and/or offsite analysis in the commercial laboratory. For gamma spectroscopy and XRF analysis in the onsite soils lab, data accuracy will be evaluated based measurements of site-specific calibration standards or references materials, agreement between onsite soils lab results and paired analysis results from the commercial laboratory, and related comparisons of field sample analysis results against estimation uncertainty reflected in the current method calibration algorithm[1] (expressed in units of activity concentration). For offsite laboratory analysis, data accuracy will be evaluated based on batch-specific measurements of certified soil reference material standards (for gamma spectroscopy) or for chemical separation methods, using the results from laboratory control samples (LCS) and matrix spikes (MS), expressed as the percent recovery or the percentage of the true (known) concentration that is measured.

Field Accuracy Objectives

Field accuracy for in-situ XRF survey measurements will be controlled and/or assessed in several ways. First, per AS-SOP 5 (Appendix 1) the following QC checks on XRF instrument performance will be performed each day:

- Energy calibration check sample

[1] Note that throughout this QAPP, use of the term “calibration algorithm” in association with gamma spectroscopy in the onsite soils lab refers to one or more statistical regressions that may be used to predict full-ingrowth Ra-226 concentrations as measured with a high-purity germanium (HPGe) detector at an offsite laboratory. The basis for this algorithm, including potential use of separate statistical regression curves for high and low ranges of sample values, and which may be non-linear for low range values, is discussed in Section S.2.2.3 of Appendix S.

- Instrument blank
- Method blank
- Calibration verification checks (NIST standard)

The details, protocols and performance/acceptance criteria for these daily QC checks are based on Method 6200 and are provided in AS-SOP 5. Secondly, accuracy can be evaluated by comparison of in-situ XRF field measurements against the estimation uncertainty reflected in the analyte-specific calibration (regression) curves established based on field in-situ measurements and offsite analyses of corresponding samples at a commercial laboratory. Specifically, 90% of onsite in-situ XRF measurements are expected to fall within two-sided 90% prediction intervals on the calibration curve for each analyte (each ROD metal parameter). This will limit the probability of under-estimation of equivalent results at the offsite lab (a Type I estimation error) to less than 5%.

The accuracy of using gamma survey measurements to estimate soil Ra-226 concentrations in-situ in the field (based on the gamma/Ra-226 correlation) will be assessed based on comparisons of mapped estimates of Ra-226 values based on gamma survey measurements, against direct soil sampling and Ra-226 analysis results in corresponding locations. Generally speaking, at least 90% of gamma survey-based estimates of Ra-226 at these specific locations should fall within the limits of two-sided 90% prediction intervals on the regression curve fitted to gamma/Ra-226 correlation data. This will limit the probability of under-estimation of direct sampling and full-ingrowth results at the offsite lab (a Type I estimation error) to less than 5%.

The accuracy of gamma survey data (in µR/hr) cannot be assessed in terms of true exposure rates without onsite calibration against a high-pressure ionization chamber (HPIC), but it can be assessed in a relative sense based on instrument calibrations against a Cs-137 source at the instrument manufacturer's calibration facility. Instrument calibrations are required on an annual basis for all instruments to be used on the project, and calibration certificates must be retained in the project records. This relative measure of accuracy is linked to measurement precision, both temporally and between various instruments. Field measurement precision is crucial for the success of the Plan. Field measurement precision can be controlled with annual instrument calibrations and can be assessed on a daily basis with QC checks on all gamma survey equipment (see Section B.4.1 and AS-SOP 6).

Laboratory Accuracy Objectives

Laboratory accuracy may be evaluated by the analysis of LCS and MS samples, with results expressed as a percentage recovery measured relative to the true (known) concentration. For Ra-226 analysis in the onsite lab, accuracy will be evaluated based on daily QC checks on measurements of site-specific calibration standards (field samples with concentrations established by the offsite laboratory) against established control limits, and by comparison of field sample analysis results against the estimation uncertainty reflected in the method calibration algorithm in use at the time of sample analysis. Specifically, at least 90% of onsite analysis results for Ra-226 in field samples should fall within the limits of two-sided 90% prediction intervals on the current method calibration algorithm (expressed in units of Ra-226 activity concentration). This will limit the probability of under-estimation of equivalent full-ingrowth results at the offsite

lab (a Type I estimation error) to less than 5%. Because onsite/offsite analytical results for the fraction of samples sent offsite for confirmation of onsite results will generally be added to the system calibration curve, this protocol will provide a conservative degree of assurance that agreement between onsite and offsite results is acceptable in a context of the project DQOs.

For intrusive XRF sample analysis in the onsite lab, accuracy will be controlled and/or assessed in several ways. First, per AS-SOP 5 (Appendix 1) the following QC checks on XRF instrument performance will be performed each day:

- Energy calibration check sample
- Instrument blank
- Method blank
- Calibration verification checks (NIST standard)

The details, protocols and performance/acceptance criteria for these daily QC checks are based on Method 6200 and are provided in AS-SOP 5. Secondly, accuracy can be evaluated by comparison of intrusive XRF sample analysis results against the estimation uncertainty reflected in respective analyte-specific calibration (regression) curves established based on intrusive XRF sample analysis results and corresponding offsite analyses of the same samples at a commercial laboratory. Specifically, 90% of onsite intrusive XRF analysis results are expected to fall within two-sided 90% prediction intervals on these calibration curves. This will limit the probability of under-estimation of equivalent results at the offsite lab (a Type I estimation error) to less than 5%.

These specifications, along with offsite laboratory LCS, MS, and matrix spike duplicate (MSD) recovery goals, as well as RPD's for laboratory and field duplicate analyses, are provided in Table 2 (additional specifications for XRF data are provided in AS-SOP 5 and Method 6200).

Table 2: Analytical Acceptance/Performance Criteria

Sample Media	Analyte	Accuracy Measures and Control Limits[1]	Precision Measures and Control Limits[1]	Completeness Goal
Soil and Sediment Samples	Total chromium (Cr), total manganese (Mn), total selenium (Se), total uranium (U), total vanadium (V); Ra-226[2], Pb-210, U-234, and U-238	<u>Offsite lab analysis:</u> LCS Recovery: 80-100% MS/MSD Recovery: 70-130% <u>Onsite lab data:</u> 1) QC measurements on site-specific calibration standards within established control limits. 2) 90% of field sample results within limits of 90% prediction Intervals on calibration curves.	<u>Offsite lab analysis:</u> Analytical Duplicate RPD: <35% MS/MSD RPD: <35% Radiochemical Analysis RER: <2.0 <u>Field duplicate RPD:</u> <50% <u>XRF measurements:</u> <20% RSD among 7 replicates	<u>Laboratory:</u> 95% <u>Gamma and XRF:</u> 100%

Sample Media	Analyte	Accuracy Measures and Control Limits[1]	Precision Measures and Control Limits[1]	Completeness Goal
		XRF measurements: Calibration Verification Check = +/- 20% of true value		

[1]Note: Due to the nature of solid samples (i.e., matrix interferences and homogeneity difficulties), a broader control limit for MS recoveries and higher RPD limits are acceptable.

[2]Chemical recovery does not apply to gamma spectroscopy (100% of the analyte is present in the original sample matrix during counting).

Completeness

Completeness is the percentage of valid measurements or data points obtained, as a proportion of the number of measurements or data points planned for the project. Completeness is affected by such factors as access to monitoring locations, sample container breakage and acceptance/non-acceptance of analytical results. Percentage completeness (C) is calculated by the following equation:

$$C(\%) = \frac{V}{P} \times 100$$

Where: V = number of valid measurements/data points obtained
P = number of measurements/data points planned

The laboratory completeness goal is 95%. For gamma or XRF field survey data, completeness should approach 100%.

Representativeness

Representativeness is a qualitative objective, defined as 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. Representativeness is achieved by collecting a sufficient number of unbiased (random) samples or field measurements with consistency in sampling and measurement technique and with proper sample collection equipment and associated decontamination procedures as applicable. The sampling and field measurement approaches developed for a project should provide for data that are representative of actual site conditions in a context of remedial objectives. Examples of representativeness for this project would include:

- When discrete surface samples accurately reflect the distribution of all values across a given survey unit and can thus be used to statistically evaluate whether the survey is compliance with ROD cleanup levels at a specified level of confidence.
- When soil or sediment samples are thoroughly homogenized such that analytical results for aliquots drawn from the bulk sample accurately reflect the true average concentration in the bulk sample.

- When composited soil samples across a gamma/Ra-226 correlation plot accurately reflect the true average Ra-226 concentration across the plot.
- When gamma readings in the general vicinity of a gamma/Ra-226 correlation plot are relatively uniform and are not influenced by non-representative gamma shine from adjacent areas.

Representativeness of analytical results is best assured by following established statistical methods for selection of sampling locations, appropriate analytical approaches and consistency in implementation of respective methodologies, adequate sampling/measurement frequencies, and QC measurements to assure analytical comparability with remedial action levels established based on previous measurements (see next section). For example, when gamma surveys are performed at 100% coverage, spatial representativeness is assured as the entire population of gamma exposure rates across the area in question is established. However, the quantitative representativeness of such scanning is still dependent on the precision or reproducibility of such measurements (e.g. when evaluating results against the gamma cutoff value or when estimating Ra-226 concentrations based on the gamma/Ra-226 correlation).

Comparability

Comparability is the confidence with which one data set can be compared to another, or with which values can be accurately compared against remedial action levels that have been established based on previous sampling or measurements. Comparability is achieved by consistency in the use of appropriate sampling methods and standard operating procedures, analytical methods and performing data evaluations. Comparability is also dependent on similar QA objectives. All data should be calculated and reported in units consistent with standard reporting procedures so that the results of the analyses can be compared with those of other laboratories, if necessary.

A.5 Special Training Requirements/Certification

The Field Program Director(s) must have specialized education, expertise and experience in designing, setting up, operating and directing radiological and XRF analysis programs as indicated in Appendix S,. The Field Program Director(s) will have expertise in environmental health physics, radiochemistry, radiological characterization methods, evaluation of radiological data (including QC related aspects), along with implementation of XRF analysis methods and related data assessments. The Field Program Director(s) will evaluate both field survey data and onsite lab data throughout the project, along with changing sampling and measurement needs as required to ensure reliable results, that the data are sufficient to meet the project DQOs, and to make correct decisions regarding compliance with ROD cleanup levels.

All field personnel that will manage and/or perform onsite gamma spectroscopy in the soils lab and official gamma surveys in the field, must be qualified to perform these functions by appropriate training, education and experience. This includes sufficient background in health physics and the ability to properly interpret radiological data, identify analytical problems, and to troubleshoot and resolve equipment issues. Similarly, personnel performing onsite XRF measurements must be

qualified by training, education and experience to perform such functions, including the safe handling of either source material or radiation generating types of XRF instruments.

Like RA workers, all personnel and sub-contractors performing onsite analytical support for RA activities must have 40 hours of OSHA HAZWOPER Training and must be updated annually with 8-hours of Refresher training. In addition, site specific training is required prior to entering the restricted area. Documentation will be kept at the facility indicating fulfillment of adequate training and yearly refresher courses.

A.6 Documentation and Records

A.6.1 Field Operation Records

Field operation records include documentation of sample collection information in field logbooks, entry of corresponding information into an electronic sample login/tracking spreadsheet in the onsite soils lab (see Appendix 1, AS-SOP 3), QC records for onsite sample analysis methods and equipment, QC records for field survey methods and equipment (gamma surveys, in-situ XRF surveys), COCs for samples to be sent offsite, and internal/external audit and corrective action reports.

Field logbook entries must be very detailed, including at minimum sampling date and time, sample location ID number, GPS coordinates, sample matrix (soil or sediment), type of sample (primary or field duplicate), sample collector's name, and notes on any observations of environmental factors or circumstances that could potentially be relevant in terms of sample analysis results and/or interpretation of those results. Calibration information for any equipment used for field measurements should be also noted in the field logbook. Other circumstances that would be required to document in the field logbook could include moving a sampling location, or if there were any circumstances at a site that prevented a sample from being collected. If a deviation in the field sampling methods or standard operating procedure (SOP) is required, it will be documented indicating what occurred, actions taken to correct the deviation (if possible), as well as the potential effect of the action on the sample in question.

COCs will be filled out for all samples to be shipped to an offsite laboratory for additional analysis and these forms will include the information discussed in Section B.3.2 and in AS-SOP 3 (see Appendix 1). Field logbook entries will be scanned to an electronic file when samples are submitted to the onsite soils lab, and the sample collection information for each sample will be entered into the sample login/tracking spreadsheet. Hardcopies of both the completed logbooks and copies of COC forms will be retained and filed per the direction of the Field Supervisor. Once the analytical results for samples analyzed offsite have been recorded in the project database, the original data reports will be scanned to electronic files and archived. All instrument calibration certificates will be archived as scanned electronic documents in the project files.

A.6.2 Laboratory Records

Laboratory records will include all of the data in the data reporting package (described in Section B.8) as well as any laboratory records generated for the project samples. In addition to the items

in the data reporting package, at a minimum, the following records will be maintained by the laboratory:

- Sample preparation logbooks
- Equipment calibration and maintenance records
- Instrument run logs, extraction logs, and digestion logs
- Certification records for standards
- Raw data

Laboratory records will be archived for the minimum period of ten years.

B. Measurement Data Acquisition

B.1 Field Surveys and Sampling Design

Field surveys and samples obtained during activities associated with the Plan, as well as specified survey measurement densities and number of samples to be collected, are described in detail in the Plan (Appendix S, Sections S.3.0 and S.4.0). The Remedial Support (Section S.3.0) and Final Status (Section S4.0) Surveys include sampling for onsite (gamma spectroscopy and insitu XRF) and offsite chemical analysis. Samples generated from the final status surveys for surface material will be collected from survey units based on the final status gamma survey, and sediment samples will be collected based on final status of XRF and gamma surveys.

The analytes for onsite and offsite testing are summarized for surface material and sediment samples in Table 4. The Remedial Support surface material samples will be analyzed for three chemical constituents, gamma emitting radionuclides and XRF metals, and for seven chemical constituents, gamma emitting radionuclides and XRF metals for the sediment samples. The Final Status surface material/soil samples will be analyzed for three chemical constituents, gamma emitting radionuclides and XRF metals, and for nine chemical constituents, gamma emitting radionuclides and XRF metals for sediment samples.

B.2 Onsite Analytical Methods Requirements

The following Standard Operation Procedures (SOPs) are designed to provide the type and quality of field measurement and onsite analysis data that will meet respective DQOs for the project:

- AS-SOP 1 - Decontamination for Field Sampling
- AS-SOP 2 - Surface Material and Sediment Sampling
- AS-SOP 3 - Onsite Sample Processing
- AS-SOP 4 - Onsite Gamma Spectroscopy
- AS-SOP 5 - XRF Procedures
- AS-SOP 6 - Gamma surveys

These SOPs are included in Appendix 1 to this QAPP, and respective procedures will be followed for all onsite surveys, sampling and analysis.

B.2.1 Onsite Sampling Handling and Custody

Sample handling and custody requirements are detailed in the following sub-sections. Corresponding procedures are detailed in AS-SOP 2 and AS-SOP 3 (see Appendix 1).

Sample Labeling and Identification

All sample collection information will be thoroughly documented in a field logbook at the time of sample collection. This information is specified in Section A.6.1 and in AS-SOP 2 (Appendix 1). Sample labels will be supplied by the laboratory. Sample labels will be completed with an indelible, waterproof marker. All samples will be labeled with date, time, sampler's initials and the

sample ID number. The sample ID number corresponds to a specific location, and respective GPS coordinates will be recorded in the field logbook. In addition, sample labeling information will include sample depth, sample matrix, sampling method and sample type. The sample IDs will be in the form of:

AAAA/BBBB/CCC/ DDD /##

Where:

AAAA = sample location ID number;
BBBB = sample depth (if applicable);
CCC = sample matrix (SED = sediment; SOI = soil);
DDD = sampling method (DIS = discrete; COM = composite)
= sample type (01=primary, 02=duplicate).

Field Logbooks

Samples collected will be documented in the field logbooks as described in Section A.6.1 Field Operation Records. Upon delivery of field samples to the onsite soils lab for processing, the pages of the logbook corresponding to the samples being delivered will be electronically copied and printed out. These hardcopies will be used to enter all logbook sampling information into the sample login/tracking spreadsheet including sample ID, location (GPS coordinates), date and time of collection, sample matrix (soil or sediment), sample type (discrete or composite) and any notes regarding observations at the field sampling location. Once a field logbook has be completely filled with sampling information, the entire logbook will be scanned into an electronic PDF and both the hardcopy and electronic copies will be filed chronologically in the project records.

Sample Containers, Preservation and Holding Times

Proper sample preparation practices will be observed to minimize sample contamination and potential repeat analyses due to anomalous analytical results. Sample containers are described in Table 3. Sample containers will be labeled as previously described in Section B.2.1.

Sample holding times are established to minimize chemical changes in a sample prior to analysis and/or extraction. A holding time is defined as the maximum allowable time between sample collection and analysis and/or extraction, based on the nature of the analyte of interest and chemical stability factors. Samples scheduled for offsite analysis will be shipped to the offsite laboratory as soon as possible after collection, onsite processing, and onsite analysis in the soils lab. Holding times for the constituents for which samples will be analyzed are summarized in Table 3. Table 3 also provides volume, container-type, and preservation specifications for solid surface material or sediment samples.

Table 3: Sample Containers, Preservation, and Holding Time Requirements for Solid Samples

Sample Media	Analysis	Sampling Container	Preservation /Prep	Minimum Sample Mass*	Holding Time
Soil, Sediment	Total Cr, Mn, Se, U and V	Quart–size freezer Zip-lock bags, gamma counting cans, and XRF soil cups	No preservation needed	150-200 g dry	180 days
	Ra-226, Pb-210, U-234, U-238	Quart–size freezer Zip-lock bags, gamma counting cans	No preservation needed	150-200 g dry	**

*The minimum combined sample mass required for all analytes is 350 g dry for any single sample collected in the field
**No established recommended holding time; analyze as soon as feasible

Onsite Sample Processing

For the purposes of the Analytical Support and Verification Plan for Surface Materials and Sediments (Appendix S), onsite sample processing refers to the following functions of the onsite soils lab:

- Sample log-in and electronic data entry of all sample collection information.
- Sample preparation for onsite analysis.
- Data management (sample login/tracking and sample analysis spreadsheets).
- Sample management and storage.
- Sample shipping for the fraction of samples that are sent to an offsite commercial laboratory.

Onsite Sample Login

After collection, samples to be delivered to the onsite soils laboratory will be labeled as described in the previous discussions, and placed in insulated hard-walled cooler for delivery to the onsite soils lab. Upon delivery to the onsite lab, all sample collection information from the logbook and sample labels will be entered into an electronic sample login/tracking spreadsheet. Procedures for tracking samples through the onsite sample preparation and analysis process, along with shipping to an offsite lab (including chain-of-custody tracking), and onsite archiving of excess sample are provided in AS-SOP 3 (Appendix 1).

Onsite Sample Preparation

One of the most important aspects of laboratory analysis of soils or sediments is sample preparation (e.g. drying and homogenization). Once samples arrive at a laboratory, sample preparation is the greatest single source of potential error in the analytical results (EPA, 2004). For solid samples (soil and sediment), homogenization is particularly crucial to ensure that aliquots taken from the bulk sample for analysis are truly representative of the bulk sample. Thorough drying of the sample will reduce analytical error due to inaccuracies in measured dry weight, as well as potential analytical interferences due to variability in soil water content (e.g. photon attenuation during gamma spectroscopy).

Field samples will be prepared onsite as indicated in Standard Operating Procedure AS-SOP 3 (Appendix 1) prior to onsite analysis by gamma spectroscopy. For intrusive XRF samples, sample preparation procedures are provided in Standard Operating Procedure AS-SOP 5. For the fraction of samples that will be sent to an offsite lab for additional analysis, the offsite lab's normal analyte-specific procedures for sample preparation will be followed as usual. Commercial laboratories typically utilize specialized grinders and riffle splitters for homogenization, and large ovens for simultaneously drying multiple samples.

Chain of Custody for Offsite Analysis

After samples have been collected in the field, they will be immediately delivered to the onsite soils lab for processing in accordance with Section B.3.5 and AS-SOP 3 (Appendix 1). Because no shipping is involved for field sampling and onsite analysis, chain of custody (COC) forms are unnecessary for the transfer of samples from the field to the onsite lab. After processing in the onsite lab, all samples will be analyzed onsite in accordance with AS-SOP 4 (gamma spectroscopy) and where called for by the Plan, Field Supervisor and/or Field Program Director, XRF analysis will also be performed in accordance with AS-SOP 5 (XRF analysis).

All samples that will be subsequently shipped to an offsite laboratory will be maintained under strict chain-of-custody protocols. Onsite soils lab personnel will be responsible for all offsite sampling shipping, and will complete a COC form for each shipping container (i.e., cooler, ice chest or other container) of samples to be delivered to the offsite laboratory for analysis. The COC will be signed by COC originator (onsite lab staff) when relinquishing the samples to anyone else. The COC for a shipping container will list only those samples in that shipping container. Each sample container will be carefully packaged in a shipping container, typically a cooler. Information contained on both the COC form and its carbonless duplicate will include the following:

- Project number
- Date and time of collection
- Sample identification number
- Sample matrix type
- Analyses requested (including method specifications as appropriate)
- Number of containers/bags for each sample (e.g. 1 sealed counting can and 1 XRF cup)
- COC originator's signature and affiliation
- Signature of persons relinquishing custody, dates, and times
- Signature of persons accepting custody, dates, and times
- Method of shipment
- Shipping air bill number (if the samples are shipped)
- Any additional instructions to the laboratory

The COC form originator will cross out any blank spaces on the COC below the last sample number listed. COC forms will be placed in a re-closeable freezer-type plastic storage bag and taped to the inside lid of the cooler. Included with the COC/analytical request form will be an

attached set of special instructions to the lab regarding sample handling and analysis requirements (see Section B.2.1.8 and AS-SOP 3 for further details).

For shipping the offsite laboratory, the coolers will be taped shut and COC seals will be attached to the outside of the cooler to ensure that the cooler cannot be opened without breaking the seal. Custody seals will be signed and dated by the sample custodian prior to shipment. If the custody seal is broken, the offsite laboratory will immediately notify Project QAM. Samples will be shipped under standard delivery unless there is a potential to exceed sample hold time (the only applicable hold time is 6 months for metals in sediments).

The onsite personnel whose signature appears on the COC is responsible for the custody of the samples from the time of sample collection until custody of the samples is transferred to a designated laboratory, a courier, or to another project employee for the purpose of transporting the sample to the designated laboratory. The sample is considered to be in custody when the sample is: (1) in the direct possession of the sample custodian; (2) in plain view of the sample custodian; or (3) is securely locked in a restricted-access area by the sample custodian.

Custody is transferred when both parties to the transfer complete the portion of the COC under "Relinquished by" and "Received by." Signatures, printed names, company names, dates and times are required. Upon transfer of custody, the sampling personnel who relinquished the samples will retain the duplicate (yellow) copy of the COC. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the COC. Copies, receipts and carbons of Bills of Lading will be retained as part of the permanent documentation in the project file. It is not necessary for courier personnel to sign the COC. These procedures will be followed for all samples delivered to offsite laboratories.

In addition to the above protocols for COC sample tracking, the onsite sample login/tracking spreadsheet and sample analysis spreadsheet will be updated to indicate which samples are being shipped to the offsite lab for analysis (see AS-SOP 3).

Corrections to Documentation

Any mistakes made in the field logbooks or chain-of-custody forms should be crossed out with a single line. Corrections should follow and include a brief explanation, if applicable.

B.3 Offsite Analytical Methods Requirements

Table 4 includes chemical and radiological analysis, methods and detection limits for the soil and sediment samples to be collected. Table3 provides holding times, preservation guidelines, and required sample amounts. A copy of the applicable analysis table and special handling and analysis instructions (Section B.2.1.8 and AS-SOP 3) will be sent with each associated batch of samples submitted to the laboratory. A copy of this QAPP will be submitted to the laboratory before the first batch of samples is received. Procedures for laboratory analysis, with any modifications, should be further documented in the laboratory SOPs, which are maintained at the laboratory and are listed in the laboratory's Quality Assurance Plans (QAP). Analytical Method QC specifications including frequency, acceptance criteria and corrective actions for each

individual method are detailed in Table 5. The QAPs for the approved laboratories are included in Appendix 3. The laboratory designated for offsite analytical support for the project must be accredited under the National Environmental Laboratory Accreditation Program (NELAP). The approved laboratory designated for offsite analysis will be selected from one or more of the following listings. Respective current NELAP certifications are included in Appendix 4.

Laboratory	Proposed Analyses Capabilities
ACZ Laboratories Inc. 2773 Downhill Drive Steamboat Springs, CO 80487 800-334-5493	All metals and general chemistry
Energy Laboratories Inc. 2393 Salt Creek Highway Casper, WY 82602 888-235-0515	All metals, general chemistry, and radiological analyses
Anatek Labs Inc. 504 E. Sprague Ste. D Spokane, WA 99202 509-838-3999	All metals and general chemistry

B.3.1 Offsite Sample Handling and Custody

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

Upon receipt by the laboratory, the samples will be inspected for sample integrity and preservation. The COC will be reviewed to verify completeness. Any discrepancies between the COC and sample labels and any problems noted upon sample receipt will be communicated immediately to the Project QAM. The laboratory will store the samples in a specially designated area which is clean and maintained for this purpose. The laboratory will be responsible for following their internal custody procedures from the time of sample receipt until sample disposal. Coolers with samples are received and processed into the laboratory using the SOP from the selected lab which is maintained on file at the facility. A Sample Receipt Checklist should be generated providing documented details of the sample receipt.

B.3.2 Special Sample Handling/Analysis Protocols for the Offsite Laboratory

For samples sent offsite for analysis, there are special requirements regarding the protocols to be followed by the offsite laboratory for sample handling and analysis. Clear instructions must be provided with the COC/analyte request forms when shipping samples. These special requirements and protocols, along with the procedures that onsite soils lab personnel must follow to ensure proper implementation, are as follows:

Surface Material Samples

1. Only the canned and sealed portion of each sample as previously analyzed onsite for Ra-226 will be shipped and analyzed at the offsite lab (archived excess sample will remain archived onsite). These canned samples must remain sealed for shipment to the offsite lab.
2. On the COC/analyte request form, the notes section must reference and include an attached set of special instructions that clearly indicate that these samples are not to be unsealed until after they have been analyzed by gamma spectroscopy for Ra-226 (Method 901.1) at least 21 days after the date the can was originally sealed in the onsite lab (as indicated on the lid of each sealed sample can), and that the dry sample weight indicated on the lid should be used to calculate the "full radon ingrowth" Ra-226 concentration. The laboratory must be clearly informed and instructed in this special protocol.
3. The protocol in item 2 above is crucial to the success of the program and prior to sample shipping the offsite lab should be contacted directly and notified in advance of these special requirements and instructions in order to help avoid confusion or mistakes.
4. After the lab has performed full-ingrowth analysis for Ra-226, the samples will be unsealed at the offsite lab, re-homogenized and analyzed for uranium and Pb-210 according to the methods specified in Appendix S and in this QAPP.
5. In cases where XRF analysis for uranium is performed onsite for a surface material sample and the XRF soil cup is sent in for confirmatory analysis, the XRF aliquot will be labeled with the same sample ID number, but with a qualifying "(Aliquot B)" designation.

Sediment Samples

1. For sediment samples, both the canned/sealed portion of each sample as previously analyzed onsite by gamma spectroscopy, as well as the smaller plastic XRF cup as previously analyzed by XRF, will be shipped together and analyzed concurrently at the offsite lab. The XRF aliquot will be labeled with the same sample ID number, but with a qualifying "(Aliquot B)" designation. Again, any additional (excess) sample previously stored onsite in original plastic sample collection baggies will remain archived onsite.
2. The above protocols for Ra-226 analysis in canned/sealed surface material samples also apply to sediment samples.
3. For sediment samples in plastic XRF cups, the lab will analyze the sample for all analytes reflected in ROD cleanup levels for sediments, with the exception of Ra-226 which will be analyzed from the canned/sealed sample aliquot per item 2 above, and Pb-210 which will be analyzed from the canned/sealed sample aliquot per item 4 above. Aside from Ra-226, all other parameters will be prepared and analyzed according to the normal laboratory protocols for the methods specified in Appendix S and in this QAPP.

Note: the above special sample handling and analysis protocols for the offsite laboratory will result in duplicate sample analysis of uranium for aliquots from canned samples and paired XRF cup

samples. This approach could provide useful information on any differences in concentrations due to differences in particle size distribution as a result of sample preparation differences (XRF samples will be sieved through a very fine 60-mesh sieve, while gamma spectroscopy samples will represent a much coarser particle size distribution).

Table 4: Soil and Sediment Laboratory Parameters, Analysis Methods, and Method Detection Limits

Sample Media	Analyte	Analytical Method	Target Method Detection Limit (mg/L)	ROD Cleanup Level
Soil	Uranium, total	EPA 3050/EPA 6020	0.5 mg/Kg	43 mg/Kg
	Lead 210	EPA 3050/EPA 909.0	5.0 pCi/g	7.5 pCi/g
	Radium 226	EPA 901.1	1.0 pCi/g	4.7 pCi/g
	Gamma Emitting Radionuclides	EPA 901.1	<1.0 pCi/g	
	Radium 226 (onsite)	NaI-based Gamma Spectroscopy	1.0 pCi/g	4.7 pCi/g
	XRF Metals	EPA Method 6200	TBD*	
Sediment	Chromium, total	EPA 3050/EPA 6020	0.5 mg/Kg	43.4 mg/Kg
	Manganese, total	EPA 3050/EPA 6020	0.5 mg/Kg	1179 mg/Kg
	Selenium, total	EPA 3050/EPA 6020	0.5 mg/Kg	1.7 mg/Kg
	Uranium, total	EPA 3050/EPA 6020	0.5 mg/Kg	93.2 mg/Kg
	Vanadium, total	EPA 3050/EPA 6020	0.5 mg/Kg	41 mg/Kg
	Lead 210	EPA 3050/EPA 909.0	5.0 pCi/g	20 pCi/g
	Radium 226	EPA 901.1	1.0 pCi/g	13 pCi/g
	Uranium 234	EPA 3050/EPA 908.0	0.4 pCi/g	41 pCi/g
	Uranium 238	EPA 3050/EPA 908.0	0.4 pCi/g	31 pCi/g
	Gamma Emitting Radionuclides	EPA 901.1	<1.0 pCi/g	
	XRF Metals	EPA Method 6200	TBD*	

*TBD = To be determined. Final detection limits for XRF metals will be calculated once the correlations between XRF measurements and laboratory confirmation samples are determined.

Table 5: Analytical Method QC Specifications

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW6020 (ICP-MS Metals)	MS tuning sample	Prior to initial calibration and calibration verification	SW6020 paragraph 5.8	Retune instrument then reanalyze tuning solution
	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	If more than one standard is used, correlation coefficient must be ≥ 0.995	If applicable, correct problem and repeat initial calibration
	Calibration blank	Before beginning a sample run, after every 10 samples and at end of the analysis sequence	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples
	Initial calibration verification (Second source standard)	After initial calibration before beginning a sample run –at a concentration other than used for calibration	All analytes within ±10% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within ±10% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration
	Low level calibration check standard (at or below RL)	Once per analytical batch prior to sample analysis unless multi-point (3+) calibration with low std at or below RL is performed	All analyte(s) with ± 50% of expected value	Correct problem then reanalyze
	Linear range calibration (high) check standard	Every three months	Analyte within ± 10% of expected value	Correct problem then reanalyze or re-set linear range
	M6020: Method blank; M200.8: Lab reagent blank (LRB)	One per analytical batch	No analytes detected ≥ RL	Correct problem reprep and analyze method blank and all samples processed with the contaminated blank
	Interference check solutions (ICS-A and ICS-AB)	At the beginning and end of an analytical run or once during an 12 hour period, whichever is more frequent	ICS-A: All non-spiked analytes < RL unless they are a verified trace impurity from one of the spiked analytes. ICS-AB: Within ±20% of true value	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples
	M6020: Lab Control Sample (LCS); M200.8: Lab fortified blank (LFB)	One per analytical batch	QC acceptance criteria, to be provided by lab	Correct problem then reanalyze; If still out, reprep and reanalyze the LCS and all samples in the affected batch
	Dilution test	Each matrix in a analytical batch (only applicable for analytes with concentrations >100X MDL)	Fivefold (1+4) dilution must agree within ±10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails, or if an analyte's concentration for all samples in a batch is less than 100X MDL	Recovery within 75-125% of expected results	Dilute the sample; reanalyze post digestion spike addition
	M6020: Matrix Spike (MS)/MSD; M200.8: Lab fortified matrix (LFM)	One MS/MSD or LFM/LFMD per every 20 project samples/ matrix	QC acceptance criteria, to be provided by lab	none
	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, to be reported by lab	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria
	Internal Standards (ISs)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in method SW6020, section 8.3
	IDL study	Every three months	Detection limits established shall be ≤ ½ x RLs	none
	MDL study	Every 12 months		
	Results reported between MDL and RL	none	none	none

Table 5: Analytical Method QC Specifications (Cont.)

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW6010 (ICP Metals)	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	If more than one standard is used, correlation coefficient must be ≥ 0.995	If applicable, correct problem and repeat initial calibration
	Initial calibration verification (second source)	Daily after initial calibration	All analytes within ±10% of expected value	Correct problem then repeat initial calibration
	Calibration verification (Instrument Check Standard – ICB, CCB)	After every 10 samples and at the end of the analysis sequence	All analyte(s) within ±10% of expected value and RSD of replicate integrations <5%	Repeat calibration and reanalyze all samples since last successful calibration
	Calibration blank	After every calibration verification	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples
	Low level calibration check standard (at or below RL)	Once per analytical batch prior to sample analysis unless multi-point (3+) calibration with low std at or below RL is performed	All analyte(s) with ± 50% of expected value	Correct problem then reanalyze
	Linear range calibration (high) check standard	Every three months	Analyte within ± 10% of expected value	Correct problem then reanalyze or re-set linear range
	M6010: Method blank; M200.7: Lab reagent blank (LRB)	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	At the beginning of an analytical run	Within ±20% of expected value	Terminate analysis; correct problem; reanalyze ICS; reanalyze all affected samples
	M6010: Lab Control Sample (LCS); M200.7: Lab fortified blank (LFB)	One per analytical batch	QC acceptance to be provided by lab criteria,	Correct problem then reanalyze; If still out, reprep and reanalyze the LCS and all samples in the affected batch
	Serial Dilution test	Each new sample matrix, at least once per analytical batch (only applicable for analytes with concentrations >50X MDL)	Fivefold (1+4) dilution must agree within ±10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails, or if an analyte's concentration for all samples in a batch is less than 50X MDL	Recovery within 75-125% of expected results	Check for instrumental problem then reanalyze post digestion spike addition if appropriate
	M6010: Matrix Spike (MS)/MSD; M200.7: Lab fortified matrix (LFM)	One MS/MSD or LFM/LFMD per every 20 project samples/ matrix	QC acceptance criteria, to be provided by lab	none
	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, to be provided by lab	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria
	MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs	none
	Results reported between MDL and RL	none	none	none

Table 5: Analytical Method QC Specifications (Cont.)

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Radiochemistry (M 901.1, 903.0, 908.0, 909.0)	Initial calibration	Prior to use if instrument –depending on type of instrumentation	Varied depending on instrument	Recount/recalibrate. Perform instrument maintenance or service instrument if needed.
	Tracer or carrier	Every sample when required by the method.	Appropriate counting statistics or measurement error if mass yield is determined.	Investigate for analyst error. Investigate use against analyte level present in samples and customer DQRs. Re-prepare and analyze affected samples. Discuss in narrative.
	Method (Prep) Blank	1 per batch (N/A for solid samples)	<Minimum Detectable Concentration (MDC), <5% sample isotope concentration or <5% decision level.	Investigate, evaluate against DQRs, correct, re-prepare and analyze as applicable.
	Laboratory Control Sample (LCS)	1 per batch	80% to 120%	Investigate and evaluate against DQRs. Correct. Re-prepare and analyze affected samples.
	Matrix Spike Sample (MS); MSD	1 per batch (N/A for Method 901.1)	75% to 125% or statistical	Investigate. If laboratory error, re-prepare and analyze. If matrix driven, evaluate against DQRs, notify client if still unacceptable. Discuss in narrative.
	Laboratory Duplicate Analysis	1 per batch	≤20% relative percent difference when the two results are >5 times the MDA or individual uncertainties are <20%. Or, alternately, the duplicates should agree within 2 standard deviations.	Evaluate. If lab error, re-prepare and analyze. If matrix driven, evaluate against DQRs, notify client if still unacceptable. Discuss in narrative.
	Minimum Detectable Concentration (MDC)	Determined for each sample based on count time and volume of sample used		
	MDL study	Once per 12 month period	Required detection limits (for drinking water) must be achieved (40 CFR 141.25)	Repeat if obvious problem occurs. Determine root causes. Repeat study.
	Counter control source	One per day as the system is used or per batch (alpha/beta and liquid scintillation). One per week or after analytical run, whichever is longer (gamma and alpha spectrometry)	Control limits: 3 sigma or ± 3%, whichever is greater. Monitor resolution, efficiency, and energy calibrations to procedure-specified limits.	Investigate. Recalibrate if necessary. Readjust if keV/channel drift occurs (gamma and alpha spectrometry).
	Instrument background (blank for liquid scintillation)	One per day as system is used (alpha beta and liquid scintillation). Minimum of every other week or after analytical run, whichever is longer (gamma spectrometry) Minimum of every 4 weeks or after analytical run, whichever is longer (alpha spectrometry)	Use background for results calculation. Monitor background for potential detector contamination.	Investigate as needed. Clean or replace parts as needed.

B.4 Quality Control Requirements

Quality control may be checked by collecting and analyzing field quality control (QC) samples and performing laboratory QC analyses. Both field and laboratory QC are necessary to control the sampling and analytical process, assess the accuracy and precision of results, and identify assignable causes for anomalous results. Project control limits for laboratory accuracy and precision measurements are listed in Table 2.

B.4.1 Field QC Procedures

To assess the measurement precision of field surveys (gamma and XRF), along with the analytical precision of sample analysis in the onsite laboratory, daily QC measurements will be performed. To evaluate data uncertainty due to variability in field sampling technique, and to provide indications of spatial heterogeneity in analyte concentrations on small spatial scales, field duplicate samples will be collected and analyzed. These protocols are described as follows:

QC for Gamma Survey Measurements

All gamma instruments used for remedial support and final status at the Site will include instrument QC measurements performed at a designated indoor location. The purpose is to quantify under controlled conditions the consistency of readings between detectors, along with temporal variability for individual detectors. Prior to initiation of field work, each detector to be used that day will be subject to QC measurements. For each detector, the mean of 10-20 individual readings of ambient background radiation, as well as radiation from a gamma check-source (e.g. a sealed Cs-137 button source), will be determined under identical indoor counting geometries and recorded on instrument QC log sheets, along with the detector ID number and date of the measurement. Under these circumstances, all data from any given set of properly calibrated and correctly functioning NaI scanning detectors should approximate a normal (Gaussian) distribution (Figure 3).

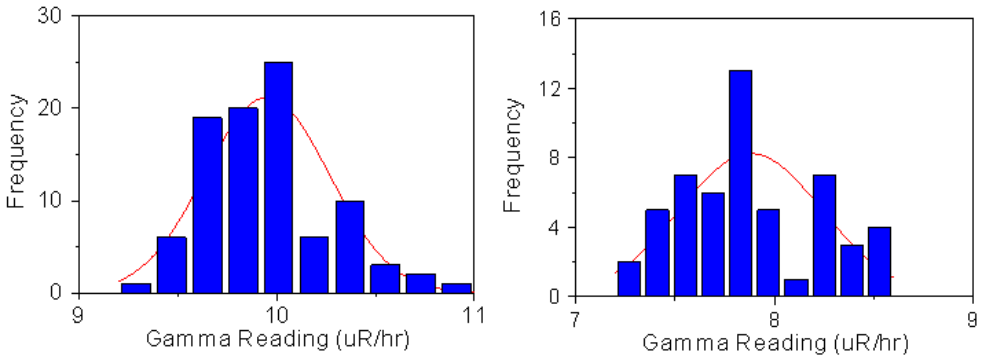


Figure 3: Example frequency histograms for several series of QC measurements from different NaI detector sets used for two separate gamma survey projects. Each series was taken indoors under controlled measurement geometries. The red lines represent theoretical normal distributions.

For normally distributed data, over 99% of measurements are expected to fall within ± 3 standard deviations from the mean. Any instrument with a QC measurement result falling outside ± 3 standard deviations from the mean of all previous QC measurements warrants investigation. If a detector exceeds the established control limits on both background and check-source control QC log sheets, it must be replaced with a factory-calibrated spare detector and sent back to the manufacturer for repair and recalibration.

Periodically, the Field Supervisor will collect all instrument QC data and plot this information on instrument control charts, the limits of which will also be periodically updated throughout the project based on cumulative data from all daily QC measurements. This protocol will allow periodic visual assessment of instrument performance over time and will also allow quantitative assessment of natural temporal variability in ambient gamma radiation due to fluctuations in atmospheric or other conditions (e.g. changes in barometric pressure, soil moisture, indoor radon decay products, etc.). The combined variability in QC data provides a means for estimating total data uncertainty from variability in both instrument response and natural fluctuations in ambient gamma exposure rates. An example of a background instrument quality control chart for various NaI detectors over time is shown in Figure 4.

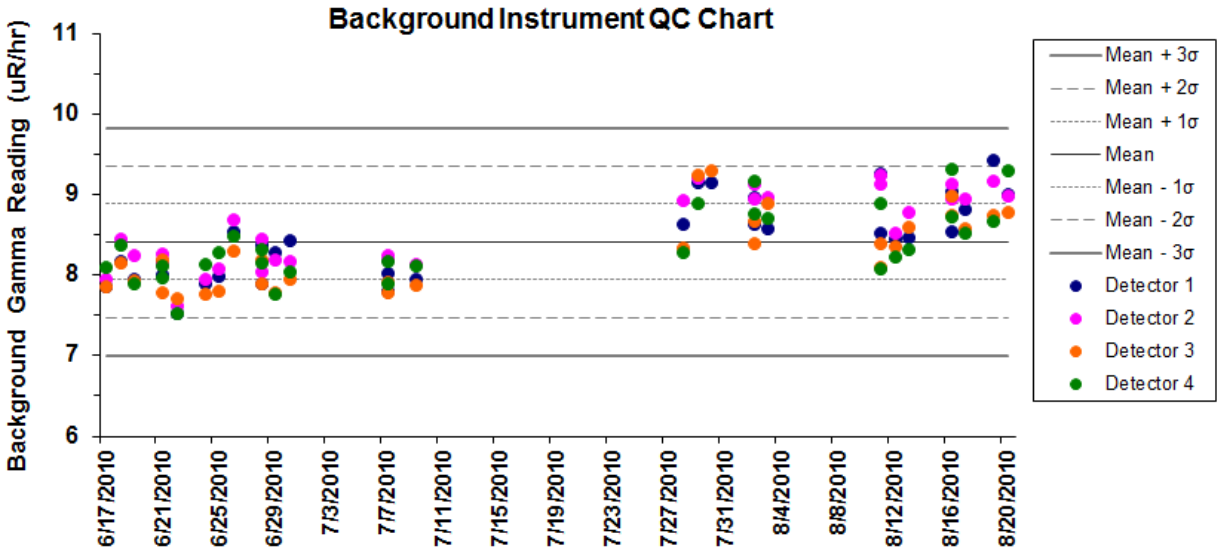


Figure 4: Example instrument background quality control chart for multiple NaI detectors.

For all recorded gamma surveys at the Site (GPS-based scanning) the actual field performance of each scanning system to be used will be tested onsite each day by obtaining average scan readings along a designated "field strip." The field strip will be in the range of 20 meters in length, and ideally will be located in an area that has relatively uniform gamma readings on all sides. Field strip scans will be conducted before each day's scanning activities in order to evaluate overall system performance and to provide quantitative information regarding the combined amount of data variability attributable to variations in instrument readings (detectors/scanning systems) and temporal variations in ambient gamma radiation in the field due to changes in atmospheric or other environmental conditions (e.g. barometric pressure, soil moisture, radon decay product concentrations in air near the ground surface). Field strip data for each scanning

system must remain within acceptable QC limits throughout the project (Figure 5). In cases where a scan system exhibits suspect performance, the system (or detector) in question will be not be used again until repaired or replaced with properly performing spare equipment (also subject to routine field strip QC measurements).

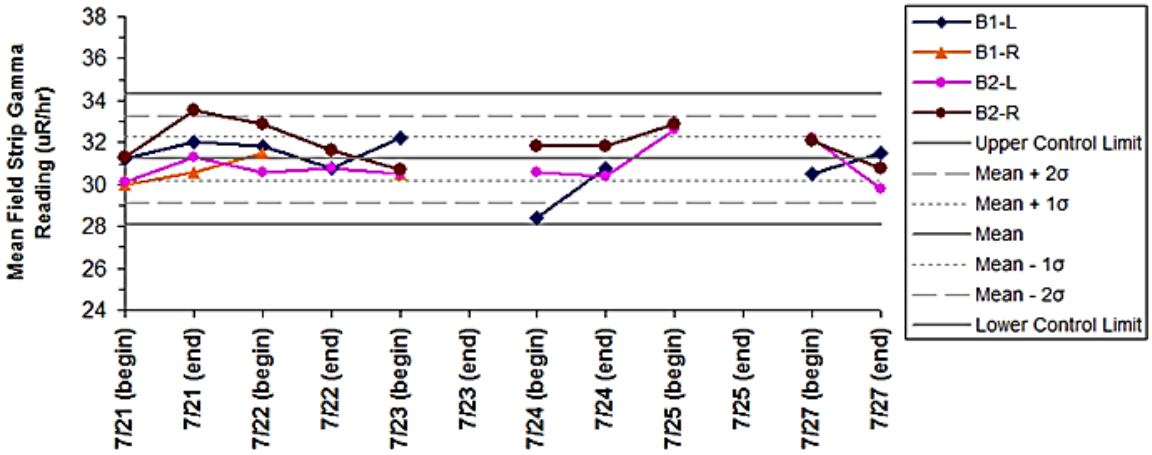


Figure 5: Example field strip control chart for multiple NaI detectors.

QC for Onsite Gamma Spectroscopy

Because NaI-based quantification of Ra-226 in soil/sediment samples will be based on system calibrations using site-specific soil Ra-226 calibration standards that have been analyzed with HPGe-based gamma spectroscopy at a commercial laboratory, the accuracy of onsite analysis of Ra-226 concentrations and associated data uncertainty will be linked to the commercial laboratory's accreditations and QC protocols. The offsite laboratory must be NELAP accredited, which requires rigorous quality assurance criteria and routine proficiency testing. The offsite laboratory must follow strict chain of custody protocols, use NIST certified standards for instrument calibrations, and perform measurements on certified (e.g. NIST traceable) reference material standards with each set of samples to provide information on measurement accuracy. The offsite lab must also perform duplicate analyses on 10% of all samples to provide information on HPGe measurement precision.

With respect to onsite soils lab QC measurements, a dedicated Cs-137 check source will be used to energy calibrate the NaI counting system at the beginning of each day and to monitor the system for spectral drift every 2-3 hours. Spectral drift can occur as a result of temperature changes in the counting room. Fine gain settings on the MCA will be adjusted as needed, and efforts will be made to keep the temperature in the counting room as consistent as possible. About 5% of samples analyzed onsite will be counted a second time as a laboratory duplicate analysis. Up to 10% of samples analyzed in the onsite soils lab will be sent to the offsite laboratory to verify and quantify the relative accuracy of soil Ra-226 concentrations as measured in the onsite soils lab. For final status survey samples, a field duplicate sample will be collected at about 5% of sampling locations (e.g. about 4 field duplicates for every 75 samples). All field duplicates will be

analyzed onsite, and at least 1 field duplicate will also be analyzed offsite, meaning that at least 1 of the 25 samples to be sent offsite from each survey unit must include a field duplicate (i.e. there will be a total of 26 samples from each survey unit to be analyzed offsite).

In addition to the above QC protocols, gamma counting system QC checks will be performed at the beginning of each day. This will include measurements of two of the Site-specific calibration standards used to develop the method calibration curve, one that is similar to typical "background" soil Ra-226 concentrations (e.g. 1-2 pCi/g), and one that represents a "source" concentration well above the cleanup criterion for Ra-226 (e.g. in the range of 15-20 pCi/g). These QC measurements will be recorded on system QC charts (Figure 6). Results greater than ± 3 standard deviations from the mean of all previously accepted QC chart measurements will indicate that the counting system is not working properly.

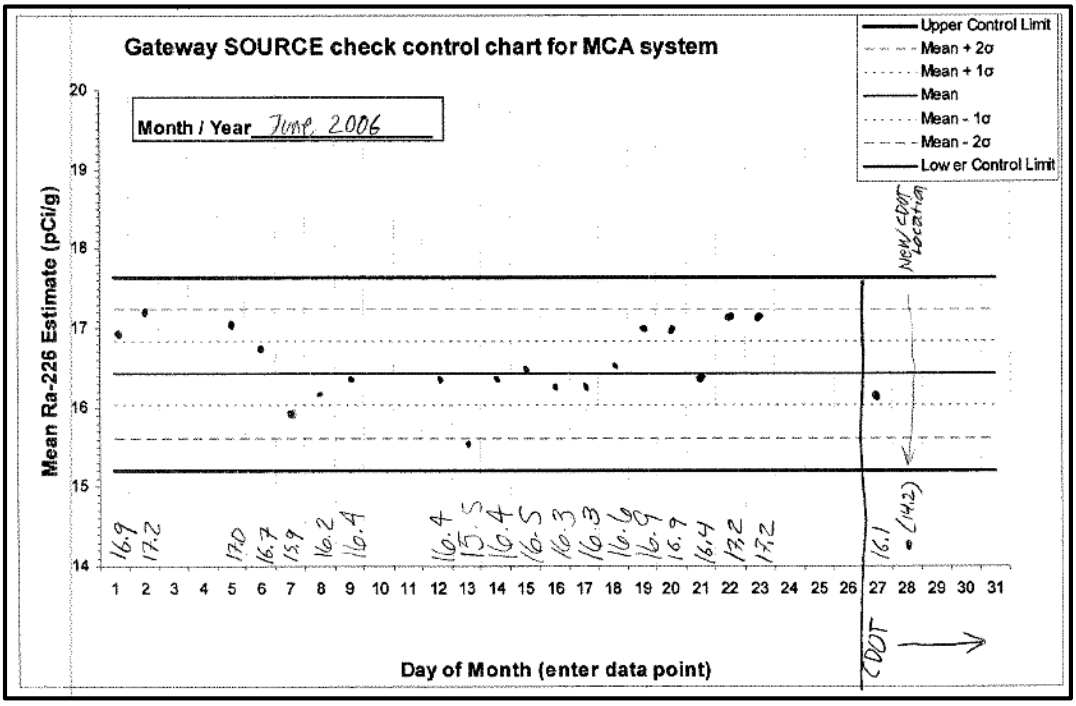


Figure 6: Example QC chart for soils lab measurements.

Determination of the optimal sample count time for onsite gamma spectroscopy measurements will be based on balancing the number of samples that can be analyzed per day against the need to achieve sufficient accuracy (i.e. optimization of spectral resolution, counting statistics, and system detection limits relative to the cleanup criterion). The minimum detectable concentration (MDC) for the system will be calculated as described in Principles of Radiological Health and Safety (Martin, 2003), based on measurements of site-specific calibration standards. The calculated MDC must be well below the ROD cleanup level for Ra-226 in surface materials (e.g. 1 pCi/g or less). If not, the sampling counting time will be increased until an acceptable MDC is achieved.

QC for XRF Analysis

The following QC samples will be run according to EPA Method 6200:

- Energy calibration check sample
- Instrument blank
- Method blank
- Calibration verification checks (NIST standard)
- Precision measurements
- Confirmation samples

The details of these QC samples and associated XRF measurements and respective performance and acceptance criteria are provided in Section A.4.1, along with standard operating procedure AS-SOP 5 and Method 6200 (see Appendix 1).

Field Duplicate Samples

A field duplicate is defined as a second sample (or measurement) from the same location, collected in immediate succession, using identical collection techniques. For final status survey samples, a field duplicate sample will be collected at about 5% of sampling locations (e.g. about 4 field duplicates for every 75 samples). All field duplicates will be analyzed onsite, and at least 1 field duplicate will also be analyzed offsite, meaning that at least 1 of the 25+ samples to be sent offsite from each survey unit must include a field duplicate (i.e. there will be a minimum total of 26+ samples from each survey unit to be analyzed offsite).

Field duplicate samples help to quantify small scale variability in concentrations at a given location, along with total propagated data uncertainty associated with variations in sampling technique, handling and sample processing, and analytical variability. In other words, field duplicates provide an indication of combined sampling and analytical measurement precision. Field duplicates will be analyzed for the same analytical parameters as the primary sample. There are no U.S. EPA criteria for evaluation of field duplicate sample comparability, however, the relative percent difference (RPD) between the original sample and field duplicate can be calculated for each parameter and compared to the precision goal. Field duplicate RPDs that are greater than the project-specified precision goal (± 50%) may be indicative of a high degree of small-scale spatial variability in concentrations at the sample location.

B.4.2 Laboratory QC Procedures

The appropriate type and frequency of laboratory QC samples will be dependent on the sample type/media, analytical methods, and the laboratory's SOPs. With each QC batch for sample analysis, the following laboratory QC samples will be analyzed in addition to the calibration samples.

Matrix Spike Samples

Laboratory matrix spike samples are used to evaluate potential matrix effects on sample analysis for inorganic parameters. Percent recoveries of target analytes from matrix spike samples should fall within control limits of 70% to 130%for solid samples. However, if other QA/QC results are acceptable, there is no requirement to qualify sample results. Matrix interference and other effects may cause low or high percent recoveries in investigative samples; matrix effects may be noted at the same time that recoveries from laboratory control samples indicate acceptable method performance.

Laboratory Control Samples

EPA (2004) guidelines specify that percent recoveries of most metals from aqueous laboratory control samples should fall within control limits of 80% to 120%. An appropriate laboratory control sample will be used by the laboratory for soil/sediment sample matrices.

Analytical Duplicate Samples

Based on EPA guidelines, laboratory replicate samples and the samples from which they are split (the field samples) should have relative percent differences (RPDs) whose absolute values do not exceed 35% (for solid samples) in cases where both sample values are greater than or equal to five times the reporting limit. The RPD is defined by the following equation:

$$RPD = \frac{\text{sample} - \text{duplicate values}}{\left(\dfrac{\text{sample} + \text{duplicate values}}{2}\right)} \times 100\%$$

If one or both values are less than five times the reporting limit, the difference between the primary and replicate values should not exceed 2x the reporting limit for solid samples.

The precision measurement for duplicate samples for radiochemistry analyses will include the Replicate Error Ratio (RER). The laboratory goal for the RER is < 2.0. The RER is defined by the following equation:

$$RER = \frac{|Sx - Dup|}{\sqrt{(Sx_{error})^2 + (Dup_{error})^2}}$$

Where: Sx = sample concentration in pCi/L

Sx_{error} = sample counting error (in pCi/L) at the 95% confidence level.

Dup = duplicate concentration in pCi/L

Dup_{error} = duplicate counting error (in pCi/L) at the 95% confidence level.

Frequency

Laboratory QA/QC samples method blank, matrix spike, and laboratory control samples should be run in a QC batch of one each per 20 field samples. If less than 20 field samples are submitted, then one set of these three QA/QC samples should be run per batch. Analytical duplicates will

be done at a frequency of one per sample media for all analytes, when sufficient sample material is available.

B.5 Instrument/Equipment Testing, Inspection and Maintenance Requirements

Field equipment will be cleaned and safely stored in between each use, and routine maintenance recommended by the equipment manufacturer will also be performed. Equipment will be inspected and the calibration checked (if applicable) before it is transported to a field setting for use. Preventative maintenance of field equipment will include routine inspection and either calibration or testing as specified in the relevant SOP or manufacturer's instructions. Laboratory preventative maintenance will include routine equipment inspection and calibration at the beginning of each day or each analytical batch, per the laboratory's internal SOPs and method requirements.

B.5.1 Field Instruments and Equipment

Equipment will be inspected before use and field instruments that fail calibration requirements or QC measurement control limits will be tagged as "nonfunctional" or "defective" and returned to the manufacturer or other supplier for re-calibration, repair or replacement. Field equipment that is worn or not functioning will be replaced immediately.

B.5.2 Laboratory Instruments

Instruments used by the laboratory will be maintained in accordance with the laboratory's QAP and method requirements. The laboratory will keep maintenance records and make them available for review, if requested, during laboratory audits.

B.5.3 Acceptance Requirements for Supplies and Consumables

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

B.6 Instrument Calibration and Frequency

B.6.1 Field Calibration Procedures

Each day, equipment used to gather, generate or measure environmental data will either be tested against QC measurement control limits or calibrated each day (consistent with manufacturer specifications) as applicable prior to use in the field in order to ensure accuracy and reproducibility of results. Field sampling and measurement equipment will be examined to certify that it is in good operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that maintenance requirements are being met. In the event that a field instrument does not meet QC measurement control limits, or cannot be calibrated to meet the manufacturer's specifications, it will be tagged "defective" and returned to the manufacturer or other supplier for service or replacement.

B.6.2 Laboratory Calibration Procedures

Instruments used by the laboratory will be calibrated in accordance with the laboratory's Quality Assurance Plan (QAP), method SOPs, and any specified EPA-method requirements. When laboratory measurement instruments do not meet the calibration criteria of the QAP, Method SOP or EPA method, then the instrument will not be used for analysis of samples submitted under this project QAPP. Calibration records should be accessible and demonstration of acceptable calibration results if requested by project personnel. Maintenance records should be available for inspection.

B.7 Data Acquisition Requirements

The offsite laboratory reporting of analysis results will include the following information. This information will be presented as an analytical hardcopy report in PDF file format and in addition, the data will also be reported as an electronic data deliverable.

- Sample identification number (see Section B.2.1)
- Sample collection date
- Analytes, concentrations and units
- Analysis date
- Analysis method used
- Laboratory qualifiers and definitions
- Results between method detection limits (MDL) and reporting limit (RL)
- Percent solids on a dry weight basis for solid samples

The laboratory QC summary (Level IV Data Package) should include:

- Laboratory case narrative summarizing any method deviations or analysis problems
- Method detection limits (MDL) and sample dilution information
- Laboratory quantification limits (PQL, RL)
- Method blank data
- Precision (duplicate) data
- Matrix spike data
- Laboratory control sample data
- Calibration data
- Raw data
- Run logs – to include all calibration samples, samples, and QC samples mentioned in Table 5
- Sample log-in information
- Copies of complete COCs

In addition to the QC data requirements listed above for all methods, laboratory QC reporting should include the following QC data specific to method:

ICP (M6010, 200.7) and ICP-MS (M6020, M200.8):

- Calibration data - to include initial calibration, calibration blanks, initial calibration verification, continuing calibration verification, low-level calibration check standard and linear range calibration check standard.
- Dilution Test
- Post-digestion spike addition
- Internal standards

ICP-MS (M6020, M200.8):

- Mass Spec tuning sample

Radiochemistry (M901.1, 903.0, 908.0 909.0)

- Aliquant size
- Tracking (link to instrument, run logs)
- Traceability of reference and standard materials
- Linkage of QC samples to project samples
- Chemical separation (yield)
- Efficiency, calibration curves and instrument background
- Spectral resolution
- Counts and duration of counts

Data reporting packages will be prepared by the Laboratory Project Manager and will be submitted to the Supervising Contractor and the Project QAM.

B.8 Data Management

Once the laboratory data has been validated and qualifications noted, the analytical data and qualifiers will be entered into the project database along with field measurements and sample information (Location ID#, sample media, sample location, and date).

C. Assessment/Oversight

C.1 Assessment and Response Actions

C.1.1 Field Measurement Data

Both quantitative and qualitative field data will be obtained for use in the project. For quantitative field measurements, accuracy is usually confirmed through calibration of measurement equipment. Measurement precision may be evaluated through replicate QC measurements. Field completeness is defined as a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions, as described in Section A.4.2.3.

Field measurement data will be reviewed daily before its incorporation into the project database. Questionable results will be addressed through a timely and appropriate corrective action (Section C.1.4). Once field data have been approved for incorporation into the project database they will also be considered acceptable for use in the project.

C.1.2 Laboratory Data

As discussed in previous sections of this QAPP, the accuracy, precision, completeness, and representativeness of analytical data will be described relative to the project's control limits. The data quality review will be documented in reports to the Supervising Contractor and any qualification of the data resulting from that review will be attached to results that are incorporated into the project database so that all data users are aware of data quality for individual results.

C.1.3 Performance and System Audits

Performance and system audits of both field and laboratory activities may be conducted to verify that sampling and analyses are performed in accordance with the procedures established in the Work Plan and QAPP. These audits are optional and not a requirement. The audits of field and laboratory activities include two independent parts: internal and external audits. Findings of these audits will be summarized in an audit report that is given to the Supervising Contractor and appropriate supervisor in charge of the audited activities (Field Program Director, Field Supervisor or Lab Manager). The Supervising Contractor will submit a reply addressing each finding cited in the report, the corrective action (if necessary) to be taken, and a schedule for implementation. Corrective action procedures are described in Section C.1.4.

Internal Field Performance and System Audits

<u>Internal Field Audit Responsibilities</u>

Internal audits of field activities, including sampling and field measurements, may be conducted by either the Field Supervisor or the QAM. Internal field audits will verify that established procedures are being followed.

Internal Field Audit Procedures

The performance and system audits will include examination of field sampling records, field instrument operating and QC records, sample collection and processing, packaging and shipping, and data handling in compliance with the established procedures and SOPs, maintenance of QA procedures, and chain-of-custody, etc. outlined in this QAPP. Follow-up audits may be conducted to correct deficiencies and to verify that QA procedures are maintained throughout the investigation. Follow-up audits will involve review of field measurement records, instrument calibration records, and sample documentation.

External Field Performance and System Audits

External field audits may be conducted by an outside regulatory agency (e.g., EPA). The external field audit process can include (but not limited to): sampling equipment decontamination procedures, sampling and sample processing procedures, examination of field surveying, sampling and safety plans, preparation for shipment, as along with field screening practices, and duplicate sample collection and analysis.

The external audit findings will be reported immediately to the Supervising Contractor who will be responsible for implementing the appropriate corrective actions if any are needed.

Internal Laboratory Performance and System Audits

Internal Laboratory Audit Responsibilities

Internal laboratory audits may be conducted by the laboratory QA Officer. The results of each performance audit will be reported to laboratory management. All performance audit results identified as unacceptable must be investigated. It is recommended that any results flagged as exceeding the warning limits, but within the control limits for the study shall also be reviewed. The findings of the investigation and corrective action will be documented. This documentation for all internal performance audits shall be provided to the agency or client supplying the audit, as well as being included in the QA report to the Supervising Contractor.

Internal Laboratory Procedures

The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The laboratory QA Officer will evaluate the analytical results of these blind performances samples to ensure the laboratory maintains acceptable QC performance.

The internal system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc., in accordance to the laboratory's QAP.

C.1.4 Corrective Actions

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or poor QC performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. Proposed corrective actions will be documented as well as the steps taken

to implement the corrective action. Corrective action should only be implemented after approval by the Supervising Contractor. If immediate corrective action is required, approvals secured by telephone from the Supervising Contractor should be documented.

Nonconforming equipment, items, activities, conditions, and unusual incidents that could affect data quality and attainment of the project's quality objectives will be identified, controlled, and reported in a timely manner. For the purpose of this QAPP, a nonconformance is defined as a malfunction, failure, deficiency, or deviation that renders the quality of an item unacceptable or indeterminate in meeting the project's data quality objectives. If the l results from field QC measurements or laboratory analytical QC samples fall outside of the measurement performance criteria, corrective actions should be initiated immediately by the field personnel or laboratory accordingly. If the situation that caused the nonconformance cannot be effectively resolved and continues to occur or is expected to occur, then the field personnel or laboratory will immediately contact the Project QAM and request instructions regarding how to proceed with sample analyses. Completion of any corrective action should be evidenced by data once again falling within prescribed measurement performance criteria. If an error in field measurement equipment, measurement technique, laboratory protocols, or sample collection and handling procedures cannot be found, the results will be reviewed by the Project QAM and Supervising Contractor to assess whether repeat measurements, reanalysis or re-sampling is required.

The need for corrective action may be identified during either data validation or data assessment. Potential types of corrective action may include resampling or reanalysis of samples. These actions are dependent upon the ability to mobilize the field team and whether the data to be collected are necessary to meet the required QA objectives. If the Field Program Director, Field Supervisor or Project QAM identifies a corrective action situation, it is the Supervising Contractor who will be responsible for approving the implementation of corrective action. All corrective actions of this type will be documented by the Field Program Director, Field Supervisor or Project QAM as appropriate.

Any corrective actions taken will be documented in writing by either the Laboratory QA Manager or the Project QAM and reported to the Supervising Contractor. Corrective action records (Appendix B) will be included in the project's files.

C.2 Quality Assurance Reports to Management

Periodic QA reports will be submitted to the Supervising Contractor from the Field Program Director and Project QAM to provide ongoing evaluation of onsite and offsite data quality. Reports will include sections that summarize the QC data collected during the program and provide a summary of data evaluation/validation results. A discussion of data usability relative to the project's quality objectives should also be included in the reports. Any anomalies or departures from the assumptions established in the planning phase of data collection will be identified.

C.2.1 Data Validation Reports

A data validation report will be issued to the Supervising Contractor from the Field Program Director and Project QAM summarizing the data validation for onsite survey data, onsite soils lab data, and offsite laboratory analysis reports as described in Section D.2. The report will summarize the data quality and include a list of any qualifications of data resulting from the data evaluation. The reports will be submitted after each field survey or sampling event.

D. Data Validation and Usability

D.1 Data Review, Validation and Verification Requirements

Field measurement values are generally reported directly in the units of final use in the field logbook without need for additional calculations (e.g., field gamma survey or onsite gamma spectroscopy QC measurements). The field data will be reviewed daily by the Field Supervisor to identify anomalous data and transcriptional and/or computational errors. Corrective actions will be initiated as appropriate; these actions may consist of re-measuring a particular parameter, collecting a new sample, or other applicable corrective action measures. Reviewed field data will be entered into the project database promptly upon completion of the review.

The offsite laboratory's calculations and data review will be performed in accordance with procedures prescribed in their own QAP and the referenced analytical method.

D.2 Validation and Verification Methods

Validation means those processes taken independently of the data-generation processes to determine the usability of data for its intended use(s). All data obtained from field and laboratory measurements will be reviewed and verified for conformance to project requirements, and then validated against the data quality objectives that are listed in Section A.4.

D.2.1 Validation/Verification of Field and Onsite Analysis Data

Gamma Survey Data

All recorded gamma survey data will be evaluated by the Field Program Director in terms of instrument calibration records and field QC measurement records to determine whether these indicators of accuracy and precision (see Section A.4.1) meet the project DQOs in terms of data usability. Descriptive statistics for raw survey data along with preliminary maps of raw data will be analyzed in a context of potential outlier readings that can on rare occasion occur due to small electronic interruptions (e.g. under jarring scan conditions or when resuming data logging after a pause in scanning). Such data are usually manifest as a single data point that is orders of magnitude higher than adjacent data. Clear evidence of invalid data points will be documented and these data will be eliminated from the usable data set. Gamma scan data will also be converted estimates of soil Ra-226 concentrations (based on the gamma/Ra-226 correlation) and this conversion will be audited to verify that the regression equation used for the conversion corresponds to the period in which the gamma survey data were collected.

The results of this data validation/verification assessment will be forwarded to the Project QAM for independent review. The Project QAM will prepare a report summarizing the results of the data validation and any qualifications of data resulting from the validation assessment. Data validation reports will be submitted to the Supervising Contractor and included in the project's files.

Gamma Spectroscopy Data

All onsite analysis results for Ra-226 concentrations in surface materials or sediments based on gamma spectroscopy in the onsite soils lab will be evaluated by the Field Program Director in terms of system QC measurement records, degree of agreement between field sample results and confirmatory offsite analysis, and related consistency with the uncertainty reflected in the system calibration algorithm (expressed in units of activity concentration) to determine whether these indicators of accuracy and precision (see Section A.4.1) meet the project DQOs in terms of data usability.

The results of this data validation/verification assessment will be forwarded to the Project QAM for independent review. The Project QAM will prepare a report summarizing the results of the data validation and any qualifications of data resulting from the validation assessment. Data validation reports will be submitted to the Supervising Contractor and included in the project's files.

XRF Analysis Data

All onsite XRF measurement results for non-radiological ROD parameters (metals) in surface materials or sediments (including both field in-situ XRF survey data and intrusive XRF sample analysis) will be evaluated by the Field Program Director in terms of system QC measurement records, degree of agreement between field sample results and confirmatory offsite analysis, and related consistency with the uncertainty reflected in analyte-specific calibration (regression) curves to determine whether these indicators of accuracy and precision (see Section A.4.1) meet the project DQOs in terms of data usability. Performance and acceptance criteria are described in detail in Section A.4.1, along with AS-SOP 5 and Method 6200 (see Appendix 1).

The results of this data validation/verification assessment will be forwarded to the Project QAM for independent review. The Project QAM will prepare a report summarizing the results of the data validation and any qualifications of data resulting from the validation assessment. Data validation reports will be submitted to the Supervising Contractor and included in the project's files.

D.2.2 Validation/Verification of Offsite Laboratory Data

Laboratory results will first of all be checked for completeness to assure that all the requested analyses were performed along with the correct methodologies and detection limits. Data will be evaluated to assess whether the measurement performance criteria for accuracy and precision (Table 1) have been achieved. The QC data summaries and the associated raw data will be reviewed for the elements listed in the QC summary reporting requirements listed in Section B.7 for each method, with the exception of the mobile laboratory data. The inorganic non-radiological

data will be validated in accordance to guidelines in EPA (2010) for inorganic parameters. EPA (2010) is only designed for inorganic methods run by ICP, ICP-MS and Mercury analysis, and therefore some but not all of the functional guidelines for review apply to the other inorganic analyses named in this QAPP. The radiological data will be validated according to MARLAP (2004) for the QC elements below, as well as the required radiological analysis elements. All of the analyses (radiological and non-radiological) will be reviewed and validated for the following:

- Preservation and holding times
- Calibration data (including calibration blanks and verification samples, when applicable)
- Blanks (laboratory method blanks)
- Matrix spike samples
- Laboratory duplicate samples
- Laboratory control samples

In addition, the ICP and ICP-MS analyses will be reviewed and validated (per EPA 2010) for:

- ICP interference check sample
- ICP serial dilution
- ICP-MS tune analysis (ICP-MS only)
- ICP-MS internal standards (ICP-MS only)

For the radiological analysis, the following elements should be present for each data package (per MARLAP 2004):

- Aliquant size
- Tracking (link to instrument, run logs)
- Traceability of reference and standard materials
- Linkage of QC samples to project samples
- Chemical separation (yield)
- Efficiency, calibration curves and instrument background
- Spectral resolution
- Counts and duration of counts

The laboratory will provide a QC summary suitable for this level of review (described in Section B.7). Review of field duplicate data will be reviewed against the measurement performance criteria for precision (Table 1).

Data that is not rejected during a validation process is generally considered usable with any qualifications noted in the validation results. The following data qualifiers as defined by EPA (2010) will be applied to both radiological and non-radiological data:

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

J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.
U	Analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
UJ	Analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

In addition, the following qualifier will be applied to the radiological data, per MARLAP (2004):

Qualifier	Definition
E	Applies to data missing any of the above required radiological analysis elements.

The data to be verified are evaluated against project specifications (Section A.4) and are checked for errors, especially errors in transcription, calculations, and data input. Any suspected errors or anomalous data will be addressed by the manager of the task associated with the data, before data validation can be completed. Potential outliers are identified by the Project QAM and Supervising Contractor by examining results for unreasonable data, or identified using computer-based statistical software. If a question arises or an error or potential outlier is identified, the Field Supervisor or the Lab Project Manager responsible for generating the data is contacted to resolve the issue. Issues that can be resolved are corrected and documented electronically or by initialing and dating the associated paperwork. If an issue cannot be corrected, the QAM consults with the Supervising Contractor to determine the appropriate course of action, or the data associated with the issue are rejected.

The Project QAM will prepare a report summarizing the results of the data validation and any qualifications of data resulting from the validation following each sampling event. In addition to the data validation results, the reports will include the laboratory report job number(s), the sample IDs associated with each laboratory report and sample collection dates. If any issues such as confirmed errors or reanalysis resulting in revised data occur, these will be noted in the report. The data validation reports will be submitted to the Supervising Contractor and included in the project's files.

E. References

MARLAP 2004. Radiochemical Data Verification and Validation. MARLAP Manual Vol. 1, Chapter 8. July 2004. (Also see complete MARLAP reference: EPA, 2004).

Miller Geotechnical Consultants, Inc. (Miller Geotechnical). 2011. Mine Waste Investigations. February 11, 2011.

MWH Americas (MWH), 2013. Basis of Design Report, 60 Percent Design, Midnite Mine Superfund Site. December 16.

SENES Consultants (SENES), 2013. Midnite Mine Superfund Site – Radiation Protection Plan for Remedial Action. 60 Percent Design. December 12, 2013.

Tetra Tech, Inc., 2009. Health and Safety Plan for the Phase I RD/RA: Interim Water Management for the Midnite Mine. Revision 1.0. Prepared for Dawn Mining Company and Newmont USA Limited. May 5.

U.S. Environmental Protection Agency (EPA), 2004. Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP). NUREG-1576/EPA 402-B-04-001A. Other sponsoring agencies include: U.S. Department of Defense; U.S. Department of Energy; U.S. Department of Homeland Security; U.S. Nuclear Regulatory Commission; U.S. Food and Drug Administration; U.S. Geological Survey; National Institute of Standards and Technology.

U.S. Environmental Protection Agency (EPA), 2010. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review. Version ISM01.2. Publication 9240.1-51, EPA 540-R-10-011. January.

Appendix 1

Standard Operating Procedures

MIDNITE MINE SUPERFUND SITE REMEDIAL ACTION PROJECT STANDARD OPERATING PROCEDURE	**AS-SOP 1** **REVISION: 1**
DECONTAMINATION FOR FIELD SAMPLING	**DATE: June 16, 2015**

CONTENTS

1.0 PURPOSE **1**

2.0 APPLICABLE REFERENCE DOCUMENTS **1**

3.0 RESPONSIBILITY **1**

4.0 EQUIPMENT AND MATERIALS **2**

5.0 PROCEDURE **2**

6.0 QUALITY ASSURANCE/QUALITY CONTROL **2**

7.0 HEALTH AND SAFETY **2**

8.0 REFERENCES **3**

1.0 PURPOSE

Samples of surface materials (soils and rock fractions) and sediments will be collected and analyzed to help guide remedial excavation of across mine-impacted areas and mine drainages and to evaluate compliance with ROD cleanup levels. This standard operating procedure (SOP) provides the methods to be used for decontamination of sampling equipment, hands, and clothing that may become soiled between collection of different surface material or sediment samples. It describes the equipment, field procedures, and quality assurance/quality control (QA/QC) protocols to be employed when collecting surface material and sediment samples in order to prevent cross-contamination of samples.

2.0 APPLICABLE REFERENCE DOCUMENTS

The procedures in this SOP pertain to Appendix S of the Preliminary (60 Percent) Basis of Design Report for remedial action at the Midnite Mine Superfund Site (MWH, 2013a). Respective citations in this SOP (where applicable) include documents that fall under the following organization and reference structure of Appendix S:

Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (SENES, 2013a)

- Attachment S1 – Technical Basis (SENES, 2013b)
- Attachment S2 – Quality Assurance Project Plan (QAPP) (WME/SENES, 2013)
    - Appendix 1 – Standard Operating Procedures (SOPs)
        - AS-SOP 1 – Decontamination for Field Sampling
        - AS-SOP 2 – Surface Material and Sediment Sampling
        - AS-SOP 3 – Sampling Processing
        - AS-SOP 4 – Onsite Gamma Spectroscopy
        - AS-SOP 5 – Field-portable XRF Procedures
        - AS-SOP 6 – Gamma Surveys
    - Appendix 2 – Corrective Action Report Form
    - Appendix 3 – Approved Laboratory Quality Assurance Plans
    - Appendix 4 – Laboratory Certification
- Attachment S3 – Determination of Bedrock during Remedial Excavation (MWH, 2013b)

3.0 RESPONSIBILITY

The Field Technician is responsible for decontaminating sampling equipment, hands and clothing as necessary to prevent cross-contamination of samples. It is the responsibility of the Field Supervisor to manage the field sampling program and to ensure that decontamination supplies are available, that these procedures are followed, and that the samples collected will meet the data quality objectives for the project. All personnel working onsite are responsible for adherence to the current project Health and Safety Plan (HASP) and Radiation Protection Plan (RPP) (Tetra Tech, 2009; SENES, 2013c). Additional general information regarding project personnel and respective responsibilities is provided in the QAPP (Attachment S2).

4.0 EQUIPMENT AND MATERIALS

The following decontamination equipment and supplies may be required during sample collection. Some items may not be necessary for every sampling event.

- Stiff bristled brushes
- Clean rinse water
- Spray bottles
- Bucket
- Alconox or similar detergent
- Liquid hand soap
- Paper towels
- Disposable gloves

5.0 PROCEDURE

1. Physically remove any visibly soiled material from sampling tools, hands and clothes with a stiff bristled brush.
2. Using a spray bottle containing diluted Alconox detergent, spray the surfaces of the sampling trowel and pick axe, then rinse with generous amount of clean rinse water from a spray bottle or other container. Rinse water is discharged at the sampling site. Dry the sampling equipment with paper towels.
3. Wash hands with liquid hand soap and clean rinse water. Dry with paper towels.
4. Collect all used paper towels in a plastic trash bag. The amount of residual soils transferred to paper towels after rinsing should be minimal and should not require special disposal protocols.
5. Because there is no need for hands to contact the soil/sediment being sampled, use of disposable latex or nitrile gloves during sampling is not mandatory provided hands are washed and free of loose/removable soil between sampling locations. However, use of disposable gloves can minimize the need for washing hands.

6.0 QUALITY ASSURANCE/QUALITY CONTROL

Assurance of proper decontamination will be addressed by following the above procedures. Adherence to these protocols may be subject to internal and/or external audit as described in the QAPP.

7.0 HEALTH AND SAFETY

Standard worker health and safety requirements for fieldwork must be observed when using this procedure. These are defined in the current Health and Safety Plan (HASP) and Radiation Protection Plan (RPP) (Tetra Tech, 2009; SENES, 2013c). This includes no eating or tobacco use while sampling at the Site. Drinking of water is necessary to prevent dehydration, but bottled

water with screw-caps is required to minimize the potential for inadvertent ingestion of solid particulates that may contain low levels of radionuclides or metals.

8.0 REFERENCES

MWH Americas (MWH), 2013a. Basis of Design Report, Preliminary (60 Percent) Design, Midnite Mine Superfund Site. December 16.

MWH Americas (MWH), 2013b. Attachment S3 – Determination of Bedrock during Remedial Excavation. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013). Memo from MWH: Defining Rock Excavation during RA Construction at the Midnite Mine Superfund Site. July 12, 2013.

SENES Consultants (SENES), 2013a. Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments. December, 2013. Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013b. Attachment S1 – Technical Basis. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013c. Midnite Mine Superfund Site – Radiation Protection Plan for Remedial Action. 60 Percent Design. December 12, 2013.

Tetra Tech, Inc., 2009. Health and Safety Plan for the Phase I RD/RA: Interim Water Management for the Midnite Mine. Revision 1.0. Prepared for Dawn Mining Company and Newmont USA Limited. May 5.

Worthington Miller Environmental, LLC. (WME) and SENES Consultants (SENES), 2013. Attachment S2 – Quality Assurance Project Plan (QAPP), Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

MIDNITE MINE SUPERFUND SITE REMEDIAL ACTION PROJECT STANDARD OPERATING PROCEDURE	**AS-SOP 2** **REVISION: 1**
SURFACE MATERIAL AND SEDIMENT SAMPLING	**DATE: June 16, 2015**

CONTENTS

1.0 PURPOSE **1**

2.0 APPLICABLE REFERENCE DOCUMENTS **1**

3.0 RESPONSIBILITY **1**

4.0 PRECAUTIONS **2**

5.0 EQUIPMENT AND MATERIALS **2**

6.0 PROCEDURE **2**

6.1 SAMPLE LOCATION SELECTION 2
6.2 DECONTAMINATION 3
6.3 DOCUMENTATION 3
6.4 SAMPLE COLLECTION 4

7.0 SAMPLE PROCESSING **5**

8.0 SAMPLE SHIPPING **5**

9.0 QUALITY ASSURANCE/QUALITY CONTROL **5**

10.0 HEALTH AND SAFETY **5**

11.0 REFERENCES **5**

1.0 PURPOSE

Samples of surface materials (soils and rock fractions) and sediments will be collected and analyzed to help guide remedial excavations across mine-impacted areas and mine drainages, and to evaluate compliance with ROD cleanup levels during final status surveys. This standard operating procedure (SOP) provides the methods to be used for collection of surface materials and sediments in accordance with Appendix S. It describes the equipment, field procedures, analysis, record keeping, and quality assurance/quality control (QA/QC) protocols to be employed when collecting surface material and sediment samples.

2.0 APPLICABLE REFERENCE DOCUMENTS

The procedures in this SOP pertain to Appendix S of the Preliminary (60 Percent) Basis of Design Report for remedial action at the Midnite Mine Superfund Site (MWH, 2013a). Respective citations in this SOP (where applicable) include documents that fall under the following organization and reference structure of Appendix S:

Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (SENES, 2013a)

- Attachment S1 – Technical Basis (SENES, 2013b)
- Attachment S2 – Quality Assurance Project Plan (QAPP) (WME/SENES, 2013)
    - Appendix 1 – Standard Operating Procedures (SOPs)
        - AS-SOP 1 – Decontamination for Field Sampling
        - AS-SOP 2 – Surface Material and Sediment Sampling
        - AS-SOP 3 – Sampling Processing
        - AS-SOP 4 – Onsite Gamma Spectroscopy
        - AS-SOP 5 – Field-portable XRF Procedures
        - AS-SOP 6 – Gamma Surveys
    - Appendix 2 – Corrective Action Report Form
    - Appendix 3 – Approved Laboratory Quality Assurance Plans
    - Appendix 4 – Laboratory Certification
- Attachment S3 – Determination of Bedrock during Remedial Excavation (MWH, 2013b)

3.0 RESPONSIBILITY

The Field Technician is responsible for collecting samples in accordance with the methods, specifications and procedures indicated in Appendix S, the QAPP (Attachment S2), in this SOP, and in accordance with the direction of the Field Supervisor and/or Field Program Director. It is the responsibility of the Field Supervisor to manage the field sampling program and to ensure that the samples collected will meet the data quality objectives for the project. All personnel working onsite are responsible for adherence to the current project Health and Safety Plan (HASP) and Radiation Protection Plan (RPP) (Tetra Tech, 2009; SENES, 2013c). Additional general information regarding project personnel and respective responsibilities is provided in the QAPP (Attachment S2).

4.0 PRECAUTIONS

- Heavy duty (freezer type) re-sealable baggies should be used for sample collection.
- It is the Field Technician's responsibility to ensure accuracy in sample labeling and records keeping, and that samples and corresponding logbook entries are delivered to the onsite lab for processing shortly after sample collection.

5.0 EQUIPMENT AND MATERIALS

The following equipment and supplies may be required during sample collection. Some items may not be necessary for every sampling event.

- Stainless steel metal shovel or hand trowel.
- Small pick axe.
- Sample containers (heavy duty quart-sized re-sealable plastic baggies).
- Water resistant sample labels.
- Water resistant ink pens, Sharpie® or equivalent indelible markers.
- Field logbook.
- Decontamination equipment and supplies (e.g. rinse water, paper towels, brush, disposable gloves).
- Handheld GPS instrument.
- Field measuring tape or digital measurement wheel.
- Personnel protective equipment.
- Gamma detector (as needed, depending on sampling objectives).
- Digital camera (as needed to document unusual observations).
- Coolers or other hard sided containers.
- Pin flags.
- First Aid Kit.

6.0 PROCEDURE

6.1 Sample Location Selection

For remedial support sampling activities, locations will be selected based on the direction of the Field Supervisor and/or Field Program Director. For final status surveys, maps of target locations will be developed by the Field Program Director and will be provided by the Field Supervisor and discussed with the Field Technician(s) in advance of the sampling event. These maps will be developed in accordance with the final status survey plans outlined in Appendix S. Target sampling locations will be determined in the field using maps, field tape measure and/or GPS device as appropriate. Locations may be adjusted based on safe access issues or surface obstructions. Significant deviations or adjustments must be approved by the Field Supervisor.

6.2 Decontamination

Clean hands and all equipment as indicated in AS-SOP 1.

6.3 Documentation

Document all required sample information in the field logbook and on the sample label as detailed below.

6.3.1 Logbook

The field logbook is crucial as this is the first hand record of each sampling event. The field logbook must be a bound field book with weather resistant and smudge resistant pages.

Required Logbook entries:

- Sample ID.
- Sampling location (GPS coordinates).
- Date of collection.
- Sample type (soil, mixed soil/rock, or sediment)
- Person(s) performing the sampling.
- Field observations:

  Sampling situations vary widely and conditions or circumstances can have a bearing on results and respective data interpretations. The best guideline is to record sufficient information such that the sampling event and relevant conditions could be reconstructed without relying on the sampler's memory. At minimum, weather conditions and any special or unusual circumstances that could have a bearing on analytical results (e.g. unusual soil appearance, texture, etc.) should be recorded. Photos may also be taken to aid with documentation of unusual observations.

Completed logbooks must be scanned into an electronic format in their entirety. Both hardcopies and electronic copies will be filed in the project archives.

6.3.2 Sample Labels

Sample labels will be water resistant and designed for this purpose. Samples will be labeled using an indelible marker before or immediately after sampling. Labels should include the following:

- Sample ID.
- Date and time of collection.

- Site name (Midnite Mine).
- Sampler initials.

The sample IDs will be in the form of: AAAA/BBBB/CCC/DDD/##

Where:

AAAA = sample ID number
BBBB = sample depth (0-15 cm for all surface samples)
CCC = sample matrix (SED = sediment; SOI = soil)
DDD = sampling method (DIS = discrete; COM = composite)
= sample type (01=primary, 02=duplicate)

6.3.3 Chain of Custody

Sample chain of custody/analysis request forms are required for all samples that will be shipped for offsite analysis at a commercial laboratory. The required information to be included on these forms, along with all custody and shipping protocols, are detailed in the QAPP (Attachment S2).

6.4 Sample Collection

6.4.1 Discrete Samples

Other than samples collected for the purpose of refining the gamma/soil Ra-226 correlation (see Appendix S), all samples will be collected as discrete grab samples. All samples will be collected to a depth of 15 cm from the ground surface (0-15 cm sample depth). Clear grass, large rocks, sticks, etc. from the sample location. Use a small pick axe to dig a 15-cm deep hole, clear soil and debris from the hole then use a hand trowel to evenly scrape about 350 grams of soil from the sides of the hole and place the sample in the properly labeled plastic re-sealable baggie. The scraping should evenly transverse the entire vertical depth of the hole in order to sample an even amount of soil across the entire 15-cm depth increment.

6.4.2 Composite Samples

For samples to be analyzed for the purpose of developing the gamma/soil Ra-226 correlation, the general procedure is detailed in Appendix S. All correlation sample labels should include a sample ID prefix of “CORR”. The above protocols for discrete sampling will be observed for composite correlation samples except that the volume of each sub-sample collected across the correlation plot (100 m^2) will be smaller (the volume for each sub-sample should be about 40 grams). The volume collected from each of the 9 sub-sampling locations should be as equal as possible to avoid introduction of any spatial bias. Combine the 9 sub-samples in a single sample baggie.

Homogenization is particularly crucial for correlation samples and thus, the sample label and logbook must each clearly indicate when a composite sample for correlation purposes is collected.

All samples should be placed into a hard sided container until returned to the onsite soils laboratory for processing.

7.0 SAMPLE PROCESSING

All samples will be promptly delivered to the onsite soils lab for processing in accordance with the provisions of the Plan (Appendix S) and the procedures indicated in AS-SOP 3.

8.0 SAMPLE SHIPPING

Protocols for all samples to be shipped to the offsite commercial laboratory will include strict chain of custody requirements, along with special procedures for instructing sample handling and analysis at the offsite lab. These requirements and procedures are specified in the QAPP (Attachment S2) and in AS-SOP 3.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Data QA/QC will be addressed by following the methods, specifications and procedures indicated in Appendix S, the QAPP (Attachment S2), and in this SOP. Adherence to these specifications may be subject to internal and/or external audit as described in the QAPP.

10.0 HEALTH AND SAFETY

Standard worker health and safety requirements for fieldwork must be observed when using this procedure. These requirements are defined in the current Health and Safety Plan (HASP) and Radiation Protection Plan (RPP) (Tetra Tech, 2009; SENES, 2013c).

11.0 REFERENCES

MWH Americas (MWH), 2013a. Basis of Design Report, Preliminary (60 Percent) Design, Midnite Mine Superfund Site. December 16.

MWH Americas (MWH), 2013b. Attachment S3 – Determination of Bedrock during Remedial Excavation. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013). Memo from MWH: Defining Rock Excavation during RA Construction at the Midnite Mine Superfund Site. July 12, 2013.

SENES Consultants (SENES), 2013a. Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments. December, 2013. Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013b. Attachment S1 – Technical Basis. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013c. Midnite Mine Superfund Site – Radiation Protection Plan for Remedial Action. 60 Percent Design. December 12, 2013.

Tetra Tech, Inc., 2009. Health and Safety Plan for the Phase I RD/RA: Interim Water Management for the Midnite Mine. Revision 1.0. Prepared for Dawn Mining Company and Newmont USA Limited. May 5.

Worthington Miller Environmental, LLC. (WME) and SENES Consultants (SENES), 2013. Attachment S2 – Quality Assurance Project Plan (QAPP), Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

MIDNITE MINE SUPERFUND SITE REMEDIAL ACTION PROJECT STANDARD OPERATING PROCEDURE	**AS-SOP 3** **REVISION: 1**
SAMPLE PROSSESSING	**DATE: June 16, 2015**

CONTENTS

1.0 PURPOSE **1**

2.0 APPLICABLE REFERENCE DOCUMENTS **1**

3.0 RESPONSIBILITY **2**

4.0 PRECAUTIONS **2**

5.0 EQUIPMENT AND MATERIALS **2**

6.0 PROCEDURE **3**

6.1 SAMPLE LOGIN AND TRACKING 3
6.2 SAMPLE PREPARATION 4
6.3 SAMPLE SHIPPING / OFFSITE ANALYSIS 5

7.0 QUALITY ASSURANCE/QUALITY CONTROL **7**

8.0 HEALTH AND SAFETY **7**

9.0 REFERENCES **7**

1.0 PURPOSE

Samples of surface materials and sediments will be collected at the Midnite Mine and analyzed to help guide remedial excavation across mine-impacted areas, and to evaluate compliance with ROD cleanup levels during final status surveys. This standard operating procedure (SOP) provides guidance regarding the sample processing protocols that will be followed when samples are received in the onsite soils laboratory. The functions of the onsite soils lab include two basic elements:

1. *Sample Processing*

   - Sample log-in and electronic data entry of all sample collection information.
   - Sample preparation for onsite analysis.
   - Data management (sample login/tracking and sample analysis spreadsheets).
   - Sample management and storage.
   - Sample shipping for the fraction of samples that are sent to an offsite commercial laboratory.

2. *Sample Analysis*

   - Onsite analysis of Ra-226 concentrations in solid samples by gamma spectroscopy.
   - Onsite analysis of the concentrations of metals in solid samples by XRF analysis.

Procedures for sample analysis are covered separately in AS-SOP 4 (onsite gamma spectroscopy) and AS-SOP 5 (XRF procedures). Because gamma spectroscopy will be performed on all samples (both soil and sediment matrices), sample preparation procedures for gamma spectroscopy are provided in this SOP. For the fraction of samples that will also be analyzed with the intrusive XRF methodology, respective sample preparation procedures are provided separately in AS-SOP 5.

All onsite soils lab functions will all be conducted in accordance with the specifications of Appendix S and data quality objectives (DQOs) indicated in the QAPP (Attachment S2). This SOP describes the requirements, equipment, procedures and quality assurance/quality control (QA/QC) protocols to be employed for sample processing in the onsite soils lab.

2.0 APPLICABLE REFERENCE DOCUMENTS

The procedures in this SOP pertain to Appendix S of the Preliminary (60 Percent) Basis of Design Report for remedial action at the Midnite Mine Superfund Site (MWH, 2013a). Respective citations in this SOP (where applicable) include documents that fall under the following organization and reference structure of Appendix S:

Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (SENES, 2013a)

- Attachment S1 – Technical Basis (SENES, 2013b)
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  - AS-SOP 3 – Sampling Processing
  - AS-SOP 4 – Onsite Gamma Spectroscopy
  - AS-SOP 5 – Field-portable XRF Procedures
  - AS-SOP 6 – Gamma Surveys
- Appendix 2 – Corrective Action Report Form
- Appendix 3 – Approved Laboratory Quality Assurance Plans
- Appendix 4 – Laboratory Certification

- Attachment S3 – Determination of Bedrock during Remedial Excavation (MWH, 2013b)

3.0 RESPONSIBILITY

The Field Technician is responsible for processing samples in accordance with the methods, specifications and procedures indicated in the QAPP (Attachment S2), in this SOP, and in accordance with the direction of the Field Supervisor and/or Field Program Director. It is the responsibility of the Field Supervisor to manage the onsite soils lab and to ensure that the samples processed will meet the data quality objectives for the project. Additional information regarding the individuals who will be involved and the tasks for which they are responsible are detailed in the QAPP (Attachment S2).

4.0 PRECAUTIONS

- Preparation of samples has the potential to create small amounts of airborne dust that may contain low levels of uranium and its decay products. Although the potential for inhalation of radiologically significant quantities of radionuclides from this activity is extremely small and does not warrant a respiratory control program (the vast majority of samples will be near or below background levels), dust masks should be worn for worker comfort and for consistency with the ALARA policy under the Radiation Protection Plan.

- It is the responsibility of the Field Technician(s) working in the onsite soils lab to ensure 1) accuracy in transcription of all sample collection data into the electronic sample login/tracking spreadsheet, 2) proper sample preparation, 3) accurate entry of sample information into the sample analysis spreadsheet, and 4) that samples are properly identified and managed throughout the sample processing life cycle to prevent sample misidentification or misplacement of samples.

5.0 EQUIPMENT AND MATERIALS

The following infrastructure, equipment and supplies will be required for sample processing in the onsite soils lab:

FACILITIES:

- Trailer or section of existing facilities that can be dedicated to the onsite lab.
- Infrastructure requirements include:

- Reliable hard power with multiple outlets
- Sufficient bench space, shelving, storage areas to support a multi-year project.
- A utility sink area with running water (if necessary, the utility sink area can be located outside of the onsite soils lab). The water supply need not be potable, but it must not contain elevated levels of radionuclides or metals.

EQUIPMENT AND SUPPLIES

- Laptop computer with standard productivity software (e.g. Microsoft Office Suite)
- Portable printer with document scanning capability
- Analytical scale (digital, with minimum precision of 0.1 grams)
- Several portable ovens (available at common stores such as Wal-Mart or Target)
- Personnel protective equipment (PPE) including dust masks, disposable gloves, safety glasses, and ear protection (if applicable)
- Deionized or distilled water for equipment decontamination (if determined necessary)
- Counting cans with lids (must match the counting cans used by the selected offsite lab)
- Size 2 mesh (1 cm) sieve for gamma spectroscopy samples
- Size 60 mesh (0.23 mm) sieve for processing XRF samples
- Mallet for breaking up large soil clods; mortar/pestle for grinding XRF samples
- Stainless steel spoons for homogenizing and transferring sample
- Aluminum pie plates
- Heavy duty aluminum foil
- Bound, water resistant field logbooks
- Chain-of-Custody/analytical request forms (usually supplied by the offsite laboratory)
- Custody seals
- Coolers for sample transfer and shipping
- Liquid dish soap and scrub brush
- Soft bristle paint brushes
- Paper towels
- Indelible markers, ink pens
- Heavy duty re-sealable plastic baggies (both gallon and quart sized)
- Paper towels
- Electrical tape
- Fire extinguisher
- First Aid Kit

6.0 PROCEDURE

6.1 Sample Login and Tracking

1. Scan the logbook entries for the samples in question and print hardcopies.
2. Verify that all sample ID information matches that indicated on the sample labels.
3. Enter all sample collection information from the logbook and sample labels into the electronic

sample login/tracking spreadsheet.

4. Verify onsite analyses to be performed for each sample and enter scheduled analysis and date
5. Create a small but thickly folded (i.e. durable) aluminum foil tag and write the sample ID number on the tag with a permanent ink marker. Place the ID tag inside the sample baggie on top of the sample. The ID tag must physically reside in the bulk soil sample throughout the sample preparation process.
6. If excess sample remains after aliquots have been extracted for gamma spectroscopy and XRF analysis, the ID tag will remain with the excess sample, which will be placed back in the original sample baggie and weighed. The weight of any excess sample to be archived will be entered into the sample login/tracking spreadsheet. Archived samples will be double bagged and stored by sample collection date. Once it is determined that an archived sample is no longer needed, it will be disposed along with other waste rock/soils/sediments being placed in the pits.
7. Before shipping of any samples offsite (see Section 6.3), the sample login spreadsheet, along with the onsite sample analysis spreadsheet, must be updated to indicate which samples are being shipped to the offsite lab for analysis. The date of shipping should be entered along with the COC form number.
8. The originator's copy of the COC form will be filed chronologically in the onsite project files.
9. Once the samples have been shipped offsite, those samples will be archived according to the laboratory's protocols (typically 6 months) unless special requests are made for longer archiving. Samples sent offsite will not be returned to the Site, and respective sample tracking, if deemed necessary, will be the responsibility of the Laboratory Project Manager.

6.2 Sample Preparation

1. Clean hands and wear disposable gloves, dust mask and safety glasses.

2. Pour the soil sample from the baggie onto an aluminum pie plate, spread the sample evenly across the plate (to maximize exposure of the bulk sample to air), and place the aluminum sample ID tag on top of the sample. Retain the original labeled sample bag for later use in archiving excess sample.

3. Place the sample in the oven until thoroughly dry [e.g. 300° F (150° C) for about 1 hour].

4. Using the steel mallet, break up any soil clods to ensure that the bulk sample will be representative after sieving.

5. Sieve the sample through the size 2 mesh (1 cm) screen to remove rocks larger than 1 cm diameter. Discard the removed rock fraction.

6. Using a stainless steel spoon, manually mix and blend the sample until thoroughly homogenized

7. On the scale, tare a counting can base (without lid).

8. Using the stainless steel spoon, systematically remove small aliquots of the sample from various representative locations across the pie plate and place in the counting can until the can is full.

    - Note: If the sample is also scheduled for intrusive XRF analysis, prepare the excess sample in accordance with the sample preparation steps indicated in AS-SOP 6 (XRF Procedures). Otherwise, return the excess sample and aluminum sample tag back to the original sample collection baggie, double bag, and place in storage (residual samples should be archived by sample collection date).

9. Place the sample and counting can base on the scale to determine the net dry sample weight. Using a permanent marker, record the sample ID, collection date, sealing date, and soil sample weight on the can lid (counting cans with label stickers pre-attached to the lids are available).

10. Enter the sample ID, collection date, sealing date, and soil sample weight in the sample analysis spreadsheet.

11. Remove any residual dust along the outside rim of the can with a paper towel and cap the can with the labeled lid. Seal the can with electrical tape, stretching the tape tightly and pressing down the tape with fingers along the lip of the lid to maximize sealing effectiveness. Wind the tape at least 2 complete times around the circumference of the can, stretching and finger-pressure-sealing as you go.

12. Count the sample on the NaI-based gamma spectroscopy system the same day that it is sealed. Enter the counting results and count date in the sample analysis spreadsheet.

13. After counting, place the canned/sealed sample in storage (organized by sample collection date).

14. Wash (with soap and water) and dry all sample preparation equipment and pie plates before using for subsequent samples.

15. Clean all sample preparation benches and related work spaces of residual soil with soft-bristled paint brushes before processing the next sample.

6.3 Sample Shipping / Offsite Analysis

Once the fraction of samples that will be sent to the offsite commercial laboratory have been selected (based on the specifications of Appendix S and the direction of the Field Program Director), the chain of custody protocols detailed in the QAPP (Attachment S2) will be strictly followed for the transfer of these samples to the offsite lab for analysis. As previously indicated, before shipping, the sample login/tracking spreadsheet and onsite sample analysis spreadsheet will be updated to indicate which samples have been shipped to the offsite lab for analysis. In

addition, there are special requirements regarding the protocols to be followed by the offsite laboratory for sample handling and analysis, and clear instructions must be provided with the COC forms when shipping samples. These special requirements and protocols, along with the procedures that onsite soils lab personnel must follow to ensure proper implementation, are as follows:

Surface Material Samples

1. Only the canned and sealed portion of each sample as previously analyzed onsite for Ra-226 will be shipped and analyzed at the offsite lab (archived excess sample will remain archived onsite). These canned samples must remain sealed for shipment to the offsite lab.
2. On the COC/analyte request form, the notes section must reference and include an attached set of special instructions that clearly indicate that these samples are not to be unsealed until after they have been analyzed by gamma spectroscopy for Ra-226 (Method 901.1) at least 21 days after the date the can was sealed in the onsite lab (as indicated on the lid of each sealed sample can), and that the dry sample weight indicated on the can label should be used to calculate the "full radon ingrowth" Ra-226 concentration. The laboratory must be clearly informed and instructed in this special protocol.
3. The protocol in item 2 above is crucial to the success of the program and prior to sample shipping the offsite lab should be contacted directly and notified in advance of these special requirements and instructions in order to help avoid confusion or mistakes.
4. After the lab has performed full-ingrowth analysis for Ra-226, the samples will be unsealed at the offsite lab, re-homogenized and analyzed for uranium and Pb-210 according to the methods specified in Appendix S and the QAPP (Attachment S2).

Sediment Samples

1. For sediment samples, both the canned/sealed portion of each sample as previously analyzed onsite by gamma spectroscopy, as well as the smaller plastic XRF cup as previously analyzed by XRF, will be shipped together and analyzed concurrently at the offsite lab. Again, any additional (excess) sample previously stored onsite in original plastic sample collection baggies will remain archived onsite.
2. The above protocols for Ra-226 analysis in canned/sealed surface material samples also apply to sediment samples. After counting the sealed sample for Ra-226 (after full radon ingrowth), the offsite lab will unseal the sample and process it for analysis of U-238, U-234, and Pb-210 according to the methods specified in Appendix S and in the QAPP (Attachment S2) for sediments.
3. For sediment samples in plastic XRF cups, the offsite lab will analyze the sample for all metals reflected in ROD cleanup levels for sediments per the methods specified in Appendix S and in the QAPP (Attachment S2).

7.0 QUALITY ASSURANCE/QUALITY CONTROL

QA/QC for sample processing will be ensured by carefully following the methods, specifications and procedures indicated in this SOP and in the QAPP (Attachment S2). The performance, accuracy and reliability of the sample processing system being implemented by onsite soils lab staff may be subject to internal and/or external audit as described in the QAPP.

8.0 HEALTH AND SAFETY

Standard worker health and safety requirements for all onsite workers must be observed when performing sample processing as described in this SOP. These are defined in the current Health and Safety Plan (HASP) and Radiation Protection Plan (RPP) (Tetra Tech, 2009; SENES, 2013c). As previously noted, sample preparation procedures can create small amounts of airborne dust. The potential for inhalation of radiologically significant quantities of radionuclides from this activity is extremely small and does not warrant a respiratory control program (the vast majority of samples will be near or below background levels), but dust masks should be worn for worker comfort and for consistency with the ALARA policy under the Radiation Protection Plan.

9.0 REFERENCES

MWH Americas (MWH), 2013a. Basis of Design Report, Preliminary (60 Percent) Design, Midnite Mine Superfund Site. December 16.

MWH Americas (MWH), 2013b. Attachment S3 – Determination of Bedrock during Remedial Excavation. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013). Memo from MWH: Defining Rock Excavation during RA Construction at the Midnite Mine Superfund Site. July 12, 2013.

SENES Consultants (SENES), 2013a. Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments. December, 2013. Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013b. Attachment S1 – Technical Basis. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013c. Midnite Mine Superfund Site – Radiation Protection Plan for Remedial Action. 60 Percent Design. December 12, 2013.

Tetra Tech, Inc., 2009. Health and Safety Plan for the Phase I RD/RA: Interim Water Management for the Midnite Mine. Revision 1.0. Prepared for Dawn Mining Company and Newmont USA Limited. May 5.

Worthington Miller Environmental, LLC. (WME) and SENES Consultants (SENES), 2013. Attachment S2 – Quality Assurance Project Plan (QAPP), Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

MIDNITE MINE SUPERFUND SITE REMEDIAL ACTION PROJECT STANDARD OPERATING PROCEDURE	**AS-SOP 4** **REVISION: 1**
ONSITE GAMMA SPECTROSCOPY	**DATE: June 16, 2015**

CONTENTS

1.0 PURPOSE 1

2.0 APPLICABLE REFERENCE DOCUMENTS 1

3.0 RESPONSIBILITY 2

4.0 PRECAUTIONS 3

5.0 EQUIPMENT 3

6.0 PROCEDURE 4

7.0 DATA QUALITY ASSURANCE/QUALITY CONTROL 5

8.0 HEALTH AND SAFETY 5

9.0 REFERENCES 5

1.0 PURPOSE

Samples of surface materials and sediments will be collected at the Midnite Mine and analyzed in an onsite soils lab to help guide remedial excavation across mine-impacted areas and to help evaluate compliance with ROD cleanup levels during final status surveys. This standard operating procedure (SOP) provides guidance regarding the systems and procedures that will be used for onsite measurement of Ra-226 concentrations in solid samples based on gamma spectroscopy methods. These methods are described in Attachment S1 to Appendix S (see Section 2.0). The functions of the onsite soils lab include two basic elements:

1. *Sample Processing*

   - Sample log-in and electronic data entry of all sample collection information.
   - Sample preparation for onsite analysis.
   - Data management (sample login/tracking and sample analysis spreadsheets).
   - Sample management and storage.
   - Sample shipping for the fraction of samples that are sent to an offsite commercial laboratory.

2. *Sample Analysis*

   - Onsite analysis of Ra-226 concentrations in solid samples by gamma spectroscopy.
   - Onsite analysis of the concentrations of metals in solid samples by XRF analysis.

The procedures for onsite sample processing and XRF analysis are covered separately in AS-SOP 3 and AS-SOP 5 respectively (see Section 2.0). Once samples have been prepared for gamma spectroscopy as indicated in AS-SOP 3, they will be counted in a lead-shielded counting well in order to calculate Ra-226 concentrations. The technical basis for the gamma spectroscopy system to be utilized is provided in Attachment S1 to Appendix S (see Section 2.0). The initial setup of the system is a complex, site-specific and project-specific process, and system setup is not the subject of this SOP (this is the responsibility of the Field Program Director). This SOP describes the basic equipment, procedures and quality assurance/quality control (QA/QC) protocols to be employed for routine sample counting by gamma spectroscopy in the onsite soils lab.

2.0 APPLICABLE REFERENCE DOCUMENTS

The procedures in this SOP pertain to Appendix S of the Preliminary (60 Percent) Basis of Design Report for remedial action at the Midnite Mine Superfund Site (MWH, 2013a). Respective citations in this SOP (where applicable) include documents that fall under the following organization and reference structure of Appendix S:

Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (SENES, 2013a)

- Attachment S1 – Technical Basis (SENES, 2013b)
- Attachment S2 – Quality Assurance Project Plan (QAPP) (WME/SENES, 2013)

- Appendix 1 – Standard Operating Procedures (SOPs)
  - AS-SOP 1 – Decontamination for Field Sampling
  - AS-SOP 2 – Surface Material and Sediment Sampling
  - AS-SOP 3 – Sampling Processing
  - AS-SOP 4 – Onsite Gamma Spectroscopy
  - AS-SOP 5 – Field-portable XRF Procedures
  - AS-SOP 6 – Gamma Surveys
- Appendix 2 – Corrective Action Report Form
- Appendix 3 – Approved Laboratory Quality Assurance Plans
- Appendix 4 – Laboratory Certification

- Attachment S3 – Determination of Bedrock during Remedial Excavation (MWH, 2013b)

3.0 RESPONSIBILITY

Field Program Director responsibilities:

- Procure all necessary analytical equipment.
- Initial setup of the gamma spectroscopy system in the onsite soils lab.
- Develop secondary soil Ra-226 reference material standards.
- Define spectral regions of interest (ROIs) for Ra-226 analysis.
- Generate and refine the system calibration between count data and Ra-226 activity.
- Determine the optimal count time for low-level samples based on calculations of the minimum detectable concentration (MDC) (e.g. following the methodology described in Martin, 2003).
- Develop a spreadsheet for data entry and calculation of sample analysis results.
- Ensure that all onsite soils lab personnel are qualified to perform the functions to which they are assigned.
- Train onsite soils lab personnel in the proper use of the gamma spectroscopy counting system and associated analysis, data generation and data management protocols.

Field Supervisor responsibilities:

- Manage onsite soils lab operations in the field.
- Ensure that the necessary supplies are available to support uninterrupted operation of onsite soils lab functions.
- Oversee/audit sample processing and counting on the gamma spectroscopy system to ensure that data generated will meet the data quality objectives for the project.
- Review data to identify additional sampling needs.
- Identify potential random or systemic sources of analytical error and implement corrective action(s) as needed to resolve related issues.
- Maintain analytical equipment, including identification, troubleshooting and resolution of improper instrument performance.

Field Technician responsibilities:

- Perform sample gamma counting in accordance with the methods, specifications and

procedures indicated in this SOP, the QAPP (Attachment S2), and in accordance with the direction of the Field Supervisor and/or Field Program Director.

- Report equipment problems or potentially problematic analysis results to the Field Supervisor.

All personnel working onsite are responsible for adherence to the current project Health and Safety Plan (HASP) and Radiation Protection Plan (RPP) (Tetra Tech, 2009; SENES, 2013c). Additional general information regarding project personnel and respective responsibilities is provided in the QAPP (Attachment S2).

4.0 PRECAUTIONS

- Rapid changes in the high voltage (HV) setting on the multi-channel analyzer (MCA) can damage the gamma detector. Do not change the pre-set system HV as established by the Field Program Director. Never exchange the detector for a new detector while the coaxial cable is plugged into the MCA and the MCA is powered on. NaI detectors are relatively durable, but can be damaged if dropped onto a hard surface or if exposed to extreme rapid temperature changes. Handle with care.
- The NaI detector to be used for this application is sensitive to temperature changes. Care must be taken to maintain a relatively consistent temperature in the onsite soils lab to the extent possible, and to perform periodic energy calibrations throughout the day to correct for spectral drift as needed. This SOP is not intended to direct the user on respective fine gain setting, or setting the high voltage power supply. These parameters and protocols will be established by the Field Program Director during initial system setup and system operators will be trained on related specifics.
- It is the responsibility of the Field Technician(s) working in the onsite soils lab to ensure 1) accuracy in transcription of all sample collection data into the electronic sample login/tracking spreadsheet, 2) proper sample preparation, 3) accurate entry of sample information into the sample analysis spreadsheet, and 4) that samples are properly identified and managed throughout the sample processing life cycle to prevent sample misidentification or misplacement of samples.

5.0 EQUIPMENT

The following equipment and supplies will be required for sample gamma counting in the onsite soils lab:

- Laptop computer with standard productivity software (e.g. Microsoft Office Suite)
- Second laptop computer for dedicated use with the gamma spectroscopy system
- Portable printer with document scanning capability
- 3×3” NaI(Tl) scintillation detector with photomultiplier tube and coaxial cable
- Small, portable multi-channel analyzer (MCA) with integrated high voltage, preamplifier

and associated software (e.g. URSA-II from Radiation Safety Associates, Inc.)

- Lead plates and rings, arranged to create a shielded counting well to house both the sample and NaI detector during counting
- Device to place/remove sample counting cans from the bottom of the counting well (e.g. a strong magnet attached the end of a wooden dowel – magnet must be strong enough to lift over 200 grams of soil in a counting can).
- Certified (NIST traceable) sealed Cs-137 check source (small button source), enclosed inside a sample counting can (taped to the center of the bottom of the can) with the can lid sealed in the same manner as a field sample.
- Two of the secondary soil Ra-226 reference material standards used for the original system calibration, one near background levels (e.g. 1-2 pCi/g) and one with a higher concentration (e.g. in the range of 15-20 pCi/g). These same two standards will be used for daily quality control (QC) measurements throughout the project.

6.0 PROCEDURE

1. Following the training and instructions provided by the Field Program Director, make sure the MCA and NaI detector are connected, start up the dedicated computer for gamma spectroscopy, and turn on the MCA power.

   Note: Rapid changes in the high voltage (HV) on the multi-channel analyzer (MCA) can damage the gamma detector. Do not change the system pre-set as established by the Field Program Director.

2. Place the canned Cs-137 check source in the counting well, insert the detector into the well on top of the check source can. Initiate counting. After 10 or 15 seconds, a well-defined photo peak will develop across channels in the region of 662 keV. Using the MCA software, display the pre-programmed energy peak markers for Cs-137 from the pull down menu of radionuclides (these markers are assigned to specific MCA channels based on the initial system energy calibration as established by the Field Program Director). Adjust the fine gain setting (±) until the centroid of the Cs-137 photo peak lines up perfectly with the Cs-137 energy peak marker. Stop the counting, discard the collected energy spectrum and remove the check source from the counting well. (Note: this procedure is specific to the URSA-II MCA and associated software. If a different MCA system is used, the specifics of the procedure may differ somewhat and the procedure will be updated accordingly to accomplish the same purpose).

3. Count the two secondary soil Ra-226 reference material standards that will serve as dedicated daily quality control (QC) checks for the system [one "background" level standard (near 1-2 pCi/g) and one higher level standard (in the range of 15-20 pCi/g) for the preset sample counting time]. For each standard, record the sum of the counts from the three ROIs on the system QC chart spreadsheet and update the QC chart. If results fall within the upper and lower system control limits (with ± 3 standard deviations of the mean of all previous QC measurements) on each QC chart, the system is working properly. If not, contact the Field Supervisor and/or Field Program Director to troubleshoot and resolve any problems with the sample counting system.

4. After sample preparation (see AS-SOP 3), count each field sample for the pre-set counting time on the same day it is sealed. Enter the sample ID, sample weight, sample collection and seal dates, along with the number of counts for each ROI in the sample analysis spreadsheet. The "full radon ingrowth" estimate of the Ra-226 concentration will be automatically calculated based on the count/activity calibration equation as established by the Field Program Director.
5. Remove the sample from the counting well and repeat the counting process for the next sample.
6. Every 2-3 hours, the system must be checked for spectral drift that can occur due to temperature changes in the counting room. If drift is apparent, the fine gain setting must be adjusted to keep the system energy calibrated throughout the day (the procedure is the same as indicated in step 2 above).
7. For samples that have been counted, follow the provisions of AS-SOP 3 for sample storage and/or shipping to an offsite lab for additional analysis.

7.0 DATA QUALITY ASSURANCE/QUALITY CONTROL

Data quality assurance (QA) for onsite analysis of Ra-226 concentrations in solid samples using gamma spectroscopy will be addressed by carefully following the methods, specifications and procedures indicated in this SOP and in the QAPP (Attachment S2). Data quality control (QC) will be addressed by daily QC measurements and monitoring for spectral drift as described in Section 6.0. The performance, accuracy and reliability of the sample counting procedures being implemented by onsite soils lab staff may be subject to internal and/or external audit as described in the QAPP.

8.0 HEALTH AND SAFETY

Standard worker health and safety requirements for all onsite workers must be observed when performing sample counting as described in this SOP. These requirements are defined in the current Health and Safety Plan (HASP) and Radiation Protection Plan (RPP) (Tetra Tech, 2009; SENES, 2013c).

9.0 REFERENCES

MWH Americas (MWH), 2013a. Basis of Design Report, Preliminary (60 Percent) Design, Midnite Mine Superfund Site. December 16.

MWH Americas (MWH), 2013b. Attachment S3 – Determination of Bedrock during Remedial Excavation. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013). Memo from MWH: Defining Rock Excavation during RA Construction at the Midnite Mine Superfund Site. July 12, 2013.

SENES Consultants (SENES), 2013a. Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments. December, 2013. Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013b. Attachment S1 – Technical Basis. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013c. Midnite Mine Superfund Site – Radiation Protection Plan for Remedial Action. 60 Percent Design. December 12, 2013.

Tetra Tech, Inc., 2009. Health and Safety Plan for the Phase I RD/RA: Interim Water Management for the Midnite Mine. Revision 1.0. Prepared for Dawn Mining Company and Newmont USA Limited. May 5.

Worthington Miller Environmental, LLC. (WME) and SENES Consultants (SENES), 2013. Attachment S2 – Quality Assurance Project Plan (QAPP), Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

MIDNITE MINE SUPERFUND SITE REMEDIAL ACTION PROJECT STANDARD OPERATING PROCEDURE	**AS-SOP 5** **REVISION: 1**
FIELD PORTABLE X-RAY FLUORESCENCE PROCEDURES	**DATE: June 16, 2015**

CONTENTS

1.0 PURPOSE AND SCOPE 1

2.0 APPLICABLE REFERENCE DOCUMENTS 1

3.0 RESPONSIBILITIES AND QUALIFICATIONS 1

4.0 HEALTH AND SAFETY ISSUES 2

5.0 RELATED STANDARD OPERATING PROCEDURES 3

6.0 PROCEDURES 3

6.1 XRF CALIBRATION AND PREPARATION 3
6.2 FIELD XRF PROCEDURES 3
6.3 INTRUSIVE XRF PROCEDURES 4

7.0 DOCUMENTATION 6

8.0 QUALITY ASSURANCE/QUALITY CONTROL 7

8.1 QUALITY CONTROL SAMPLES 7

9.0 SAMPLE STORAGE, HANDLING AND CUSTODY 9

10.0 REFERENCES 9

ATTACHMENTS

Attachment 1 EPA Method 6200, Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Revision 0, February 2007

Attachment 2 ThermoFisher Scientific Inc. User's Guide for the ThermoFisher Scientific Niton XL3t GOLDD+

1.0 PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure is to outline the proper procedures for use with a field portable X-Ray Fluorescence (XRF) Analyzer in support of XRF measurements and sample collection and analysis described in the 60% Remedial Design, Appendix S Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments.

The following procedures describe the protocol for performing in-situ and intrusive analysis of soil using a field-portable, ThermoFisher Scientific Niton XL3t GOLDD+ (or equivalent model) XRF Analyzer, in accordance with EPA Method 6200, Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Revision 0, February 2007 (Attachment 1). The Niton XL3t instrument uses a 50 kV X-Ray tube source for the analysis of inorganic metal concentrations. These procedures will also be used in conjunction with the ThermoFisher Scientific Inc. User's Guide for the instrument (Attachment 2). Any changes or modifications to these procedures will be documented by the Field Technician and approved by the Field Program Director.

2.0 APPLICABLE REFERENCE DOCUMENTS

The procedures in this SOP pertain to Appendix S of the Preliminary (60 Percent) Basis of Design Report for remedial action at the Midnite Mine Superfund Site (MWH, 2013a). Respective citations in this SOP (where applicable) include documents that fall under the following organization and reference structure of Appendix S:

Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (SENES, 2013a)

- Attachment S1 – Technical Basis (SENES, 2013b)
- Attachment S2 – Quality Assurance Project Plan (QAPP) (WME/SENES, 2013)
    - Appendix 1 – Standard Operating Procedures (SOPs)
        - AS-SOP 1 – Decontamination for Field Sampling
        - AS-SOP 2 – Surface Material and Sediment Sampling
        - AS-SOP 3 – Sampling Processing
        - AS-SOP 4 – Onsite Gamma Spectroscopy
        - AS-SOP 5 – Field-portable XRF Procedures
        - AS-SOP 6 – Gamma Surveys
    - Appendix 2 – Corrective Action Report Form
    - Appendix 3 – Approved Laboratory Quality Assurance Plans
    - Appendix 4 – Laboratory Certification

Attachment S3 – Determination of Bedrock during Remedial Excavation (MWH, 2013b)

3.0 RESPONSIBILITIES AND QUALIFICATIONS

The Field Supervisor is responsible for assuring that this and any other appropriate procedures are followed by all project personnel. The project staff assigned to use field portable XRF and collect related samples are responsible for completing their measurements according to this and other

appropriate procedures. Only qualified personnel will be allowed to perform these procedures. Qualifications are based on education, previous experience, and on-the-job training and supervision by another qualified person.

4.0 HEALTH AND SAFETY ISSUES

Proper training for safe use of the instrument and radiation training will be completed by the user prior to use of the instrument. Information and procedures contained herein are specific to the operation of the Niton XRF Analyzer. The user will also refer to the user's manual for the Niton XL3t XRF Analyzer instrument for proper operation of that instrument (Attachment 2). The instrument user should also be aware of local, state and national regulations that pertain to the use and storage of radiation producing equipment and radioactive materials. Compliance with all applicable regulations is required.

The best precaution to prevent radiation exposure is distance and shielding. Safety precautions for use of the XRF instrument are as follows:

- Never point the XRF at yourself or anybody else with the shutter open
- Stand to the rear or side of the XRF when the shutter is open. Do not operate the instrument in a seated position; this may expose your lower body to radiation.
- Do not fix the shutter in an open position (except in provided test stands)
- Do not leave the XRF unattended
- Only trained people will operate an XRF
- Open the shutter only with the sample in place
- Never open the probe
- Store the XRF in a safe place. Do not drop the machine (or put the instrument in a position where it will be likely to be dropped).
- Wear a dosimeter badge (if required)
- Perform wipe tests, per manufacturer's instructions
- Women of child bearing age should be aware of the potential damage to a developing fetus from radiation exposure
- Transport XRF in a shock-proof case
- Follow all manufacturer's training and instructions

OSHA exposure limits are presented below.

Whole body exposure:	5,000 mrem/yr	1,250 mrem/quarter
Extremities:	50,000 mrem/yr	18,750 mrem/quarter

Some states have specified lower limits for public exposure. The lowest exposure limits were found to be 100 mrem/yr and 1 mrem/day.

More detailed information and procedures are contained in EPA Method 6200, Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment,

February 2007 (Attachment 1).

5.0 RELATED STANDARD OPERATING PROCEDURES

This procedure is intended to be used with the following SOPs:

- AS-SOP 1 Decontamination for Field Sampling
- AS-SOP 2 Surface Material and Sediment Sampling
- AS-SOP 3 Sample Processing

6.0 PROCEDURES

This section provides procedures for two types of measurement of metal concentrations with the portable XRF instrument according to procedures recommended in EPA Method 6200. The field in-situ XRF measurements are performed directly on the soil surface in the field to provide real-time analysis of metal concentrations. The intrusive XRF measurements are performed on samples returned from the field under the controlled interior conditions of a laboratory or work space in cases where the in-situ soils have greater than 20% moisture content or are collected at depth to be analyzed.

6.1 XRF Calibration and Preparation

The XRF will be calibrated daily prior to use for each the in-situ analysis and the intrusive analysis, according to procedures in the manufacture's user guide (Attachment 2), using calibration checks with certified reference materials and field blanks as described in EPA Method 6200.

QC samples will also be analyzed prior to sample analysis, as described in Section 8.

6.2 Field XRF Procedures

The following procedures outline the steps for in-situ XRF analysis of undisturbed soils in the field.

6.2.1 <u>In-Situ XRF Measurement</u>

Attach the test guard to the XRF detector. Determine and prepare the location to be sampled. Remove any debris on the soil surface consisting of rocks, pebbles, leaves, vegetation, twigs or roots. Level and smooth the soil surface with a stainless-steel or plastic trowel so that the probe window is in direct contact with the soil surface. Lightly tamp the soil surface with the trowel to increase soil density and compactness. The soil should not be saturated or have a moisture content exceeding approximately 20%. Where soils have a moisture content of greater than 20%, a sample should be collected and dried according to Section 5.3.4 prior to XRF measurement.

When ready for analysis, press the XRF down on the soil surface, thus opening the XRF shutter. Maintain the XRF shutter open for the specified count time (60 seconds is recommended) then remove/release the XRF from the sample to stop the analysis. The

measured metal concentrations are recorded by the XRF data logger.

6.2.2 Decontamination

After every test the XRF detector shutter and the test guard will be wiped clean with tissue or wipes. If a stainless-steel trowel or other non-disposable equipment is used for the XRF in-situ measurements, decontamination procedures that are provided in AS-SOP 1 will be followed.

6.3 Intrusive XRF Procedures

The following method outlines procedures for preparing soil samples collected in the field for intrusive XRF measurements, according to procedures recommended in EPA Method 6200.

6.3.1 Supplies

General Sample Supplies:	XRF Equipment and Supplies (obtained from the XRF manufacturer):
Stainless steel bowls	XRF instrument and mini lab kit
Stainless steel trowel or spoon	Sieves (60-mesh/0.2 mm)
Disposable plastic spoons	Mortar and pestle (ceramic)
Paper towels	Polyethylene sample cups, collar, and bottom
Toaster oven	X-ray window film (Mylar, or similar)
Deionized water	
Alconox detergent (or similar)	
Scrub brushes	
Sample bags/containers	
Field logbook	

6.3.2 Sample Collection and Preparation

Soil samples will be collected in such a manner that the sample is representative of the soil matrix analyzed at the field in-situ XRF measurement location. Sample collection procedures are described in AS-SOP 2. A minimum of 350 grams of surface soil or sediment will be collected to a depth of six inches. A larger sample volume may be required to provide a sufficient sample for drying depending on the soil texture and moisture content, and if necessary splitting for onsite soil laboratory analysis, offsite laboratory analysis, or quality control (QC) testing. The sample will be processed according to procedures outlined in AS-SOP 3.

6.3.3 Drying the Sample

If the sample is visibly wet, the sample will be air-dried or dried in a conventional or toaster oven at a temperature no greater than 150 degrees Celsius. A microwave oven will not be used to dry the sample. If the sample is air-dried, it will be allowed to dry in a protected

environment to prevent contamination by dust deposition. The sample will be inspected for any remaining foreign debris (rocks, metal, wood, etc.); any such debris will be removed. Drying time may need adjustment, depending on the initial moisture content of the sample.

6.3.4 Grinding and Sieving

Once the sample is dried, transfer a split of the sample to a 60-mesh sieve to obtain a sample of uniform particle size. After sieving transfer the material retained on the 60-mesh sieve to the mortar and pestle and grind until soil particles are broken up. Once the sample is ground, pass the sample again through a 60-mesh sieve. The material retained on the sieve after it has been ground and sieved a second time shall be discarded. If more processed material is needed for data analysis the above process shall be repeated with another split of the original sample. In order to reduce variation between collected samples and calibration standards the same sieve size should be used to prepare calibration standards and all collected samples.

6.3.5 Sample Cups

Prepare the polyethylene sample cup by placing a piece of Mylar X-ray film over the sample cup body. Next, secure the film to the body by pressing the polyethylene collar down over the Mylar film and cup body. Make sure that the film has been “stretched” taut (no slack) and there are no wrinkles on the window portion of the film. Turn the sample cup over so the film is resting on the table. Next, transfer the ground and sieved sample to the cup to a level where a minimum of 3/8-inches of sample is contained in the cup. If the sample size is not large enough to fill the cup, place a small piece of filter paper over the soil and then fill the remaining area with polyethylene cotton. The cotton should take up any remaining space in the cup so that the sample will stay pressed against the Mylar film. Next, press the cup bottom on the sample cup so that it lies flush against the cup body. Write the sample identification number on the cup bottom with an indelible marker. Next, turn the sample cup over. There should be neither slack in the Mylar film nor space between the Mylar film and the soil sample in the cup. The sample is ready for XRF analysis.

6.3.6 XRF Analysis

Place the sample cup in the soil test platform with the Mylar film/soil side up. Next place the XRF detector on the soil test platform. When ready for analysis, depress the XRF into the test stand, opening the shutter. The soil test platform has a lock down devise and will hold the XRF in place. Keep the XRF shutter open for a specified count time (60 seconds is recommended) then remove/release the XRF from the sample to stop the analysis. Count times are the seconds the sample is analyzed. The same count time should be used for calibration standards and samples for the same matrix. Count times may vary depending on the required results and may range from 20 seconds to over 600 seconds. The longer the count time the lower the detection limit obtained. Record the results in the field logbook

for the analytes of interest, including the +/- variance value.

6.3.7 Decontamination

Any reusable equipment will be decontaminated prior to reuse, according to AS-SOP 1 Decontamination. Equipment, including stainless steel bowls, mortar and pestle, sieves, reusable trowels or spoons, etc. shall be decontaminated prior to reuse. Decontamination procedures will consist of wiping with a clean paper towel or dry brushing loose soil from each piece of equipment. Next, rinse and/or scrub equipment with a DI and Alconox mixture using a clean scrub brush. Rinse with DI or distilled water, and then wipe dry with clean paper towels or air dry. If air drying is used, ensure the area is clean and away from areas where recontamination by air deposition is possible. Store equipment in plastic or other protective covering to keep clean. The XRF detector and soil test platform may be wiped with a clean paper towel. The work area should be kept clean and clear of unnecessary equipment at all times. It is recommended that plastic be used to cover the work surface so that it may easily be replaced with new and clean plastic whenever necessary.

7.0 DOCUMENTATION

A field logbook will be maintained to document all sampling activities. All notes will be made in indelible ink. Entries on each page will be initialed at the end of each page by the sampling crew member who entered the information. If any changes are made to the record, the original notation will be crossed out with a single line and initialed. At each measurement location, the following information will be recorded in the field logbook or on a designated field form:

- Names of field personnel
- Date/time of measurement
- Measurement location identifier
- Global Positioning System (GPS) coordinates
- General weather conditions (e.g., hot, windy, no precipitation)
- Surface material description, including color and texture (e.g., red-brown, sandy silt with occasional gravel) and relative moisture content (e.g., dry, moist, wet)
- Description and location of stormwater drainage paths, if present near the measurement location
- General description of vegetation conditions
- Sample identification, sample collection location, and collection time, if sample is collected
- Any problems encountered or deviations in XRF operation or sample collection methods
- Description of any unusual circumstances
- Photo documentation details, if necessary

In addition, the following information will be recorded in the field logbook at least on a daily basis to document the field in-situ XRF measurements:

- XRF make and model number
- Documentation and results of instrument performance checks
- Certified reference materials (NIST standards) and blanks used for calibration purposes
- Site-specific calibration standards used
- Any problems encountered with instrument setup and operation
- QC samples, their origin and type
- Samples collected for intrusive XRF analysis
- Sample preparation method, sieve size used, if any
- Confirmation samples submitted to the laboratory for analyses
- Any problems encountered in instrument set up and operation or sample preparation and analysis

8.0 QUALITY ASSURANCE/QUALITY CONTROL

QA/QC measures include analyses of QC samples in order to describe precision and accuracy of field in-situ and intrusive XRF data. Definitions of precision and accuracy are included in the QAPP under Measurement Performance Criteria. Calibration check samples, soil blanks and certified reference material (NIST standards) will be provided with the Niton XRF instrument. NIST soil standards should include a low, medium, and high range of analyte concentrations. The NIST standard and soil blanks provided by Niton will be measured every time the instrument is calibrated to ensure the accuracy the XRF.

8.1 Quality Control Samples

The following QC samples will be run according to EPA Method 6200.

- Energy calibration check sample
- Instrument blank
- Method blank
- Calibration verification checks (NIST standard)
- Precision measurements
- Confirmation samples

8.1.1 Energy Calibration Check

An energy calibration check sample determines whether the characteristic x-ray lines are shifting, which would indicate drift within the instrument. Energy calibration check samples should be run according to manufacturer's recommendations as indicated in the user's manual. Generally this would be the beginning of each working day, after the batteries are changed or the instrument turned off, at the end of the working day, and at any other time when the instrument operator believes that drift is occurring during analysis.

8.1.2 Instrument Blank

The instrument blank is used to verify that no contamination exists in the spectrophotometer or in the probe window. The instrument blank can consist of silicon dioxide, PTFE block, a quartz block, "clean" sand or lithium carbonate. The instrument blank should be analyzed on each working day before and after analysis are conducted, and once per 20 samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. No element concentrations above the established lower limit of detection (LLD) should be found in the instrument blank.

8.1.3 Method Blank

The method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed a least once daily. The method blank should be less than the LLD or < 10% of the lowest sample concentration for the analyte, whichever is greater.

8.1.4 Calibration Verification Check

Calibration verification check samples are used to check the accuracy of the instrument. If a site-specific check sample is not available, than a NIST certified standard reference material that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within +/- 20% of the true value. A check sample should be analyzed at the beginning of each working day, during sample analysis, and at the end of the working day.

8.1.5 Precision Measurements

Duplicate in-situ and intrusive XRF measurements for precision will be done as a minimum of one per day, measured 7x in replicate, per Method 6200. The relative standard deviation (RSD) of the sample mean is used to assess method precision. The RSD should be < 20 % for metals (< 30% for chromium). The equation for calculating the RSD is:

$$RSD = (SD/\text{Mean conc.}) \times 100$$

where:
SD = Standard deviation of the concentration for the analyte
Mean conc. = mean concentration for the analyte

8.1.6 Confirmation Samples

Confirmation samples taken from the intrusive samples are to be sent to off-site analytical laboratory should be taken at a minimum of 1 sample for each 20 intrusive prepared samples. The confirmatory samples will be splits of the homogenized material that has passed through a 60-mesh sieve; in most cases this would be the soil cups used for the intrusive analysis. The correlation coefficient (r) calculated for the results of the intrusive XRF data vs. the confirmatory laboratory analysis should be 0.7 or greater for the XRF data to be screening level data. If the r is 0.9 or greater and inferential statistics indicate the XRF data and the confirmatory data are statistically equivalent at a 99% confidence level, the

data could meet definitive level data criteria.

9.0 SAMPLE STORAGE, HANDLING AND CUSTODY

Samples collected in the field for intrusive analysis will be stored according to AS-SOP 3 under secured conditions at the onsite laboratory or other designated secure storage area before and during analysis. Confirmatory samples collected for off-site laboratory analysis will be handled, preserved, and shipped to the analytical laboratory under strict chain-of-custody protocols according to the procedures outlined in the project QAPP.

10.0 REFERENCES

MWH Americas (MWH), 2013a. Basis of Design Report, Preliminary (60 Percent) Design, Midnite Mine Superfund Site. December 16.

MWH Americas (MWH), 2013b. Attachment S3 – Determination of Bedrock during Remedial Excavation. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013). Memo from MWH: Defining Rock Excavation during RA Construction at the Midnite Mine Superfund Site. July 12, 2013.

SENES Consultants (SENES), 2013a. Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments. December, 2013. Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013b. Attachment S1 – Technical Basis. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013c. Midnite Mine Superfund Site – Radiation Protection Plan for Remedial Action. 60 Percent Design. December 12, 2013.

Tetra Tech, Inc., 2009. Health and Safety Plan for the Phase I RD/RA: Interim Water Management for the Midnite Mine. Revision 1.0. Prepared for Dawn Mining Company and Newmont USA Limited. May 5.

Worthington Miller Environmental, LLC. (WME) and SENES Consultants (SENES), 2013. Attachment S2 – Quality Assurance Project Plan (QAPP), Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

Attachment 1

EPA Method 6200, Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Revision 0, February 2007

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples. The three electron shells include the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α), beta (β), or gamma (γ) etc., which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than

the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments, specifically, in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

3.6 SRM -- Standard reference material; a standard containing certified amounts of metals in soil or sediment.

3.7 eV -- Electron volt; a unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup (i.e., against the cup window), the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95 and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As K_β line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as Method 3050, or a total digestion procedure, such as Method 3052, is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project-specific data quality objectives (DQOs).

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the DQOs of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Sec. 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10° F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) and leak tests should be present with the instrument at all times and available to local and national authorities upon request.

X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs or badges should be worn in the area of maximum exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 FPXRF spectrometer -- An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation sources -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron Fe-55 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of

accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample presentation device -- FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors -- The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 L. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the

units or from PCs. Once the data–storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film -- Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 µm thick.

6.5 Mortar and pestle -- Glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers -- Glass or plastic to store samples.

6.7 Sieves -- 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels -- For smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags -- Used for collection and homogenization of soil samples.

6.10 Drying oven -- Standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Pure element standards -- Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if designated for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.3 Site-specific calibration standards -- Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 °C. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 g of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.4 Blank samples -- The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Follow the manufacturer’s instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting,

which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.2 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank samples -- Two types of blank samples should be analyzed for FPXRF analysis, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetraflurorethylene (PTFE) block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the established lower limit of detection should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration verification checks -- A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision measurements -- The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

The equation for calculating RSD is as follows:

$$RSD = (SD/\text{Mean Concentration}) \times 100$$

where:

RSD	=	Relative standard deviation for the precision measurement for the analyte
SD	=	Standard deviation of the concentration for the analyte
Mean concentration	=	Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

9.7 Confirmatory samples -- The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on project-specific data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument calibration -- Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments, namely: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental parameters calibration -- FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are necessary, although site-specific samples with confirmed and validated analytical results for all elements present could be used.

- Cost is reduced because fewer confirmatory laboratory results or calibration standards are necessary.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Sec. 7.3. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective energy FP calibration -- The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference
C_k = Certified concentration of standard sample
C_s = Measured concentration of standard sample

10.2.2 BFP calibration -- BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended

count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical calibration -- An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Sec. 7.3; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is necessary. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are necessary to perform an adequate empirical calibration. The exact number of standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton normalization method -- The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline reading. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_α peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later during analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide example performance data for this method, this modest amount of sample preparation was found to take less than 5 min per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for in situ analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only “screening” type data.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 g or 250 cm^3, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Sec. 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 °C. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 min per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from "nondetect" to tens of thousands of mg/kg. These data are provided for guidance purposes only.

13.3 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode

detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.4 All example data presented in Tables 4 through 8 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from "nondetects" to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the in situ homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square,

measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy measurements -- Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 6. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability -- Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4–intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 8 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 8 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Sec. 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer," Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, February 24-26, 1993, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex, X-MET 920 User's Manual.

2. Spectrace Instruments, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.

3. TN Spectrace, Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, received from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: Refs. 1, 2, and 3
These data are provided for guidance purposes only.

TABLE 2

SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Refs. 1, 2, and 3

TABLE 3

SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Ref. 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4

EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84[a]	NR	24.80[a]	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85[a]	NR	24.92[a]	20.92[a]	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69[a]	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32[a]	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These data are provided for guidance purposes only.
Source: Ref. 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

These data are provided for guidance purposes only.
Source: Ref. 4

[a] These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.

ND Not detected.

NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Ref. 4. These data are provided for guidance purposes only.
n: Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD: Standard deviation; NA: Not applicable; only two data points, therefore, a SD was not calculated.
%Rec.: Percent recovery.
-- No data.

TABLE 7

EXAMPLE ACCURACY FOR TN 9000[a]

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Ref. 4. These data are provided for guidance purposes only.
[a] All concentrations in milligrams per kilogram.
%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.
-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY[1]

	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

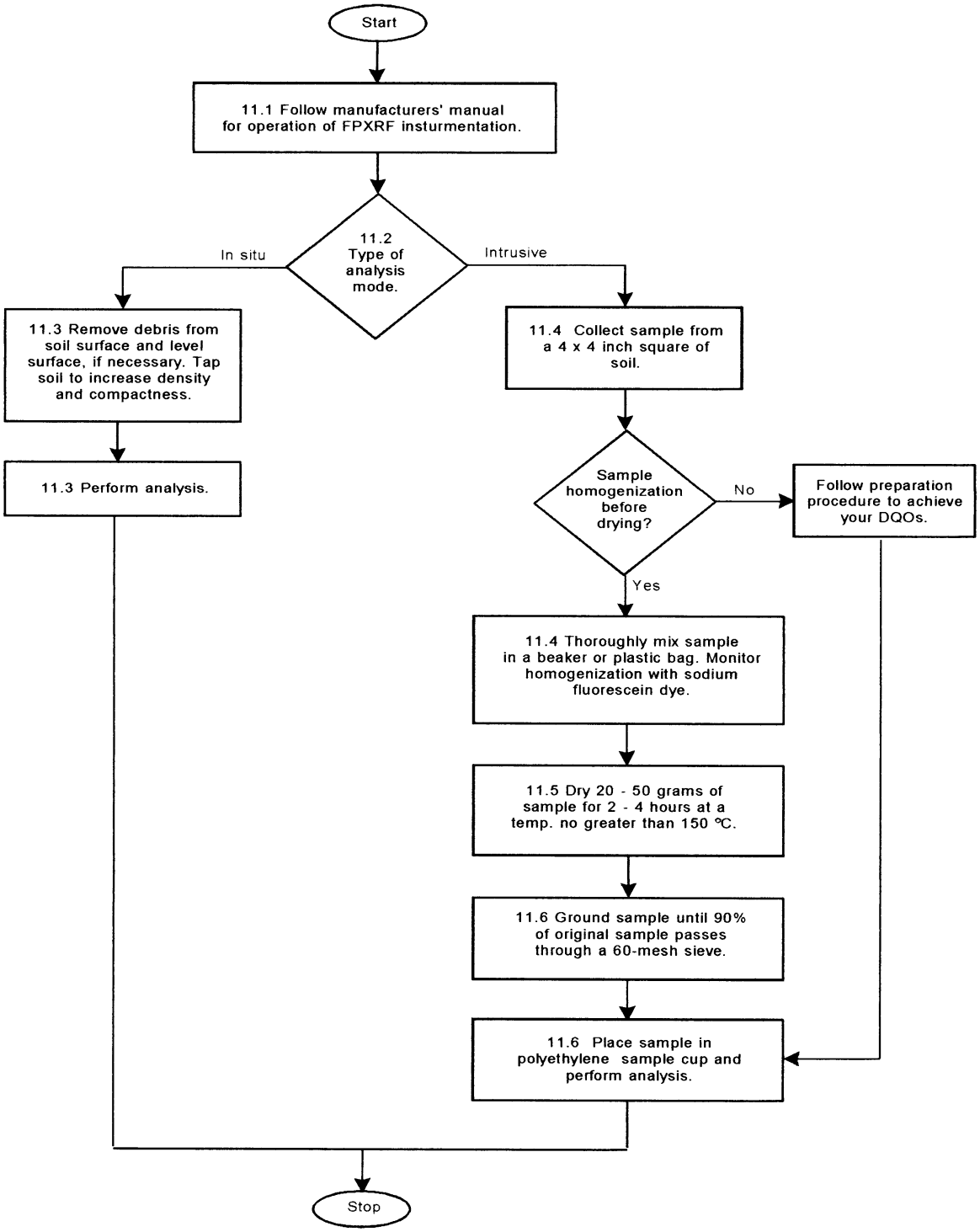
Source: Ref. 4. These data are provided for guidance purposes only.

[1] Log-transformed data

n: Number of data points; r^2: Coefficient of determination; Int.: Y-intercept

— No applicable data

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



Attachment 2

User's Guide for the ThermoFisher Scientific Niton XL3t GOLDD+

Due to its size, this user's guide is included in the electronic submittal only.

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MIDNITE MINE SUPERFUND SITE REMEDIAL ACTION PROJECT STANDARD OPERATING PROCEDURE	**AS-SOP 6** **REVISION: 1**
GAMMA SURVEYS	**DATE: June 16, 2015**

CONTENTS

1.0 PURPOSE **1**

2.0 APPLICABLE REFERENCE DOCUMENTS **1**

3.0 RESPONSIBILITY **1**

4.0 PRECAUTIONS **2**

5.0 EQUIPMENT AND MATERIALS **2**

6.0 PROCEDURE **3**

6.1 GAMMA SURVEY AREA PLANNING 3
6.2 SCAN SYSTEM MOUNTING CONFIGURATION 3
6.3 PRE-SURVEY QC MEASUREMENTS 3
6.4 FIELD GAMMA SCANNING 4

7.0 QUALITY ASSURANCE/QUALITY CONTROL **4**

8.0 HEALTH AND SAFETY **4**

9.0 REFERENCES **5**

1.0 PURPOSE

Gamma surveys will be performed to help guide remedial excavation of across mine-impacted areas and mine drainages and to help evaluate compliance with ROD cleanup levels during final status surveys. This standard operating procedure (SOP) provides the methods to be used for gamma scanning in accordance with the Remedial Support and Verification Plan (Appendix S). It describes the equipment, field procedures, data logging (where required), and quality assurance/quality control (QA/QC) protocols to be employed when conducting gamma scanning.

2.0 APPLICABLE REFERENCE DOCUMENTS

The procedures in this SOP pertain to Appendix S of the Preliminary (60 Percent) Basis of Design Report for remedial action at the Midnite Mine Superfund Site (MWH, 2013a). Respective citations in this SOP (where applicable) include documents that fall under the following organization and reference structure of Appendix S:

Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (SENES, 2013a)

- Attachment S1 – Technical Basis (SENES, 2013b)
- Attachment S2 – Quality Assurance Project Plan (QAPP) (WME/SENES, 2013)
    - Appendix 1 – Standard Operating Procedures (SOPs)
        - AS-SOP 1 – Decontamination for Field Sampling
        - AS-SOP 2 – Surface Material and Sediment Sampling
        - AS-SOP 3 – Sampling Processing
        - AS-SOP 4 – Onsite Gamma Spectroscopy
        - AS-SOP 5 – Field-portable XRF Procedures
        - AS-SOP 6 – Gamma Surveys
    - Appendix 2 – Corrective Action Report Form
    - Appendix 3 – Approved Laboratory Quality Assurance Plans
    - Appendix 4 – Laboratory Certification
- Attachment S3 – Determination of Bedrock during Remedial Excavation (MWH, 2013b)

3.0 RESPONSIBILITY

The Field Technician is responsible for performing gamma surveys in accordance with the methods, specifications and procedures indicated in Appendix S, the QAPP (Attachment S2), in this SOP, and in accordance with the direction of the Field Supervisor and/or Field Program Director. It is the responsibility of the Field Supervisor to manage the gamma survey program and to ensure that the data collected will meet the data quality objectives for the project. All personnel working onsite are responsible for adherence to the current project Health and Safety Plan (HASP) and Radiation Protection Plan (RPP) (Tetra Tech, 2009; SENES, 2013c). Additional general information regarding project personnel and respective responsibilities is provided in the QAPP (Attachment S2).

4.0 PRECAUTIONS

- "Official" gamma scanning (i.e. recorded surveys) should not be conducted during or immediately following precipitation events or when the ground is significantly wetted (e.g. when rainfall is sufficient to cause muddy conditions). Precipitation can cause atmospheric scrubbing of airborne radon decay products and temporarily elevate gamma readings in a manner that is not representative of soil radionuclide concentrations. Conversely, high soil moisture can slightly shield gamma emissions from in-situ soil radionuclides.

- Gamma scanning will often require traversing rough, uneven terrain and loose surface materials on foot while carrying considerable equipment. Slips and falls are a continual risk and serious injury and/or damage to equipment can occur. Scanning can be physically demanding. Hot weather conditions can increase risk of dehydration and can lead to heat stress. Working in the vicinity of heavy operating equipment, as well as alone in remote areas of the Site, can create the potential for additional health and safety hazards. These issues are discussed in Section 8.0 and respective cautions and procedures, along with all Site safety rules and precautions as detailed in the current HASP and RPP (Tetra Tech, 2009; SENES, 2013c) must be observed.

5.0 EQUIPMENT AND MATERIALS

The following equipment and supplies may be required during gamma scanning, depending on whether the scanning is for remedial support purposes (generally unrecorded) or for final status survey purposes (always recorded).

- Ludlum Model 44-10 2x2" NaI Scintillation detector
- Ludlum Model 2350 Rate Meter/Scalar
- Coaxial cable
- RS-232 connector cable and USB adaptor (if applicable)
- Gamma scanning computer with scanning software
- Computer carrying case (shoulder harnessed)
- WAAS-enabled GPS receiver
- Rigid framed backpack, assorted bungee cords
- Paint can holder with extension wand (example at right) to mount detector to backpack
- Electrical tape
- Field Logbook
- First Aid Kit
- Bottled water
- 2-way radio

6.0 PROCEDURE

6.1 Gamma Survey Area Planning

For remedial support scanning activities, the gamma survey area will generally be unrecorded and conducted in the immediate vicinity of remedial excavation activities (see Appendix S). These surveys will only involve use of a gamma detector as the purpose is only to determine when excavation has met the gamma cutoff value and may be ready for final status surveys. In addition, early in the cleanup, the Field Supervisor and/or Field Program Director will direct recorded gamma surveys in outlying areas of the site for further evaluation of potential impacts and refinement of the boundaries for Class 1 and Class 2 areas. For final status surveys, the survey area will include a given and previously defined survey unit upon determination that the survey unit is ready for final status surveys. Further specification of gamma survey areas and target coverage is detailed in Appendix S.

6.2 Scan System Mounting Configuration

A photo of an example gamma survey system mounting configuration for conducting recorded, GPS-based backpack scanning is shown in Figure 1. Using bungee cords, zip ties, electrical tape or other means as appropriate, mount the paint can holder/extension wand, detector, rate meter, and GPS receiver in a manner similar to that shown in Figure 1. The mounting configuration and system to be used for walkover surveys at the Midnite mine may differ somewhat from that shown in Figure 1, but the detector height must remain at about 1 meter above the detector, rate meter and GPS specifications indicated in section 4.0 are mandatory. The Ludlum 2350 rate meter must be programmed to provide integrated readings every 1 second, and the data acquisition software should be capable of capturing paired gamma/GPS readings every 1-2 seconds (the data capture rate for these systems can vary slightly depending on computer processing speed). It is possible that a small all-terrain vehicle (ATV) may alternatively be used for recorded gamma scanning (depending on terrain) but again, the detector height must remain the same and for all final status surveys, scan speed must be conducted at typical walking speed (2-3 mph).

Figure 3: Backpack gamma scanning system.

6.3 Pre-survey QC measurements

The technical details and requirements for gamma survey QC measurements are provided in the QAPP. Respective procedures are as follows:

1. Prior to initiation of any field work involving gamma surveys on any given day, first check the voltage on the 2350 rate meters to be used. If the voltage is below about 5.5 volts on any instrument, replace the batteries with fresh ones as loss of battery power for more than several minutes will result in a need to reprogram the instrument.
2. Also for each detector/rate meter pairing to be used in the field, determine the mean of 10-20 individual readings of ambient background radiation, as well as radiation from a designated gamma check-source (e.g. the Cs-137 check source for gamma spectroscopy, see AS-SOP 4), as measured at a permanently designated location (and fixed counting geometry) inside of the onsite soils lab. For each detector, record the date, detector ID number, individual readings, and mean readings on the instrument QC log sheet that will be maintained in the lab. Mean values must fall within the control limits indicated on the current QC log sheet.
3. Prior to any recorded gamma surveys, perform a test gamma scan along a designated field strip (about 20 meters in length) outside the soils lab (see QAPP for further details). Either save the field strip scan data to the scan computer as a separate file for later data processing and analysis, or if the software has an automated averaging algorithm, this function can be used to automatically calculate the average reading along the field strip. In the latter case, record the date, detector ID number, and mean field strip reading in the field logbook.

6.4 Field Gamma Scanning

Perform gamma surveys in accordance with the methods, specifications and procedures indicated in Appendix S, and in accordance with the direction of the Field Supervisor and/or Field Program Director.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

All NaI detector/rate meter pairings must have been properly calibrated against a Cs-137 calibration source at a qualified calibration facility within one year prior to use on the project (calibration certificates must be retained in the project files). Calibrated detector/rate meter pairings must remain paired when used at the site ("swapping" a detector with a different rate meter would invalidate subsequent data). In addition, data QA/QC will be addressed by following all methods, specifications and procedures indicated in Appendix S, the QAPP (Attachment S2), and in this SOP. Adherence to these specifications may be subject to internal and/or external audit as described in the QAPP.

8.0 HEALTH AND SAFETY

Gamma scanning will often involve traversing rough, uneven terrain and loose surface materials on foot. Slips and falls are a continual risk and serious injury and/or damage to equipment can occur. Personal protective equipment (work boots, rugged field clothing, leather gloves and safety glasses) must be worn at all times and great care should be taken to move slowly and deliberately across potentially dangerous terrain. Safety must be prioritized over target scan

coverage in all circumstances.

When scanning in the vicinity of heavy operating equipment, hard hats must also be worn at all times. When scanning away from heavy operating equipment, hard hats will not protect against head injuries from falls, and may create greater risk of heat stress in hot weather conditions. Scanning across rugged terrain is a physically demanding activity and it is important to stay well hydrated, especially in hot weather conditions.

Professional judgment should be used to anticipate the circumstances and conditions that can reasonably be expected during scanning and to take the necessary steps to minimize potential safety hazards accordingly. Other field staff must be notified of planned scan areas, expected duration of scanning (especially when scanning in remote areas of the Site), and a 2-way radio should always be carried when scanning alone in remote areas of the Site. These and other safety precautions as detailed in the current HASP and RPP (Tetra Tech, 2009; SENES, 2013c) must be observed.

9.0 REFERENCES

MWH Americas (MWH), 2013a. Basis of Design Report, Preliminary (60 Percent) Design, Midnite Mine Superfund Site. December 16.

MWH Americas (MWH), 2013b. Attachment S3 – Determination of Bedrock during Remedial Excavation. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013). Memo from MWH: Defining Rock Excavation during RA Construction at the Midnite Mine Superfund Site. July 12, 2013.

SENES Consultants (SENES), 2013a. Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments. December, 2013. Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013b. Attachment S1 – Technical Basis. Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

SENES Consultants (SENES), 2013c. Midnite Mine Superfund Site – Radiation Protection Plan for Remedial Action. 60 Percent Design. December 12, 2013.

Tetra Tech, Inc., 2009. Health and Safety Plan for the Phase I RD/RA: Interim Water Management for the Midnite Mine. Revision 1.0. Prepared for Dawn Mining Company and Newmont USA Limited. May 5.

Worthington Miller Environmental, LLC. (WME) and SENES Consultants (SENES), 2013. Attachment S2 – Quality Assurance Project Plan (QAPP), Appendix S (SENES, 2013a), Basis of Design Report, Preliminary 60 Design (MWH, 2013).

Appendix 2
Corrective Action Report Form

CORRECTIVE ACTION REPORT (CAR)

Section1: (to be filled out by the Initiator)

Project/Location: _____ OPEN DATE: _____

Activity/Instrument: _____ Samples Affected: _____

Description of Non-Compliance (attach extra pages, data, chromatograms, etc., as necessary):

Signature: _____ Date: _____

Section 2: (To Project QA Manager)

Corrective Action:

Is data acceptable? Why?

Signature: _____ Date: _____

Section 3: (To Project Manager)

A) The variance is being handled to my satisfaction. Yes _____ No

B) Data will be accepted. Yes _____ No

C) Client Contacted. Yes _____ No

Contact: _____ Date: _____ Time: _____

D) Actions to be taken:

Signature: _____ Date: _____

Section 4: (To Project QA Manager)

Follow-up/Discussion:

Signature: _____ Date: _____

Appendix 3
Approved Laboratory Quality Assurance Plans

Due to their size, the Laboratory Quality Assurance Plans are included in the electronic submittal only.

UNCONTROLLED

ACZ Laboratories, Inc.

QUALITY ASSURANCE PLAN
v.17

Effective Date: May 22, 2014

Last Review: May, 2014

APPROVAL AUTHORITIES[1]:

Christopher Wakefield, President/CEO
Reserve Technical Director – Organic Chemistry

Billy Grimes, Production Manager
Reserve Technical Director – Metals, WetChemistry Manual, WetChemistry Instrument

Matt Sowards, QA Officer
Reserve Technical Director – Radiochemistry

TECHNICAL DIRECTORS & SIGNATORIES

Scott Habermehl, Senior Project Manager

Max Janicek, Project Manager

Sue Webber, Project Manager

Isaac Klaus, Technical Director – Organic Chemistry

Steve Pulford, Technical Director – Metals

Alyssa Dybala, Technical Director –WetChemistry Manual

Jennifer Fox, Technical Director – WetChemistry, Instrument

Brett Dalke, Technical Director – Geochemistry

Mark McNeal, Technical Director – Radiochemistry

[1] Emails & read receipt constitute approval signatures.

UNCONTROLLED

TABLE OF CONTENTS

1 INTRODUCTION 3
2 QUALITY SYSTEM OBJECTIVES & COMPONENTS 4
3 ETHICAL AND LEGAL RESPONSIBILITY 14
4 PERSONNEL AND RESPONSIBILITIES 15
5 TECHNICAL TRAINING 24
6 SAMPLE COLLECTION & HOLDING TIMES 26
7 SAMPLE CUSTODY & SAMPLE HANDLING 27
8 PROCUREMENT, INVENTORY & TRACEABILITY OF SUPPLIES 29
9 MAINTENANCE & CALIBRATION OF INSTRUMENTATION & EQUIPMENT 32
10 CONTROL & STORAGE OF RECORDS & DOCUMENTS 34
11 ELEMENTS OF QUALITY CONTROL 41
12 EVALUATING QUALITY CONTROL SAMPLES 48
13 VALIDATION & REVIEW OF ANALYTICAL DATA 52
14 DETECTION LEVELS 55
15 SAMPLE DILUTIONS 56
16 ERROR CORRECTION PROTOCOL 56
17 COMPUTER / AUTOMATED PROCESSES 57
18 CLIENT SERVICES 58
19 FACILITIES 61
20 RADIOCHEMISTRY 64
21 CLINICAL LABORATORY IMPROVEMENT AMENDMENTS (CLIA) 67
22 CERTIFICATIONS 67
APPENDIX A REQUIRED CONTAINER TYPE, PRESERVATION TECHNIQUES, AND HOLDING TIMES 68
APPENDIX B – FORMS FOR MANAGEMENT REVIEW OF THE QUALITY SYSTEM 70
APPENDIX C REFERENCES UTILIZED BY ACZ 74
APPENDIX D DEFINITIONS OF TERMS 75
APPENDIX E TECHNICAL DIRECTORS 82

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1 INTRODUCTION

ACZ Laboratories, Inc. is an environmental testing laboratory that provides data to clients primarily for regulatory purposes. Samples are analyzed for compliance with federal programs including the Resource Conservation Recovery Act (RCRA), Safe Drinking Water Act (SDWA), and Clean Water Act (CWA). Environmental compliance and management decisions are based on the analytical data provided, which are critical to the expenditure of large amounts of money; are important to public health safety; are important in evaluating, monitoring, and protecting the environment; and are often essential in litigation. To this effect, analytical data must always be technically sound, accurate, and legally defensible or it is useless to the end user.

An effective Quality Assurance and Quality Control program is the cornerstone of the generation of reliable analytical data. ACZ's Quality Assurance Plan (QAP) outlines the quality assurance and quality control objectives, policies, and procedures determined to be necessary to meet the requirements of the EPA, federal government entities, state agencies, other regulatory authorities, and our clients. This document provides the necessary guidelines to ensure all ACZ employees have sufficient knowledge and training to perform their job responsibilities in a manner that guarantees all data reported to ACZ's clients is accurate, reliable, technically sound, legally defensible, and impartial.

For data to be accurate, it must be of known and documented quality. The word "quality" has many different meanings, but for the purposes of environmental testing activities can be stated simply as "conformance to requirements." Conforming to requirements allows objective measurements to be applied, rather than subjective opinions, to determine when work is of good quality. *Quality control* refers to all activities that ensure accuracy (i.e. good quality) of the data. It requires action(s) to be taken and is typically included as part of the procedure. *Quality assurance* provides the records of the results obtained from the required action(s) and refers to the ability of the laboratory to demonstrate or prove to an outside party that the quality of the data is what the laboratory states it is. Quality assurance relies heavily on documentation, and to be effective, the documentation must: (1) assure the quality control procedures are being implemented as required; (2) assure the reported data reflect the sample as it was received, meaning sample mix-up was avoided, the sample was properly preserved prior to analysis, etc.; (3) facilitate traceability of an analytical result; and (4) be subjected to reasonable precautions to protect data from loss, damage, theft, and internal or external tampering.

Quality Policy Statement: To maintain an effective QA program, continually improve the quality of our environmental testing services, and consistently provide clients with technically sound and legally defensible data in a timely manner, the management of ACZ recognizes the importance of its commitment to:

- Ensuring good professional practice by well-trained and qualified employees with the necessary experience and skills to carry out their organizational functions and to meet or exceed ACZ's standards for the quality and reliability of its testing services.
- Ensuring the data provided to our clients is of known and documented quality, and is accurate and impartial.
- Ensuring that all quality assurance and quality control policies and procedures are communicated to and understood by all employees, and that they are implemented by all employees in their work.
- Ensuring that all aspects of the business operations are conducted in a manner that adheres to the TNI Standards and all of ACZ's policies and procedures documented in the QAP, SOPs, emails, memos, etc.
- Upholding the spirit and intent of ACZ's Data Integrity Program and implementing the requirements of the program.

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2 QUALITY SYSTEM OBJECTIVES & COMPONENTS

ACZ's QAP provides a framework that guides all technical staff and administrative personnel. The information presented is necessary to ensure all employees perform their duties in a manner that allows the company to achieve its objectives, thereby ensuring the precision, accuracy, completeness, and consistency of the analytical data reported to our clients. This framework is referred to as the Quality System. The Quality System encompasses every documented quality assurance (QA) and quality control (QC) policy and procedure and guides all business functions and laboratory operations by specifying standardized protocols to control both the short-term and long-term activities that influence the quality and defensibility of our testing services.

The Quality System is designed to be appropriate to the type, range, and volume of the environmental testing undertaken. The Quality System is not a static entity and must function in a manner that allows for continuous evolution of all aspects of ACZ's business when improvements have been identified and have been determined to be necessary or beneficial. ACZ management recognizes that the staff is comprised of people who possess varied experience and knowledge and can contribute valuable insight and suggestions regarding these improvements. All employees are encouraged to be involved in this process. The following six (6) key elements form the foundation of ACZ's Quality System:

- Documents & Records
- SOPs
- Training
- Audits
- Corrective Actions
- Management Review of the Quality System

2.1 Documents & Records

The entire history of any sample must be readily understood through the associated documentation. To this extent, a formal and systematic control of documents and records is necessary for accurately reconstructing all events pertaining to any sample and for guaranteeing the quality and defensibility of the data. All information relating to the laboratory facility's equipment, analytical test methods, and related laboratory activities (such as sample receipt, sample preparation, data verification and data reporting) must be documented, and all records, including those pertaining to calibration and test equipment, certificates and reports, must be maintained. Documents and records must be safely stored (protected against fire, theft, loss, deterioration, and vermin), and must be held secure and in confidence to the client for a minimum of 10 years. Refer to §10 for details regarding the storage and control of ACZ's documents and records.

2.1.1 Documents

All controlled documents are reviewed for accuracy, approved for release by authorized personnel, and properly distributed. A document control system subsequently ensures that employees use only the correct and effective version of any form, Standard Operating Procedure (SOP), or other document, which are maintained through ACZ's LabWeb intranet. LabWeb is a computerized document control system based in HTML that can be accessed from any network computer within the facility. Documents can be queried by department and then organized in several ways by clicking the appropriate header. Click on the title of the document to view it as an Adobe Acrobat (*.pdf) file. The PDF has a "read only" qualifier and does not allow changes. Users may view SOPs but the documents may not be saved to another network drive and may not be printed. Forms may be viewed and printed but may not be saved to another network drive.

All documents are categorized by department and are assigned a unique document ID that is displayed in either the header or footer section. The ID nomenclature starts with either SOP (procedure) or FRM (form), followed by the 2-letter department code, the unique document number, the month and year of issue, and the revision. The effective date for any SOP or other document is included on the title page and header section of each subsequent page and indicates the implementation date.

The QA Officer has full responsibility of the Document Control System. Documents can be changed, overwritten, or saved as a different document only by employees with the appropriate computer access (primarily IS and QA

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staff). A new or revised document is reviewed, and following approval, the document control number is updated and the SOP or form is uploaded to Labweb. When a new version of an SOP is added to Labweb, the previous version is removed from the active list, date-stamped and electronically archived in a designated location on the network. This automatic process guarantees that ACZ can retrieve the version that was in effect at any given time.

2.1.2 Records

A record is any information or data on a particular subject that is collected and preserved. Records are produced on a daily basis and contain original, factual information from an activity or study. For ACZ's purpose, this information may be recorded by the following means: LIMS database, logbooks, raw instrument data, worksheets, and notes (or exact copies thereof) that are necessary for the reconstruction and evaluation of the report of the activity or study. The record management system provides control of records for data reduction, validation, reporting and storage, and also provides control of all laboratory notebooks and logbooks. The system must allow for historical reconstruction of all laboratory activities that produced analytical data, must document the identity of personnel involved in sample receipt, preparation, calibration, and testing; and must facilitate the retrieval of all working files and archived records for inspection and verification purposes. At a minimum, the following criteria for records must be met:

1) Instrument logbooks must be kept up-to-date on a daily basis. In general, document all relevant activities when the event occurs.

2) Dilution factors and observations must be recorded at the time they are made, and notes regarding the sample(s) or analysis must be identifiable to the specific task.

3) A detailed description of any departure from a documented procedure, and the reason for the departure, must be provided at the time it is performed.

4) All generated data must be recorded either by an automated data collection system or must be recorded directly, promptly and legibly in permanent ink (blue or black is preferred).

5) Erroneous entries (hard copy or electronic) cannot be destroyed by methods such as erasures, overwritten files or markings. Refer to §16 for ACZ's error correction protocol.

6) Any change(s) to hard copy records must be clearly initialed and dated by the responsible staff. Changes to electronic records must also be traceable to the individual who made the change, and the reason for the change must be provided.

7) Records generated by computers must have hard copy or write-protected backup copies.

2.2 Standard Operating Procedures

A documented procedure is required for all phases of ACZ's business operations, from sample log-in through sample disposal. A Standard Operating Procedure (SOP) is a written document that details the manner in which an operation, analysis, or action is performed and thoroughly prescribes the techniques and procedures, which are the accepted process for performing certain routine or repetitive tasks. Analytical SOPs must be written with adequate detail to allow someone similarly qualified, other than the analyst(s) who routinely performs the procedure, to reproduce the procedure used to generate the test result. To the extent possible, administrative SOPs [non-technical] must include specific requirements pertaining to the process; however, the procedure itself may be a more general description so as to lend a degree of necessary flexibility to account for client requests and other circumstances which may be outside of ACZ's control.

Proposed revisions to any test SOP shall be submitted by the pertinent department supervisor (exceptions may be granted on a case by case basis) and be reviewed and approved by QA prior to implementation. Changes to provide additional clarification, correct typographical errors, etc. do not require formal approval and/or training. Analytical SOPs must be reviewed annually using the SOP Review Form (FRMQA035), and Administrative SOPs must be reviewed regularly and revised if necessary to ensure the information is accurate and reflects current practice. Documenting changes in the controlled copy of any SOP is prohibited. Refer to §10.5.1 for additional information on SOPs.

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SOPs are proprietary documents and ACZ does not distribute them freely. Any copy sent electronically or otherwise to an outside party is considered uncontrolled, and the recipient understands that additional changes can be made without prior notification. The use of uncontrolled copies of SOPs is not permitted on site unless approved by QA, and such documents will be initialed and dated by QA personnel when issued.

Unless the reference method is followed exactly and contains sufficient detail to ensure consistent application, an SOP must be developed before a new procedure, application, or instrument can be implemented. The introduction of a new method must be a planned activity directed by the Production Manager, assigned to the appropriate technical director(s), and overseen by QA staff. Exceptions may be made when the client provides specific procedural instructions. In this event, the client's instructions must be followed exactly and appended to ACZ's test report package. Exceptions are primarily related to the preparation of solid materials for analytical testing (refer to SOPAD043 for additional details). An SOP template (SOPAD025) may be obtained from QA. If a client requests a procedure for which there is no published method or existing SOP, ACZ will utilize the process described in the SOP *Client Service Policies and Procedures* (SOPAD043). Analytical SOPs are written in accordance with the TNI Standards and must include or reference the following items, where applicable:

1) identification of the test method
2) summary, scope & application of the test method, including matrices & parameters to be analyzed
3) references, including documents provided by instrument / equipment manufacturer
4) sample collection, preservation, & storage
5) equipment & supplies
6) reagents & standards, including storage conditions & shelf-life for each
7) safety
8) interferences
9) complete procedure, including details and acceptance criteria for initial & continuing calibration
10) data review & assessment, including protocols for handling out-of-control or unacceptable data
11) quality control, including acceptance criteria & corrective action for handling failed quality control
12) calculation equations (dilution factors, RPD, % recovery, etc.) & calibration formulas
13) method detection limit & reporting limit
14) method performance, including Demonstration of Capability and Method Detection Limit procedures
15) pollution prevention & waste management
16) definitions
17) tables, diagrams, flowcharts

2.3 Training

It is the responsibility of ACZ's management to ensure the competence of all employees who perform environmental tests and other specific duties, operate equipment or instrumentation, give opinions and interpretations, evaluate results, and sign test reports. Additionally, ACZ management is responsible for formulating the goals and policies with respect to the necessary education, training, and skills of all personnel and for providing training that is relevant to the company's present and anticipated tasks.

Employees must possess the appropriate combination of education, experience, and skills to adequately demonstrate a specific knowledge of their particular functions and to carry out those functions in a manner that meets ACZ's standards and expectations. Additionally, each staff member must demonstrate an understanding of laboratory operations, test methods, related quality assurance and quality control procedures, and management of records and documents to the extent necessary to successfully perform their job duties.

All full-time and part-time personnel must complete a formal training process for Safety, Ethics, Quality Assurance / Quality Control, Quarantined Materials, and Sexual Harassment on the first day of hire and are subsequently responsible for complying with all requirements that pertain to their organizational functions. For all technical staff, training for analytical procedures must be completed prior to independent generation of client data, including Proficiency Testing samples. In general, any staff member who is undergoing training must be provided with appropriate supervision. It is the responsibility of each supervisor or manager to ensure personnel within his or her department are supervised, competent, and working in accordance with ACZ's Quality System.

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2.3.1 Safety Training

Safety training is scheduled with ACZ's Chemical Hygiene Officer (CHO) and includes viewing a video of general laboratory safety, a complete review of ACZ's Chemical Hygiene Plan, and a building tour to identify the location of Safety Data Sheets, emergency showers, eye wash stations, and emergency exits. Following completion of the training, the employee takes an exam, which allows the CHO to evaluate their understanding of the material covered.

2.3.2 Data Integrity Training

ACZ is committed to fostering and enforcing an ethically sound work environment that encourages the conscientious production of accurate, technically sound, and legally defensible data. Data integrity training is required for all full-time and part-time employees (permanent or temporary) as described in ACZ's SOP *Data Integrity Principles & Policies* (SOPAD039). Initial training provides an orientation of ACZ's Ethics program, ACZ's Code of Conduct, Code of Ethics, and zero-tolerance policy. Each new employee is introduced to the company's Ombudsman. Data integrity training is provided for all current employees on an annual basis. At a minimum, employees must review ACZ's Data Integrity Principles & Policies (SOPAD039) and sign form FRMQA023 to indicate they have read, understood, & agree to adhere to them.

2.3.3 QA Training

All full-time and part-time employees attend an initial orientation session which is based on the most current version of ACZ's Quality Assurance Plan [QAP] and focuses on the relationship between quality control, quality assurance, environmental testing, and environmental monitoring.

2.3.4 Sexual Harassment Training

Sexual Harassment training is required for each new employee and includes viewing a video that demonstrates the identification, reporting, and remediation of harassment issues in the work place. Any complaints of sexual harassment must be brought to the attention of ACZ's CEO as soon as possible.

2.3.5 Technical personnel must be thoroughly trained in the analytical techniques and operating principles for all pertinent method procedures. Under no circumstances may any analyst independently generate or review client data for a test procedure before completing the required training and receiving the explicit approval of the QA department. §5 provides details of ACZ's technical training program.

2.3.6 An employee performing only data AREV or SREV functions must be appropriately trained regarding QC requirements, corrective action(s), and data qualification criteria stated in the effective version of the test SOP. The trainee must first read the SOP, and then review all pertinent information with the department supervisor. Items covered during training must be documented using the appropriate form, and both the supervisor and the trainee must sign the form. Thereafter, the effective version of the test SOP must always be used for data review.

2.3.7 Continuing training must be documented and, at a minimum, the documentation must certify that the employee has read, understands, and agrees to follow the effective version of the applicable SOP and any associated revision(s) or other in-house document. Training is documented using either FRMQA023, FRMQA012, or through email, whichever is most appropriate.

2.3.8 Training is required for all employees whose activities are affected by any procedural change(s) to an SOP and is considered to be complete once each employee has submitted documentation attesting they have read, understand, and agree to follow the revised policy.

2.3.9 ACZ recognizes the benefit of continuing education and encourages employee participation in advanced training courses, seminars, and professional organizations and meetings.

2.4 Audits

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The purpose of an audit is to verify performance and compliance to documented Quality Assurance and Quality Control policies and procedures, and to identify discrepancies when they exist. In the latter case, any problems shall be addressed and resolved in an appropriate manner to assure the Quality System is continuously improved on all levels.

2.4.1 External Audits

External audits are conducted to ascertain compliance with rules, regulations, and additional criteria for certification, and will have a higher degree of formality than internal audits. Where records are required, compliance will be critically evaluated. Issues of non-compliance identified in previous audits are usually reviewed to verify the laboratory has remediated them effectively. The ease with which important records and information can be retrieved is a criterion for judgment of the management practices of a laboratory and may dictate the depth of the audit. Individual state agencies, laboratory Accrediting Bodies, and current and potential clients routinely audit ACZ.

The on-site assessment is generally a two to four day process during which the regulating agency conducts an entrance interview and tours the facility before performing an in-depth review of documents, workgroups, reports, electronic data files, etc. A critical aspect of the on-site assessment is review and verification of bench-level documentation and analyst interviews to determine actual laboratory practices. ACZ's policy is to always have QA personnel present during an interview. If necessary, the President, Executive Director or Production Manager may attend the interview. An exit interview is conducted upon completion of all on-site assessments, during which observations and findings are reviewed. The agency will submit a final report to ACZ, generally within 30 days, detailing all pertinent findings and recommendations.

Upon receipt and review of the agency's report, the QA department will meet with department managers to develop a corrective action plan, which must be submitted to the agency by the date indicated in their report. Each finding or group of similar findings is addressed as a major corrective action as described in §2.5.2. Employees may not make changes to any laboratory or other practice based on comments or opinions expressed by the regulating agency during an interview or any other stage of the on-site assessment without first obtaining approval from QA. ACZ will revise policies and procedures as necessary as part of the major corrective action process.

2.4.2 Internal Audits

ACZ is responsible for the quality of its data and must take reasonable efforts to assure itself and all interested parties confidence can be placed in it. ACZ shall conduct internal audits of its activities to verify compliance with the Quality System. It is the responsibility of the QA Officer (QAO) to plan, direct, and organize internal audits; however, a trained and qualified individual, independent from the area or system being audited, may be designated by the QAO to conduct an internal audit. The area of activity audited, the audit findings, and subsequent corrective actions shall be documented.

The internal audit program shall address all elements of the management system. At least one test method shall be audited annually for each analytical laboratory division. Method audits encompass both qualitative evaluation of the operational details of the QA program and quantitative evaluation of the accuracy of data generated by the laboratory staff. Test method audits include step by step witnessing of the procedure. Laboratory Divisions:

- Wet Chemistry Manual
- Wet Chemistry Instrument (Prep and Analytical)
- Metals (Instrument & Prep)
- Soils
- Radiochemistry (Prep and Analytical)
- Organics (Prep and Analytical)

When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test or calibration results, the laboratory shall take timely corrective action, and shall notify customers in writing if investigations show that the laboratory results may have been meaningfully affected. Client contact should normally be initiated within two months of discovering the error(s). If data impact assessment

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cannot be completed in this timeframe management shall set a deadline commensurate to the demands of the assessment.

More frequent internal audits may be scheduled depending on the following criteria:

- Number and type of corrective actions filed for a method or activity
- Client complaints
- Continued failure to achieve acceptable results for a Proficiency Testing sample
- Findings from an external audit
- Request from management

All findings from internal audits are directed through ACZ's corrective action system. Each finding is assigned a corrective action number (similar findings may be combined). A general description of the process is as follows:

1) Findings and observations are summarized in a report.

2) The report is distributed to the department supervisor, Production Manager, and President or Executive Director.

3) The supervisor reviews the report and composes a plan of corrective action (POC) and projected completion dates for each finding. The POC should be proportional to the finding and the projected completion date commensurate with the demands of the tasks required for the corrective action.

4) The supervisor submits the plan of corrective action to the QAO or designee for review and approval.

5) The QAO or designee reviews the plan of corrective action for each internal audit finding. Once the plan of corrective action is accepted, a major corrective action number is assigned to each planned corrective action or group of similar corrective actions.

6) The supervisor negotiates the corrective action and submits a Corrective Action Report (FRMQA001) for each major corrective action number to the QA department for final review.

7) Once all corrective actions associated with the internal audit have been completed and approved, the internal audit process is complete.

An in-depth review will be conducted if there is any evidence of inappropriate actions or vulnerabilities related to data integrity. This review shall be handled in a confidential manner until a follow up evaluation, full investigation, or other appropriate actions have been completed and the issue(s) clarified. Refer to ACZ's SOP *Data Integrity Policies & Procedures* (SOPAD039).

2.4.3 Proficiency Testing (PT) Program

ACZ is required to participate in a formal Proficiency Testing Program at the frequency stipulated by regulating agencies. These "performance evaluations" are facilitated through the introduction of blind samples, purchased from approved vendors. ACZ analyzes PT samples for most accredited parameters twice in a calendar year, with each study being approximately six (6) months apart. These tests are matrix, technology, and analyte specific, and provide useful information regarding the accuracy of the analytical data being produced. At a minimum, ACZ participates in the Water Supply (WS) study for SDWA, the Water Pollution (WP) study for CWA, a Soil study for RCRA, and a Radiochemistry PT study for Drinking Water. Refer to SOPAD011 for additional details.

2.5 Corrective Action

Corrective action shall be performed when any aspect of ACZ's testing and/or calibration work, or the results of this work, do not conform to established procedures or the agreed requirements of the customer. Corrective actions are a fundamental element of ACZ's QA Program, as a successful Quality System requires the identification of deficiencies and depends on the

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development, implementation, and documentation of effective contingency plans and resolutions to effectively remediate the deficiencies. Corrective actions are classified as minor, major, or technical.

2.5.1 Minor Corrective Action

Minor corrective actions address problems or issues isolated to a specific data set or group of data sets that do not meaningfully impact reports *already* issued to clients. The minor corrective action report (FRMQA001) allows for complete documentation of any temporary deviation from the SOP or other protocol. The employee who initiates the corrective action will complete Section 1 of the report. Documentation must be accurate and must provide a complete detailed explanation of the situation for future reference. The need to qualify data shall be critically assessed and appropriately addressed. The department supervisor should always be informed of the need for a minor corrective action and may provide additional information in the appropriate section. The project manager may also provide additional information in the appropriate section if necessary. QA does not need to close a minor corrective action; however, the employee may review the report with QA personnel and request their signature in the appropriate section. Minor corrective actions do not require follow-up.

Complete documentation may be provided either on the workgroup bench sheet or on the data review checklist in lieu of using FRMQA001. Use FRMQA001 if the deviation applies to many workgroups and attach a copy of the completed form to each workgroup before the workgroup is scanned. If the report is generated after the workgroups have been scanned, then the workgroup must be retrieved and rescanned with the report included as part of the data package. In this case, a note is made on the front page of the workgroup package indicating the reason the workgroup was rescanned (i.e. “CAR attached, WG rescanned”).

2.5.2 Major Corrective Action

Major corrective actions address problems which are systematic or meaningfully impact reports which have been issued to clients. It is the responsibility of the QAO to notify laboratory management in writing of departures from the Quality System, and it is the responsibility of the laboratory management to ensure remediation is completed by the assigned due date or to negotiate an extended deadline.

A major corrective action is initiated whenever a system failure has been identified or whenever an audit finding or other circumstance casts doubt on the correctness or validity of the analysis result(s). The client must be notified in writing if their work is significantly affected. The QA department will work with the Project Manager to determine if a revised report must be issued to the client. See ACZ’s SOP *Client Service Policies and Procedure* (SOPAD043) for details. A major corrective action may also be initiated when the need for preventive action has been identified (refer to §2.5.4).

Only QA department personnel may open and close a major corrective action. When opened, the corrective action shall be assigned a unique tracking number (referred to as the CAR number) to ensure that ACZ maintains a complete and accessible record of all Quality System deviations or failures, root cause determinations and subsequent resolutions, and preventive actions. A remediation deadline shall be assigned for all major corrective actions.

Examples of circumstances requiring a major corrective action include, but are not limited to:

- Contamination trends as indicated by blanks routinely above acceptable levels
- Spikes, surrogates and lab control samples continually outside acceptance limits
- Change to the MDL and/or PQL for a procedure
- “Not Acceptable” Proficiency Testing results
- Findings from internal or external audits
- Discrepancies between what was reported to clients and what should have been reported to clients due to equation errors or incorrect LIMS configuration.
- Hold times or deadlines routinely missed

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- Evidence of insufficient or inadequate training

Following initiation, the procedure for a major corrective action proceeds to an investigation by the assigned individual to determine the root cause of the problem and identify possible resolutions to rectify the problem. The action(s) most likely to eliminate the problem and prevent recurrence of the problem must be selected, documented and implemented, and pertinent staff members must be trained, if necessary. Changes resulting from the corrective action will be monitored, if necessary, to ensure the resolution(s) are effective. A general outline of the procedure is as follows:

1) Initiation: Any employee may initiate a corrective action by notifying QA. The department manager should be notified first so that they can assess the need for a major corrective action. If determined to be necessary, QA personnel will open a corrective action, assign a unique tracking number, and a deadline for remediation. Deadlines shall be assigned based on the anticipated demands of remediation and potential threat posed to data integrity.

2) Assignment: QA assigns the corrective action to the person(s) responsible for problem characterization, Root Cause Analysis (RCA), Data Impact Assessment (DIA), corrective (including preventive) actions. Sections 1 and 2 of FRMQA001 shall be completed by the assignee(s).

3) Immediate action shall be taken to eliminate propagation of errors. Stopgap measures may be employed, including but not limited to: subcontracting analyses, imposing a moratorium on data reporting, manual data transformation. Immediate action shall be to a degree commensurate with the magnitude and risk of the problem.

4) Investigation and Action: Must be completed by the assigned deadline. Deadline extensions shall be negotiated with the QA department.

   a. The assigned individual(s) launch an investigation of the problem. There are three major components of the investigation
      1) Characterization of the problem: A thorough, but succinct, description of the problem must be composed. Whenever possible, this includes determination of the exact timeframe during which the error was present and what workgroups and samples were affected.
      2) Root Cause Analysis (RCA): Focuses on establishing the sequence of events or causal chain leading up to the problem, identifying contributing factors and elucidating relationships between them, and determining where intervention could be reasonably implemented to change performance and prevent an undesirable outcome. The depth of the RCA shall be commensurate with the risk and magnitude of the problem.
      3) Data Impact Assessment (DIA): Once the problem has been fully characterized, it shall be evaluated to determine whether client data may have been significantly impacted by the error. The DIA shall be commensurate with the risk and magnitude of the problem.

   b. A resolution to correct the problem and prevent its recurrence must be determined & implemented. Resolution may be done solely by the person(s) who investigated the root cause or it may require input from one or more additional departments.

      1) Conduct additional training if necessary. Training must be documented using the appropriate form and must include a description provided by the person who conducts the training. All trainees are required to sign and date the form to acknowledge he/she has received training, understands the change(s) and agrees to adhere to any change(s) in a policy or procedure.
      2) Revise SOP(s) as necessary. Proposed revisions must be approved by QA prior to implementation.
      3) Configure or enhance automated systems (e.g. LIMS) to correct problems or support preventive measures.
      4) Correct data in ACZ's LIMS as deemed necessary by Technical Directors, QA Staff, & Project Managers.

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5) Perform additional measures (e.g. instrument or support equipment purchase, etc.) as necessary.
6) Document implementation dates for each corrective action.
7) Attach or reference all supporting documentation in the corrective action report.

5) Project Manager Review: If necessary, the PM will determine whether affected data will be accepted or rejected, contact the client, and issue revised reports accordingly. Project Manager review may not be required for every major corrective action.

6) QA reviews the corrective action. If satisfactory, the corrective action is approved and closed.

7) If deemed necessary, QA conducts follow-up. Follow-up is scheduled after sufficient time has elapsed to observe the efficacy of the corrective action and may need to be done multiple times. If the corrective action is determined to be ineffective, a new major corrective action will be initiated and the process repeated. QA follow-up may be documented on the corrective action report or the CAR spreadsheet located on ACZ's network.

2.5.3 Technical Corrective Actions

Technical corrective actions apply to departures or deviations from the quality control parameters stated in individual test SOPs. Each test SOP must include all required quality control that applies to the procedure (as stipulated by the method and other regulatory agencies) as well as the performance frequency, acceptance criteria and corrective action for handling failed quality control measurements. Each SOP must describe the procedures to be followed for reviewing and assessing data, including corrective action for handling out-of-control or unacceptable data. The required protocol for technical corrective actions is summarized below. ACZ's protocols are included within the [ ].

1) Identify the individual responsible for assessing each nonconformance and initiating or recommending corrective action [analyst who performs AREV]

2) Define how the analyst must treat data if associated quality control measurements are unacceptable [section 12 of SOP]

3) Specify how non-conformance and subsequent corrective actions are to be documented [data review checklist]

4) Specify how management reviews the corrective actions [reviewed during SREV]

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control then the corrective action described in the SOP must be performed. Alternatively, report data with the appropriate qualifier if reprocessing and reanalysis is not possible. The qualifier must be assigned to any sample associated with the failed quality control measure. A current list of all extended qualifiers is available in the LIMS database and may be accessed by all employees.

2.5.4 Preventive Action

Preventive action is a pro-active process to identify opportunities for improvement rather than reacting to the identification of problems or complaints. Needed improvements and potential source(s) of any nonconformance, either technical or concerning the Quality System, must be identified and addressed. Examples of preventive action include but are not limited to: maintaining a cross-trained staff; maintaining a supply of spare consumable parts; monitoring the performance of support equipment; performing routine maintenance on instruments; maintaining an adequate supply of standards/reagents; ordering supplies before running out; completing log-in review in a timely manner; ensuring ACZ can perform work before samples are accepted; correcting quotes before samples are logged in; and analyzing samples by the appropriate method.

2.6 Management Review of the Quality System

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At least once per calendar year, ACZ's management conducts a review of its Quality System and all activities related to its environmental testing services to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. At a minimum, the review must take the following into account:

- Status, review, and discussion of major corrective actions
- Results of recent PT studies and corrective actions initiated / completed
- Review of recent external audits
- Review of internal audits
- Presentation of ideas to improve efficiency and productivity
- Presentation of ideas to improve service and data quality
- Status of state certifications
- Feedback from clients
- Feedback from employees
- Ethics Program
- Ombudsman
- Changes in the volume and type of work undertaken
- Customer feedback from annual client survey
- Other pertinent issues
- Suitability of policies and procedures.
- The effectiveness of training.

2.6.1 Department Reports

Each department manager completes a Department Report (FRMQA041) prior to the Management Review meeting. Each item on the report is to be evaluated as it pertains to the individual department. FRMQA041 is provided in APPENDIX B – Forms for Management Review of the Quality System
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2.6.2 Management Review Report

The completed department reports are submitted to ACZ's President or Executive Director by the specified due date, and the information from each report is reviewed and compiled to complete the Management Review Report (FRMQA042). A copy of the completed report is issued to each manager in advance of the Management Review meeting. At a time specified by the President or Executive Director, the CEO, CFO, QAO, Production Manager, IS Manager, Senior Project Manager, and Business Development Manger meet as a group to discuss the report. Other formats may be utilized at the President or Executive Director's discretion. All reviews will be appropriately documented. FRMQA042 is provided in APPENDIX B – Forms for Management Review of the Quality System

2.6.3 Customer Feedback

ACZ solicits customer feedback on an annual basis through the use of a client survey distributed and received via email. The survey asks for feedback regarding customer service, data quality, staff, value, timeliness, and laboratory standing compared to other labs. Feedback is compiled by the CEO and discussed during management review of the quality system.

2.6.4 When a systemic failure is identified through the management review process ACZ's corrective action protocol shall be initiated.

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3 ETHICAL AND LEGAL RESPONSIBILITY

All ACZ employees have an ethical and legal responsibility to produce data that is accurate, reliable, and legally defensible. ACZ's proactive program for the prevention and detection of improper, unethical, or illegal actions includes an Ombudsman who acts as a neutral party and serves as a confidential liaison between ACZ employees and upper management regarding questions, problems, complaints, suggestions, or ethical dilemmas.

Initial employee orientation includes ACZ's Code of Conduct, Code of Ethics, and zero-tolerance policy. Employees are informed of the processes in place to ensure employees are free from undue internal or external commercial, financial, or other pressures that may adversely affect the quality of an employee's work, endanger the trust in the independence of ACZ's judgment, or compromise the integrity of ACZ's environmental testing activities. A more detailed description of all aspects of the ethics program is provided in ACZ's SOP *Data Integrity Principles & Policies* (SOPAD039).

ACZ will not tolerate unethical or improper activities or behavior. Violation of company policies may lead to repercussions ranging from a warning to termination and possible criminal prosecution if warranted by the situation. ACZ has access to many resources that may be utilized at any time to help clarify any situation determined to be a "gray area." Employees are strongly encouraged to seek further guidance from a supervisor, ACZ's Ombudsman, President, Executive Director, or QA staff whenever doubt is raised. Activities that will not be tolerated include, but are not limited to:

- **Misrepresentation of a procedure or documentation** – Intentionally performing a job duty in a manner that does not comply with a documented procedure, including but not limited to a test SOP or method used for sample analysis; providing inaccurate and misleading documentation associated with a data package or failing to provide the necessary documentation as part of a data package.

- **Falsifying Records** – Providing false information on personal credentials, resumes or educational transcripts, logbooks, raw data and client reports, or creating data without performing the procedure (also known as dry labbing).

- **Improper peak integration** – Intentionally performing improper integration of data chromatograms so quality control samples meet acceptance criteria. This is also known as peak shaving or peak enhancing.

- **Improper clock setting** – Readjusting the computer clock so that it appears samples were analyzed within hold times. Also referred to as time traveling.

- **Improper representation of Quality Control samples** – Failing to treat batch quality control samples in the same manner as client samples (including Proficiency Testing samples) or misrepresenting any type of quality control sample associated with the preparation batch and/or analytical batch.

- **Improper calibration** – Intentionally performing improper manipulation of calibration data or forging tune data so that it meets acceptance criteria.

- **File Substitution** – Replacing invalid data with valid data from a different time so the analysis appears to be successful.

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4 PERSONNEL AND RESPONSIBILITIES

ACZ Laboratories, Inc. is a Sub chapter S corporation with a single owner.

Due to the nature of regulatory oversight and the increasing demands of the environmental lab industry, QA issues permeate all aspects of our business, the largest and most critical of which are operations (production). On a daily basis, QA and Production must efficiently function together to consistently provide clients with technically sound and legally defensible data, and to ensure the Quality System remains an integral part of all areas within ACZ. The President or Executive Director must rely on regular input and feedback from ACZ's QAO and Production Manager, and to this effect, upper management is defined as ACZ's President or Executive Director, QAO, and Production Manager. It is the responsibility of upper management to document company policies, objectives, systems, programs, procedures, and instructions to the extent necessary to assure the quality and defensibility of all data.

ACZ is organized such that the President or Executive Director also works directly with and relies on input and feedback from the Senior Project Manager, Business Development Manager, Production Supervisors, Log-In Supervisor, Document Control Supervisor, IS Manager, Chief Financial Officer, Chemical Hygiene Officer, Radiation Safety Officer, and Hazardous Waste Coordinator. These individuals are responsible for managing both the day-to-day operations and long-term goals within their respective areas. It is the responsibility of all managers to ensure that all documented ACZ policies and procedures, including those in the QAP and associated SOPs, are communicated to, understood by, made available to, and implemented by ACZ personnel.

ACZ only uses personnel who are employed by or under contract to the laboratory.

Refer to FRMAD072 for ACZ's current organizational chart.

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4.1 President/CEO/Executive Director[2]

The President or Executive Director is ultimately responsible for all analytical and operational activities of the laboratory and must ensure that 1) the laboratory carries out all environmental activities in such a way as to meet the requirements of the TNI Standards and 2) the laboratory satisfies the needs of the client and the regulatory authorities. General duties involve budgeting for all departments, making decisions on capital equipment and automation; developing company policies and benefits; addressing personnel issues such as hiring, firing, and promotions; and working with clients on various matters. Day-to-day responsibilities include providing direction to all laboratory departments including laboratory operations, accounting, marketing, QA, and client services. Additional responsibilities are as follows:

- Work directly with ACZ's Ombudsman to provide and maintain a mechanism for confidential reporting of ethical/data integrity issues as well as issues that may directly affect current ACZ policies.
- Define the minimal level of qualification, experience, and skills necessary for all laboratory positions.
- Provide the QA Officer with defined responsibility and authority for ensuring the successful development, implementation, and management of ACZ's Quality System.
- Provide the Production Manager with defined responsibility and authority for ensuring the technical operations and provision of resources needed to maintain the required quality of laboratory operations.
- Provide adequate supervision of environmental staff by persons familiar with methods and procedures, purpose of each test, and assessment of the test results.
- Ensure all technical staff has demonstrated capability in the activities for which they are responsible and ensure that the training of each member of the technical staff is kept up-to-date.
- Ensure the QA Officer has access to the highest level of management at which decisions are made on laboratory policy or resources.
- Provide managerial staff the authority and resources needed to discharge their duties.
- Provide technical personnel the resources needed to discharge their duties.
- Specify and document the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests.
- Implement appropriate and current guidelines for all lab methods and procedures to ensure data quality and efficiency of analyses. Ensure all method protocols utilized by ACZ meet the QC requirements as established by EPA or other governing agency.
- Document all policies and procedures related to the analytical and operational activities of the laboratory.
- Provide support to technical staff to ensure timely completion of all laboratory work, and develop contingency plans to ensure workflow progresses as planned.
- Meet quarterly (or more often) with the QA Officer and Production Manager.

[2] If more than one Executive Director is appointed, the duties described in this section will be performed jointly by these individuals.

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4.2 QA Officer (QAO)

The QA Officer reports directly to the President or Executive Director; however, the QA department is considered a separate entity from operations in order to ensure data is evaluated objectively. The QA Officer has direct access to the President or Executive Director, and is therefore able to discuss and/or resolve all concerns, policies, etc. related to quality assurance or quality control. The primary responsibility of the QAO is to develop, implement, and manage all aspects of ACZ's Quality System, and he/she may take any action necessary to ensure all ACZ employees adhere to all policies, procedures, and objectives documented in ACZ's QAP, SOPs, memorandums, emails, etc. If warranted, the QAO has the authority to halt the performance of a single method or the production of a department, and if necessary, the operations of the entire laboratory, and will grant permission to resume when satisfied that the issue(s) have been resolved. Additional responsibilities include but are not limited to those stated in FRMAD060 and the following:

- Review and revise ACZ's QAP and provide training for all employees following approval of a new version.
- Provide QA orientation to new employees.
- Meet quarterly (or more often) with the President or Executive Director and Production Manager.
- Work with department managers to develop and improve training protocols.
- Conduct department training sessions as needed to address specific problems and questions.
- Arrange for or conduct internal audits; notify management of deficiencies; and track corrective actions.
- Organize all external audits; notify management of deficiencies; and assign and track corrective actions.
- Review and approve SOPs.
- Meet at least quarterly with Production Supervisors to provide information, respond to questions, etc.
- Manage Proficiency Testing (PT) program.
- Coordinate and maintain all regulatory and client certification programs.
- Review and validate a determined percentage of all data packages from Log-in to Reporting.
- Work with marketing/client service representatives on QA aspects of proposals.
- Work with Project Managers and the Production Manager to resolve client feedback regarding data quality.
- Review and maintain records and documentation for audits, certifications and all other QA issues.

  Qualifications:

  - General knowledge of the analytical test methods
  - Documented training and/or experience in QA procedures
  - Knowledge of the Quality System as defined under TNI

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4.3 QA Coordinator

The QA Coordinator reports directly to the QAO and assists with the development, implementation, and management of the Quality System. Primary job responsibilities are as follows:

- Review and maintain records/documentation for employee training including DOCs, MDLs, etc.
- Provide initial QA orientation to new employees.
- Coordinate annual data integrity training.
- Schedule analyses and compile and report data for Proficiency Testing (PT) program, including DMRQA.
- Initiate and track corrective actions related to PT samples and manage all documentation associated with analyses.
- Review and approve SOPs.
- Conduct internal audits, notify management of deficiencies; and track corrective actions.
- Conduct department training sessions as needed to address specific problems and questions.
- Update control chart-generated QC limits in the LIMS database as needed.
- Monitor control & calibration of support equipment
- Assist QAO with management of certifications.
- Manage ACZ's resume compilation.
- Update ACZ organizational chart as necessary.

Qualifications:

- General knowledge of the analytical test methods
- Documented training and/or experience in QA procedures
- Knowledge of the Quality System as defined under TNI

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4.4 Production Manager

The Production Manager reports directly to the President or Executive Director. General duties involve working with analytical department supervisors on a daily basis to prioritize client projects and QA deadlines and to track sample analyses in order to maintain acceptable turn-around-times for project completion. The Production Manager also addresses personnel, instrumentation, and reagent/supply issues that may affect the completion of the scheduled work and works directly with the QA department to ensure all Quality System requirements pertaining to production are successfully completed in a timely manner. Additional responsibilities are described in FRMAD060.

- Conduct weekly meeting with Production Supervisors to discuss current and upcoming workload, scheduling, priority projects, QC requirements, instrument / equipment issues, personnel, etc.
- Schedule QA work (MDL studies, DOCs, PT sample analysis, SOP revisions, etc.) with department supervisors in order to ensure QA requirements are kept up-to-date.
- Meet at least quarterly with the President or Executive Director and QA Officer.
- Communicate with Project Managers regarding project/instrument status. Notify PMs if problems exist that may affect the project completion date.
- Work with marketing/client service representatives on production aspects of proposals.
- Work with Project Managers and the QA Officer to resolve client feedback regarding data quality.
- Perform checks of sample status using LIMS database to help the laboratory staff meet all established hold times and to determine that analyses can proceed as scheduled to meet required turnaround times.
- Provide hands-on support to analysts when necessary to ensure timely completion of all laboratory work, and develop contingency plans to ensure workflow progresses as planned.
- Work with QA Officer to develop and improve training protocols, conduct department work sessions to address specific problems and questions.

  Qualifications:

  - General knowledge of the analytical test methods
  - Minimum four (4) years of laboratory experience
  - Minimum two (2) years of supervisory experience
  - General knowledge of lab-wide systems (including but not limited to log-in and reporting)

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4.5 Production Supervisor

Each Production Supervisor is a full-time employee who reports to the Production Manager and exercises day-to-day oversight of laboratory operations for their specific area(s) of expertise. Each supervisor must be familiar with the test methods and related theory and instrumentation, as well as the assessment of results. In addition to monitoring the standards of performance, validity of all analyses, and quality of all data generated in their respective department(s), each supervisor is also responsible for ensuring that a new analyst has successfully completed all training requirements and is adequately prepared to commence work on client samples. Additional responsibilities are described in FRMAD060. If any supervisor is absent for more than 15 consecutive calendar days then another full-time staff member meeting the required qualifications will be assigned to perform the supervisor's duties.

Required Qualifications for a Production Supervisor:

1) Chemical analyses (Organics & Metals): BS or BA in chemical, environmental, biological sciences, physical sciences or engineering, with a minimum of 24 college semester credit hours in chemistry and at least two (2) years of experience in the environmental analysis of representative inorganic and organic analytes for the which the laboratory seeks or maintains accreditation. A masters or doctoral degree in one of the above disciplines may be substituted for one (1) year of experience.

2) Inorganic Chemical analyses (other than Metals): At least an earned associate's degree in the chemical, physical, or environmental sciences, or two (2) years of equivalent and successful college education, with a minimum of 16 college semester credit hours in chemistry and at least two (2) years of experience performing such analyses.

3) Radiological analyses: BS or BA in chemistry, environmental, biological sciences, physical sciences, or engineering, with at least 24 college semester credit hours in chemistry and at least two (2) years of experience in the radiological analyses of environmental samples. A masters or doctoral degree may be substituted for one (1) year of experience.

4.6 Business Development Manager

ACZ's Business Development Manager reports directly to the President or Executive Director and supervises all Client Service Representatives, each of who conducts marketing and sales efforts on behalf of ACZ with potential, new and existing clientele, and develops and maintains long-term relationships with customers by working with Project Managers when necessary. Additional responsibilities of the Business Development Manager are described in FRMAD060. ACZ's Client Service staff is authorized to review all contractual agreements with clients, review all proposals and develop price quotations for routine and non-routine analytical projects.

4.7 Senior Project Manager (PM)

The Senior Project Manager reports directly to the President or Executive Director and is responsible for overseeing the PM department. Additional responsibilities of the Senior PM are described in FRMAD060. Each Project Manager serves as the primary laboratory contact for each ACZ client, handles all client service requests, and investigates and resolves any problem brought to ACZ's attention by the customer. In order to provide consistency, each PM is assigned a list of clients, and it is the primary responsibility of each PM to ensure all of their client project needs are managed on a day-to-day basis and met in a timely manner and that all data submitted to the client is of high quality. All PMs work directly with the Production Manager and Production Supervisors regarding client data issues (due dates, hold times, retests, data quality, etc.), with Document Control regarding client reports and with the QA department regarding data quality questions or concerns.

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4.8 Instrument Operator

Instrument operators report directly to the respective Production Supervisor. The position involves the analysis of various matrices for trace level contaminants using specialized and technical instrumentation. Each operator must be capable of performing all job duties in an accurate and proficient manner. Education will be verified by providing a copy of a college transcript or diploma, which is maintained in the employee's personnel file. Experience is verified by ACZ's CFO prior to completing the hiring process (verbal or documented verification provided by each reference listed on a resume or application is acceptable). The operator must demonstrate understanding of related theory, mathematics, analytical instrumentation, and data interpretation. This work is predominantly intellectual and involves the continuous use of professional and sound judgment. The employee must meet or exceed all requirements for generation of litigation-quality data and must also continue to demonstrate increased proficiency regarding the interpretation of the data as well as the operation and troubleshooting of the assigned instrument(s). These improvements should be attainable through ongoing efforts in-house as well as through specialized instruction at off-site locations. Prerequisites regarding education and experience, as well as job responsibilities and performance expectations are described in FRMAD059. Exceptions pertaining to experience or education will be made on a case-by-case basis.

Qualifications:

- BA or BS in Chemistry or related science or a minimum of 3 years of relevant experience in lieu of degree
- Prior laboratory experience is preferred but is not required.
- Successful completion of training by supervisor or proficient instrument operator

4.9 Laboratory Analyst [Technician]

The laboratory technician reports directly to the respective Production Supervisor. The position involves analysis of various matrices using appropriate analytical techniques and support equipment as well as preparation of samples for instrument analyses. Each technician must be capable of performing all job duties in an accurate and proficient manner. Education will be verified by providing a copy of a college transcript or diploma, which is maintained in the employee's personnel file. Experience is verified by ACZ's CFO prior to completing the hiring process (verbal or documented verification provided by each reference listed on a resume or application is acceptable). The technician must demonstrate understanding of related principles and mathematics, must possess common sense and mechanical skills, and must seek professional judgment from the supervisor as necessary. The employee must meet or exceed all requirements for generation of litigation-quality data as well as sample preparation tasks and routine analyses, and must also continue to demonstrate continuous improvements. These improvements should be attainable through ongoing training efforts in-house as well as through training opportunities at off-site locations. Prerequisites regarding education and experience, as well as job responsibilities and performance expectations, are described in FRMAD058. Exceptions pertaining to experience or education will be made on a case-by-case basis.

Qualifications:

- BA or BS in Chemistry or related science is preferred but is not required
- Prior laboratory experience is preferred but is not required
- Successful completion of training period by supervisor or proficient technician

4.10 Information Services (IS) Manager

The Information Technology Manager reports directly to the President or Executive Director and is responsible for the oversight of the IS department regarding the installation and maintenance of ACZ's computer network and all hardware and software and related equipment deployed on the premise. Additional responsibilities are described in FRMAD060. The department is also responsible for developing, maintaining, and improving custom written applications for laboratory automation and efficiency as well as for ACZ's LIMS, Intranet (Labweb), Internet and electronic data deliverables (EDDs).

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4.11 Log-In Supervisor

The Log-In Supervisor reports directly to the President or Executive Director and is responsible for the oversight and management of all department personnel and operations. Primary responsibilities include fulfillment and shipment of bottle orders to the client's destination in a timely manner, receipt of all incoming samples, evaluation of all incoming samples against ACZ's Sample Acceptance Policy, entering samples into the LIMS database, and performing timely review of all logged samples. Additional responsibilities are described in FRMAD060.

4.12 Document Control Supervisor

The Document Control Supervisor reports directly to the President or Executive Director and is responsible for the oversight of the Document Control department. Primary responsibilities include the generation of client reports and EDDs and the maintenance, organization and control of all hard copy data and records, including workgroup data, client reports, CCOCs, and QA records and documents. Additional responsibilities are described in FRMAD060.

4.13 Chemical Hygiene Officer (CHO)

The Chemical Hygiene Officer is primarily responsibilities for oversight of ACZ's documented Chemical Hygiene Plan, conducting initial and refresher safety training for all employees, monitoring exposures, and maintaining records for Safety Data Sheets, injury reports, chemical exposure reports, etc. Additional responsibilities include working with management to develop and implement policies to improve the program. The person designated as CHO must have completed at least one basic laboratory safety course and have one year of experience performing laboratory work, preferably with responsibility for at least one area of laboratory safety.

4.14 Hazardous Waste Coordinator (HWC)

The HWC is responsible for managing and, with the collaboration of management, enforcing all aspects of ACZ's Hazardous Waste Management Plan (SOPAD007). The HWC must insure the HWP is compliant with relevant requirements in the US Code of Federal Regulations as well as any additional state regulations. Additional duties include bulking and labeling hazardous materials, filling out required documentation, and arranging for disposal; these activities may be delegated to qualified individuals under the supervision of the HWC. The HWC must know and understand the specific waste streams that ACZ uses and be able to determine how to dispose of unknown chemicals. This is best done by attending a training course on "Laboratory Waste Management." The individual responsible for hazardous waste disposal and signing the waste manifest must maintain HAZWOPER and DOT hazmat certification.

4.15 Radiation Safety Officer (RSO)

ACZ's Radioactive Materials License (RML) requires the laboratory have an RSO. The President or Executive Director appoints a Radiation Safety Officer to act as his/her representative in implementing the Radiation Safety Program. The RSO's responsibilities include developing radiation safety guidelines in accordance with Nuclear Regulatory Commission (NRC) and Colorado state rules and regulations, and for assuring compliance with those guidelines by ACZ personnel. The RSO will work with ACZ's administration to implement policies and seek ways to improve the safety program. The person designated as RSO must have completed a Radiation Safety Course or have at least 3 years of experience prior to being officially designated as the RSO. The RSO reports directly to the President or Executive Director of ACZ.

4.16 Chief Financial Officer (CFO)

ACZ's Chief Financial Officer is primarily responsible for all financial matters including payroll, accounts receivable, accounts payable and financial statements; monthly and annual balance and profit and loss statements; and assisting with annual budget preparation. In addition, the CFO maintains and monitors the security system and electronic time clock; invoices client projects from the database; updates customer account information; acts as the administrator for 401k/Profit Sharing Plan; maintains and executes the Employee Benefits Manual; and assists in hiring process by posting job openings, scheduling qualified candidates for interviews, checking references, and ensuring a new employee provides proof of education.

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4.17 Purchasing Agent

Primary responsibilities include final review and approval of requisitions and submitting and tracking all subsequent purchase orders.

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5 TECHNICAL TRAINING

Prior to the independent generation or review of data for client samples (including PT samples), all analysts must undergo a formal, documented training process. Technical personnel must be thoroughly trained in the analytical techniques and operating principles and procedures for the methods utilized by ACZ. This process includes but is not limited to: reading the associated published method, reading all related SOPs, improving laboratory skills, learning troubleshooting, maintenance, calibration and operating procedures for pertinent equipment and instruments, and creating workgroups and reviewing data through the LIMS database.

It is the responsibility of the department supervisor to determine that a new analyst is properly trained, has successfully completed all initial training requirements and is prepared to commence work on client samples. Under no circumstances may an analyst independently generate client data before receiving the explicit approval of the QA department.

5.1 The effective version of the test SOP provides the training framework for all sample preparation and analysis. The SOP is typically based on published approved methodologies (EPA or other) and incorporates any necessary activities and protocols not included in the published method(s) as well as requirements stipulated by other regulatory agencies.

5.2 Training for data AREV or SREV only must be documented as specified in §2.3.6. For analysts, approval to perform a procedure includes approval to perform AREV for the procedure. For supervisors and technical directors, approval to perform a procedure includes both AREV and SREV approval. SREV-specific training documentation is required for analysts.

5.3 Each employee must be trained either by the department supervisor or by an analyst within the department who is proficient in the area of testing and has been designated by the supervisor. Whenever possible, anyone performing training must meet the following requirements:

1) Documentation of training on the effective version of the test SOP.
2) Documented approval for the analysis.
3) A current IDOC or CDOC.

Exceptions may be granted on a case-by-case basis as approved by the QAO.

5.4 Initial training is documented using the Initial Method Training form (FRMQA004). The General Lab Practice Training Form (FRMQA047) is also required for an analyst's first procedure. Once training has been completed, the trainee and the instructor fill out the form together to ensure all pertinent information has been addressed and to ensure the trainee comprehends the material and is provided an opportunity to ask questions or request additional training. The trainee's signature is an attestation that he/she has read, understands, and agrees to follow the effective version of the SOP.

5.5 To demonstrate an aptitude for the procedure, the analyst must perform a successful Initial Demonstration of Capability (IDOC) prior to independent preparation and/or analysis of client samples. Performance is documented using FRMAD023. The data is reviewed initially by the department supervisor and the analyst (AREV), and both individuals must initial and date the review checklist. A new IDOC is required if an analyst does not perform the method within 12 months.

5.6 SREV for any preparation workgroup is performed by the department supervisor or a qualified analyst, and SREV for any analytical workgroup is performed by QA.

5.7 Prior to performing an IDOC, a new analyst should be provided sufficient opportunity to practice the procedure. This confirms the analyst understands the procedure and feels comfortable performing the procedure independently. Data associated with any practice is not submitted to QA.

5.8 It is not necessary for the first IDOC attempt to pass; however, the supervisor needs to review the analyst's techniques if multiple attempts do not pass.

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5.9 A thorough review of the raw data is performed as part of initial method training and should include particular attention to details not presented in LIMS or on the final report, such as generating final sample concentration from the instrument response provided in the raw data (if applicable), verifying correct standard and reagent traceability.

5.10 Where specified by the method or a regulating entity, and as stated in the test SOP, successful demonstration of performance such as Linear Calibration Range determination (LCR) or Method Detection Limit (MDL) study must be completed prior to independent analysis of client samples.

5.11 All initial training documentation must be submitted to the QA department as a complete package. At a minimum, the package must include:

1) Initial Method Training form (FRMQA004), signed by the trainee and instructor (or department supervisor).

2) IDOC documentation:

   - ✓ Completed and signed certification statement (FRMAD023)
   - ✓ Workgroup bench sheet, raw data, and all supporting documentation

3) If applicable, an MDL study. Complete FRMAD031 and attach all related raw data and supporting documentation.

4) If applicable, calibration range study. Complete FRMQA029 and attach all related raw data and supporting documentation.

5) For all determinative methods utilizing a calibration curve or average response factor, the Method Calibration Form (FRMQA050).

5.12 Following review of all pertinent training documentation, QA will issue procedure-specific clearance for the trainee to independently generate and review data for client samples. This permission is tracked and may be viewed on a designated location on the public network drive.

1) Approval for preparation procedures is granted after the instrument data has been reviewed and approved.

2) An unapproved analyst who is “shadowing” the trainer (observing, learning the organization of the lab, reagent room, etc.) may not assist with the procedure, and the workgroup documentation must bear only the initials of the trainer, who is fully responsible for the data.

3) If the analyst has successfully completed training for a procedure and generates client data or reviews client data prior to QA approval, then any workgroup(s) or data review checklist must also bear the initials of a proficient analyst, with current approval for the method, who oversees the analyst’s work for the procedure and assumes full responsibility for the data. The primary analyst must always be aware that he/she is responsible for the workgroup. The use of another employee’s initials without their explicit approval is expressly prohibited.

5.13 The supervisor is responsible for ensuring the training of each analyst is kept up-to-date. Each analyst must read, understand, and agree to follow the effective version of the SOP and continued proficiency must be demonstrated and documented annually for each analyst. A one month grace period is allowed for submitting CDOC documentation. Thereafter, the analyst is prohibited from performing the procedure until a successful CDOC or IDOC is submitted to QA.

5.14 Each production supervisor routinely conducts department meetings to discuss procedures, work schedules, resources, questions and concerns, problems, QA, etc.

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6 SAMPLE COLLECTION AND HOLDING TIMES

Sample collection procedures are well documented by the EPA and other agencies. ACZ's clients are instructed to provide representative samples whenever possible. ACZ supplies its clients with the containers and other materials necessary to maintain sample integrity (to the extent possible) from the time of collection through analysis. Although ACZ does not perform sample collection activities, each project manager or client service representative will assist a client with specific sampling requirements as needed. When necessary, will direct a client to other resources. The following sections include general information on sample containers, preservatives. and holding times. These are essential components in preserving the chemical and physical properties possessed by the sample at the time of collection.

6.1 Sampling Containers and Preservatives

The EPA outlines the requirements for sample container types, sample volume, and preservation. ACZ's inventory includes various sizes of plastic and glass containers that range from pre-sterilized to certified-clean by the supplier. Amber bottles are used when specified by the method. Glass containers are obtained from vendors that specialize in the sales of environmental sample containers, and all non-certified bottles are purchased from reputable lab/industry vendors. Refer to FRMAD045 and FRMAD046 for bottles types and preservation techniques for specific analyses. Refer also to APPENDIX A Required Container Type, Preservation Techniques, and Holding Times for additional information regarding EPA requirements for container types and preservation.

All sample containers shipped to our clients are new, contain the appropriate preservative(s), and are color-coded to identify preservation and storage. Out-going containers are packed in clean coolers with a copy of ACZ's Sample Acceptance Policy, general directions for sample collection, bottle labels, ice packs, sampling information, blank chain of custody, return shipping labels, and custody seals. Trip blanks and rinsette water are included when requested by the client or when mandated by a specific analytical method. After samples have been collected they are cooled to a temperature ≥ 0 °C and ≤ 6.0 °C. Samples that require thermal preservation must be maintained within this temperature range until all analyses have been completed.

6.2 Holding Times

The EPA has conducted lengthy studies of sample degradation versus time to establish a maximum holding time for each parameter, and the results of these studies are compiled into holding-time tables to provide guidelines for litigation purposes. Data for a sample prepared / analyzed outside of the established holding time may be rejected by regulators as unusable. Holding times will vary slightly from regulation to regulation, thus further emphasizing the need for a client to consult with their Project Manager prior to sample collection. The holding time typically begins at the time or date of collection in the field. APPENDIX A Required Container Type, Preservation Techniques, and Holding Times outlines holding times (a hold time stated in 40 CFR supersedes the published method).

If ACZ Laboratories, Inc. receives samples past holding times or near the expiration of the holding time, sample analysis will proceed unless the client has indicated on the CCOC that an attempt to contact the client must first be made. Analyses performed outside of holding time will be appropriately qualified on the final report. Holding times $\leq$ 72 hours are calculated based on the <u>hour</u> of the sample date/time. Holding times > 72 hours are calculated based on the <u>day</u> of the sample date/time.

In general, and unless otherwise noted in the test SOP, sample preparation and analysis must be completed within the stated holding time. For analyses that extend beyond the intended scope of the method for an analyte or matrix, the hold time stated in the SOP must be met or the samples must be appropriately qualified.

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7 SAMPLE CUSTODY & SAMPLE HANDLING

Sample custody begins with receipt of sample containers from the client and continues beyond preparation and analysis to the proper disposal of primary and secondary sub-samples. Complete and accurate documentation must be provided at all stages of custody. There are many key elements to sample custody including laboratory security, chain of custody records, sample storage, internal custody logs, sample tracking within the laboratory, control of subcontracted work, and sample disposal. Unless otherwise specified, ACZ is contractually committed to retain samples for a minimum of 30 days after the invoice of a project[3].

7.1 Sample Receipt and Log-in

Refer to SOPAD045, *Sample Receipt & Log-In Procedure / Maintenance of Sample Integrity*, for the details of ACZ's sample receipt and log-in procedures. Upon delivery of samples to ACZ, Log-In personnel evaluate the condition of the cooler and custody seals. The custody seals are then broken to retrieve the Chain of Custody (COC), which must be signed by the sample custodian to document transfer of sample possession to ACZ.

Sample conditions are evaluated and any problems, such as expired hold times, lack of preservative or improper cooler temperature, are noted. Clients are notified of problems as soon as possible so that a contingency plan can be initiated if necessary. Samples are logged-in and are delivered to the assigned storage areas. Samples (including subsamples, extracts, etc.) must be stored away from standards, reagents, food, and other potential contaminants. Following log-in, every project is reviewed by the assigned PM. Upon completion of the review, the client receives an electronic summary, referred to as the "Login Review Report" that details the project information. This summary allows the client an opportunity to make changes to the project before samples are analyzed. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for additional information.

7.2 Internal Custody Logs

Some clients may specify additional custody tracking of the samples once they have been logged in. Internal custody may require that samples are stored in a manner that ensures limited access. The internal custody log (FRMQA015) shall accompany the samples from log-in through completed analysis. The person responsible for the work signs and dates each entry and/or page in the logbook. When all data from a sample set is compiled, copies of all logbook entries shall be included in the final report package. For projects requiring internal custody, ACZ will adhere to the procedure described in the SOP *Client Service Policies and Procedures* (SOPAD043).

7.3 Sample Tracking

Sample flow through the laboratory is facilitated by the use of an Oracle-based LIMS database (Laboratory Information Management System). Every product (requested analysis) logged into the LIMS for a sample has a specific, pre-determined department path. All products have default paths of at least Login Review and Reporting. Between these two departments, a product may go through, for example, Soil Prep and Metal Analysis or Soil Prep, Organic Prep and GC Analysis. At each department step in a product's path, the status can be updated and viewed at any time. Analytical product statuses are defined below. Additional information regarding sample tracking is available in the SOP *Client Service Policies and Procedures* (SOPAD043).

NEED	Prep or Analysis has not been started
WIP	Prep or Analysis has been started (Work In Progress)
PREP	Sample preparation is complete and sample is ready for analysis
UPLD	Analytical data has been uploaded into LIMS
AREV	Analyst has reviewed and accepted analytical data
SREV	Supervisor has reviewed and accepted analytical data
DONE	Analysis or task has been completed

[3] Currently, samples scheduled for any radiochemistry parameters are held for a minimum of 90 days to facilitate radioactive material inventory monitoring. This policy is subject to change at any time in harmony with ACZ's Radiation Safety Plan.

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REDO	Sample requires reanalysis
REDX	Sample requires re-digestion/extraction
CANT	Sample preparation or analysis cannot be performed
NREV	Project requires PM review before work can begin
HOLD	Prep or analysis postponed
SENT	A final report has been issued to the client

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8 PROCUREMENT, INVENTORY AND TRACEABILITY OF SUPPLIES

8.1 Procurement / Inventory

All consumable supplies are purchased from reputable vendors that have been evaluated for service, quality, and price. To the extent possible, materials traceable to national or international standards of measurement are purchased for use in technical operations. Supplies are purchased using ACZ's purchase order (PO), remote inventory management system (RIMS), and the Aestiva ordering system. The Purchasing Agent is not permitted to make a substitution for any material specifically requested unless the department supervisor approves the substitution. Upon receipt, reagents, chemicals, standards, and other laboratory consumables are stored in the Chemical & Supply Room, which has limited access, or are delivered to the laboratory. Refer to ACZ's SOP *Purchase, Receipt, and Storage of Consumable Materials for Technical Operations* (SOPAD037) for additional information.

8.2 Glassware

ACZ uses only laboratory grade glassware. Prior to use, glassware is cleaned to meet the sensitivity of the method. Refer to individual test SOPs for detailed cleaning procedures.

8.3 Other Supplies

Routine consumables (centrifuge tubes, autosampler tubes, pipette tips, etc.) are purchased through an automatic system managed by Fisher (RIMS). All other supplies are purchased on an as-needed basis through ACZ's Purchase Order and the Aestiva ordering system. Refer to SOPAD037 for additional information.

8.4 Traceability of Standards and Reagents

To provide complete traceability, each data package must reference every standard and reagent used for sample preparation or analysis, including but not limited to acids, bases, preservatives, color reagents, pH indicators, buffers, and instrument reagents. Each PCN and/or SCN must be documented either on the workgroup bench sheet, data review checklist, or a current standard/reagent form. The open date for all original containers is not tracked in LIMS; however, good laboratory practice dictates the open date be noted on the sample container.

8.4.1 Primary Control Number (PCN)

Upon receipt, all stock chemicals, standards, and reagents are assigned a unique PCN in LIMS for tracking and traceability purposes. A label with the PCN and the expiration date is affixed to both the container and the Certificate of Analysis (if applicable). Document Control enters the data for each PCN using the certified value(s) supplied by the vendor, as indicated on the Certificate of Analysis. Because the certified value is entered, the final concentrations for prepared standards may vary slightly from the theoretical value indicated in the test SOP. Certified values shall be used for standards when available. If certified values are not available, informational values may be used. Document Control maintains certificates of Analysis, and a copy of the PCN report is generated and maintained. If the certified reference values for any PCN are changed after the PCN has been used in the laboratory, then complete documentation must be provided as a major corrective action (FRMQA001).

NOTE: Only Document Control and QA personnel are authorized to enter or edit PCN data.

8.4.2 Secondary Control Number (SCN)

To ensure complete traceability, a unique SCN must be created when any intermediate or working standard is prepared from one or more stock solutions, stock chemicals, or intermediate solutions. A standardized format is used for creating the SCN: a two-letter code indicates the lab section and is followed by the prep date and then by a daily sequential number. For example, the SCN **II051128-2** denotes the second standard prepared on November 28, 2005 in the Inorganic Instrument lab. An acceptable alternative is to let LIMS assign a unique number when prompted.

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An SCN for any working standard subjected to a LIMS calculation must be created electronically in LIMS. The initial volume and concentration of each constituent and the final volume of the prepared solution are entered in the SCN Wizard program to calculate the final concentration(s) of each analyte using the formula $C_1V_1 = C_2V_2$. The preparation date, expiration date, and preparer's initials are included as part of this electronic record. A hard copy of the SCN report may be affixed to the standard/reagent logbook, depending on individual department practice; however, it is not required.

Prepared reagents do not require an SCN be created electronically in LIMS; however, preparation must be recorded in the department's designated logbook. At a minimum, the logbook entry must clearly identify what reagent was prepared, its subcomponents, the preparer's initials, the preparation date, and the expiration date. This information is sufficient for color reagents, buffer solutions, instrument reagents, etc. because details of the preparation are stated in the test SOP.

8.5 Preparation and Expiration of Standards and Reagents

8.5.1 Preparation of Standards and Reagents

Refer to individual test SOPs for detailed information regarding standard and reagent preparation.
In general, either Class A pipettes or mechanical pipettes are used to measure and dispense aliquots of any solution used to prepare a standard or reagent. Accurate delivery of mechanical pipettes must first be verified as described in ACZ's SOP *Control, Calibration, and Maintenance of Measuring and Test Equipment* (SOPAD013).

All containers of prepared reagents and standards stored for more than one day must be properly labeled with the SCN (or other unique identifier), name/description, preparation date, and expiration date. Preparation of reagents and standards must be documented as described in §8.4.2.

8.5.2 Expiration of Purchased Standards and Chemicals (PCNs)

When provided, the manufacturer's expiration date will be assigned. If the manufacturer does not provide an expiration date, an expiration date of 5 years from receipt is assigned.

An expired stock material may continue to be used only if its reliability can be verified. For the purpose of ensuring transparency, the rationale for extending the expiration date must be documented on FRMQA051 and submitted to the QA department for approval. If the extension is granted, FRMQA051 is filed with the certificate of analysis. Unusable materials must be replaced and the standard or reagent remade as soon as possible. Remove the container from the lab or the supply room and dispose of properly. Contact ACZ's HWC for assistance.

8.5.3 Expiration of Prepared Standards

Storage conditions and shelf life for prepared standards are provided in the individual test SOPs. The following guidelines may be used to determine the shelf life for a prepared standard if the method does not prescribe a shelf life:

1) A standard that has been prepared in-house may continue to be used after its assigned expiration date for as long as its reliability can been verified. Whenever possible, reliability should be verified by comparison to another, unexpired standard containing the same constituents. For applicable procedures, instrument response may be considered when determining whether or not a solution is still reliable.

    - In cases where reliability has been verified, the expiration date of the SCN must be updated in LIMS and/or the standard/reagent logbook. The rationale for extending the expiration must be documented on FRMQA051 and submitted to the QA department for approval.

    - In the event the solution was used prior to updating the SCN then documentation must be provided as part of the workgroup to indicate the solution was used past the shelf life stated in the SOP (a minor corrective action or FRMQA051 may be used if more than one workgroup is affected). The expired standard must be remade as soon as its reliability becomes questionable – it is the responsibility of the analyst to use their best judgment.

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2) The shelf life of any prepared standard with any analyte concentration < 10 mg/L is 90 days from the preparation date. This is a general guideline – if any constituent does not remain in solution for 90 days, then the standard must be prepared more often. If the manufacturer's expiration date for any stock standard is sooner, then the expiration date of the SCN is the manufacturer's expiration date for a single analyte solution or the earliest manufacturer's expiration date for a multiple analyte solution.

3) The shelf life of a prepared standard with analyte concentration ≥ 10 mg/L is one year from the preparation date. This is a general guideline – if any constituent does not remain in solution for one year, then the standard must be prepared more often. If the manufacturer's expiration date for any stock standard is sooner, then the expiration date of the SCN is the manufacturer's expiration date for a single analyte solution or the earliest manufacturer's expiration date for a multiple analyte solution.

4) In general, prepared Radiochemistry standards expire one year from the preparation date. The solution may be re-evaluated using control charts, efficiency checks, or other criteria and the expiration date extended by year intervals if the solution is still deemed usable. Refer to the specific test SOP for details.

8.5.4 Expiration of Reagents

In general, a reagent is a solution, which does not contain the target analyte(s). Storage conditions and shelf life are stated in the individual test SOPs. The expiration date can be extended for a prepared reagent provided its reliability can be verified. LCS/LFB performance (QC criteria met) may be used to verify reagent stability if the control standard is a valid indication of the reagent's continued functionality/stability. Reagents used to treat samples for interference may not be verified this way. Reagents used to dissociate complexed target analytes may not be verified this way unless the LCS is an appropriate complex. FRMQA051 must be submitted to QA for approval whenever an expiration extension is requested.

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9 MAINTENANCE AND CALIBRATION OF INSTRUMENTATION & EQUIPMENT

9.1 Maintenance of Instruments and Support Equipment

The best protocol for producing quality work is to prevent errors and non-conformances rather than react to and correct problems after they occur. An essential part of this protocol is ensuring that all laboratory instrumentation and equipment used for the generation of data have been optimized and are functioning properly before commencing work on client samples. Performing routine maintenance and optimizing instrument-operating conditions prior to sample analysis minimizes instrument downtime, thereby improving productivity and ensuring quality of the data. It is the responsibility of the designated analyst(s) to perform and properly document daily and routine maintenance, instrument optimization, troubleshooting, instrument service or repair, and repair or replacement of parts.

All manufacturer-prescribed inspection and maintenance shall be performed according to the schedule indicated in the operator's manual (or similar) provided by the manufacturer and must be documented in the instrument logbook, a separate maintenance logbook, or on the instrument maintenance checklist (available in LabWeb). ACZ management recognizes that performing all maintenance procedures at the frequency indicated by the manufacturer may not be necessary to sustain instrument optimization. Therefore, at a minimum, instrument part(s) and optimization shall be inspected according to the schedule. The analyst must use their professional judgment to determine if maintenance or replacement is necessary at that time. Decisions to deviate from the manufacturer's schedule shall be documented.

All support equipment (any device that may not be the actual test instrument, but is necessary to support laboratory operations) must be monitored regularly to confirm proper functioning. The temperature of all drying ovens, refrigerators, freezers, and incubators must be checked each day the equipment is in use and each check recorded on the associated Temperature Logsheet. Refer to SOPAD013 for more detail.

Equipment that does not meet performance specifications must be taken out of service and FRMAD029 attached to indicate the instrument or equipment is waiting for repair and cannot be used. During this downtime the department supervisor, Production Manager, and Project Manager may collectively determine it is necessary to sub-contract samples until correct performance of the repaired instrument or equipment has been demonstrated by a successful calibration or other suitable test. Document all contact with the manufacturer, as well as all repairs and other services, in the instrument or maintenance logbook to be used as a reference for solving future instrument problems. Transport and storage of measuring equipment shall be done in accordance with manufacturer recommendations. Additionally, when instrumentation or equipment goes outside of the direct control of the laboratory, the functioning and calibration status must be checked and shown to be satisfactory before it is returned to service. Refer to SOPAD013 for additional information.

To minimize downtime and prevent analytical delays, each laboratory should maintain an adequate inventory of reagents, stock standards, glassware, etc. and should keep a sufficient supply of extra "critical" parts in-house. Instrument redundancy should be established for all analyses and Instrument Qualification (IQ) should be maintained on backup instruments.

9.2 Instrument Calibration

The accuracy of all instrument-generated data relies on proper calibration. In general, calibration or standardization involves defining the relationship between instrument response and the amount or concentration of analyte introduced into the instrument. The graphical depiction of this relationship is referred to as the calibration curve.

Calibration frequency must be performed in accordance with the manufacturer's guidelines, test method or other regulatory requirements, or client contract stipulations, whichever is most stringent. Every calibration or standardization must meet the acceptance criteria stated in the SOP and shall be subsequently verified by analyzing an initial calibration verification standard (ICV) or other control standard (if specified in the SOP) that contains all target analytes and has been prepared or obtained from a different source than the one used to prepare the calibration standards.[4] Calibration standards and the second-source

[4] If a second source standard is not available calibration shall be verified using a standard from a different lot. If a different lot is not available, an analyst who did not prepare the calibration standards may prepare the calibration verification standard. For some standards, it is important to consider whether manufacturer's have obtained their material from the same lot.

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verification standard should be prepared on different days. If they are prepared concurrently, then another qualified analyst should prepare the second-source verification standard. This minimizes the risk of both solutions being prepared consistently incorrectly.

A continuing calibration verification standard (CCV) containing all analytes of interest must be analyzed at the frequency stated in the test SOP to ensure the stability of the initial calibration curve has not varied over time due to any change in the analytical instrument and its detection system, such as instability of standards, instrument cleanliness, column performance, matrix effects, flow changes, and changes within the laboratory environment.

For applicable methods, all initial and continuing calibration steps must be clearly detailed in the test SOP. Additionally, each test SOP must specify the frequency and acceptance limits for the calibration and subsequent verification (ICV and CCV). In general, acceptance criteria are method-specific; however, the SOP may also include requirements of other regulatory agencies. Prior to resuming sample analysis, immediate corrective action must be taken if the calibration, ICV, or CCV is outside of the acceptance criteria. Technical corrective actions are described in the individual test SOPs. Refer also to §11.2 for additional information.

General calibration guidelines are listed below. Additional information is provided in the individual test SOP's and ACZ's SOP *Control of Measuring & Test Equipment* (SOPAD013).

- Understand the method requirements for calibration (minimum number of standards, etc.)
- Use the correct calibration model (linear, second-order, etc.)
- Include all target analytes in the calibration standards and second-source standard
- Analyze a calibration standard with a concentration less than or equal to the reporting limit.[5]
- Do not remove points from the middle of the calibration (only high or low standards may be dropped).
- Calibration is a single-event process. A retest of a calibration standard must be performed immediately.
- Documentation and resolution of calibration abnormalities is critical

[5] In general, the concentration of the low calibration standard is equal to the reporting limit, because lesser values are qualified as estimated; however, actual lab practice may differ and must be stated in the test SOP.

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10 CONTROL AND STORAGE OF RECORDS AND DOCUMENTS

A formal and systematic control of records and documents is necessary to accurately reconstruct the entire history of any sample and guarantee the quality and defensibility of the data. All information pertaining to instrumentation and equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, data verification, audits, corrective actions, method validation, and data reporting must be documented, must identify all personnel involved, and must be readily understood. All records, including those pertaining to calibration and test equipment, certificates and reports, must be maintained, and the management system must facilitate the retrieval of all working files and archived records for inspection and validation purposes. Documents and records must be safely stored (protected against fire, theft, loss, environmental deterioration, and vermin) and must be held secure and in confidence to the client for a minimum of 10 years. The hard copy of all records and documents must be maintained in a designated storage area with limited access. To the extent possible, hard copies for the most recent two (2) years are stored on-site, and if necessary, may be moved to off-site storage after two years. Off-site storage conditions must meet the same criteria that apply to on-site storage.

10.1 Workgroups

10.1.1 Changes made to any workgroup record (hardcopy or electronic) must be documented.

1) If a workgroup is “dissolved” or its data deleted from LIMS, the analyst is prompted in LIMS to provide an explanation of why he/she is performing the task.

2) Changes to upload files must be documented on the hard copy of the workgroup.

10.1.2 Workgroup data that is re-uploaded *for any reason* must first be deleted. If any of the data changes, the Run Approval report shall be corrected. The workgroup shall be rescanned if necessary.

10.1.3 Document Control or other administrative personnel use a multi-page scanner with its own PDF scanning software to scan all hardcopy portions of workgroups.

1) Before the workgroup is scanned, the top page is reviewed to make sure it has both the AREV and SREV initials and dates.

2) The person scanning the workgroup must initial in the lower right hand corner of the front page of the workgroup. This provides an indication the document has been scanned.

3) The workgroup is scanned to the designated network directory and is then moved through an automated process to the appropriate read-only LabWeb directory. This directory is accessible to all employees. When a workgroup is rescanned, the previous file is maintained. A copy will be automatically created so as not to overwrite any files and will have a letter appended; starting with “A” the first time the workgroup is rescanned. <u>The most current file will not have a letter appended</u>.

10.1.4 The hard copy is filed by workgroup number in a file cabinet in the locked supply room by the front office. When capacity is reached, the workgroups are boxed and prepared for long-term storage. The front of the full storage box is labeled with the year and the workgroups contained in the box. The first box of each new calendar year is “1.” Full boxes are consecutively numbered, transferred to a designated location and stored in numerical order. The storage room is locked at all times and access is limited to authorized staff.

10.1.5 When workgroups moved to storage are accessed a checkout card must hold the place of the workgroup in the file and must indicate who removed the workgroup, the workgroup number, and the date the workgroup was removed. The checkout card is removed when the workgroup is returned.

10.2 Electronic File Retention & Storage

All electronic records, stored either on instrument computers or on the network, are systematically backed up to

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both fixed and removable media. These records include Oracle data, instrument raw data, workgroups, client reports, instrument upload files, SOPs and other controlled documents, voice mails, and department data.

10.2.1 Critical system data is protected by Microsoft's Data Protection Manager. The Data Protection Manager is configured to maintain data for a period of 10 years.

10.2.2 All archived data is moved to a secondary machine on a weekly basis. From there, it is backed up to removable media to provide additional data redundancy.

10.2.3 The removable media from the first week of the month is pulled from service and moved to ACZ's safe deposit box at a local bank. The most recent 6 months of tapes are kept in the bank safe deposit box. Months 7 through 12 are placed in a secure, data rated, 4-hour fireproof safe that is located in the CFO's office. Note that this removable media only contains data from December 1st of the previous year to the present date.

10.2.4 At the conclusion of the calendar year, a master copy is made that comprises all of the data from December 1st of the previous year through January 31st of the following (current) year. This 14-month span of data is then moved to ACZ's safe deposit box at a local bank. At that time, the removable media that has aged 10 years is removed from the safe deposit box and its contents are destroyed. All data on the secondary machine from prior to December 1st of the previous year is removed from the system so that it is no longer included on the weekly backup.

10.2.5 Data that has aged 5 years is deleted from the Oracle Database on a monthly basis.

10.3 Instrument Data Files

Instrument raw data files are backed up by ACZ's Instrument Data Backup Application (IDBA). IDBA is a program that accesses local directories from instrument computers. Each morning the program retrieves and backs up individual data files from the specified directory on each instrument computer. Refer to ACZ's SOP *Backup and Archive of Instrument Data Files* (SOPAD044) for details.

10.4 Client Reports

10.4.1 Client reports are generated and signed electronically and are automatically stored as a PDF at a designated location on the network that has limited access. If a copy of any report exists on the network, and a new report is generated, then the existing copy will be renamed so that it is not overwritten. This way ACZ maintains a copy of all reports generated for a client.

10.4.2 Hardcopy documentation associated with a client project (CCOC, invoice, Login Review Form, etc) is filed by project number and stored in the document storage location.

10.4.3 Electronic Data Deliverables (EDD) are stored on the network at a designated location.

10.4.4 Once a project has been invoiced, the working directory is moved to the designated storage network location as a read-only PDF. If a project is un-invoiced, the project folder is copied back to the working directory where changes may take place. If an invoice is altered, a revised invoice is included with the project hardcopy.

10.4.5 In general, changes are not allowed to projects (including compilation) if the project has been invoiced. If a change needs to be made, the project must first be un-invoiced. At the time of un-invoicing, the user must provide a reason in LIMS to explain why the project was un-invoiced. This information is then stored in the Oracle database.

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10.4.6 If a test report requires amendment after it has been issued to the client, the entire report shall be re-issued with the amendment. The amended report shall include a case narrative describing change(s) from the original report. Amended reports shall be uniquely identified and contain a reference to the original report. Typically, the laboratory number assigned to the project serves as the link to the original report. Amendments shall meet all the requirements of ACZ's quality system.

10.5 Documents

10.5.1 Standard Operating Procedures

10.5.1.1 Refer to §2.2 for additional information pertaining to SOPs.

10.5.1.2 The original master copy of each SOP is stored as a PDF in a secured public directory. The cover page indicates approval authorities. Approval is documented through emails. Whenever an SOP or SOP revision is approved, QA emails all staff whose job activities intersect the SOP or SOP revision with training instructions and a request for read receipt. The pertinent technical director shall be included in the email distribution list. This email constitutes QA's signature of approval. Read receipts constitute training signatures and the technical director's signature of approval. Emails requesting read receipts for training documentation purposes shall contain a statement that by sending read receipt the sender attests they have read, understand, and agree to follow the identified policy. MS Outlook's read receipt is mimicked for this process. To send read receipt, trainees and approval authorities reply to the sender and replace "Re" with "Read" at the beginning of the subject field. Emails documenting training and/or approval shall be converted to PDF's and stored in the same directory as the SOP master copy or a subdirectory therein. If applicable, an SOP Control Form (FRMQA003) is kept with the master copy. This form shall indicate each controlled copy of the SOP that was issued, the issue date, and which lab division the copy was distributed to

SOP master copies pre-dating the above paperless policy are located in the document control office.

10.5.1.3 All controlled copies of the previous version are collected and disposed of when a new version of any SOP becomes effective. The collect date is documented on the SOP Control Form maintained with the associated master copy SOP. LabWeb is configured so that only QA & IT staff can print or copy text from SOPs.

10.5.1.4 A printed controlled copy of any SOP may be requested from QA.

1) Each lab or department will be issued a controlled copy of any relevant test SOPs, upon request. Issued controlled copies must not be removed from the assigned area for an extended period of time and may not be photocopied. Additional controlled copies of any SOP or individual replacement pages of an SOP will be also distributed upon request.
2) To ensure outdated information is not inadvertently used as a reference, an uncontrolled copy of any SOP or SOP excerpt is not allowed unless issued by QA. Additionally, an electronic copy of any SOP becomes obsolete and must be deleted from a network drive or email once the effective version has been uploaded to LabWeb.

10.5.1.5 SOP Revisions: Any revision to a procedure must be approved by QA before changes may be implemented.

1) SOP revision proposals shall be submitted by the pertinent department supervisor (exceptions may be granted on a case by case basis) to QA through email. The email shall include:
   a. The full document ID & title in the subject line.
   b. A desired effective date.

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c. The section(s) with proposed revisions. Strikethrough red font shall indicate proposed deletions. Blue font shall indicate proposed additions. (An editable copy or section of the SOP may be requested from QA to facilitate this activity.)

2) If QA approves the revision(s), an email will be sent to all pertinent staff stating the specific revision(s) & requesting a read receipt to document training.

3) The revision will then be incorporated into the electronic SOP stored on LabWeb.

4) On the effective date, all controlled copies of the SOP will be collected from the laboratory and new ones issued with the revision(s) incorporated. This activity shall be documented on the SOP control form.

5) The email from QA (#2 above) and records of read receipt shall be converted to PDF's, compiled, and stored in the same directory as the SOP master copy. The name of the PDF compilation shall be the effective date of the revision.

10.5.2 When documents are found to contain conflicting policies or procedures, the most recent document will be followed unless the conflict is prescribed as an exception to general protocol by a document more specific to the application.

10.5.3 All controlled forms must be printed from LabWeb and may not be stored on a separate network drive. If photocopies are used then any unused copies of the expired version must be disposed of as soon as a new version is uploaded to LabWeb. This ensures that the effective version of any controlled form is in use at all times. Exceptions may be granted by the QAO on a case by case basis.

10.5.4 Any controlled SOP(s) issued to an employee must be collected upon resignation or termination.

10.5.5 Only Document Control and QA personnel are authorized to enter or edit data for a PCN.

10.5.6 The hard copy of each PCN report generated in LIMS is stored in a three-ring binder that is maintained by the Document Control department.

10.5.7 The original certificate of analysis for any stock material, if provided, is attached to the hard copy PCN report.

10.5.8 Accreditation certificates are stored as PDF files to a designated network location. Original copies are maintained by QA. Certificates are also posted to ACZ's website.

10.5.9 Original calibration certificates and related documentation for support equipment (including but not limited to pipettes, thermometers, and glass micro liter syringes) are maintained by the QA Department.

10.5.10 LIMS and other problems pertaining to IT are documented and managed by the electronic system called Help Desk. If an employee encounters a problem that requires attention, that employee will submit a request through Help Desk. The request requires a priority be assigned. This system allows ACZ to track all changes made to computer systems. Help Desk requests are evaluated regularly by IT and upper management.

10.6 Records

10.6.1 Records include, but are not limited to: all logbooks; phone logs; raw data, derived data, and calibration data; training documentation (training forms, MDL studies, DOCs, etc.); proficiency testing results; calibration and certification records; internal audit reports; external audit reports; corrective action reports; management reports; and regulatory correspondence.

10.6.2 Records related to sample log-in are maintained as described in SOPAD045.

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10.6.3 Records related to support equipment calibration and calibration verification are maintained as described in SOPAD013.

10.6.4 Certificates of cleanliness and volumetric accuracy received with consumable supplies (e.g. sample containers, centrifuge tubes) shall be submitted to and maintained by QA. Any other type of certificate that does not have a defined storage location shall be submitted to QA.

10.6.5 Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, dictated observations, and recorded data from automated instruments.

10.6.6 Original copies of records, except those pertaining to analytical data, are maintained by the QA department or Document Control, and access is limited.

10.6.7 Relevant qualifications, training skills, and experience of technical personnel are maintained in the employee's training file.

10.6.8 Records such as transcripts, applications for employment, performance evaluations, etc. are maintained in the personnel files, which are stored in the secured office of the CFO.

10.6.9 The DOC certification statement (FRMAD023), initial method training form (FRMQA004), General Lab Practice Training Form (FRMQA047), and Method Calibration Training Form (FRMQA050) are filed with the workgroup if the DOC was logged-in; otherwise, the DOC package is filed in the method files. An analyst training spreadsheet referencing training dates and documentation locations is maintained on a public drive.

10.6.10 Each employee's legal name, legal signature, and initials are documented on the New Employee Checklist (FRMAD043). The form is maintained in the employee's personnel file, which is stored in the CFO's office. Additionally, employee names, signatures, and initials are documented in a logbook maintained by ACZ's CFO. In the event an employee legally changes their name, the CFO is responsible for garnering new signatures and initials in the logbook; FRMAD043 is not updated in this event.

10.6.11 Each Organic Instrument ICAL data package is scanned to the designated network directory as a PDF and the hard copy stored in labeled boxes. Alternatively, a PDF may be generated directly from the instrument files. ICAL information that needs to be attached to any subsequent workgroup(s) must be printed from the PDF.

10.6.12 Logbooks shall be maintained and controlled as described in SOPAD013.

10.6.13 Project Managers are responsible for maintaining all emails pertaining to a client and/or project. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043).

10.6.14 Changes to electronic records must be traceable to the individual who made the correction, and the reason for the change must be provided. Erroneous entries cannot be destroyed by methods such as overwritten files.

10.6.15 Record Storage and Retention

10.6.15.1 The minimum record retention period of 10 years may be increased dependent upon client request, regulatory requirement, or civil action order.

10.6.15.2 Records stored by a computer must have hard copy or software backup copies.

10.6.15.3 Records stored only on electronic media must be supported by the hardware and software necessary for their retrieval and utilization in the proper format.

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10.6.15.4 Records stored on electronic media must be stored in a way to provide protection from electronic or magnetic sources.

10.6.15.5 If there is a change in ownership and/or a change in location, all records and documents will be made available to clients for 10 years. Under no circumstances shall any records or documents be destroyed – all records and analyses performed that pertain to TNI accreditation are subject to inspection by the TNI accrediting authorities for a 5 year period. (The 10 year record retention policy is client driven, TNI standards require records be retained for a minimum of 5 years.) A new owner of ACZ will assume possession of all records and documents.

10.6.15.6 If ACZ goes out of business, all records and documents will be stored and maintained according to protocol in a location to be determined at the time of closure.

10.6.16 Access to Archived Records

10.6.16.1 Access to archived information must be documented with an access log. A log is kept in each storage location, and any person entering a storage location must provide the required information in the log.

10.6.16.2 Hard copy records are stored in a locked environment with limited access. When a record is removed from its location, a "checkout card" must be filled out to indicate who removed the record, the date the record was taken, and a description of the record. The card marks the place in the storage box, and when the record is returned the card is pulled from the box.

10.6.16.3 Any changes to be made to archived electronic data will require assistance from IT to do so.

10.6.16.4 Electronic data that has been archived to removable media is stored in a bank safety deposit box. Access is limited to ACZ's CEO and CFO and recorded in a logbook maintained by the CEO.

10.6.17 Record Disposal

10.6.17.1 Records are disposed of in a manner that ensures client confidentiality.

10.6.17.2 Stored records will be reviewed to determine which ones can be destroyed in compliance with ACZ's record retention policies.

10.7 Computer Data and Records

10.7.1 Network File Server

Computer files pertaining to all aspects of ACZ's business are stored on a series of file servers. To gain access, an employee logs on to the "LAB" domain. Each employee has a unique network user name so that security rules may be enforced. No "guest" logon is permitted. Every employee belongs to a specific "group" and directory security is enforced through privileges granted to these groups. An employee is granted access to files that pertain to their job functions. Other files will be granted read-only or no access as appropriate to the employee's position.

Data generated and reported by ACZ is extremely confidential and the company may be liable for the consequences of the release of this data to any unauthorized person. The implementation of password security is not arbitrary and ensures data is protected and cannot be disclosed to outside parties. Weak passwords that are not changed frequently make this scenario more likely.

In general, the network will prompt employees to change their password every 30 days. The password must be at least five (5) characters. Numeric characters are optional. Passwords may not be shared with other employees, unless necessary for work purposes. The use of another employee's password without permission from ACZ's

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CEO, QAO, or IS Manager (with the exception of common passwords for shared computers) is grounds for disciplinary action.

10.7.2 LIMS Server

a. Information stored in LIMS consists of all sample and client information needed for day-to-day production activities. The information is stored in an Oracle database. Access is controlled through membership in "groups." Employees may update and change database records according to their job responsibilities. Otherwise, information is restricted to read-only access or no access.

b. No modifications to data can be made through applications not authorized by ACZ's IT department unless a CAR or Help Desk ticket is submitted or documentation is provided on the hardcopy of the workgroup. Unauthorized applications include any form of direct database manipulation.

c. Tracked changes will be audited on a regular basis by the QA department or its designee to ensure sufficient information is being supplied as to why changes occur. The explanations must be both professional and specific.

10.7.3 Docs Server

Access to the docs server is read-only and is permitted through Internet Information Services (IIS) authentication and is logged in IIS log files. The server is updated on a regular basis by automated scripts.

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11 ELEMENTS OF QUALITY CONTROL

A critical focus of ACZ's quality control policies and protocols involves monitoring sample preparation and measurement processes to determine matrix effects and to evaluate laboratory performance. Quality control samples are typically analyzed with every batch of environmental samples. Each test SOP provides detailed information regarding quality control sample types, acceptance criteria, and corrective actions, if applicable to the procedure, and reflects the requirements of the method and/or other regulatory authorities.

Performance control samples demonstrate precision or accuracy and expose out-of-control events. Matrix-specific control samples indicate possible effects of the matrix on method performance and may also identify data as in-control or out-of-control. Data that is out-of-control dictates corrective action ranging from re-preparation and re-analysis to reporting data with qualifiers. In general, the corrective action specified in the SOP shall be performed if any quality control sample does not meet the acceptance criteria.

To the extent possible, client samples are reported only if all quality control measures are acceptable. If any measure is outside of the acceptance criteria, and the data will be accepted and reported to the client, then the appropriate data qualifier(s) must be assigned to all associated samples. The list of current extended qualifiers is maintained in the LIMS database.

11.1 Method Performance

11.1.1 Negative Control – Prep Blank (Method Blank)

A prep blank or method blank shall be analyzed at a minimum of one per batch. The blank shall be processed along with and under the same conditions as the associated samples. Method blanks are not applicable for certain analyses, such as pH, Conductivity, Flash Point, and Temperature.

The prep blank is used to assess possible contamination introduced during sample processing steps. A prep blank is prepared using Type I water or other similar matrix that is free of the target analyte(s) and contains all reagents in the same volumes used to prepare the client samples. Unless specified in the test SOP, sample concentration may not be corrected for the prep blank value.

While the goal is to have no detectable contaminants, each prep blank must be carefully evaluated as to the nature of the interference and the effect on the analysis of each sample in the batch. Contamination in the prep blank results from four principle sources: the environment the analysis is performed in; the reagents used; the supplies and apparatus used; and the analyst performing the analysis. Contamination sources vary and the test SOP must be referenced to determine appropriate corrective action.

When method blanks fail acceptance criteria, potential sources shall be investigated and measures taken to correct, minimize or eliminate the problem, and associated client samples must be reprocessed and reanalyzed. Alternatively, data may be reported with the appropriate qualifier if reprocessing and reanalysis is not possible or if one of the following criteria is met:

i) The concentration of a target analyte in the blank is at or above the acceptance limit and the measured concentration of the analyte in an associated sample is greater than 10 times the measured concentration of analyte in the blank.

ii) The concentration of the target analyte in the associated sample is less than the MDL.

iii) Corrective actions could not be performed or are ineffective. Thoroughly document any corrective action taken and the outcome.

11.1.2 Positive Control (however named): Laboratory Fortified Blank (LFB), Laboratory Fortified Blank Duplicate (LFBD), Laboratory Control Sample Water (LCSW), Laboratory Control Sample Water Duplicate (LCSWD), Laboratory Control Sample Solid (LCSS), Laboratory Control Sample Solid Duplicate (LCSSD).

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1) The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps.

2) The LCS is a quality system matrix, known to be free of the analytes of interest, spiked with known concentrations of analytes. Alternatively, an appropriate Certified Reference Material (CRM) containing the analytes of interest may be used.

3) If no separate preparation method is used (e.g. dissolved metals), an ICV or CCV may double as the LCS. If different acceptance criteria are specified, the most stringent criteria shall be observed.

4) Each test SOP must define the positive control to be used for the procedure, the required frequency, acceptance criteria, and contingencies for corrective action.

5) Unless the reference method specifies a different frequency, the LCS shall be analyzed at a minimum of one per batch, not to exceed 20 environmental samples.

6) Any affected samples associated with a failing LCS shall be re-processed for analysis or the results reported with appropriate data qualification. A failing LCS may be re-tested once to confirm the failure. Additional re-tests must be accompanied by documented corrective action taken between tests. For example, the instrument did not sample from the correct tray position in the first two tests; alignment was corrected for the third test.

   Note: In general, qualification of data for LCS failures is only permitted if there is insufficient sample for re-analysis, the data is extremely time sensitive, or the LCS failed high but the analyte was not detected above the reporting limit in the sample.

7) The components to be included in the LCS shall be as specified by the method, regulation, or as requested by the client. **In the absence of such specifications, the following rules shall be observed (Radiochemistry excluded)** :

   1) For those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike shall be chosen that represents the chemistries and elution patterns of the components to be reported.

   2) For those methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected shall be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. **However, the laboratory shall insure that all targeted components are included in the spike mixture over a two (2) year period**:

      i. For methods that include one (1) to ten (10) targets, spike all components.

      ii. For methods that include eleven (11) to twenty (20) targets, spike at least ten (10) or 80%, whichever is greater.

      iii. For methods with more than twenty (20) targets, spike at least sixteen (16) components.

8) An LCSW duplicate may be prepared and analyzed with the batch, typically in lieu of a matrix duplicate or spike duplicate. Data is acceptable if the LCSW and/or LCSWD is within the acceptance limits and the RPD passes. Associated samples must be re-prepped and reanalyzed if either of the following occurs:

   a) LCSW/D RPD fails the acceptance criteria specified in the SOP.

   b) % R of both the LCSW and LCSWD is outside the acceptance limits.

9) For a solid or semi-solid matrix, an LCSS and LCSSD are often prepared and analyzed.[6] The data is acceptable if the LCSS and/or LCSSD are within the acceptance limits and the RPD passes. Associated samples must be re-prepped and reanalyzed if any of the following occurs:

   a) LCSS/D RPD fails the acceptance criteria specified in the SOP.

[6] Corrective action for Recommendation #5 cited in the 2002 ADHS audit report.

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b) % R of both the LCSS and LCSSD is outside the acceptance limits.

10) When the acceptance criteria for the LCS are exceeded [i.e. high bias] then any associated client sample with a measured concentration less than the reporting limit (MDL or PQL) may be accepted and reported with the appropriate qualifier.

11) Refer to §11.1.3.3 for additional information regarding data assessment for solid-matrix workgroups prepared with both LCSS/LCSSD and MS/MSD.

12) An LCS is not required for those analytes for which no spiking solution is available.

13) The following apply to radiochemistry only:

a) The activity of the LCS shall be at least 10 times the Lower Limit of Detection (LLD) or Minimum Detectable Activity (MDA). Note: this requirement does not apply to DOCs.

b) Whenever possible, the standards used to prepare the laboratory control sample shall be from a source independent of the standards used for instrument calibration.

c) Where a radiochemical method, other than gamma-ray spectroscopy, has more than one reportable analyte isotope (e.g. plutonium, 238Pu and 239Pu, using alpha-particle spectrometry), only one of the analyte isotopes needs to be included in the laboratory control sample at the indicated activity level. However, where more than one analyte is detectable, each shall be assessed against the specified acceptance criteria.

d) Where gamma-ray spectrometry is used to identify and quantify more than one analyte, the laboratory control sample shall contain gamma-emitting radionuclides that represent the low (e.g., Am^{241}), medium (e.g., Cs^{137}) and high (e.g., Co^{60}) energy range of the analyzed gamma-ray spectra. As indicated by these examples, the nuclides need not exactly bracket the calibrated energy range or the range over which nuclides are identified and quantified.

e) The laboratory control sample shall be prepared with similar aliquot size to that of the routine samples for analyses.

11.1.3 Sample Specific Controls

The effect of different sample matrices on the performance of any method can be profound; therefore, matrix spikes, duplicates, and surrogate compounds are analyzed to evaluate matrix effects on data quality. Each SOP includes specific information regarding the usage and evaluation of matrix-specific QC samples and also states the required corrective action to take if any matrix QC fails.

ACZ provides analytical services to numerous and varied clients. Therefore, the possibility of routinely favoring one client or sample is highly unlikely, and over time the samples from all routine sources should be fortified. ACZ recommends that analysts, to the extent possible, select samples to spike or duplicate that are representative of the workgroup. Analysts are not to associate QC with a client sample known to be or believed to be any type of blank or Proficiency Testing sample. Several exceptions exist for selecting samples for spiking or duplicating:

1 A sample is not spiked or duplicated if the volume is inadequate and the client sample and QC sample(s) would require dilution; however, if no other option is available then the client sample and Duplicates should be prepared and analyzed on the same dilution whenever possible. Matrix spikes will not be accepted on different dilutions (minor d.f. variations in soils samples are acceptable) unless no other alternative exists. The data must be qualified in this event.

2 Use the same weights (as close as possible) to prepare duplicates of solid matrix samples.

3 A client may require that one or more of their samples be spiked or duplicated. A “RUN QC” comment is added when the sample is logged in to notify the analyst that QC must be performed for a specific sample or project. Alternatively, the Special Instructions function in ACZ’s LIMS may be used to communicate the request. If a client requests that their sample(s) be spiked or duplicated, ACZ will accommodate the client for a fee.

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4 A reactive sample is unpredictable and is a poor choice for spiking or duplicating.

5 A PT sample is not a real-world sample and is a poor choice for spiking or duplicating, because the data does not provide any useful information about possible matrix effects. When selecting samples for batch QC such as spikes or duplicates, PT samples should be avoided. If insufficient volume exists to spike or duplicate any other samples in the batch, it is appropriate to select a PT sample. It is better to use the PT sample for a duplicate than a spike if this choice is presented. If a batch consists solely of a PT sample, QC designed to assess matrix effects is not required (e.g. spike, SDL); an assessment of precision is still required and may be accomplished by duplicating the PT sample, or preferably, running a duplicate of the positive control.

11.1.3.1 Surrogates

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but are not normally found in environmental samples. Surrogates are included in the scope of Organic methods and are used to evaluate accuracy, method performance and extraction efficiency and are added to environmental samples, controls, and blanks, in accordance with the method requirements.

When surrogate recoveries fail acceptance limits, corrective action stated in the test SOP shall be performed. If corrective action cannot be performed or is ineffective, reported data must be appropriately qualified.

11.1.3.2 Matrix Spike Samples

A matrix spike sample (however named) is used to determine the level of bias (accuracy) associated with a particular matrix. For the purposes of this document, “MS” designates a matrix spike, and “MSD” designates a matrix spike duplicate. Spikes are prepared by adding a known and appropriate quantity of each target analyte to a replicate aliquot of client sample.

The required analytical frequency is specified by the method or other regulating entity and is indicated in the test SOP. Each result is evaluated against the acceptance criteria, and matrix effects are determined and reported to the client. The following evaluation criteria apply to spikes that are subjected to processing steps and post-digestion spikes (analytical spikes).

- Percent Recovery (%R) is considered for all spikes.
- %R is evaluated only if the theoretical concentration in the spiked aliquot is greater than or equal to the reporting limit; otherwise, each associated client sample must be reported with the appropriate qualifier, regardless of %R.
- If %R for the MS and/or the MSD is outside of the acceptance limits, the RPD passes, and all other pertinent prep and instrument QC passes, each associated client sample may be accepted and reported with appropriate qualification.

11.1.3.2.1 The components to be included in the MS & MSD shall be as specified by the method, regulation, or as requested by the client. **In the absence of such specifications, the following rules shall be observed (Radiochemistry excluded)**:

1) For those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike shall be chosen that represents the chemistries and elution patterns of the components to be reported.

2) For those methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected shall be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. **However, the laboratory shall**

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insure that all targeted components are included in the spike mixture over a two (2) year period:

a) For methods that include one (1) to ten (10) targets, spike all components.

b) For methods that include eleven (11) to twenty (20) targets, spike at least ten (10) or 80%, whichever is greater.

c) For methods with more than twenty (20) targets, spike at least sixteen (16) components.

11.1.3.3 Matrix Duplicates and Matrix Spike Duplicates

The matrix-specific precision associated with an analysis is determined through the use of a matrix duplicate (DUP) or spike duplicate (MSD), which are performed at a frequency specified by the method or other regulating entity (refer to the specific test SOP). If the method does not prescribe a frequency, a duplicate shall be included in each workgroup, not to exceed 20 samples. The results are evaluated, and the matrix affect on precision are determined and reported to the client.

- Relative Percent Difference (RPD) is used to evaluate precision, unless the test SOP specifies a different technique (§12.4.6).
- RPD for a spike duplicate is evaluated only if the observed concentration is greater than or equal to the reporting limit; otherwise each associated client sample must be reported with the appropriate qualifier.
- RPD for a matrix duplicate is evaluated only if the observed concentration is greater than 10 times the MDL or 2 times the PQL if an MDL has not been established; otherwise each associated client sample must be reported with the appropriate qualifier, regardless of RPD.
- In the absence of other contributing factors, a DUP failure for a solid or semi-solid matrix is attributed to non-homogeneity of the sample, and each associated client sample may be reported with the appropriate qualifier.
- For an aqueous matrix, if the DUP fails then all associated samples must be retested. If permitted by the instrument software the sample and DUP can be reanalyzed at the end of the analysis in lieu of retesting all associated samples.
- For an aqueous matrix, if the MS/MSD RPD fails then the associated samples must be reanalyzed. If permitted by the instrument software the sample and MS/MSD can be reanalyzed at the end of the analysis in lieu of retesting all associated samples.
- If applicable, evaluate the LCS/LCSD if the RPD fails for a matrix duplicate or spike duplicate. Each associated client sample may be reported with the appropriate qualifier if the LCS/LCSD meets the criteria stated in §11.1.3.2.
- For a solid or semi-solid matrix, if both the LCSS and LCSSD recoveries pass but the RPD fails, then acceptable precision may be demonstrated by a passing RPD for the MS/MSD, and each associated client sample may be reported with the appropriate qualifier.
- A sample and duplicate may only be re-analyzed once before additional corrective action is required. If more than one re-analysis is performed, the workgroup documentation must include justification.

11.2 Instrument Specific Controls

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All data must be associated with a passing instrument calibration and initial calibration verification. To the extent possible, all data must be associated with passing continuing calibration verification. If the initial calibration verification results (ICV/ICB) are outside of the acceptance criteria, then the source of the failure must be identified, necessary corrective action performed, and the instrument recalibrated if necessary before proceeding with sample analysis.

If the continuing calibration verification results (CCV/CCB) do not meet the acceptance criteria, then the source of the failure must be identified and corrective action performed, including recalibration if necessary, before continuing with sample analysis. If reanalysis of any sample associated with failing calibration verification is not possible and results will be reported, the data shall be appropriately qualified.

For instruments that permit the analysis of subsequent workgroups using the most recent calibration, two (2) consecutive attempts of the opening CCV/CCB are allowed. If both attempts fail to produce acceptable results then the sources of failure must be identified and corrective action performed, including recalibration if necessary, before commencing sample analysis.

If a CCV or ICV retest fails and the instrument is not recalibrated, 2 consecutive passing CCVs or ICVs are required before continuing with analysis.

Unless stated otherwise by the test SOP, passing calibration verification must bracket all batch quality control samples, and results for additional instrument check standards, if applicable, must be within the acceptance criteria stated in the SOP. However, when the acceptance criteria for a CCV or CCB are exceeded (i.e. high bias) any associated client sample with a measured concentration less than the MDL may be accepted and reported with the appropriate qualification. This exception is not allowed if the workgroup contains a batch LCS (however named) which fails low.

11.3 Other Control Indicators

11.3.3 Internal Standards

Internal Standards (IS) are measured amounts of certain compounds added after preparation or extraction of a sample to be analyzed. The IS is an analyte not likely to be found in the environment and is used in a calibration method to correct sample results affected by column injection losses, purging losses or viscosity effects. The IS is added to client samples, controls and blanks in accordance with the method requirements. When the results are outside of the acceptance limits for applicable quality control samples, corrective actions shall be performed. Once system control has been reestablished, all samples analyzed while the system was malfunctioning shall be reanalyzed. If corrective actions could not be performed or are ineffective and associated sample results will be reported, the data must be appropriately qualified.

11.3.2 Trip Blank

The trip blank is a sample container filled in the laboratory with Type I water that is shipped to the collection site in the sample cooler, returned to the laboratory, logged-in, and analyzed in the same manner as the client samples. With the exception of Hg-1631, trip blanks are not opened in the field.

11.3.3 Instrument Blank

The instrument blank is an aliquot of Type I water processed only through the instrument steps of sample analysis and is used to determine presence of instrument contamination. For Organic instrument methods, neither surrogate nor IS standards are added.

11.3.4 Equipment Blank

An equipment blank is provided by the client and is used to assess the effectiveness of equipment decontamination procedures. Type I water is poured into (or over) or pumped through the sampling device, collected in a sample

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container and transported to the lab to be analyzed for all parameters requested for the environmental samples collected at the site.

11.3.5 Ambient Blank

The ambient blank consists of Type I water poured into a VOA vial at the sampling site (in the same vicinity as the associated samples). It is handled like an environmental sample and transported to the laboratory for analysis. Ambient blanks are prepared when samples are to be analyzed for VOA analytes and are used to assess the potential introduction of contaminants from ambient sources (e.g. active runways, engine test cells, gasoline motors in operation) to the samples during sample collection. The frequency of collection for ambient blanks is specified in the client's field-sampling plan. Ambient blanks are not required for all projects.

11.3.6 Radiological Tracers & Carriers

Radiological tracers and carriers are used for radiological analyses. The control reacts in the same manner as the target isotope and is used to assess analyte recovery. The tracer is added to client samples and QC in accordance with the requirements stipulated in the test SOP. Because the tracer recovery has a direct impact on the LLD, the recovery must be high enough to yield LLDs that are within the scope of the project or meet ACZ's acceptance criteria. Refer to the test SOP for evaluation criteria and corrective action(s) for out-of-control tracer recovery.

11.4 Titrants – Where applicable, test SOPs shall include procedures for verifying the concentration of titrants prepared by the laboratory. Verification is not required for purchased titrants with certified values. If a purchased titrant is diluted, verification is required.

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12 EVALUATING QUALITY CONTROL SAMPLES

In general, acceptance criteria for quality control samples are method-specific; however, compliance with the requirements of clients and regulatory or other accrediting agencies must also be demonstrated. Immediate corrective action must be taken if any quality control is outside of the acceptance criteria. Appropriate corrective actions are described in the test SOP. To the extent possible, client samples are reported only if all quality control measurements are acceptable. If a quality control measure is outside of acceptance criteria, and the data will be reported, then all samples associated with the failed QC must be appropriately qualified. Clients will occasionally request limits different from those in a published method. Deviations from ACZ's policies pursuant to client request must be explicitly noted on client reports. ACZ will not be held liable in the event such deviations do not meet client regulatory needs.

Unless otherwise stated, for the purpose of determining conformance to specifications, ACZ employs the rounding method described in ASTM E29. When using this method, observed values are rounded to the same decimal place limits are expressed before assessing conformance. For example, if the calculated percent recovery for an LCS is 89.5% and the QC limits are 90 to 110%, the percent recovery would be rounded to 90% and evaluated as passing. (Note: double rounding is NOT permitted, e.g. 89.48 rounds to 89.5 rounds to 90.) Conversely, if limits were expressed as 90.0 to 110.0%, the same LCS would be evaluated as failing acceptance criteria. Analysts must consider whether the QC limits expressed in the test method cohere in a technically sound manner with the rounding method. If they do not, the SOP must express the limits to a technically sound numerical place value, or the absolute method must be employed. The absolute method takes a limit of 6˚C, 6.0˚C, and 6.000˚C all to mean the same thing, exactly 6 degrees Celsius.

For methods that permit the use of control charts or do not specify acceptance criteria for quality control measurement, limits may be generated by plotting historical data obtained from analytical processes considered in control. Whenever practical, a minimum of 20 data points is used. The process of rejecting data points relies heavily on the statistician's judgment and control chart activities are therefore restricted to supervisors and experienced analysts. All points must be associated with passing calibrations and calibration verification(s). Data points with known anomalies must be rejected. Data points should not be rejected solely because they fail acceptance criteria. Control chart documentation must clearly indicate rejected data points. ACZ's LIMS has a utility for querying and retrieving historical data for control chart applications. Control chart limits are typically set at ± 3σ. All control chart limits are reviewed and approved by the QA department prior to implementation. When possible, a comparison to previous limits is included in the review and may form the basis for rejecting new limits and requiring an investigation of the analytical system's condition. Previous limits are archived in a network folder. Default acceptance criteria established by the Arizona Department of Health Services (ADHS) may be used in lieu of generating a control chart to establish limits; however the SOP must specify which limits are in use.[7] **NOTE:** For all data evaluation, final results ending with 1 – 4 are rounded down and results ending with 5 – 9 are rounded up.

12.1 Accuracy

Accuracy is defined as "The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations". Control samples (LCS or LFB) and spiked samples are analyzed with every batch of samples or as stipulated by the specific test SOP to assess accuracy and matrix effects.

- Percent Recovery (%R) for a control sample is calculated as follows:

$$\%R = \frac{M}{S_p} \times 100$$

Where: M = Measured concentration of the control sample
S_p = True value of the control sample

[7] Arizona Administrative Code (A.A.C.), Title 9, Ch. 14, Article 6, Exhibit II (December, 2006)

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- Percent Recovery (%R) for a spike is calculated as follows:

$$\%R = \frac{M - S}{S_p} \times 100$$

Where: M = Measured concentration of the spiked sample
S = Measured concentration of the sample aliquot
S_p = True value of the spike concentration

12.2 Precision

Precision is defined as "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." Matrix duplicates and spike duplicates are analyzed with every batch of samples or as stipulated by the test SOP to determine the precision associated with the analysis. If any method does not specify acceptance criteria for the RPD, then default criteria of RPD $\leq$ 20 is used (a value that rounds to 20 is acceptable). [8] The Relative Percent Difference (RPD) as an absolute value is calculated as follows:

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

Where: S = Sample Value
D = Duplicate Value

12.3 Other Calculations

- Solids Dilution Factor:

$$\text{Dilution Factor} = \frac{V}{(W)(\%\ solid)}$$

Where: V = Final digestate volume, in mL
W = Sample weight used, in g
%solid = %solid of the sample as a fraction

- Sample Concentration for Solids:
  - wet weight [biota tissue, fruit or vegetable matter, etc.]: $mg/Kg = \frac{DF * C * V}{W}$
  - dry weight [plant matter, grasses, soil, sludge, etc.]: $mg/Kg = SF * C * DF$

Where: DF = instrument dilution factor
C = raw data value, in mg/L
V = Final volume of digestate, in L
W = sample (as received) weight used, in Kg
SF = soil dilution factor

- Percent Difference for Serial Dilution (SDL):

$$|\%D| = \frac{[I - (s * 5)]}{I} \times 100$$

Where: I = initial sample result
s = serial dilution result (raw data value)

For SDL calculations in LIMS, "s" is multiplied by 5 and the resulting "reg value" is compared to the "found value" to calculate %D.

[8] ADHS Information Update #87 (July 7, 2005)

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12.4 Radiochemistry Calculations: (**NOTE**: Specifications in the individual test SOPs supercede the information detailed below.)

12.4.3 Activity

The results of radioactivity are typically reported in terms of activity per unit volume or mass. Units are normally expressed in picocuries (pCi), which equal 2.22 disintegrations per minute (dpm). Specific formulas to determine activity are in the SOP for each method. The general formula is as follows:

$$C = \frac{R_{net}}{(e)(y)(i)(v)(u)}$$

Where: C = activity per unit volume (pCi/L)
R_{net} = net counts per minute
e = counting efficiency, cpm/dpm
y = chemical yield
i = ingrowth correction factor
v = volume or mass being counted (L)
u = units correction factor, 2.22 for cpm to pCi

12.4.4 Counting Error

Radiochemical data are considered incomplete without reporting associated random and systematic errors. For this reason all radiochemical results should be accompanied by a counting error at the 95% confidence level (1.96*standard deviation). The general counting error formula is as follows:

$$E = \frac{1.96(R_o / t_1 + B / t_2)^{1/2}}{(e)(y)(i)(v)(u)}$$

Where: E = counting error
R_o = gross sample, cpm
t_1 = sample count duration, min
B = background, cpm
t_2 = background count duration, min
e, y, i, v, and u are as previously defined.

12.4.5 Lower Limit of Detection (LLD)

LLD (also referred to as Minimum Detectable Activity or MDA) is considered the smallest quantity of sample radioactivity that will yield a net count for which there is a pre-determined level of confidence that radioactivity is present. At the 95% confidence level, the following equation calculates the LLD for any single nuclide. The calculation uses the standard deviation for the background counting rate, assuming the sample and background counting rates should be very similar at the LLD. The formula for determining LLD is as follows:

$$\mathrm{LLD}_{95} = \frac{4.66 S_b}{(e)(y)(i)(v)(u)}$$

Where : LLD_{95} = Lower limit of detection at the 95% confidence interval
S_b = Standard deviation of the instrument background counting rate, cpm
e, y, i, v, and u are as previously defined

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12.4.6 Precision

The normalized absolute difference, or Replicate Error Ratio (RER), between the sample and the laboratory duplicate, given by the following equation shall be used to determine that results do not differ significantly when compared to their respective 2* sigma uncertainty.

$$RER = \frac{|Sx - Dup|}{\sqrt{(Sx_{error})^2 + (Dup_{error})^2}} \leq 2.0$$

Where: Sx = sample concentration in pCi/L
Sx_{error} = sample counting error (in pCi/L) at the 95% confidence level.
Dup = duplicate concentration in pCi/L
Dup_{error} = duplicate counting error (in pCi/L) at the 95% confidence level.

NOTE: For Radchem samples, both RPD and RER may be used to evaluate precision. RPD is the default assessment for Drinking Water samples; RER is the default assessment for non-Drinking Water samples. Data for both RER and RPD are uploaded to LIMS for all analyses. Use the following guidelines to correctly assess precision. Further details are provided in ACZ's Wiki and should be consulted to ensure data for each workgroup is correctly evaluated. Go to LabWeb \ Wiki \ Analytical Departments \ Radio Chemistry.

Drinking Water:

RPD $\leq$ 20, RER < 2.0 – Precision is judged to be in control
RPD $\leq$ 20, RER > 2.0 – Precision is judged to be in control;
RPD > 20, [sx] < 5x [LLD], RER < 2.0 – Precision is judged to be in control; qualify data.
RPD > 20, [sx] > 5x [LLD], RER> 2.0 – Precision of the prep batch is questionable.
RPD > 20, [sx] > 5x [LLD], RER < 2.0 – Precision of the prep batch is questionable.

Non-Drinking Water:

RER < 2.0, RPD $\leq$ 20 – Precision is judged to be in control.
RER < 2.0, RPD > 20 – Precision is judged to be in control;
RER > 2.0, RPD $\leq$ 20 – Precision of the sample prep batch is questionable.
RER > 2.0, RPD > 20 – Precision of the sample prep batch is questionable.
RER > 2.0, [sx] > 5x [LLD], RPD $\leq$ 20 – Precision is judged to be in control; qualify data.

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13 VALIDATION AND REVIEW OF ANALYTICAL DATA

ACZ has a responsibility to provide the best data possible to ensure our clients can make sound and cost-effective decisions regarding public health and the environment. In order to generate and report reliable data, the analytical systems used need to be properly functioning, and the review process must be conducted in a manner that is logical and reasonable and would be defensible if subjected to legal scrutiny. Decisions regarding data quality must be meaningful and must be backed by good science and sound professional judgments.

The entire validation and review process encompasses more than solely evaluating the final results for client and quality control samples. To this extent, the necessary steps must also be performed *prior* to sample preparation or analysis to ensure the quality of the data. Following sample analysis, data is uploaded to the LIMS database and then submitted to a variety of process chains such as calculations, rounding, application of qualifiers, etc. A multi-level data review process is utilized to verify the uploaded analytical data meets all documented ACZ requirements as well as any client-specific quality objectives. At a minimum, the validation process must include the following steps, as applicable:

- Monitor the expiration dates for all stock, intermediate, and working standards, reagents, and chemicals.

- Prior to analysis, determine that holding times have not been exceeded. Unless otherwise specified by the test SOP, sample preparation and analysis must be completed within the holding time.

- Prior to analyzing samples, verify the correct set-up and operation of the instrument or equipment. Perform calibration, maintenance, and optimization as necessary to ensure proper functioning.

- QC Association
  1) In general, for QC frequency of 1 per10 or less client samples, the first set of QC is associated with samples 1 – 10. If there are fewer than 20 samples in the workgroup, then the remaining client samples are associated with the second set of QC.
  2) If sample characteristics or amount dictate that 2 of the first 10 samples be spiked or dup'd, then the first spike or DUP is associated with samples 1 through 11 excluding the 2nd sample spiked or dup'd, and the 2nd spike or dup is associated with itself and samples 12-20. For example, if samples 3 & 5 are spiked in a 20 sample batch, sample 3 is associated with 1-4 & 6-11, and sample 5 is associated with 5 and 12-20. The same principle applies if both spiked or dup'd samples reside in the 2nd set of ten within the workgroup sequence.
  3) Variations to the QC association rules noted above are permitted but must be documented with the WG. The documentation must define the altered QC association and provide a compelling, technically sound reason for the deviation. QC association may **not** be changed after data has been acquired.
  4) QC association must be properly defined in LIMS.

- Before completing workgroup creation, verify the correct PCNs and/or SCNs have been entered. Percent recovery for control samples and spikes is calculated using the information in LIMS for each.

- Verify the proper sub-sample (green dot, yellow dot, etc.) is being used for preparation or analysis.

  - Notify the supervisor or Production Manager as soon as possible if a sample cannot be located.
  - Document on the bench sheet if a sub-sample other than the type indicated in the SOP is used.

- Clearly label tubes, beakers, autosampler cups, etc. to identify the sample (and dilution factor, if applicable).

- Manage sample volume to ensure all analyses from a bottle type can be completed.

- Document all dilution factors at the time the dilution is performed.

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- Record complete and accurate observations when an analysis, sample preparation, or sample matrix is unusual or problematic.

- Ensure transcription errors do not occur. Verify all data manually entered into LIMS is correct before completing the upload process.

- The calibration workgroup must be associated with all subsequent workgroups. Record the calibration workgroup number (or calibration file name) on the data review checklist.

- Provide complete traceability for all standards and reagents used for sample preparation and analysis.

- Batch quality control samples must be treated in the same manner as client sample, including preparation.

- If it is necessary to perform a calculation manually, use the values in the raw data [do not truncate] and then round the final result to the appropriate numerical place value. If the final result passes the acceptance criteria then pass the QC in LIMS and note on the data review checklist that it passes.

- LIMS performs several additional QC calculations on the approved data including cation/anion balance (CAB) checks, calculated TDS versus actual TDS ratios, and Total versus Dissolved ratios. The Project Manager may update the status of pertinent samples to REDO if one of these calculations indicates a discrepancy with the associated data.

- If two attempts fail to produce acceptable data then notify the supervisor or Production Manager before taking further action. It may be necessary to first determine if a larger problem is interfering with the analysis. Investigate the problem before qualifying the associated data.

- If there is an indication the analytical system is out of control, the issue must be investigated. Notify the supervisor immediately. Conduct troubleshooting in a systematic, organized manner.

- All data must be reviewed initially in LIMS [AREV] by the analyst who performed the analysis or another individual authorized to perform or AREV the procedure. The department supervisor or another individual authorized for SREV performs the secondary review [SREV]. The following are data review guidelines:

  1 A data review checklist must be completed during the review process. Verify all items listed and note any errors, problems or non-compliances and the corrective action(s) taken.

  2 If applicable, review the raw data to verify the analytical system was in control and to ensure no anomalies exist. Check for notes on the bench sheet regarding the preparation or analysis.

  3 For client samples and quality control samples, ensure all results are within the measurement range and are bracketed by a passing calibration and passing calibration verification [ICV/ICB or CCV/CCB]. Sample values outside of the measurement range must be appropriately qualified if reanalysis is not possible.

  4 The corrective action specified in the SOP must be performed if any quality control sample does not meet the acceptance criteria.

  5 Data generated after the hold time has elapsed may not be usable for the client. If reprep or reanalysis will be conducted outside of the holding time, check first with the supervisor.

  6 Confirm all dilutions are appropriate. A reasonable explanation must be provided on the bench sheet if a sample was diluted and the value is less than the quantitation limit (refer also to §15).

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7 If a spike fails, determine if the sample concentration is disproportionate to the spike added. If the analyte concentration in the sample is more than 4 times the spike concentration, note the failure on the checklist and appropriately qualify the associated samples.

8 If a spike recovery suggests the sample was not spiked, matrix interference must be confirmed prior to qualifying samples. If matrix interference cannot be confirmed, then re-prep and/or re-test all associated samples.

9 Each associated client sample must be appropriately qualified if the matrix spike, matrix duplicate, or spike duplicate data cannot be used for validation purposes.

10 Confirm failed QC by verifying the correct PCN or SCN was entered. Make corrections if necessary before proceeding with data review.

11 Verify all assigned qualifiers are appropriate. Does use of a particular qualifier make sense? Could data be defended using the qualifier assigned to the scenario or problem?

12 If a case narrative is necessary, the reason for accepting and reporting the data must be sound and logical. Provide sufficient and accurate verbiage to ensure the data is legally defensible.

13 If a sample was retested in the same workgroup, verify the correct data will be reported. All other data for the sample must be failed; LIMS cannot report multiple datum for the same sample-product-analyte combination.

14 Confirm all samples have the correct status (PASS, FAIL, REDO, REDX) before completing the review process. For multi-parameter workgroups, all analytes must have the correct status.

15 Refer also to §11 for data evaluation criteria.

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14 DETECTION LEVELS

Current practice identifies several detection levels, each of which has a defined purpose: Instrument Detection Limit (IDL), Method Detection Limit (MDL), Reporting Limit (RL), and Practical Quantitation Limit (PQL). The MDL and PQL are stated in each test SOP and are adjusted accordingly in LIMS when data is uploaded to reflect the use of smaller sample volume (dilution) or larger sample volume (concentration).

14.1 Instrument Detection Limit (IDL)

The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the selected analytical mass(es). (EPA Method 200.8 definition.)

14.2 Method Detection Limit (MDL)

The EPA defines the MDL as the "minimum concentration of substance that can be measured by a specific testing protocol and reported with 99% confidence that the analyte concentration is greater than zero…" This confidence interval means that any substance detected at a concentration equal to the MDL is 99% likely to be present, but it also means there is a 1% chance that the substance will be considered falsely present (false positive). The MDL procedure is designed so that the probabilities of both false positive and false negative errors are acceptably small; however, the procedure has limitations. Data users must understand the limitations when evaluating low level data and must proceed with caution when interpreting data reported between the MDL and PQL in order to minimize the risk of making poor environmental decisions.

MDLs are dependent on variables (temperature, instrument conditions, analysts, matrix, etc.) and are typically determined by processing, preferably over the course of several days, at least seven individual replicates of a fortified blank sample through the method's preparation and analytical schemes. MDLs determined for the same method / matrix / technology must be compared to ensure they are in agreement.

ACZ maintains a current MDL for each applicable method. A qualitative verification of the MDL must be performed annually for each applicable method, analyte, instrument, and matrix and before a new instrument or method is utilized for client samples. Refer to ACZ's SOP *Demonstration of Capability & Method Detection Limit Studies* (SOPAD001) for additional information.

14.3 Practical Quantitation Limit (PQL)

The PQL represents the lowest quantitative level that can be reported with a specified degree of confidence. Data reported at or above the PQL is considered reproducible, allowing for comparison of analytical results over a relatively long period of time, which is important to the monitoring of environmental data. ACZ *typically* defines the PQL as a value 2 – 10 times the MDL with an accuracy of 70 to 130% in a matrix free of interferents. The low calibration standard shall be at or below the PQL. Reported values less than the PQL are qualified as estimated. The region between the MDL and PQL is a continuum of uncertainty, lacking distinct cutoff points, and the error below the PQL is increased to the extent that the statistical validity of the result is questionable.

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15 SAMPLE DILUTIONS

Sample dilution may be necessary for one or more of the following reasons: (1) sample concentration exceeds the established measurement range of the procedure/method; (2) sample volume or material is limited; (3) matrix interference is indicated or suspected; (4) sample matrix is reactive; (5) aqueous sample contains high sediment; (6) color, odor or other physical characteristics are present; (7) For ICP and ICPMS, TDS is greater than 2000 mg/L. In all cases, the analyst must use good professional judgment when determining the most appropriate dilution. Whenever possible, prepare and analyze client samples and any complimentary duplicates or spikes on the same dilution.

For samples that contain high concentration of analyte(s), the analyst will use their knowledge of the measurement range of the procedure to determine an optimal dilution that yields quantifiable data with minimal error propagation. In general, prepare the dilution so the final concentration is near the mid-point of the measurement range. A sample must be retested on a smaller dilution if analyte concentration is less than the reporting limit; exceptions must be explained on the bench sheet. For multi-parameter analyses, it may not be practical to report all analytes within the desired range, and the analyst must use their best judgment when determining a reasonable dilution factor.

The following requirements pertain to all dilutions:

- Document all dilution factors, and the reason for dilution, when the dilution is performed.
- Assign the appropriate "D" qualifier if data for the diluted sample is less than the reporting limit
- Retest sample on smaller dilution if the result is less than the reporting limit (or document justification for accepting the data on the bench sheet or data review checklist)
- Document the reason for any dilution on the bench sheet [not required for sample values that exceed the measurement range of the procedure]

16 ERROR CORRECTION PROTOCOL

When an error occurs in any type of record it must be crossed out with a **single line**. The error must not be erased, deleted, overwritten, obliterated, or made illegible. Alterations to make data legible are considered error corrections. The correct value must be entered alongside. All changes to hard copy records must be initialed and dated by the person making the correction. [9]Under no circumstances may White-Out® or any other substance be used to conceal data. Concealing or improperly altering data is fraudulent and may be grounds for termination from ACZ. Equivalent measures must be taken to avoid loss or change of original data in the case of records stored electronically. Refer to §10 for details of corrections made to electronic records. The following is an example of proper error correction:

fleece BWC 10-20-06
Mary had a little lamb, it's ~~feet~~ as white as snow. And everywhere that ~~Lary~~ went, the lamb was sure to go.
Mary BWC 10-20-06

[9] There is one exception to this rule. Client identification may be obliterated from a record if it's presence compromises client confidentiality (e.g. client ID is mistakenly entered in a logbook). In this event, the rationale for obliteration must be clearly stated and initialed and dated by the person making the correction.

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17 COMPUTER / AUTOMATED PROCESSES

ACZ employs its proprietary LIMS (Laboratory Information Management System) to acquire, record, process, store, and archive data. LIMS is the primary application for all employees and encompasses the combination of hardware and software throughout the entire facility. Tasks performed with LIMS include but are not limited to creating workgroups, reviewing data, and generating client reports. ACZ implements the defined standards of Good Automated Laboratory Practices (GALP) to establish a uniform set of procedures to assure that all LIMS data used by our clients is reliable, credible, and legally defensible.

17.1 Software

The software used to achieve GALP goals is a combination of industry standard commercial off the shelf (COTS) software and internally developed applications. COTS software is purchased through professional and well-developed companies such as Oracle, Microsoft, and Lab Vantage Systems that complete sufficient testing and quality control to assure their products function properly. Internal applications undergo testing before being implemented and distributed throughout the laboratory.

Electronic records are protected, backed up, and archived to prevent unauthorized access or amendment. Refer to §10 of this document and ACZ's SOP *Backup and Archive of Instrument Data Files* (SOPAD044) for details.

17.2 Hardware

ACZ deploys many servers using industry standard architecture. All servers run standard enterprise operating systems such as Microsoft Windows Server and SuSE Linux. All data residing on network servers is routinely backed up.

To the extent possible, instrument PCs comply with at least the minimum recommendations of the instrument manufacturer and they are connected to ACZ's network. This allows transparent backup and access to computers by system administrators.

17.3 Security

GALP security is controlled through a set of passwords. A log-in name and password are required to access ACZ's network. User passwords must be at least five characters and must be changed when the user is prompted. Each user has a given set of network rights and is restricted to software necessary to complete their job functions as well as his/her own documents. Refer also to §10.7.1 for additional information.

A firewall protects the network from internet traffic. The only traffic permitted access to the internal network is protocols approved by ACZ such as IMAP, SMTP and HTTP. Incoming and outgoing E-mails are scanned for viruses and content. Email that fails this automated scan is quarantined for further review. Web traffic that is potentially harmful or inappropriate is automatically blocked by ACZ's proxy server.

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18 CLIENT SERVICES

18.1 Contracting Services

ACZ's sales representatives and project managers are responsible for reviewing requests, preparing quotes, and entering contractual commitments with clients. Prior to accepting new work, it must be verified that the laboratory has appropriate facilities and resources to meet client needs. To the extent possible and pragmatic, ACZ shall use the latest valid edition of a standard. This is dictated largely by what ACZ's accrediting authorities will issue certification for. Where an older standard is universally recognized by ACZ's accrediting authorities but the latest is not, ACZ will typically use the older standard. As necessary, sales representatives and project managers must collaborate with ACZ's Production Manager, QAO, and/or technical directors to evaluate laboratory capacity, capability, and resources. Refer to SOPAD043 for additional details.

18.2 Subcontracting

ACZ utilizes subcontract labs to perform analyses for various reasons. A subcontracted lab must meet the clients DQOs and laboratory certification requirements for the subcontracted analysis. When applicable, ACZ advises its clients in writing of its intentions to subcontract any portion of the testing to another party. Any non-accredited tests shall be clearly identified as such to the client. ACZ scans this report as an attachment to be included as part of ACZ's final report. A comment is added to ACZ's final report indicating which subcontracted laboratory performed the analyses, if the name is not indicated on the attachment. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for additional information.

18.3 Data Reporting

Once all analyses and the entire review process have been completed, a client report is generated and submitted for final validation by the Project Manager. If necessary, a case narrative is written describing the details of the project and any non-conformances or other relevant issues. The PM electronically signs the report, and the Document Control department sends the report to the client in an electronic format. At a minimum, the following information appears on an ACZ analytical report:

Client Name	Sample Matrix
Client Address	Parameter/Analyte
Client Contact	Method Reference
Lab Sample ID	Result
Client Sample ID	Units
Client Project ID	LIMS Qualifier (U, B, J, H)
ACZ Report ID	MDL or LLD
Date/Time Sampled	PQL
Date/Time Received	Analyst's Initials
Date/Time Analyzed	Extended Qualifiers (as separate page)

A complete electronic data package contains the analytical reports, the external chain of custody records, sample shipping documentation, and any other relevant project information. Department Reference Sheets explaining acronyms, qualifiers, and method references are also included. All of these documents are an integral part of the final data package and must always be viewed as a whole. To prevent the separation of reports, each page identifies the project number, the sequential page number, and the total number of pages in the data package. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for more detail.

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If requested by a client, custom and standard Electronic data deliverables (EDDs) are generated by the Document Control department. These deliverables, containing data in client specified format, are sent by e-mail with the client report. EDDs and analytical reports access data from the same Oracle tables, thus eliminating the possibility of inconsistent results. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for more detail.

Results may be reported in a simplified manner for internal customers or in accordance to a written agreement with a customer.

18.3.1 ACZ Report Packages

ACZ provides different levels of data packages based on client request. ACZ defines the different levels as follows:

Level 2: Standard analytical reports

Level 3: Standard analytical reports; Electronic Data Deliverable (EDD); Standard QC summary

Level 4A: Standard analytical reports, Extended QC Summary (standard QC plus calibration verification checks, interference checks and serial dilutions) EDD, raw data and run logs. This package can be provided either on a disk or in a full paginated data package with the raw data

Level 4B: "CLP like" data package: CLP like forms 1-12; Run Logs and raw data incorporated into the full paginated data package.

NOTE: Surcharges apply for non-standard reports.

18.4 Data Confidentiality

ACZ has an obligation to each client to maintain custody of samples, data, and reports and to keep all data or other information confidential. To uphold this responsibility, ACZ retains custody of the information at all times – data or other client information obtained by ACZ is not allowed to leave the premises. This includes but is not limited to Chains of Custody, raw data, workgroups, run logs, logbooks, reports, QC summaries, data packages and other media containing data. Client data cannot be released to anyone except the client (as directed on the Chain of Custody) or the client's designated representative, and project data, including any client information, is not to be discussed with anyone other than ACZ employees and/or the client without first receiving written permission from the client. Additionally, client-specific information is not to be documented on raw data, workgroups, logbooks, or other records that may be provided to any client as part of an extended data package. All information must be referenced using only the ACZ log-In number. Refer to ACZ's SOP *Data Integrity Principles and Policies* (SOPAD039) for additional details of policies pertaining to confidentiality.

External access to the ACZ network is limited to employees that may need to access information remotely. Employees requiring such access use ACZ's Virtual Private Network (VPN). The VPN client is setup on the employee's computer so that it adheres to ACZ security standards. These standards include (1) a unique user name (2) a password with at least 12 characters, and (3) 128 bit encryption of data to and from the client from the ACZ servers. After the VPN server has authenticated the employee, the employee must logon to the ACZ domain through normal domain security in order to access any ACZ network resources. Most employees initiate a "Remote Desktop" connection to their office PCs, thus ensuring that ACZ data is never accessible from the client PC hard drive.

18.5 Client Feedback

Handling client feedback is a joint effort between QA, Project Managers, Production Supervisors, and Client Service representatives. If a client has a concern or complaint, either a Project Manager or Client Service Representative takes the call and initiates the feedback procedure by documenting the complaint or problem and requesting the assistance of the Production Supervisor and/or QA Officer. If the issue cannot be easily resolved, then it must be documented using FRMAD024, which is routed from the initiator to other appropriate parties, including the QAO if necessary. All client feedback is submitted to upper

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management as part of the Management Review of the Quality System. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for additional information.

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19 FACILITIES

ACZ Laboratories, Inc. inhabits a modern 31,000 square foot laboratory facility architecturally designed and specifically organized to ensure efficient operation and meet the needs of a large capacity analytical laboratory. Complete lists of instrumentation, balances, thermometers, & weight sets are maintained on a network drive. Incompatible activities are effectively separated.

19.1 Accommodation of Environmental Test Conditions

19.1.1 Temperature and room pressure are controlled by an HVAC system which maintains 19 independent zones. The clean room, metals lab, and organic instrument lab are kept under positive pressure to prevent contaminant infiltration. The radiochemistry and organic prep labs are kept under negative pressure to prevent the migration of fire, smoke, and chemical releases from the laboratory space. All other zones are maintained at a neutral pressure.

19.1.2 In humid environments, a sudden rise in temperature can result in condensation on microcircuitry leading to problems such as reduced life cycle, inaccurate readings, corrosion, etc. Due to the laboratory's location at 6730 feet above sea level, these concerns are irrelevant and humidity monitoring is only required for desiccators and the clean room.

19.1.3 Servers have a 20 minute backup power supply. If there is an interruption in power, the IS Manager receives a text. This provides sufficient time to ramp down the servers.

19.2 Security

A secure facility is essential to maintaining sample and data integrity and to providing safety to employees and visitors. ACZ has an electronic security system, which controls and limits access to only authorized personnel. The following steps have been taken to ensure this security:

- All entryways are secured. ACZ has three entries equipped with proximity readers which allow access to an employee only after he/she presents their access card. Access to the front visitor entry is controlled by an interior push button monitored by ACZ staff.
- All employees are required to use their access cards to enter and exit the building.
- If any employee does not have their access card, they must sign in at the front desk and notify ACZ's CFO. This ensures a record is maintained of which personnel were in the building at any time. A temporary access card will then be activated and issued to the employee for the day. These access cards are identified by the word "Temporary" written on a scenic background.
- During normal business hours, public access into the building can be made at the front entrance and the west shipping entrance. Both doors are equipped with a buzzer.
- Visitors must enter and exit through the main entrance and must sign the register at the front desk upon arrival and before departure. A visitor pass is issued at sign in and collected at sign out. There are two types of visitor passes. A red pass identified by the word "Visitor", will not function as an access card and symbolizes the visitor requires an escort. The other visitor pass is identified by the word "Visitor Pass" written on a scenic background and will not function as an access card. This visitor does not require an escort. The determination of which pass the visitor gets is made first, by the visitor's trust level and, second, by the visitors access needs. Visitor passes must be collected when the visitor leaves for the day.
- Companies or individuals under contract to perform recurring or extensive work for ACZ are assigned an access card similar to employees. Contractor passes function as an access card for a defined period of time commensurate with the contract work.
- Emergency Exit doors are to be used only for emergency purposes. If a door is opened, an alarm will sound.
- Loaning or transferring access cards to anyone, including other ACZ employees, is prohibited.

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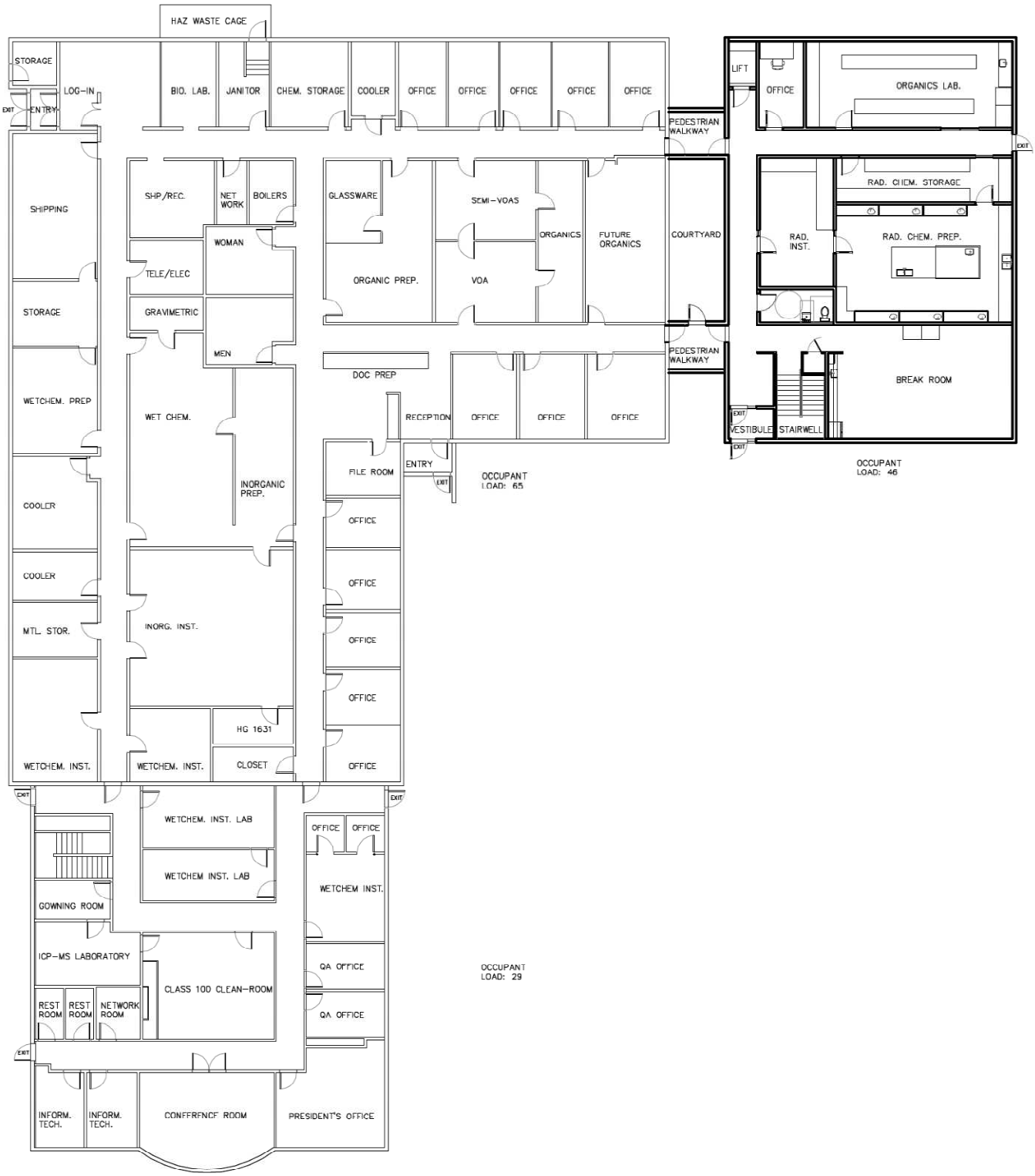

ACZ LABORATORY INC. MAIN LEVEL PLAN

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ACZ LABORATORY INC. BASEMENT LEVEL PLAN

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20 RADIOCHEMISTRY

20.1 DATA TRANSFORMATION

Unlike other laboratory divisions, ACZ's radiochemistry department utilizes excel spreadsheets to transform instrument response into final results. Spreadsheet equations are locked and password protected in order to reduce the likelihood of inadvertent modifications. Additionally, spreadsheet equations are validated by the radiochemistry supervisor or a sufficiently experienced analyst on an annual basis. Initial validation must be performed by hand calculating results. Annual validation may be performed by populating the current template with data that has been hand calculated in a previous validation and comparing the calculated results from the current template to the hand calculated results from the previous validation. Documented secondary review is required for all updates to spreadsheet templates (e.g. incorporating new mass attenuation coefficients).

20.2 INSTRUMENTATION

Radioanalytical instrumentation is located adjacent to the radiochemistry prep lab. In order to maintain appropriate temperature control in the instrument lab, separation must be maintained. The door between the two lab areas must be kept closed when not in use. Except as noted, instrument checks and other determinations must be performed and documented annually, or more often if necessary.

NOTE: To eliminate potential contamination, planchets must be stored in a covered container or in a drawer.

20.2.1 Gas-Flow Proportional Counter

20.2.1.1 Instrument Reliability Test (Voltage Plateau Determination) – The proper voltage plateau for alpha and beta is where the counting rate is consistent (should not exceed > 5% over a 150 volt change in anode voltage).

20.2.1.2 Cross Talk (Carryover) Check - Cross talk is defined as the percentage of alpha counts represented on the beta plateau. Once the amount of cross talk is determined, the cross talk settings are adjusted on the instrument to eliminate cross talk.

20.2.1.3 Detector Efficiency Curve (Self Absorption) - Efficiency curves are graphs plotting counts versus sample residue density and determine the efficiency of the alpha and beta counter as a function of sample residue density. This factor is part of the overall determination of sample activity.

20.2.1.4 Background Determination - Characteristic of most detectors is a background or instrument count rate attributed to cosmic radiation, radioactive contaminants in instrument parts, counting room construction material and/or the proximity of radioactive sources. The background is determined weekly by counting an empty planchet for 12 hours. On each day of use the instrument is checked for background drift by counting an empty planchet for 90 minutes. Background counts must fall within established control chart limits or corrective action must be taken before analyzing samples. Although most radiation measurement systems are noteworthy for their stability, sudden changes can occur due to instrument component failure, loss of gas pressure, vacuum, or contamination of a detector or sample chamber from a high activity sample. Subsequently, instrument drift in detector efficiency and background must be checked both before and after measuring samples used for drinking water compliance monitoring. Refer to individual test SOPs for additional details.

20.2.1.5 Instrument-Response Check (Performance Check) – This continuing calibration check verifies the instrument response and stability and is performed daily for each detector. For a performance check measurement, the same calibration sources must be used as for the calibration measurement in order to verify the current measuring results still match the results of the calibration measurement stored last. At the end of the check the count rates and the relative deviations from older calibration measurements are displayed. The system signals "OK" if the deviations do not exceed the maximum deviation defined by

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the user. Samples used for drinking water compliance monitoring must be bracketed by passing performance checks. Refer to individual test SOPs for additional detail.

20.2.2 Liquid Scintillation Counter

20.2.2.1 *Optimal Window* - When determining radionuclides by liquid scintillation, it is necessary to select the optimal window by counting a standard for five minutes and generating a sample spectrum. For better clarity, a log scale for the channel number axis should be used. On the graph, the region of interest is determined by the energy of the peak one is trying to quantitate. The optimal window is formed by extending this region by 10% on each side of the alpha peaks.

20.2.2.2 *Efficiency Quench Curve* – The liquid scintillation instrument, a Beckman LS 6000TA, automatically corrects for quenching by the H - Method. Refer to SOPRC010 for details.

20.2.2.3 *Background Check* - Three background blanks are run with every batch. The first two are run immediately after calibration. The third, the CCB, is employed as a measurement of instrument drift and is run immediately before the final LCS. For both checks, the counting duration must be equivalent to the longest sample counting duration.

20.2.2.4 *Instrument-Response Check Source* - This continuing calibration check verifies instrument response and stability and must be performed daily. If the source count is within two standard deviations (sigma) of the previously determined average count rate, instrument reliability and stability is established. If the source rate is outside the ±2 sigma-warning limit then the variability should be further investigated. If the source check is outside the ±3 sigma out of control limits, then no further samples should be analyzed until the problem is resolved. Resolution might include a new efficiency curve, background checks, and/or instrument maintenance. If insufficient data exists for control charts, ±10% of the initial source value is considered acceptable. The source for this check is a Tritium standard.

20.2.3 Alpha Spectrometer

20.2.3.1 *Energy vs. Channel Calibration* - Each alpha spectrometer has a set number of channels associated with it. To associate these channels to a specific alpha particle, the channels must be calibrated. One known calibrated solid source is placed into the detector and analyzed for five minutes to determine its associated channel to its calibrated energy peak. Since the energy is linear across the channels, all of the channels now have an associated energy. This determination is performed on an annual basis, or whenever maintenance is performed that could potentially affect the calibration.

20.2.3.2 *Background Checks* - Characteristic of most detectors is a background or instrument count rate attributed to cosmic radiation, radioactive contaminants in instrument parts, counting room construction material and/or the proximity of radioactive sources. Placing an empty sample tray in the counting chamber and counting it for as long as the longest sample-counting duration can determine the background rate (or a background check can be completed overnight). An overnight background determination must be completed at least quarterly.

20.2.3.3 *Instrument-Response Check Source* - This continuing calibration check verifies the instrument response and stability and is performed daily. If the source count is within two standard deviations (sigma) of the previously determined average count rate, instrument reliability and stability is established. If the source rate is outside the ±2 sigma-warning limit, then the variability should be further investigated. If the source check is outside the ±3 sigma out of control limits, then no further samples should be analyzed until the problem is resolved. Resolution might include a background check, and/or instrument maintenance. If insufficient data exists for control charts then ±10% of the true value is considered acceptable.

20.2.4 Gamma Spectrometer

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20.2.4.1 *Background Checks* –Characteristic of most detectors is a background or instrument count rate attributed to cosmic radiation, radioactive contaminants in instrument parts, counting room construction material and/or the proximity of radioactive sources. A cave background must be measured monthly and the background gross activity recorded. The cave background is determined by counting the empty cave for a period of time at least as long as the longest sample-counting duration. When drinking water samples are present in the batch, and additional background check is measured at the end of the batch to monitor instrument drift.

20.2.4.2 *Instrument-Response Check (Performance Check)*- The total activity of a calibration or check source will check the efficiency calibration currently in use and the general operating parameters of the system, including source positioning, contamination, library values, and energy calibration. This activity calculation uses the general analysis program to ensure that the total system is checked. This check is performed for every workgroup. If the performance check is within the defined acceptance limits, instrument reliability and stability is established. If the performance check does not meet acceptance criteria, then no further samples should be analyzed until the problem is resolved. Samples used for drinking water compliance monitoring must be bracketed by acceptable performance checks. Resolution might include a background check, and/or instrument maintenance. Refer to SOPRC016 for additional information.

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21 CLINICAL LABORATORY IMPROVEMENTS

Pursuant to Section 353 of the Public Health Services Act (42 U.S.c. 263a) as revised by the Clinical Laboratory Improvement Amendments (CLIA), ACZ Laboratories, Inc. may accept human specimens for the purposes of performing laboratory examinations or procedures. ACZ's clinical laboratory services are currently limited to the analysis of metals in blood and Uranium in urine.

ACZ adheres to the following guidelines as they pertain to work under the Clinical Laboratory Improvement Amendments (CLIA)

- ACZ adheres to the Health Insurance Portability and Accountability Act (HIPPA) regarding client confidentiality and the release of patient information.
- Client ID's for CLIA samples are transcribed from the CoC and adhere to the following naming protocol: **LAST NAME**_SPACE_ **FIRST NAME**_SPACE_ **DATE OF BIRTH (MM/DD/YY)**_SPACE_**SEX (M/F)**. (i.e. William F Grimes, DOB July 5, 1977, Sex: Male: would be expressed as "GRIMES WILLIAM 07/05/77 M"). In rare instances where the number of characters is greater than the Client ID field's max character length, the First Name is truncated to accommodate the other characters.
- Competency assessments: Employees analyzing samples associated with CLIA regulation are observed and reviewed for competency in performing the relevant tests semi-annually in their first year of analysis and annually thereafter. A General Supervisor or Technical supervisor is required to perform the assessment which is documented by completing the CLIA Personnel Assessment Form (FRMQA061).
- For continuing method validation of analyses that fall under CLIA regulations, ACZ analyzes CRM samples and/or single-blind spiked samples biannually. See the associated test SOP for additional ongoing analyst and method Quality Control requirements.

22 CERTIFICATIONS

ACZ has primary or secondary (reciprocal) certification with numerous states and EPA regions. Current certificates can be viewed at http://acz.com/certifications/. Each certificate contains a scope of accreditation listing each method the laboratory is accredited for by the issuing authority.

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APPENDIX A Required Container Type, Preservation Techniques, and Holding Times

Parameter	Container	Preservation [a, b]	Maximum Holding Time [c]
Alkalinity	HDPE or Glass	≤ 6.0 °C	14 days
Acidity	HDPE or Glass	≤ 6.0 °C	14 days
Ammonia (N-NH_3)	HDPE or Glass	≤ 6.0 °C; H_2SO_4 to pH < 2	28 days
Anions	HDPE	≤ 6.0 °C	28 days (Br^-, F^-, Cl^-, SO_4^{2-})
As(III), As(V)	Amber HDPE	EDTA and Acetic Acid	28 days
BOD, CBOD	HDPE or Glass	≤ 6.0 °C	48 hours
COD	HDPE or Glass	≤ 6.0 °C; H_2SO_4 to pH < 2	28 days
Color	HDPE or Glass	≤ 6.0 °C	48 hours
Conductivity	HDPE or Glass	≤ 6.0 °C	28 days
Cyanide	HDPE or Glass	≤ 6.0 °C; NaOH to pH > 12	14 days
Chromium (VI)	HDPE or Glass	≤ 6.0 °C	Refer to SOP for holding time
Dissolved Oxygen	Glass	None required	Analyze immediately
Metals (unless specified otherwise)	HDPE or [10]Glass	HNO_3 to pH < 2	180 days
Mercury (CVAA, ICP/MS)	HDPE or Glass	HNO_3 to pH < 2	28 days
Mercury (CVAFS)	Glass	5 mL 12N HCl and 5 mL/L BrCl	90 days
N – NO_2 / NO_3	HDPE or Glass	≤ 6.0 °C; H_2SO_4 to pH < 2	28 days (48 hours if unpreserved)
N – NO_3	HDPE or Glass	≤ 6.0 °C	48 hours
N – NO_2	HDPE or Glass	≤ 6.0 °C	48 hours
Nitrogen, Total Kjeldahl	HDPE or Glass	≤ 6.0 °C; H_2SO_4 to pH < 2	28 days
Oil & Grease	Glass	≤ 6.0 °C; HCl or H_2SO_4 to pH < 2	28 days
Orthophosphate	HDPE or Glass	≤ 6.0 °C	Filter within 15 minutes, analyze within 48 hours
pH	HDPE or Glass		Analyze within 15 minutes
Phenols	Glass	≤ 6.0 °C; H_2SO_4 to pH < 2	28 days
Phosphorus (Total)	HDPE or Glass	≤ 6.0 °C; H_2SO_4 to pH < 2	28 days
Sulfide	HDPE or Glass	≤ 6.0 °C; Zn acetate + NaOH to pH > 9	7 days
Sulfite	HDPE or Glass	≤ 6.0 °C; EDTA	Analyze immediately
Settleable Solids	HDPE or	≤ 6.0 °C	48 hours

[10] Glass is not a suitable container for Boron or Silicon.

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Parameter	Container	Preservation [a, b]	Maximum Holding Time [c]
	Glass		
Total Organic Carbon	HDPE or Glass	≤ 6.0 °C; HCl, H_2SO_4, or H_3PO_4 to pH < 2	28 days
Total & Dissolved Inorganic Carbon	HDPE or Glass	≤ 6.0˚C	28 days
Turbidity	HDPE or Glass	≤ 6.0 °C	48 hours
Total Dissolved Solids	HDPE or Glass	≤ 6.0 °C	7 days
Total Suspended Solids	HDPE or Glass	≤ 6.0 °C	7 days
Total Solids	HDPE or Glass	≤ 6.0 °C	7 days
Total Volatile Solids	HDPE or Glass	≤ 6.0 °C	7 days
Radon-222	Glass Vial [d]		4 days
Total Volatile Hydrocarbons	Glass Vial or jar [d]	≤ 6.0 °C; HCl to pH < 2 (water)	Refer to SOP for holding times
Total Petroleum Hydrocarbons	Amber Glass	≤ 6.0 °C	Refer to SOP for holding times
BTEX / MTBE	Glass Vial or jar [d]	≤ 6.0 °C; HCl to pH < 2 (water)	Refer to SOP for holding times
Organochlorine Pesticides	Glass Vial or jar [d]	≤ 6.0 °C; pH 5 – 9	Refer to SOP for holding times
PCBs	Amber Glass	≤ 6.0 °C	Refer to SOP for holding times
PAHs	Amber Glass	≤ 6.0 °C	Refer to SOP for holding times
BNAs (semi-volatiles)	Amber Glass	≤ 6.0 °C	Refer to SOP for holding times
VOAs (volatiles)	Glass Vial or jar [d]	≤ 6.0 °C; HCl to pH < 2 (water)	Refer to SOP for holding times
TCLP	Glass [d]	≤ 6.0 °C	Refer to SOP for holding times
Radchem (except Rn)	HDPE cube	HNO_3 to pH < 2	180 days
Metals in blood	Vial	EDTA ≤6.0˚C or -10 to -20˚C	180 days
Metals in urine	HDPE specimen jar	HNO_3 to pH <2 ≤6.0˚C	30 days

a. No pH adjustment for soil
b. Preservation with 0.008% $Na_2S_2O_3$ required only when residual chlorine is present.
c. Unless otherwise specified in the test SOP, complete sample preparation and analysis within holding time.
d. Teflon-lined septa or lid
e. Aqueous samples must be preserved at ≤6.0 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤ °C" is used in place of the "4 °C" and "< 4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

Refer to test SOPs for additional details and considerations.

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APPENDIX B – Forms for Management Review of the Quality System

ACZ Laboratories, Inc.
2773 Downhill Drive
Steamboat Springs, CO 80487

DEPARTMENT REPORT FOR MANAGEMENT REVIEW OF THE QUALITY SYSTEM

Department:_____ Quarter Ending:_____

1) **OPERATIONS:**
EVALUATE ALL OPERATIONS FROM LOG-IN TO REPORTING AS IT PERTAINS TO YOUR DEPT.

a. What company wide operations-related issues has the department encountered during the last quarter? Were any of the issues reoccurring?

b. What actions were taken to resolve the issues?

c. What actions can be taken to reduce/eliminate these issues in the future?

2) **RESOURCES & PERSONNEL:**
EVALUATE RESOURCES & PERSONNEL AS THEY PERTAIN TO THE DEPARTMENT

a. Did the department have adequate resurces (supplies, instrumentation, facilitied, etc.) and properly trained staff for the volume of work?

b. What resources must be available for the work expected next quarter?

c. What obstacles do employees within the department routinely experience that hinder efficiency/productivity?

3) **QUALITY ASSURANCE & QUALITY CONTROL:**
EVALUATE QA/QC AS THEY PERTAIN TO THE DEPARTMENT

a. Are any failed QC indicators reoccurring? If so, describe.

b. Were any changes to policies/procedures implemented during the past quarter as a result of corrective and/or preventive actions? If yes, were they effective? If no, what changes would be effective?

4) **CLIENT COMPLAINTS/ FEEDBACK**

a. What client feedback has the department encountered during the last quarter? Where any of the issues reoccurring?

5) **MISC: PROVIDE ADDITIONAL FEEDBACK**

FRMQA041.11.08.03

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Steamboat Springs, CO 80487

MANAGEMENT REVIEW OF THE QUALITY SYSTEM

DATE OF REVIEW: _____

Attendees:

SUITABILITY OF POLICIES & PROCEDURES: **SUMMARY OF MEETING**
(REVIEW FRM QA041 SECTIONS 1A, 1B, 1C, 2A, 2C, 3A, 3B, 4A)

- Do ACZ's policies and procedures accurately reflect management's Quality Policy Statement?
- Are ACZ's policies and procedures effective? If no, what changes are necessary?

REVIEW OF STAFF RESOURCES & TRAINING:
(REVIEW FRM QA041 SECTIONS 2A, 2C)

- Did ACZ have appropriate staff to handle the volume of work received during the past quarter?
- Was all staff properly trained and was training documented before independently analyzing client samples?

Geochemistry

Clean Room / Prep

Inorganic Inst / Prep

Wet Chemistry Instrument

Wet Chemistry Manual

Organics

Radiochemistry

Log-In

Client Services (Sales / PMs)

Document Control

Information Systems

REVIEW OF INSTRUMENTATION / EQUIPMENT, SUPPLIES & CONSUMABLES:
(REVIEW FRM QA041 SECTIONS 1A, 2B) (COMPARE EXPENSES TO REVENUE)

- Did ACZ have necessary and properly functioning instrumentation and equipment to perform the volume of work received last quarter?
- Did ACZ have adequate supplies and consumables on hand to perform the volume of work?

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MANAGEMENT REVIEW OF THE QUALITY SYSTEM

Geochemistry

Clean Room / Prep

Inorganic Inst / Prep

Wet Chemistry Instrument

Wet Chemistry Manual

Organics

Radiochemistry

Log-In

Client Services (Sales / PMs)

Document Control

Information Systems

REVIEW OF RECENT INTERNAL AUDITS: **QA OFFICER**
(REVIEW FRM QA041 SECTIONS 3A, 3B)

- Did the QA/QC department adhere to its internal audit schedule?
- Did any department(s) have significant issues?
- Were corrective actions completed properly and within the agreed time frame?
- Was follow-up completed for all corrective actions?

REVIEW OF RECENT EXTERNAL AUDITS: **QA OFFICER**

- Did the audit report(s) cite any repeat deficiencies?
- Did any department(s) have significant issues?
- Were all corrective actions completed properly and within the agreed time frame?
- Was follow-up completed for all corrective actions?

REVIEW OF RECENT PROFICIENCY TESTING STUDIES: **QA OFFICER**

A) Were all analyte values reported for each study?

WP	NA ☐	Yes ☐	No ☐
WS	NA ☐	Yes ☐	No ☐
RAD	NA ☐	Yes ☐	No ☐
Soil/UST	NA ☐	Yes ☐	No ☐

B) Did ACZ passed 2 out of the 3 most recent PT studies for each analyte?

WP	NA ☐	Yes ☐	No ☐
WS	NA ☐	Yes ☐	No ☐
RAD	NA ☐	Yes ☐	No ☐
Soil/UST	NA ☐	Yes ☐	No ☐

C) Were corrective actions completed within the agreed time frame for all studies?

WP	NA ☐	Yes ☐	No ☐
WS	NA ☐	Yes ☐	No ☐
RAD	NA ☐	Yes ☐	No ☐
Soil/UST	NA ☐	Yes ☐	No ☐

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MANAGEMENT REVIEW OF THE QUALITY SYSTEM

REVIEW OF RECENT CORRECTIVE / PREVENTATIVE ACTIONS: **QA OFFICER**

- What corrective / preventive actions were implemented during the past quarter?
- What trends are apparent?
- Were corrective actions completed within the agreed time frame?
- Have changes resulting from corrective / preventive actions been implemented? Are they effective?

REVIEW OF CLIENT COMPLAINTS/FEEDBACK:
(REVIEW FRM QA041 SECTIONS 4A)

- Did ACZ receive client feedback (positive or negative) during the past quarter?
- For complaints received, did ACZ adhere to its client complaint policy?
- Were complaints handled in a manner satisfactory to the client?
- Were all complaints resolved?
- What quality indicators are repeated?

REVIEW OF CHANGES IN VOLUME / TYPE OF WORK:
(REVIEW FRM QA041 SECTIONS 1A, 1B, 1C, 2A, 2B)

- Did ACZ experience a significant change in volume and/or type of work last quarter? How did we do?
- What improvements can be made?
- Is ACZ prepared for volume and/or type of work expected next quarter?

REVIEW OF ETHICS PROGRAM:

- Were all Ombudsman issues addressed in a timely manner?
- Were any data intergrity issues/concerns brought to the attention of the Ombudsman issues or dilemmas?
- Were all employees trained on ACZ's Ethics and Proactive Prevention Program (SOPAD039)?
- Was follow-up training conducted within the time frame stated in SOPAD039?

REVIEW OF DEPARTMENTS:

Geochemistry

Clean Room / Prep

Inorganic Inst / Prep

Wet Chemistry Instrument

Wet Chemistry Manual

Organics

Radiochemistry

Log-In

Client Services (Sales / PMs)

Document Control

Information Systems

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APPENDIX C REFERENCES UTILIZED BY ACZ

- “TNI Standards,” National Environmental Laboratory Accreditation Conference, (current version).
- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act," USEPA, Federal Register Vol. 67, No. 205, October 23, 2002.
- "Manual for the Certification of Laboratories Analyzing Drinking Water," USEPA, (current version).
- "Methods for the Chemical Analysis of Water and Wastes," USEPA, EPA-600/4-79-020, March 1983.
- "Test Methods for Evaluating Solid Waste," USEPA, SW-846 Third Edition, Update IV, January 2008.
- “Guidelines in Establishing Test Procedures for the Analysis of Wastewater Pollutants,” Code of Federal Regulations 40, Parts 136, 141, 143.
- "Quality Assurance of Chemical Measurements,” Taylor, J., Lewis Publishers, Michigan, 1987
- "Annual Book of Standards, Water Analysis," ASTM, 1989.
- "Quality Control in Analytical Chemistry," Kateman, G., Vol. 60, 1985.
- "Principles of Environmental Analysis, Analytical Chemistry," Keith, L.H., et al., Vol. 55, 1983.
- "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," USEPA, 1979.
- “Guidance for the Data Quality Assessment: Practical Methods for Data Analysis,” USEPA, EPA 600/R-96-084, July 2000.
- “Methods for the Determination of Metals in Environmental Samples,” USEPA, EPA 600/4-91-010, June 1991.
- “Methods for the Determination of Metals in Environmental Samples,” Supplement I [to EPA 600/4-91-010], USEPA, EPA 600/R-94-111, May 1994.
- “Methods for the Determination of Inorganic Substances in Environmental Samples,” USEPA, EPA 600/R-93-100, August 1993.
- "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," USEPA, EPA 821/B-96-005, December 1996.
- "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," USEPA, EPA 600/4-80-032. August 1980.
- “Determination of Lead-210, Thorium, Plutonium and Polonium-210 in Drinking Water: Methods 909, 910, 911, 912,” 01A0004860 (Region 1 Library), March 1982.
- “Good Automated Laboratory Practices - Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations” USEPA, 2185, 1995.
- “Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications”, ASTM E29-08

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APPENDIX D DEFINITIONS OF TERMS

Acceptance Criteria: specified limits places on characteristics of an item, process, or service defined in requirement documents.

Accreditation: verification by a competent, disinterested, third party that a laboratory possesses the capability to produce accurate test data, and that it can be relied upon in its day-to-day operations to maintain high standards of performance.

Accrediting Body: The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.

Accreditation body: Authoritative body that performs accreditation.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Aliquot: A discrete, measured, representative portion of a sample taken for analysis.

Analyte: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Spike (AS): an aliquot of client sample to which a known amount of target analyte is added and that demonstrates the absence or presence of interference in the matrix. The AS is prepared exactly the same way as the LFB, only spiking into sample instead of reagent blank, and is not prepped (digested) prior to analysis. The AS may also be referred to as a post-digestion spike.

Analytical Spike Duplicate (ASD): a second replicate analytical spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Analytical System: the combination of events, techniques, and procedures used to generate analytical results.

Analyst Review (AREV): See Primary Review.

Atomization: A process in which a sample is converted to free atoms.

Audit: a systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

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 20 environmental samples of the same matrix, meeting the above 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 20 or less prepared environmental samples (extracts, digestates or concentrates) that are analyzed together as a group. QC samples (e.g. LCS, MS, MSD) do not count towards the maximum of 20.

All required QC samples must be prepared and/or analyzed with each batch at the frequency required by the method, even if there are less than 20 client samples in the batch. If the workgroup has more than 20 samples, then sufficient batch QC must be analyzed for additional samples. Every batch of environmental samples is assigned a unique (i.e. traceable) six-digit numerical identifier called the LIMS Workgroup number.

Blank: a sample that has not been exposed to the analyzed sample stream utilized to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. See also Equipment Blank, Field Blank, Instrument Blank, Method Blank, Reagent Blank. Refer to §11.3 for types of blanks.

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Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst or laboratory may know the identity of the sample but not its composition. It is used to test the analyst or laboratory's proficiency in the execution of the measurement process.

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 applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration Curve: the graphical relationship between the known values, such as concentrations, or a series of calibration standards and their instrument responses.

Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.

Case Narrative: Additional documentation provided in the client report that describes any abnormalities and deviations that may affect the analytical results and summarizes any issues in the data package that need to be highlighted for the data user to help them assess the usability of the data.

Certified Reference Material (CRM): 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.

Chain of Custody Form: a legal 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; the collector; time of collection; preservation; and requested analyses.

Client: Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations. (ANSI/ASQ E4-2004)

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 confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors; or
- Additional cleanup procedures. (TNI)

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)

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

Continuing Calibration Blank (CCB): the same solution as the calibration blank, it detects baseline drift in the calibration of the instrument. When specified by the method, analyze a CCB immediately after each CCV, including the final CCV.

Continuing Calibration Verification (CCV): a solution of method analytes of known concentrations used to confirm the continued calibration of the instrument. The CCV is analyzed at the frequency indicated in the test SOP.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e. the data meet specified acceptance criteria)

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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.

Definitive Data: Analytical data of known quality, concentration, and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making. (UFP-QAPP)

Demonstration of Capability (DOC): a procedure to establish the ability of the analyst to generate acceptable accuracy [and precision, if applicable].

Detection Limit: the lowest concentration or amount of 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).

Digestion: A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form.

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.

Eluent: A solvent used to carry the components of a mixture though a stationary phase. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Elute: To extract; specifically, to remove (adsorbed material) from an adsorbent by means of a solvent. (Merriam-Webster's Collegiate Dictionary, 2000)

Elution: A process in which solutes are washed though a stationary phase by the movement of a mobile phase. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Equipment Blank: a sample of analyte-free media that has been used to rinse common sampling equipment to check the effectiveness of decontamination procedures.

False Positive (Type I or alpha error): concluding that a substance is present when it truly is not.

False Negative (Type II or beta error): concluding that a substance is not present when it truly is.

Field Blank: a blank prepared in the field by filling a clean container with Type I water and appropriate preservative, if any, for the specific sampling activity being undertaken.

Holding Time (Maximum Allowable Holding Time): the maximum time that samples may be held prior to analysis and still be considered valid or not compromised.

Homologue: One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, CH3OH (methanol), C2H5OH (ethanol), C3H7OH (propanol), C4H9OH (butanol), etc., form a homologous series. (*The Condensed Chemical Dictionary* G.G. Hawley, ed. 1981)

Initial Calibration Blank (ICB): a solution identical to the calibration blank and confirms the absence of background contamination in the calibration blank. When specified by the method, an ICB is analyzed immediately after the ICV.

Initial Calibration Verification (ICV): a solution of method analytes of known concentrations intended to determine the validity of the instrument calibration. The ICV must be analyzed immediately after each calibration and must be prepared from a source independent of the calibration standards, preferably purchased from a different manufacturer.

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Instrument Blank: an aliquot of Type I water or solvent processed through the instrument steps of the measurement process; used to determine presence of instrument contamination.

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. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Interference, chemical: Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Internal Standard (IS): 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.

Isomer: One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane. (Websters)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, 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.

Laboratory Fortified Blank (LFB): a reagent blank spiked with a known concentration of analyte. The LFB is analyzed exactly like a sample and determines whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the client. These protocols include the use of a Chain of Custody form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Linear Dynamic Range (LDR): concentration range over which the instrument response to analyte is linear.

Matrix Duplicate (DUP): a second aliquot of a client sample that is prepared and analyzed in the same manner as all other samples in the same workgroup. The DUP demonstrates the precision of the method.

Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known amount of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes (MS or LFM) are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate: a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Maximum Contamination Limit (MCL): the numerical value expressing the maximum permissible level of contaminant in water that is delivered to any user of a public water system.

May: denotes permitted action, but not required action.

Measurement Quality Objectives (MQOs): The desired sensitivity, range, precision, and bias of a measurement.

Measurement System: A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).

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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 client samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for the sample analyses.

Method Detection Limit: the minimum concentration of an analyte, in a given fortified matrix, that can be measured and reported with 99% confidence that the concentration is greater than zero.

Method of Standard Additions: A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Must: denotes a requirement.

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Nonconformance: An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.

Outlier (Statistical): an observation or data point that deviates markedly from other members of the population.

Performance Audit: the routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

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.

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.

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

Primary Review (AREV): The first level of data review conducted after data has been generated and uploaded to LIMS. Primary review is typically conducted by the analyst who generated the data but may be performed by another authorized individual. Quality control and corrective actions are evaluated as part of this review. Where acceptance criteria fails, samples are scheduled for re-preparation and/or re-analysis or data is appropriately qualified.

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Testing Study Provider: Any person, private party, or government entity that meets stringent criteria to produce and distribute TNI PT samples, evaluate study results against published performance criteria and report the results to the laboratories, primary accrediting authorities, PTOB/PTPA, and TNI.

Proficiency Test Sample (PT): 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)

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Protocol: a detailed written procedure [SOP] for laboratory operation that must be strictly followed.

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.

Quality Control: 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 users.

Quality Manual [QAP]: a document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control.

Quantitation Limit [Limit of Quantitation, Practical Quantitation Limit]: level, concentration, or quantity of a target variable (i.e. target analyte) below which data is reported as estimated. The quantitation limit may or may not be statistically determined, or may be an estimate that is based upon analyst experience or judgment.

Quantity Sufficient (QS): Refers to the addition of appropriate diluent to the solution to achieve the final volume.

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 reconstructing and evaluating the report of the activity or study.

Reagent Blank (method reagent blank): a sample consisting only of Type I water and 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.

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so (EPA, etc.). The reference method is included on the client report.

Reporting Limit (RL): The lowest analyte level (concentration or mass) the laboratory will report as a detected result. ACZ's default reporting limit is the MDL; however the RL may be defined as the PQL or another level dependent on project needs.

Retention Time: The time between sample injection and the appearance of a solute peak at the detector. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Sample: Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis

Sample Tracking: procedures employed to record the possession of the samples from the time of sampling until analysis, reporting, and archiving. These procedures include the use of a Chain of Custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.

Secondary Review (SREV): The second level of data review conducted after primary review (AREV) has been completed. Secondary review is typically conducted by the pertinent department supervisor but may be performed by another authorized individual. Quality control and corrective actions are evaluated as part of this review. Data qualifiers and sample statuses assigned at AREV are evaluated and corrected if necessary.

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

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Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (i.e. concentrations) of a variable of interest.

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there is no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.

Should: denotes a guideline of recommendation whenever noncompliance with the specification is permissible.

Signal to Noise Ratio (S/N): a dimensionless measure of the relative strength of an analytical signal (S) to the average strength of the background instrumental noise (N) for a particular sample.

Spike: a known amount of target analyte added to a blank sample or client sub-sample; used to determine the recovery efficiency or for other quality control purposes.

Standard Deviation: the measure of the degree of agreement (precision) among replicate analyses of a sample. The population standard deviation (n degrees of freedom) should only be used for more than 25 data points; otherwise, when referenced, standard deviation implies sample standard deviation (n-1 degrees of freedom).

Standard Operating Procedure (SOP): a written document which details the manner in which an operation, analysis, or action is performed. The techniques and procedures are thoroughly prescribed in the SOP and are the accepted process for performing certain routine or repetitive tasks.

Supervisor [however named]: the individual designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training, and experience to perform the required analyses.

Surrogate (SURR): 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.

Test Method: adoptions of a scientific technique for a specific measurement problem, as documented in a laboratory SOP or published by a recognized authority.

The NELAC Institute (TNI): a voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories.

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.

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

Validation: The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification: Confirmation by examination and provision of evidence that specified requirements have been met.

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APPENDIX E TECHNICAL DIRECTORS

Name	Department	Degree
Christopher Wakefield	Organics (reserve)	BS, Chemistry
Isaac Klaus	Organics	BS, Biological Sciences, Minor in Chemistry
Steve Pulford	Metals	BS, Chemical Engineering, Minor in Biochemistry
Billy Grimes	Wet Chemistry Instrument (reserve) Metals (reserve) Wet Chemistry Manual (reserve)	BA, Biology
Alyssa Dybala	Wet Chemistry Manual	BS, Pharmaceutical Marketing
Matt Sowards	Radiochemistry (reserve)	BA, Neuroscience
Jennifer Fox	Wet Chemistry Instrument	BA, Environment Studies, Minor in Mathematics
Brett Dalke	Geochemistry	BA, Geology & English
Mark McNeal	Radiochemistry	BS, Biology
If a technical director is absent for a period exceeding fifteen calendar days, another qualified full time employee shall be assigned to temporarily fulfill the duties of technical director. Defined reserve technical directors shall assume these duties by default. Where reserves are not yet defined, management shall appoint a qualified individual as necessary. If a technical director is absent for more than 34 days, it is the QAO's (or delegee's) responsibility to notify accrediting bodies in writing.		

Anatek Labs, Inc.

Quality Assurance Plan

1282 Alturas Drive
Moscow, ID 83843
(208) 883-2839

504 E Sprague Ave. #D
Spokane, WA 99202
(509) 838-3999

Revision No. 11

Effective Date: 10 Aug 2015

Approvals:

Mike Pearson
Lab Director
Date: 8/3/2015

Kathy Sattler
Lab Manager, Spokane
Date: 8-4-15

John Coddington
Lab Manager, Moscow
Date: 7/27/2015

Gene Solomon
QA Officer
Date: 7/23/2015

Todd Taruscio
Technical Director, Moscow
Date: 7/27/15

Erin Linskey
Technical Director, Moscow
Date: 7-27-15

Melissa Lewis
QA Officer/Technical Director, Spokane
Date: 8-4-15

Signature Page (1)

Anatek Labs, Inc.

The undersigned have read this Quality Assurance Plan and understand the requirements set forth in it. By signing this document, these individuals acknowledge their responsibility to follow the procedures outlined. The annual review of this manual is mandatory for all employees and will be construed as training for all matters contained within.

Mike Pearson Lab Director		Initials: MTP	Date: 3 AUG 2015
Gene Solomon QA Officer		Initials: GS	Date: 7/23/2015
John Coddington Lab Manager, Moscow		Initials: JC	Date: 7/27/2015
Kathy Sattler Lab Manager, Spokane	Kathy Sattler	Initials: KAS	Date: 8-4-15
Melissa Lewis QA Officer, Spokane	Melissa Lewis	Initials: ML	Date: 8-4-15

Name: (Please Print)	Signature:	Initials:	Date:
Todd Tarusco		TT	7/27/15
Erin T Linskey		ER	7-27-15
Connor Williams		CW	8-5-15
Mark Havrilla		MH	8-5-15
Kristopher Grows		KG	8/5/15
Brandon McGoogan		BMM	8/5/15
Mark Ritari		MER	8/5/15
Helen Westbrook		HW	8/5/15
Kelly Cavanaugh		KAC	8/5/15
John M. Ingston		JI	8/5/2015
Terrill W. Settles		TWS	8/5/2015
Justin Doty		JD	8-5-15

Signature Page (2)

Anatek Labs, Inc.

The undersigned have read this Quality Assurance Plan and understand the requirements set forth in it. By signing this document, these individuals acknowledge their responsibility to follow the procedures outlined. The annual review of this manual is mandatory for all employees and will be construed as training for all matters contained within.

Name: (Please Print)	Signature:	Initials:	Date:
Stuart Tolman	Stuart Tolman	ST	8/5/15
Travis Keane	Travis Keane	TK	8/5/15
KScott	KScott	KTS	8-11-15
Katie Boger	Katie Boger	KEB	8/11/15
Jessica Oakland	Jessica Oakland	JO	8/11/15
Bonnie Thompson	Bonnie Thompson	BT	8/11/15
Rebecca Watson	Rebecca Watson	RAW	8-11-15
Andrew Mendez	Andrew Mendez	AM	8-11-15
Wendy Ozminkowski	Wendy Ozminkowski	WO	8-11-15
John Beattie	Beattie	JB	8-12-15
Justin Koch	Justin Koch	JK	8/13/15
Cheri L. Price	Cheri L. Price	CP	08/14/2015

Table of Contents

Introduction **7**
Quality Assurance Policy Statement **7**
Confidentiality Policy Statement **7**
Code of Ethics/Conduct **7**
Data Integrity, Fraud Prevention & Detection **8**
Policy on Waste, Fraud and Abuse **8**
Customer Service **8**
New Work Requests and Contracts 8
Quality Assurance Program Management and Implementation **8**
QA Committee 8
QA Officer 9
Laboratory Organization, Responsibilities and Personnel Qualifications **9**
Laboratory Director 9
Laboratory Manager 9
QA Officer 10
Inorganic Supervisor 10
Microbiology Supervisor 11
Organic Supervisor 11
Radiation Safety Officer 11
Technical Staff (Chemists, Lab Technicians, etc.) 11
Staff 12
Sample Procedures **13**
(Sample Collection, Storage, Handling and Acceptability) **13**
Collection 13
Sample Containers 13
Preservation Methods 13
Transportation 13
Hand Delivery 13
Shipped Samples 14
Sample Acceptability 14
Sample Logging and Tracking **14**
Logging 15
Tracking 15
Sample Custody and Legal Defensibility **15**
Collection 15
Analytical Procedures **16**
Data Generation – Data Reduction, Validation and Reporting **16**
Data Reduction 16
Verification / Validation 16
Timely Reporting 16
Reporting Results 16
Uncertainty of Measurement 17
Notification of MCL Violations 17
Internal Quality Control **17**
Precision 17
Accuracy 18
Completeness 18

Analytical Batch 18
Method Blank (a.k.a. Laboratory Reagent Blank) 19
Calibration Blanks 19
Continuing Calibration Blanks 19
Calibration Standards 19
Initial (or Independent) Calibration Verification Standards 19
Continuing Calibration Verification Standards (CCV) 19
Internal Standards 19
Surrogates 20
Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix) 20
Laboratory Duplicates and Duplicate Matrix Spikes 20
Laboratory Control Samples (a.k.a. Laboratory Fortified Blanks or Quality Control Samples) 20
Interference Check Samples 21
Post Digestion Spikes 21
Source and Preparation of Standard Reference Materials 21
Practical Quantitation Limits (PQL) 21
Method Detection Limits (MDL) 22
Control Charting 22
Quality Document Control 22
Standard Operating Procedures (SOPs) and Laboratory Notebooks 22
Deviation from Standard Operating Procedures 23
Modified Procedures 23
Policy on Manual Integration 23
Integration Procedure and Review 23
System and Performance Audits 24
Management System Reviews (MSRs) 24
Technical System Audits (TSAs) 24
Performance Evaluation (PE) 25
Data Quality Audits (DQAs) 25
IT Systems Auditing 25
Corrective Action 25
Preventative Action 25
Training & Personnel Qualifications 26
Demonstration of Capability (DOC) 26
Facilities, Equipment and Services 26
Calibration Procedures and Frequency 26
Analytical Instrumentation 26
Preventive Maintenance 27
Temperature Record Keeping 27
Analytical Balances 27
Water Purification System 28
Glassware Washing 28
Services and Supplies 28
Waste Disposal 28
Subcontracting of Laboratory Services 28
Termination or Transfer of Business 28

Tables, Figures, and Appendices in separate document
(located at http://www.anateklabs.com/wp-content/uploads/QA-Plan-Docs/TablesFigsApp.pdf)
In lieu of page numbers, click the links in the PDF document

Tables .....

1a Analytical Equipment Utilized at Anatek Labs, Moscow .....
1b Analytical Equipment Utilized at Anatek, Labs, Spokane .....
2 Summary of Analytical Parameters, Method, Sample Containers, Preservation Methods, Holding Times and Estimated Working time .....

Figures .....

1 Anatek Labs Organizational Chart.....
2 Anatek Labs, Moscow Personnel.....
3 Anatek Labs, Moscow Floor Plan.....
4 Anatek Labs, Spokane Personnel.....
5 Anatek Labs, Spokane Floor Plan.....

Appendices .....

A Index of Standard Operating Procedures .....
B Example of Sample Submission Form.....
C Quick Reference for Chemical Safety .....
D Laboratory Staff Resumes / Qualifications.....
E Current State Certifications.....
F Backup, Fault Tolerance, Disaster Recovery and Data Archive of Mission-critical Information Storage and Services.....
G Control Chart Information .....
H Method Detection Limit.....
I Calibration Methods and Equations.....

Introduction

Anatek Labs, Inc. is a private, full service, multi-state certified analytical laboratory. We are committed to providing the highest quality environmental, agricultural, residential, and industrial testing services in a timely and cost-effective manner. We have established quality systems to ensure the quality and integrity of our work, and we are committed to enacting these quality measures and ensuring compliance with applicable TNI standards.

Anatek Labs, Inc. has integrated many Quality Assurance (QA) practices into its measurement activities. These QA practices are designed to generate high quality data in an efficient and cost effective manner. Anatek Labs, Inc. has a laboratory-wide Quality Assurance (QA) Program designed to assess and monitor the ongoing quality of the testing performed in its facilities. Its purpose is to identify and correct problems as they occur and, if possible, to determine in advance potential problem areas and institute measures for their resolution. The Quality Assurance Committee will oversee all QA activities to assure the accurate, reliable and prompt reporting of testing results. This document describes Anatek Labs, Inc.'s Quality Assurance Plan as it relates to operations within the laboratory. While this document strives to be inclusive, much of the Anatek Labs, Inc. quality plan is incorporated in the laboratory and method SOPs referenced in the Appendix.

This QA Plan addresses all the minimum required elements described in the Guidelines and Specifications for Preparing Quality Assurance Program Plans (QAMS-004 / 80), Interim Guidelines and Specifications For Preparing Quality Assurance Project Plans (QAMS-005 / 80) and Guidance on Preparation of Laboratory Quality Assurance Plans (EPA 910 / 9-92-0332).

Quality Assurance Policy Statement

It is the policy of Anatek Labs, Inc. that there shall be sufficient Quality Assurance activities conducted to ensure that all data generated and processed will be scientifically valid and of known and documented quality. In addition, the use of all aspects of this quality system will continually improve the effectiveness of the laboratory and the quality system. All data generated by Anatek Labs, Inc., unless acknowledged and authorized by the submitting party, will be of known precision and accuracy and legally defensible. Quality assurance activities are designed in the most cost-effective fashion possible without compromising data quality objectives. The laboratory staff adheres to the requirements and specifications stated in this Quality Assurance Plan. All data reported meets the applicable requirements for The NELAC Institute, EPA and/or any State specific methods used. For specific method requirements refer to SOPs, current EPA methods, the most current edition of Standard Methods, and/or state specific methods.

All employees must read, understand, and follow the provisions of this Quality Assurance Plan.

Confidentiality Policy Statement

All client information at Anatek Labs, Inc. is considered confidential. No information will be given out without the express verbal or written permission of the client. All reports generated will be held in the strictest of confidence and issued only to the client. The exceptions to this policy would be those mandated by law (e.g., positive *E. coli* in public water systems that are required to be reported to State Regulatory Agencies). All employees of Anatek Labs, Inc. will at all times adhere to this policy.

Code of Ethics/Conduct

The Anatek Organization is a team and each team member is expected to maintain a high level of professionalism. Each employee is responsible for his or her work, and that work must be conducted ethically, legally, and in accordance with standard operating procedures and applicable methods and regulations. Employees are expected to perform their duties with excellence, and to contribute to an environment where their co-workers can efficiently perform their duties and maintain focus on the overall benefit of the Anatek team, our customers, public health, and the environment. The penalties for violating the Code of Ethics can range from verbal reprimands to loss of position. No person at Anatek Labs, Inc. will in any way be put under undue pressure, financial or other, to complete their assigned tasks in violation of this code.

Data Integrity, Fraud Prevention & Detection

Anatek Labs, Inc. actively works to insure that the data it produces is of the highest quality and legally defensible. The data integrity system includes data integrity training for all new employees and annual refresher training for all employees, signed data integrity documentation, in-depth monitoring and review of data, and proficiency testing samples. At a minimum, 10% of all data packets generated are reviewed by the QAU or laboratory supervisors. If discrepancies are found management is notified. Blind samples are prepared as needed to check for fraud. If an employee is found to have committed a fraudulent act they will be dismissed.

Investigations resulting from data integrity issues will be conducted in a confidential manner until they are completed.

Policy on Waste, Fraud and Abuse

Under no circumstances is the willful act or fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to management for appropriate corrective action. Reported acts will be assessed on an individual basis, resulting actions will be consistent with Anatek Labs, Inc. policies and could result in dismissal.

Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented and appropriate remedies and punitive measures will be taken toward those individuals responsible.

Customer Service

Anatek Labs, Inc. reputation has been built upon service to the customer. The laboratory is always willing to communicate and cooperate with customers to ensure their requirements are met, provided confidentiality to other customers can be ensured. Feedback from customers, including complaints, is used to improve laboratory operations and services.

New Work Requests and Contracts

It is the policy of Anatek Labs, Inc. to never turn away customer requests. The lab is constantly expanding its abilities by seeking new certifications, purchasing new equipment, and hiring quality personnel. New work requests are reviewed to ensure that the lab has the appropriate facilities, resources, and expertise to perform the analyses required.

Most of the work contracted to Anatek Labs, Inc. is for regulated projects analyzed by accredited methods. Specialty projects are reviewed and established by the Lab Director, Lab Manager, and/or Technical Directors, and the technical, legal, and financial considerations are established via correspondence and contracts, and reviewed as necessary. Project–related documentation (e-mail, contracts, quotes, etc.) is maintained (and reviewed as necessary) by the Lab Manager and/or Lab Director.

If Anatek Labs is unable to perform a particular analysis we will find a certified lab to subcontract the job. The above policy is at the discretion of the Lab Director and is subject to change.

Quality Assurance Program Management and Implementation

Overall responsibility for quality assurance lies with the Laboratory Director. The primary QA management of the laboratory rests with the Laboratory Manager. To provide technical assistance to the Laboratory Manager, a QA Officer is appointed by the Laboratory Director. The QA Officer is granted sufficient resources to ensure the proper execution of the QA Plan and to recommend and implement specific QA policies and procedures. A Quality Assurance Committee exists to further facilitate adherence and development of QA policies and procedures.

QA Committee

The Quality Assurance Committee is comprised of the QA Officer, Laboratory Manager, Laboratory Director, and Laboratory Supervisors. The QA Committee is responsible for overseeing lab-wide QA Plan implementation and addressing QA complaints or concerns brought to its attention from internal or external sources. The QA Committee directs the efforts of the QA Officer and enforces any necessary corrective actions. The QA Committee shall convene if needed to assess and/or address lab-wide QA concerns and any actions taken by the QA Officer. Any QA complaints or problems that are received from outside the laboratory will be referred to the committee in order to resolve such issues.

QA Officer

The QA Officer is appointed by the Laboratory Director to oversee specific QA policy and procedure development, implementation, and adherence of Anatek Labs, Inc. The QA Officer is responsible for auditing internal operations and ensuring compliance with QA criteria established by this QA Plan and other documented policies and procedures. The QA Officer assesses all QA systems on an annual basis. Results of all findings are documented and corrective action recommendations, if any, are submitted to the QA Committee, Laboratory Manager and affected staff members.

The QA Officer is responsible for documentation and evaluation of specific policies and procedures. Standard Operating Procedures are kept on file documenting specific procedures employed to ensure the validity and acceptability of data generated at Anatek Labs. Materials purchased for Quality Control purposes are received with a Certificate of Authenticity from the manufacturer. Certificates are kept on file for review if necessary. The QA Officer is responsible for coordinating and reporting for all performance evaluation samples, maintaining and updating certifications and accreditations, and monitoring corrective actions.

Laboratory Organization, Responsibilities and Personnel Qualifications

All employees maintain a copy of all training certificates and diplomas on file with certificates of capability for each method they perform. Job descriptions are maintained in employee training files. Organizational charts for Anatek Labs, Inc. are shown in the Appendices.

Laboratory Director

The Laboratory Director is responsible for overall technical direction and business leadership of Anatek Labs, Inc. The Laboratory Director is responsible for the implementation and evolution of QA policy and SOPs within the laboratory, based on the current market, technological advances in equipment, and methods.

The Laboratory Director is responsible for assuring that the provisions of this QA Plan are met, and that adequate resources are available for technical operations and quality systems oversight.

The Laboratory Director is a direct liaison to the Corporation's Board of Directors (BOD) and must attend a BOD meeting at least once a year, or as necessary to discuss equipment purchases, managerial changes, contracts, and major SOP and QA changes.

All Laboratory Managers, Technical Directors, and Systems Managers report directly to the Laboratory Director.

The Laboratory Director is required to attend managerial and/or staff meetings at which the topic of QA is discussed. These meetings will be held as often as necessary or at the direction of the Laboratory Director.

The Laboratory Director may serve as Laboratory Manager, Technical Director, Systems Manager, and/or Analyst if these positions are not filled for any reason.

The Laboratory Director must have a minimum BS in a science or engineering field and 5 years of managerial experience in an environmental laboratory or an equivalent combination of education and experience.

Laboratory Manager

The Laboratory Manager is responsible for overseeing the daily operations of Anatek Labs, Inc. The Laboratory Manager, in conjunction with the Technical Director(s), is responsible for coordinating the activities within the laboratory with the overall goal of efficiently producing high quality data in a reasonable time. The Laboratory

Manager is responsible for monitoring the validity of the analyses performed and data generated and for monitoring standards of performance in quality control and quality assurance.

The Laboratory Manager reports directly to the Laboratory Director and may act as Interim Director during extended absence of the Laboratory Director.

Additionally, the Laboratory Manager will provide technical support to customers and coordinate project to meet specific customer needs.

The Laboratory Manager is responsible for the maintenance of standards and materials in accordance with the QA Plan, to ensure uninterrupted operation of the laboratory.

All Section Managers and Analysts not reporting to the Technical Director(s) report directly to the Laboratory Manager.

In events where employee scheduling or current workload is such that new work cannot be incorporated without missing holding times or data quality objectives, the Laboratory Manager has authority to refuse samples, modify employee scheduling, or re-schedule projects.

The Laboratory Manager, in coordination with the Section Managers, QC/QA Managers, and Technical Director, is responsible for determining in which QA Proficiency Programs the laboratory will participate, and which accreditations the laboratory will pursue. It is the responsibility of the Laboratory Manager to ensure that the laboratory sections perform the tasks necessary to complete the proficiency testing required to maintaining certification and accreditation

The Laboratory Manager will attend managerial and/or staff meetings at which the topic of QA is discussed.

The Laboratory Manager can act as Analyst, Section Manager, or Technical Director if, for any reason, the positions are not filled.

The Laboratory Manager is responsible for all human resource decisions within the laboratory except for employees reporting directly to the Technical Director or Laboratory Director.

If the Laboratory Manager is to be absent for more than 15 days, the Technical Director (or other supervisor) will serve as the temporary Laboratory Manager.

The Laboratory Manager must have a minimum BS in a science or engineering field and 5 years of managerial experience in an accredited environmental laboratory, or an equivalent combination of education and experience.

QA Officer

The QA Officer is a member of Anatek Labs, Inc. QA Committee and is designated by the Laboratory Director. The QA Officer serves as the focal point for QA/QC and is responsible for the oversight and/or review of quality control data. The QA Officer functions independently from laboratory operations (answering directly to the Lab Director), and is able to evaluate data objectively without managerial influence. The QA Officer is responsible for conducting internal audits, and for notifying management of any deficiencies in the quality system, and for monitoring corrective actions. The QA Officer is responsible for maintaining the currency of this QA Plan, and will help develop, implement, and maintain Standard Operating Procedures appropriate to the procedures employed within Anatek Labs, Inc. The QA Officer is responsible for ensuring all applicable regulatory agency requirements are met.

The QA Officer must have a minimum BS in a science or engineering field and two years experience in QA/QC or an equivalent combination of education and experience.

Inorganic Supervisor

The Inorganic Supervisor is responsible for training, overseeing and assisting inorganic technical staff in their daily duties. The Inorganic Supervisor assures adherence to Standard Operating Procedures and Quality Assurance activities. The Inorganic Supervisor should be a Chemist II or III.

The Inorganic Supervisor must have a minimum BS in a science or engineering field and 2 years of laboratory experience, or an equivalent combination of education and experience.

Microbiology Supervisor

The Microbiology Supervisor is responsible for quality microbiological results by maintaining the laboratory microbiology program and services within the framework of the lab QA guidelines. He/She is responsible for maintaining certifications and training/supervising the microbiology technicians.

Microbiology Supervisor must have at least two years experience performing microbiological analysis in an environmental laboratory.

Organic Supervisor

The Organic Supervisor is responsible for overseeing and assisting organic chemistry technical staff in their daily duties. The Chemistry Supervisor assures adherence to Standard Operating Procedures and Quality Assurance activities. The Organic Supervisor should be a Chemist III.

The Chemistry Supervisor must have a minimum BS in a science or engineering field and 2 years of Laboratory experience, or an equivalent combination of education and experience.

Radiation Safety Officer

The Radiation Safety Officer is responsible for overseeing and assisting radiochemistry technical staff in their daily duties, and for radiation safety training for employees. The Radiation Safety Officer assures adherence to NRC, EPA, and NELAC regulations relating to radionuclides, as well as to the Anatek Radiation Safety Plan and applicable SOPs.

The Radiation Safety Officer Chemistry Supervisor must have a minimum BS in a science or engineering field, 2 years of radionuclide experience, or an equivalent combination of education and experience. In addition, the RSO must have completed Radiation Safety Officer training.

Technical Staff (Chemists, Lab Technicians, etc.)

Technical Director

The company Technical Directors are responsible for providing scientific leadership and vision. One or more Technical Directors will manage and coordinate activities of laboratory departments as designated by the Laboratory Director. The Technical Directors will work with the Laboratory Manager to ensure that all lab QA/QC practices are being satisfied in order to produce data that exceeds the quality objectives of the QA Plan, specific customers or projects, and regulatory requirements.

The Technical Directors will provide technical support to laboratory staff and investigate new areas of interest to the company by utilizing methods development and technical advancements. The Technical Directors will be available as needed to fill in for Chemist(s) in the event of absence or job opening.

The Technical Directors will be the primary contact for specified customers and provide project management and advice to those customers. Additionally, the Technical Directors will prepare final customer reports as needed.

The Technical Directors will be responsible for non-routine instrument maintenance and troubleshooting and, if needed, obtain outside technical assistance for equipment maintenance or repairs as necessary.

The Technical Directors must have at least 5 years of applicable laboratory experience and a minimum of a BS in a science or engineering field.

Chemist (I, II, III)

The Chemist is responsible for the analysis of samples and the generation of high quality data in accordance with the laboratory SOPs and QA/QC guidelines in a reasonable time as prescribed by laboratory standard turnaround schedules or as directed by the Laboratory Director or Laboratory Manager.

The Chemist is responsible for making sure all data generated by him/her is entered into the appropriate database in the correct manner and that raw data packs are signed and archived properly.

The Chemist reports daily to the Laboratory Manager and will inform the Lab Manager as to the material needs of the laboratory, specifically pertaining to analyses performed by the Chemist.

Additional duties of the Chemist may include, but not be limited to, preparation of samples for analysis, maintenance of lab equipment, and providing technical assistance to lower-level laboratory staff. The Senior Chemist in the laboratory may be asked to perform supervisory duties as related to operational aspects of the laboratory. In the event that this is required for any reason, these supervisory duties will be assigned by the Lab Manager and/or Lab Director. The Chemist may perform all of the duties of Laboratory Technician.

The position of Chemist is a full time or part time hourly position and may be divided into three levels, Chemist I, II, and III. Chemist I must have the equivalent of a Bachelors degree in Chemistry or a closely related science. Additionally, Chemist II must have at least 2 years of environmental or closely related lab experience. Chemist III must have a Bachelors degree plus 5 years of environmental or closely related lab experience.

Laboratory Technician/Microbiology Technician

The Laboratory Technician (chemistry or microbiology) is responsible for providing support in the form of sample analyses, sample preparation, and general lab maintenance. This may include tasks such as filling out daily maintenance logs, chemical inventories, and laboratory cleaning (glassware, etc).

The Laboratory Technician reports to the Laboratory Manager or the Microbiology Supervisor in the case of Microbiology Technician.

The Laboratory Technician may be divided into three levels, Technician I, II and III. Lab Technician I must have a high school diploma or GED. Lab Technician II must have at least 1 year of experience in an environmental lab or equivalent secondary education. Lab Technician III must have a bachelor's degree in chemistry or closely related science or equivalent work experience.

Staff

Systems Administrator

The Systems Administrator is responsible for overseeing all information systems infrastructure. Infrastructure is defined as all hardware including computer workstations, servers and IT support equipment plus all laboratory equipment that interfaces with the network or database as well as all software and applications resident on the system.

The Systems Administrator reports directly to the Laboratory Director. The Systems Administrator must have a minimum BS in a computer science or information technology field or an equivalent combination of education and experience.

Customer Service Manager

The Customer Service Manager is responsible for all phases of customer service including but not limited to answering telephones, assisting customers with Anatek Labs forms, distributing sample kits, and receiving samples from walk-in customers.

The Customer Service Manager must have a minimum AA in Business Administration, Accounting or other relevant field and 5 years experience in office administration/supervision or an equivalent amount of education and experience.

Bookkeeper

The Bookkeeper is responsible for: invoicing customers, processing payments, invoices and packing slips, paying A/P invoices, payroll, payroll taxes, federal and state taxes, issuance of purchase orders, and deposits.

The Bookkeeper is responsible for all Human Resource management including insurance and 401K.

The Bookkeeper must have a minimum AA in Business Administration, Accounting or other relevant field and 1 year experience in business accounting or an equivalent amount of education and experience.

Sample Custodian, Shipping/Receiving

The Sample Custodian is responsible for the log-in and tracking of all samples throughout the laboratory. The Sample Custodian is additionally responsible for tracking of all samples sent to sub labs. All shipping and receiving is done/monitored by the sample custodian, to include sampling kits, trip blanks and prepreserved sample battles. The Sample Custodian takes customer orders and insures that incoming samples are correct. If a discrepancy is noted the Sample Custodian will contact the customer to resolve any questions or problems. The Sample Custodian is an integral part of the customer service team.

The Sample Custodian must have a high school diploma or equivalent.

Sample Procedures
(Sample Collection, Storage, Handling and Acceptability)

All samples sent to Anatek Labs, Inc. are received, logged in and distributed by the Sample Custodian. Samples that are unsatisfactory are not analyzed unless authorized by the customer. Any such sample will be noted on the Chain of Custody form and with a qualifying statement on the final report noting unsatisfactory sample submission. Corrective measures to ensure proper specimen collection and/or handling on future sample sets will be supplied to the customer(s).

Collection

Samples must adhere to requirements for container, preservation and holding times described on the Anatek Labs, Inc. website (www.anateklabs.com). Consult the Standard Operating Procedure, EPA SW-846 Manual on Test Methods for Evaluating Solid Waste, the Federal Register on EPA Test Methods Determining Contaminants in municipal and industrial wastes, Standard Methods or other appropriate documentation for specific instructions on sample collection.

Sample Containers

Most sampling containers are supplied to the customer by the laboratory. Containers are generally used only once and discarded. Some analytical methods utilize containers of a type that is conducive to recycling. In these cases, containers are cleaned according to Standard Operating Procedures to ensure cleanliness. Samples must not be exposed to interfering materials. Consult the laboratory for the proper container material and size for a specific analysis or project. If the samples are collected and stored for transport in inappropriate types of containers, the Laboratory may not be able to accurately quantify the amount of the desired components. In this case resampling may be required.

Preservation Methods

All samples should be preserved according to the type of matrix, analysis required and data objectives. If the samples are not properly preserved the analytical results may be inaccurate due to loss by volatilization and/or degradation. Anatek Labs, Inc. provides sample containers with appropriate preservatives already in the container, when possible. Table 2 contains information on appropriate preservation methods.

Transportation

Samples should be transported to the laboratory by the fastest means possible. In general, samples should be chilled.

Hand Delivery

Personal delivery is preferred, as it is the most secure. A Chain-of-Custody record must accompany the transfer of the sample with the identification information if results will potentially be used as evidentiary. The field sampler is responsible for the proper packaging and dispatch of their samples. This responsibility includes sample preservation and the completion of all necessary documents concerning custody.

Shipped Samples

A sealed container should be used to ship samples via a common carrier. Samples within these containers should also be properly sealed, identified and accompanied by appropriate paperwork such as a Chain-of-Custody record or a test request form. The Laboratory will retain all bills of lading for use to supplement the accompanying Chain-of-Custody documentation.

Sample Acceptability

Samples received after holding times have expired, in inappropriate containers, or lacking appropriate preservative measures are generally not accepted for testing. Occasionally, a customer will request that a sample be processed even if it is received in an unacceptable condition. In such a case, testing will only proceed after the customer has provided written or verbal acknowledgement of the unacceptable status of the sample and authorized continued testing. Further, a comment, narrative, or explanation of possible negative effects of unacceptable sample submission is placed on the report or attached as a more detailed description.

Sample Logging and Tracking

Standard Operating Procedures have been established for the receiving of samples into the laboratory (SOPs ALI-02 & ALI-18). These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received. Documentation of all sample storage is maintained in order to preserve the integrity of the samples.

Samples delivered to the lab are received by a designated Sample Custodian(s). Verification of sample integrity by the Sample Custodian includes the following activities:

- Assessment of custody seal presence/absence, location and signature
- Temperature of sample containers upon receipt
- Chain-of-Custody documents properly completed (entries in ink, signature present, etc.)
- Sample containers checked for integrity (broken, leaking, etc.)
- Sample is clearly marked and dated (bottle labels complete with required information)
- Appropriate containers (size, type) are received for the requested analyses
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.)
- Assessment of proper sample preservation (if inadequate, corrective action is employed)
- VOC containers are inspected for the presence/absence of headspace bubbles (No assessment of proper preservation is performed for VOC containers at time of receipt; preservation is checked after analysis to avoid loss of sample)

Any anomalies or discrepancies observed during the initial assessment are recorded on the chain of custody documents and/or in the LIMS sample tracking software. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When a satisfactory resolution has been reached by coordination with the client, the log-in process may commence and analysis may begin. Any changes on documentation resulting from these discussions are documented and authorized directly by the customer. During the log-in process, each sample is given a unique laboratory code and an analysis request form is generated. The analysis request contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates and analysis due dates and other pertinent information.

Facility security and access is important in maintaining the integrity of samples received at Anatek Labs, Inc. Access to the laboratory is limited to authorized personnel except for the sample receipt area that is manned during business hours.

Samples are stored appropriate to the analysis requested until they undergo analysis. Anatek Labs, Inc. stores samples in one of many refrigerators, freezers or other storage locations, depending on the type of analysis and the matrix of the sample. Anatek Labs, Inc. has several refrigerators for storage of samples. These refrigerators are segregated by matrix type (soil or water) and method of analysis. Drinking water, wastewater and soil samples are segregated and placed in separate refrigerators. The samples are further separated into dedicated refrigerated storage of VOC samples. A walk-in refrigerator is used for sample archival. An additional freezer provides additional frozen storage capacity for miscellaneous samples. The temperature of each sample storage unit used at Anatek Labs, Inc. is monitored daily during operations and the data recorded in a file for future reference.

Samples and sample extracts are retained for up to six weeks then disposed of unless other arrangements have been made in advance. All samples are either returned to the client or disposed of according to approved disposal practices.

Logging

Samples are assigned a unique laboratory identification number. All samples are assigned a number with the following format: YYMMDDZZZ-XXX
Where:

YY = year (16=2016)
MM = month (02=February)
DD = day of the month
ZZZ = sample batch (for that log-in day)
XXX = sample number

For example, 160107001-001 would be the first sample of the first batch of samples received on January 7, 2016.

Tracking

Samples are tracked by their individual log-in numbers. As testing is completed the LIMS is updated and the data archived.

Sample Custody and Legal Defensibility

Anatek Labs, Inc. routinely tests samples used as legal evidence. A primary consideration for the legal credibility of analytical data is the ability to demonstrate that samples were obtained, reached the laboratory and analyzed without improper alteration or contamination. In most instances, Chain-of-Custody forms function only as a sample receipt form and initiate normal, standard sample handling procedures. Samples whose testing results may become evidentiary utilize Chain-of-Custody protocol where evidence of sample collection, shipment, laboratory receipt and laboratory custody until disposal are documented. Chain-of-Custody forms document how physical custody of a particular sample is maintained, how custody is transferred and the identity of individuals responsible for sample collection, shipping, receipt, analysis, storage and disposal. Formal Chain-of-Custody protocol is not automatically initiated due to the accompaniment of a Chain-of-Custody form. Formal Chain-of-Custody protocol must be specifically requested by the sample submitter.

The Sample Custodian is responsible for receiving Chain-of-Custody linked samples. Upon receipt of these samples, the Sample Custodian immediately inspects the documentation and the samples to ensure the integrity of the sample shipping container, sample bottles, custody seals and cooler temperature upon receipt. Samples received in broken or leaking containers are noted on the Chain-of-Custody form and specific instructions for the lab are then requested of the submitter. If discrepancies between accompanying documentation and information on labels or sample containers exist, clarification is requested from the submitting party in writing and a notation is placed on the Chain-of-Custody form explaining the discrepancy.

After receipt in the laboratory, samples are logged into the internal tracking system. Samples are stored in appropriate refrigerators according to matrix until analyzed. After analysis samples are stored for up to six weeks in designated areas in the walk-in refrigerator.

Collection

All samples should be collected using standard field sampling techniques. The sample container should be labeled with the following information:

1. Date and time of collection
2. Source of sample
3. Preservative used (if any)
4. Name of person collecting sample
5. Sample ID and project name

When appropriate, the container should be sealed so that it cannot be opened without disrupting the seal. Gummed tape or another type of sealant is recommended. The person collecting the sample should date and initial the seal, particularly across the junction of the tape to ensure a tamper-proof seal.

Pertinent data concerning each sample should be entered into a field log book. This information may be used to refresh the memory in the event that the collector is summoned as a witness.

The sample should be kept in the custody of the collector or a designated custodian. A sample is in a person's custody if:

1. It is in one's physical possession, or
2. It is in one's view after being in one's physical possession, or
3. It has been placed into a locked area to which the custodian retains the key.

Analytical Procedures

Analytical Standard Operating Procedures are based upon methods appearing in a variety of publications. Most commonly, procedures are adopted from EPA publications, "Methods and Guidance for Analysis of Water," "Test Methods for Evaluating Solid Waste: SW-846,", or "Standard Methods for the Examination of Water and Wastewater" online edition. Refer to the Appendices for a listing of the test procedures utilized at Anatek Labs, Inc.

Data Generation – Data Reduction, Validation and Reporting

Data Reduction

Test results are calculated manually and electronically as specified in the method-specific SOP and SOP ALI-05. Formulae are contained in the manual testing procedures and algorithms are contained in software controlled procedures. All data and calculations are verified by the analyst and posted to summary reports or the computer system for review by the Laboratory Manager.

Verification / Validation

Some procedures utilize additional visual confirmation and validation of values obtained electronically in the form of strip charts or other printouts. Where possible verification is made using interrelated analytes, i.e., the concentration of one analyte theoretically cannot exceed the concentration of another. Validation in gas chromatography is accomplished through the use of two dissimilar columns or the use of one or more compound specific detectors.

Data quality indicators such as blank results, duplicate reproducibility (precision), matrix spike and quality control sample recoveries (accuracy), and known sample or project histories are checked to verify result validity. Refer to the individual method SOPs for acceptability criteria.

Timely Reporting

Samples are typically tested consecutively as received unless holding times or special arrangements require expedited testing schedules. All testing is scheduled so that accepted holding times can be met.

Reporting Results

The analyst generating the test results posts them to manual worksheets, the computer tracking system, and/or summary reports depending upon the analysis. A final report is generated after all testing for a particular sample is completed and the Laboratory Manager reviews, signs, and dates the reports for distribution to the customer and any regulatory agency requiring copies.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with an appropriate data qualifier. Failure to meet established analytical controls prompts corrective action. Corrective action may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the Laboratory Manager, area supervisors, or the QA Officer may examine and pursue alternative solutions. Resumption of work subsequent to extensive corrective action (i.e., outside the scope of the method or SOP) shall be determined by the Lab Manager or area supervisor. In addition, an assessment will be made in order to ascertain if contact with the client is necessary.

Uncertainty of Measurement

Anatek Labs attempts, when possible, to identify all the components of measurement uncertainty, and estimate uncertainty of measurements. For most analyses, this is accomplished by following well-recognized and established test methods, and meeting method and SOP-specified quality control measures (blanks, CCVs, matrix spikes, etc.) Successful analysis of quality control samples helps to establish the certainty of an analytical measurement. To reduce the uncertainty of measurement, results are generally not reported below the lower limit of quantitation or above the upper limit of quantitation. If results outside these ranges must be reported, qualifiers, flags, or explanations are used to identify the increased quantitative uncertainty.

Radionuclide analysis incorporates a measure of uncertainty into reported results. The uncertainty of a measurement is a factor of background, counting times, and instrument considerations, and is calculated according to equations in the analytical method, and reported alongside the analytical results.

Notification of MCL Violations

If analysis of a drinking water sample indicates nitrate, coliform or E. coli results in excess of Maximum Contaminant Levels (MCLs), the client and the appropriate regulatory agency or agencies shall be notified within 24 hours of the validation of the analytical run. For other results exceeding the MCL, notification shall take place within 48 hours or two business days after validation of the sample result. This notification may be by phone, fax, or e-mail, depending upon the requirements of the regulatory agency.

Internal Quality Control

An Internal Quality Control program has been designed to ensure systematic in-house production of high quality analytical data. The objectives of this program are:

1. To provide a measure of the precision of analytical methods;
2. To maintain a continuing assessment of the accuracy, precision and completeness of individual analyses performed in the laboratory;
3. To identify methods that are weak and provide a source of research problems to overcome these deficiencies and weaknesses;
4. To detect training needs within the analytical group;
5. To provide a permanent record of instrument performance as a basis for validating data and projecting repair and replacement needs;
6. To upgrade the overall quality of laboratory performance.

Precision

Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability or random error in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: ***repeatability*** – the random error associated with

measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions and, ***reproducibility*** - the random error associated with measurements made by different test operators in different laboratories, using the same method but different equipment to analyze identical samples of test material. At Anatek Labs, Inc. our "within batch" precision is measured through the use of replicate samples of QC analyses and is expressed as the relative percent difference (RPD) between replicate measurements. The "Batch to Batch" precision is calculated from the variance observed in results from analysis of standard solutions of laboratory control samples from multiple analytical batches.

Accuracy

Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of standard reference materials, standard solutions, and laboratory-fortified blanks. In addition, laboratory-fortified (matrix spike) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (%Rec.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination). Anatek Labs, Inc. utilizes several quality control samples and independent calibration verification standards. Because bias can be positive or negative and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement.

Completeness

Completeness is a measure of the amount of valid data that is obtained, compared to the amount that is expected. For the purposes of this plan, completeness is calculated by dividing the number of samples having valid data by the total number of samples in the project, expressed as a percentage. Anatek Labs, Inc.'s objective for completeness is 100%.

The specific types, frequencies and processes for quality control sample analysis are described in detail in method-specific standard operating procedures. These sample types and frequencies are described below. In addition, a number of other quality control processes that may impact analytical results are also described below.

Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that Anatek Labs, Inc. has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples, are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner.

The minimum requirements of an analytical batch are:

1. The number of (field) samples in a batch is not to exceed that specified in the Standard Operating Procedure for the procedure being employed (typically 20).
2. All (field) samples in a batch are typically of the same matrix.
3. The QC samples to be processed with the (field) samples typically include:
    a. Method Blank (aka Laboratory Reagent Blank)
       Function: Determination of laboratory contamination
    b. Laboratory Control Sample (aka Laboratory Fortified Blank)
       Function: Assessment of method performance
    c. Matrix Spiked (field) Sample-when sufficient sample is supplied (aka Laboratory Fortified Sample Matrix)
       Function: Assessment of matrix problems
    d. Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample – when sufficient sample is supplied (aka Laboratory Duplicate)
       Function: Assessment of batch precision
4. A single lot of reagents is used to process the batch of samples.
5. Each operation within the analysis is performed by a single analyst/technician/chemist or by a team of analysts/technicians/chemists.

6. (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic compounds, it begins when the samples are extracted.
7. The QC samples are to be analyzed in conjunction with the associated field samples prepared with them.
8. Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.
9. Specific project, program or method SOP requirements may be exceptions to these definitions. If project, program or method SOP requirements are more stringent than these laboratory minimum requirements, then the project, program or method SOP requirements will take precedence.

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

The method blank is either analyte-free water or analyte-free soil (when available), subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand or an acceptable substitute may be used instead. The method blank is analyzed to demonstrate that the analytical system itself is not contaminated with the analyte(s) being measured. The method blank results should be below the Method Reporting Limit (MRL) or, if required, less than the Method Detection Limit (MDL) for the analyte(s) being tested, otherwise, corrective action must be taken. At least one method blank is included with the analysis of every analytical batch as stated in the method Standard Operating Procedure.

Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve.

Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of analyte-free water, reagent, or solvents that are analyzed in order to verify the system is contamination-free when continuing calibration standards are analyzed. The frequency of CCB analysis is either once every ten (10) samples or as indicated by the method, whichever is greater.

Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard solutions that are, in turn, prepared from stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used. Refer to Anatek Labs, Inc. SOP ALI-08 for policies regarding standards.

Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are second-source standards that are analyzed after a calibration and prior to sample analysis, to verify the calibration curve. ICVs should be run every time a new calibration is prepared. The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards. ICVs are also analyzed in accordance with method-specific requirements.

Continuing Calibration Verification Standards (CCV)

Continuing Calibration Verification Standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. Many modern method revisions require low-level (reporting level) CCVs to verify method performance at the reporting limit. The frequency of CCV analysis is indicated in the reference method and the Standard Operating Procedure.

Internal Standards

Internal Standards consist of known amounts of specific compounds that are added to each sample following sample preparation or extraction. Internal standards are generally used for procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by certain matrix effects. The integrated area

of the internal standard compared to the initial calibration average or most recent CCV should vary by no more than the limits specified in each method.

Surrogates

Surrogates are organic compounds that are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards and samples (including duplicate, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate and recovery is a measurement of the overall method performance. The percent recovery must meet the limits set forth in the SOP or determined from control charting.

Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are field samples to which a known amount of the target analyte (or analytes) has been added. The samples are then prepared and analyzed in the same analytical batch in exactly the same manner as routine samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

Percent Recovery = ((S – A) x 100) / T

Where:
S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

Matrix spiked samples are prepared and analyzed at the levels and frequency noted in the Standard Operating Procedure for the particular analysis. When matrix spike recoveries fall outside of method or control-charted acceptance limits, the analytical results should be qualified as potentially affected by the matrix.

Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate sample aliquot or a matrix spiked sample and duplicate matrix spiked sample (MS/MSD) are analyzed. The relative percent difference between duplicate analyses or between an MS and MSD is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

Relative Percent Difference = (S1 – S2) x 100 / S_{avg}

Where S1 and S2 = the observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and S_{avg} = the average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Duplicates or MS/MSD analyses are performed at the level and frequency outlined in the Standard Operating Procedure for the analysis being performed.

Duplicates or MS/MSD's are selected on the basis of volume or matrix. Samples with enough volume are selected unless a matrix problem is suspected in which case a sample with enough volume and an appropriate matrix is selected.

Laboratory Control Samples (a.k.a. Laboratory Fortified Blanks or Quality Control Samples)

The laboratory control sample (LCS) is an aliquot of analyte-free matrix to which known amounts of the target analyte(s) is (are) added. A standard reference material of known matrix type, containing certified amounts of target analytes, may also be used as a LCS. The LCS sample is prepared and analyzed in the same analytical batch and in exactly the same manner as the other routine samples. Stock solutions used for LCS's are purchased or prepared independently of calibration standards. The percent recovery (%Rec) of the target analytes in the LCS assists in

determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy. Acceptance criteria for LCS analyses are either specified in the analytical method or obtained through the use of control charts. A LCS is prepared and analyzed at a minimum frequency specified in the Standard Operating Procedure for the specific method being employed. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, occasionally a duplicate LCS (LCSD) will be prepared and analyzed. Laboratory Control Samples are also referred to as Laboratory Fortified Blanks (LFB) or Quality Control Samples (QCS), depending upon the method.

Interference Check Samples

An interference check sample (ICS) is a solution containing interfering elements of known concentration that can be analyzed to verify background and inter-element correction factors in metals analyses.

Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the instrument detection limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

Source and Preparation of Standard Reference Materials

All analytical measurements generated at Anatek Labs, Inc. are performed using materials and/or processes that are traceable to a Standard Reference Material. Standard Operating Procedures are utilized to trace all quantitative and qualitative determinations to certified reference materials. All metrology equipment (analytical balances, thermometers, etc.) is calibrated using materials traceable to the National Institute of Standards and Technology (NIST) and maintained on a schedule to ensure accuracy.

All Sampling containers provided to the client by the laboratory are assured to be free of interfering contaminants by:

1. The container is purchased as pre-cleaned (Level 1) with certificates of analysis available for each bottle type; or
2. The container is cleaned by the laboratory using Standard Operating Procedures; or
3. The specific bottle type and manufacturer has been proven through study to be free of interfering materials; and/or
4. A blank is prepared with a surrogate bottle using laboratory reagent water at the time of sample collection to provide information on possible interferences or contamination resulting from the sample container.

Consumable materials routinely purchased by the laboratory (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem. Services, Inc., Absolute Standards, Aldrich Chemical Co., J.T. Baker, Spex, E.M. Science, Fisher Scientific, etc. are examples of the vendors used by Anatek Labs, Inc. All reference materials that are received are recorded by the technical staff in the appropriate logbook(s) and are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e., vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. In addition, the date that the standard is received in the laboratory is marked on the container. When the material container is opened for use the first time, the date of opening is recorded on the container. Stock solutions and/or calibration standard solutions are prepared fresh as often as necessary according to Standard Operating Procedures. After preparation, all standard solutions are properly labeled as to analyte concentration, solvent, date, preparatory analyst and expiration date; these entries are also recorded in the appropriate logbook(s). Prior to introduction into the analytical system / process, all in-house prepared reference materials are verified with a second, independent source of the material. Once the reference material has been verified to be accurate, it may then be used for instrument calibration and subsequent quantitative purposes. In addition, an independent source of reference material is also used to check the calibration standards for signs of deterioration (i.e., Control or QC samples).

Practical Quantitation Limits (PQL)

The practical quantitation limit (PQL) is defined as minimum concentration of a substance that can be definitively quantified by a method. In general, the practical quantitation limit is the lowest calibration standard concentration. Results reported below the PQL (and above the MDL – see below) are qualified on the final report as estimated concentrations.

Practical quantitation limit is also referred to in some methods or regulations as the Limit of Quantitation (LOQ), Minimum or Method Reporting Limit (MRL), or Reporting Limit (RL).

Method Detection Limits (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. Method detection limits are determined for most analyses performed at Anatek Labs, Inc. Refer to Appendix I for specific information on the development of MDLs.

The MDL is also referred to in some methods or regulations as Minimum Detection Limit or Limit of Detection (LOD).

In general, Anatek Labs, Inc. reports results to the lowest calibration point (PQL). Some clients or projects will request reporting to the MDL. Such results are qualified on the final report as estimated concentrations, or 'J-flagged.'

Control Charting

Control charts are used to establish laboratory-specific, performance-generated acceptance limits for many analytical methods. The generation of control charts is routinely performed at Anatek Labs, Inc. Refer to Appendix H for specific information on the generation and use of control charts.

Quality Document Control

All Standard Operating Procedures and Quality Assurance Plans are maintained under the control of the QA Officer. The QA Officer is responsible for maintaining all official/authorized versions of all Standard Operating Procedures and Quality Assurance Plans. The official, master version of each Standard Operating Procedure is maintained by the QA Officer. Copies of SOPs are available to analysts and other personnel via PDF links on each computer workstation. This QA Plan is maintained in the possession of the QA Officer and a copy is distributed to the Laboratory Manager. All original signatures are maintained on the QA Officer's master copy. Any copies or versions of these documents that are distributed outside the laboratory will not be controlled and updated during annual reviews.

Additional quality systems documents (bench sheets, facilities maintenance forms, etc.) are tracked in a Master List of Quality Systems Documents, which records the revision number and effective date of approved forms.

All instrument activity log books and manuals are maintained by the analysts with the equipment. IALs are periodically inspected by the QA Officer to ensure compliance with standard operating procedures (refer to ALI-15). When full, IALs are archived and retired with the piece of equipment.

Analytical and support records, both electronic and hard-copy, are retained in accordance with Anatek Labs data archiving SOPs.

Standard Operating Procedures (SOPs) and Laboratory Notebooks

Anatek Labs, Inc. maintains a database of SOPs for use in both technical and administrative functions. SOPs are written following the format and content requirements described in the SOP for preparation of SOPs (ALI-01). Each SOP has been reviewed and approved by a minimum of two authorities, the Laboratory Manager and the QA Officer. All SOPs undergo a documented annual review to make sure current practices are described. The QA Officer maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of SOP is being used for guidance and instruction. The QA Manual, SOPs, standards preparation logbooks, run logbooks, etc., are all considered crucial to consistent operations at Anatek Labs, Inc. and

all analysts are instructed on the proper usage of each. Anatek Labs, Inc. maintains a current file, accessible to all laboratory staff, of the promulgated methodology (EPA, Standard Methods, etc.) used to perform analyses as well as this QA Plan and applicable Standard Operating Procedures. (For specific IAL procedures refer to SOP ALI-15.)

Deviation from Standard Operating Procedures

Anatek Labs, Inc. recognizes that occasionally a modification to a Standard Operating Procedure may be necessary. In such cases, a written record of the deviation is retained with the sample data and if the deviation affects the data integrity an appropriate data qualifier comment is noted on the analytical report. An example of this would include a special preparative step or procedure not normally performed but perhaps mandated by special matrix concerns.

Modified Procedures

Anatek Labs, Inc. strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

Policy on Manual Integration

Automated integration data reduction software currently available is generally accurate when performing peak integration for chromatographic analyses. However, instances occur where the instrument software does not yield the proper integration and the analytical data is inaccurate. Some examples include, but are not limited to, peak splitting, coeluting interferences, peak detection failure, peak tailing, and failure to separate peaks. Accurate measurements require an analyst to review peak integration and evaluate if adjustments need to be made.

Manual integration is never appropriate when performed solely for the purpose of meeting method QC criteria or compliance requirements, avoiding rework or instrument maintenance. Inappropriate manual integrations include peak shaving, peak enhancement, and baseline manipulation.

Violation of this policy is subject to disciplinary action up to and including termination of employment.

Integration Procedure and Review

All data are generated and reduced following the procedures specified in the methods and/or SOPs. Chromatograms are evaluated for chromatography performance criteria, including:

Baseline noise (3 to 1 signal to noise ratio)
Peak resolution
Peak tailing (good column performance should produce symmetrical peaks with minimum tailing for most compounds)
Peak splitting
Co-elutions
Negative spikes in baselines

Corrective action must be taken when the chromatography has deteriorated. Corrective actions include:

Trimming head of column
Guard column replacement
Cleaning detectors and/or ion source
Cleaning injector ports, replacing ferrule, liner, gold seal or washer
Identifying leaks
Replacing the column
Changing trap
Change suppressor
Change eluent

Change regenerant for IC systems

The analyst must review all automatic integrations for all parameters in the method. This review must include:

Relative retention time/retention time shifts
Identification of peaks
Mass spectrum primary ion abundance – secondary ions maximize within one scan of primary ion (for GC/MS analyses)
Peak shape
Interference
Consistency
Verification that baseline is clearly visible
Inspection of auto and manual integration for proper technique and necessity of manual integration

When auto integration is determined to be incorrect (for example, peak splitting, coeluting interferences, peak detection failure, peak tailing, and failure to separate peaks), the peak(s) must be manual integrated to correct the area response. Integration must be consistent throughout the analytical run for samples, QC samples, blanks, and calibration standards.

Quality control will include reviewing chromatograms and verifying that manual integrations, when performed, are appropriate and analytically sound.

System and Performance Audits

Laboratory Evaluations and Audits are to be conducted under the authorization of the QA Committee and all findings and recommendations are submitted to the QA Committee for decisive action. System Audit requests are generated internally and externally. Internal audits are generally scheduled at the frequency noted under the type of review, however concerns brought to the attention of the QA Committee may necessitate an unscheduled systemic review at the discretion of the QA Committee. External audit requests are referred to the QA Committee for authorization and scheduling of external auditors to review systems.

The following evaluations are performed at Anatek Labs, Inc.:

Management System Reviews (MSRs)

MSRs are external audits conducted at Anatek Labs, Inc. Idaho Dept. of Health Bureau of Laboratories audits Anatek Labs, Inc. to assess the adequacy of the overall QA Plan. FL DOH (for NELAP) and ID Bureau of Laboratories perform rigorous on-site inspections of Anatek Labs, Inc.'s QA Plan, adequacy of facilities, Quality Control Records, Performance Evaluations, Standard Operating Procedures and Analyst abilities, and submit audit reports to Anatek Labs, Inc. These reports and any corrective actions plans are maintained at Anatek Labs, Inc. Washington Department of Ecology does a complete inspection as well for WA wastewater and soil/solid certification. Anatek Labs, Inc. also makes the facilities available for customer or regulatory agency inspection of Management Systems as well. Anatek Labs, Inc.'s QA Committee reviews all MSR reports and recommendations. If reports indicate the necessity for corrective action, the QA Committee or its designee will prepare and implement a Corrective Action Plan. The Corrective Action Plan will itemize the specific action necessary to correct the deficiency and define the time frames and responsible parties for implementation and follow-up.

Technical System Audits (TSAs)

TSAs are both internal and external audits conducted at Anatek Labs, Inc. Florida Dept. of Health (NELAP), Idaho Dept. of Health, WA Dept. of Ecology, and Arizona Department of Health Services evaluate Anatek Labs, Inc.'s technical systems. The above listed agencies review calibration records, sampling and measurement procedures, general lab cleanliness, support systems, equipment and facilities, maintenance and repair records, control charts and general operation of the lab. The inspecting agencies prepare audit reports to Anatek Labs, Inc. and these reports and all responses and corrective action plans are maintained at Anatek Labs, Inc. Additionally, Anatek Labs, Inc. staff performs internal TSAs annually. The QA Officer performs an annual inspection of Standard Operating Procedures, quality systems, calibration and maintenance records, and employee training records. TSA audit reports are prepared by the external auditor or QA Officer and given to the QA Committee or the analyst as appropriate

(reference SOP ALI-16). If a report indicates the necessity for corrective action a Corrective Action Plan will be prepared and implemented according to SOP ALI-07. The Corrective Action Plan will itemize the specific action necessary to correct the deficiency and define the time frames and responsible parties for implementation and follow-up. The results from all corrective action plans will be compiled and forwarded to management and/or the QA Committee as necessary.

Performance Evaluation (PE)

PE's are performed according to regulatory requirements for NELAP and the various testing regimens employed at Anatek Labs, Inc. Anatek Labs, Inc. participates in at least two Water Supply (WS), two Water Pollution (WP) and two solids/soils (RCRA) Performance Evaluations annually. All PE Sample materials are procured from a NIST/NVLAP approved provider. Acceptable results for each analyte and method used to perform regulatory testing are demonstrated semi-annually. In the event that an unacceptable result is received for a particular analyte, corrective actions are employed. Blind studies may be initiated by the lab to verify performance. Additionally double-blind studies may be conducted at Anatek Labs, Inc. when initiated by customers.

Data Quality Audits (DQAs)

Peer review of acceptable blank results, QA/QC sample recovery, matrix spike and matrix spike duplicate recoveries, reproducibility of duplicate samples, and verification of sample calculations are performed on approximately 10% of analytical batches. Any errors or deviations from acceptable criteria are noted and data is returned to the generating analyst for correction. In the event that QA/QC criteria for a particular sample or batch of samples cannot be met, all associated sample reports are noted that QA/QC criteria were not met and may include other relevant discussions. If numerous problems are found with a particular method or analyst, more data packets will be reviewed.

IT Systems Auditing

IT Systems Auditing of Anatek Labs, Inc. is conducted both internally and externally. The external auditing is conducted by IDOH, WA DOE and FL DOH. The agencies review Anatek Labs, Inc.'s IT SOPs, IT documentation, access security and backup/restore plans during their inspections. Internal audits are performed at least once a year by Anatek Labs, Inc.'s QAU with assistance from IT personnel. The internal audits will inspect the network security, network throughput performance, server storage available, backup/restore plan testing and general documentation of the IT systems. An IT systems auditing report is prepared by the external auditor or QA Officer and given to the QA Committee. If a report indicates that corrective actions are necessary, a Corrective Action Plan will be prepare and implemented by IT personnel. The Corrective Action Plan shall specifically address the areas of deficiency and the actions to be taken.

Corrective Action

Corrective action is a function of the laboratory as a whole and is dependent upon the nature of the problem. Corrective action is initiated when deviations or non-conformances with laboratory or regulatory practices are identified. Some examples include unacceptable PT results, internal or external audit findings, data or record review findings, and customer complaints.

Corrective action may take several forms. Some findings (for example, matrix spike recoveries that fail recovery limits) may only require a note on the data or the final report, while other findings will initiate a documented Corrective Action Report. Corrective action reports identify the problem noted, an investigation, a root cause analysis of the problem, any actions taken to correct or prevent the problem, and followup activities.

Customer complaints will be directed to the Laboratory Manager, QA Manager, and/or section supervisors. Every reasonable effort should be made to address (and correct, if necessary) customer complaints. If the lab is found to be at fault, a corrective action form should be initiated and completed.

Preventative Action

Anatek Labs, Inc. and its personnel always strive to improve the laboratory procedures, including analysis, record-keeping, and customer service. Preventative action is a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints. When improvement opportunities or preventative actions are identified, action plans will be developed, implemented, and evaluated for effectiveness. All Anatek Labs employees are encouraged to look for and identify potential improvements to lab safety, efficiency, and customer service.

Training & Personnel Qualifications

All personnel involved in any function affecting data quality will have sufficient training and technical expertise to effectively execute their job requirements. The laboratory evaluates all prospective job applicants for scientific knowledge and experience as noted in the job descriptions for the position considered.

New employees receive documented training on the Quality Assurance Plan, laboratory safety, standard operating procedures, and data integrity, as well as method-specific training. A record of specialized training received by or given by the staff is kept in the Personnel Training folders.

In addition to prior work and educational experience, Anatek Labs, Inc. actively encourages its employees to expand and refine their job skills and knowledge through participation in a variety of educational programs. Time off is granted to attend seminars and training sessions put on by instrument manufacturers, regulatory agencies, professional business and scientific organizations, etc. Additionally Anatek Labs, Inc. conducts in-house training on related topics. Anatek Labs, Inc. also encourages continuing education through a tuition reimbursement program.

Demonstration of Capability (DOC)

Analysts perform an Initial Demonstration of Capability (IDOC) when performing a new method or a method they have not performed in a 12-month period. In general, for an IDOC, mean recovery and standard deviation from four replicates of a quality control sample are compared to method or SOP acceptance criteria. Demonstrations of capability are verified annually, either by an on-going demonstration of capability (ODOC), performed similarly to the IDOC, or by successful performance of a blind proficiency testing sample.

Facilities, Equipment and Services

Anatek Labs, Inc. was founded in 1992. Anatek Labs, Inc. is a full service environmental testing laboratory serving the Inland Empire and the Pacific Northwest. Anatek Labs operates facilities in Moscow, Idaho and Spokane, Washington.

A listing of major analytical equipment used at Anatek Labs, Inc. can be found in Tables 1a & 1b of the Appendices this document.

Calibration Procedures and Frequency

All equipment and instruments used at Anatek Labs, Inc. are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Personnel who have been properly trained in these procedures perform operation and calibration. Documentation of calibration information is maintained in data archives (see SOP ALI-14, Data Archiving) or Instrument Activity Logs (IALs). Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. More information is contained in laboratory SOPs.

Analytical Instrumentation

Each instrument utilized at Anatek Labs, Inc. is calibrated against traceable standards. Standard Operating Procedures are used to document the procedure for ensuring traceability of stock reference materials. Standard Operating Procedures also specify specific instrument settings, calibration concentrations and frequency, instrument linear ranges, specific required QA/QC measures and a number of other related technical issues.

Preventive Maintenance

Preventive maintenance is a crucial element of Anatek Labs, Inc.'s Quality Assurance program. Qualified in-house personnel maintain instruments, such as GC/MS systems, spectrometers, analytical balances and gas and liquid chromatographs. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument activity logbooks (IALs). The IALs contain extensive information about the instruments used at the laboratory.

When an instrument is acquired at the laboratory, the following information is noted in a maintenance notebook specifically associated with the new equipment:

1. The equipment's serial number;
2. Date the equipment was received;
3. Date the equipment was placed into service;
4. Condition of equipment when received (new, used, reconditioned, etc.); and
5. Prior history of damage, malfunctions, modification or repair (if known).

Preventative maintenance procedures, frequencies, etc. are available for each instrument. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase. Responsibility for ensuring that routine maintenance is performed lies with the Laboratory Manager. The Laboratory Manager may perform the maintenance, assign the maintenance task to a qualified bench level analyst or acquire on-site manufacturer repair.

When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem (attempted repairs, etc.) is also recorded in the IAL. Typical logbook entries include the following information:

1. Details and symptoms of the problem;
2. Repairs and/or maintenance performed;
3. Description and/or part number of replaced parts;
4. Source(s) of the replaced parts; and/or
5. The analyst's initials and date.

Temperature Record Keeping

Temperatures are monitored and recorded for all of our temperature-regulating devices including ovens, incubators and refrigerators when in use. An electronic monitoring system is used to track temperatures in a number of refrigerators and freezers. Liquid thermometers are available as a backup. The following are instruments that are documented daily (during operations) and the associated acceptable average temperature limits:

Sample Archive	$0 – 4^0C$
Drinking Water Storage	$0 – 6^0C$
Drinking Water VOC Storage	$0 – 6^0C$
Non Drinking Water Storage	$0 – 6^0C$
Waste Water VOC Storage	$0 – 6^0C$

All thermometers are checked annually against a National Institute of Standards and Technology (NIST) certified thermometer.

Analytical Balances

Analytical balances are serviced on an annual basis by a professional metrology organization. New certificates of calibration for each balance are issued to the laboratory on an annual basis. The calibration of each analytical balance is verified daily. As needed, the balances are recalibrated using the manufacturer's recommended operating procedures. Records are kept that contain the recorded measurements, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks.

Water Purification System

There are a variety of water purification systems used at Anatek Labs, Inc. A filtration system is in place to provide deionized water throughout the Laboratory. The system is monitored and provides a purity of at least 1 MΩ (up to approximately 18 MΩ). When purity falls below 1 MΩ, the system is serviced by Culligan (Moscow) or King Soft Water (Spokane) and new filters are installed. Additionally there is a filter system that provides 18 MΩ purity water. The specifications, preventative maintenance schedules and other information for particular water purification systems are explained in detail in the applicable Standard Operating Procedure.

Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at Anatek Labs, Inc. undergoes a rigorous cleansing procedure prior to every usage. Refer to SOP ALI-03 and method specific SOPs for specific glassware cleaning procedures.

Services and Supplies

Anatek Labs, Inc. purchases services and supplies from reputable vendors, and ensures that supplies meet or exceed standards established in the analytical methods. A list of approved vendors is maintained, and updated annually.

Waste Disposal

All samples received at Anatek Labs, Inc. remain in the ownership of the submitting party. Unless analysis of the samples demonstrates hazardous/regulated levels of contaminants, liquid samples are routinely disposed of by disposal in the sanitary sewer after adjustment to a pH specified by the local wastewater treatment facility. Solid samples are disposed of using the solid waste sanitation services. Samples demonstrated to be inappropriate (hazardous) to be disposed of by routine means are returned to the client for disposal/treatment at the original sampling location or retained in a manner consistent with mineral acid, solvent or other hazardous materials storage and disposal activities within Anatek Labs, Inc.

All mineral acids, solvents and other hazardous materials used in the daily operation of the laboratory are collected in designated areas until sufficient material is collected for cost-effective disposal at a licensed disposal facility.

Subcontracting of Laboratory Services

Analytical services may be subcontracted when the requested analyses are not performed by Anatek Labs, Inc. Subcontracting of laboratory services is done only with the knowledge and approval of the client.

The acceptability of subcontracting laboratories is assessed using the following criteria:

1. The subcontracting laboratory is certified for the analysis requested if results are for regulatory purposes;
2. The subcontracting laboratory has an approved/audited Quality Assurance Plan and/or an established reputation for providing quality services;
3. The subcontracting laboratory agrees to perform and provide specific Quality Control measures outlined by the project manager or sample submitter;
4. The subcontracting laboratory agrees to retain records for a period of time no less than outlined by the project manager or sample submitter.

Termination or Transfer of Business

In the event Anatek Labs goes out of business or ownership is transferred, available clients will be contacted, and customer records will be dealt with according to client instructions and state and regulatory requirements. For those clients who cannot be contacted, customer records will be destroyed in the event the lab goes out of business or transferred in the case of new ownership.

Anatek Labs, Inc.

Quality Assurance Plan
Tables, Figures & Appendices

Items below are linked – click on a heading to proceed to that section

Tables .....

1a Analytical Equipment Utilized at Anatek Labs, Moscow .....
1b Analytical Equipment Utilized at Anatek, Labs, Spokane .....
2 Summary of Analytical Parameters, Method, Sample Containers, Preservation Methods, Holding Times and Estimated Working time .....

Figures.....

1 Anatek Labs, Moscow Organizational Chart.....
2 Anatek Labs, Moscow Personnel.....
3 Anatek Labs, Moscow Floor Plan.....
4 Anatek Labs, Spokane Organizational Chart.....
5 Anatek Labs, Spokane Personnel.....
6 Anatek Labs, Spokane Floor Plan.....

Appendices .....

A Index of Standard Operating Procedures .....
B Examples of Sample Submission Form .....
C Quick Reference for Chemical Safety .....
D Laboratory Staff Resumes / Qualifications.....
E Current State Certifications.....
F Backup, Fault Tolerance, Disaster Recovery and Data Archive of Mission-Critical Information Storage and Services.....
G Control Chart Information .....
H Method Detection Limit.....
I Calibration Methods and Equations.....
J Authorized Signatures.....

Tables

Tables 1a & 1b

Instrument Inventory

Table 1a

Anatek Moscow Equipment List

Type	*Description*	*Manufacturer*	*Model*
API-3000	TRIPLE QUAD MS/MS	PE SCIEX	API-3000
DISCRETE ANALYZER	Spectrophotometer	SYSSTEA	EASY CHEM PLUS
ECD1	GC SYSTEM	HP	HP6890
ECD2	GC SYSTEM	HP	HP6890
FIA	ANALYZER	OI ANALYTICAL	FS 3000
FID1	GC SYSTEM	HP	6890
FID2	GC SYSTEM	AGILENT	6890N
HPLC #2	PCX	PICKERING	PINNACLE PCX
	DIODE ARRAY DETECTOR	SHIMADZU	SPD M20A
	FLUORESCENCE DETECTOR	SHIMADZU	RF20A
ICP	ICP-OES	VARIAN	720-ES
ICP-MS	ICP-MS	AGILENT	G3155A
IC	COMPACT IC	METROHM	761
Mercury Analyzer	Hg Analyzer	CETAC	M8000
MSD2	MSD	AGILENT	5973N
	GC SYSTEM	AGILENT	6890N
OIL & GREASE	EXTRACTOR	HORIZON	3000
SATURN 2100	GC/MS	Varian	2100T
SEMIVOC	GC SYSTEM	AGILENT	6890
	MSD	AGILENT	5973N
VARIAN 1200	GC	VARIAN	CP-3800
	QUADRUPOLE MS/MS	VARIAN	1200
VARIAN 4000	MS/MS	VARIAN	4000
	GC SYSTEM	VARIAN	4000
VOC1	GC	Agilent	6890N
	MS	Agilent	5975

TABLE 1B

Anatek Labs Equipment List - Spokane

Type	*Description*	*Manufacturer*	*Model*
Autoclave		Market Forge Sterilmatic	STM-EL
BOD	LBOD 101 Probe	Hach	HQ40d
BTU	Oxygen Bomb Calorimeter	Parr	1341EB
Chlorine	Colorimeter	Hach	58700-00
COD	Digestor	HACH	
Coliform		IDEXX Quanti-tray 2X	89-10894-00
Conductivity	Meter	Hach	HQ40d
FIA	Analyzer	Lachet	QUIKCHEM 8500
	TKN Digestor Block	Lachet	BD-46
	Cyanide Distillation System	Lachet	65454
FOG		CPI Instruments	
GC/ECD	GC	Hewlett Packard	5890 SERIES II
GC/FID	GC	Hewlett Packard	5890 SERIES II
GC/FID	GC	Hewlett Packard	6890 SERIES II
GC/MS	Purge and Trap	Encon	ENCON EV
	Autosampler	Centurion	CENT272011509
	GC	Hewlett Packard	5890 SERIES II PLUS
	MS		
GC/MS	Purge and Trap	TEKMAR	3000
	Autosampler	Varian Archon	
	GC	Hewlett Packard	5890 SERIES II PLUS
	MS		
GC/PID/FID	Concentrator Purge and Trap	Hewlett Packard	4430
Gross a and ß counter	Gas Flow Proportional Counter	Protean Instrument	IPC650
HPLC	Autosampler	Agilent Technology	G1329B
	UV	Agilent Technology	G1314B
IC	ICS-4000 Capillary HPIC	Thermo Scientific	4000
ICPMS	ICPMS	Agilent	7500cx
Mercury Analyzer	Analyzer	Cetac	M-7600
pH meter		Denver Instruments	225
pH		Hach	HQ40d
Salmonella		SEWARD STOMACHER	400
Spectrophotometer	Analyzer	HACH	DR/3000
TOC		DOHRMANN PHOENIX	8000
TOX	Analyzer	MITSUBISHI MCI	
Turbidimeter		HACH	2100AN
		WTW	TURB355IR

Type	Description	Manufacturer	Model
GC/PID/FID	Concentrator Purge and Trap	Hewlett Packard	4430
	Lamp Power Supply	Hewlett Packard	
	Autosampler	TEKMAR	ALS 2016
		TEKMAR	LSC 2000
Gross α and β counter	Windowless Gas Flow Proportional Counter	Protean Instrument	IPC650
Hoods		Genie Scientific	400SLBB
		Genie Scientific	400SLBB
		Kewaunee Scientific	
		LABCON	70198-90
		LABCON	70198-90
		Genie Scientific	500SLBB
IC	Compact IC	Metrohm Peak	761
	Autosampler	Metrohm Peak	788
ICPMS	ICPMS	Agilent	7500cx
	Autosampler	Agilent	ASX-500
	Recirculating Pump	Agilent	6106T / G3292A
Incubator		THERMOLYNE BLUE	B2730-Q
		THERMOLYNE	142325
Media Dispensor		TRITECH Research	POURBOY 4
N-Evap		Organimation	111
pH/Cyanide		Denver Instruments	225
	ATC	ATC	300729.1
	Probe	VWR	SYMPHONY
	Cyanide Probe	ORION	9406BN
	Cyanide Probe	ORION	900200
pH		Hach	HQ40d
	Probe	Hach	HQ40d
Pipettes	0.5-10	VWR EHP	
	0.5-10	VWR	
	10-100	VWR	
	10-100	Mettler Toledo VoluMate	
	20-200	VWR EHP	
	20-200	VWR EHP	
	100-1000	Drummond	
	100-1000	Drummond	
	100-1000	VWR	
	100-1000	Mettler Toledo VoluMate	
	100-1000	Mettler Toledo VoluMate	
	100-1000	Mettler Toledo VoluMate	
	100-1000	Mettler Toledo VoluMate	
	100-1000	Mettler Toledo VoluMate	
	100-1000	Eppendorf Reference	
	500-5000	Mettler Toledo VoluMate	
	1000-5000	VWR EHP	
	1000-5000	VWR	
	1000-10000	VWR	
	1000-10000	VWR	
Refrigerators	WET CHEM FRIDGE 1	Kenmore	253.60722000
	524 FRIGDE		
	VOC FRIDGE	Kenmore	253.60722004
	VOC FREEZER	Kenmore	253.24202102
	STANDARDS FRIDGE	Kenmore	253.60722004
	WET CHEM FRIDGE 2	Kenmore	253.60722007
	ICE PACK FREEZER	Kenmore	WB15021458
	WALK IN REFRIGERATOR		

Type	Description	Manufacturer	Model
Salmonella		SEWARD STOMACHER	400
Sonicator		VIBRA CELL SONICS MATERIALS	VC375
		Branson 2510 Ultrasonic	2510R-MT
Spectrophotometer	Analyzer	HACH	DR/3000
Stirplates		isotemp - Fisher Scientific	
		VWR-Thermolyne Corp	320
		Nuora II Stirrer	SI8525
		SH-3 Stirring/Hot Plate	
		Fischer Scientific Stirrer	
		VWR 375 Stirring/Hot Plate	
Thermometer	Infra Red K	VWR	RS232
TOC		DOHRMANN PHOENIX	8000
TOX	Analyzer	MITSUBISHI MCI MICROCOULOMETER	
	Sample Preparator		TOX-10-A
Turbidimeter		HACH	2100AN
		WTW	TURB355IR
Turbovap II		Zymark	47570/0
UV Light		Spectroline	EA160
Vacuum Pump		GAST	M100GX
Vortexer		Fisher Scientific	12810-1
		VWR	
Waterbath		VWR	1275 PC
Weights	10mg-100g weight set	TROMNER	SN 19151
	10mg weight	Mettler Toledo	11123047

Anatek Labs, Inc.

Table 2

Summary of Analytical Parameters, Method, Sample Containers, Preservation Methods, and Holding Times

Test Type	Parameter	Method	Container	Preservative	Holding Time
Organics - Drinking Water					
COMPOST	Herbicide/ Pesticide Residue in Soil/Compost		4 oz glass	n/a	14 days
DBP	Haloacetic Acids	SM 6251B	2 x 40 mL amber vial no headspace	4 mg Ammonium Chloride	28 days
DBP	Total Trihalomethane Potentials (THMP)	EPA 524.3	3 x 40 mL amber vial no headspace	Ambient Temperature	ASAP
DBP	Total Trihalomethanes (TTHM)	EPA 524.3	2 x 40 mL amber vial no headspace	25 mg ascorbic acid, 200 mg maleic acid	14 days
DIOXIN	Dioxin				
DIQUAT	Diquat	EPA 549	500 mL HDPE	10 mg Sodium Thiosulfate	7 days
EDB	EDB/DBCP	EPA 504.1	2 x 40 mL amber vial	3 mg Sodium Thiosulfate	14 days
ENDOTHALL	Endothall	EPA 548.1	250 mL amber glass	20 mg Sodium Thiosulfate	14 days
GAS	Petroleum in Soil-HCID WA TPH-HCID	NWTPH-Gx/EPA 8015	4 oz glass	n/a	14 days
GLYPHOSATE	Glyphosate	EPA 547	40 mL amber vial	3 mg Sodium Thiosulfate	14 days
HERB1	Herbicides Regulated & Unregulated	EPA 515.4	250 mL amber glass	20 mg Sodium Thiosulfate	14 days
HERB2	Private Herbicide	EPA 515.4	250 mL amber glass	Sodium Thiosulfate	14 days
HERB2	Private Herbicide	EPA 515.4	1 L amber glass	Sodium Thiosulfate	14 days
CARB	Carbamates Regulated & Unregulated	EPA 531.2	40 mL amber vial	6mg Sodium Thiosulfate / 560mg Potassium Dihydrate Citrate	28 days
NWTPH-HCID	Petroleum	NWTPH-HCID	1L amber bottle	HCl pH<2, Cool to <4°C	7 days
PERCHLORATE	Perchlorate	EPA 331.0/6850	125 mL HDPE	Cool to <4°C	14 days
PEST1	Chlorinated Pesticides/PCBs	EPA 505	2 x 40 mL amber vial	3 mg Sodium Thiosulfate	14 days
SOC	(SOC) Haloacetic Acids	SM 6251B	2 x 40 mL amber vial	4 mg NH4CL	14 days
SOC	Full SOC	Multiple methods			14 days
SOC	Synthetic Organic (SOC) Screen - Washington	Multiple methods	1 L amber glass and 1 x 40 mL amber vial	50 mg Sodium Sulfite 4 mg Sodium Thiosulfate	14 days
SOC	Synthetic Organic (SOC) Screen	Multiple methods	1 L amber glass	50 mg Sodium Sulfite	14 days
SOC	WA Short SOCs	Multiple methods			14 days
SOC Phase II	(Phase II SOC) Carbanates Regulated & Unregulated	EPA 531.2	40 mL amber vial	6mg Sodium Thiosulfate / 560mg Potassium Dihydrate Citrate	28 days
SOC Phase II	(Phase II SOC) Chlorinated Pesticides/PCBs	EPA 505	2 x 40mL amber vial	50 mg Sodium Thiosulfate	14 days
SOC Phase II	(Phase II SOC) EDB/DBCP	EPA 504.1	2 x 40 mL amber vial	3 mg Sodium Thiosulfate	14 days
SOC Phase II	(Phase II SOC) Herbicides Regulated & Unregulated	EPA 515.4	250 mL amber glass	20 mg Sodium Thiosulfate	14 days
SOC Phase II	(Phase II SOC) Semivolatiles	EPA 525.2	1 L amber glass	50 mg Sodium Sulfite	14 days
SOC Phase V	(Phase V SOC) Diquat	EPA 549.2	500mL HDPE	Sodium Thiosulfate	21 days
SOC Phase V	(Phase V SOC) Endothall	EPA 548.1	250 mL amber glass	Sodium Thiosulfate	14 days
SOC Phase V	(Phase V SOC) Glyphosate	EPA 547	2 x 40 mL amber vial	Sodium Thiosulfate	14 days
TOC	Total Organic Carbon (TOC)	SM 5310B	2 x 40 mL amber vial	H2SO4 or HCl pH<2	14 days
VOC	(VOC) Regulated & Unregulated Full List	EPA 524.3	3 x 40 mL amber vial	25 mg ascorbic acid, 200 mg maleic acid	14 days
VOC	(VOC) Single Volatile Compound (e.g. TCE or MTBE)	EPA 524.3	3 x 40 mL amber vial	25 mg ascorbic acid, 200 mg maleic acid	14 days
VOC	(VOC) Total Trihalomethanes (Total THM)	EPA 524.3	3 x 40 mL amber vial	25 mg ascorbic acid, 200 mg maleic acid	14 days
VOC	(VOC) Trihalomethanes (THMP)	EPA 524.3	3 x 40 mL amber vial	n/a	14 days
VOC1 Chlorinated	Regulated & Unregulated Full List	EPA 524.3	3 x 40 mL amber vial no headspace	25 mg ascorbic acid, 200 mg maleic acid	14 days
VOC1 Not chlorinated	Regulated & Unregulated Full List	EPA 524.3	3 x 40 mL amber vial no headspace	25 mg ascorbic acid, 200 mg maleic acid	14 days
VOC2	Private Volatiles (VOC)	EPA 524.3	3 x 40 mL amber vial no headspace	25 mg ascorbic acid, 200 mg maleic acid	14 days
VOC2	Single Volatile Compound (i.e. TCE or MtBE)	EPA 524.3/624/8270	3 x 40 mL amber vial no headspace	25 mg ascorbic acid, 200 mg maleic acid	14 days

Test Type	Parameter	Method	Container	Preservative	Holding Time
Inorganics - Drinking Water					
ASBESTOS	Asbestos in Insulation or Tile		100 grams, Ziploc bag (no vermiculite)	Cool to <4°C	6 months
ASBESTOS	Asbestos in Insulation or Tile		100 grams, Ziploc bag (no vermiculite)	n/a	6 months
FLUORIDE	Fluoride	EPA 300.0	125 mL HDPE	n/a	7 days
ID IOC Package	Arsenic & Sodium	EPA 200.8 / 200.7	125 mL HDPE	n/a	6 months
ID IOC Package	Fluoride	EPA 300.0	125 mL HDPE	n/a	28 days
ID IOC Package	Idaho Primary IOC package with cyanide waiver	Multiple methods	1 L HDPE	n/a	28 days
ID IOC Package	Idaho Secondary/Optional IOC package	Multiple methods	1 L HDPE	n/a	ASAP
ID IOC Package	Nitrate	EPA 300.0	125 mL HDPE	n/a	48 hours
ID IOC Package	Nitrate/Nitrite	EPA 300.0	125 mL HDPE	n/a	48 hours
ID IOC Package	Phase II IOC metals	EPA 200.7/200.8	500 mL HDPE	n/a	28 days
ID IOC Package	Phase V IOC	Multiple methods	500 mL HDPE	n/a	6 months
IOC	Alkalinity	SM 2320B	125 mL HDPE	n/a	14 days
IOC	Ammonia	SM 4500 NH3-G	125 mL HDPE	H2SO4 pH<2	28 days
IOC	Anionic Surfactants	SM 5540C	250 mL HDPE	n/a	48 hours
IOC	Anions: (Cl, F or SO4)	EPA 300.0	125 mL HDPE	n/a	28 days
IOC	Anions: (NO2, NO3 or PO4)	EPA 300.0	125 mL HDPE	n/a	48 hours
IOC	Chlorine - Total Residual	SM 4500 Cl-G	2 x 40mL clear vial no headspace	n/a	15 minutes
IOC	Color	SM 2120B	125 mL HDPE	n/a	48 hours
IOC	Conductivity	SM 2510B	125 mL HDPE	n/a	28 days
IOC	Corrosivity - Langlier		1 L HDPE	n/a	ASAP
IOC	Cyanide	EPA 335.4/SM 4500-CNE	250mL HDPE	NaOH pH > 12	14 days
IOC	Hardness	EPA 200.7 / SM 2340B	125 mL HDPE	HNO# pH <2	6 months
IOC	Hydrogen Sulfide		1 L HDPE	1 Tablet NaOH/L + Zn Acetate, pH>9	7 days
IOC	Iron	EPA 200.7	125 mL HDPE	HNO3 pH < 2	6 months
IOC	Lead & Copper (Pb/Cu) Rule	EPA 200.8	1 L HDPE	HNO3 Lab Preserves	6 months
IOC	Mercury by CVAFS	EPA 245.7/EPA 1631E	I L glass or 250 mL fluorinated plastic	HCl pH < 2	6 months
IOC	Metals (single metals)	EPA 200.7/200.8/6010/6020	125 mL HDPE	HNO3 pH < 2	6 months
IOC	Odor		1 L HDPE	n/a	ASAP
IOC	pH	SM 4500 H+B	125 mL HDPE	n/a	15 minutes
IOC	Phenolics	EPA 420.1	1L amber bottle	H2SO4 pH<2	28 days
IOC	Silica/Silicon - Dissolved Only	EPA 200.7	125 mL HDPE	n/a	6 months
IOC	Surfactants	SM 5540C	1 L amber glass	n/a	48 hours
IOC	TDS	SM 2540C	1 L HDPE	n/a	7 days
IOC	TSS	SM 2540D	1 L HDPE	n/a	7 days
IOC	Turbidity	EPA 180.1	125 mL HDPE	n/a	48 hours
IOC	Washington Complete IOC	Multiple methods	2 x 1 L HDPE	NaOH for CN in 1L	48 hours
IOC	Idaho Complete IOC	Multiple methods	1 L HDPE	n/a	48 hours
LEAD	Lead in Paint	EPA 6020	10 grams, Ziploc bag	Cool to <4°C	6 months
NITRATE	Nitrate	EPA 300.0	125 mL HDPE	n/a	48 hours
Spokane Co. Test Package	Coliform Bacteria, Nitrate	EPA 9223B/EPA 300.0	125 mL HDPE and Sterile 125 mL bottle	Sodium Thiosulfate (in Sterile bottle only)	30 hours
Tri-County Area Test Package	Coliform Bacteria, Nitrate, Lead, Arsenic	Multiple methods	1 Sterile 125 mL HDPE and 1 125 mL HDPE	Sodium Thiosulfate (in Sterile 125 mL HDPE only)	30 hours

Test Type	Parameter	Method	Container	Preservative	Holding Time
Private Well - Drinking Water					
PRIVATE WELL	Private Well List (with bacteria)	Multiple methods	125 mL HDPE and Sterile 125 mL bottle	Cool to <4°C	30 hours
PRIVATE WELL	Private Well List (without bacteria)	Multiple methods	125 mL HDPE	Cool to <4°C	48 hours
Microbiology - Drinking Water					
Bacteriological	Coliform P/A as Count	SM 9223B	Sterile 125 mL bottle	Sodium Thiosulfate	30 hours
Bacteriological	Heterotrophic Plate Counts (HPC)	SM 9215B	Sterile 125 mL bottle	Sodium Thiosulfate	30 hours
Bacteriological	Iron Bacteria		125 mL HDPE	n/a	30 hours
Bacteriological	Coliform Presence/Absence	SM 9223B	Sterile 125 mL bottle	Sodium Thiosulfate	30 hours
Bacteriological	Coliform Bacteria	SM 9223B	Sterile 125 mL HDPE	Sodium Thiosulfate	30 hours
Bacteriological	Coliform Bacteria - Waste Water	SM 9223B	Sterile 125 mL HDPE	Sodium Thiosulfate	6 hours
BOD	BOD	SM 5210B	1 L plastic	n/a	2 days

Microbiology - Waste Water

Test	Method	Container	Preservative	Holding Time
COLIFORM MPN	SM9223B	Plastic	Na2S2O3	6 Hours
E COLI	SM9221F	Plastic	Na2S2O3	8 Hours
E COLI MPN	SM9223B	Plastic	Na2S2O3	6 Hours
FECAL COLIFORMS	SM9221E	Plastic	Na2S2O3	8 Hours
HETEROTROPIC PLATE COUNT	SM9215B	Plastic	Na2S2O3	8 Hours
TOTAL COLIFORMS	SM9221B	Plastic	Na2S2O3	6 Hours
TOTAL/ECOLI COLILERT	SM9223B	Plastic	Na2S2O3	24 Hours

Organics - Waste Water

Test	Method	Container	Preservative	Holding Time
624 VOLATILES IN WW	EPA 624	40mL Amber Vial	HCl	14 Days
AMINOPYRALID	HPLC/MS/MS		n/a	10 Days
BTEX	EPA 624/8260	40mL Amber Vial	HCL	14 Days
CLOPYRALID	GC/MS/MS	Plastic or glass	n/a	14 Days
DIESEL	NWTPHD/EPA 8015D	1L Amber Glass	HCl	14 Days
EPA 8141A	EPA 8141A	1L Amber Glass	n/a	7 Days
EPH - WA DOE	WTPHEPH	G	n/a	7 Days
EXPLOSIVES GC	EPA 8095	1L Amber Glass	n/a	7 Days
EXPLOSIVES HPLC	EPA 8330	1L Amber Glass	n/a	7 Days
GASOLINE	NWTPH-G/EPA 8015D	40mL Amber Vial	HCl	14 Days
Glyphosate-LC/MS	EPA 8321A	250mL Amber Glass	n/a	14 Days
HERBICIDES	EPA 8151A	1L Amber Glass	n/a	14 Days
KEROSENE MOSC	EPA 8015D	1L Amber Glass	HCl	14 Days
METHAMPHETAMINE	HPLC/MS/MS			28 Days
OC PEST 8081A	EPA 8081A	G	n/a	7 Days
OP PESTS	EPA 8270Dm	1L Amber Glass	n/a	7 Days
PAH 8270D MOSC	EPA 8270D	1L Amber Glass	n/a	7 Days
PCB 8082	EPA 8082	G	n/a	7 Days
PERCHLORATE 331.0	EPA 331.0	125mL HDPE	n/a	28 Days
PEST SCREEN 8270D	EPA 8270D	1L Amber Glass	n/a	7 Days
SEMIVOLATILES 625	EPA 625	1L Amber Glass	n/a	7 Days
SVOC 8270D	EPA 8270D	1L Amber Glass	n/a	7 Days
TCLP HERBICIDES	EPA 8151A	1L Amber Glass	n/a	7 Days
TCLP PESTICIDES	EPA 8081A		n/a	14 Days
TCLP SVOC	EPA 8270D	1L Amber Glass	n/a	14 Days
TCLP VOLATILES	EPA 8260B	40mL Amber Vial	HCl	14 Days
TOC/DOC	SM 5310B	40mL Clear Vial	HCl	28 Days
VOLATILE ACIDS GC	EPA 8015D	40mL HCL Vial	HCl	14 Days
VOLATILES 8260	EPA 8260B	40mL HCL Vial	HCl	14 Days
VOLATILES MISC GC/MS	EPA 8260B	40mL HCL Vial	HCl	14 Days
Volatiles Priority Pollutant	EPA 624	40mL HCL Vial	HCl	14 Days
WA Fertilizer Metals	N/A			14 Days

Inorganics - Waste Water

Test	Method	Container	Preservative	Holding Time
ALKALINITY	SM2320B	125mL HDPE	n/a	48 Hours
AMMONIA-NITROGEN	SM4500NH3G	125mL HDPE	H2SO4	28 Days
CARBONATE	SM2320B	125mL HDPE	n/a	48 Hours
CHLORIDE	EPA 300.0	125mL HDPE	n/a	28 Days
CHROMIUM VI (HEXAVALENT)	SM3500CR	1 L Plastic	n/a	28 Days
CN AMENABLE TO CHLORINATION	SM4500CNG	Plastic or glass	NaOH	14 Days
COLOR	SM 2120B	125mL HDPE	n/a	7 Days
CONDUCTIVITY	SM2510B	Plastic or glass	n/a	7 Days
CYANIDE FREE SM 4500 CN-E	SM4500CNE	250mL HDPE	NaOH	14 Days
CYANIDE REACTIVE	SW846 CH7	250mL HDPE	n/a	14 Days
CYANIDE TOTAL EPA	EPA 335.4	250mL HDPE	NaOH	14 Days
CYANIDE WAD	SM4500CNI	250mL HDPE	NaOH	14 Days
DISSOLVED MERCURY-CVAFS	EPA 245.7		HNO3	28 Days
DISSOLVED MERCURY-TRACE	EPA 1631e	PTFE/Glass	HCl / BrCl	180 Days
DISSOLVED METALS - ICP	EPA 200.7/6010B	Plastic or glass	HNO3	28 Days
DISSOLVED METALS ICP-MS	EPA 200.8/6020a	Plastic or glass	HNO3	28 Days
DISSOLVED NITRATE	EPA 300.0		n/a	48 Hours
DISSOLVED NITRATE + NITRITE AS N	EPA 300.0		n/a	48 Hours
DISSOLVED NITRITE	EPA 300.0		n/a	48 Hours
DISSOLVED PHOSPHATE/P FIA	SM4500PF	Plastic or glass	H2SO4	28 Days
DISSOLVED SULFATE	EPA 300.0		n/a	28 Days
DISSOLVED TOTAL P FIA	SM4500PF	Plastic or glass	H2SO4	28 Days
DOC - MOSC	SM 5310B	40mL Clear Vial	HCl	28 Days
FLUORIDE	EPA 300.0	125mL HDPE	n/a	28 Days
FOG - HEM	EPA 1664A	1L Amber Glass	H2SO4 or HCl	28 Days
FOG - NON POLAR	EPA 1664	1L Amber Glass	H2SO4 or HCl	28 Days
GLYCOLS - EG & PG	EPA 8015	G	n/a	14 Days
HARDNESS	EPA 200.7	125mL HDPE	HNO3	180 Days
MERCURY-CV-7471A	EPA 7471A			28 Days
MERCURY-CVAFS	EPA 245.7	40mL Amber Vial	HCl	28 Days
MERCURY-ICPMS	EPA 200.8	125mL HDPE	HNO3	180 Days
MERCURY-TRACE	EPA 1631e	PTFE/Glass	HCl / BrCl	180 Days
METALS ICP	EPA 200.7/6010B	Plastic or glass	HNO3	180 Days
METALS ICP-MS	EPA 200.8	Plastic or glass	HNO3	180 Days
NITRATE/NITRITE FIA	SM 4500 NO3F	125mL HDPE	H2SO4	48 Hours
NITRATE/NITRITE	EPA 300.0	125mL HDPE	n/a	48 Hours
NITRITE - TOTAL N+N FIA	SM 4500 NO3F	125mL HDPE	H2SO4	28 Days
pH	SM 4500pH-B	Plastic or glass	n/a	48 Hours
PHENOLICS TOTAL	EPA 420.1	1L Amber Glass	H2SO4	28 Days
PHOSPHATE - SRP	SM4500PF	Plastic or glass		28 Days
PHOSPHATE/P	EPA 300.0	Plastic or glass	n/a	48 Hours
PHOSPHATE/P FIA	SM4500PF	Plastic or glass	H2SO4	28 Days
SOLIDS - TDS/TSS/TS/TVS/VSS	SM 2540C	Plastic or glass	n/a	7 Days
SOLIDS - FDS	EPA 160.4	1L HDPE	n/a	7 Days
SULFATE	EPA 300.0	Plastic or glass	n/a	28 Days
SULFIDE REACTIVE	SW846 CH7	250ml HDPE	NaOH / ZnAc	7 Days
SURFACTANTS	SM5540C	Plastic or glass	H2SO4	48 Hours
TANNIN & LIGNIN	SM5550B	Plastic or glass	n/a	28 Days
TCLP MERCURY BY 245.7	EPA 245.7			28 Days
TCLP Metals	EPA 6020A	Plastic or glass		14 Days
TCLP METALS	EPA 6020A		n/a	14 Days
TCLP Metals ICP	EPA 6020A	Plastic or glass		14 Days
TKN	SM4500NORGC	125mL HDPE	H2SO4	28 Days
TOTAL 4	N/A	Plastic or glass	HNO3	14 Days
TOTAL P FIA	SM4500PF	125mL HDPE	H2SO4	28 Days
TOTAL VOLATILE ACIDS	SM5560C	Plastic or glass	n/a	14 Days
TURBIDITY	EPA 180.1	Plastic or glass	n/a	48 Hours

Microbiology - Soils/Solids

Test	Method	Container	Preservative	Holding Time
E COLI	SM9221F	Plastic	n/a	24 Hours
FECAL COLIFORMS	SM9221E	Plastic	n/a	24 Hours
HETEROTROPIC PLATE COUNT	SM9215B	Plastic	n/a	24 Hours
TOTAL COLIFORMS	SM9221B	Plastic	n/a	24 Hours

Organics - Soil/Solids

Test	Method	Container	Preservative	Holding Time
ACROLEIN	EPA 8260B	Glass	Cool to 4°C	14 Days
AMINOPYRALID	HPLC/MS/MS	Plastic or glass	Cool to 4°C	10 Days
BTEX 8260	EPA 8260B	Glass	Cool to 4°C	14 Days
CARBAMATE PESTICIDES	EPA 8318	Glass	Cool to 4°C	28 Days
CLOPYRALID	GC/MS/MS	Plastic or glass	Cool to 4°C	28 Days
DIESEL	NWTPH-DX/EPA 8015D	Glass	Cool to 4°C	14 Days
EPA 6850 perchlorate	EPA 331.0	4oz glass jar	Cool to 4°C	14 Days
EPA 8141A	EPA 8141A		Cool to 4°C	7 Days
EPH - WA DOE	WTPHEPH	Glass	Cool to 4°C	14 Days
EXPLOSIVES GC	EPA 8096	Glass	Cool to 4°C	14 Days
EXPLOSIVES HPLC	EPA 8330	Glass	Cool to 4°C	14 Days
GASOLINE	NWTPH-GX/EPA 8015D	Glass	Cool to 4°C	14 Days
GLYPHOSATE IN SOIL	HPLC/MS/MS	Glass	Cool to 4°C	28 Days
HERBICIDES	EPA 8151A	Glass	Cool to 4°C	14 Days
KEROSENE	EPA 8015D		Cool to 4°C	14 Days
METHAMPHETAMINE	HPLC/MS/MS		Cool to 4°C	28 Days
OC PEST 8081A	EPA 8081A	Glass	Cool to 4°C	14 Days
OP PESTS	EPA 8270Dm/EPA 8141	Glass	Cool to 4°C	14 Days
PAH 8270D MOSC	EPA 8270D	1L Amber Glass	Cool to 4°C	7 Days
PCB 8082	EPA 8082	Glass	Cool to 4°C	14 Days
PERCHLORATE 6850	EPA 6850	8 OZ JAR	Cool to 4°C	14 Days
SEMIVOLATILES 625	EPA 625	Glass	Cool to 4°C	7 Days
SEMIVOLATILES MISC GC/FID	GC/FID		Cool to 4°C	7 Days
SEMIVOLATILES MISC GC/MS	EPA 8270CMOD		Cool to 4°C	14 Days
SEMIVOLATILES MISC GC/MS/MS	EPA 8270CMOD		Cool to 4°C	7 Days
SEMIVOLATILES MISC HPLC	HPLC		Cool to 4°C	14 Days
SEMIVOLATILES MISC LC/MS/MS	EPA 8321A		Cool to 4°C	14 Days
SPLP TPHDx	EPA 8015D		Cool to 4°C	14 Days
SVOC 8270D MOSC	EPA 8270D	1L Amber Glass	Cool to 4°C	7 Days
TCLP HERBICIDES	EPA 8151A		Cool to 4°C	14 Days
TCLP PCB	EPA 8082		Cool to 4°C	14 Days
TCLP PESTICIDES	EPA 8081A		Cool to 4°C	14 Days
TCLP SVOC	EPA 8270D		Cool to 4°C	14 Days
TCLP VOLATILES	EPA 8260B		Cool to 4°C	14 Days
VOLATILES 8260	EPA 8260B	Glass	Cool to 4°C	14 Days

Inorganics - Soil/Solids

Test	Method	Container	Preservative	Holding Time
ALKALINITY	SM2320B	Plastic or glass	Cool to 4°C	48 Hours
AMMONIA-NITROGEN	SM4500NH3G	Plastic or glass	Cool to 4°C	28 Days
CATION EXCHANGE CAPACITY	EPA 9081	Plastic or glass	Cool to 4°C	28 Days
CHLORIDE	EPA 300.0	Plastic or glass	Cool to 4°C	28 Days
CN AMENABLE TO CHLORINATION	SM4500CNG	Plastic or glass	Cool to 4°C	14 Days
CONDUCTIVITY	ASTM G57A		Cool to 4°C	7 Days
CYANIDE REACTIVE	SW846 CH7	Plastic or glass	Cool to 4°C	14 Days
CYANIDE TOTAL	EPA 335.4/EPA 9012B	Plastic or glass	Cool to 4°C	14 Days
CYANIDE TOTAL EPA 9012B	EPA 9012B		Cool to 4°C	14 Days
CYANIDE TOTAL SM	SM4500CNE	Plastic or glass	Cool to 4°C	14 Days
CYANIDE WAD	SM4500CNI	Plastic or glass	Cool to 4°C	14 Days
FLUORIDE	EPA 300.0	Plastic	Cool to 4°C	28 Days
FOG - HEM	EPA 1664A	Glass	Cool to 4°C	28 Days
FOG - NON POLAR	EPA 1664	Glass	Cool to 4°C	28 Days
GLYCOLS - EG & PG	EPA 8015	Glass	Cool to 4°C	14 Days
HEX CHROM - SOIL	SM3500 CrD		Cool to 4°C	Days
MERCURY-CV-7471A	EPA 7471A		Cool to 4°C	28 Days
MERCURY-ICPMS	EPA 6020A	Plastic or glass	Cool to 4°C	180 Days
METALS ICP	EPA 200.7/6010B		Cool to 4°C	180 Days
METALS ICP-MS	EPA 6020A	Plastic or glass	Cool to 4°C	180 Days
NITRATE FIA	SM 4500 NO3F	Plastic or glass	Cool to 4°C	48 Hours
NITRATE/N	EPA 300.0	Plastic or glass	Cool to 4°C	48 Hours
NITRATE+ NITRITE AS N	EPA 300.0	Plastic or glass	Cool to 4°C	28 Days
NITRATE+NITRITE FIA	SM 4500 NO3F	Plastic or glass	Cool to 4°C	28 Days
NITRITE FIA	SM 4500 NO3F	Plastic or glass	Cool to 4°C	48 Hours
NITRITE/N	EPA 300.0	Plastic or glass	Cool to 4°C	48 Hours
pH 1:5	EPA 9045	Plastic or glass	Cool to 4°C	14 Days
PHENOLICS TOTAL	EPA9065	Glass	Cool to 4°C	28 Days
PHOSPHATE/P	EPA 300.0	Plastic or glass	Cool to 4°C	48 Hours
PHOSPHATE/P FIA	SM4500PF	Plastic or glass	Cool to 4°C	48 Hours
SOLIDS - TSS/TVS	SM2540D	Plastic or glass	Cool to 4°C	7 Days
SPLP MERCURY BY 245.7	EPA 245.7		Cool to 4°C	28 Days
SPLP Metals	EPA 6020A	Plastic or glass	Cool to 4°C	14 Days
SULFATE	EPA 300.0	Plastic or glass	Cool to 4°C	28 Days
SULFIDE - EXTRACTABLE	EPA 9031		Cool to 4°C	Days
SULFIDE REACTIVE	SW846 CH7		Cool to 4°C	Days
SURFACTANTS	SM5540C	Plastic or glass	Cool to 4°C	48 Hours
SURFACTANTS	SM5540C	Plastic or glass	Cool to 4°C	48 Hours
TCLP 8 METALS	N/A		Cool to 4°C	14 Days
TCLP MERCURY BY 245.7	EPA 245.7		Cool to 4°C	14 Days
TCLP MERCURY BY 6010B	EPA 6010B		Cool to 4°C	180 Days
TCLP Metals	EPA 6020A	Plastic or glass	Cool to 4°C	14 Days
TCLP Metals ICP	EPA 6010B		Cool to 4°C	180 Days
TKN	SM4500NORGC	Plastic or glass	Cool to 4°C	28 Days
TOTAL 4/8	N/A		Cool to 4°C	14 Days
TOTAL P FIA	SM4500PF	Plastic or glass	Cool to 4°C	28 Days
TOTAL SOLIDS	SM2540G		Cool to 4°C	28 Days

Figures

Anatek Labs, Inc.
Organizational Chart

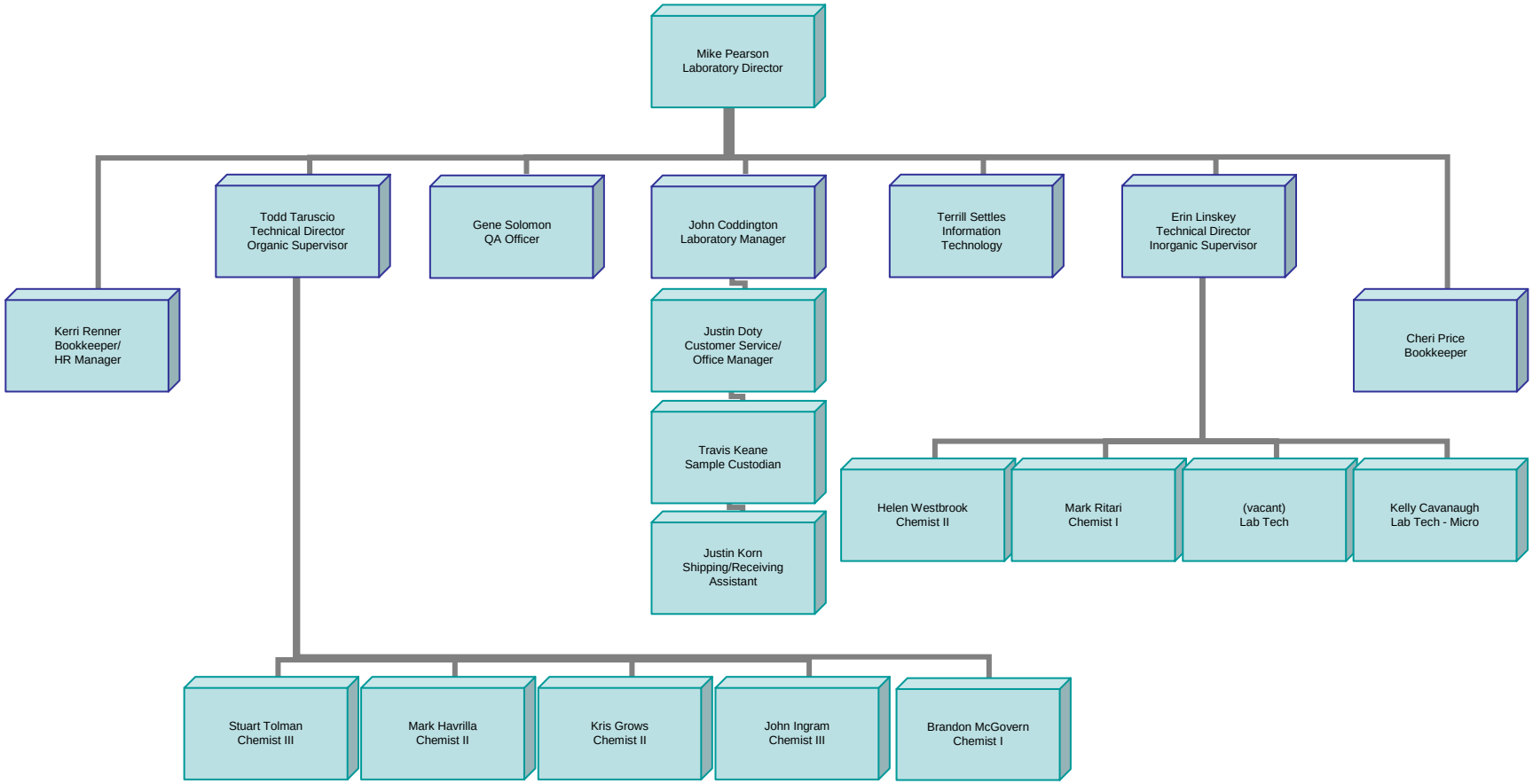
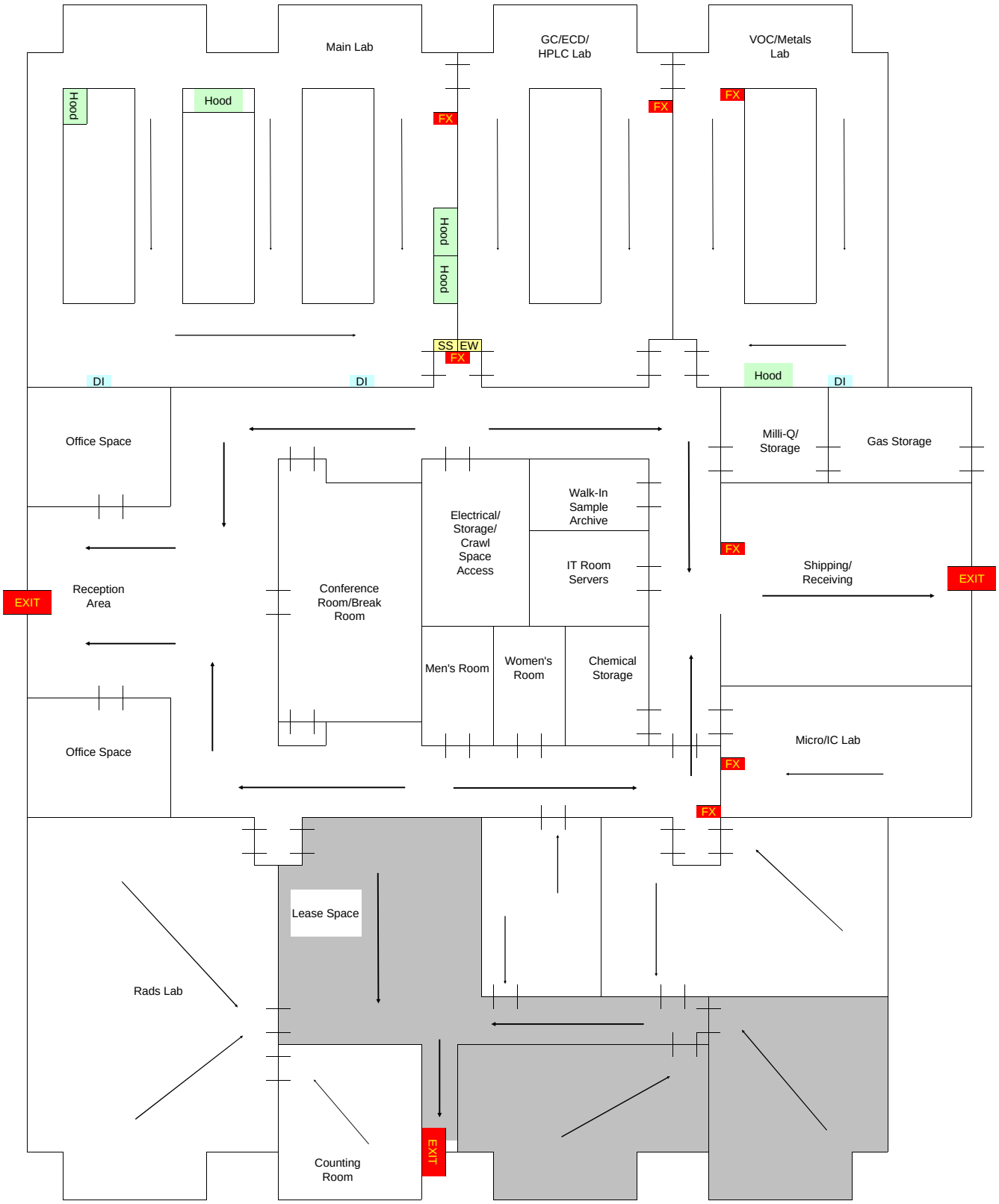


Figure 2

Anatek Labs, Moscow Personnel

Position	Employee	Degree
Laboratory Director	Mike Pearson	B.S Elec. Eng.
Laboratory Manager	John Coddington	Ph.D. Chemistry
Technical Director - Organics Supervisor	Todd Taruscio	Ph.D. Zoophysiology
Technical Director - Inorganics Supervisor	Erin Linskey	B.S. Biology
Chemist III	Stuart Tolman	M.S. Food Science
Chemist III	John Ingram	M.S. Chemistry/MBA
Chemist II	Mark Havrilla	B.S. Chemistry
Chemist II	Kris Grows	B.S. Chemistry
Chemist II	Helen Westbrook	M.S. Chemistry/B.S. Biochemistry
Chemist I	Brandon McGovern	B.S. Science Microbiology
Chemist I	Mark Ritari	B.S. Chemistry
Lab Tech	Kelly Cavanaugh	B.S. Microbiology/Molecular Biology
Lab Tech	Angela Crabtree	B.S. Chemistry/Microbiology
QA Officer	Gene Solomon	B.A. Economics
Customer Service Manager	Justin Doty	
Sample Custodian	Travis Keane	
Shipping/Receiving Asst.	Justin Korn	
IT Manager	Terrill Settles	
Bookkeeper	Cheri Price	
Bookkeeper	Kerri Renner	


Main Lab
GC/ECD/
HPLC Lab
VOC/Metals
Lab
Hood
Hood
FX
FX
FX
Hood
Hood
SS
EW
FX
DI
DI
Hood
DI
Office Space
Milli-Q/
Storage
Gas Storage
Walk-In
Sample
Archive
Electrical/
Storage/
Crawl
Space
Access
IT Room
Servers
FX
Shipping/
Receiving
EXIT
EXIT
Reception
Area
Conference
Room/Break
Room
Men's Room
Women's
Room
Chemical
Storage
Office Space
Micro/IC Lab
FX
FX
Lease Space
Rads Lab
Counting
Room
EXIT

Anatek Labs, Inc.

Figure 4

Anatek Labs, Spokane
Organizational Chart

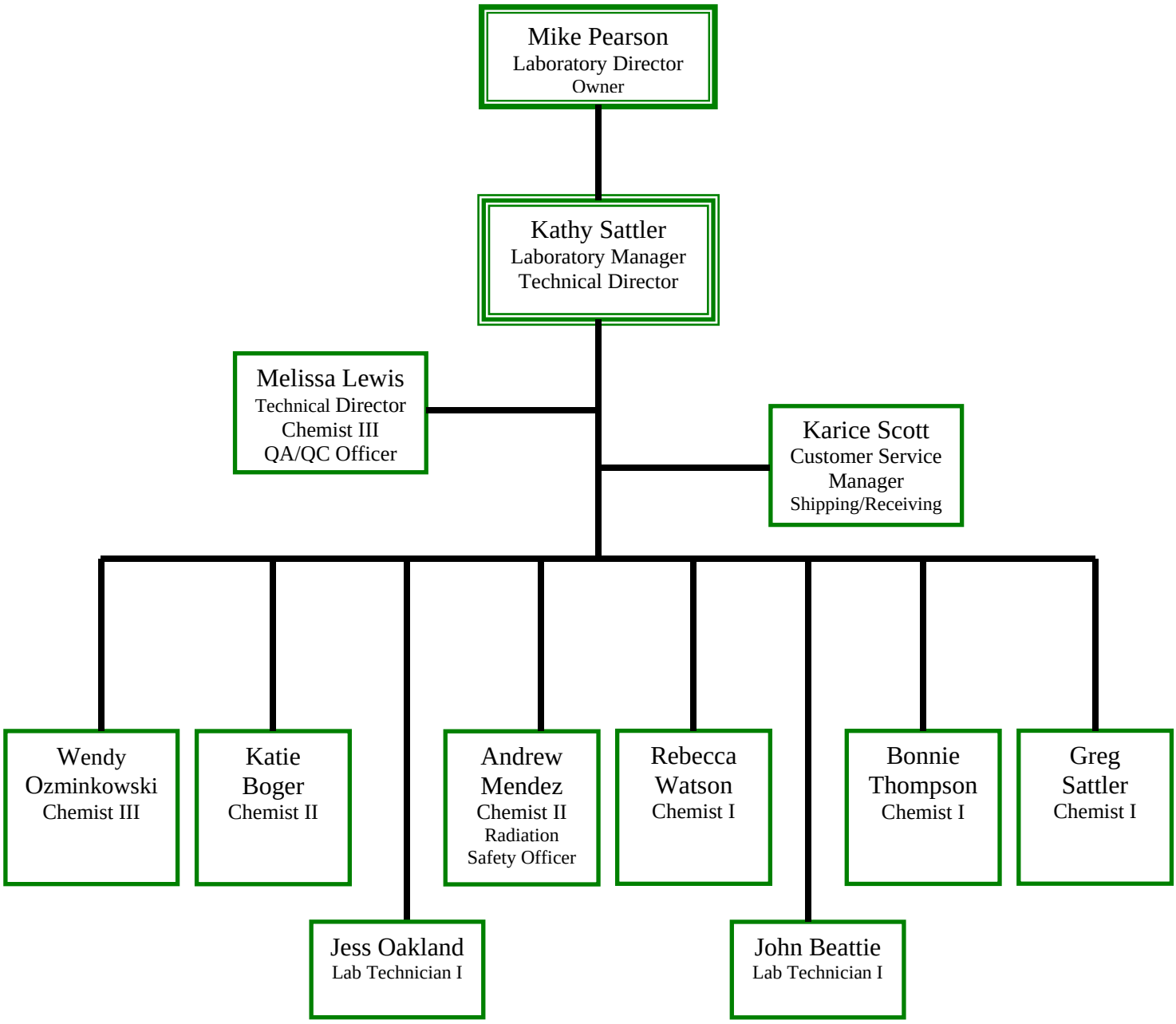


Figure 5

Anatek Labs, Spokane
Personnel

Position	Employee	Degree
Laboratory Director	Mike Pearson	B.S. Electrical Engineering
Laboratory Manager	Kathy Sattler	B.S. Microbiology
QA Officer	Melissa Lewis	B.S. General Studies/Natural Sciences
Chemist III	Wendy Ozminkowski	B.S. Chemistry
Chemist II	Katie Boger	B.S. Chemistry and Biochemistry
Chemist II	Andrew Mendez	B.A. Chemistry
Chemist I	Rebecca Watson	B.S. Biology
Chemist I	Bonnie Thompson	B.S. Biochemistry
Chemist I	Greg Sattler	B.S. Microbiology
Customer Service Manager	Karice Scott	
Lab Technician I	Jess Oakland	
Lab Technician I	John Beattie	

Anatek Labs, Spokane Floor Plan and Safety Plan

Figure 6

Anatek Labs, Inc.

Appendices

Anatek Labs, Inc.

Appendix A

Index of Standard Operating Procedures

Moscow Index is followed by Spokane Index

Anatek Labs, Inc.
Standard Operating Procedures

Laboratory Director:
Mike Pearson

1 Aug 2015

The SOP's contained herein are for the use of Anatek Labs Employees and are not to be removed from the premises without the prior approval of the Lab Director or Lab Manager. Original signed copies are maintained by the QA Officer. Any changes should be submitted to the QA Officer for implementation.

General (00)

ALI-01: Preparing and Maintaining Standard Operating Procedures
ALI-02: Sample Login, Handling and Custody
ALI-03: Glassware Cleaning
ALI-04: Waste Disposal
ALI-05: Data Entry
ALI-06: Complaints
ALI-07: Corrective Action Reports
ALI-08: Standards and Reagents – Labeling, Logging, Storage, and Expiration Dates
ALI-09: Instrument Maintenance and Calibration
ALI-10: Laboratory Safety
ALI-11: Data Handling
ALI-12: Laboratory Blind Samples
ALI-13: Personnel Training Records
ALI-14: Data Archiving
ALI-15: Instrument Activities Logbooks & Laboratory Notebooks
ALI-16: Internal Inspections and Reporting
ALI-17: Procedure for QA Audits of Instrument Activity Logs, IDC's & MDL's
ALI-18: Sample Receiving
ALI-19: Temperature Monitoring and Thermometer Calibrations
ALI-20: No longer in use
ALI-21: Customer Notification
ALI-22: Training
ALI-23: No longer in use
ALI-24: Performing Records Inspections
ALI-25: Performance of IDCs, MDLs, and PQLs
ALI-26: Data Reporting
ALI-27: IT Systems Documentation
ALI-28: QC Acceptance Ranges
ALI-29: PT Reporting
ALI-30: Authorized Signatures
ALI-31: Master List of Quality Systems Documents

Office Manual (00)

ALI-OM-01: Customer Service
ALI-OM-02: Custodial Services
ALI-OM-03: No longer in use
ALI-OM-04: Telephone Systems Procedures
ALI-OM-05: Mail Handling
ALI-OM-06: Purchasing
ALI-OM-07: Shipping
ALI-OM-08: Office Equipment
ALI-OM-09: Accounting
ALI-OM-10: Credit Accounts
ALI-OM-11: Employees
ALI-OM-12: Website Maintenance

Radiochemistry – General (00)

ALI-R-01: Radiation Safety Plan
ALI-R-02: Disposal by Sanitary Sewarage
ALI-R-03: Radiochemical Method Validation

Analytical (000)

Drinking Water (100)

ALI-A-101: EDB/DBCP/1,2,3-TCP Analysis by EPA Method 504.1
ALI-A-102: Haloacetic Acids Analysis by SM 6251B
ALI-A-103: Organochlorine Pesticides and PCB's by EPA Method 505
ALI-A-104: Herbicides Analysis by EPA Method 515.4
ALI-A-105: Volatile Organic Analysis by EPA Method 524.2
ALI-A-106: Semi-volatiles Analysis by EPA Method 525.2
ALI-A-107: Carbamates Analysis by EPA Method 531.2
ALI-A-108: Glyphosate Analysis by EPA Method 547
ALI-A-109: Endothall Analysis by EPA Method 548.1
ALI-A-110: Diquat/Paraquat Analysis by EPA Method 549.2
ALI-A-111: Explosives by GC/MS – EPA Method 529
ALI-A-112: Pesticides and Flame Retardants by GC/MS – EPA Method 527
ALI-A-113: Acetanilide Degradates by LC/MS/MS – EPA Method 535
ALI-A-114: Nitrosamines by GC/MS/MS - EPA Method 521
ALI-A-115: Semi-volatiles Analysis by EPA Method 525.2 – UCMR2 List
ALI-A-116: Volatile Organic Analysis by EPA Method 524.3
ALI-A-117: 1,4-Dioxane by EPA Method 522
ALI-A-118: Perfluorinated Alkyl Acids by EPA Method 537
ALI-A-119: Hormones Analysis by EPA Method 539

Non Drinking Water Organic (200)

ALI-A-201: Pressurized Fluid Extraction By EPA Method 3545
ALI-A-202: No longer used
ALI-A-203: Pesticides/PCB's by EPA Method 608/608.2
ALI-A-204: Carbamates/Urea Pesticide Analysis by HPLC-UV by EPA Method 8321B & EPA Method 632
ALI-A-205: Herbicides by EPA Method 8151A/615
ALI-A-206: Semivolatile Organic Compounds Analysis by GC/MS by EPA Method 625/8270D
ALI-A-207: Volatile Organic Analysis by EPA Method 8260C
ALI-A-208: Volatile Organic Analysis by EPA Method 624

ALI-A-209: Explosives and Explosive By-products by EPA Method 8330B
ALI-A-210: No longer used
ALI-A-211: Organophosphorus Pesticide Analysis by GC/MS by EPA Method 8141B Modified/614/614.1
ALI-A-212: Pesticides/PCB's by EPA Method 8081B/8082A
ALI-A-213: Soil Herbicides by EPA Method 8151A Modified
ALI-A-214: No longer used
ALI-A-215: See SOP ALI-A-340
ALI-A-216: Volatile Organic Analysis by EPA Method 8260B
ALI-A-217: Semivolatile Organic Compounds Analysis by EPA Method 8270C
ALI-A-218: Triclopyr and 2,4-D by HPLC/MS/MS by EPA Method 8321A
ALI-A-219: Closed Purge-and-Trap Extraction for VOCs by EPA Method 5035A
ALI-A-220: Continuous Liquid-Liquid Extraction by EPA Method 3520C

Inorganic and Wet Chemistry (300)

ALI-A-301: Orthophosphate/Total Phosphorus (SM4500P-F/EPA 365.4) Flow Injection Analysis
ALI-A-302: No longer in use
ALI-A-303: Phenolics by Manual Colorimetry by EPA Method 420.1/SM5530C/EPA 9065
ALI-A-304: Ammonia Nitrogen (SM4500NH3-G/EPA 350.1) and TKN (SM4500NorgC) by Flow Injection Analysis
ALI-A-305: Cation Exchange Capacity of Soils by EPA Method 9081
ALI-A-306: Residual Chlorine by SM 4500Cl-G
ALI-A-307: No longer in use
ALI-A-308: Cyanide (Total, Amenable, and Weak Acid Dissociable) by Semi-Automated Colorimetry SM 4500-CN^-E-G-I
ALI-A-309: Total Cyanide by Semi-Automated Colorimetry by EPA Method 335.4/9012B
ALI-A-310: pH by EPA Method 150.1 & EPA Method 9045C & SM4500 H^+B
ALI-A-311: Alkalinity by EPA Method 310.1/SM 2320B
ALI-A-312: Conductivity by EPA Method 120.1/SM 2510B
ALI-A-313: Hardness by SM 2340B
ALI-A-314: Trace Metal Analysis by EPA Method 200.8
ALI-A-315: Trace Metal Analysis by EPA Method 6020A
ALI-A-316: Ultra Trace Metal Analysis by EPA Method 1638
ALI-A-317: No longer in use
ALI-A-318: Trace Mercury Analysis by EPA Method 1631/245.7/7471A
ALI-A-319: Nitrate/N and Nitrite/N (SM4500NO3-F & EPA 353.2) Flow Injection Analysis
ALI-A-320: Ions (Nitrate, Nitrite, Chloride, Sulfate, Fluoride, Phosphate) By EPA Method 300.0
ALI-A-321: Chlorate Analysis by EPA Method 300.1
ALI-A-322: Turbidity by EPA Method 180.1
ALI-A-323: TSS by SM 2540-D and TDS by SM 2540-C
ALI-A-324: Color (Platinum-Cobalt Method) by SM 2120B
ALI-A-325: Total Volatile Solids by EPA Method 160.4/SM 2540E & Total Fixed Solids (%Ash) by EPA Method 1684
ALI-A-326: Total Volatile Acids by SM 5560 C
ALI-A-327: Anionic Surfactants by Method 5540 C
ALI-A-328: Tannin and Lignin by SM 5550 B
ALI-A-329: No longer in use
ALI-A-330: Toxicity Characteristic Leaching Procedure by SW 846 Method 1311
ALI-A-331: No longer in use
ALI-A-332: Sulfide (SM4500-S2 F) Iodometric Titration
ALI-A-333: Ammonia Nitrogen by SM4500 NH3-G (Discrete Analyzer)
ALI-A-334: Low Level Orthophosphate/Total P by SM4500P-F (Discrete Analyzer)
ALI-A-335: Mid Range Orthophosphate/Total P by SM4500P-F (Discrete Analyzer)

ALI-A-336: TKN by SM4500NorgC (Discrete Analyzer)
ALI-A-337: Synthetic Precipitation Leaching Procedure by SW-846 Method 1312
ALI-A-338: Trace Metals Analysis by EPA Method 200.7
ALI-A-339: Trace Metals Analysis by EPA Method 6010B
ALI-A-340: Perchlorate by EPA Method 331.0 (HPLC/ESI/MS)
ALI-A-341: Perchlorate by EPA Method 6850
ALI-A-342: TOC/DOC by SM5310B
ALI-A-343: Trace Metals Analysis by EPA Method 200.8 – UCMR3 Analysis
ALI-A-344: Acid Digestion of Sediments, Sludges, and Soils by EPA Method 3050B
ALI-A-345: Odor by SM 2150B

UST Petroleum Methods (400)

ALI-A-401 No longer used
ALI-A-402: No longer used
ALI-A-403: No longer used
ALI-A-404: Gasoline Analysis by EPA Method 8015 (Modified)/NW TPHG(X)
ALI-A-405: TPH-D & HCID-NW TPH-D & NW TPH-HCID – EPA 8015D
ALI-A-406: No longer used
ALI-A-407: Hexane Extractable Material (FOG) by EPA Method 1664B
ALI-A-408: Extractable Petroleum Hydrocarbons (EPH) Massachusetts Method) and Diesel Range Organics (DRO)
ALI-A-409: No longer used
ALI-A-410: No longer used
ALI-A-411: Flashpoint by EPA Method 1010
ALI-A-412: C_{10}-C_{32} Hydrocarbons in Soil by 8015AZ
ALI-A-413: Glycols by EPA 8015D

Coliform and Bacteria (500)

ALI-A-501: SM 9223B-MPN (Quanti-tray) Procedure
ALI-A-502: SM 9223B-PA Procedure
ALI-A-503: Heterotrophic Plate Count by Method 9215 B
ALI-A-504: No longer used
ALI-A-505: SM 9223E-MPN Fecal Coliform and E. coli Count by Multiple Tube Fermentation
ALI-A-506: SM9221B-E-F-MPN Total Coliform, Fecal Coliform, and E. coli Count by Multiple Tube Fermentation

Special (600)

ALI-A-601: Quantitation of Clopyralid in Finished Compost
ALI-A-602: Determination of Methamphetamine from Wipe Samples and Other Matrices
ALI-A-603: Glyphosate and AMPA in Soil & Solids

Radiochemistry - Analytical (700)

ALI-A-701: Gross Alpha-Beta Radioactivity by EPA Method 900.0
ALI-A-702: Radium-226 by EPA Method 903.1
ALI-A-703: Radium-228 by EPA Method 904.0
ALI-A-704: Wipe Testing for Gross Alpha-Beta Radioactivity

Anatek Labs, Inc.
Standard Operating Procedures

Laboratory Director: Mike Pearson
Laboratory Manager: Kathy Sattler

Revised October 15, 2012

The SOP's contained herein are for the use of Anatek Labs employees and are not to be removed from the premises without the prior approval of the Lab Director or Lab Manager. Original signed copies are maintained by the QA Officer. Any changes should be submitted to the QA Officer for implementation.

Office Manual (00)

ALI-OM-01.01	Customer Service
ALI-OM-02.01	Custodial Services
ALI-OM-03.01	Sample Handling Procedures
ALI-OM-04.01	Telephone Systems Procedures
ALI-OM-05.01	Mail Handling
ALI-OM-06.01	Purchasing
ALI-OM-07.01	Shipping
ALI-OM-08.01	Office Equipment
ALI-OM-09.01	Accounting
ALI-OM-10.01	Credit Accounts
ALI-OM-11.01	Employees

General (00)

ALI-01 .02	Preparing and Maintaining Standard Operating Procedures
ALI-02 .03	Sample Login, Handling, and Custody
ALI-03 .01	Glassware Cleaning
ALI-04	Waste Disposal
ALI-05	Data Entry Guidelines
ALI-06	Complaints
ALI-07 .02	Corrective Action Reports
ALI-08	Labeling, Logging and Storage of Standards and Commercial Reagents/Solvents
ALI-09	Instrument Maintenance and Calibrations
ALI-10	Chemical Hygiene Plan
ALI-11	Data Handling

General (00) cont.

ALI-12	Laboratory Blind Sample
ALI-13	Personnel Training Records
ALI-14	Data Archiving
ALI-15	Instrument Activities Logbook
ALI-16	Internal Inspections and Reporting
ALI-17	Procedure for QA Audits of Instrument Activity Logs, IDC's and MDL's
ALI-18	Sample Receiving
ALI-19	Calibration and Monitoring of Thermometers
ALI-20	IDEXX Bottle Volume and Sterility Test
ALI-21	Customer Notification
ALI-22	Training
ALI-23	Shipping and Receiving
ALI-24	Performing Records Inspections
ALI-25.00	Performance of IDCs, MDLs, and PQLs
ALI-26.00	Data Reporting – pending
ALI-27.00	IT Systems Documentation
ALI-28.00	Control Charting – pending
ALI-29.00	PT Reporting – pending
ALI-30.00	Authorized Signatures

Analytical (000)

Organics (100)

ALI-S-101	Volatile Organic Analysis by EPA Method 524.2
ALI-S-102	Volatile Organic Analysis by EPA Method 624.4
ALI-S-103	Volatile Organic Analysis by EPA Method 8260B
ALI-S-104	Total Organic Carbon by SM 5310C
ALI-S-105	Total Organic Halides by EPA Method 9020B
ALI-S-106	Extractable Organic Halides by EPA Method 9023
ALI-S-107	Haloacetic Acids by SM 6251B

Inorganic and Wet Chemistry (200)

ALI-S-201	Total Phosphorus by SM4500-PF
ALI-S-202	Total Residual Chloride by SM4500Cl-G
ALI-S-203	Total Cyanide by SM 4500CN-F
ALI-S-204	pH by EPA Method 150.1
ALI-S-205	Alkalinity (Carbonate & Bicarbonate) by SM 2320 B/EPA 310.1
ALI-S-206	Conductivity by SM 2510 B
ALI-S-207	Hardness by SM 2340 B

Inorganic and Wet Chemistry (200) cont.

ALI-S-208	Anions (NO_3, NO_2, SO_4, Cl, F, PO_4, Br) by EPA Method 300.0
ALI-S-209	Ions (Bromate, Chlorate, Chlorite) by EPA Method 300.0 Part B (Currently not used)
ALI-S-210	Turbidity by EPA Method 180.1
ALI-S-211	TSS (Total Suspended Solids) by EPA Method 160.2/SM 2540 D
ALI-S-212	TDS (Total Dissolved Solids) by EPA Method 160.1/SM 2540 C
ALI-S-213	TS (Total Solids) by EPA Method 160.3
ALI-S-214	BOD/DO/CBOD by SM 5210 B
ALI-S-215	COD by EPA Method 410.4
ALI-S-216	Percent Solids
ALI-S-217	pH (non-aqueous) by EPA Method 9045D
ALI-S-218	Color by SM 2120 B
ALI-S-219	Ammonia Nitrogen by SM 4500 NH3-G
ALI-S-220	Ortho-phosphate by SM 4500-PF
ALI-S-221	Sulfide by SM4500-S^{2-}F
ALI-S-222	Acidity by EPA 310.1 / SM2310-B
ALI-S-223	Sulfite by SM4500-SO_3^{2-}B
ALI-S-224	pH (in Soil and Waste) by EPA Method 9040C
ALI-S-225	Resistivity by ASTMG57A
ALI-S-226	Metals by EPA 200.8
ALI-S-227	Metals by EPA 6020
ALI-S-228	TKN by SM 4500N_{ORG} C
ALI-S-229	Specific Gravity/Density by
ALI-S-230	Chlorine Demand by SM
ALI-S-231	MBAS by SM 5540C
ALI-S-232	Carbon Dioxide
ALI-S-233	Sulfide by SM4500-S^{2-}D
ALI-S-234	CN in DW by SM 4500 CN F / EPA 335.4
ALI-S-235	Total CN by SM 4500 CN N / EPA 335.4

Hazardous Waste/Waste Oil (300)

ALI-S-301	PCB's by EPA Method 8082
ALI-S-302	BTU – Heat of Combustion
ALI-S-303	Total Chlorine by EPA Method 9076

UST Petroleum Methods (400)

ALI-S-401	Gasoline Analysis by NW TPHG(x)
ALI-S-402	BTEX by EPA Method 8021 (Modified)
ALI-S-403	VPH by MADEP-VPH-98-1
ALI-S-404	VPH by WA VPH Method
ALI-S-405	Oxyfuel Analysis by ASTM D 4815
ALI-S-406	Diesel and Lube Oil Analysis by NW-TPHDx & HCID
ALI-S-407	Gasoline Range Organics (GRO) by EPA Method 8015B

Microbiology (500)

ALI-S-501	Total and E. coli by SM 9223B-PA Quanti-Tray
ALI-S-502	Total and E. coli by SM 9223B-PA
ALI-S-503	Archived
ALI-S-504	Multi-tube MPN Total and Fecal coliform SM 9221 B
ALI-S-505	Multi-tube MPN Fecal and E.coli SM 9221 E
ALI-S-506	Multi-tube MPN Fecal and E.coli in soil SM 9221 E
ALI-S-507	Heterotrophic Plate Count by SM 9215 B
ALI-S-508	HPC by SimPlate (unit dose) SM 9221 B
ALI-S-509	HPC by SimPlate (unit dose) SM 9221 B
ALI-S-510	Fecal Coliform Membrane Filtration by SM 9222D
ALI-S-511	Fecal in Biosolids by EPA 1680
ALI-S-512	Salmonella in Biosolids by EPA 1682
ALI-S-513	Fecal Streptococcus and Enterococcus by Multi-Tube Fermentation SM 9230 B

Radionuclides (600)

ALI-S-601	Gross Alpha and Gross Beta by EPA 900.0

Appendix B

Example of Sample Submission Form

Anatek Labs, Inc.

Chain of Custody Record

1282 Alturas Drive, Moscow ID 83843 (208) 883-2839 FAX 882-9246
504 E Sprague Ste D, Spokane WA 99202 (509) 838-3999 FAX 838-4433

Anatek
Log-In #

Company Name:	Project Manager:
Address:	Project Name & # :
City: State: Zip:	Email Address :
Phone:	Purchase Order #:
Fax:	Sampler Name & phone:

Turn Around Time & Reporting

Please refer to our normal turn around times at:
http://www.anateklabs.com/services/guidelines/reporting.asp

__Normal
__Next Day*
__2nd Day*
__Other*_____

*All rush order requests must be prior approved.

__Phone
__Mail
__Fax
__Email

Provide Sample Description / List Analyses Requested

Lab ID	Sample Identification	Sampling Date/Time	Matrix	# of Containers	Sample Volume	Preservative:									

Note Special Instructions/Comments

Inspection Checklist

Received Intact?	Y	N
Labels & Chain Agree?	Y	N
Containers Sealed?	Y	N
VOC Head Space?	Y	N

Temperature (C): _____

Preservative: _____

Inspected By: _____

Date & Time:

	Printed Name	Signature	Company	Date	Time
Relinquished by					
Received by					
Relinquished by					
Received by					
Relinquished by					
Received by					

Appendix C

Quick Reference for Chemical Safety

ACIDS	Hazards	First Aid (Skin)	Fire-Fighting	Spillage
Acetic Acid (CH_3COOH)	Flammable, corrosive	Water spray	CO_2, Powder	Neutralize with weak base
Hydrochloric acid (HCl) (concentrated)	Not combustible, corrosive	Rinse with plenty of water	Any extinguishing agent	Report to supervisor
Hydrochloric acid (HCl) (diluted, 50%<)	Corrosive, not combustible	Rinse with plenty of water	Any extinguishing agent	Neutralize with weak base
Hydrofluoric Acid (HF) (concentrated)	Corrosive, not combustible	Rinse with plenty of water	NO hydrous agent	Report to supervisor
Hydrofluoric Acid (HF) (diluted 30%<)	Corrosive, not combustible	Rinse with plenty of water	NO hydrous agent	Neutralize with weak base
Nitric Acid (HNO_3) (concentrated)	Corrosive, not combustible	Rinse with plenty of water	NO FOAM	Report to supervisor
Nitric Acid (HNO_3) (diluted, 50%<)	Corrosive, not combustible	Rinse with plenty of water	NO FOAM	Neutralize with weak base
Phosphoric Acid (H_3PO_4) (diluted or concentrated)	Corrosive, not combustible	Rinse with plenty of water	Any extinguishing agent	Neutralize with weak base
Sulfuric Acid (H_2SO_4) (diluted or concentrated)	Corrosive, not combustible	Rinse with plenty of water	Powder, CO_2, NO WATER	Neutralize with weak base
BASES				
Ammonium Hydroxide (NH_4OH)	Corrosive, not combustible	Rinse with plenty of water	Any extinguishing agent	Neutralize with weak acid

(diluted)				
Potassium Hydroxide (KOH) (diluted)	Corrosive, not combustible	Rinse with plenty of water	Any extinguishing agent	Neutralize with weak acid
Sodium Hydroxide (NaOH) (diluted)	Corrosive, not combustible	Rinse with plenty of water	Any extinguishing agent	Neutralize with weak acid
SOLVENTS	**Hazards**	**First Aid (Skin)**	**Fire-Fighting**	**Spillage**
Acetone	Highly flammable	Rinse with water	CO_2, Powder	Ventilate, Collect in container
Acetonitrile	Flammable	Rinse with plenty of water	CO_2, Powder	Ventilate, Collect in container
Carbon Disulfide	Highly flammable	Rinse with plenty of water	CO_2, Powder	Evacuate, report to supervisor
Chloroform	Drowsiness, not combustible	Rinse with plenty of water	Any extinguishing agent	Evacuate, Collect in container
Diethyl Ether	Extremely flammable, drowsiness	Rinse with water	CO_2, Powder	Evacuate, Collect in container
Ethanol	Highly flammable	Rinse with water	CO_2, Powder, Water	Collect in container
Ethyl Acetate	Drowsiness, Highly flammable	Rinse with water	CO_2, Powder	Evacuate, Collect in container
Ethylene Glycol	Combustible	Rinse with water	CO_2, Powder	Collect in container
Hexane	Drowsiness, Highly flammable	Rinse with water	CO_2, Powder	Ventilate, Collect in container
Hydrogen Peroxide	Corrosive, not combustible	Rinse with plenty of water	Water	Ventilate, wash with plenty of water
Isooctane	Drowsiness, Highly flammable	Rinse with water	CO_2, Powder	Evacuate, Collect in container

Methanol	Highly flammable	Rinse with water	CO_2, Powder, Water	Evacuate, Collect in container
Methylene Chloride	Drowsiness, combustible	Rinse with water	Any extinguishing agent	Ventilate, Collect in container
MTBE	Highly flammable	Rinse with water	CO_2, Powder	Ventilate, Collect in container
THF	Highly flammable	Rinse with water	CO_2, Powder, Water	Ventilate, Collect in container
Toluene	Highly flammable	Rinse with water	CO_2, Powder	Collect in container

Appendix D

Laboratory Management and Staff CVs/Qualifications

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Appendix E

Current State Certifications
Anatek - Moscow

Florida Department of Health (NELAP # E87893)
(Primary Accrediting Authority for NELAP)
Idaho Department of Health (EPA ID00013)
Washington Department of Ecology (C595)
Oregon Department of Environmental Quality (ORELAP # ID200001)
Nevada Department of Conservation and Natural Resources (ID00013)
Montana Department of Public Health and Human Services (CERT0028)
New Mexico Environment Department, Drinking Water Bureau (ID00013)
Arizona Department of Health Services (AZ0701)
EPA Region 8/Wyoming (EPA ID00013)

Current State Certifications
Anatek - Spokane

Florida Department of Health (NELAP # E871099)
(Primary Accrediting Authority for NELAP)
Idaho Department of Health (EPA WA00169)
Washington Department of Ecology (C585)
Montana Department of Public Health and Human Services (CERT0095)
Washington State Liquor Control Board (Cert# 0010)

Current scopes of accreditation are available at
www.anateklabs.com/certifications

Anatek Labs, Inc.

Appendix F

Backup, Fault Tolerance, Disaster Recovery and Data Archive of Mission-critical Information Storage and Services

Terrill Settles
Information Systems Manager

Updated 8/12/2015

Introduction

Mission-critical information storage and services are those that a business cannot afford to lose. Loss of such data or interruption of such services will seriously impact the daily operations of the business and incur monetary loss.

Fault Tolerance

Fault tolerance in data storage involves redundant storage disks to tolerate certain faults in the hardware. For example, in RAID 5 implementation, in case of failure of one disk, the remaining disks in the array still maintain the data. But with the loss of a disk the array is in a critical state and its performance is greatly reduced until the failed disk is replaced and the array is rebuilt.

Fault tolerance only tolerates hardware faults. It does not cover human or application software faults. For example, accidental or intentional deletion by operators or file damage caused by misbehaving applications is not covered by fault tolerance. So fault tolerance does not replace backups and the archiving of the data.

Backup

Backup is the process of copying data to other media creating an extra copy. The media can be copied to hard disk drives, tape drives, USB drives, CD and DVD disks, and even to the internet "cloud." In case of loss of or damage to current files, if they were backed up, and the media is available, the data can be restored. Backup usually occurs during off-peak hours, normally nights and weekends.

Backups generally cannot copy certain open system files and database files such as SQL databases and Exchange Information Store. However, by using the built-in backup tools within these programs a backup can be created to a file or folder. After these backups have occurred this data can now be backed up to another media.

System Disaster Backup and Recovery

A system disaster is an incident that causes the loss of the server operating system and other data due to hardware or software failure. If set up correctly the downtime due to system disaster can be minimized due to a well-planned, and rehearsed, recovery model.

Normal data backup works well for regular data files but does not back up the system files. The system files are used by the server's operating system. In case of loss of the server system such as an operating system crash, the server cannot be restored the same as restoring normal data files. In a disaster recovery scenario the operating system will need to be installed, then any additional programs will need to be installed. Finally, any configurations and data files will need to be restored as well.

A disaster recovery plan involves recognizing and documenting what programs are installed on the system, backing up of the system files and other programs configuration files, and having available a recovery plan and the backups of the files.

A backup of the system has to be made during a downtime made available for system maintenance. There are different possibilities backing up of the system files. One could be an image of the server created while off-line. Backups can also be accomplished by backing up the system files using other manufacturers' offline backup programs.

Data Archiving

Infrequently used older data should be archived onto other media. Data archiving serves dual purposes. It frees online disk storage, and, if it is still necessary to put a copy online, the archive provides a backup.

Identification of mission-critical information storage and services

Analytical data and results acquired by the instruments:

The analytical data resides on a server with RAID 5 fault tolerance. Current and recent data files are in a share folder called "AnatekData." Archived data is copied to CD/DVD ROMs and is available in IT and in off-site storage. These files are backed up nightly Monday through Friday, and again on Sunday night.

SQL Server running on Windows server:

SQL server databases for LIMS and other miscellaneous databases: LIMS databases provide data entry, storage and reporting. Databases and their logs reside on RAID drives. Further, database files and log files reside on separate disk spindles for maximum recoverability in case of disk failure. SQL provides its own backup nightly -- the files are also backed up nightly Monday through Friday, and again on Sunday night.

Exchange Server running on Windows server:

Microsoft Exchange Provides e-mail, tasking, scheduling and other collaborating functions to the company. The Exchange data is located on a RAID drive. Exchange provides its own backup nightly – the files are also backed up nightly Monday through Friday, and again on Sunday night.

Windows Domain Controller Servers:

These servers provide Active Directory services which includes user authentication, security, and sharing functions for the domain. Active Directory services provide the platform on which SQL, Exchange, FTP, IIS, and other services reside.

Standard Windows File Server:

Provide general file service for user shares.

IIS on Windows Server:

Provides a web service for Internet, Intranet and Extranet.

QuickBooks files on Windows File Server:

Stores all accounting information of the company, including banking, purchasing, receivable, payable and payroll. These files are stored on the RAID disk of Windows file server.

Data security/integrity implementations

Fault Tolerance

Each Windows Server has been configured with RAID 5 or RAID 10 for storage of critical data files. All disks are hot swappable. The domain controllers have redundant power supplies.

Backup

The backup software currently in use is Macrium Reflect and the Windows NTbackup utility. Backup hardware is a removable hard disk drive for disk-to-disk backup. Macrium Reflect runs on server Treasure copying the following files: Analysis Data from server Treasure and other Instrument PCs; SQL and FTP backups from server Bobwhite; department and user personal files from Grouse; Quickbook and Payclock backup files from PC Moonstone; and Quickbook files from Magpie. Data backups on Friday run a full backup. Data backups Sunday through Thursday run incremental backups.

Exchange runs its own backup and places the backup files on server Bobwhite in folder "ExchangeBackup."

Quickbooks backups are copied from server Magpie in the folder "Quickbooks." PC Moonstone also runs a windows backup to copy the files every Sunday through Friday at 9AM.

SQL runs its own backups and they occur Sunday through Thursday at 1AM and are placed on server Bobwhite in folder "SQL2K8\Backup".

Disaster recovery

We have Symantec Ghost 7.5 as an imaging tool. The entire hard disks of the Windows servers and majority of workstations have been taken as a snapshot. The images of those disks were either burned onto DVD disks or storage on removable drives.

As imaging the servers requires taking them off-line, the imaging is not a regularly scheduled task. However, when major changes are made to servers, re-imaging should be done once the servers have been tested to be running satisfactorily.

Workstation imaging is updated when there is major configuration change in hardware or software.

Data archiving

The older data files on the analytical data storage server (older than 6 or 12 months, as dictated by data volume) are archived to CD/DVD at intervals of 6 or 12 months, or when deemed necessary, for permanent archival. The permanent archive has two copies, one kept on-site and another off-site. The DVD/CD disks, if properly stored, should have a lifetime of at least 50 years. A few randomly selected DVD/CD disks are checked for readability annually to ensure the availability of older data files.

A copy of the archived data files is kept available for read-only, as long as the storage server has enough space for them. When the space approaches depletion, the oldest files will be purged.

The shared location for the older data is secured so only system administrators can change them. All regular users have read-only permission.

Summary

We have hardware redundancy to protect against disk failure for the most important data. Our backup scheme is disk-to-disk technology and CD/DVD archival. The disk backups are on a four week off-site rotation. The servers and majority of workstations are imaged for quick disaster recovery. Data archiving is done on a regular basis and two permanent copies of archived data are kept.

The locations for the older data are secured so only system administrators can change them. All regular users have read only permission.

Summary

We have hardware redundancy to protect against disk failure for the most important data. Backup scheme is a combination of D2D and tape technology, which offers both off-site backup media storage and fast restore. The tape backup is up to one week old. The servers and majority of workstations are imaged for quick disaster recovery. Data archiving is done on a regular basis and two permanent copies of archived data are kept.

Appendix G

Control Chart Information

Control Charting

Control charting is a useful way to determine accuracy and precision data for specific repeated recovery calculations (surrogates, LFBs, CCVs, etc.). It is most useful to calculate acceptance criteria from the most recent data, and allows comparison to written method requirements if they exist.

At minimum, control charts must be made for control standards. For methods that require the addition of surrogate compounds, control charts are also required for the surrogate recoveries.

<u>Definitions:</u> Let X_1, X_2, X_3, X_4,.....X_n ($n \geq 20$) represent the first n time ordered determinations for an analyte, and then define the following:

$$X = \text{average} = \frac{1}{n}\,(X_1 + X_2 + X_3 + \ldots\ldots\ldots\ldots + X_n)$$

$$S = \text{Standard Deviation of the Group} = \left[\frac{\Sigma(X_n - X)^2}{n-1}\right]^{\frac{1}{2}}$$

Based on the average and standard deviation information of this n number of trials a control chart can be plotted using the formulas outlined in Table 1. An example of a control chart is shown in Figure 1 with X = 99 % and S = 4 %. Such plot can then be used to determine if one or a set of trial is out of control.

Table 1: Control Chart Formula.

Parameter	**Symbol**	**Formula**
Centerline	CL	X
Upper Control Limit	UCL	X + 3S
Lower Control Limit	LCL	X – 3S
Upper Warning Limit	UWL	X + 2S
Lower Warning Limit	LWL	X – 2S

<u>Criteria for an Out-of-Control Situation</u>

A process is considered out of statistical control whenever one of the following conditions is demonstrated from control charting.

a. Any one point is outside of the control limits.

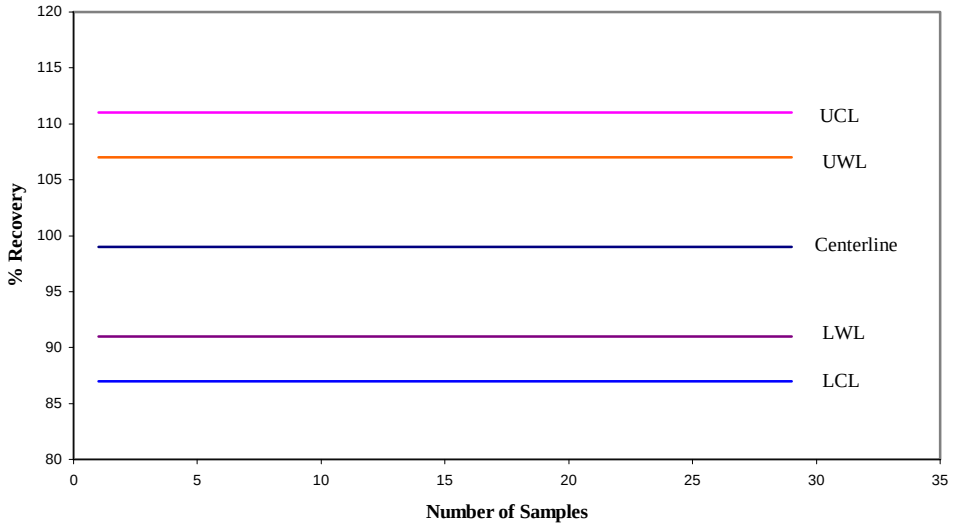
b. Any three consecutive points are outside the warning limits.

c. Any ten consecutive points are on the same side of the centerline.

d. Any six consecutive points are such that each deviation is greater than its predecessor.

e. Any obvious cyclic pattern is seen in the points.

Figure 1: A sample control chart.



Corrective Action

When a process is out of control as determined by control chart monitoring, an immediate solution must be found before processing more samples. An example might be the slow deterioration of the PID lamp, which might cause recoveries to slowly decrease. This problem may easily be remedied by more frequent cleaning or perhaps more frequent calibration.

Appendix H

Method Detection Limit

USEPA DEFINITION AND METHOD FOR MDL

From: 40 CFR (7–1–95 Edition) Part 136, Appendix B

APPENDIX B TO PART 136 — DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT — REVISION 1.11

Definition

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 and is determined from analysis of a sample in a given matrix containing the analyte.

Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit. The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample. The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

Procedure

1. Make an estimate of the detection limit using one of the following:
(a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve. (d) Instrumental limitations. It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The

interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4. If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit. If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

> (1) Obtain another sample with a lower level of analyte in the same matrix if possible.
>
> (2) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data: (1) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL. (2) If these measurements indicate the sample is not in correct range, reestimate the MDL, obtain new sample as in

3 and repeat either 4a or 4b.

5. Calculate the variance (S2) and standard deviation (S) of the replicate measurements, as follows:

$$s^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} x_i^2 - \left(\frac{\left(\sum_{i=1}^{n} x_i\right)^2}{n}\right)\right]$$

$$s = (s^2)^{1/2}$$

where: Xi; i = 1 to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and
S refers to the sum of the X values from i = 1 to n.

6. (a) Compute the MDL as follows:

MDL = t(n-1, 1-alpha = 0.99) (S)

where: MDL = the method detection limit
t(n-1, 1-alpha = .99) = the students' t value appropriate for a 99% confidence level and alpha standard deviation estimate with n-1 degrees of freedom. See Table.
S = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (X^2/df).

LCL = 0.64 MDL
UCL = 2.20 MDL

where: LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL and proceed through the procedure starting with Step 4.

(b) If this is the second or later iteration of the MDL calculation, use S^2 from the current MDL calculation and S^2 from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger S^2 into the numerator S^2_A and the other into

the denominator S^2_B. The computed F-ratio is then compared with the F-ratio found in the table which is 3.05 as follows: if $S^2_A / S^2_B < 3.05$, then compute the pooled standard deviation by the following equation:

$$S_{pooled} = [(6S^2_A = 6S^2_B) / 12]^{½}$$

if $S^2_A / S^2_B > 3.05$, respike at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c) Use the Spooled as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681 (S_{pooled})$$

where 2.681 is equal to t(12, 1-alpha = .99).

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

LCL = 0.72 MDL
UCL = 1.65 MDL

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLES OF STUDENTS' t VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Number of replicates	Degrees of freedom (n-1)	t(n-1, .99)
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
00	00	2.326

Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Re-port the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery. If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL. [49 FR 43430, Oct. 26, 1984; 50 FR 694, 696, Jan. 4, 1985, as amended at 51 FR 23703, June 30, 1986]

Interpretation of Data Index
Pesticide National Synthesis Project Home Page
Contact: Robert J. Gilliom, rgilliom@usgs.gov
Maintained: Tamara Shelton, tshelton@usgs.gov
URL: ***http://ca.water.usgs.gov/pnsp/rep/interpret/.html***
Last Modified: Thu Feb 17 2000

Appendix I

CALIBRATION METHODS AND EQUATIONS

This chapter provides an overview of calibration models and equations commonly used by methods performed at Anatek Labs, Inc. Calibration information is provided here as a reference, so that definitions and equations do not have to be provided in each individual analytical SOP. Review of this information shall be considered adequate on-going training in calibration models and equations.

This chapter consists of the following sections:

- Definitions
- General Criteria for All Calibration Models
- External Standard Calibration
- Internal Standard Calibration
- Response/Calibration Factor Model
- Linear Calibration Using A Least Square Regression
- Weighted Least Squares Regression

DEFINITIONS

Calibration can be defined in several ways:

- **Reference standards with known values for selected points covering the chosen concentration range are measured with the instrument. A functional relationship is then established between the values of the standards and the corresponding measurements.**

- **Calibration: set of operations that establish, under specific conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.**

Instrument calibration is intended to eliminate or reduce bias in an instrument's readings over a range for all continuous values.

Precision is a measure of the degree of agreement among replicate analyses of a sample, usually expressed as a standard deviation.

Bias is consistent deviation of measured values from the true value, caused by systematic errors in a procedure.

Accuracy is the combination of bias and precision of an analytical procedure, which reflects the closeness of a measured value to a true value

An acceptable calibration assures that an instrument will produce results which meet or exceed some defined criteria with a specified degree of confidence.

GENERAL CRITERIA FOR ALL CALIBRATION MODELS

- **Mid-points cannot be dropped to meet criteria**
- **Points can be reanalyzed, original run must be discarded**
- **Analyzing additional standards and discarding some to achieve a better correlation coefficient value is prohibited**
- **Narrowing of curve on either end is allowed**

Initial Calibration

Perform initial calibration with a minimum of three concentrations of standards for linear curve, a minimum of five concentrations for nonlinear curves, or as specified by the method of choice.

At the beginning of each day that samples are to be analyzed, a calibration curve covering the sample concentration range and all target analytes should be generated according to the approved SOP. Depending on concentration ranges, the curve should be composed of three or more points. The reporting limit should be included in the calibration range.

Daily Verification Standard

Where the determinative time is extensive and the instrument is very stable, the calibration curve should be initially developed. Thereafter, each day analyses are performed, this curve should be verified by analysis of at least one standard for each of the target analytes at the expected concentration range. This verification should be done at both the beginning and end of the analyses.

Calibration Plot

A working curve is a plot of the instrument response as a function of analyte concentration. The concentration of an unknown sample is determined by correlating its response to the mathematical relationship of concentration to the instrument response established by the curve.

Response Factor/Calibration Factor

Both calibration factors and response factors are measures of the slope of the calibration relationship. Each calibration or response factor represents the slope of the line between the response for a given standard and the origin. Under ideal conditions, the factors will not vary with the concentration of the standard. In practice, some variation is to be expected.

If response factors or calibration factors are used, the calculated % RSD for each analyte of interest must be less than or equal the method-specified value. Refer to the applicable method for the calibration procedure and acceptance criteria on the response factors or calibration factors for each analyte.

Correlation Coefficient

The correlation coefficient is a measure of the degree with which the independent variable and its partner move either together or in opposition. A positive result indicates direct correlation and a negative result indicates an inverse correlation.

If linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation coefficient is not specified then a minimum value of 0.995 is recommended. The appropriate linear or nonlinear correlation coefficient for standard concentration to instrument response should be > 0.995.

It is not necessarily true that a relationship measured by r is meaningful. There must be a rational relationship of the two variables under investigation.

The sample on which the data is based must be large enough to ensure that the influence of chance causes of variation is minimized.

Coefficient of Determination

In a correlation analysis, r2 (occasionally called the “correlation index”) may be calculated most simply by squaring the correlation coefficient, r. It may be described as the amount of variability in one of the variables accounted for by correlating that variable with the second variable. As in regression analysis, r2 may be considered to be a measure of the strength of the straight-line relationship. In order for the linear regression model to be used for quantitative purposes, r, COD, or r2 must be greater than or equal to 0.99

EXTERNAL STANDARD CALIBRATION

For an external standard quantitation, known data from a calibration standard and unknown data from the sample are combined to generate a quantitative report.

It is called external standard because the standard or known material is separate or external to the unknown material. External standard calibration is one of the most common approaches to calibrations. It involves a simple comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards.

Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

$$CF = (Ax)/(Cx)$$

Where: Ax = Area of the compound
Cx = Concentration of the compound

Advantages:

- The advantages of external standard calibration are that it is simple to perform this type of calibration and it can be applied to a wide variety of methods.

Disadvantages:

- The primary disadvantage is that it is greatly affected by the stability of the chromatographic detector system and the presence of chromatographic interferences in a sample or sample extract.

INTERNAL STANDARD CALIBRATION

Internal standard calibration involves the comparison of the instrument responses from the target compounds in the sample to the responses of reference standards added to the sample or sample extract before injection.

The response of the target compound is normalized to the response of the reference standard. This reference standard is called an *internal standard* because it is contained within the aliquot of the sample or sample extract that is actually injected into the instrumentation. A constant amount of the internal standard is added to all samples or extracts. That same amount of the internal standard is also included in each of the calibration standards.

The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard.

This ratio is termed the response factor (RF) or relative response factor (RRF), indicating that the target compound response is calculated relative to that of the internal standard.

$$\mathbf{RF = ((Ax)(Cis))/((Ais)(Cx))}$$

Where: **Ax = Area of the compound**
Cx = Concentration of the compound
Ais = Area of the internal standard
Cis = Concentration of the internal standard

Selection of Internal Standards

- Internal standards that are similar in analytical behavior to the compounds of interest, and not expected to be found in the samples
- The analyst needs to demonstrate that the measurement of the internal standard is not affected by target analytes, surrogates, or by matrix interferences
- This is not as useful for GC and HPLC methods with non-MS detectors, unless the internal standards could be separated from target compounds chromatographically.

Advantages:

- Accounts for routine variation in the response of the chromatographic system
- Accounts for the variations in the exact volume of sample or sample extract introduced into the chromatographic system
- The retention times of the target compound and the internal standard may be used to calculate the relative retention time (RRT) of the target compound and can then be used to compensate for small retention time shifts

Disadvantages:

- The principal disadvantage to internal standard calibration is that the internal standards must be compounds that are not found in the samples to be analyzed and they must produce an unambiguous response on the chromatographic detector system.

RESPONSE/CALIBRATION FACTOR MODEL

Calibration Factor: A measure of the chromatographic response of a target analyte relative to the mass injected.

Response Factor: A measure of the relative mass spectral response of an analyte compared to its internal standard.

Each calibration or response factor represents the slope of the line between the response for a given standard and the origin. The average calibration factor or response factor of the standards for each analyte is then used to calculate the concentration of the sample.

When the variation, measured as the relative standard deviation (RSD) of the factors, is less than or equal to 20%, then the slopes of the lines for each standard are sufficiently close to one another that the use of the linear model is generally appropriate over the range of standards that are analyzed. A relative standard deviation (RSD) of 25% or less is considered linear.

Response/Calibration Factor Equations

External Standard Equation

$$CF = (Ax)/(Cx)$$

or

Internal Standard Equation

$$RF = ((Ax)(Cis))/((Ais)(Cx))$$

Where: Ax = Area of the compound
Cx = Concentration of the compound
Ais = Area of the internal standard
Cis = Concentration of the internal standard

Response/Calibration Factor Statistical Equations

Average RF or CF: $RFAVE = (S\ RFi / n)$

Standard Deviation (s): $s = \sqrt{\{ [S (RFi - RFAVE)2] / (n-1) \}}$

Relative Standard Deviation (RSD): $RSD = s / RFAVE * 100$

Where: n = number of pairs of data
RFi = Response Factor for each level
RFAVE = Average of all the response factors
S = the sum of all the individual values

In the equations above RF can be replaced with CF

Response/Calibration Factor Equations for Concentration

External Standard Equation
$$Cx = Ax/CFAVE$$

or

Internal Standard Equation
$$Cx = ((Ax)*(Cis)) / ((Ais)*(RFAVE))$$

Advantages

- Assumes linearity through the origin, no negative calculated concentrations.
- Simple calculation

Disadvantages

- Linearity of the curve is required.
- May not reflect actual detector response curve.

LINEAR CALIBRATION USING A LEAST SQUARE REGRESSION

According to NIST Linear least squares regression is by far the most widely used modeling method. It is what most people mean when they say they have used "regression", "linear regression" or "least squares" to fit a model to their data.

Least square regression is a method of determining the curve that best describes the relationship between expected and observed sets of data by minimizing the sums of the squares of deviation between observed and expected values. The regression calculations attempt to minimize this sum of the squares, hence the name “least squares regression.”

A linear calibration model based on a least squares regression may be employed based on past experience or a prior knowledge of the instrument response and at the discretion of the analyst. This approach may be used for analytes that do meet the RSD Limits. The linear calibration model is most easily achieved by performing a linear least squares regression of the instrument response versus the mass of the analyte.

Correlation Coefficient Definition: A measure of the interdependence of two random variables that ranges in value from −1 to +1, indicating perfect negative correlation at −1, absence of correlation at zero, and perfect positive correlation at +1. Also called coefficient of correlation.

Linear Equation

$$y = mx + b$$

Where: y = Response Ax for External Standard
Or
Ax/Ais for Internal Standard

x = Concentration Cx for External Standard
or
Cx/Cis for Internal Standard
m = Slope
b = Intercept

Linear Regression Statistical Equations

Slope (m)

$$m = \frac{[(Swxiyi * Sw) - (Swxi * Swyi)]}{[(Sw * Swxi2) - (Swxi * Swxi)]}$$

Intercept (b)

$$b = yAVE – (m * (xAVE))$$

Correlation Coefficient (r)

$$r = \frac{[(Sw * Swxiyi) - (Swxi * Swyi)]}{\sqrt{\{[(Sw * Swxi2) - (Swx * Swxi)] * [(Sw * Swyi2) – (Swyi * Swyi)]\}}}$$

Coefficient of Determination (r2)

$$r2 = r * r$$

Where:
- n = number of x, y pairs
- xi = individual values for the independent variable
- yi = individual values for the dependent variable
- w = weighting factor, for equal or no weighting w = 1
- xAVE = average of the x values
- yAVE = average of the y values
- S = the sum of all the individual values

Equations for Concentration

External Standard Equation

$$Cx = \{Ax-b\} /m$$

or

Internal Standard Equation

$$Cx = [\{(Ax)/(Ais)\}-b] /m *Cis$$

Advantages

- This technique is the simplest and most commonly applied form of Linear Curve
- Computation of coefficients and standard deviations is easy

Disadvantages

- If least squares regression (linear and non-linear) is used for curve construction it is usually noticed that the lower levels of the calibration may fail the re-fit criteria (<20% D) even when the r/COD/r2 criteria have been met.

- Analysts that use least squares regression and rely only on the r/COD/r2 criteria for curve acceptance may not be aware of this potential problem at the lower calibration levels.

WEIGHTED LEAST SQUARE REGRESSION

Each term in the weighted least squares criterion includes an additional weight that determines how much each observation in the data set influences the final parameter estimates and it can be used with functions that are either linear or nonlinear in the parameters.

One of the common assumptions underlying most process modeling methods, including linear and nonlinear least squares regression, is that each data point provides equally precise information about the deterministic part of the total process variation. In other words, it is assumed that the standard deviation of the error term is constant over all values of the predictor or explanatory variables. This assumption clearly does not hold, even approximately, in every modeling application.

In a weighted fit, less weight is given to the less precise measurements and more weight to more precise measurements when estimating the unknown parameters in the model. Using weights that are inversely proportional to the variance at each level of the explanatory variables yields the most precise parameter estimates possible.

Method 8000C describes variance as the difference between the observed instrument response for the ith calibration standard and the predicted or calculated response for the ith calibration standard.

Weighting the sum of the squares of the differences may significantly improve the ability of the least square regression to fit the linear model to the data.

$$\sum w_i (y_i - y'_i)^2$$

where:

w_i = Weighting factor for the ith calibration standard (w=1 for unweighted least square regression)

y_i = Observed instrument response for the ith calibration standard

y'_I = Predicted (or calculated) response for the ith standard

$\sum$ = The sum of all individual values

The mathematics used in unweighted least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated will tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels.

Examples of weighting factors which can place more emphasis on numbers of smaller value are:

$$w_i = 1/y_i \quad \text{or} \quad w_i = 1/y_i^2$$

where,

wi = weighting factor for the ith calibration standard (wi=1 for unweighted least squares regression).

y_i = observed instrument response (area or height) for the ith calibration standard.

Different Types Of Weights

No Weights:	Default higher weighting of higher amounts or signal values
1/Amount:	Nearly cancels out the weighting of higher amounts
1/Amount^2 :	Causes over-proportional weighting of smaller amounts
1/Response:	Nearly cancels out the weighting of higher signal values
1/Response^2:	Causes over-proportional weighting of smaller signal values
1/RSD:	Weights signal values with small relative standard deviations more than those with large relative standard deviations
1/RSD ^{2} :	Weights signal values with small relative standard deviations clearly more than those with large relative standard deviation.

Advantages

- Weighted least squares is an efficient method that makes good use of small data sets. It also shares the ability to provide different types of easily interpretable statistical intervals for estimation, prediction, calibration and optimization.

- The main advantage that weighted least squares enjoys over other methods is the ability to handle regression situations in which the data points are of varying quality.

Disadvantages

- The biggest disadvantage of weighted least squares is probably the fact that the theory behind this method is based on the assumption that the weights are known exactly. The exact weights are almost never known in real applications, so estimated weights must be used instead. The effect of using estimated weights is difficult to assess, but experience indicates that small variations in the weights due to estimation do not often affect a regression analysis or its interpretation.

- When the weights are estimated from small numbers of replicated observations, the results of an analysis can be very badly and unpredictably affected. This is especially likely to be the case when the weights for extreme values of the predictor or explanatory variables are estimated using only a few observations. It is important to remain aware of this potential problem, and to only use weighted least squares when the weights can be estimated precisely relative to one another.

- Weighted least squares regression is also sensitive to the effects of outliers. If potential outliers are not investigated and dealt with appropriately, they will likely have a negative impact on the parameter estimation and other aspects of a weighted least squares analysis.

- If a weighted least squares regression actually increases the influence of an outlier, the results of the analysis may be far inferior to an unweighted least squares analysis.

APPENDIX J

Authorized Signatures

The individuals listed below are authorized to sign/approve documents in the classes listed. See also SOP ALI-30.

Approval Signatures - Moscow

Quotes/Contracts/RFP Submissions

- John Coddington, Lab Manager
- Todd Taruscio, Technical Director
- Erin Linskey, Technical Director
- Mike Pearson, Lab Director
- Gene Solomon, QA Officer

Analytical Reports

- John Coddington, Lab Manager
- Todd Taruscio, Technical Director
- Erin Linskey, Technical Director
- Mike Pearson, Lab Director

SOPs

- John Coddington, Lab Manager
- Gene Solomon, QA Officer
- Justin Doty, Customer Service Manager (for office procedures)
- Erin Linskey/Todd Taruscio, Technical Director (in Lab Manager's absence)
- Mike Pearson, Lab Director (in Lab Manager's absence)

Corrective Action Reports

- John Coddington, Lab Manager
- Todd Taruscio, Technical Director
- Erin Linskey, Technical Director
- Mike Pearson, Lab Director
- Gene Solomon, QA Officer

Demonstrations of Capability (IDC/DOC)

- John Coddington, Lab Manager
- Gene Solomon, QA Officer
- Erin Linskey/Todd Taruscio, Technical Director (in Lab Manager's absence)
- Mike Pearson, Lab Director (in Lab Manager's absence)

Approval Signatures - Spokane

Quotes/Contracts/RFP Submissions

- Kathy Sattler, Lab Manager
- Melissa Lewis, QA Officer
- Mike Pearson, Lab Director

Analytical Reports

- Kathy Sattler, Lab Manager
- Melissa Lewis, QA Officer
- Mike Pearson, Lab Director

SOPs

- Kathy Sattler, Lab Manager
- Melissa Lewis, QA Officer
- Mike Pearson, Lab Director
- Karice Scott, Office Manager (for office procedures)
- Mike Pearson, Lab Director (in Lab Manager's absence)

Corrective Action Reports

- Kathy Sattler, Lab Manager
- Melissa Lewis, QA Officer
- Mike Pearson, Lab Director

Demonstrations of Capability (IDC/DOC)

- Kathy Sattler, Lab Manager
- Melissa Lewis, QA Officer
- Mike Pearson, Lab Director (in Lab Manager's absence)

ENERGY LABORATORIES-CASPER, WY
QUALITY ASSURANCE MANUAL

Revision May 15, 2015

Energy Laboratories, Inc.
Main Building Address: 2393 Salt Creek Hwy.
Radiochemistry Building Address: 2325 Kerzell Lane

PO Box 247
Casper, Wyoming 82602

Toll Free (888) 235-0515
Local: (307) 235-0515
Fax: (307) 234-1639
E-mail: casper@energylab.com

Daniel W. Lashbrook
Casper Laboratory Manager

Digitally signed by
Daniel W. Lashbrook
Date: 2015.05.26 08:47:10 -06:00

Laboratory Manager
Daniel Lashbrook

Date:

Technical Director

Digitally signed by
Steve Vien
Date: 2015.05.27 08:44:31 -06:00

Laboratory Technical Director
Steve Vien, Ph.D.

Date:

Quality Assurance Manager

Digitally signed by
Donny Juarez
Date: 2015.05.18 10:22:01 -06:00

Quality Assurance Officer
Donny Juarez

Date:

TABLE OF CONTENTS

Quality Assurance Manual 1
Table of Contents 2
ELI Commitment 4
Introduction 4
Chapter 1 – Quality Control Program 6
Quality Policy Statement 6
Quality Assurance Program 6
Estimation of Uncertainty 9
Maintenance of Performance Records 9
Method Quality Control Specifications 10
Chapter 2 – Quality Assessment Program 11
Proficiency Testing (PT) Samples 11
Quality Control Check Samples 12
Quality Assurance Audits 12
Chapter 3 – Laboratory Facilities 14
Chapter 4 – Personnel Requirements and Laboratory Organization 15
Laboratory Organization 15
Personnel Requirements 16
Laborator Manager 16
Quality Assurance Officer 17
Technical Director 17
Laboratory Supervisor 17
Analysts 17
Laboratory Technicians 18
Approved Signatories 18
Chapter 5 – Sampling Procedures 19
Chapter 6 – Sample Handling 21
Sample Receipt 21
Chain-of-Custody 21
Sample Tracking 22
Sample Disposal 22
Subcontracting Policy 22
Chapter 7 – Instrument Operation and Calibration 24
Chapter 8 – Records and Reporting 26

Document Management 26
Laboratory Notebooks 26
Records 27
Data Reduction 27
Validation 28
Reporting 28
Chapter 9 – General Laboratory Practices 30
Chemicals and Reagents 30
Reagent Interference 30
Glassware Preparation 31
Laboratory Pure Water 31
Employee Training 31
Data Integrity 32
Standard Operating Procedures 33
Client Confidentiality 33
Chapter 10 – Quality Control Monitoring 34
Routine Monitoring 34
Instruments/Methods 34
Chapter 11 – Corrective Action 36
Procedure for Dealing with Complaints 37
Penalty for Improper, Unethical or Illegal Actions 38
Chapter 12 – Management of Change 39
Chapter 13 – Major Equipment and Methods 40
Chapter 14 – Preventive Maintenance 41
Chapter 15 - References 42
Chapter 16 – Glossary of Terms 44
Acronyms and Abbreviations 49
APPENDIX A 51
Quality System Controlled Documents 52
APPENDIX B 53
Quality Assurance / Quality Control Specifications 54
APPENDIX C 57
Organizational Charts 58
APPENDIX D 60
Curricula Vitae of Key Laboratory Personnel 61
APPENDIX E 83
Equipment and Methods List 84
Record of Review 85

ELI COMMITMENT

Energy Laboratories, Inc. Strives Toward:

1. Being highly skilled in the field of analytical chemistry.
2. Delivering quality and service with integrity.
3. Encouraging the professional development of our staff.
4. Offering our employees a safe and positive work environment.
5. Being profitable and using resources wisely for a sustainable future.

INTRODUCTION

Energy Laboratories, Inc. provides chemical, industrial hygiene, and environmental analytical services to private industry, agricultural industry, engineering consultants, government agencies, and private individuals. Analytical services include: analysis of waters and soils for inorganic and organic constituents, aquatic toxicity testing, hazardous waste analysis, radiochemistry, industrial hygiene, microbiology, soils and water physical parameters, and petroleum analysis.

Founded in 1952, Energy Laboratories currently incorporates five separate testing laboratories. The corporate headquarters are located in Billings, MT, with branch laboratories located in Casper, WY; Gillette, WY; College Station, TX and Helena, MT.

ELI, as a coordinated company of five participating branches, has developed a QA program that takes into account the various method types and EPA programs, while also considering sample matrices, to develop a single comprehensive set of QA guidance. We have used scientific approaches, Good Laboratory Practices, EPA Methods and Guidance documents, and accreditation audit guidance to develop our overall QA Program.

The Quality Assurance Program establishes acceptable performance criteria for all routine analytical procedures being performed by laboratory personnel. The Quality Assurance Assessment Program provides a formal system for evaluating the quality of data being generated and reported. The ELI Laboratory Safety Manual & Chemical Hygiene Plan defines the safety and monitoring procedures used by laboratory personnel in laboratory operations. These, in addition to the experience and expertise of our analysts, provide a comprehensive Quality Assurance Program. Energy Laboratories, Inc., in Casper, Wyoming, is certified under the Safe Drinking Water Act by Region (SDWA) VIII EPA. Individual state approval for SDWA is managed through reciprocal certifications when required by a specific state. ELI-Casper also holds accreditation for Clean Water Act, Safe Drinking Water Act and Resource Conservation Recovery Act (RCRA) parameters through the National Environmental Laboratory Accreditation Program (NELAP), which is supported by the EPA. The NELAP certification is maintained through the state of Florida. Individual state approval for CWA (NPDES),SDWA, and RCRA is managed through the Federal/State DMRQA program or through reciprocal certifications when required by a specific state. To perform radon testing, ELI is certified under the National Radon Proficiency Program administered by the National Environmental Health Association. Branch laboratories of ELI are certified in their own state and in additional states. Copies of ELI's certificates for all laboratories are maintained on ELI's website: www.energylab.com.

The ELI Quality Assurance Manual, the ELI Qualifications Manual, and the ELI Technical Services and Fee Schedule together are used to outline the ELI Quality Assurance/Quality Control Program. This Quality Assurance Manual is appropriate to all departments of Energy Laboratories-Casper. The procedures discussed or referenced in this manual describe our day-to-day laboratory practices and adhere to USEPA Safe Drinking Water Act, and TNI (The NELAC Institute) requirements as well as Good Laboratory Practices (GLPs). Information on ELI-Casper's, and all other ELI branch laboratories', applicable accreditations and certifications are maintained on the ELI website at www.energylab.com. Where possible, ELI uses EPA, AOAC, ASTM, APHA, NIOSH, OSHA, or published analytical methods and follows the procedures with strict adherence to described protocol and recommended QA/QC parameters. The analytical methods approved and in use are described in Standard Operating Procedures, and are available for review at the laboratory. Vital parts of our Quality Assurance Program, Quality Control and Quality Assessment programs are outlined in Chapters One and Two of this manual.

To generate data that will meet project-specific requirements, it is necessary to define the type of decisions that will be made and identify the intended use of the data. Data Quality Objectives (DQOs) are an integrated set of specifications that define data quality requirements and the intended use of the data. Project-specific DQOs will be established as needed for both field and lab operations. Through the DQO process, appropriate reporting limits, extraction/digestion methods, clean-up methods, analytical methods, target analytes, method quality control samples, sample security requirements, quality control acceptance ranges, corrective action procedures, reporting formats and reporting limits can be specified. Professional laboratory project managers are available to assist clients in specifying appropriate laboratory analyses and reporting procedures necessary to meet project requirements.

Client-specific DQOs can be coordinated with the laboratory through our Project Managers via quotations or contracts, or with relevant documentation provided to the laboratory prior to (or at time of) sample receipt. Client-specific requirements are communicated to analysts and final report validators through the laboratory LIMS system. By default, our methods, analytes, and QC parameters are set up to meet the DQOs specified in the referenced method and/or federal/state regulations. ELI encourages clients to provide ELI documentation of any client-specific, regulatory or project monitoring requirements.

Certain types of requests may not be suitable to standardized analytical methods. These custom requests are handled individually with laboratory management and staff scientists. Project-specific methods and reporting packages are available. Attention to documentation of the analytical procedure and use of suitable QC parameters is maintained according to good scientific discipline and Good Laboratory Practice guidelines.

The ELI-Casper Laboratory Manager, or their designee, will evaluate all new contracts to determine that the laboratory is capable of performing the requested work. This process includes ensuring that the laboratory maintains the required accreditation, equipment and resources. In the event that sample analysis is not performed at our Casper location, clients are notified, on the laboratory analytical report, if the work is subcontracted to a qualified branch laboratory or an outside laboratory (See Subcontracting Policy – Chapter 6 in this QA Manual).

This Quality Manual and related quality documentation meet requirements of the National Environmental Laboratory Accreditation Program (NELAP), which is an EPA approved accreditation program.

CHAPTER 1 – QUALITY CONTROL PROGRAM

Quality Policy Statement

Energy Laboratories, Inc. is committed to producing laboratory data of known and documented quality that is scientifically valid, meets method specifications, satisfies regulatory requirements, and accomplishes the data quality objectives of the client and project. Management ensures that the laboratory maintains current certifications and is in compliance with accreditations through USEPA, State Agencies, and NELAP. Those method, regulatory, and client requirements (as well as the policies, procedures, and all referenced documents) are incorporated into our Quality Assurance Program; which is outlined within this Quality Assurance Manual. Our Quality Systems are designed to comply with the standards as defined by the most current version of the NELAC accreditation standard and ISO 17025 standards. To ensure compliance with these standards, all laboratory personnel are required to be familiar with quality documentation and implement those policies and procedures in their work. ELI is dedicated to the continual improvement of the management system's effectiveness by providing appropriate corporate resources to set objectives, offering training opportunities, and monitoring the quality performance of our staff. ELI also provides facilities and equipment adequate and appropriate to these objectives.

Quality Assurance Program

The purpose of the Quality Assurance Program is to ensure that the analytical services provided by Energy Laboratories are of high quality, data is within established accuracy and precision limits (required by the referenced method or Standard Operating Procedure), and each analytical result produced meets or exceeds our accreditation requirements. Management ensures that the integrity of the management system is maintained. The Technical Directors, or their designees, ensure that changes to the management systems are planned, implemented and documented.

Management establishes and maintains data integrity by providing the following to ELI's data integrity system:

1) Data Integrity Training (Including the highest standards of ethical behavior)
2) Periodic review of data integrity procedural documentation
3) Annual review of data integrity procedures with updates as needed
4) Periodic, in-depth monitoring of data integrity
5) Maintenance of signed data integrity documentation for all laboratory employees

All employees are expected to implement and follow the policies contained within the Quality Assurance Program. Internal documents (controlled and associated with the Quality Assurance Program) are listed in Appendix A.

The quality systems in the program consist of the policies and procedures, and all referenced documents, described in this Quality Assurance Manual. The Quality Control Program also functions to maintain the laboratory's compliance with accreditations through USEPA, State Agencies, and NELAP.

The Quality Control Program requires that the following points be met for each applicable analytical method:

- Performance of any analytical method requires that the proper equipment and instrumentation are available. A list of major equipment is listed in Appendix E. The procedure for operation of an analytical instrument is described in the equipment manufacturer's operating manual, and may also be supplemented with a specific Standard Operating Procedure (SOP) for the instrument and/or the method.

- Specific SOPs cover operation of the instrument including the sequence of operations involved in instrument start-up, calibrating, analyzing, and shutting down. Chapter Thirteen of this manual includes recommended preventative maintenance, and/or a list of parameters used to identify other types of maintenance. SOPs outline any special safety precautions for operation of the instrument.

- SOPs of well-detailed Standard Methods, EPA, ASTM, NIOSH, APHA, OSHA, or published procedures include, as appropriate, a list of any method-specific items or variances, a list of QC parameters and their recommended method performance ranges, recommended or example analytical sequences, specific or unique safety information, method references, and a signed signature page. SOPs details, and format of method SOPs, follow NELAP requirements. Detailed SOPs may be prepared for those procedures that do not have published methods. Further details of SOP format and information required in method SOPs can be found in the ELI SOP, *Preparation, Numbering, Use, and Revision of Standard Operating Procedures.* Written Standard Operating Procedures referenced within this manual are available at the laboratory for review. (ELI SOPs are considered confidential proprietary information and ELI does not allow copies to be removed).

- For radiochemical analysis performed at Casper and ELI's branch labs, each method undergoes Method Validation as outlined in EPA's specific method and/or the Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), Chapter 6.

- The required detection level (RDL) for radiochemical analysis of drinking water samples is calculated based on the requirements in 40 CFR 141.25(c), which is a sample specific determination. The equation is specific for each method and noted in the method-specific SOP.

- The initial test method evaluation for chemical analysis involves Method Detection Limit (MDL) studies, (refer to ELI SOP, *Determination of Method Detection Limits (MDL) and Quantitation Limits*), confirmation of the Limit of Detection (LOD) and/or Practical Quantitation Limit (PQL), also known as the Limit of Quantitation (LOQ), an evaluation of method performance (using four or more replicates of quality control samples), evaluation of the selectivity of the method, and any additional method-specific requirements

- ELI demonstrates that laboratory staff are qualified and capable of performing the method. Analysts are assigned duties based on their skills and experience. Training records are maintained for all analysts. Curricula vitae of supervisory and senior analysts are described in Appendix D and the ELI *Qualifications Manual*.

- It is the responsibility of the analyst to become thoroughly familiar with the methodology and instrument operation before performing the analysis. It is the responsibility of the person providing training to monitor all laboratory results generated for a reasonable time. The amount of time necessary may vary depending on the method and the experience of the analyst. At a minimum, the analyst's performance is to be monitored until the analyst demonstrates the ability to generate results of acceptable accuracy and precision according to the method.

- All analysts are required to demonstrate and maintain a record of proof of competency by routinely analyzing quality control samples appropriate to the analytical procedures they perform. Competency in analyzing these control samples is documented in analysts' training files per NELAP requirements (for more information, see ELI SOP, *Personnel Training and Training Records*). For those analyses where external proficiency testing (PT) samples are not routinely analyzed, competency is documented by including the results of routine analysis of method-specific quality control samples (prepared by laboratory staff) and/or a verifying statement of procedural review by a supervisor or trained analyst.

- Each analytical method is subjected to quality control monitoring. The purpose is to demonstrate that results generated meet acceptable accuracy and precision criteria for the method. Precision and bias are determined for standard and non-standard methods. Precision and bias are determined for standard methods through control charting of data from quality control samples. Precision and bias using non-standard, modified standard or laboratory-developed methods are compared to the criteria established by the client (when requested), the method, or the laboratory.

- Quality control requirements are outlined in the methods and ELI, at a minimum, follows the guidelines specified in the methods used. Additional QC requirements are also added as appropriate. Performance is periodically evaluated against method requirements using control charts.

- Quality control monitoring to measure accuracy for each method generally requires that five to ten percent of all samples analyzed be fortified (spiked) with a known concentration of target analytes tested by the method. The percent recovery is then calculated. This provides a means for monitoring method accuracy and evaluating sample matrix effects. Where appropriate, surrogates are included in the method to monitor method performance on each individual sample. Blank spike samples replace matrix spike samples for certain methods, or when there is insufficient sample for a matrix spike analysis. Historical, routine batch QC sample performance can be used to estimate the precision and accuracy of the method.

- Quality control monitoring to measure precision for each method requires replicate samples be prepared and analyzed when appropriate. Actual requirements are outlined in the specific SOP. When replicate samples or matrix spike duplicates are analyzed, relative percent difference is calculated and used to monitor precision of the method. In instances where there are no specific method requirements, it is the policy of this laboratory to analyze five to ten percent of all samples in duplicate. Duplicate test results must be within the control limits established for each analysis type or data is

qualified. Acceptance limits generally follow specifications listed in the method. Matrix spike duplicates replace sample duplicates for most methods.

- When not defined in the method, and as appropriate, method blanks and/or instrument blanks are analyzed one in every 20 samples at a minimum. Method blanks are used to verify that contamination from laboratory reagents and glassware is not present in the analytical sample process. Generally, the method blank should be less than the reporting limit, or 10 times less than the concentration amount in the sample, for the analytical parameter being tested, whichever is greater.

- When not defined in the analytical method and as appropriate, method spikes (blank spikes) are analyzed one in every 20 samples, at a minimum.

- Calibration standards are analyzed and calibration curves are developed for all applicable methods. For additional information on instrument calibration, see Chapter Seven of this QA manual.

- The initial calibration is continuously monitored by analyzing a continuing calibration standard every 10 to 20 samples, or within a specified time frequency, and at the end of each analytical sequence; depending on the method and instrumentation. Results must be within an established range as described by the method SOP. Initial calibrations are verified against a standard from a second source.

- Proficiency testing samples and further quality control check samples may be required for various methods. Refer to Chapter Two of this QA manual for further details.

Estimation of Uncertainty

The estimation of uncertainty consists of the sum of the uncertainties of the individual steps or processes of an analytical procedure. The variability of the sampling plan, sample heterogeneity, extraction procedure, instrument calibration, instrument drift, systematic bias, and many other factors all contribute to the uncertainty of a measurement or result.

ELI estimates uncertainty utilizing Confidence Intervals defined as ±2σ (95%) and ±3σ (99%) where σ is the standard deviation of the recovery of quality control samples. The confidence intervals calculated from these QC samples are based on the spike level concentrations for each method. Uncertainty at low concentrations may be one to three times the quantitation limit. Real world samples, depending on matrix interferences, may have a greater amount of uncertainty associated. Due to limitations in assessing the uncertainty for each matrix type, the confidence intervals calculated from method QC samples provides an estimate of uncertainty.

Energy Laboratories, Inc. uses the procedures outlined in ELI SOP, *Control Chart Generation and Maintenance*, for the purpose of evaluating estimation of uncertainty for chemical analyses and uses the determination of uncertainty on a sample-specific basis for all radiochemistry measurements. These estimates of uncertainty have formulas documented in the individual SOP.

Maintenance of Performance Records

All quality control monitoring is recorded and documented. Quality control data is recorded in laboratory notebooks, electronic summary files, and/or analysis sheets. Generally, review of QC data and trends is managed within the Laboratory LIMS system. QC data management and control chart generation, maintenance, and usage are described in ELI SOP, *Control Chart Generation and Maintenance*. It is the responsibility of the analyst to see that all results are recorded in a timely manner.

All quality control data is filed and available for inspection and assessment by analysts, supervisors, management, and quality control personnel.

Method Quality Control Specifications

A general summary of Quality Assurance/Quality Control specifications are outlined in Appendix B. Exact details of method QC can be found in the applicable method SOPs. These types of QA parameter tables are available upon approved request for our clients to use in the preparation of Quality Assurance Project Plans (QAPPs).

CHAPTER 2 – QUALITY ASSESSMENT PROGRAM

The function of the Quality Assessment Program is to provide formal evaluation of the quality of data being generated and reported by the laboratory. External and internal quality control measures are used in this assessment. These measures include proficiency testing samples, laboratory quality control check samples, and routine internal and external audits on methodology and documentation procedures.

Proficiency Testing (PT) Samples

PT samples are supplied by an outside entity and contain known amounts of constituents. The laboratory does not have access to known values of the samples. Only the PT provider has knowledge of constituent levels prior to the formal publishing of the test results.

PT samples are received on a minimum bi-annual basis, with results sent to the providing entity for evaluation. Proficiency Testing (PT) samples for USEPA, NELAP and various State certifications are Water Pollution Study samples (WP or DMRQA), Water Supply Study samples (WS), and LPTP Soil PT samples provided by either Resource Technology Corporation (RTC) and/or Environmental Resource Associates (ERA); both being NELAP approved PT providers. Routine participation in LPTP, WS and WP PT sample studies is used to maintain certifications for Safe Drinking Water Act (SDWA), Clean Water Act (CWA), National Pollutant Discharge Elimination System (NPDES), Discharge Monitoring Report Quality Assurance (DMRQA), permit monitoring analyses, Resource Conservation and Recovery Act (RCRA) analyses, as well as other states and projects requiring method accredited parameter analyses. The samples are analyzed in the same manner as any routine sample in the laboratory. Acceptable results are those that fall within a defined range as determined by the vendor/EPA/ NELAP; based on multi-laboratory study results. The provider sends results to USEPA and other certifying agencies as requested by ELI-Casper. PT study results are posted on the ELI website www.energylab.com.

A copy of the certificate for our primary certifications to perform drinking water analyses issued by the Environmental Protection Agency (USEPA) and the NELAP certificate from Florida Department of Health are maintained on the ELI website at www.energylab.com.. The EPA certification includes a list of parameters/methods for which drinking water certification has been granted. The NELAP certificate also includes RCRA methods used for hazardous waste characterizations and CWA parameters/methods which are used for NPDES monitoring permits. ELI also participates in the Federal/State DMRQA programs for clients which require/request this with their NPDES permits. Reciprocal accreditation in other states is based on either of these, or both, depending on specific state certification requirements/parameters. A list of current certifications is maintained on the ELI website at www.energylab.com.

Blind Quality Control Check Samples are samples submitted as regular lab samples and are processed through the system in the same manner as any other sample. The analysts do not know the true values of these samples when performing the analyses. Method performance reports are returned to the analysts. Clients occasionally submit these types of samples for their QAPP.

Inter-Laboratory comparison samples are samples containing known/unknown quantities of analytes that are split and analyzed by more than one laboratory.

Quality Control Check Samples

Quality Control Check Samples are performance evaluation samples used for routine method performance monitoring. As appropriate, analytical procedures include the analysis of a quality control sample with every sample batch analyzed. The materials are obtained from a commercial source when available, or they may be prepared in-house. Acceptable results are within a defined range based on certified ranges, or against statistically determined control limits, method-defined criteria or client defined Data Quality Objectives. Routinely used methods not subjected to PT sample monitoring are evaluated with Quality Control Check Samples, as appropriate.

QC samples are processed through the system in the same manner as any other sample, except the analyst is aware of the source, concentration, and acceptance ranges of target analytes and calculates analyte recoveries to evaluate method performance in real time.

Quality Assurance Audits

Quality Assurance Audits consist of internal and external laboratory inspections designed to monitor adherence to Quality Systems and quality control requirements. These audits check general laboratory operations, overall Quality Systems, adherence to QA program requirements, sample tracking procedures, sample holding times, storage requirements, adherence to procedures during analysis, calculations, completion of required quality control samples within the group surrounding the sample, and proper record-keeping.

Internal quality control audits are conducted or coordinated by the Quality Assurance Officer of the laboratory. See ELI SOP, *Internal Quality Assurance Audits*, for further information. ELI conducts internal inspections on a regular basis to monitor adherence to quality control requirements. Results of formal audits are given to management with possible recommendations for corrective action in the event any discrepancies are found. As necessary, a follow-up review is conducted to determine that identified problems have been addressed. Annually, the overall quality systems of the laboratory are reviewed and a summary report is prepared.

Per NELAP/ISO 17025-2005 requirements, the management of the laboratory will conduct an annual review of the Quality System, including policies, procedures and environmental testing activities. This is done to ensure the continuing suitability and effectiveness of the QA systems, as well as provide the opportunity to introduce necessary changes or improvements. The review shall take into account, at a minimum, the following:

- The suitability of policies and procedures
- Reports from managerial and supervisory personnel
- The outcome of recent internal audits
- Corrective and preventative actions
- Assessments by external bodies
- The results of inter-laboratory comparisons or proficiency tests
- Changes in the volume and type of work

- Client feedback
- Complaints
- Recommendations for improvement
- Other relevant factors, such as quality control activities, resources and staff training

The findings from management reviews and the corrective actions that arise from these findings shall be recorded. The management shall ensure that any corrective actions are carried out within an appropriate, pre-determined time frame.

ELI also conducts Peer Audits as part of an internal auditing program established within the company. This process utilizes analysts and supervisors from other branch laboratories to evaluate a designated ELI branch. The Peer Audits serve to not only address conformance issues, but also provide ELI with a tool to continuously improve process and consistency throughout the company. The goals of the Peer Audits are to:

- Encourage relationships between analysts
- Transfer technical knowledge between peers
- Establish consistency of analytical process/method between branch laboratories
- Identify the depth of analysts' knowledge at each position by observing what analysts are doing at the bench
- Determine training needs of personnel
- Document process/method and verify that issues are being corrected when found
- Work with, and in support of, QA department efforts

Depending on the size of the laboratory, a large number of methods and processes are examined during a Peer Audit. Results from these audits are provided to the branch management, as well as Corporate Management. Corrective Action Plans of a Peer Audit are initiated with the assistance of the Corporate Quality Assurance Officer for resolution of any findings.

ELI welcomes external Quality Assurance Audits, by qualified outside auditors, for review and comment on the overall QA program. To maintain certifications, accrediting authorities from the USEPA, and NELAP conduct periodic comprehensive external audits. External audits to meet Quality Assurance Project Plans (QAPPs), as applicable to environmental remediation projects, or for major industries, are conducted as requested. For more information, see ELI SOP, *External Quality Assurance Audits*.

CHAPTER 3 – LABORATORY FACILITIES

The Energy Laboratories, Inc. – Casper, WY facilities consists of five buildings; three of the buildings are on the same property with the main building address of 2393 Salt Creek Highway, Casper, WY 82601. The fourth and fifth buildings, located at 2325 Kerzell Lane, are the radiochemistry laboratory facilities.

The phone number for Casper's Energy Laboratories, Inc. is (307) 235-0515, the fax number is (307) 234-1639, the toll free number is 888-235-0515, and the email address is casper@energylab.com.

Laboratory space includes adequate bench top and floor space to accommodate periods of peak work load. Working space includes sufficient bench top area for processing samples; storage space for reagents, chemicals, glassware, bench and portable equipment items; floor space for stationary equipment; and adequate associated area for cleaning glassware. Laboratory departments are organized and the facilities are designed for specific laboratory operations in order to protect the safety of analysts and to minimize potential sources of contamination between and within department areas (for more information, see ELI SOP, *Facility Description, Access, and Security*).

The laboratory is appropriately ventilated and illuminated, and is not subject to excessive temperature changes. Specific laboratory areas are temperature and humidity controlled as required. Ample cabinets, drawers and shelves are available for storage and protection of glassware. Exhaust fume hoods are available as needed for use during preparation, extraction, and analysis of samples. Employee exposure monitoring is conducted to provide a safe working environment.

To maintain security, all visitors must enter their name on the ELI sign-in log at the front desk and wear a visitor's badge.

The laboratory has provisions for the disposal of chemical, radiochemical and microbiological wastes. These provisions are described in Standard Operating Procedures as well as outlined in the Laboratory Safety Manual & Chemical Hygiene Plan along with other safety and health guidelines. For more information, see ELI SOPs, *General Laboratory Waste Disposal*, and *Radiochemistry Waste Disposal*.

CHAPTER 4 – PERSONNEL REQUIREMENTS AND LABORATORY ORGANIZATION

Relationship between Management, Technical Operations, Support Services and the Quality System

Laboratory Organization

The corporate organization of the five ELI laboratories located in Montana (2), Wyoming (2), and Texas is provided in Appendix C. The Billings laboratory is the center for all corporate functions. Each laboratory is managed and operated individually under the supervision of a Laboratory Manager. Branch laboratories have fiscal and QA/QC responsibilities to Corporate, as well as general operating policies and goals. Quality Assurance Manuals are prepared individually for each branch and follow the QA/QC program outlined in the ELI-Billings QA manual.

The ELI-Casper Organizational Chart is also included in Appendix C with curricula vitae of key ELI-Casper laboratory personnel maintained in Appendix D of this manual. A Personnel Summary for all ELI employees listing title, academic background, and years of relevant experience is alsomaintained in the Corporate Qualifications Manual. Job descriptions are maintained by the Human Resources Department.

Quality Assurance receives direct support from senior management. Branch Quality Assurance Officers report directly to the Corporate Quality Assurance Officer as well as the Laboratory Manager. Quality Assurance Officers provide independent oversight of Quality Systems within the overall Energy Laboratories structure. When Quality Assurance Officers fill more than one role within the organization, they operate independently of direct environmental data generation while fulfilling quality assurance responsibilities. Quality Assurance Officers facilitate development of and maintain the branch Quality Assurance Manual, provide assistance to personnel on quality assurance / quality control issues, maintain a quality assurance training program, and review quality documentation including SOPs.

Management ensures the development and implementation of programs and policies to continuously improve the effectiveness of ELI's QA Program and Management Systems. Management performs an annual review of the laboratory's Quality System (policies, procedures, work instructions) to assure their continuing suitability and effectiveness (See ELI SOP: *Management Reviews*, for detailed procedures). As appropriate, management identifies and implements any necessary changes or improvements. Corrective and preventive actions are detailed in a Corrective Action Report and filed with the QA Department. (Refer to ELI SOP: *Nonconformance Procedures and Corrective/Preventive Action Reports,* for detailed procedures.) In addition, management performs meetings with supervisory and key staff members throughout the year. Supervisors and QA personnel provide input on their specific areas of responsibility and evaluate the following:

1) Client-Related Items
2) Internal and External Audit Reports
3) Proficiency Testing Results
4) Review of Performance by Department
5) Corrective and Preventive Actions

6) Personnel Training Needs
7) Quality System Policies and Procedures
8) Resources including Personnel, Equipment and Facilities

Laboratory Management Review findings are compiled into a summary report. The report includes deficiencies identified and areas for improvement. The QA department ensures items from the Management Review are tracked, including actions that must be addressed, assignment of parties responsible for the actions to be taken, and recommendations on improvements to the Quality System. The Technical Director, Laboratory Manager, QA Manager or designee, shall assign specific persons to address management review findings and establish deadlines for their completion. The Technical Director, Laboratory Manager, QA Manager or designee, reviews and approves all documents issued to personnel in the laboratory as part of the management system. The Technical Director, or designee, has overall responsibility for the technical operations of the laboratory. Any procedural deviations to SOPs that are client or project-specific must receive approval either from the Technical Director, Laboratory Manager, or Quality Assurance Officer. Work is stopped when identification of any of the following is made: unapproved departures from the management system, unauthorized deviations from the procedures for performing tests and/or calibrations, and data quality or data integrity issues. The Technical Director, Laboratory Manager, QA Manager, or designee, is responsible for providing authorization for the work to resume once the identified issue has been addressed.

Personnel Requirements

ELI maintains experienced staff and management. Below is a summary of the primary roles, responsibilities and qualifications for the designated positions. Laboratory experience can be substituted for academic requirements. At ELI's smaller laboratory operations, the technical director may serve multiple roles. Detailed job descriptions are maintained by the Human Resources Departmet. Specific titles of employees are at the discretion of the Laboratory Manager.

Laboratory Manager

The Laboratory Manager is required to have education equivalent to a Bachelor of Science degree in Chemistry or a related science. Five years of relevant laboratory experience is required.

The Laboratory Manager is responsible for all operations, client management, analysis scheduling, equipment acquisition, as well as compliance with all employment, safety, environmental and NELAP/ISO 17025 regulations. The Laboratory Manager may delegate daily activities of these work aspects to appropriate personnel. The Laboratory Manager reports directly to the Corporate Operations Manager. All Laboratory Managers have both technical and management responsibilities.

Quality Assurance Officer

The Quality Assurance Officer is required to have an education or experience equivalent to a Bachelor's of Science degree in Chemistry or a related science. Five years of relevant laboratory experience is preferred.

The Quality Assurance Officer is responsible for quality systems development, implementation, and management. The Quality Assurance Officer is also responsible for maintaining and improving compliance with all applicable state and federal regulations as well as maintaining compliance with NELAP/ISO 17025 regulations regarding Quality Systems. The Quality Assurance Officer or his/her designee manages the laboratory's certification programs to meet government regulatory requirements. The QA program is implemented in cooperation with all levels of management and staff. Quality Assurance Officers report directly to the Corporate Quality Assurance Officer. The Laboratory Manager will direct daily laboratory-specific QA/QC requirements.

Technical Director

The Technical Director is required to have a Bachelor of Science degree in Chemistry or a related science. Five years of relevant laboratory experience is required.

The Technical Director is responsible for ensuring compliance with all laboratory policies and that the analyses conducted under their supervision are compliant with all state, EPA, and NELAC/ISO17025 standards. The Technical Director reports directly to the Laboratory Manager.

The Technical Director may serve multiple roles. The Laboratory Manager can also serve as one of the branch technical directors.

Laboratory Supervisor

A Laboratory Supervisor is required to have education or experience equivalent to a Bachelor of Science degree in Chemistry or related science. Two years of relevant laboratory experience is required.

ELI's Laboratory Supervisors are responsible for the day-to-day operation of the laboratories: scheduling testing, assigning work, and completing the technical review of laboratory data. Supervisors are responsible for ensuring compliance with all laboratory policies and ensure that the analyses conducted under their supervision are compliant with all state, EPA, and NELAC/ISO17025 standards. They report directly to the Laboratory Manager.

Analysts

Laboratory Analysts are required to have an education equivalent to a Bachelor of Science degree in Chemistry (or related science), or a High School diploma with experience as an analyst in training. New analysts require a minimum of six months of on-the-job training, under direct supervision of a qualified analyst, in the measurements being considered for certification. After the initial training period, and on a continuing basis thereafter, the analyst must demonstrate acceptable skills through the successful participation in the analysis of applicable performance evaluation and quality control samples.

Laboratory Analysts perform the following duties: Preparation of samples and reagents, analysis and preliminary data input, as well as various other tests. Laboratory Analysts are responsible for complying with all laboratory policies and procedures.

Laboratory Technicians

Laboratory Technicians are required to have a High School Diploma or equivalent. Laboratory Technicians work under the supervision of the primary analyst performing general laboratory tests.

Under the supervision of a primary analyst, Laboratory Technicians perform the following duties: preparation of samples and reagents, analysis, and preliminary data input, as well as various other tests.

Laboratory Technicians are responsible for complying with all laboratory policies and procedures.

Approved Signatories

Signatures for policies are based on appropriate individuals, roles and responsibilities as determined by the policy being reviewed and approved. A list of significant signatories is included below. Additional signatures may be required for specific procedures.

- Laboratory Manager
- Technical Director
- Quality Assurance Officer
- Radiation Safety Officer (RSO)Corporate Officer- Board of Directors

A master list including signatures and initials for all employees is maintained for reference and signature verification.

CHAPTER 5 – SAMPLING PROCEDURES

Private individuals or companies, who are responsible for using proper collection procedures, collect most of the samples processed in this laboratory. Members of the staff are acquainted with proper sample collection and handling procedures and advise those who need help in this area. Instructions and forms for initiating Chain-of-Custody are available from ELI. Laboratory procedures for logging in samples for analysis and maintaining Chain-of-Custody are described in ELI SOP, *Sample Receipt, Login, and Labeling*.

When the laboratory has been assigned the responsibility of sample collection, there is strict adherence to correct sampling protocols, initiation of chain-of-custody, sampling documentation, complete sample identification, and prompt transfer of sample(s) to the laboratory. Procedures are described in ELI SOP, *Field Sampling*.

This laboratory provides proper sample containers and preservatives as specified for the procedure. Certified sample bottles may be ordered upon request. Sample containers, preservatives, coolers for shipping, re-sealable plastic bags for ice containment, trip blanks for monitoring contamination during shipping, temperature blanks for accurately monitoring sample receiving temperatures, Chain-of-Custody forms, Chain-of-Custody seals, sample bottle labels, instructions for sampling, sample labeling, sample preservation, and sample packaging/shipping are provided upon request. Sample container type, sample volume, preservation requirements, and maximum holding times, are detailed for each analyte/method in the ELI Technical Services and Fee Schedule. See the ELI website, www.energylab.com for the current pricing.

Energy Laboratories maintains a strict Sample Acceptance Policy. The client is immediately notified (as appropriate) upon sample receipt if there is any doubt concerning the sample's suitability for testing, including but not limited to, when:

- Samples are out of temperature compliance;
- Samples are received in unacceptable containers;
- Samples have not been properly preserved;
- Samples have labels or chain-of-custody procedures that are incomplete;
- Samples cannot be analyzed within method recommended holding time; or
- The custody seal has been broken.

Notification of sample receipt condition is available through the final report, Energy Source, Email, telephone and/or voice.

Samples not collected or documented properly can be rejected for any regulatory-based analysis with re-sampling recommended. If re-sampling is not possible, or the client cannot be contacted, the sample may be analyzed, and if analyzed, the sample will be clearly qualified in the data package.

The laboratory will preserve samples at the time of sample login if samples are unpreserved and preservation is required by the methodology. Aqueous samples for volatile analysis are checked for preservation at the time of analysis. Samples for microbiological analysis are collected in pre-sterilized 120 mL plastic bottles containing sodium thiosulfate.

Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at collection. Refer to ELI Technical Services and Fee Schedule for detailed information on sample preservation requirements per applicable method and regulatory requirements.

The laboratory initiates a sample condition report titled Login Prep Checklist at the time of sample receipt. The sample condition report contains Chain-of-Custody procedures, sample preservation status, carrier used for sample shipment, sample receipt temperature, and provides general comments concerning sample condition. In addition, the samples may be screened for potential radioactivity. The sample condition report is provided with the analytical data report package. For more information, see ELI SOP, *Sample Receipt, Login, and Labeling*.

When any sample is shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements as described in the ELI Technical Services and Fee Schedule, the Office of Hazardous Materials, Material Transportation Bureau, and Department of Transportation has determined the Federal Hazardous Materials Regulations do not apply to the following:

A) Hydrochloric Acid - (HCl) in water solutions of 0.04 % by weight or less (pH of 1.96 or greater).
B) Nitric Acid - (HNO_3) in water solutions of 0.15 % by weight or less (pH of 1.62 or greater).
C) Sulfuric Acid - (H_2SO_4) in water solutions of 0.35% by weight or less (pH of 1.15 or greater).
D) Sodium Hydroxide - (NaOH) in water solutions of 0.080% by weight or less (pH of 12.30 or less).

For regulatory compliance monitoring, it is required that all samples be analyzed within the prescribed holding times. Holding times are the maximum times allowed between sampling and analysis for results to still be considered valid. Samples should be delivered to the laboratory as soon as possible following collection to assure that holding times can be met. Samples are analyzed as soon as possible after sample receipt. When maximum holding times cannot be met, re-sampling is requested. If samples are analyzed out of hold, data is appropriately qualified.

To ensure that drinking water analysis for radiochemistry is met, the requirements for sample handling, preservation, and instrumentation for radiochemical analysis are included in ELI SOP: "*Sample Receipt, Log-In and Labeling*". (For additional information, refer to "Manual for the Certification of Laboratories Analyzing Drinking Water", Table VI-2: Sample Handling, Preservation, and Instrumentation, EPA 5th Edition, January 2005).

CHAPTER 6 – SAMPLE HANDLING

The ELI laboratory utilizes a sample tracking policy that includes client-initiated chain of custody. Upon receipt, the security of the samples is maintained by the implementation of the laboratory access and security policies. See ELI SOP, *Facility Description, Access and Security*.

Sample Receipt

All samples arriving at the laboratory are logged in the Laboratory Information Management System (LIMS). Each sample container is given a unique laboratory sample number. The sample receipt checklist evaluates Chain-of-Custody procedures, sample preservation status, carrier used for sample shipment, sample temperature, and provides general comments concerning sample condition. The completed checklist is provided with the analytical report package. Chain-of-Custody forms are checked for pertinent information. If necessary information has been omitted, the collector is notified, if possible, and the missing information is requested.

Samples requiring preservation are checked to determine if the client performed preservation. If requested, ELI staff will preserve or filter samples as appropriate. Samples that degrade quickly or cannot be opened (such as aqueous samples for volatiles) are not preserved at the time of sample login. If samples are improperly preserved, or the maximum holding times are exceeded upon arrival at the laboratory, the client is notified and re-sampling is requested.

Samples are stored per method specifications, or as method/parameter storage requirements are updated per later EPA guidance in Federal Regulations posted in 40CFR (Method Update Rules).

During sample login, all sample information such as sample description, client name and address, analyses requested, special requirements, etc. are entered into the computer database of the Laboratory Information Management System (LIMS). Requested analysis parameters and special requirements are communicated to the analysts via their LIMS work lists. Project-specific requirements are maintained in the LIMS for any samples received from a special project. This process ensures that individual requirements are maintained.

Chain-of-Custody

Evidence level internal chain-of-custody (COC) procedures are available on a project-specific basis. For these procedures, internal COC sample custody is maintained down to the individual analyst level. When transferring the possession of the samples, the transferee must sign and record the date and time on the chain-of-custody record. Every person who takes custody must fill in the appropriate section of the chain-of-custody record. When received by ELI, sample identification information on the sample containers is compared to the custody report form. The sample is inspected and information regarding the condition of the sample and seal (if used) is recorded on a report form; the method of shipping is also documented on the report form. A copy of the report form is kept with the sample data file and a copy is sent to the client with the analysis report. Internal chain-of-custody forms are used to document the progress of the sample through the laboratory. ELI's routine COC policy is maintained at the laboratory level

through our laboratory access and security policies. See ELI SOP, *Facility Description, Access, and Security*.

Sample Tracking

Samples are tracked through the analytical process by the LIMS. Completed analyses, which have been approved by the appropriate reviewer as valid data, are reported in the LIMS. When all analyses are complete, the data is reviewed as a whole to ensure results pass data quality checks. The completed report is signed by an approved signatory. The signed report is sent to the client via requested delivery format. Generation of the invoice automatically completes the work order in the LIMS and removes the samples from the status report. For more information, see ELI SOP, *Document and Record Management, Control and Archiving*.

Sample Disposal

It is preferred that remaining hazardous sample material be returned to the originator (client) for disposal. When this is not possible or reasonable, ELI will dispose of remaining hazardous sample materials with a waste disposal surcharge added to the cost of the analysis.

The disposal of laboratory wastes will be performed in accordance with local, state, and federal regulations which apply to such activities. Each method SOP addresses waste minimization and management specific to the method procedure. See ELI SOPs, *General Laboratory Waste Disposal,* and *Radiochemistry Waste Disposal* for more information.

Subcontracting Policy

The ELI Casper laboratory utilizes the expanded branch laboratory capability and expertise to provide comprehensive analytical services. This occurs when the laboratory is requested to perform an analysis outside of the laboratory's capabilities (If sample overload is experienced; if equipment is out of service; or when the laboratory is not accredited for the particular analysis). Upon completion of the analyses, the branch laboratories report the sample results, and their quality control package, to the primary laboratory. The results are reviewed before being reported.

Branch laboratories are certified to perform drinking water analysis in their state and in neighboring states. Samples are forwarded to our branch laboratories only if the laboratory is certified in the state from which the sample originated. Individual branch laboratory Quality Assurance Programs are consistent with the Corporate Quality Assurance Program and are monitored through internal laboratory audits.

To support Energy Laboratories, Inc. Casper analytical services, ELI branch laboratories (which maintain specific instrumentation for specialized analysis) are utilized to provide complete analytical services. All branch laboratory certificates are available on the Energy Laboratories website at www.energylab.com.

In the event that ELI is dependent on the service of an outside laboratory for analyses not available through our facility or our branch laboratories, the client is notified that their samples

are subcontracted to an outside laboratory. The outside laboratory reports the results to ELI and these results become part of the final report. Any external or internal subcontracted analyses that require accredited analyses will be performed by a laboratory accredited for those parameters in the State from which the sample originated. All final reports indicate where the analyses were performed.

CHAPTER 7 – INSTRUMENT OPERATION AND CALIBRATION

Laboratory instruments and equipment are operated and calibrated according to the manufacturer's instructions and according to the requirements of the method being used. Exact calibration procedures are outlined in the appropriate SOP. For most instruments, a calibration curve composed of three to five standards covering the concentration range of the samples is prepared. The acceptance criteria for the calibration curves are listed in the individual methods. Unless otherwise specified in the method, at least one of the standards is at or below the practical quantitation limit (PQL) of the method. Routine PQLs for each method are given in the ELI Technical Services and Fee Schedule. Calibration standards are routinely compared to second source calibration standards to verify accuracy. These second source standard results must fall within an established range, as described by the SOP, to be accepted. Whenever possible, the laboratory uses calibration standards prepared from certified stock standards. Initial instrument calibration curves are verified and routinely monitored by analyzing a continuing calibration standard every 10 to 20 samples (or within a specified time frequency) and at the end of every analytical sequence, depending on the analysis method and instrumentation. When applicable to the method, high-level samples, which produce an analytical response outside the calibrated range of the instrument, are diluted (or reduced in mass) and re-analyzed until a response within the calibrated range is obtained and/or the result is appropriately qualified.

System cleanliness is verified through the analysis of reagent/instrument blanks prior to analysis, between highly contaminated samples, and at regular intervals during the analysis.

Use of measuring equipment and reagents (glassware, water, chemical reagents, and industrial gases) conform to Good Laboratory Practice guidelines. Good Laboratory Practices (GLPs) are laboratory guidelines which were established by the Food and Drug Administration and published in the Federal Register (21 CFR, part 58). The GLP guidelines were adopted by the Environmental Protection Agency. SOPs are developed in accordance with GLP and NELAP guidelines. Laboratory volumetric glassware conforms to National Institute of Standards and Technology (NIST), American Society for Testing and Materials (ASTM) Class A or B standards. All mechanical pipettes are calibrated at least quarterly. Laboratory balances are serviced annually and calibrated by certified technicians. Calibration checks of balances are performed each day of use, using ASTM Class 1 or 2 weights. Laboratory thermometers are calibrated annually against a NIST traceable thermometer. Laboratory drying ovens, incubators, freezers, refrigerators, and water bath temperatures are monitored and recorded each working day, or at frequencies as described in the specific SOP. Laboratory pure water is generated by commercial water purification systems and is monitored and documented each working day in accordance with specifications needed for applicable methods. The routine analysis of laboratory blanks is used to verify laboratory water quality and the suitability of sampling containers. Chemical reagents and gases meet or exceed purity requirements for their intended uses. Laboratory stock and working standards are derived from ISO 17025 and/or 9001 (or equivalent-certified) commercially available primary standards whenever possible. Standard preparation notebooks document the reagent/standard type, source, purity, content, concentrations, preparation date, and analyst. All calibration standards are documented in the analytical records such that they are uniquely identified and traceable to stock standards and their source.

Standard Operating Procedures (SOPs) detail the sequence of operations involved in instrument start-up, calibration, analysis, shut-down, and routine maintenance. Suggestions for

corrective action are included with the SOPs and parameters are identified which dictate certain types of maintenance. Instrument and method detection limit studies are performed at the method required frequency or whenever there is a significant change in instrumentation. Method Detection Limits are determined according to EPA guidelines found in 40 CFR, part 136, Appendix B for general chemistry and 40 CFR 141.25 (c) for radiochemistry (except for the few methods that are not amenable to MDLs). Refer to ELI's Technical Services and Fee Schedule for practical quantitation limits (method reporting limits). Acceptable instrument response/performance criteria are based upon the manufacturer or the analytical method specifications. SOPs exist for all major pieces of analytical equipment/methods.

Instrument logbooks and/or electronic logbooks are used to document instrument maintenance and repairs. Instruments that are no longer being utilized are documented in the applicable instrument logbook as "out-of-service" with the date the instrument was taken out of use noted. All out-of-service instruments are labeled with an out-of-service tag that identifies the effective date the instrument was taken out of use.

Laboratory analysts record and document all instrumental runs in Laboratory Instrument Logbooks or computer files. Instrument Logbooks and/or dated computer files record instrument performance data, analytical sequences, instrument maintenance, calibration standards data, and any other additional information pertinent to operation of the instrument.

CHAPTER 8 – RECORDS AND REPORTING

Document Management

Energy Laboratories Inc. QA manages three types of documents: 1) controlled, 2) approved, and 3) obsolete.

A CONTROLLED document is one that is uniquely identified, issued, tracked, and kept current as part of the Quality System. Controlled documents may be internal documents or external documents. A list of ELI's controlled documents is listed in Appendix A. All ELI controlled documents are written and reviewed by personnel technically competent to perform that procedure and approved for use by the Laboratory Manager as well as the Quality Assurance Officer.

APPROVED documents have been reviewed, signed and dated by the technical reviewer, the Quality Assurance Officer and the Laboratory Manager or designee.

OBSOLETE documents are documents that have been superseded by more recent versions. Obsolete documents are retained for legal use or historical knowledge preservation. Old or archived SOPs are available for review using the laboratory's electronic document system. ELI's OBSOLETE document records are maintained for at least ten years.

Documents are reviewed on an annual basis to ensure their contents are suitable and in compliance with the current quality systems requirements, and accurately describe current operations. SOPs include a Record of Review/Revision page, which details revisions or reviews. The branch Quality Assurance Officer maintains a master list of controlled documents (which include title, author, and date of issue).

Procedures for identification, collection, access, filing, storage, and disposal of records are found in ELI SOP, *Document and Record Management Control and Archiving*.

Laboratory Notebooks

Several different types of Laboratory Notebooks are maintained at the ELI Laboratory. These include, but are not limited to, the following:

- Method/Parameter Notebooks
- Project Notebooks
- Instrument/Equipment Use and Maintenance Notebooks
- Standard Preparation Logbooks
- Balance Calibration Logbooks
- Pipet Calibration Logbooks
- General Logbooks

The general purpose of maintaining each of these Laboratory Notebooks is to record the details that may be important in repeating a procedure, interpreting data, or documenting certain operations. Entries in the notebook may include data such as standard and sample weights, pH measurements, instrument operating parameters, preparation of calibration curves, analytical run sequences, calculations, recording of instrument operating parameters, sample condition,

etc. The analyst's notebook is particularly important in documenting analyses that deviate in any way from routine or standard practices. It can also be an important training record. All pertinent data is to be recorded directly in the notebook. Some notebooks or data records are maintained in electronic format (LIMS, spreadsheets, or databases). Electronic data records are duplicated using hardcopy and/or alternate electronic backup techniques.

It is the responsibility of each analyst to maintain a laboratory notebook according to Good Laboratory Practices (GLP) Guidelines. All physical laboratory notebooks are assigned a unique logbook control number and are assigned to an analyst and/or supervisor. These notebooks remain the responsibility of the ELI staff member's supervisor to whom they are assigned until they are formally transferred to another staff member, until they are completely filled and returned to the ELI QA Department for archiving. ELI staff members, other than the individual to whom the laboratory notebook is issued to, may make entries in the notebook as long as those entries are consistent with the intended use of the notebook and such entries are initialed and dated. Procedures for use and maintenance of laboratory notebooks are detailed in ELI SOP, *Laboratory Notebooks*.

Records

The laboratory maintains records of all chemical analyses, including all quality control records, for a minimum of ten years. In the event that Energy Laboratories, Inc., or any individual laboratory transfers ownership or goes out of business, the records will be transferred to the new owners. If a branch laboratory is closed, records will be maintained by Energy Laboratories Corporate office in Billings, Montana. Energy Laboratories, Inc. reserves the right to offer the records to the clients in the event of complete closure. Details are described in ELI SOP, *Document and Record Management, Control and Archiving*.

Data Reduction

Data reduction refers to the process of converting raw data to reportable units. The reporting units used and analytical methods performed are described in the ELI Technical Services and Fee Schedule.

Wherever possible, the instrument is calibrated to read out directly in the units reported. In this case, the value is recorded directly into a laboratory notebook, logbook, bench sheet, or electronic file and presented for review.

In cases such as titration, gravimetric measurements, or other techniques that require calculation prior to reporting, raw data is recorded in the appropriate laboratory notebook or electronic file, or on the appropriate laboratory form. The calculations specified in the methods are used to determine the reported value. That value is also entered into the laboratory notebook or bench sheet. Most calculations are automated to reduce the chance of arithmetic or transcription errors.

Wherever possible, electronic data results are transmitted throughout the laboratory via the LIMS computer network. This process is intended to minimize manual data transcriptions within the laboratory. Additional advantages include the opportunity for rapid comprehensive data validation by supervisors, and more rapid data reporting.

Validation

Data validation includes the procedures used to ensure that the reported values are consistent with the raw data, calculated values, sample type, sample history, and other analysis parameters requested.

The data recorded is validated with several review steps. The analyst who submits the analytical results checks all the values reported for omissions and accuracy. Elements of this review also evaluate all instrument and method QC results. Automated data management programs are designed with an interactive step allowing data review by the analyst. Results to be reported are approved by the analyst.

The report is reviewed for the suitability of the data according to project and method performance specifications. Analytical results for each requested parameter may be evaluated against other requested parameters, project specifications, other samples within the set, historical files associated with the project/client, and/or any other information provided with the sample.

The reports are generated, proofread, and reviewed by designated reporting staff.

The Laboratory Manager, project managers, supervisors, Quality Assurance Manager or their designees, may also examine the data included in the final report.

Internal and external laboratory audits review selected sets of data to ensure that the analytical results are correct and accurate, analytical methods are appropriate, documentation and record keeping procedures are complete, and that there is compliance to the overall objectives of the Quality Assurance Program. Data integrity is being monitored on an on-going basis.

All controlled automated programs used to process and report data are initially verified using manually calculated results. Whenever a modification is performed to a program, re-verification of overall software function is performed.

One step of the Quality Control process involves data outlier detection; data that falls outside of established limits. If an outlier is observed, corrective action is taken as appropriate, to investigate and/or correct the cause. Actions to correct these causes may include, but are not limited to, inspection of the instrumentation, checking calibrations, checking sample numbers or dilutions, re-analyzing samples or calibrations.

Reporting

One copy of the report is distributed to the client, via requested delivery format, after the report is validated and signed. A standardized report format is used unless otherwise specified. Client-specified report formats are available upon request. Results can be sent via physical media, email, EDD, website FTP and/or FAX when requested by the client. Energy Laboratories, Inc. offers its clients access to electronic records through our Energy Source Portal.

Various levels of data reporting are available. All analytical results, regardless of the level of reporting used, have record keeping procedures which allow an appropriate "data validation package" to be produced. Note that a comprehensive "data validation package" is most easily generated at the time of sample analysis. Example data packages are available upon request.

Safe Drinking Water Act (SDWA) compliance monitoring samples for microbiological and chemistry samples that exceed the SDWA maximum contaminant level (MCL) may require notification to the appropriate state agencies. Generally, notification to the client, and to the state, of any SDWA MCL exceedance must be within 24 hours of completion of analysis/review, or by noon the next business day. If requested by the client, additional copies of the report will be sent to a specified address or person.

The final copy of a completed report is maintained in an electronic format. An electronic copy of this file is available upon request. Energy Source is a client resource of ELI that provides secure online access for clients to view their data and documents. Clients are able to access their electronic files through ELI's secure website at https://energysource.energylab.com/. For more information, see ELI SOP, *Document and Record Management, Control and Archiving*.

In addition to traditional ink signatures, Energy Laboratories has approved the use of electronic signatures within our company-produced PDF documents. These signatures comply with Title 15 of the US Code Section 101 regarding legal requirements of a digital signature.

Electronic signatures verify that the document has not changed after it was produced. Upon opening the document, notifications automatically display to inform the recipient of the validity of the sender's electronic signature and all included certificates. Should any changes be detected, an alert message is automatically displayed, noting that the signatures cannot be validated due to changes made to the document. Detailed instruction on how to view/validate ELI's electronic signatures is available.

CHAPTER 9 – GENERAL LABORATORY PRACTICES

Chemicals and Reagents

When available and appropriate, chemicals used in the laboratory are analytical reagent grade (AR) chemicals purchased from reliable suppliers. Reagents are prepared, standardized, and made fresh as mandated by the method, their stability, and according to Good Laboratory Practices. Procedures for purchasing of materials may be found in ELI SOP, *Property Procurement, Inventory, and Control*.

Normalized standards are checked regularly against independently prepared reference materials.

All standards and reagents are dated when received, opened, or prepared, and each is labeled with an expiration date when applicable. Standards and reagents are checked for discoloration or signs of degradation and are discarded if these are observed.

Certified primary standards are obtained from ISO accredited commercial sources when available. Standards used for calibration are verified against second source standards. Secondary and working standards are accurately prepared with volumetric flasks, or other calibrated glassware, from primary standards and stored in appropriate containers.

ELI has determined 10 years to be a reasonable expiration date for stable salts where the manufacturer does not supply such information. Titrants, standards, and other solutions used for analytical purposes are frequently standardized upon preparation with certified or traceable standards. Method SOPs specify if standardization is necessary. The date and analyst's initials must be recorded on the container whenever re-standardized and these records are maintained in a laboratory notebook or in the LIMS.

Individual SOPs may also provide additional details for reagent requirements.

Reagent Interference

To determine the extent of reagent interference, method blanks are analyzed prior to sample analysis whenever appropriate.

If any interference cannot be eliminated, the magnitude of the interference is considered when calculating the concentration of the specific constituent in the sample, but only when permitted within the applicable method.

If reagents, materials, or solvents contain substances that interfere with a particular determination, they are replaced.

Individual method SOPs may also provide additional requirements for handling reagent interferences.

Glassware Preparation

All glassware used for inorganic and radiochemical analysis is washed in warm detergent solution and thoroughly rinsed in tap water. Glassware is then rinsed well three times with laboratory-purified water. This cleaning procedure is sufficient for many analytical needs, but individual SOPs detail additional procedures when necessary. Glassware washing procedures for various analysis are described in ELI SOPs, *Cleaning of Glassware Used in Inorganic Analyte Sample Preparation and Analysis*, *Cleaning of Glassware Used in Volatile and Semivolatile Analyte Sample Preparation and Analysis*, and *Cleaning of Glassware Used in Radiochemical Sample Preparation and Analysis.*

All glassware used for organic analysis is washed in warm synthetic detergent solution and thoroughly rinsed in tap water. The glassware is then rinsed well with laboratory-purified water, followed by rinses with acetone to remove any residual organics. Prior to use, the glassware is rinsed three times with the organic solvent to be used with the glassware.

All glassware used for microbiological analysis is washed in warm detergent solution. The detergent must be proven to contain no bacteriostatic or inhibiting substances. The glassware is rinsed thoroughly with laboratory-purified water. Specific details are described in method specific SOPs.

Disposable, glassware/plasticware is preferred for many procedures in the laboratory. The cleanliness and suitability of disposable glassware/plasticware is continuously evaluated for each test with the routine analysis of method blanks.

All volumetric glassware used in precise measurements of volume is Class A or laboratory calibrated.

Laboratory Pure Water

Laboratory-purified water is used in the laboratory for dilution, preparation of reagent solutions and final rinsing of glassware. For organic analysis, organic-free water is prepared and used. Energy Laboratories, Inc. uses water purification systems that are designed to produce deionized water that meets the requirements of the methods. Use and maintenance of laboratory reagent water systems are described in ELI SOP, *Use and Maintenance of the Laboratory Purified Water System.*

Water quality is monitored for acceptability in the procedure in which it is used. Specific details are listed in the appropriate SOPs.

Employee Training

All new ELI employees and contract personnel are given an initial general orientation and tour of the laboratory facilities. Personnel are shown the locations of safety equipment such as safety showers, eye wash fountains, fire extinguishers, and first aid supplies. Personal protective equipment such as lab coats, disposable gloves, and safety glasses (if applicable) are issued at this time.

Safety considerations are a vital part of the training process. All hazards associated with the performance of a procedure or with the operation of an instrument are to be understood by the

trainee before training can be considered complete. General laboratory safety procedures are a part of the new and current employee training. Specific safety procedures are outlined in SOPs and in instrument Operator's Manuals. Training in use of protective clothing, eye protection, ventilation, and general safety are provided to each employee. Each employee is required to read and sign the *Laboratory Safety Manual & Chemical Hygiene Plan*.

All new and existing employees must demonstrate capability prior to performing an analytical procedure independently (see Chapter One). Method performance on Quality Control Samples is used to document employee training and work quality. Employees are required to read the Quality Assurance Manual and all appropriate SOPs. Employees are required to sign a Quality Assurance Manual Acknowledgement form which states that they have read, understood, and will comply with the requirements of the Quality Assurance Manual. Employees also are required to sign, for all applicable SOPs, a Record of Acknowledgement Form that states they have read, understood, and agree to abide by the SOP. In the case of method SOPs, the employees sign a Record of Acknowledgement form that states they have read, understood, and agree to abide by the SOP.

Employees also receive training on general laboratory policies including ethics and conflict of interest. All employees are required to read, understand and comply with the Corporate Compliance & Ethics Manual. Data integrity training is provided for all employees initially upon hire and annually thereafter. In addition to the *Corporate Compliance & Ethics Manual*, the ELI Quality Assurance department maintains a *Laboratory Ethics & Data Integrity Manual*, which supplements the corporate manual and provides specific training on data integrity. All employees are required to read, understand and comply with the ELI *Laboratory Ethics & Data Integrity Manual*. An annual Ethics training course is given to all laboratory employees. Attendance is required and is recorded with a signature attendance sheet or other form of documentation that demonstrates all staff have participated and understand their obligations related to data integrity and ethics policies. For details pertaining to ethics training and additional ethical procedures and policies refer to ELI SOP, *Personnel Training and Training Records*.

ELI encourages attendance at courses, workshops and other forms of continuing education available from on-site seminars, private institutions, local schools, and State and Federal regulatory agencies. Staff and department meetings are held routinely to communicate company policies and procedures. All training on procedures and policies is documented, per NELAP guidelines, in employee training files. For more information see ELI SOP, *Personnel Training and Training Records*.

Data Integrity

In order to provide for the integrity of ELI and client data, the laboratory has multiple controls on the network, LIMS and applications used. These controls limit access to and the ability to change data as well as provide for redundancy in case of loss.

These include but are not limited to:

- Users connecting to ELI computer systems are authenticated through a user name and password combination.
- Passwords are required to be changed on a regular basis.

- Permissions within ELI applications are role based with different roles having various levels of access and control. Users (analysts, supervisors, and managers) are assigned to these roles.
- In the LIMS, analytical data locks after a period of time and cannot be modified without special handling.
- Certain information has been identified for additional tracking and logging. Changes to this information is not only tracked in an audit log but also reported to select personnel.
- Information on ELI servers including the ELI LIMS system is backed up and recoverable.

Standard Operating Procedures

Laboratory operations and procedures are documented in Standard Operating Procedures (SOPs). SOPs provide information on the consistent and safe operation of the laboratory... For analytical methods, SOPs provide information on the details of the analysis that is not specified in a published analytical method. For routine procedures other than analytical methods, SOPs define the steps required in accomplishing a given task. All SOPs are reviewed and updated periodically to reflect any changes in laboratory operations. Method SOPs follow NELAP requirements. For more information on generation and distribution of SOPs, see ELI SOP, *Preparation, Numbering, Use, and Revision of Standard Operating Procedures*.

Client Confidentiality

Each employee has the responsibility to maintain confidentiality in all matters pertaining to our clients, samples submitted, and Energy Laboratories, Inc. Information obtained during employment with this laboratory, regarding the specific business of this laboratory, or its clients shall at no time be revealed to any outside sources without permission from the owner of the data.

Sample submittal, analysis and the report contents are considered confidential information of the client. When requested to provide results (either in person, via telephone or email), the employees shall verify that the requestor is either the person associated with the project, on the COC, or on a list provided by the client who are authorized to receive data. If a person who is not associated with the project personnel (or is not on the approved list), the base client will be contacted to inquire about authorization to release data. These contacts are documented and associated with the work order in the LIMS system to provide archival proof of authorization to release data. If the client does not authorize a release of data, the requestor will be contacted and informed of this decision.

Client confidentially is maintained electronically through the use of password-protected logins on all laboratory computer systems. Additionally, the laboratory maintains network security such as anti-virus programs and firewalls that prevent any unauthorized outside access. All copies of the original report are stored on the laboratory's document archival system, which is also protected from unauthorized use by the network security systems. Raw data, reports, and LIMS records are kept in a secure location of the laboratory or off-site. All client confidential paper waste, including printouts, is shredded.

CHAPTER 10 – QUALITY CONTROL MONITORING

Routine Monitoring

Temperatures of incubators, water baths, refrigerators, and ovens are checked and recorded according to a prescribed schedule using an automated monitoring system. In the event that the automated monitoring system is inoperable for more than twelve hours the appropriate temperatures will be recorded manually on instrument specific forms.

Conductivity of the laboratory-purified water is continuously monitored using an automated monitoring system and as method blanks in routine analytical runs. In the event that the automated monitoring system is inoperable for more than twelve hours the recorded method blanks will serve as the required monitoring.

Reagents are dated and initialed at the time of receipt. Expirations dates are assigned as a fundamental component of their receipt and/or preparation. Reagents are not used after manufacturer's expiration date is exceeded.

Balances are checked daily, or as required, against ASTM Class 1 or 2 NIST traceable weights and are calibrated and serviced by certified technicians annually.

SOPs are reviewed periodically for accuracy.

Laboratory Notebooks are reviewed periodically for correctness and accuracy by supervisors.

Proficiency Testing (PT) Samples are analyzed as required (See Chapter Two of this QA Manual).

Quality Control Check Samples are analyzed with each analytical batch.

Internal and external audits are performed as specified or requested (See Chapter Two of this QA Manual for additional discussion).

Additional monitoring requirements may also be specified in individual SOPs.

The Laboratory maintains an active fraud protection program that is implemented through the laboratory ethics policy. Additionally, the potential of fraud is monitored through analyst supervision, management supervision, regular internal audits, PT study participation, and an active quality assurance program.

Instruments/Methods

Calibration is performed as outlined in Chapter Seven of this QA Manual.

Generally, and depending on method requirements, the standard curve is verified with a known second source reference sample. The reference sample results must fall within the appropriate target range for the calibration to be accepted.

In most cases, the calibration stability is checked by analyzing a continuing calibration standard every 10 to 20 samples, depending on the analysis and instrumentation. The verification standard results must fall within an established range as described by the SOP.

All laboratory instruments are subjected to preventive maintenance schedules. Preventive maintenance schedules are specified in instrument maintenance logbooks.

As appropriate, instrument and/or method detection limits are determined annually, or more frequently if changes in instrument performance are noted or per method requirements. Procedures for the determination of instrument detection and method detection limits are described in ELI SOP, *Determination of Method Detection Limits (MDL) and Quantitation Limits*.

Precision and accuracy requirements for each method are specified in the SOPs. General guidelines are given below.

- Each analytical batch will contain QC samples to measure the accuracy of the method. Each QC sample result is monitored to be within QC specifications of the method. Results of blank spiked sample analysis must be within the established control limits. Quality Control Limits are specified in the SOPs and meet recommended QC limits as described in the referenced method.

- Each analytical batch will contain QC samples to measure the precision of the method. (See Chapter One for discussion on duplicate sample analysis.) Criteria for duplicate sample acceptance are found in the SOP and are generally taken from the referenced method.

- Each analytical batch will contain QC samples to measure the performance of the method on the sample matrix. These are typically identified as a matrix spike analysis and may be performed in duplicate to assess method precision. Typically the sample is fortified with a known amount of target analyte and spike recoveries are calculated. Results outside of method QC guidance are flagged. Quality control limits and appropriate corrective actions steps are specified in the method SOP.

- Several methods are considered to be concurrent methods in that they are either nearly identical or are identical to a method with a different citation. Even if two methodologies are identical in procedure, slight differences in the QC requirements might be the only difference between the two methodologies. These types of methods may also be considered "concurrent" if the procedures are identical and the more stringent of the two method criteria are used. During data reduction and reporting, the referenced method specifications and criteria will always take priority.

As appropriate, the performance trends of QC sample results are evaluated with Quality Control Charts. Suitability of existing QC limits is evaluated and possibly adjusted, but not to exceed method specification.

CHAPTER 11 – CORRECTIVE ACTION

When the quality control checks indicate that an analysis is not within the established control limits, corrective action is needed. This section gives general guidelines for corrective action. Corrective actions for each method or instrument are detailed in individual SOPs. Records are maintained of non-conformances requiring corrective action to show that the root cause(s) was investigated, and includes the results of the investigation. The QA Officer will monitor implementation and documentation of the corrective action to assure that the corrective actions were effective.

Method QC samples that fail to fall within QC control limits may be analyzed again to verify if a problem exists. However, matrix spike or matrix spike duplicate QC samples are not required to be re-analyzed if the performance can be attributed to matrix effects; data results are then reported and flagged.

If the repeat analysis is not within control limits, the particular instrument or procedure is checked according to the specific protocols outlined in the method or according to the instrument manufacturer's guidelines. Once results are within control limits, analysis of all samples that were analyzed while the procedure was out of control are repeated, i.e., all analyses are repeated back to the previous acceptable control sample. In the case of radiochemical analysis, the term “analyze again” means to recount the final sample on the same (or different) detector.

If the analyst is unable to achieve acceptable results after following the corrective action guidelines detailed in the SOP, a supervisor and/or the Technical Director is consulted. If necessary, the appropriate service personnel are contacted if the problem is determined to be due to instrument error, and cannot be resolved. It is also possible that the result is due to statistical variation of the results based on the tolerable error rate that has been determined for the analysis (usually 0.05). In certain cases, where control limits are exceeded, it is possible that problems cannot be corrected to satisfy QC criteria. This could be due to problems such as matrix interference, instrument problems, lack of sufficient sample, missed holding times, high blank contamination, etc. If all possible solutions available to correct the problem are examined and the sample results are still considered valid, qualifying comments are attached to the sample report describing the non-compliance and probable cause.

In the case of a single radiochemistry detector being returned to service, this refers only to the samples counted on that detector. For example, an individual gas proportional counter instrument may have up to 16 detectors; if only one does not pass the QC check the others are still valid and sample analyses performed on the others do not need to be repeated.

In the event that a QC audit or other informational review shows an analysis report to be incorrect, incomplete, or adversely compromised, a revised report and explanation is submitted to the client within ten business days unless otherwise communicated to the client with another time period. The report will clearly be identified as a revised report. As appropriate, an explanation submitted to the client should give a detailed review of the problem and document any unapproved deviations from the regulations, standard operating procedures, or project-specific scope of work that may have caused it. The explanation to the client may include, but not be limited to, the following components:

1) What actions have been taken regarding the affected data set(s),
2) Identification of the cause, and
3) Corrective action(s) taken to prevent future occurrence.

In the event that a QC check fails, the analyst will follow the procedures outlined in the QA/QC summary of the SOP.

Quality Control Checks for each method or instrument may vary. Energy Laboratories Inc. follows the QC checks set by each governing method. Due to the wide variations between methods, specifics are listed within each SOP for the given method. Please reference the SOP for specific QC checks for the given method. The QC checks may include: ICV, MB, CCV, CCB, LCS, LCSD, LOD, MS, MSD or others specific to that method.

A general summary of Quality Assurance/Quality Control specifications is outlined in Appendix B. Exact details of method QC can be found in the applicable method SOPs.

Procedure for Dealing with Complaints

DEFINITIONS

Complaint: For the purposes of this procedure, a complaint comes from a client, a user of our data, or employee. The complaint might cover issues about the quality of our data, sample turnaround time, method used, pricing, or other expectations.

Client: The client is a person or company that ordered and paid for the services.

Procedure: The staff person receiving the complaint exercises judgment in deciding the severity and disposition of every complaint. The judgment must be used to decide whom, if anyone, is alerted to the complaint and what actions are appropriate. The complaint issued should be handled with a high degree of discretion and tact by the supervisor or manager involved. The individual handling the complaint is instructed to follow ELI's guidelines provided in this section on how to handle the complaint. This involves listening to the client and getting adequate information so the complaint can be investigated and resolved. The appropriate laboratory staff is notified and a solution to the problem, as well as a timeline for action, is given.

After the complaint is investigated or resolved, as necessary, the client is made aware of the results and determination is made as to what further actions are needed. Complaints and investigations may result in the need to submit a revised report or invoice. Complaints that are straightforward and can be resolved using the resources available to the person handling the complaint should be resolved there. These include such things as minor revisions of reports or invoices. If other decisions need to be made, the appropriate person should be contacted.

It may be appropriate to initiate or prepare a non-compliance report. This report should be completed with the intention of informing the affected staff about the problem from which everyone may learn, change our procedures and improve our service. A procedure to document non-compliance reports is documented in ELI SOP, *Nonconformance Procedures and Corrective/Preventive Action Reports.*

If an employee sees an issue, they are encouraged to report concerns regarding Quality Systems, unethical behavior, and/or financial mismanagement. This issue should initially be brought to the attention of their supervisor. The supervisor will take appropriate action to resolve the concern. If the employee is uncomfortable with approaching their supervisor or feels that the issue was not properly dealt with, they may approach higher levels of management with their issue.

Energy Laboratories, Inc., has also implemented a program to facilitate confidential reporting to upper management. This tool allows employees to report situations or behaviors that they consider to be unethical, immoral, or improper. It also allows the reporting of suggestions or comments. The program has been implemented at ELI so that anyone reporting a situation can be assured that there will not be retaliation for reporting. It is meant to encourage parties to communicate with upper management when there appears to be no alternative for resolving the types of issues already described. . Access to the program is available on the ELI internal and external websites as well as through a 24-hour telephone hotline number (877-874-8416).

Penalty for Improper, Unethical or Illegal Actions

Energy Laboratories, Inc. employees are expected to work in an ethical, proper, and legal manner. They are expected to perform laboratory analyses according to the cited method(s) and in conjunction with the SOP and the Quality Assurance Plan. Employees are expected and required to report any violations of this policy. All employees are mandated to participate in an ethics-training program as part of their orientation upon hire.

Improper, unethical, or illegal actions by an employee will be addressed on a case-by-case basis as determined by the seriousness of the offense. Corrective actions may include disciplinary action up to and including discharge.

CHAPTER 12 – MANAGEMENT OF CHANGE

Management of change is the process used to review and manage proposed changes to materials, technology, equipment, procedures, personnel and facility operations. These changes may be permanent or temporary depending on circumstances. Change is managed, communicated, and documented as appropriate to the level of change, by the Laboratory Manager and the Supervisors of each department. Significant revisions to controlled documents may require employees to sign a record of acknowledgement.

- New Equipment Validation – Documented in the Instrument Maintenance Module. Supporting studies are documented in the LIMS.
- Implementation of new test methods and method updates – Documented in the method SOP and Instrument Maintenance Module. Supporting studies are documented in the LIMS.
- The QA Manual and SOPs – Documented in the Record of Revision and stored in the Document Control Software.
- Work order changes are documented in the work order report and stored in the LIMS or Document Control Software.
- LIMS changes - documented in a version control repository.

  Personnel changes - documented in employee training records or personnel records.

CHAPTER 13 – MAJOR EQUIPMENT AND METHODS

A summarized listing of major instrumentation utilized is included in Appendix E. Refer to the ELI Technical Services and Fee Schedule, located on the ELI website at http://energylab.com, for a list of all methods and analyte parameters that Energy Laboratories, Inc. performs.

CHAPTER 14 – PREVENTIVE MAINTENANCE

Preventive maintenance is performed on laboratory equipment according to the manufacturer's guidelines and our operational experience. Repairs and maintenance are accomplished in-house by experienced laboratory personnel whenever possible. Other than consumable equipment items, an inventory of spare parts is not maintained. Spare parts are available from outside vendors on an as needed basis. (To ensure method capability, some methods have more than one instrument available). An example of maintenance performed follows:

Instrument	**Maintenance**	**Frequency – Note that Daily is based on use.**
Balances	Check with Class I weights	Daily
	Independent Service	Annually
Pipettes	Check volume	Quarterly/Daily
ICP-Atomic Emission	Check Pump Tubing	Daily
	Check Coolant Levels	Daily
	Lubricate Autosampler	Quarterly
	Air Filter	Quarterly
	Optics Servicing	As needed
ICP-Mass Spectrometry	Check Pump Tubing	Daily
	Check Coolant Levels	Daily
	Check Electron Multiplier	Monthly
	Lubricate Autosampler	Quarterly
	Air Filter	Quarterly
Gas Chromatograph	Change Septum	As needed
	Check Injection Liner	Daily
	Clean Detector	As needed
	Change Gas Cylinders	At 200 psi
	Change Column	As needed
Auto Analyzers	Check Bath Temperature	Daily
	Check For Leaks	Daily
	Change Tubing	When wear is visible
	Align Flow Cell	Quarterly
	Lubricate Pumps	Annually
	Lubricate Sampler	Annually
Mass Spectrometers	Monitor Vacuum Pressures	Daily
	Monitor Background Levels	Daily
	Monitor Electron Multiplier	Daily
	Change Pump Oil	Annually
Microbiology	Monitor Room Temperature	Twice daily
	Monitor Incubator Temperature	Twice daily
	Autoclave Maintenance	Annually
	Monitor Water Bath Temperature	Twice daily
Reagent Water Systems	Change/Check Cartridges	Quarterly, or as needed
Compressed Gases	Change Gas Cylinders	At 200 psi, monitor daily
Liquid Chromatograph	Flush System	Daily
	Change Filters	As needed
	Change Lamps	As needed
	Replace Seals	As needed
Radiochemistry Detection Equipment	Check Background	Daily
	Check Efficiency	Daily
	Clean Window	As needed
	Change Gas Cylinders	At 200 psi

CHAPTER 15 - REFERENCES

ANSI N42.23-1996, American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories.

ASTM Annual Book of Standards, Part 31 (water), American Society for Testing and Materials.

ASTM D 7282-06 Standard Practices for Set-up, Calibration, and Quality Control of Instruments Used for Radioactive Measurements.

Handbook for Analytical Quality Control in Water and Wastewater Laboratories, Environmental Protection Agency. EPA 600/4-79-019

ELI Technical Services and Fee Schedule, Current Revision, Energy Laboratories, Inc.

Manual for the Certification of Laboratories Analyzing Drinking Water, 5th Ed., EPA 815-R-05-004, 2005.

Manual for the Certification of Laboratories Analyzing Drinking Water, Supplement to 5th Ed., EPA 815-F-08-006, June 2008.

Methods for Chemical Analysis of Water and Wastes Environmental Protection Agency, 600/4-79-020.

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

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

Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, December 1998.

Methods for the Determination of Organic Compounds in Drinking Water – Supplement I, EPA/600/4-90/020, July 1990.

Methods for the Determination of Organic Compounds in Drinking Water – Supplement II, EPA/600/R-92/129, August 1992.

NELAC Chapter 5: Quality System Standard, 2003 or most current version approved by Florida and Texas NELAC Accreditation program.

NELAP, National Environmental Laboratory Accreditation Program http://www.nelac-institute.org/newnelap.php

Qualifications Manual, Current Revision, Energy Laboratories, Inc.

Standard Methods for the Examination of Water and Wastewater; 20th, 21st and -22nd Editions, APHA.

Technical Notes on Drinking Water Methods, EPA/600/R-94/173, October 1994.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW846), Environmental Protection Agency. http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm

TNI Standard, Volume 1 (EL-V1-2009), The NELAP Institute.

CHAPTER 16 – GLOSSARY OF TERMS

Accuracy - The degree of agreement between an observed value and an accepted reference value.

Analyst - The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Sample - Any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification, and continuing calibration blank.

Audit or Assessment- A systematic evaluation to determine the conformance to quantitative specifications of some operational function or activity.

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 matrix, meeting the criteria above. An analytical batch is composed of prepared environmental samples, extracts, digestates, or concentrates, which are analyzed together as a group.

Blank (BLK) - A sample of clean water that accompanies the samples through different aspects of sampling and/or sample preparation. It is used to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value. There are various types of blanks: equipment blank, field blank, instrument blank, method blank, and reagent blank.

Blank Spike - See Laboratory Fortified Blank.

Blind QC Check Samples - Samples whose analyte concentrations are not known to the analyst. That the sample is a QC check sample may or may not be known to the analyst.

Calibration - The set of operations that establish, under specified conditions, the relationship between values indicated by the measuring instrument and the corresponding known value of the property being measured.

Calibration Blank - A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

Calibration Check Standard - See Check Standard.

Calibration Curve – The graphical relationship between the known values and the instrument responses for a series of calibration standards.

Calibration Standard - A solution of known concentration used in the calibration of an analytical instrument.

Chain of Custody Form- A 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; collector; time of collection; preservation; and requested analyses.

Check Standard - A material of known composition that is analyzed concurrently with test samples to evaluate a measurement process.

Clean Water Act - Public Law PL 92-500. Found at 40 CFR 100-140 and 400-470. The act regulates the discharge of pollutants into surface waters.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) - The enabling legislation (42 USC 9601 - 9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 USC 9601 et seq.), to eliminate the health and environmental threats posed by hazardous waste sites.

Continuing Calibration Blank (CCB) – See Check Standard.

Continuing Calibration Standard - See Check Standard.

Continuing Calibration Verification (CCV) - See Check Standard.

Control Limits - A range within which specified measurement results must fall to be compliant.

Control Standard - See Check Standard.

Corrective Action (CA) - An action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Data Quality Objectives (DQO) - An integrated set of specifications that define data quality requirements and the intended use of the data.

Demonstration of Capability (DOC) - A procedure to establish the ability of the analyst to generate data of acceptable quality.

Detectability – For radiochemical analysis, detectability as a Lower Limit Detection (LLD) or Minimum Detection Concentration (MDC), is assessed based on the requirements of 40 CFR 141.25(c) and is a sample-specific determination. The equation is specific for each method and noted in the method SOP.

Detection Limit - See Practical Quantitation Limit and Method Detection Limit. Reporting of detection in radiochemistry is based on specific formulas identified in individual procedures. Single activity point standards are used for efficiency calibration. When required, multiple energy emitters are used for energy calibration.

Document Control - The act of ensuring that documents and revisions 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.

Duplicate (DUP) - A second aliquot of a sample that is treated the same as the original sample to determine the precision of the method.

Duplicate Sample - See Duplicate.

Fortified Sample - See Matrix Spike.

Holding Times (Maximum Allowable Holding Times) - The maximum time that samples may be held prior to analysis and still be considered valid or not compromised.

Initial Calibration Verification (ICV) - A sample of known concentration, from a source other than that of the calibration standards, analyzed following calibration to demonstrate validity of the calibration.

Instrument Blank - See Calibration Blank.

Internal Standard – 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.

Laboratory Control Sample (LCS) – A sample with a known concentration prepared and/or analyzed as a measure of accuracy for the method.

Laboratory Fortified Blank (LFB) – An aliquot of reagent water to which known quantities of specific compounds are added and which is analyzed as a measure of method recovery.

Laboratory Inter-comparison Sample - A performance evaluation sample analyzed by numerous laboratories. Acceptance criteria are often based statistically on the analysis results.

Limit of Detection (LOD) - For chemical analysis, the LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent.

Limit of Quantitation (LOQ) – For chemical analysis, the LOQ is an estimate of the minimum amount of a substance that can be reported with a specified degree of confidence. An LOQ is an evaluation of precision and bias.

LIMS - Laboratory Information Management System.

Matrix – The substrate of a test sample.

Matrix Spike - (MS) – An aliquot of a sample to which known quantities of specific compounds are added, and which is carried through the entire analytical process to determine the effect of the matrix on the methods recovery efficiency.

Matrix Spike Duplicate (MSD) – A second aliquot of a sample to which known quantities of specific compounds are added, and which is carried through the entire analytical process to determine the effect of the matrix on the method's recovery efficiency and the precision of the method.

Maximum Contaminant Level (MCL) – Regulatory action levels for a contaminant of concern.

Method Blank (MBLK)- A clean sample processed simultaneously with, and under the same conditions as, samples being tested for an analyte of interest through all steps of the analytical procedure.

Method Detection Limit (MDL) - A measure of the limit of detection for an analytical method determined according to the procedure given in 40 CFR Part 136 Appendix B.
Method Validation - The confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled (NELAC 2003) (MARLAP 2004 for radiochemical methods).

NELAC - National Environmental Laboratory Accreditation Conference.

NELAP - National Environmental Laboratory Accreditation Program.

NPDES - National Pollutant Discharge Elimination System- A discharge permit system authorized under the Clean Water Act.

Performance Evaluation (PE) Sample - A sample with a composition unknown to the analyst that is provided to test whether the analyst/laboratory can produce analytical results within specified limits.

Practical Quantitation Limit (PQL) – 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.

Precision - The degree to which a set of observations or measurements of the same property conform to themselves.

Preservation - Refrigeration and/or reagents added at the time of sample collection to maintain the chemical and/or biological integrity of the sample.

Proficiency Testing (PT) Sample - A sample with a composition unknown to the analyst which is provided to test whether the analyst/laboratory can produce analytical results within specified limits.

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.

Quality Assurance Project Plan (QAPP) - A formal document describing the detailed quality control procedures pertaining to a specific project. For environmental clean-up projects, this is typically produced by an engineering firm with references to include a laboratory's Quality Assurance Manual.

Quality Control – 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 users.

Quality Control Sample – A sample used to assess the performance of all, or a portion, of the measurement system.

Replicate - See Duplicate.

Reporting Limit (RL) –. The lowest level of concentration reported for an analyte.

Resource Conservation and Recovery Act (RCRA) - The enabling legislation under 42 USC 321 et seq. (1976) that gives EPA the authority to control hazardous waste.

Safe Drinking Water Act (SDWA) - The enabling legislation, 42 USC 300f et seq. (1974), which requires the USEPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sample (SAMP) - A portion of material to be analyzed.

Spiked Sample – See Matrix Spike.

Standardization - See Calibration.

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 certain routine or repetitive tasks.

TNI – The NELAC Institute

Traceability – The property of a result of a measurement whereby it can be related to appropriate standards.

Trip Blank - One type of Field Blank. An aliquot of analyte-free water or solvent transported to the field in a sealed container and returned to the laboratory with the sample containers.

Acronyms and Abbreviations

AA	-	Accrediting Authority
AB	-	Accrediting Body
ANSI	-	American National Standards Institute
AOAC	-	The Scientific Association Dedicated to Analytical Excellence
APHA	-	American Public Health Association
ASQC	-	American Society for Quality Control
ASTM	-	American Society for Testing and Materials
Bq	-	Becquerel
BLK	-	Blank
Bg	-	Background
°C	-	Degrees Celsius
Cal	-	Calibration
CAS	-	Chemical Abstract Service
CCB	-	Continuing Calibration Blank
CCV	-	Continuing Calibration Verification
COC	-	Chain of Custody
DOC	-	Demonstration of Capability
DO	-	Dissolved Oxygen
DQO	-	Data Quality Objectives
DMRQA	-	NPDES Discharge Monitoring Report Quality Assurance
DUP	-	Duplicate
ELI	-	Energy Laboratories, Inc.
EPA	-	Environmental Protection Agency
FDA	-	Food and Drug Administration
g/L	-	Grams per Liter
GC	-	Gas Chromatography
GC-MS	-	Gas Chromatography-Mass Spectrometry
ICP-AES	-	Inductively Coupled Plasma Atomic Emission Spectrophotometry
ICP-MS	-	Inductively Coupled Plasma-Mass Spectrometry
ICV	-	Initial Calibration Verification
ISO	-	International Organization for Standardization
LCS	-	Laboratory Control Sample
LFB	-	Laboratory Fortified Blank
LIMS	-	Laboratory Information Management System
LLD	-	Low Limit Detection
LOD	-	Limit of Detection
LOQ	-	Limit of Quantitation
MDC	-	Minimum Detection Concentration
MDL	-	Method Detection Limit
MBLK	-	Method Blank
MS/MSD	-	Matrix Spike/Matrix Spike Duplicate
NELAC	-	National Environmental Laboratory Accreditation Conference
NELAP	-	National Environmental Laboratory Accreditation Program
NIOSH	-	National Institute for Occupational Safety and Health
NIST	-	National Institute of Standards and Technology
NPDES	-	National Pollutant Discharge Elimination System
OSHA	-	Occupational Safety and Health Administration
pCi/L	-	Picocuries per Liter
PT	-	Proficiency Testing
QA/QC	-	Quality Assurance / Quality Control
QS	-	Quality Systems
QAM	-	Quality Assurance Manual
QAPP	-	Quality Assurance Project Plan

RDL	-	Required Detection Level
RCRA	-	Resource Conservation and Recovery Act
RL	-	Reporting Limit
RPD	-	Relative Percent Difference
RSD	-	Relative Standard Deviation
SOP	-	Standard Operating Procedure
SPK	-	Spike
Std	-	Standard
SVOC	-	Semi-Volatile Organic Compound
TNI	-	The NELAC Institute
ug/L	-	Micrograms Per Liter
UV/VIS	-	Ultraviolet/Visible Spectroscopy
VOC	-	Volatile Organic Compound
WET	-	Whole Effluent Toxicity

APPENDIX A

Quality Systems Controlled Documents

Quality Systems Controlled Documents

- Quality Assurance Manual
- Radiation Safety Manual
- Laboratory SOPs
    - Organization and Personnel (10-Series)
    - General Facility Operations (20-Series)
    - General Laboratory Procedures (30-Series)
    - Equipment Use and Maintenance (40-Series)
    - Analytical Methods (50-Series)
- Laboratory Notebooks (Quality Assurance Department)
- Qualifications Manual (Corporate Document)
- Energy Laboratories Technical Services and Fee Schedule (Controlled by Corporate Marketing Department)
- Employee Policies and Procedures Package (Controlled by Corporate Human Resource Department)
- Corporate Safety Policies and Programs (Controlled by Corporate Safety Officer)

APPENDIX B

QUALITY ASSURANCE / QUALITY CONTROL SPECIFICATIONS

**These are general QA/QC parameters. The table must be tailored for each specific method, as not all sample types, frequency or acceptance criteria may apply. Where frequency, acceptance criteria or comments have multiple options separated by "OR", please choose only one of the options. Use the verbiage and formatting of this table wherever possible. **

Method QA/QC Parameters
Method Name
Method Number

QA SAMPLE/ SAMP TYPE CODE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Instrument Calibration	Daily OR Monthly OR Every 6 months After maintenance or when needed due to peak shifts or QC failures.	r ≥ 0.995 Also includes visual interpretation for quadratic or higher order calibration fit types.	1) Prepare/Purchase new standards 2) Recalibrate 3) Perform instrument maintenance	Establishes calibration curve over a range of analyte concentrations to quantify analytes of interest. Minimum 3 calibration points and a blank (depending on method) required.
Linear Calibration/Dynamic Range (LCR/LDR)	Initially, then every 6 months. Or with major changes in equipment. Or as required by method.	Residuals (Percent Recovery of standards) recommended being within CCV limits.	1) Evaluate alternate non-linear calibration models, especially for lowest and highest calibration points.	LCR/LDR is the linear portion of a calibration curve. See the ELI LDR procedure on the wiki.
Linear Dynamic Range (LDR) For metals methods only.	Initially, then every 6 months.	%Rec = 90-110	1) Re-establish/verify LDR 2) Dilute samples within the calibration range.	Sets the upper limits of the calibration range. Must include at least 3 points, with one outside the upper range of the curve.
Retention Time (RT) window position establishment	Initially with instrument set up. Recommend verifying annually.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	1) For shifting retention times, adjust according to initial CCV (mid-range). 2) Follow method requirements.	Calculated for each analyte.
Retention Time (RT) window width	Initially with instrument set up. Recommend verifying annually.	IC: RT width is ±3 times standard deviation for each analyte RT from the 24-hour period. GC and HPLC: RT width is ±3 times standard deviation for each analyte RT from the 72-hour period. GC/MS: RT of each reported analyte within ± 0.06 RT units.	1) For shifting retention times, adjust according to initial CCV (mid-range). 2) Follow method requirements.	Calculated for each analyte.
Initial Calibration Verification (ICV)	Immediately following calibration, daily when used as Analytical Sequence LCS for analyses without Prep.	%Rec = 90-110 (Limits may be set statistically depending on method.)	1) Repour and rerun. 2) Prepare fresh calibration standards and/or ICV. 3) Recalibrate and rerun.	Evaluates calibration accuracy and method performance. Must be prepared from second source standard.

Method QA/QC Parameters
Method Name
Method Number

QA SAMPLE/ SAMP TYPE CODE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Initial Calibration Blank (ICB/MBLK)	Immediately follows ICV	< Lowest reporting limit	1) Prepare fresh blank 2) Re-pour blanks, recalibrate, and rerun.	Evaluates calibration accuracy, reagent/ glassware contamination, and instrument carryover.
Continuing Calibration Verification (CCV)	Run every 10 samples and at end of run. (Methods with internal standards do not require and ending CCV.)	%Rec = 90-110 (Limits may be set statistically depending on method.)	1) Remake and rerun. 2) Recalibrate and rerun samples since last valid CCV	Evaluates instrument drift throughout analytical sequence. Typically uses midpoint calibration standard.
Continuing Calibration Blank (CCB)	Run after every CCV. (Run every 10 samples and at the end of run)	< Lowest reporting limit	1) Check for high conc. sample. 2) Prepare fresh blank. 3) Rerun samples since last valid CCB.	Evaluates baseline drift, contamination in the analytical system, and analyte carryover.
Instrument Blank	Daily prior to sample analysis.	< Lowest reporting limit	1) Repour and rerun. 2) Perform instrument maintenance	Evaluates baseline drift, contamination in the analytical system, and analyte carryover. The method blank may be substituted; not required for methods with CCB criteria. Generally necessary for organics methods. Not necessarily imported to Omega.
Method Blank (MBLK)	1/preparation batch	<Lowest reporting limit	1) Re-digest samples from batch, or 2) Qualify sample data	Evaluates overall method including possible contamination in reagents and glassware utilized in preparatory batch.
Laboratory Control Sample (LCS)	1/preparation batch	Reference material specified limits or laboratory statistical limits of +/- 3 standard deviations.	1) Repeat analyses once 2) Correct problem 3) Re-extract and re-analyze all samples associated with LCS	Evaluates overall method accuracy/bias for the Preparatory Batch. Must be second source. If prepared the same as MS/MSD will evaluate the spiking technique.
Laboratory Fortified Blank (LFB)	1/daily sequence	%Rec = 90-110 (Limits may be set statistically depending on method.)	1) Re-prep and rerun. 2) Recalibrate and rerun.	Evaluates spiking technique. Can be primary or secondary source depending on the method. LCS or ICV are preferred QC Types.
Duplicate Sample (DUP)	1/10 samples OR 1/20 samples	% RPD ≤ 5 OR 10 OR 20 or < PQL OR < 2X PQL (Appropriate limits must be evaluated for each method.)	1) Rerun sample pair, evaluate for sample homogeneity or 2) Report with qualifiers***	Evaluates method precision. MSD duplicate analyses preferred on some methods.

Method QA/QC Parameters
Method Name
Method Number

QA SAMPLE/ SAMP TYPE CODE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Matrix Spike (MS/MSD)	1/10 samples OR 1/20 samples	Water %Rec = 90-110, %RPD ≤ 20 Soil Statistical Limits OR Soil Post Digestion Spike %Rec= 80-120 %RPD ≤ 20	LCS OR LFB OR ICV must be passing 1) If matrix interference suspected report as found, or 2) Re-analyze and re-spike if no matrix interference suspected, or 3) Use "A" qualifier for sample amount > 4X spike level.	Evaluates effect of matrix on method performance. MSD also evaluates method precision.
Post Digestion Spike (PDS/PDSD)	1/10 samples	Soil Statistical Limits OR Soil Post Digestion Spike %Rec= 80-120 %RPD ≤ 20	LCS OR LFB OR ICV must be passing 1) If matrix interference suspected report as found, or 2) Re-analyze and re-spike if no matrix interference suspected, or 3) Use "A" qualifier for sample amount > 4X spike level.	Evaluates effect of matrix on method performance. PDSD also evaluates method precision. Use the same solution and concentration as LFB.
External PE Samples	Biannual WS and/or WP and internal blind and double blind samples.	PT sample defined acceptance limits (Must pass 2 out of last 3 PT studies.)	1) Complete corrective action report 2) Repeat with another make-up study (for failure of 2 out of 3)	External review of analytical method accuracy.
MDL Studies	Bi-annually OR Annually per method requirement or whenever method changes might affect sensitivity.	<PQL, Spike Level < 10X MDL	1) Repeat if obvious problem occurs 2) Adjust reporting limit to > MDL	Evaluates overall method detection limits in clean sample matrix. Actual samples may have higher MDL.
LOD Verification	Bi-annually OR Annually per method MDL requirement following each MDL Study	Positive Result, S/N greater than 3 (above typical Method Blank performance)	1) Examine method or preparatory steps, 2) Verify MDL study, 3) Repeat analysis 4) Consult QA	Spike at 2-3X calculated MDL for single analyte test. OR Spike at 1-4X MDL for multiple analyte tests.
Control Charting and Proof of Competency	Annual statistical review of method.	Data statistically within control limits.	1) Trend Analysis/ Method Review 2) Correct method/instrument problem.	For statistical process control.
Batch Definition	Water = Each daily analytical sequence Prepped Samples = Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent.	Must pass all method QC criteria	Re-analyze batch or qualify results	A group of samples and associated QC

*** DUP Qualifier (Canned Comment) for use when values are low and the % RPD criteria does not apply.
Since the difference between the analytical result for the sample and its duplicate is less than the reporting limit, the RPD variance is not considered significant.

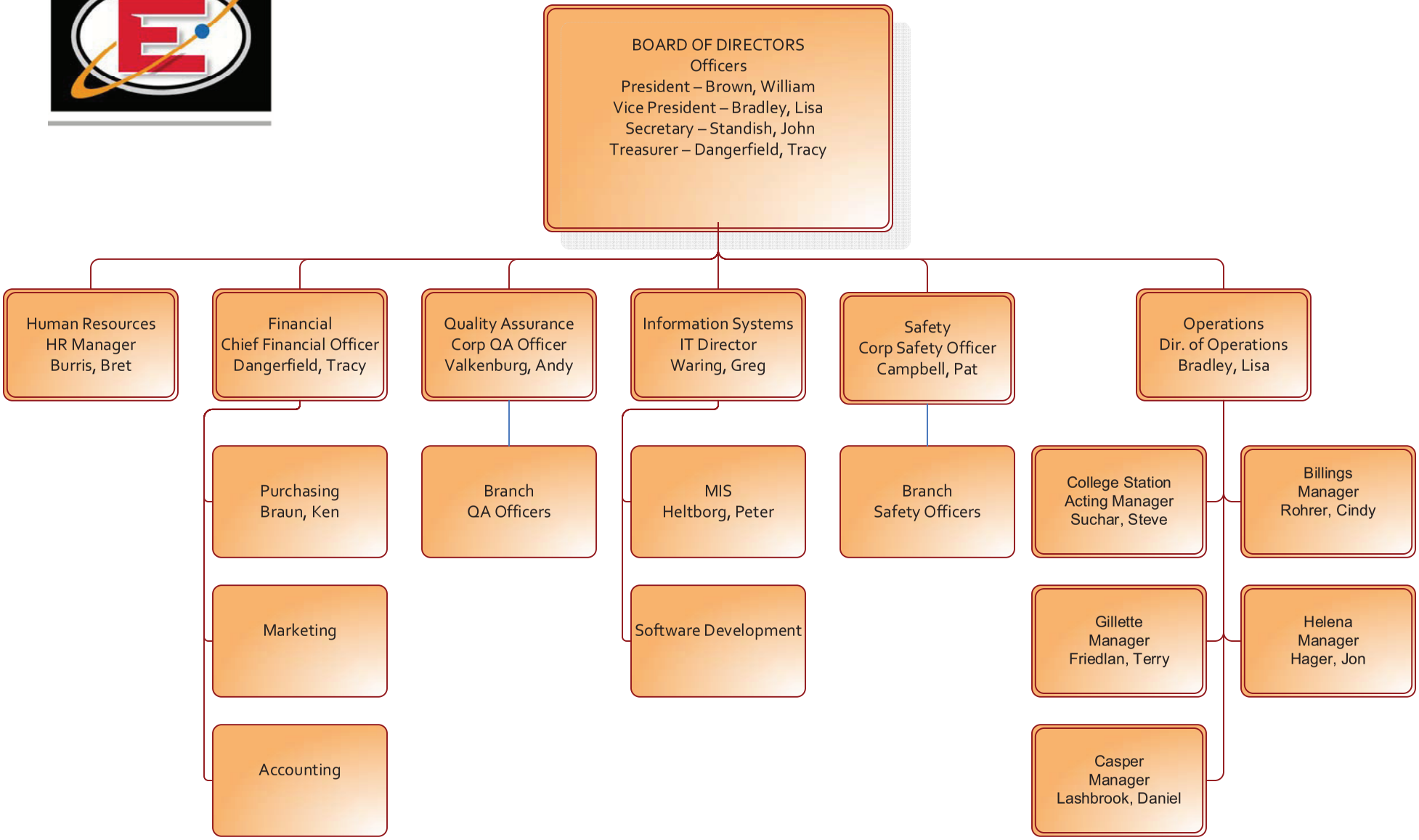
APPENDIX C

ORGANIZATIONAL CHARTS

Corporate Organizational Chart

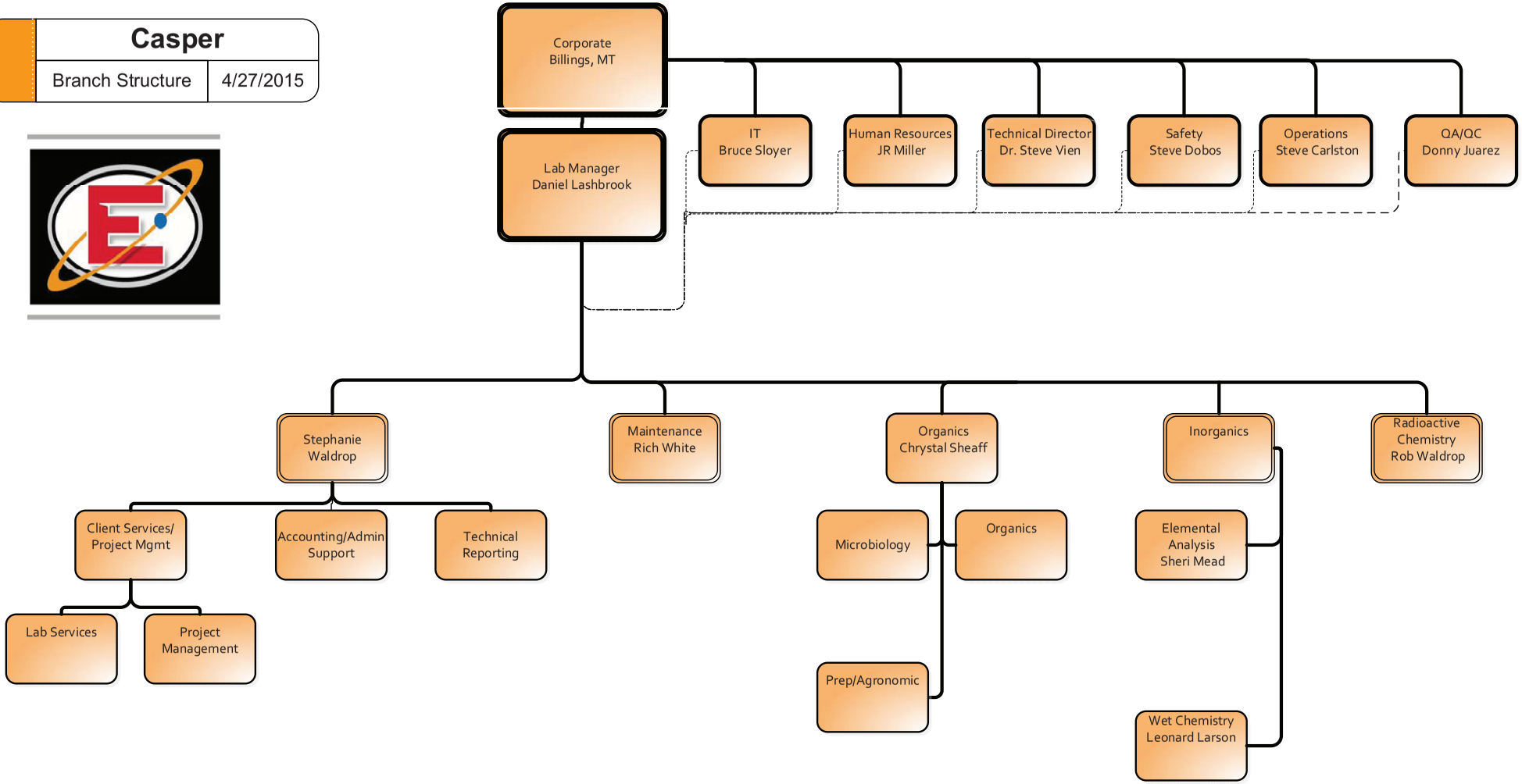
Casper Branch Lab Organizational Chart

Energy Laboratories / Corporate Structure



Casper

Branch Structure | 4/27/2015



APPENDIX D

CURRICULA VITAE OF KEY LABORATORY PERSONNEL

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APPENDIX E

EQUIPMENT AND METHODS LIST

ENERGY LABORATORIES, INC – CASPER, WYOMING

Equipment	Quantity	Methods
Gas Chromatograph-FID with auto sampler	2	8015 DRO, TPH
Gas Chromatograph-PID/FID with purge and trap & auto sampler	2	8015 GRO, 8021 MBTEXN
Gas Chromatograph-Dual ECD with auto sampler	1	504.1
Gas Chromatograph-Mass Spectrometer with purge and trap & auto sampler	3	524.2, 624, 8260B
Closed Cup Flashpoint Analyzer	2	SW1010
Atomic Absorption Fluorescence Spectrophotometer with cold vapor apparatus	1	7470A, 7471A, SM 3112B, 245.7
Atomic Absorption Spectrophotometer with flame apparatus	1	SM 3114B
Inductively Coupled Argon Plasma Spectrophotometer	2	200.7, 6010B
Inductively Coupled Argon Plasma Spectrophotometer-Mass Spectrometer	2	200.8, 6020A
Quick Trace Mercury Analyzer - Cold Vapor Atomic Absorption Analyzer	1	245.1, 7470, 7471, SM3112B, 3114B
Ion Chromatograph	2	300.0
Conductivity and pH	1	A2510B, A4500-H B
Turbidimeter	2	Turbidity, A4500-SO4
Auto Titrator / ISE	1	SM 2320B, A4500-F C, A2510B, A4500-H B
Manual Solid-Phase Extractor	1	1664A
Spectrophotometer	2	Hach 8000, SM4500-NO2 B, 420.1, A3500-Cr B
Autoanalyzer (FIA)	2	353.2, A4500-NH3
TOC Analyzer	3	A5310 B, A5310 C, SW9060
Liquid Chromatography (HPLC)	2	549.2, 531.1, 547, 632, 8316
Liquid Scintillation Counter	2	906.0, ASTM D 5072 92, 909.0M
Alpha / Beta Gas Proportional Counters Detectors	6 69	900.0, 900.1, 903.0, 905.0, RA-05
Gamma Ray Spectrometers (2 HPGe, 3 NaI(TI))	5	901.1
Alpha Spectrometers Detectors	2 40	907.0
BOD/DO Analyzer	2	5210B
TCLP Extractor/Rotator	4	1311
Microscope	1	Microbiology
(*Serial numbers and associated support equipment are located in the ELI-Casper's LIMS database).		

ATTACHMENT 1

RECORD OF REVIEW/REVISION

ATTACHMENT 1
RECORD OF REVIEW/REVISION
CASPER, WYOMING
Quality Assurance Manual

Date of Review/ Revision	Revision Number	Performed By	QA Manager Approval Initials/Date	Action (Review with no changes/ Detailed modifications)
02-2-2011	**7.15.2010.1**	**J. Judge**	**J. Judge 2-2-11**	**Edit TOC, Update Organization Chart, Add Leonard Larson to Curricula Vitae (Remove Judy Larson). Update Certifications.**
11/15/11	9/20/11	J. Judge /A. Valkenburg	J. Judge 9-22-11	Replaced Dave Poelstra with Steve Carlston as interim Branch Manager. Updated Org. charts. Replaced "EPA" with "USEPA". INTRO Section: Moved ELI commitment section to Introduction, removed Quality Policy Statement, moved "Relationship between Mgmt…and Quality System" paragraph to Ch 4 (Personnel Requirements). Added Client specific DQO information, Moved information @ "Written SOPs available at laboratory" to Ch1-Method QC Specifications. Ch1: Replaced "SOPs" with "Chapter 13 of this manual" includes recommended preventative maintenance…Added to Method QC Specifications Paragraph, "ELI SOPs are considered confidential proprietary information and ELI does not provide copies of SOPs off ELI premises." Moved "For radiochemical analyses, each method undergoes Method Validation as outlined in EPA's specific method and the Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), Chapter 6. " and "The required detection level (RDL) for radiochemical analyses of drinking water samples is calculated based on the requirements in 40 CFR 141.2~~7~~5(c), which is a sample specific determination. The equation is specific for each method and noted in the method-specific SOP." Updated ELI MDL SOP reference. CH 4: Added "and laboratory experience/training appropriate to the procedures they are performing" to analysts. Added electronic signature info to Approved signatories section. CH 5: Added: To ensure that drinking water analysis for radiochemistry are met, the requirements for sample handling, preservation, and instrumentation for radiochemical analysis are included in ELI SOP, "Sample Receipt, Log-In and Labeling". (For additional information, refer to "Manual for the Certification of Laboratories Analyzing Drinking Water, Table VI-2: Sample Handling, Preservation, and Instrumentation, EPA 5th Edition, January 2005)". Ch 7: Added to laboratory stock/working stds: "are

Date of Review/ Revision	Revision Number	Performed By	QA Manager Approval Initials/Date	Action (Review with no changes/ Detailed modifications)
				derived from ISO 17025 and/or 9001 (or equivalent-certified)" commercially available. Removed "for drinking water samples" from MDL are determined according to EPA guidelines… and added (except for the few methods that are not amenable to MDLs)." Replaced MDL with "practical quantitation limits". Removed "General method detection limits are given in Appendix D". Ch 8: Added information @Record of Review/Revision page included on SOPs. Removed from Laboratory Notebooks, "Laboratory notebooks are controlled with unique identifiers. They are indexed and maintained at a commercial storage facility when completed". Added to Reporting section, "Clients are able to access their electronic files through ELI's secure website @ https://energysource.energylab.com/. CH 10: Removed, " Balances are checked monthly against a second set of NIST traceable ASTM Type I weights." Replaced "periodically" with annually for when SOPs are reviewed. CH 11: Replaced ELI's website "global compliance" with "Alert". CH 13: Added, "Repairs and maintenance are accomplished in-house by experienced laboratory personnel. Other than consumable equipment items, an inventory of spare parts is not maintained. Spare parts are available from outside vendors on an as needed basis. (To ensure method capability, some methods have more than one instrument available)." References Section: Added ASTM D7282-06 Radiochem reference. Appendices: Added General Radiochemistry QC elements. Removed David Poelstra from Curricula Vitae.
11/15/11	9/20/2011	J. Judge/ J. Erickson	J.Judge 11/15/11	Title Page: Changed Branch Technical Director to Steve Dobos. Introduction Section: Added information on the procedure for all new work acquired by lab. Chapter 4, Personnel Requirements: Added "refer to ELI SOP Method Deviations for details regarding the specific process for any lab deviations. Appendix E: Added "Technical Director" to Steve Dobos Curricula Vitae.
11/8/12	11/8/12	J.Judge /J. Erickson	J.Judge 11/08/12	Title Page: Added Steve V. as technical director. **CH 5:** Added "Aqueous" to Samples for microbiological analysis." Added, "Solid samples are collected in pre-sterilized 120mL plastic bottles for microbial testing without preservation. "**CH 12:** Updated Equipment list. **Attachments:** Updated Org charts, NELAP/NELAC, EPA, RadChem Certificates. Curricula Vitea: Removed Randy Ogden and Donny J. Added Steve Vien, Kerri Schroeder and James Ritchie, updated Steve Dobos.
7-26-13	7/26/13	J Judge	J Judge 7-26-13	New P.O. Box Number front page. Update ELI Org Chart Added annual ethics training Chapter11. Edited last paragraph Chapter 11, Corrective Action regarding deviations from method. Update Chapter12

Date of Review/ Revision	Revision Number	Performed By	QA Manager Approval Initials/Date	Action (Review with no changes/ Detailed modifications)
				Update Chapter 13 – Compressed gasses change set at 200 PSI Chapter 14, added newest SW846 version Update Appendix C Update Appendix D Remove John Standish, add Lisa Bradley Remove Dave Blaida Update Rob Waldrop Add Chuck Patterson Update NELAP & EPA Certifications
3-7-14	3-7-14	J Judge	J Judge 3-7-14	Major Update – Entire QAM is now based on Billings Corporate QAM.
5/15/2015	5/15/2015	D Juarez	D Juarez 5/15/2015	Updated Laboratory Manager and Quality Assurance Officer Changed all reference to “Branch Manager” to “Laboratory Manager” Removed all reference to the Laboratory Policy SOP Removed all reference to the Roles and Responsibilities SOP Removed reference to Rapid City Lab Updated the Table of Contents Removed Appendix A and renamed Appendix B, C, D, E and F to A, B, C, D and E Updated the Introduction Section Updated Chapter 4 Updated Chapter 6 Updated Chapter 7 Updated Chapter 8 Updated Chapter 11 Major update Chapter 12 moved equipment list the Appendix E Updated Chapter 15 Updated all Appendices Removed Attachment 2 Multiple grammatical corrections See redline document for all changes Added Management of Change Chapter

Appendix 4
Laboratory Certification

Due to its size, the Laboratory Certification is included in the electronic submittal only.

State of Utah

Department of Health

Environmental Laboratory Certification Program

Certification is hereby granted to

ACZ Laboratories, Inc.

2773 Downhill Drive
Steamboat Springs, CO 80487

Has conformed with the
2009 TNI Standard
Scope of accreditation is limited to the
State of Utah accredited fields that accompany
this Certificate

EPA Number:	CO00028
Expiration Date:	7/31/2016
Certificate Number:	CO000282015-12

Robyn M. Atkinson, Ph.D, HCLD
Director, Utah Public Health Laboratory

NELAP RECOGNIZED TNI ACCREDITATION BODY

Continued accredited status depends on successful ongoing participation in the program.

UTAH DEPARTMENT OF HEALTH

State of Utah
Gary R Herbert
Governor
Gregory S Bell
Lieutenant Governor

Utah Department of Health
W. David Patton Ph.D
Executive Director

Division of Disease Control and Prevention
Robyn M. Atkinson, Ph.D, HCLD
Director, Utah Public Health Laboratory

EPA Number: ***CO00028*** **Attachment to Certificate Number: CO000282015-12**

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***CWA (Non Potable Water)***			
Method ASTM D516-02			
Sulfate	8/1/2015	7/31/2016	UT
Method ASTM D516-07			
Sulfate	8/1/2015	7/31/2016	UT
Method ASTM D6888-04			
Total cyanide	8/1/2015	7/31/2016	UT
Method ASTM D6888-09			
Total cyanide	8/1/2015	7/31/2016	UT
Method ASTM D7065-06			
Nonyl phenol	8/1/2015	7/31/2016	UT
Nonylphenol diethoxylate	8/1/2015	7/31/2016	UT
Nonylphenol ethoxylate	8/1/2015	7/31/2016	UT
Method ASTM D7511-09e2			
Total cyanide	8/1/2015	7/31/2016	UT
Method EPA 160.4			
Residue-volatile	8/1/2015	7/31/2016	UT
Method EPA 1631E			
Mercury	8/1/2015	7/31/2016	UT
Method EPA 1664A			
Oil & Grease	8/1/2015	7/31/2016	UT
Method EPA 1664A (SGT-HEM)			
Hexane Extractable Material - Silica Gel Treated (HEM-SGT)	8/1/2015	7/31/2016	UT
Method EPA 180.1			
Turbidity	8/1/2015	7/31/2016	UT
Method EPA 200.7			
Aluminum	8/1/2015	7/31/2016	UT
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Boron	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Calcium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***CWA (Non Potable Water)***			
Cobalt	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Iron	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Lithium	8/1/2015	7/31/2016	UT
Magnesium	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Molybdenum	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Phosphorus, total	8/1/2015	7/31/2016	UT
Potassium	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silica as SiO2	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Sodium	8/1/2015	7/31/2016	UT
Strontium	8/1/2015	7/31/2016	UT
Tin	8/1/2015	7/31/2016	UT
Titanium	8/1/2015	7/31/2016	UT
Vanadium	8/1/2015	7/31/2016	UT
Zinc	8/1/2015	7/31/2016	UT
Method EPA 200.8			
Aluminum	8/1/2015	7/31/2016	UT
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT
Cobalt	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Mercury	8/1/2015	7/31/2016	UT
Molybdenum	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Thallium	8/1/2015	7/31/2016	UT
Thorium	8/1/2015	7/31/2016	UT
Uranium	8/1/2015	7/31/2016	UT
Vanadium	8/1/2015	7/31/2016	UT
Zinc	8/1/2015	7/31/2016	UT
Method EPA 245.1			
Mercury	8/1/2015	7/31/2016	UT
Method EPA 300.0			
Bromide	8/1/2015	7/31/2016	UT
Chloride	8/1/2015	7/31/2016	UT
Fluoride	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***CWA (Non Potable Water)***			
Sulfate	8/1/2015	7/31/2016	UT
Method EPA 335.4			
Total cyanide	8/1/2015	7/31/2016	UT
Method EPA 350.1			
Ammonia as N	8/1/2015	7/31/2016	UT
Method EPA 351.2			
Kjeldahl nitrogen - total	8/1/2015	7/31/2016	UT
Method EPA 353.2			
Nitrate as N	8/1/2015	7/31/2016	UT
Nitrate-nitrite	8/1/2015	7/31/2016	UT
Nitrite as N	8/1/2015	7/31/2016	UT
Method EPA 365.1			
Orthophosphate as P	8/1/2015	7/31/2016	UT
Phosphorus, total	8/1/2015	7/31/2016	UT
Method EPA 375.4			
Sulfate	8/1/2015	7/31/2016	UT
Method EPA 410.4			
Chemical oxygen demand	8/1/2015	7/31/2016	UT
Method EPA 420.4			
Total phenolics	8/1/2015	7/31/2016	UT
Method EPA 624			
1,1,1-Trichloroethane	8/1/2015	7/31/2016	UT
1,1,2,2-Tetrachloroethane	8/1/2015	7/31/2016	UT
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	8/1/2015	7/31/2016	UT
1,1,2-Trichloroethane	8/1/2015	7/31/2016	UT
1,1-Dichloroethane	8/1/2015	7/31/2016	UT
1,1-Dichloroethylene	8/1/2015	7/31/2016	UT
1,2-Dichlorobenzene (o-Dichlorobenzene)	8/1/2015	7/31/2016	UT
1,2-Dichloroethane (Ethylene dichloride)	8/1/2015	7/31/2016	UT
1,2-Dichloropropane	8/1/2015	7/31/2016	UT
1,3-Dichlorobenzene	8/1/2015	7/31/2016	UT
1,4-Dichlorobenzene	8/1/2015	7/31/2016	UT
1,4-Dioxane (1,4- Diethyleneoxide)	8/1/2015	7/31/2016	UT
1-Chlorohexane	8/1/2015	7/31/2016	UT
2-Chloroethyl vinyl ether	8/1/2015	7/31/2016	UT
4-Isopropyltoluene (p-Cymene,p-Isopropyltoluene)	8/1/2015	7/31/2016	UT
Acetonitrile	8/1/2015	7/31/2016	UT
Acrolein (Propenal)	8/1/2015	7/31/2016	UT
Benzene	8/1/2015	7/31/2016	UT
Bromodichloromethane	8/1/2015	7/31/2016	UT
Bromoform	8/1/2015	7/31/2016	UT
Carbon tetrachloride	8/1/2015	7/31/2016	UT
Chlorobenzene	8/1/2015	7/31/2016	UT
Chlorodibromomethane	8/1/2015	7/31/2016	UT
Chloroethane (Ethyl chloride)	8/1/2015	7/31/2016	UT
Chloroform	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***CWA (Non Potable Water)***			
cis-1,3-Dichloropropene	8/1/2015	7/31/2016	UT
Ethylbenzene	8/1/2015	7/31/2016	UT
Iodomethane (Methyl iodide)	8/1/2015	7/31/2016	UT
Methyl bromide (Bromomethane)	8/1/2015	7/31/2016	UT
Methyl chloride (Chloromethane)	8/1/2015	7/31/2016	UT
Methylene chloride (Dichloromethane)	8/1/2015	7/31/2016	UT
m-Xylene	8/1/2015	7/31/2016	UT
n-Hexane	8/1/2015	7/31/2016	UT
o-Xylene	8/1/2015	7/31/2016	UT
p-Xylene	8/1/2015	7/31/2016	UT
Tetrachloroethylene (Perchloroethylene)	8/1/2015	7/31/2016	UT
Toluene	8/1/2015	7/31/2016	UT
trans-1,2-Dichloroethylene	8/1/2015	7/31/2016	UT
trans-1,3-Dichloropropylene	8/1/2015	7/31/2016	UT
trans-1,4-Dichloro-2-butene	8/1/2015	7/31/2016	UT
Trichloroethene (Trichloroethylene)	8/1/2015	7/31/2016	UT
Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	8/1/2015	7/31/2016	UT
Vinyl chloride	8/1/2015	7/31/2016	UT
Xylene (total)	8/1/2015	7/31/2016	UT
Method EPA 625			
1,2,4-Trichlorobenzene	8/1/2015	7/31/2016	UT
2,4,6-Trichlorophenol	8/1/2015	7/31/2016	UT
2,4-Dichlorophenol	8/1/2015	7/31/2016	UT
2,4-Dimethylphenol	8/1/2015	7/31/2016	UT
2,4-Dinitrophenol	8/1/2015	7/31/2016	UT
2,4-Dinitrotoluene (2,4-DNT)	8/1/2015	7/31/2016	UT
2,6-Dinitrotoluene (2,6-DNT)	8/1/2015	7/31/2016	UT
2-Chloronaphthalene	8/1/2015	7/31/2016	UT
2-Chlorophenol	8/1/2015	7/31/2016	UT
2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	8/1/2015	7/31/2016	UT
2-Nitrophenol	8/1/2015	7/31/2016	UT
3,3'-Dichlorobenzidine	8/1/2015	7/31/2016	UT
4-Bromophenyl phenyl ether	8/1/2015	7/31/2016	UT
4-Chloro-3-methylphenol	8/1/2015	7/31/2016	UT
4-Chlorophenyl phenylether	8/1/2015	7/31/2016	UT
4-Nitrophenol	8/1/2015	7/31/2016	UT
Acenaphthene	8/1/2015	7/31/2016	UT
Acenaphthylene	8/1/2015	7/31/2016	UT
Anthracene	8/1/2015	7/31/2016	UT
Benzo(a)anthracene	8/1/2015	7/31/2016	UT
Benzo(a)pyrene	8/1/2015	7/31/2016	UT
Benzo(b)fluoranthene	8/1/2015	7/31/2016	UT
Benzo(g,h,i)perylene	8/1/2015	7/31/2016	UT
Benzo(k)fluoranthene	8/1/2015	7/31/2016	UT
bis(2-Chloroethoxy)methane	8/1/2015	7/31/2016	UT
bis(2-Chloroethyl) ether	8/1/2015	7/31/2016	UT
bis(2-Chloroisopropyl) ether	8/1/2015	7/31/2016	UT
Butyl benzyl phthalate	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***CWA (Non Potable Water)***			
Chrysene	8/1/2015	7/31/2016	UT
Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	8/1/2015	7/31/2016	UT
Dibenz(a,h) anthracene	8/1/2015	7/31/2016	UT
Diethyl phthalate	8/1/2015	7/31/2016	UT
Dimethyl phthalate	8/1/2015	7/31/2016	UT
Di-n-butyl phthalate	8/1/2015	7/31/2016	UT
Di-n-octyl phthalate	8/1/2015	7/31/2016	UT
Fluoranthene	8/1/2015	7/31/2016	UT
Fluorene	8/1/2015	7/31/2016	UT
Hexachlorobenzene	8/1/2015	7/31/2016	UT
Hexachlorobutadiene	8/1/2015	7/31/2016	UT
Hexachlorocyclopentadiene	8/1/2015	7/31/2016	UT
Hexachloroethane	8/1/2015	7/31/2016	UT
Indeno(1,2,3-cd) pyrene	8/1/2015	7/31/2016	UT
Isophorone	8/1/2015	7/31/2016	UT
Naphthalene	8/1/2015	7/31/2016	UT
Nitrobenzene	8/1/2015	7/31/2016	UT
n-Nitrosodimethylamine	8/1/2015	7/31/2016	UT
n-Nitrosodi-n-propylamine	8/1/2015	7/31/2016	UT
n-Nitrosodiphenylamine	8/1/2015	7/31/2016	UT
Pentachlorophenol	8/1/2015	7/31/2016	UT
Phenanthrene	8/1/2015	7/31/2016	UT
Phenol	8/1/2015	7/31/2016	UT
Pyrene	8/1/2015	7/31/2016	UT
Method EPA 900			
Gross-alpha	8/1/2015	7/31/2016	UT
Gross-beta	8/1/2015	7/31/2016	UT
Method EPA 901.1			
Cesium-134	8/1/2015	7/31/2016	UT
Cesium-137	8/1/2015	7/31/2016	UT
Method EPA 903			
Radium-226	8/1/2015	7/31/2016	UT
Total alpha radium	8/1/2015	7/31/2016	UT
Method EPA 903.1			
Radium-226	8/1/2015	7/31/2016	UT
Method EPA 904			
Radium-228	8/1/2015	7/31/2016	UT
Method OIA 1677-09			
Free cyanide	8/1/2015	7/31/2016	UT
Method SM 2310 B-1997			
Acidity, as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2310 B-2011			
Acidity, as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2320 B-1997			
Alkalinity as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2320 B-2011			

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***CWA (Non Potable Water)***			
Alkalinity as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2340 B-1997			
Total hardness as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2340 B-2011			
Total hardness as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2510 B-1997			
Conductivity	8/1/2015	7/31/2016	UT
Method SM 2510 B-2011			
Conductivity	8/1/2015	7/31/2016	UT
Method SM 2540 B-1997			
Residue-total	8/1/2015	7/31/2016	UT
Method SM 2540 B-2011			
Residue-total	8/1/2015	7/31/2016	UT
Method SM 2540 C-1997			
Residue-filterable (TDS)	8/1/2015	7/31/2016	UT
Method SM 2540 C-2011			
Residue-filterable (TDS)	8/1/2015	7/31/2016	UT
Method SM 2540 D-1997			
Residue-nonfilterable (TSS)	8/1/2015	7/31/2016	UT
Method SM 2540 D-2011			
Residue-nonfilterable (TSS)	8/1/2015	7/31/2016	UT
Method SM 2540 F-1997			
Residue-settleable	8/1/2015	7/31/2016	UT
Method SM 2540 F-2011			
Residue-settleable	8/1/2015	7/31/2016	UT
Method SM 3114 B-1993			
Selenium	8/1/2015	7/31/2016	UT
Method SM 3114 B-2009			
Selenium	8/1/2015	7/31/2016	UT
Method SM 3114 B-2011			
Selenium	8/1/2015	7/31/2016	UT
Method SM 3114 C-2009			
Selenium	8/1/2015	7/31/2016	UT
Method SM 3114 C-2011			
Selenium	8/1/2015	7/31/2016	UT
Method SM 3500-Cr B-2009			
Chromium VI	8/1/2015	7/31/2016	UT
Method SM 3500-Cr B-2011			
Chromium VI	8/1/2015	7/31/2016	UT
Method SM 3500-Cr D-1990			
Chromium VI	8/1/2015	7/31/2016	UT
Method SM 3500-Fe B-1997			
Iron-(II) (Ferrous Iron)	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***CWA (Non Potable Water)***			
Method SM 3500-Fe B-2011			
Iron-(II) (Ferrous Iron)	8/1/2015	7/31/2016	UT
Method SM 4500-Cl E-1997			
Chloride	8/1/2015	7/31/2016	UT
Method SM 4500-Cl E-2011			
Chloride	8/1/2015	7/31/2016	UT
Method SM 4500-CN$^-$ C-1999			
Total cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-CN$^-$ C-2011			
Total cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-CN$^-$ E-1999			
Cyanide	8/1/2015	7/31/2016	UT
Total cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-CN$^-$ E-2011			
Cyanide	8/1/2015	7/31/2016	UT
Total cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-CN$^-$ I-2011			
Weak Acid Dissociable Cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-F$^-$ C-1997			
Fluoride	8/1/2015	7/31/2016	UT
Method SM 4500-F$^-$ C-2011			
Fluoride	8/1/2015	7/31/2016	UT
Method SM 4500-H+ B-2000			
pH	8/1/2015	7/31/2016	UT
Method SM 4500-H+ B-2011			
pH	8/1/2015	7/31/2016	UT
Method SM 4500-S2$^-$ D-2000			
Sulfide	8/1/2015	7/31/2016	UT
Method SM 4500-S2$^-$ D-2011			
Sulfide	8/1/2015	7/31/2016	UT
Method SM 4500-SO4$^-$ D-1997			
Sulfate	8/1/2015	7/31/2016	UT
Method SM 4500-SO4$^-$ D-2011			
Sulfate	8/1/2015	7/31/2016	UT
Method SM 5210 B-2001			
Biochemical oxygen demand	8/1/2015	7/31/2016	UT
Carbonaceous BOD, CBOD	8/1/2015	7/31/2016	UT
Method SM 5210 B-2011			
Biochemical oxygen demand	8/1/2015	7/31/2016	UT
Carbonaceous BOD, CBOD	8/1/2015	7/31/2016	UT
Method SM 5310 B-1996			
Total organic carbon	8/1/2015	7/31/2016	UT
Method SM 5310 B-2000			
Total organic carbon	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***CWA (Non Potable Water)***			
Method SM 5310 B-2011			
Total organic carbon	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

Program/Matrix: ***RCRA (Biological Tissue)***

	Start Date	Expires	AB
Method EPA 6010B			
Aluminum	8/1/2015	7/31/2016	UT
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Boron	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Calcium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT
Cobalt	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Iron	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Lithium	8/1/2015	7/31/2016	UT
Magnesium	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Molybdenum	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Potassium	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Sodium	8/1/2015	7/31/2016	UT
Strontium	8/1/2015	7/31/2016	UT
Thallium	8/1/2015	7/31/2016	UT
Tin	8/1/2015	7/31/2016	UT
Titanium	8/1/2015	7/31/2016	UT
Vanadium	8/1/2015	7/31/2016	UT
Zinc	8/1/2015	7/31/2016	UT
Method EPA 6020			
Aluminum	8/1/2015	7/31/2016	UT
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT
Cobalt	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Molybdenum	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Thallium	8/1/2015	7/31/2016	UT
Uranium	8/1/2015	7/31/2016	UT
Vanadium	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Biological Tissue)***			
Zinc	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Non Potable Water)***			
Method EPA 1010A			
Ignitability	8/1/2015	7/31/2016	UT
Method EPA 1311			
Toxicity Characteristic Leaching Procedure (TCLP)	8/1/2015	7/31/2016	UT
Method EPA 1312			
Synthetic Precipitation Leaching Procedure (SCLP)	8/1/2015	7/31/2016	UT
Method EPA 3005A			
Metals Sample Prep	8/1/2015	7/31/2016	UT
Method EPA 3010A			
Acid Digestion of Aqueous samples and Extracts for Total Metals	8/1/2015	7/31/2016	UT
Method EPA 3510C			
Seperatory Funnel Liquid-Liquid Extraction	8/1/2015	7/31/2016	UT
Method EPA 3520C			
Continuous Liquid-Liquid Extraction	8/1/2015	7/31/2016	UT
Method EPA 3630C			
Silica Gel Clean-up	8/1/2015	7/31/2016	UT
Method EPA 5030C			
Aqueous-phase Purge & Trap	8/1/2015	7/31/2016	UT
Method EPA 6010B			
Aluminum	8/1/2015	7/31/2016	UT
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Boron	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Calcium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT
Cobalt	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Iron	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Lithium	8/1/2015	7/31/2016	UT
Magnesium	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Molybdenum	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Phosphorus, total	8/1/2015	7/31/2016	UT
Potassium	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silica as SiO2	8/1/2015	7/31/2016	UT
Silicon	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Sodium	8/1/2015	7/31/2016	UT
Strontium	8/1/2015	7/31/2016	UT
Tin	8/1/2015	7/31/2016	UT

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Non Potable Water)***			
Titanium	8/1/2015	7/31/2016	UT
Vanadium	8/1/2015	7/31/2016	UT
Zinc	8/1/2015	7/31/2016	UT
Method EPA 6020			
Aluminum	8/1/2015	7/31/2016	UT
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT
Cobalt	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Molybdenum	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Thallium	8/1/2015	7/31/2016	UT
Uranium	8/1/2015	7/31/2016	UT
Vanadium	8/1/2015	7/31/2016	UT
Zinc	8/1/2015	7/31/2016	UT
Method EPA 7196A			
Chromium VI	8/1/2015	7/31/2016	UT
Method EPA 7470A			
Mercury	8/1/2015	7/31/2016	UT
Method EPA 8015D			
Diesel range organics (DRO)	8/1/2015	7/31/2016	UT
Gasoline range organics (GRO)	8/1/2015	7/31/2016	UT
Method EPA 8021B			
Benzene	8/1/2015	7/31/2016	UT
Ethylbenzene	8/1/2015	7/31/2016	UT
m-Xylene	8/1/2015	7/31/2016	UT
o-Xylene	8/1/2015	7/31/2016	UT
p-Xylene	8/1/2015	7/31/2016	UT
Toluene	8/1/2015	7/31/2016	UT
Xylene (total)	8/1/2015	7/31/2016	UT
Method EPA 8082A			
Aroclor-1016 (PCB-1016)	8/1/2015	7/31/2016	UT
Aroclor-1221 (PCB-1221)	8/1/2015	7/31/2016	UT
Aroclor-1232 (PCB-1232)	8/1/2015	7/31/2016	UT
Aroclor-1242 (PCB-1242)	8/1/2015	7/31/2016	UT
Aroclor-1248 (PCB-1248)	8/1/2015	7/31/2016	UT
Aroclor-1254 (PCB-1254)	8/1/2015	7/31/2016	UT
Aroclor-1260 (PCB-1260)	8/1/2015	7/31/2016	UT
Aroclor-1262 (PCB-1262)	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Non Potable Water)***			
Aroclor-1268 (PCB-1268)	8/1/2015	7/31/2016	UT
Method EPA 8260B			
1,1,1,2-Tetrachloroethane	8/1/2015	7/31/2016	UT
1,1,1-Trichloroethane	8/1/2015	7/31/2016	UT
1,1,2,2-Tetrachloroethane	8/1/2015	7/31/2016	UT
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	8/1/2015	7/31/2016	UT
1,1,2-Trichloroethane	8/1/2015	7/31/2016	UT
1,1-Dichloroethane	8/1/2015	7/31/2016	UT
1,1-Dichloroethylene	8/1/2015	7/31/2016	UT
1,1-Dichloropropene	8/1/2015	7/31/2016	UT
1,2,3-Trichlorobenzene	8/1/2015	7/31/2016	UT
1,2,3-Trichloropropane	8/1/2015	7/31/2016	UT
1,2,4-Trichlorobenzene	8/1/2015	7/31/2016	UT
1,2,4-Trimethylbenzene	8/1/2015	7/31/2016	UT
1,2-Dibromo-3-chloropropane (DBCP)	8/1/2015	7/31/2016	UT
1,2-Dibromoethane (EDB, Ethylene dibromide)	8/1/2015	7/31/2016	UT
1,2-Dichlorobenzene (o-Dichlorobenzene)	8/1/2015	7/31/2016	UT
1,2-Dichloroethane (Ethylene dichloride)	8/1/2015	7/31/2016	UT
1,2-Dichloropropane	8/1/2015	7/31/2016	UT
1,3,5-Trimethylbenzene	8/1/2015	7/31/2016	UT
1,3-Dichlorobenzene	8/1/2015	7/31/2016	UT
1,3-Dichloropropane	8/1/2015	7/31/2016	UT
1,4-Dichlorobenzene	8/1/2015	7/31/2016	UT
1,4-Dioxane (1,4- Diethyleneoxide)	8/1/2015	7/31/2016	UT
1-Chlorohexane	8/1/2015	7/31/2016	UT
2,2-Dichloropropane	8/1/2015	7/31/2016	UT
2-Butanone (Methyl ethyl ketone, MEK)	8/1/2015	7/31/2016	UT
2-Chloroethyl vinyl ether	8/1/2015	7/31/2016	UT
2-Chlorotoluene	8/1/2015	7/31/2016	UT
2-Hexanone	8/1/2015	7/31/2016	UT
4-Chlorotoluene	8/1/2015	7/31/2016	UT
4-Isopropyltoluene (p-Cymene,p-Isopropyltoluene)	8/1/2015	7/31/2016	UT
4-Methyl-2-pentanone (MIBK)	8/1/2015	7/31/2016	UT
Acetone	8/1/2015	7/31/2016	UT
Acetonitrile	8/1/2015	7/31/2016	UT
Acrolein (Propenal)	8/1/2015	7/31/2016	UT
Acrylonitrile	8/1/2015	7/31/2016	UT
Benzene	8/1/2015	7/31/2016	UT
Bromobenzene	8/1/2015	7/31/2016	UT
Bromochloromethane	8/1/2015	7/31/2016	UT
Bromodichloromethane	8/1/2015	7/31/2016	UT
Bromoform	8/1/2015	7/31/2016	UT
Carbon disulfide	8/1/2015	7/31/2016	UT
Carbon tetrachloride	8/1/2015	7/31/2016	UT
Chlorobenzene	8/1/2015	7/31/2016	UT
Chlorodibromomethane	8/1/2015	7/31/2016	UT
Chloroethane (Ethyl chloride)	8/1/2015	7/31/2016	UT
Chloroform	8/1/2015	7/31/2016	UT

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Non Potable Water)***			
cis-1,2-Dichloroethylene	8/1/2015	7/31/2016	UT
cis-1,3-Dichloropropene	8/1/2015	7/31/2016	UT
Dibromomethane (Methylene bromide)	8/1/2015	7/31/2016	UT
Dichlorodifluoromethane (Freon-12)	8/1/2015	7/31/2016	UT
Ethylbenzene	8/1/2015	7/31/2016	UT
Hexachlorobutadiene	8/1/2015	7/31/2016	UT
Iodomethane (Methyl iodide)	8/1/2015	7/31/2016	UT
Isopropylbenzene	8/1/2015	7/31/2016	UT
Methyl bromide (Bromomethane)	8/1/2015	7/31/2016	UT
Methyl chloride (Chloromethane)	8/1/2015	7/31/2016	UT
Methyl tert-butyl ether (MTBE)	8/1/2015	7/31/2016	UT
Methylene chloride (Dichloromethane)	8/1/2015	7/31/2016	UT
m-Xylene	8/1/2015	7/31/2016	UT
Naphthalene	8/1/2015	7/31/2016	UT
n-Butylbenzene	8/1/2015	7/31/2016	UT
n-Hexane	8/1/2015	7/31/2016	UT
n-Propylbenzene	8/1/2015	7/31/2016	UT
o-Xylene	8/1/2015	7/31/2016	UT
p-Xylene	8/1/2015	7/31/2016	UT
sec-Butylbenzene	8/1/2015	7/31/2016	UT
Styrene	8/1/2015	7/31/2016	UT
tert-Butylbenzene	8/1/2015	7/31/2016	UT
Tetrachloroethylene (Perchloroethylene)	8/1/2015	7/31/2016	UT
Toluene	8/1/2015	7/31/2016	UT
trans-1,2-Dichloroethylene	8/1/2015	7/31/2016	UT
trans-1,3-Dichloropropylene	8/1/2015	7/31/2016	UT
trans-1,4-Dichloro-2-butene	8/1/2015	7/31/2016	UT
Trichloroethene (Trichloroethylene)	8/1/2015	7/31/2016	UT
Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	8/1/2015	7/31/2016	UT
Vinyl acetate	8/1/2015	7/31/2016	UT
Vinyl chloride	8/1/2015	7/31/2016	UT
Xylene (total)	8/1/2015	7/31/2016	UT
Method EPA 8270C			
1,2,4-Trichlorobenzene	8/1/2015	7/31/2016	UT
1,2-Dichlorobenzene (o-Dichlorobenzene)	8/1/2015	7/31/2016	UT
1,3-Dichlorobenzene	8/1/2015	7/31/2016	UT
1,4-Dichlorobenzene	8/1/2015	7/31/2016	UT
2,4,5-Trichlorophenol	8/1/2015	7/31/2016	UT
2,4,6-Trichlorophenol	8/1/2015	7/31/2016	UT
2,4-Dichlorophenol	8/1/2015	7/31/2016	UT
2,4-Dimethylphenol	8/1/2015	7/31/2016	UT
2,4-Dinitrophenol	8/1/2015	7/31/2016	UT
2,4-Dinitrotoluene (2,4-DNT)	8/1/2015	7/31/2016	UT
2,6-Dinitrotoluene (2,6-DNT)	8/1/2015	7/31/2016	UT
2-Chloronaphthalene	8/1/2015	7/31/2016	UT
2-Chlorophenol	8/1/2015	7/31/2016	UT
2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	8/1/2015	7/31/2016	UT
2-Methylnaphthalene	8/1/2015	7/31/2016	UT

Program/Matrix: ***RCRA (Non Potable Water)***

	Start Date	Expires	AB
2-Methylphenol (o-Cresol)	8/1/2015	7/31/2016	UT
2-Nitroaniline	8/1/2015	7/31/2016	UT
2-Nitrophenol	8/1/2015	7/31/2016	UT
3,3'-Dichlorobenzidine	8/1/2015	7/31/2016	UT
3-Methylphenol (m-Cresol)	8/1/2015	7/31/2016	UT
3-Nitroaniline	8/1/2015	7/31/2016	UT
4-Bromophenyl phenyl ether	8/1/2015	7/31/2016	UT
4-Chloro-3-methylphenol	8/1/2015	7/31/2016	UT
4-Chloroaniline	8/1/2015	7/31/2016	UT
4-Chlorophenyl phenylether	8/1/2015	7/31/2016	UT
4-Methylphenol (p-Cresol)	8/1/2015	7/31/2016	UT
4-Nitroaniline	8/1/2015	7/31/2016	UT
4-Nitrophenol	8/1/2015	7/31/2016	UT
Acenaphthene	8/1/2015	7/31/2016	UT
Acenaphthylene	8/1/2015	7/31/2016	UT
Anthracene	8/1/2015	7/31/2016	UT
Azobenzene (1,2-Diphenylhydrazine)	8/1/2015	7/31/2016	UT
Benzo(a)anthracene	8/1/2015	7/31/2016	UT
Benzo(a)pyrene	8/1/2015	7/31/2016	UT
Benzo(b)fluoranthene	8/1/2015	7/31/2016	UT
Benzo(g,h,i)perylene	8/1/2015	7/31/2016	UT
Benzo(k)fluoranthene	8/1/2015	7/31/2016	UT
Benzoic acid	8/1/2015	7/31/2016	UT
Benzyl alcohol	8/1/2015	7/31/2016	UT
bis(2-Chloroethoxy)methane	8/1/2015	7/31/2016	UT
bis(2-Chloroethyl) ether	8/1/2015	7/31/2016	UT
bis(2-Chloroisopropyl) ether	8/1/2015	7/31/2016	UT
Butyl benzyl phthalate	8/1/2015	7/31/2016	UT
Chrysene	8/1/2015	7/31/2016	UT
Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	8/1/2015	7/31/2016	UT
Dibenz(a,h) anthracene	8/1/2015	7/31/2016	UT
Dibenzofuran	8/1/2015	7/31/2016	UT
Diethyl phthalate	8/1/2015	7/31/2016	UT
Dimethyl phthalate	8/1/2015	7/31/2016	UT
Di-n-butyl phthalate	8/1/2015	7/31/2016	UT
Di-n-octyl phthalate	8/1/2015	7/31/2016	UT
Fluoranthene	8/1/2015	7/31/2016	UT
Fluorene	8/1/2015	7/31/2016	UT
Hexachlorobenzene	8/1/2015	7/31/2016	UT
Hexachlorobutadiene	8/1/2015	7/31/2016	UT
Hexachlorocyclopentadiene	8/1/2015	7/31/2016	UT
Hexachloroethane	8/1/2015	7/31/2016	UT
Indeno(1,2,3-cd) pyrene	8/1/2015	7/31/2016	UT
Isophorone	8/1/2015	7/31/2016	UT
Naphthalene	8/1/2015	7/31/2016	UT
Nitrobenzene	8/1/2015	7/31/2016	UT
n-Nitrosodimethylamine	8/1/2015	7/31/2016	UT
n-Nitrosodi-n-propylamine	8/1/2015	7/31/2016	UT
n-Nitrosodiphenylamine	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Non Potable Water)***			
Pentachlorophenol	8/1/2015	7/31/2016	UT
Phenanthrene	8/1/2015	7/31/2016	UT
Phenol	8/1/2015	7/31/2016	UT
Pyrene	8/1/2015	7/31/2016	UT
Method EPA 9012B			
Amenable cyanide	8/1/2015	7/31/2016	UT
Total cyanide	8/1/2015	7/31/2016	UT
Method EPA 9040C			
pH	8/1/2015	7/31/2016	UT
Method EPA 9070A			
Oil & Grease	8/1/2015	7/31/2016	UT
Method EPA 9310			
Gross alpha-beta	8/1/2015	7/31/2016	UT
Method EPA 9315			
Total alpha radium	8/1/2015	7/31/2016	UT
Method EPA 9320			
Radium-228	8/1/2015	7/31/2016	UT
Method SM 4500-CN$^-$ C-1999			
Total cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-CN$^-$ E-1999			
Total cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-CN$^-$ I-2011			
Weak Acid Dissociable Cyanide	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

	Start Date	Expires	AB
Method ASTM D5744-13			
Preparation/Extraction	8/1/2015	7/31/2016	UT
Method ASTM E2242-12			
Meteoric water mobility procedure	8/1/2015	7/31/2016	UT
Method EPA 1010A			
Ignitability	8/1/2015	7/31/2016	UT
Method EPA 1311			
Toxicity Characteristic Leaching Procedure (TCLP)	8/1/2015	7/31/2016	UT
Method EPA 1312			
Synthetic Precipitation Leaching Procedure (SCLP)	8/1/2015	7/31/2016	UT
Method EPA 3050B			
Acid Digestion of Solids	8/1/2015	7/31/2016	UT
Method EPA 3051A			
Microwave Digestion of Solids	8/1/2015	7/31/2016	UT
Method EPA 3052			
Preparation/Extraction	8/1/2015	7/31/2016	UT
Method EPA 3060A			
Preparation/Extraction	8/1/2015	7/31/2016	UT
Method EPA 3540C			
Soxhlet Extraction	8/1/2015	7/31/2016	UT
Method EPA 3546			
Microwave Extraction	8/1/2015	7/31/2016	UT
Method EPA 3550C			
Ultrasonic Extraction	8/1/2015	7/31/2016	UT
Method EPA 3580A			
Waste Dilution	8/1/2015	7/31/2016	UT
Method EPA 3630C			
Silica Gel Clean-up	8/1/2015	7/31/2016	UT
Method EPA 5035			
Solid-Phase Purge & Trap	8/1/2015	7/31/2016	UT
Method EPA 6010B			
Aluminum	8/1/2015	7/31/2016	UT
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Boron	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Calcium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT
Cobalt	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Iron	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Lithium	8/1/2015	7/31/2016	UT

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Solid & Hazardous Material)***			
Magnesium	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Molybdenum	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Phosphorus, total	8/1/2015	7/31/2016	UT
Potassium	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silica as SiO2	8/1/2015	7/31/2016	UT
Silicon	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Sodium	8/1/2015	7/31/2016	UT
Strontium	8/1/2015	7/31/2016	UT
Thallium	8/1/2015	7/31/2016	UT
Tin	8/1/2015	7/31/2016	UT
Titanium	8/1/2015	7/31/2016	UT
Vanadium	8/1/2015	7/31/2016	UT
Zinc	8/1/2015	7/31/2016	UT
Method EPA 6020			
Aluminum	8/1/2015	7/31/2016	UT
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT
Cobalt	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Molybdenum	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Thallium	8/1/2015	7/31/2016	UT
Uranium	8/1/2015	7/31/2016	UT
Vanadium	8/1/2015	7/31/2016	UT
Zinc	8/1/2015	7/31/2016	UT
Method EPA 7196A			
Chromium VI	8/1/2015	7/31/2016	UT
Method EPA 7471A			
Mercury	8/1/2015	7/31/2016	UT
Method EPA 7473			
Mercury	8/1/2015	7/31/2016	UT
Method EPA 8015D			
Diesel range organics (DRO)	8/1/2015	7/31/2016	UT
Gasoline range organics (GRO)	8/1/2015	7/31/2016	UT
Method EPA 8021B			

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Solid & Hazardous Material)***			
Benzene	8/1/2015	7/31/2016	UT
Ethylbenzene	8/1/2015	7/31/2016	UT
m-Xylene	8/1/2015	7/31/2016	UT
o-Xylene	8/1/2015	7/31/2016	UT
p-Xylene	8/1/2015	7/31/2016	UT
Toluene	8/1/2015	7/31/2016	UT
Xylene (total)	8/1/2015	7/31/2016	UT
Method EPA 8082A			
Aroclor-1016 (PCB-1016)	8/1/2015	7/31/2016	UT
Aroclor-1221 (PCB-1221)	8/1/2015	7/31/2016	UT
Aroclor-1232 (PCB-1232)	8/1/2015	7/31/2016	UT
Aroclor-1242 (PCB-1242)	8/1/2015	7/31/2016	UT
Aroclor-1248 (PCB-1248)	8/1/2015	7/31/2016	UT
Aroclor-1254 (PCB-1254)	8/1/2015	7/31/2016	UT
Aroclor-1260 (PCB-1260)	8/1/2015	7/31/2016	UT
Aroclor-1262 (PCB-1262)	8/1/2015	7/31/2016	UT
Aroclor-1268 (PCB-1268)	8/1/2015	7/31/2016	UT
Method EPA 8260B			
1,1,1,2-Tetrachloroethane	8/1/2015	7/31/2016	UT
1,1,1-Trichloroethane	8/1/2015	7/31/2016	UT
1,1,2,2-Tetrachloroethane	8/1/2015	7/31/2016	UT
1,1,2-Trichloroethane	8/1/2015	7/31/2016	UT
1,1-Dichloroethane	8/1/2015	7/31/2016	UT
1,1-Dichloroethylene	8/1/2015	7/31/2016	UT
1,1-Dichloropropene	8/1/2015	7/31/2016	UT
1,2,3-Trichlorobenzene	8/1/2015	7/31/2016	UT
1,2,3-Trichloropropane	8/1/2015	7/31/2016	UT
1,2,4-Trichlorobenzene	8/1/2015	7/31/2016	UT
1,2,4-Trimethylbenzene	8/1/2015	7/31/2016	UT
1,2-Dibromo-3-chloropropane (DBCP)	8/1/2015	7/31/2016	UT
1,2-Dibromoethane (EDB, Ethylene dibromide)	8/1/2015	7/31/2016	UT
1,2-Dichlorobenzene (o-Dichlorobenzene)	8/1/2015	7/31/2016	UT
1,2-Dichloroethane (Ethylene dichloride)	8/1/2015	7/31/2016	UT
1,2-Dichloropropane	8/1/2015	7/31/2016	UT
1,3,5-Trimethylbenzene	8/1/2015	7/31/2016	UT
1,3-Dichlorobenzene	8/1/2015	7/31/2016	UT
1,3-Dichloropropane	8/1/2015	7/31/2016	UT
1,4-Dichlorobenzene	8/1/2015	7/31/2016	UT
2,2-Dichloropropane	8/1/2015	7/31/2016	UT
2-Butanone (Methyl ethyl ketone, MEK)	8/1/2015	7/31/2016	UT
2-Chloroethyl vinyl ether	8/1/2015	7/31/2016	UT
2-Chlorotoluene	8/1/2015	7/31/2016	UT
2-Hexanone	8/1/2015	7/31/2016	UT
4-Chlorotoluene	8/1/2015	7/31/2016	UT
4-Methyl-2-pentanone (MIBK)	8/1/2015	7/31/2016	UT
Acetone	8/1/2015	7/31/2016	UT
Acrylonitrile	8/1/2015	7/31/2016	UT
Benzene	8/1/2015	7/31/2016	UT

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Solid & Hazardous Material)***			
Bromobenzene	8/1/2015	7/31/2016	UT
Bromochloromethane	8/1/2015	7/31/2016	UT
Bromodichloromethane	8/1/2015	7/31/2016	UT
Bromoform	8/1/2015	7/31/2016	UT
Carbon disulfide	8/1/2015	7/31/2016	UT
Carbon tetrachloride	8/1/2015	7/31/2016	UT
Chlorobenzene	8/1/2015	7/31/2016	UT
Chlorodibromomethane	8/1/2015	7/31/2016	UT
Chloroethane (Ethyl chloride)	8/1/2015	7/31/2016	UT
Chloroform	8/1/2015	7/31/2016	UT
cis-1,2-Dichloroethylene	8/1/2015	7/31/2016	UT
cis-1,3-Dichloropropene	8/1/2015	7/31/2016	UT
Dibromomethane (Methylene bromide)	8/1/2015	7/31/2016	UT
Dichlorodifluoromethane (Freon-12)	8/1/2015	7/31/2016	UT
Ethylbenzene	8/1/2015	7/31/2016	UT
Hexachlorobutadiene	8/1/2015	7/31/2016	UT
Isopropylbenzene	8/1/2015	7/31/2016	UT
Methyl bromide (Bromomethane)	8/1/2015	7/31/2016	UT
Methyl chloride (Chloromethane)	8/1/2015	7/31/2016	UT
Methyl tert-butyl ether (MTBE)	8/1/2015	7/31/2016	UT
Methylene chloride (Dichloromethane)	8/1/2015	7/31/2016	UT
m-Xylene	8/1/2015	7/31/2016	UT
Naphthalene	8/1/2015	7/31/2016	UT
n-Butylbenzene	8/1/2015	7/31/2016	UT
n-Propylbenzene	8/1/2015	7/31/2016	UT
o-Xylene	8/1/2015	7/31/2016	UT
p-Xylene	8/1/2015	7/31/2016	UT
sec-Butylbenzene	8/1/2015	7/31/2016	UT
Styrene	8/1/2015	7/31/2016	UT
tert-Butylbenzene	8/1/2015	7/31/2016	UT
Tetrachloroethylene (Perchloroethylene)	8/1/2015	7/31/2016	UT
Toluene	8/1/2015	7/31/2016	UT
trans-1,2-Dichloroethylene	8/1/2015	7/31/2016	UT
trans-1,3-Dichloropropylene	8/1/2015	7/31/2016	UT
Trichloroethene (Trichloroethylene)	8/1/2015	7/31/2016	UT
Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	8/1/2015	7/31/2016	UT
Vinyl acetate	8/1/2015	7/31/2016	UT
Vinyl chloride	8/1/2015	7/31/2016	UT
Xylene (total)	8/1/2015	7/31/2016	UT
Method EPA 8270C			
1,2,4-Trichlorobenzene	8/1/2015	7/31/2016	UT
1,2-Dichlorobenzene (o-Dichlorobenzene)	8/1/2015	7/31/2016	UT
1,3-Dichlorobenzene	8/1/2015	7/31/2016	UT
1,4-Dichlorobenzene	8/1/2015	7/31/2016	UT
2,4,5-Trichlorophenol	8/1/2015	7/31/2016	UT
2,4,6-Trichlorophenol	8/1/2015	7/31/2016	UT
2,4-Dichlorophenol	8/1/2015	7/31/2016	UT
2,4-Dimethylphenol	8/1/2015	7/31/2016	UT

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

	Start Date	Expires	AB
2,4-Dinitrophenol	8/1/2015	7/31/2016	UT
2,4-Dinitrotoluene (2,4-DNT)	8/1/2015	7/31/2016	UT
2,6-Dinitrotoluene (2,6-DNT)	8/1/2015	7/31/2016	UT
2-Chloronaphthalene	8/1/2015	7/31/2016	UT
2-Chlorophenol	8/1/2015	7/31/2016	UT
2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	8/1/2015	7/31/2016	UT
2-Methylnaphthalene	8/1/2015	7/31/2016	UT
2-Methylphenol (o-Cresol)	8/1/2015	7/31/2016	UT
2-Nitroaniline	8/1/2015	7/31/2016	UT
2-Nitrophenol	8/1/2015	7/31/2016	UT
3,3'-Dichlorobenzidine	8/1/2015	7/31/2016	UT
3-Methylphenol (m-Cresol)	8/1/2015	7/31/2016	UT
3-Nitroaniline	8/1/2015	7/31/2016	UT
4-Bromophenyl phenyl ether	8/1/2015	7/31/2016	UT
4-Chloro-3-methylphenol	8/1/2015	7/31/2016	UT
4-Chloroaniline	8/1/2015	7/31/2016	UT
4-Chlorophenyl phenylether	8/1/2015	7/31/2016	UT
4-Methylphenol (p-Cresol)	8/1/2015	7/31/2016	UT
4-Nitroaniline	8/1/2015	7/31/2016	UT
4-Nitrophenol	8/1/2015	7/31/2016	UT
Acenaphthene	8/1/2015	7/31/2016	UT
Acenaphthylene	8/1/2015	7/31/2016	UT
Anthracene	8/1/2015	7/31/2016	UT
Azobenzene (1,2-Diphenylhydrazine)	8/1/2015	7/31/2016	UT
Benzo(a)anthracene	8/1/2015	7/31/2016	UT
Benzo(a)pyrene	8/1/2015	7/31/2016	UT
Benzo(b)fluoranthene	8/1/2015	7/31/2016	UT
Benzo(g,h,i)perylene	8/1/2015	7/31/2016	UT
Benzo(k)fluoranthene	8/1/2015	7/31/2016	UT
Benzoic acid	8/1/2015	7/31/2016	UT
Benzyl alcohol	8/1/2015	7/31/2016	UT
bis(2-Chloroethoxy)methane	8/1/2015	7/31/2016	UT
bis(2-Chloroethyl) ether	8/1/2015	7/31/2016	UT
bis(2-Chloroisopropyl) ether	8/1/2015	7/31/2016	UT
Butyl benzyl phthalate	8/1/2015	7/31/2016	UT
Chrysene	8/1/2015	7/31/2016	UT
Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	8/1/2015	7/31/2016	UT
Dibenz(a,h) anthracene	8/1/2015	7/31/2016	UT
Dibenzofuran	8/1/2015	7/31/2016	UT
Diethyl phthalate	8/1/2015	7/31/2016	UT
Dimethyl phthalate	8/1/2015	7/31/2016	UT
Di-n-butyl phthalate	8/1/2015	7/31/2016	UT
Di-n-octyl phthalate	8/1/2015	7/31/2016	UT
Fluoranthene	8/1/2015	7/31/2016	UT
Fluorene	8/1/2015	7/31/2016	UT
Hexachlorobenzene	8/1/2015	7/31/2016	UT
Hexachlorobutadiene	8/1/2015	7/31/2016	UT
Hexachlorocyclopentadiene	8/1/2015	7/31/2016	UT
Hexachloroethane	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***RCRA (Solid & Hazardous Material)***			
Indeno(1,2,3-cd) pyrene	8/1/2015	7/31/2016	UT
Isophorone	8/1/2015	7/31/2016	UT
Naphthalene	8/1/2015	7/31/2016	UT
Nitrobenzene	8/1/2015	7/31/2016	UT
n-Nitrosodimethylamine	8/1/2015	7/31/2016	UT
n-Nitrosodi-n-propylamine	8/1/2015	7/31/2016	UT
n-Nitrosodiphenylamine	8/1/2015	7/31/2016	UT
Pentachlorophenol	8/1/2015	7/31/2016	UT
Phenanthrene	8/1/2015	7/31/2016	UT
Phenol	8/1/2015	7/31/2016	UT
Pyrene	8/1/2015	7/31/2016	UT
Method EPA 9012B			
Amenable cyanide	8/1/2015	7/31/2016	UT
Total cyanide	8/1/2015	7/31/2016	UT
Method EPA 903.1			
Radium-226	8/1/2015	7/31/2016	UT
Method EPA 9045D			
pH	8/1/2015	7/31/2016	UT
Method EPA 9071B			
Oil & Grease	8/1/2015	7/31/2016	UT
Method EPA 9310			
Gross alpha-beta	8/1/2015	7/31/2016	UT
Method EPA 9315			
Total alpha radium	8/1/2015	7/31/2016	UT
Method EPA 9320			
Radium-228	8/1/2015	7/31/2016	UT
Method SM 4500-CN^{-} C-1999			
Total cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-CN^{-} E-1999			
Total cyanide	8/1/2015	7/31/2016	UT
Method SM 4500-CN^{-} I-2011			
Weak Acid Dissociable Cyanide	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

Program/Matrix: ***SDWA (Potable Water)***

	Start Date	Expires	AB
Method ASTM D6888-04			
Cyanide	8/1/2015	7/31/2016	UT
Method EPA 180.1			
Turbidity	8/1/2015	7/31/2016	UT
Method EPA 200.7			
Aluminum	8/1/2015	7/31/2016	UT
Calcium	8/1/2015	7/31/2016	UT
Iron	8/1/2015	7/31/2016	UT
Magnesium	8/1/2015	7/31/2016	UT
Manganese	8/1/2015	7/31/2016	UT
Silica as SiO2	8/1/2015	7/31/2016	UT
Sodium	8/1/2015	7/31/2016	UT
Strontium	8/1/2015	7/31/2016	UT
Zinc	8/1/2015	7/31/2016	UT
Method EPA 200.8			
Antimony	8/1/2015	7/31/2016	UT
Arsenic	8/1/2015	7/31/2016	UT
Barium	8/1/2015	7/31/2016	UT
Beryllium	8/1/2015	7/31/2016	UT
Cadmium	8/1/2015	7/31/2016	UT
Chromium	8/1/2015	7/31/2016	UT
Copper	8/1/2015	7/31/2016	UT
Lead	8/1/2015	7/31/2016	UT
Nickel	8/1/2015	7/31/2016	UT
Selenium	8/1/2015	7/31/2016	UT
Silver	8/1/2015	7/31/2016	UT
Thallium	8/1/2015	7/31/2016	UT
Uranium	8/1/2015	7/31/2016	UT
Method EPA 245.1			
Mercury	8/1/2015	7/31/2016	UT
Method EPA 300.0			
Bromide	8/1/2015	7/31/2016	UT
Chloride	8/1/2015	7/31/2016	UT
Fluoride	8/1/2015	7/31/2016	UT
Sulfate	8/1/2015	7/31/2016	UT
Method EPA 335.4			
Cyanide	8/1/2015	7/31/2016	UT
Method EPA 353.2			
Nitrate as N	8/1/2015	7/31/2016	UT
Nitrate-nitrite	8/1/2015	7/31/2016	UT
Nitrite as N	8/1/2015	7/31/2016	UT
Method EPA 900.0			
Gross-alpha	8/1/2015	7/31/2016	UT
Gross-beta	8/1/2015	7/31/2016	UT
Method EPA 903			
Radium-226	8/1/2015	7/31/2016	UT

ACZ Laboratories, Inc.

	Start Date	Expires	AB
Program/Matrix: ***SDWA (Potable Water)***			
Total alpha radium	8/1/2015	7/31/2016	UT
Method EPA 903.1			
Radium-226	8/1/2015	7/31/2016	UT
Method EPA 904			
Radium-228	8/1/2015	7/31/2016	UT
Method SM 2320 B-1997			
Alkalinity as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2320 B-2011			
Alkalinity as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2340 B-1997			
Total hardness as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2340 B-2011			
Total hardness as CaCO3	8/1/2015	7/31/2016	UT
Method SM 2510 B-1997			
Conductivity	8/1/2015	7/31/2016	UT
Method SM 2510 B-2011			
Conductivity	8/1/2015	7/31/2016	UT
Method SM 2540 C-1997			
Residue-filterable (TDS)	8/1/2015	7/31/2016	UT
Method SM 2540 C-2011			
Residue-filterable (TDS)	8/1/2015	7/31/2016	UT
Method SM 4500-F⁻ C-1997			
Fluoride	8/1/2015	7/31/2016	UT
Method SM 4500-F⁻ C-2011			
Fluoride	8/1/2015	7/31/2016	UT
Method SM 4500-H+ B-1996			
pH	8/1/2015	7/31/2016	UT
Method SM 4500-H+ B-2011			
pH	8/1/2015	7/31/2016	UT
Method SM 5310 B-1996			
Total organic carbon	8/1/2015	7/31/2016	UT
Method SM 5310 B-2011			
Total organic carbon	8/1/2015	7/31/2016	UT

The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data users to verify the most current certification letter for the authorized method.

The analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. It is the certified laboratory's responsibility to review this letter for discrepancies. The certified laboratory must document any discrepancies in this letter and send notice to this bureau within 15 days of receipt. This certificate letter will be recalled in the event your laboratory's certification is revoked.

State of Florida
Department of Health, Bureau of Public Health Laboratories
This is to certify that

E87893

ANATEK LABS, INC.
1282 ALTURAS DR.
MOSCOW, ID 83843

has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

DRINKING WATER - GROUP I UNREGULATED CONTAMINANTS, DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - GROUP III UNREGULATED CONTAMINANTS, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, DRINKING WATER - RADIOCHEMISTRY, DRINKING WATER - SYNTHETIC ORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2015 Expiration Date: June 30, 2016

Carina Blackmore, DVM, PhD, Dipl. ACVPM, CPM
Chief, Bureau of Public Health Laboratories
DH Form 1697, 7/04
NON-TRANSFERABLE E87893-58-07/01/2015
Supersedes all previously issued certificates

Laboratory Scope of Accreditation

Attachment to Certificate #: E87893-58, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87893** EPA Lab Code: **ID00013** **(208) 883-2839**

E87893
Anatek Labs, Inc.
1282 Alturas Dr.
Moscow, ID 83843

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,1,1-Trichloroethane	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
1,1,2,2-Tetrachloroethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,1,2-Trichloroethane	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
1,1-Dichloroethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,1-Dichloroethylene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
1,1-Dichloropropene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,2,3-Trichlorobenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,2,3-Trichloropropane	EPA 504.1	Synthetic Organic Contaminants	NELAP	4/7/2008
1,2,3-Trichloropropane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,2,4-Trichlorobenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,2,4-Trimethylbenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	4/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	4/1/2003
1,2-Dichlorobenzene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
1,2-Dichloroethane	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
1,2-Dichloropropane	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
1,3,5-Trimethylbenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,3-Dichlorobenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,3-Dichloropropane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
1,4-Dichlorobenzene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
2,4,5-T	EPA 515.4	Group I Unregulated Contaminants	NELAP	9/29/2014
2,4-D	EPA 515.4	Synthetic Organic Contaminants	NELAP	9/29/2014
2,4-DB	EPA 515.4	Group I Unregulated Contaminants	NELAP	9/29/2014
2,4-Dinitrotoluene (2,4-DNT)	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
2-Chlorotoluene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
3-Hydroxycarbofuran	EPA 531.2	Group I Unregulated Contaminants	NELAP	10/29/2012
4,4'-DDD	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
4,4'-DDE	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
4,4'-DDT	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
4-Chlorotoluene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
4-Isopropyltoluene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Acenaphthylene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Acetochlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Acifluorfen	EPA 515.4	Group I Unregulated Contaminants	NELAP	9/29/2014

Laboratory Scope of Accreditation

Attachment to Certificate #: E87893-58, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87893** EPA Lab Code: **ID00013** **(208) 883-2839**

E87893
Anatek Labs, Inc.
1282 Alturas Dr.
Moscow, ID 83843

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Alachlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	4/1/2003
Aldicarb (Temik)	EPA 531.2	Group I Unregulated Contaminants	NELAP	10/29/2012
Aldicarb sulfone	EPA 531.2	Group I Unregulated Contaminants	NELAP	10/29/2012
Aldicarb sulfoxide	EPA 531.2	Group I Unregulated Contaminants	NELAP	10/29/2012
Aldrin	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Alkalinity as CaCO3	SM 2320 B	Primary Inorganic Contaminants	NELAP	8/25/2005
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
alpha-Chlordane	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	7/7/2010
Aluminum	EPA 200.8	Secondary Inorganic Contaminants	NELAP	4/1/2003
Anthracene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Aroclor-1016 (PCB-1016)	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Aroclor-1221 (PCB-1221)	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Aroclor-1232 (PCB-1232)	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Aroclor-1242 (PCB-1242)	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Aroclor-1248 (PCB-1248)	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Aroclor-1254 (PCB-1254)	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Aroclor-1260 (PCB-1260)	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Arsenic	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Atrazine	EPA 525.2	Synthetic Organic Contaminants	NELAP	4/1/2003
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Benzene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Benzo(a)anthracene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Benzo(a)pyrene	EPA 525.2	Synthetic Organic Contaminants	NELAP	4/1/2003
Benzo(b)fluoranthene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Benzo(g,h,i)perylene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Benzo(k)fluoranthene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 525.2	Synthetic Organic Contaminants	NELAP	4/1/2003
Bromacil	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Bromoacetic acid	SM 6251 B	Group I Unregulated Contaminants	NELAP	4/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Bromobenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Bromochloroacetic acid	SM 6251 B	Group I Unregulated Contaminants	NELAP	4/1/2003
Bromochloromethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Bromodichloromethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Bromoform	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Butachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Butyl benzyl phthalate	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Carbaryl (Sevin)	EPA 531.2	Group I Unregulated Contaminants	NELAP	10/29/2012
Carbofuran (Furadan)	EPA 531.2	Synthetic Organic Contaminants	NELAP	10/29/2012
Carbon disulfide	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Carbon tetrachloride	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Chlordane (tech.)	EPA 505	Synthetic Organic Contaminants	NELAP	4/1/2003
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	5/7/2003
Chloroacetic acid	SM 6251 B	Group I Unregulated Contaminants	NELAP	4/1/2003
Chlorobenzene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Chloroform	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Chlorothalonil	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Chrysene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
cis-1,2-Dichloroethylene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
cis-1,3-Dichloropropene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	11/26/2007
Conductivity	SM 2510 B	Primary Inorganic Contaminants	NELAP	8/25/2005
Copper	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Copper	EPA 200.8	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	4/1/2003
Cyanazine (Bladex)	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Dacthal Acid Metabolites	EPA 515.4	Group I Unregulated Contaminants	NELAP	9/29/2014
Dalapon	EPA 515.4	Synthetic Organic Contaminants	NELAP	9/29/2014
delta-BHC	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Di(2-ethylhexyl)adipate	EPA 525.2	Synthetic Organic Contaminants	NELAP	4/1/2003
Diazinon	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005

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Anatek Labs, Inc.
1282 Alturas Dr.
Moscow, ID 83843

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibenz(a,h)anthracene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Dibromoacetic acid	SM 6251 B	Group I Unregulated Contaminants	NELAP	4/1/2003
Dibromochloromethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Dibromomethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Dicamba	EPA 515.4	Group I Unregulated Contaminants	NELAP	9/29/2014
Dichloroacetic acid	SM 6251 B	Group I Unregulated Contaminants	NELAP	4/1/2003
Dichlorodifluoromethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Dichloroprop (Dichlorprop)	EPA 515.4	Group I Unregulated Contaminants	NELAP	9/29/2014
Dieldrin	EPA 505	Group I Unregulated Contaminants	NELAP	8/25/2005
Diethyl ether	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Diethyl phthalate	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Dimethyl phthalate	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Di-n-butyl phthalate	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 515.4	Synthetic Organic Contaminants	NELAP	9/29/2014
Diquat	EPA 549.2	Synthetic Organic Contaminants	NELAP	4/1/2003
Endothall	EPA 548.1	Synthetic Organic Contaminants	NELAP	4/1/2003
Endrin	EPA 505	Synthetic Organic Contaminants	NELAP	10/20/2006
EPTC (Eptam, s-ethyl-dipropyl thio carbamate)	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Ethylbenzene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Fluorene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Fluoride	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	4/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 505	Synthetic Organic Contaminants	NELAP	4/1/2003
gamma-Chlordane	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Glyphosate	EPA 547	Synthetic Organic Contaminants	NELAP	4/1/2003
Hardness	SM 2340 B	Secondary Inorganic Contaminants	NELAP	6/5/2013
Heptachlor	EPA 505	Synthetic Organic Contaminants	NELAP	4/1/2003
Heptachlor epoxide	EPA 505	Synthetic Organic Contaminants	NELAP	4/1/2003
Hexachlorobenzene	EPA 525.2	Synthetic Organic Contaminants	NELAP	5/7/2003
Hexachlorobutadiene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Hexachlorocyclopentadiene	EPA 525.2	Synthetic Organic Contaminants	NELAP	4/1/2003
Indeno(1,2,3-cd)pyrene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Iodomethane (Methyl iodide)	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	7/7/2010
Isopropylbenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003

Laboratory Scope of Accreditation

Attachment to Certificate #: E87893-58, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87893** EPA Lab Code: **ID00013** **(208) 883-2839**

E87893
Anatek Labs, Inc.
1282 Alturas Dr.
Moscow, ID 83843

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	7/7/2010
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	4/1/2003
Mercury	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Mercury	EPA 245.7	Primary Inorganic Contaminants	NELAP	11/26/2007
Methiocarb (Mesurol)	EPA 531.2	Group I Unregulated Contaminants	NELAP	10/29/2012
Methomyl (Lannate)	EPA 531.2	Group I Unregulated Contaminants	NELAP	10/29/2012
Methoxychlor	EPA 505	Synthetic Organic Contaminants	NELAP	10/20/2006
Methyl bromide (Bromomethane)	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Methyl chloride (Chloromethane)	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Methyl tert-butyl ether (MTBE)	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Methylene chloride	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Metolachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Metribuzin	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Molinate	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
m-Xylene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Naphthalene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
n-Butylbenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Nitrate as N	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/1/2003
Nitrate as N	SM 4500-NO3 F	Primary Inorganic Contaminants	NELAP	4/1/2003
Nitrate-nitrite	SM 4500-NO3 F	Primary Inorganic Contaminants	NELAP	7/7/2010
Nitrite	SM 4500-NO3 F	Primary Inorganic Contaminants	NELAP	4/1/2003
Nitrite as N	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/1/2003
n-Propylbenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Orthophosphate as P	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/1/2003
Oxamyl	EPA 531.2	Synthetic Organic Contaminants	NELAP	10/29/2012
o-Xylene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
PCBs	EPA 505	Synthetic Organic Contaminants	NELAP	4/1/2003
Pentachlorophenol	EPA 515.4	Synthetic Organic Contaminants	NELAP	9/29/2014
Perchlorate	EPA 331.0	Secondary Inorganic Contaminants	NELAP	7/7/2010
pH	SM 4500-H+-B	Primary Inorganic Contaminants	NELAP	11/26/2007
Phenanthrene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
Picloram	EPA 515.4	Synthetic Organic Contaminants	NELAP	9/29/2014
Potassium	EPA 200.7	Secondary Inorganic Contaminants	NELAP	7/7/2010

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Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Prometon	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Propachlor (Ramrod)	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Propoxur (Baygon)	EPA 531.2	Group I Unregulated Contaminants	NELAP	10/29/2012
p-Xylene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Pyrene	EPA 525.2	Group III Unregulated Contaminants	NELAP	8/25/2005
sec-Butylbenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Silver	EPA 200.8	Secondary Inorganic Contaminants	NELAP	4/1/2003
Silvex (2,4,5-TP)	EPA 515.4	Synthetic Organic Contaminants	NELAP	9/29/2014
Simazine	EPA 525.2	Synthetic Organic Contaminants	NELAP	4/1/2003
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2010
Styrene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Sulfate	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	4/1/2003
Surfactants - MBAS	SM 5540 C	Secondary Inorganic Contaminants	NELAP	7/7/2010
Terbacil	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
tert-Butylbenzene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Tetrachloroethylene (Perchloroethylene)	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/1/2003
Toluene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Total cyanide	EPA 335.4	Primary Inorganic Contaminants	NELAP	8/25/2005
Total dissolved solids	SM 2540 C	Secondary Inorganic Contaminants	NELAP	11/26/2007
Total haloacetic acids (HAA5)	SM 6251 B	Synthetic Organic Contaminants	NELAP	4/1/2003
Total nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/1/2003
Total organic carbon	SM 5310 B	Primary Inorganic Contaminants	NELAP	2/25/2013
Total trihalomethanes	EPA 524.3	Other Regulated Contaminants	NELAP	9/29/2014
Toxaphene (Chlorinated camphene)	EPA 505	Synthetic Organic Contaminants	NELAP	4/1/2003
trans-1,2-Dichloroethylene	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
trans-1,3-Dichloropropene	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
trans-Nonachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	8/25/2005
Trichloroacetic acid	SM 6251 B	Group I Unregulated Contaminants	NELAP	4/1/2003
Trichloroethene (Trichloroethylene)	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Trichlorofluoromethane	EPA 524.3	Group II Unregulated Contaminants	NELAP	8/29/2013
Turbidity	EPA 180.1	Secondary Inorganic Contaminants	NELAP	3/12/2010
Uranium	EPA 200.8	Radiochemistry	NELAP	11/26/2007
Vinyl chloride	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013

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Moscow, ID 83843

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Xylene (total)	EPA 524.3	Other Regulated Contaminants	NELAP	8/29/2013
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	4/1/2003

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E87893
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Moscow, ID 83843

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	11/14/2003
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	11/14/2003
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	11/14/2003
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	11/14/2003
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	11/14/2003
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2003
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	8/25/2005
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	11/14/2003
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2003
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	8/25/2005
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	11/14/2003
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	11/14/2003
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	8/25/2005
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	11/14/2003
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2003
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	8/25/2005
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	11/14/2003

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1282 Alturas Dr.
Moscow, ID 83843

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2003
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	8/25/2005
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	8/25/2005
2,2'-Oxybis(1-chloropropane),bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether	EPA 625	Extractable Organics	NELAP	8/25/2005
2,2'-Oxybis(1-chloropropane),bis(2-Chloro-1-methylethyl)ether (fka bis(2-Chloroisopropyl) ether	EPA 8270	Extractable Organics	NELAP	8/25/2005
2,3,4,6-Tetrachlorophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	11/26/2007
2,4,5-Trichlorophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2003
2,4,6-Trichlorophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	11/26/2007
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	11/26/2007
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	10/15/2014
2,4-Dichlorophenol	EPA 8270	Volatile Organics	NELAP	10/15/2014
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	4/1/2003
2,4-Dimethylphenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2003
2,4-Dinitrophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	4/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	8/25/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	3/19/2012
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	3/19/2012
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	8/25/2005
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	11/14/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	12/1/2011
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	12/1/2011
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2003
2-Chlorophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	8/25/2005
2-Hexanone	EPA 8260	Volatile Organics	NELAP	8/25/2005
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2003
2-Methyl-4,6-dinitrophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	8/25/2005
2-Methylphenol (o-Cresol)	EPA 8270	Volatile Organics	NELAP	8/25/2005

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	8/25/2005
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2003
2-Nitrophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	4/1/2003
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	8/25/2005
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	8/25/2005
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	8/25/2005
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	8/25/2005
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	4/1/2003
4-Chloro-3-methylphenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	8/25/2005
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	8/25/2005
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	8/25/2005
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	8/25/2005
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	8/25/2005
4-Methylphenol (p-Cresol)	EPA 8270	Volatile Organics	NELAP	8/25/2005
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	8/25/2005
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2003
4-Nitrophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
Acenaphthene	EPA 625	Extractable Organics	NELAP	4/1/2003
Acenaphthene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Acenaphthylene	EPA 625	Extractable Organics	NELAP	4/1/2003
Acenaphthylene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Acetone	EPA 8260	Volatile Organics	NELAP	8/25/2005
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	8/25/2005
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	8/25/2005
Acrylonitrile	EPA 624	Volatile Organics	NELAP	8/25/2005
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	8/25/2005
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	1/6/2009
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Aluminum	EPA 200.7	Metals	NELAP	7/7/2010
Aluminum	EPA 200.8	Metals	NELAP	4/1/2003
Aluminum	EPA 6010	Metals	NELAP	7/7/2010
Aluminum	EPA 6020	Metals	NELAP	8/25/2005
Ammonia as N	SM 4500-NH3 G	General Chemistry	NELAP	8/12/2003
Aniline	EPA 8270	Extractable Organics	NELAP	8/25/2005
Anthracene	EPA 625	Extractable Organics	NELAP	4/1/2003
Anthracene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Antimony	EPA 200.7	Metals	NELAP	7/7/2010
Antimony	EPA 200.8	Metals	NELAP	4/1/2003
Antimony	EPA 6010	Metals	NELAP	7/7/2010
Antimony	EPA 6020	Metals	NELAP	8/25/2005
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/1/2009
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/1/2009
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	12/19/2006
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	12/19/2006
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/1/2009
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/1/2009
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/3/2008
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/3/2008
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/1/2009
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/1/2009
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/29/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/29/2007
Arsenic	EPA 200.7	Metals	NELAP	7/7/2010
Arsenic	EPA 200.8	Metals	NELAP	4/1/2003
Arsenic	EPA 6010	Metals	NELAP	7/7/2010
Arsenic	EPA 6020	Metals	NELAP	8/25/2005
Azinphos-methyl (Guthion)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Barium	EPA 200.7	Metals	NELAP	7/7/2010
Barium	EPA 200.8	Metals	NELAP	4/1/2003

Laboratory Scope of Accreditation

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State Laboratory ID: **E87893** EPA Lab Code: **ID00013** **(208) 883-2839**

E87893
Anatek Labs, Inc.
1282 Alturas Dr.
Moscow, ID 83843

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Barium	EPA 6010	Metals	NELAP	7/7/2010
Barium	EPA 6020	Metals	NELAP	8/25/2005
Benzene	EPA 624	Volatile Organics	NELAP	11/14/2003
Benzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Benzidine	EPA 625	Extractable Organics	NELAP	4/1/2003
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	4/1/2003
Benzo(a)anthracene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	4/1/2003
Benzo(a)pyrene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2003
Benzo(b)fluoranthene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	4/1/2003
Benzo(g,h,i)perylene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2003
Benzo(k)fluoranthene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	8/25/2005
Beryllium	EPA 200.7	Metals	NELAP	7/7/2010
Beryllium	EPA 200.8	Metals	NELAP	4/1/2003
Beryllium	EPA 6010	Metals	NELAP	7/7/2010
Beryllium	EPA 6020	Metals	NELAP	8/25/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	8/25/2005
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	8/25/2005
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	8/25/2005
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	8/25/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	4/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	8/25/2005
Boron	EPA 200.7	Metals	NELAP	7/7/2010
Boron	EPA 6010	Metals	NELAP	7/7/2010
Bromobenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	11/14/2003
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
Bromoform	EPA 624	Volatile Organics	NELAP	11/14/2003
Bromoform	EPA 8260	Volatile Organics	NELAP	8/25/2005

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2003
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	8/25/2005
Cadmium	EPA 200.7	Metals	NELAP	7/7/2010
Cadmium	EPA 200.8	Metals	NELAP	4/1/2003
Cadmium	EPA 6010	Metals	NELAP	7/7/2010
Cadmium	EPA 6020	Metals	NELAP	8/25/2005
Calcium	EPA 200.7	Metals	NELAP	7/7/2010
Calcium	EPA 6010	Metals	NELAP	7/7/2010
Calcium	EPA 6020	Metals	NELAP	8/25/2005
Carbazole	EPA 8270	Extractable Organics	NELAP	8/25/2005
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	8/25/2005
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	11/14/2003
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	8/25/2005
Carbophenothion	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Chloride	EPA 300.0	General Chemistry	NELAP	4/1/2003
Chlorobenzene	EPA 624	Volatile Organics	NELAP	11/14/2003
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Chloroethane	EPA 624	Volatile Organics	NELAP	8/25/2005
Chloroethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
Chloroform	EPA 624	Volatile Organics	NELAP	11/14/2003
Chloroform	EPA 8260	Volatile Organics	NELAP	7/7/2010
Chromium	EPA 200.7	Metals	NELAP	7/7/2010
Chromium	EPA 200.8	Metals	NELAP	4/1/2003
Chromium	EPA 6010	Metals	NELAP	7/7/2010
Chromium	EPA 6020	Metals	NELAP	8/25/2005
Chrysene	EPA 625	Extractable Organics	NELAP	4/1/2003
Chrysene	EPA 8270	Volatile Organics	NELAP	8/25/2005
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	8/25/2005
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	11/14/2003
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Cobalt	EPA 200.7	Metals	NELAP	7/7/2010
Cobalt	EPA 200.8	Metals	NELAP	4/1/2003
Cobalt	EPA 6010	Metals	NELAP	7/7/2010
Cobalt	EPA 6020	Metals	NELAP	8/25/2005

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Moscow, ID 83843

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Conductivity	SM 2510 B	General Chemistry	NELAP	1/6/2009
Copper	EPA 200.7	Metals	NELAP	7/7/2010
Copper	EPA 200.8	Metals	NELAP	4/1/2003
Copper	EPA 6010	Metals	NELAP	7/7/2010
Copper	EPA 6020	Metals	NELAP	8/25/2005
Coumaphos	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	11/26/2007
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Demeton-s	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	4/1/2003
Dibenz(a,h)anthracene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	8/25/2005
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	11/14/2003
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
Dibromomethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	11/26/2007
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	11/26/2007
Dichlorovos (DDVP, Dichlorvos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Diethyl ether	EPA 8260	Volatile Organics	NELAP	8/25/2005
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	8/25/2005
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2003
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	8/25/2005
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2003
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	8/25/2005
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2003
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	8/25/2005
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	11/26/2007
Dioxathion	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
EPN	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Ethylbenzene	EPA 624	Volatile Organics	NELAP	11/14/2003
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Fensulfothion	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Fenthion	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2003
Fluoranthene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Fluorene	EPA 625	Extractable Organics	NELAP	4/1/2003
Fluorene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Fluoride	EPA 300.0	General Chemistry	NELAP	4/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Hardness	SM 2340 B	General Chemistry,Metals	NELAP	8/25/2005
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2003
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	8/25/2005
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	4/1/2003
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	8/25/2005
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	4/1/2003
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	8/25/2005

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Moscow, ID 83843

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachloroethane	EPA 625	Extractable Organics	NELAP	4/1/2003
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	8/25/2005
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	4/1/2003
Indeno(1,2,3-cd)pyrene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	8/25/2005
Iron	EPA 200.7	Metals	NELAP	7/7/2010
Iron	EPA 6010	Metals	NELAP	7/7/2010
Iron	EPA 6020	Metals	NELAP	8/25/2005
Isophorone	EPA 625	Extractable Organics	NELAP	4/1/2003
Isophorone	EPA 8270	Extractable Organics	NELAP	8/25/2005
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Kjeldahl nitrogen - total	SM 4500-NorgC	General Chemistry	NELAP	11/26/2007
Lead	EPA 200.7	Metals	NELAP	7/7/2010
Lead	EPA 200.8	Metals	NELAP	4/1/2003
Lead	EPA 6010	Metals	NELAP	7/7/2010
Lead	EPA 6020	Metals	NELAP	8/25/2005
Lithium	EPA 200.7	Metals	NELAP	7/7/2010
Lithium	EPA 6010	Metals	NELAP	7/7/2010
m/p-Xylenes	EPA 8260	Volatile Organics	NELAP	7/7/2010
Magnesium	EPA 200.7	Metals	NELAP	7/7/2010
Magnesium	EPA 6010	Metals	NELAP	7/7/2010
Magnesium	EPA 6020	Metals	NELAP	8/25/2005
Malathion	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Manganese	EPA 200.7	Metals	NELAP	7/7/2010
Manganese	EPA 200.8	Metals	NELAP	4/1/2003
Manganese	EPA 6010	Metals	NELAP	7/7/2010
Manganese	EPA 6020	Metals	NELAP	8/25/2005
Mercury	EPA 1631	Metals	NELAP	4/7/2003
Mercury	EPA 200.8	Metals	NELAP	4/1/2003
Mercury	EPA 245.7	Metals	NELAP	11/26/2007
Mercury	EPA 6020	Metals	NELAP	8/25/2005
Methoxychlor	EPA 608.2	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	11/14/2003
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	8/25/2005
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	11/14/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/7/2010
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	8/25/2005
Methylene chloride	EPA 624	Volatile Organics	NELAP	7/7/2010
Methylene chloride	EPA 8260	Volatile Organics	NELAP	8/25/2005
Mevinphos	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Molybdenum	EPA 200.7	Metals	NELAP	7/7/2010
Molybdenum	EPA 200.8	Metals	NELAP	4/1/2003
Molybdenum	EPA 6010	Metals	NELAP	7/7/2010
Molybdenum	EPA 6020	Metals	NELAP	8/25/2005
Monocrotophos	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Naled	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Naphthalene	EPA 625	Extractable Organics	NELAP	4/1/2003
Naphthalene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Naphthalene	EPA 8270	Volatile Organics	NELAP	8/25/2005
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Nickel	EPA 200.7	Metals	NELAP	7/7/2010
Nickel	EPA 200.8	Metals	NELAP	4/1/2003
Nickel	EPA 6010	Metals	NELAP	7/7/2010
Nickel	EPA 6020	Metals	NELAP	8/25/2005
Nitrate as N	EPA 300.0	General Chemistry	NELAP	4/1/2003
Nitrate as N	SM 4500-NO3 F	General Chemistry	NELAP	4/1/2003
Nitrate-nitrite	SM 4500-NO3 F	General Chemistry	NELAP	7/7/2010
Nitrite	SM 4500-NO3 F	General Chemistry	NELAP	4/1/2003
Nitrite as N	EPA 300.0	General Chemistry	NELAP	4/1/2003
Nitrobenzene	EPA 625	Extractable Organics	NELAP	4/1/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	8/25/2005
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	4/1/2003
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	8/25/2005
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	4/1/2003
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	8/25/2005
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	4/1/2003
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	8/25/2005
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Oil & Grease	EPA 1664A	General Chemistry	NELAP	4/1/2003
Orthophosphate as P	EPA 300.0	General Chemistry	NELAP	10/15/2014

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Orthophosphate as P	SM 4500-P F	General Chemistry	NELAP	4/1/2003
o-Xylene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/22/2010
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2003
Pentachlorophenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
Perchlorate	EPA 6850	General Chemistry	NELAP	7/7/2010
pH	SM 4500-H+-B	General Chemistry	NELAP	11/26/2007
Phenanthrene	EPA 625	Extractable Organics	NELAP	4/1/2003
Phenanthrene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Phenol	EPA 625	Extractable Organics	NELAP	4/1/2003
Phenol	EPA 8270	Volatile Organics	NELAP	8/25/2005
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Phosmet (Imidan)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Phosphorus, total	SM 4500-P F	General Chemistry	NELAP	4/1/2003
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Potassium	EPA 200.7	Metals	NELAP	7/7/2010
Potassium	EPA 6010	Metals	NELAP	7/7/2010
Potassium	EPA 6020	Metals	NELAP	8/25/2005
Pyrene	EPA 625	Extractable Organics	NELAP	4/1/2003
Pyrene	EPA 8270	Volatile Organics	NELAP	8/25/2005
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	11/26/2007
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	11/26/2007
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/7/2010
Selenium	EPA 200.7	Metals	NELAP	7/7/2010
Selenium	EPA 200.8	Metals	NELAP	4/1/2003
Selenium	EPA 6010	Metals	NELAP	7/7/2010
Selenium	EPA 6020	Metals	NELAP	8/25/2005
Silver	EPA 200.7	Metals	NELAP	7/7/2010
Silver	EPA 200.8	Metals	NELAP	4/1/2003
Silver	EPA 6010	Metals	NELAP	7/7/2010
Silver	EPA 6020	Metals	NELAP	8/25/2005
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	11/26/2007
Sodium	EPA 200.7	Metals	NELAP	7/7/2010
Sodium	EPA 6010	Metals	NELAP	7/7/2010
Sodium	EPA 6020	Metals	NELAP	8/25/2005
Styrene	EPA 8260	Volatile Organics	NELAP	8/25/2005

Laboratory Scope of Accreditation

Attachment to Certificate #: E87893-58, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87893** EPA Lab Code: **ID00013** **(208) 883-2839**

E87893
Anatek Labs, Inc.
1282 Alturas Dr.
Moscow, ID 83843

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Sulfate	EPA 300.0	General Chemistry	NELAP	4/1/2003
Sulfotepp	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Terbufos	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	11/14/2003
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	8/25/2005
Tetrachlorvinphos (Stirophos, Gardona)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Thallium	EPA 200.7	Metals	NELAP	7/7/2010
Thallium	EPA 200.8	Metals	NELAP	4/1/2003
Thallium	EPA 6010	Metals	NELAP	7/7/2010
Thallium	EPA 6020	Metals	NELAP	8/25/2005
Tin	EPA 200.7	Metals	NELAP	7/7/2010
Tin	EPA 6010	Metals	NELAP	7/7/2010
Titanium	EPA 200.7	Metals	NELAP	11/30/2010
Titanium	EPA 6010	Metals	NELAP	11/30/2010
Toluene	EPA 624	Volatile Organics	NELAP	11/14/2003
Toluene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Total cyanide	EPA 335.4	General Chemistry	NELAP	8/25/2005
Total nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	6/9/2008
Total organic carbon	SM 5310 B	General Chemistry	NELAP	2/25/2013
Total phenolics	EPA 420.1	General Chemistry	NELAP	4/7/2003
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	8/25/2005
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	11/14/2003
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	8/25/2005
trans-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	11/14/2003
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	8/25/2005
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	8/25/2005
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	11/14/2003
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	8/25/2005
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	11/14/2003
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	8/25/2005
Turbidity	EPA 180.1	General Chemistry	NELAP	8/25/2005
Uranium	EPA 200.8	Metals	NELAP	4/1/2003
Vanadium	EPA 200.7	Metals	NELAP	7/7/2010
Vanadium	EPA 200.8	Metals	NELAP	4/1/2003

Laboratory Scope of Accreditation

Attachment to Certificate #: E87893-58, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87893** EPA Lab Code: **ID00013** **(208) 883-2839**

E87893
Anatek Labs, Inc.
1282 Alturas Dr.
Moscow, ID 83843

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Vanadium	EPA 6010	Metals	NELAP	7/7/2010
Vanadium	EPA 6020	Metals	NELAP	8/25/2005
Vinyl chloride	EPA 624	Volatile Organics	NELAP	11/14/2003
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	8/25/2005
Xylene (total)	EPA 624	Volatile Organics	NELAP	11/14/2003
Xylene (total)	EPA 8260	Volatile Organics	NELAP	8/25/2005
Zinc	EPA 200.7	Metals	NELAP	7/7/2010
Zinc	EPA 200.8	Metals	NELAP	4/1/2003
Zinc	EPA 6010	Metals	NELAP	7/7/2010
Zinc	EPA 6020	Metals	NELAP	8/25/2005

Florida HEALTH

State of Florida

Department of Health, Bureau of Public Health Laboratories

This is to certify that

E871099

ANATEK LABS, INC. - SPOKANE
504 E. SPRAGUE STE. D
SPOKANE, WA 99202

has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

DRINKING WATER - RADIOCHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - RADIOCHEMISTRY

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2015 Expiration Date: June 30, 2016

Carina Blackmore, DVM, PhD, Dipl. ACVPM, CPM
Chief, Bureau of Public Health Laboratories
DH Form 1697, 7/04
NON-TRANSFERABLE E871099-06-07/01/2015
Supersedes all previously issued certificates

Rick Scott
Governor

HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E871099-06, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871099 **EPA Lab Code:** WA00169 (509) 838-3999

E871099
Anatek Labs, Inc. - Spokane
504 E. Sprague Ste. D
Spokane, WA 99202

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Gross-alpha	EPA 900.0	Radiochemistry	NELAP	12/10/2012
Gross-beta	EPA 900.0	Radiochemistry	NELAP	1/14/2015

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2015 **Expiration Date: 6/30/2016**

Laboratory Scope of Accreditation

Attachment to Certificate #: E871099-06, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871099 **EPA Lab Code:** WA00169 (509) 838-3999

E871099
Anatek Labs, Inc. - Spokane
504 E. Sprague Ste. D
Spokane, WA 99202

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aluminum	EPA 200.8	Metals	NELAP	12/10/2012
Aluminum	EPA 6020	Metals	NELAP	12/10/2012
Antimony	EPA 200.8	Metals	NELAP	12/10/2012
Antimony	EPA 6020	Metals	NELAP	12/10/2012
Arsenic	EPA 200.8	Metals	NELAP	12/10/2012
Arsenic	EPA 6020	Metals	NELAP	12/10/2012
Barium	EPA 200.8	Metals	NELAP	12/10/2012
Barium	EPA 6020	Metals	NELAP	12/10/2012
Beryllium	EPA 200.8	Metals	NELAP	12/10/2012
Beryllium	EPA 6020	Metals	NELAP	12/10/2012
Cadmium	EPA 200.8	Metals	NELAP	12/10/2012
Cadmium	EPA 6020	Metals	NELAP	12/10/2012
Calcium	EPA 6020	Metals	NELAP	12/10/2012
Chromium	EPA 200.8	Metals	NELAP	12/10/2012
Chromium	EPA 6020	Metals	NELAP	12/10/2012
Cobalt	EPA 200.8	Metals	NELAP	12/10/2012
Cobalt	EPA 6020	Metals	NELAP	12/10/2012
Copper	EPA 200.8	Metals	NELAP	12/10/2012
Copper	EPA 6020	Metals	NELAP	12/10/2012
Lead	EPA 200.8	Metals	NELAP	12/10/2012
Lead	EPA 6020	Metals	NELAP	12/10/2012
Magnesium	EPA 6020	Metals	NELAP	12/10/2012
Manganese	EPA 200.8	Metals	NELAP	12/10/2012
Manganese	EPA 6020	Metals	NELAP	12/10/2012
Mercury	EPA 200.8	Metals	NELAP	12/10/2012
Mercury	EPA 6020	Metals	NELAP	12/10/2012
Molybdenum	EPA 200.8	Metals	NELAP	12/10/2012
Molybdenum	EPA 6020	Metals	NELAP	12/10/2012
Nickel	EPA 200.8	Metals	NELAP	12/10/2012
Nickel	EPA 6020	Metals	NELAP	12/10/2012
Potassium	EPA 6020	Metals	NELAP	12/10/2012
Selenium	EPA 200.8	Metals	NELAP	12/10/2012
Selenium	EPA 6020	Metals	NELAP	12/10/2012
Silver	EPA 200.8	Metals	NELAP	12/10/2012
Silver	EPA 6020	Metals	NELAP	12/10/2012
Sodium	EPA 6020	Metals	NELAP	12/10/2012

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2015 **Expiration Date: 6/30/2016**

Laboratory Scope of Accreditation

Attachment to Certificate #: E871099-06, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871099 **EPA Lab Code:** WA00169 (509) 838-3999

E871099
Anatek Labs, Inc. - Spokane
504 E. Sprague Ste. D
Spokane, WA 99202

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Thallium	EPA 200.8	Metals	NELAP	12/10/2012
Thallium	EPA 6020	Metals	NELAP	12/10/2012
Total alpha	EPA 900.0	Radiochemistry	NELAP	12/10/2012
Total beta	EPA 900.0	Radiochemistry	NELAP	12/10/2012
Uranium	EPA 200.8	Metals	NELAP	12/10/2012
Uranium	EPA 6020	Metals	NELAP	12/10/2012
Vanadium	EPA 200.8	Metals	NELAP	12/10/2012
Vanadium	EPA 6020	Metals	NELAP	12/10/2012
Zinc	EPA 200.8	Metals	NELAP	12/10/2012
Zinc	EPA 6020	Metals	NELAP	12/10/2012

State of Florida
Department of Health, Bureau of Public Health Laboratories
This is to certify that

E87641

ENERGY LABORATORIES INC.
2393 SALT CREEK HWY
CASPER, WY 82602

has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

DRINKING WATER - GROUP I UNREGULATED CONTAMINANTS, DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, DRINKING WATER - RADIOCHEMISTRY, DRINKING WATER - SYNTHETIC ORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 02, 2015 Expiration Date: June 30, 2016

Carina Blackmore, DVM, PhD, Dipl. ACVPM, CPM
Chief, Bureau of Public Health Laboratories
DH Form 1697, 7/04
NON-TRANSFERABLE E87641-35-07/02/2015
Supersedes all previously issued certificates

Laboratory Scope of Accreditation

Attachment to Certificate #: E87641-35, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87641** EPA Lab Code: **WY00002** **(307) 235-0515**

E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/22/2009
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
1,2,3-Trichloropropane	EPA 504.1	Group II Unregulated Contaminants	NELAP	7/7/2009
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
1,2,4-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	7/22/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	7/22/2002
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
3-Hydroxycarbofuran	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/22/2002
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
Aldicarb (Temik)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/22/2002
Aldicarb sulfone	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/22/2002
Aldicarb sulfoxide	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/22/2002
Alkalinity as CaCO3	SM 2320 B	Primary Inorganic Contaminants	NELAP	7/22/2002
Aluminum	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Aluminum	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/7/2009
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Arsenic	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Arsenic	SM 3114 B	Primary Inorganic Contaminants	NELAP	7/22/2002

Laboratory Scope of Accreditation

Attachment to Certificate #: E87641-35, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87641** EPA Lab Code: **WY00002** **(307) 235-0515**

E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Boron	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Bromide	EPA 300.0	Primary Inorganic Contaminants	NELAP	6/24/2015
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
Bromodichloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Bromoform	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Calcium	ELI-C-SOP 50-018-07/ICP-MS	Secondary Inorganic Contaminants	NELAP	1/12/2012
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/22/2002
Carbaryl (Sevin)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/22/2002
Carbofuran (Furadan)	EPA 531.1	Synthetic Organic Contaminants	NELAP	7/22/2002
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	7/22/2002
Chloride	SM 4500 Cl- B	Secondary Inorganic Contaminants	NELAP	7/22/2002
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	4/6/2012
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Chloroform	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	1/12/2012
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	1/12/2012
Conductivity	SM 2510 B	Primary Inorganic Contaminants	NELAP	1/12/2012
Copper	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Copper	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Corrosivity (langlier index)	SM 2330 B	Secondary Inorganic Contaminants	NELAP	1/12/2012
Dibromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002

Laboratory Scope of Accreditation

Attachment to Certificate #: E87641-35, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87641** EPA Lab Code: **WY00002** **(307) 235-0515**

E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diquat	EPA 549.2	Synthetic Organic Contaminants	NELAP	7/22/2002
Dissolved organic carbon (DOC)	SM 5310 C	Primary Inorganic Contaminants	NELAP	1/12/2012
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
Fluoride	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	7/22/2002
Fluoride	SM 4500 F-C	Secondary Inorganic Contaminants	NELAP	7/22/2002
Glyphosate	EPA 547	Synthetic Organic Contaminants	NELAP	7/22/2002
Hardness	SM 2340 B	Secondary Inorganic Contaminants	NELAP	1/12/2012
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
Iron	ELI-C-SOP 50-018-07/ICP-MS	Secondary Inorganic Contaminants	NELAP	1/12/2012
Iron	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Magnesium	ELI-C-SOP 50-018-07/ICP-MS	Secondary Inorganic Contaminants	NELAP	1/12/2012
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/22/2002
Manganese	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Manganese	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/7/2009
Mercury	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	7/7/2009
Mercury	SM 3112 B	Primary Inorganic Contaminants	NELAP	7/22/2002
Methiocarb (Mesurol)	EPA 531.1	Group I Unregulated Contaminants	NELAP	1/12/2012
Methomyl (Lannate)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/22/2002
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Molybdenum	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/7/2009
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Nitrate as N	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/22/2002
Nitrate as N	EPA 353.2	Primary Inorganic Contaminants	NELAP	7/22/2002
Nitrite as N	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/22/2002
Nitrite as N	SM 4500-NO2-B	Primary Inorganic Contaminants	NELAP	7/22/2002
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009

Laboratory Scope of Accreditation

Attachment to Certificate #: E87641-35, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87641** EPA Lab Code: **WY00002** **(307) 235-0515**

E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Orthophosphate as P	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/10/2009
Oxamyl	EPA 531.1	Synthetic Organic Contaminants	NELAP	7/22/2002
pH	SM 4500-H B	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	7/22/2002
Potassium	ELI-C-SOP 50-018-07/ICP-MS	Secondary Inorganic Contaminants	NELAP	1/12/2012
Potassium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Propoxur (Baygon)	EPA 531.1	Group I Unregulated Contaminants	NELAP	1/12/2012
Residue-filterable (TDS)	SM 2540 C	Secondary Inorganic Contaminants	NELAP	7/22/2002
sec-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/7/2009
Selenium	SM 3114 B	Primary Inorganic Contaminants	NELAP	1/12/2012
Silica as SiO2	EPA 200.7	Primary Inorganic Contaminants	NELAP	5/12/2009
Silver	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Silver	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/7/2009
Sodium	ELI-C-SOP 50-018-07/ICP-MS	Secondary Inorganic Contaminants	NELAP	1/12/2012
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/22/2002
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
Sulfate	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	7/22/2002
Sulfate	SM 4500 SO4-E	Secondary Inorganic Contaminants	NELAP	7/22/2002
Surfactants - MBAS	SM 5540 C	Secondary Inorganic Contaminants	NELAP	1/12/2012
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/7/2009
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/22/2002
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
Total nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/22/2002
Total nitrate-nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	7/22/2002
Total organic carbon	SM 5310 C	Primary Inorganic Contaminants	NELAP	7/22/2002
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
trans-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/22/2002
Turbidity	SM 2130 B	Secondary Inorganic Contaminants	NELAP	1/12/2012
Uranium	EPA 200.8	Radiochemistry	NELAP	2/17/2006

Attachment to Certificate #: E87641-35, expiration date June 30, 2016. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87641** EPA Lab Code: **WY00002** **(307) 235-0515**

E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Vanadium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Vanadium	EPA 200.8	Primary Inorganic Contaminants	NELAP	7/7/2009
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	4/6/2012
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	7/22/2002
Zinc	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/7/2009
Zinc	EPA 200.8	Primary Inorganic Contaminants	NELAP	6/24/2015

Laboratory Scope of Accreditation

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State Laboratory ID: **E87641** EPA Lab Code: **WY00002** **(307) 235-0515**

E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	3/19/2010
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	1/24/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	1/24/2001
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003

Laboratory Scope of Accreditation

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	6/24/2015
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	1/24/2001
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	1/24/2001
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	1/24/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Aluminum	EPA 200.7	Metals	NELAP	1/24/2001
Aluminum	EPA 200.8	Metals	NELAP	1/24/2001
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Ammonia as N	SM 4500-NH3 G	General Chemistry	NELAP	1/24/2001
Antimony	EPA 200.7	Metals	NELAP	7/7/2009
Antimony	EPA 200.8	Metals	NELAP	1/24/2001
Antimony	EPA 6010	Metals	NELAP	7/7/2009
Antimony	EPA 6020	Metals	NELAP	7/1/2003
Arsenic	EPA 200.7	Metals	NELAP	9/2/2002
Arsenic	EPA 200.8	Metals	NELAP	1/24/2001
Arsenic	EPA 6010	Metals	NELAP	7/7/2009
Arsenic	EPA 6020	Metals	NELAP	7/1/2003
Arsenic	SM 3114 B	Metals	NELAP	6/24/2015
Barium	EPA 200.7	Metals	NELAP	1/24/2001
Barium	EPA 200.8	Metals	NELAP	1/24/2001
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Benzene	EPA 624	Volatile Organics	NELAP	1/24/2001
Benzene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	7/22/2002
Beryllium	EPA 200.8	Metals	NELAP	1/24/2001
Beryllium	EPA 6010	Metals	NELAP	7/7/2009
Beryllium	EPA 6020	Metals	NELAP	7/1/2003

Laboratory Scope of Accreditation

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E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	1/24/2001
Boron	EPA 200.7	Metals	NELAP	7/22/2002
Boron	EPA 200.8	Metals	NELAP	1/12/2012
Boron	EPA 6010	Metals	NELAP	7/1/2003
Boron	EPA 6020	Metals	NELAP	1/12/2012
Bromide	EPA 300.0	General Chemistry	NELAP	1/24/2001
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	1/24/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	EPA 624	Volatile Organics	NELAP	1/24/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Cadmium	EPA 200.7	Metals	NELAP	7/22/2002
Cadmium	EPA 200.8	Metals	NELAP	1/24/2001
Cadmium	EPA 6010	Metals	NELAP	7/7/2009
Cadmium	EPA 6020	Metals	NELAP	7/1/2003
Calcium	ELI-C-SOP 50-018-07/ICP-MS	Metals	NELAP	1/12/2012
Calcium	EPA 200.7	Metals	NELAP	1/24/2001
Calcium	EPA 6010	Metals	NELAP	7/1/2003
Calcium	EPA 6020	Metals	NELAP	1/12/2012
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	1/24/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	1/24/2001
Chemical oxygen demand	HACH 8000	General Chemistry	NELAP	1/24/2001
Chloride	EPA 300.0	General Chemistry	NELAP	1/24/2001
Chloride	SM 4500 Cl- B	General Chemistry	NELAP	1/24/2001
Chlorobenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroethane	EPA 624	Volatile Organics	NELAP	1/24/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroform	EPA 624	Volatile Organics	NELAP	1/24/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chromium	EPA 200.7	Metals	NELAP	7/22/2002
Chromium	EPA 200.8	Metals	NELAP	1/24/2001

Laboratory Scope of Accreditation

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E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium	EPA 6010	Metals	NELAP	7/7/2009
Chromium	EPA 6020	Metals	NELAP	7/1/2003
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	1/24/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Cobalt	EPA 200.7	Metals	NELAP	7/22/2002
Cobalt	EPA 200.8	Metals	NELAP	1/24/2001
Cobalt	EPA 6010	Metals	NELAP	7/7/2009
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Conductivity	SM 2510 B	General Chemistry	NELAP	1/24/2001
Copper	EPA 200.7	Metals	NELAP	7/22/2002
Copper	EPA 200.8	Metals	NELAP	1/24/2001
Copper	EPA 6010	Metals	NELAP	7/7/2009
Copper	EPA 6020	Metals	NELAP	7/1/2003
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	1/24/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	1/24/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Fluoride	EPA 300.0	General Chemistry	NELAP	1/24/2001
Fluoride	SM 4500 F-C	General Chemistry	NELAP	1/24/2001
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Hardness	SM 2340 B	Metals	NELAP	7/17/2002
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ignitability	EPA 1010	General Chemistry	NELAP	7/1/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	EPA 200.7	Metals	NELAP	1/24/2001
Iron	EPA 200.8	Metals	NELAP	11/22/2010
Iron	EPA 6010	Metals	NELAP	7/1/2003
Iron	EPA 6020	Metals	NELAP	1/12/2012

Laboratory Scope of Accreditation

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E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Lead	EPA 200.7	Metals	NELAP	7/22/2002
Lead	EPA 200.8	Metals	NELAP	1/24/2001
Lead	EPA 6010	Metals	NELAP	7/7/2009
Lead	EPA 6020	Metals	NELAP	7/1/2003
Magnesium	EPA 200.7	Metals	NELAP	1/24/2001
Magnesium	EPA 200.8	Metals	NELAP	11/22/2010
Magnesium	EPA 6010	Metals	NELAP	7/1/2003
Magnesium	EPA 6020	Metals	NELAP	1/12/2012
Manganese	EPA 200.7	Metals	NELAP	1/24/2001
Manganese	EPA 200.8	Metals	NELAP	1/24/2001
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/7/2009
Mercury	EPA 200.8	Metals	NELAP	1/24/2001
Mercury	EPA 245.1	Metals	NELAP	7/7/2009
Mercury	EPA 245.7	Metals	NELAP	1/12/2012
Mercury	EPA 6020	Metals	NELAP	1/12/2012
Mercury	EPA 7470	Metals	NELAP	7/1/2003
Mercury	SM 3112 B	Metals	NELAP	1/24/2001
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	1/24/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	1/24/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylene chloride	EPA 624	Volatile Organics	NELAP	1/24/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Molybdenum	EPA 200.7	Metals	NELAP	7/22/2002
Molybdenum	EPA 200.8	Metals	NELAP	1/24/2001
Molybdenum	EPA 6010	Metals	NELAP	7/1/2003
Molybdenum	EPA 6020	Metals	NELAP	7/7/2009
Naphthalene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.7	Metals	NELAP	7/22/2002

Laboratory Scope of Accreditation

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E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nickel	EPA 200.8	Metals	NELAP	1/24/2001
Nickel	EPA 6010	Metals	NELAP	7/7/2009
Nickel	EPA 6020	Metals	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	1/24/2001
Nitrate as N	EPA 353.2	General Chemistry	NELAP	1/24/2001
Nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	1/24/2001
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	1/24/2001
Nitrite as N	EPA 300.0	General Chemistry	NELAP	1/24/2001
Nitrite as N	SM 4500-NO2-B	General Chemistry	NELAP	1/24/2001
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Oil & Grease	EPA 1664A	General Chemistry	NELAP	1/24/2001
Orthophosphate as P	EPA 300.0	General Chemistry	NELAP	1/24/2001
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
pH	SM 4500-H+-B	General Chemistry	NELAP	1/24/2001
Phosphorus	EPA 6020	Metals	NELAP	1/12/2012
Phosphorus, total	EPA 200.7	Metals	NELAP	7/22/2002
Phosphorus, total	EPA 6010	Metals	NELAP	7/1/2003
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	ELI-C-SOP 50-018-07/ICP-MS	Metals	NELAP	1/12/2012
Potassium	EPA 200.7	Metals	NELAP	1/24/2001
Potassium	EPA 6010	Metals	NELAP	7/1/2003
Potassium	EPA 6020	Metals	NELAP	1/12/2012
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	1/24/2001
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	7/7/2009
Residue-total	SM 2540 B	General Chemistry	NELAP	1/12/2012
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.7	Metals	NELAP	7/22/2002
Selenium	EPA 200.8	Metals	NELAP	1/24/2001
Selenium	EPA 6010	Metals	NELAP	7/1/2003
Selenium	EPA 6020	Metals	NELAP	7/7/2009
Selenium	EPA 7742	Metals	NELAP	1/12/2012
Selenium	SM 3114 B	Metals	NELAP	1/24/2001
Silicon	EPA 200.7	Metals	NELAP	1/24/2001
Silicon	EPA 6010	Metals	NELAP	7/1/2003
Silicon	EPA 6020	Metals	NELAP	1/12/2012

Laboratory Scope of Accreditation

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E87641
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Casper, WY 82602

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Silver	EPA 200.7	Metals	NELAP	7/7/2009
Silver	EPA 6010	Metals	NELAP	7/7/2009
Sodium	ELI-C-SOP 50-018-07/ICP-MS	Metals	NELAP	1/12/2012
Sodium	EPA 200.7	Metals	NELAP	1/24/2001
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Sodium	EPA 6020	Metals	NELAP	1/12/2012
Strontium	EPA 200.7	Metals	NELAP	7/7/2009
Strontium	EPA 200.8	Metals	NELAP	1/12/2012
Strontium	EPA 6010	Metals	NELAP	7/1/2003
Strontium	EPA 6020	Metals	NELAP	7/7/2009
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Sulfate	EPA 300.0	General Chemistry	NELAP	1/24/2001
Sulfate	SM 4500 SO4-E	General Chemistry	NELAP	1/24/2001
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	1/24/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrahydrofuran (THF)	ELI-C-SOP 50-073-05/GC-MS	Volatile Organics	NELAP	1/12/2012
Thallium	EPA 200.8	Metals	NELAP	1/24/2001
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thorium	EPA 200.8	Metals	NELAP	7/7/2009
Thorium	EPA 6020	Metals	NELAP	7/7/2009
Tin	EPA 200.8	Metals	NELAP	1/12/2012
Tin	EPA 6020	Metals	NELAP	7/7/2009
Titanium	EPA 200.7	Metals	NELAP	7/7/2009
Titanium	EPA 200.8	Metals	NELAP	1/12/2012
Titanium	EPA 6010	Metals	NELAP	7/7/2009
Titanium	EPA 6020	Metals	NELAP	7/7/2009
Toluene	EPA 624	Volatile Organics	NELAP	1/24/2001
Toluene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Total hardness as CaCO3	EPA 200.7	Metals	NELAP	1/24/2001
Total organic carbon	SM 5310 C	General Chemistry	NELAP	1/24/2001
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	EPA 1664A	General Chemistry	NELAP	5/10/2010
Total phenolics	EPA 420.1	General Chemistry	NELAP	1/12/2012

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State Laboratory ID: **E87641** EPA Lab Code: **WY00002** **(307) 235-0515**

E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	1/24/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	1/24/2001
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	1/24/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	1/24/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Uranium	EPA 200.8	Metals	NELAP	1/24/2001
Uranium	EPA 6020	Metals	NELAP	7/7/2009
Vanadium	EPA 200.7	Metals	NELAP	7/22/2002
Vanadium	EPA 200.8	Metals	NELAP	1/24/2001
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Vanadium	EPA 6020	Metals	NELAP	7/7/2009
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	1/24/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Xylene (total)	EPA 624	Volatile Organics	NELAP	7/7/2009
Xylene (total)	EPA 8021	Volatile Organics	NELAP	7/1/2003
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Zinc	EPA 200.7	Metals	NELAP	7/22/2002
Zinc	EPA 200.8	Metals	NELAP	1/24/2001
Zinc	EPA 6010	Metals	NELAP	7/7/2009
Zinc	EPA 6020	Metals	NELAP	7/1/2003

Laboratory Scope of Accreditation

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State Laboratory ID: **E87641** EPA Lab Code: **WY00002** **(307) 235-0515**

E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	3/16/2010
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	1/24/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	6/24/2014
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	1/24/2001
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	6/24/2015
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	1/24/2001
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	1/24/2001
2-Hexanone	EPA 8260	Volatile Organics	NELAP	1/24/2001
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	1/24/2001
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Acetone	EPA 8260	Volatile Organics	NELAP	1/24/2001
Acetonitrile	EPA 8260	Volatile Organics	NELAP	1/24/2001
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	1/24/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Aluminum	EPA 6010	Metals	NELAP	1/24/2001
Aluminum	EPA 6020	Metals	NELAP	1/24/2001
Antimony	EPA 6010	Metals	NELAP	1/12/2012

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E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Antimony	EPA 6020	Metals	NELAP	1/24/2001
Arsenic	EPA 6010	Metals	NELAP	7/7/2009
Arsenic	EPA 6020	Metals	NELAP	1/24/2001
Barium	EPA 6010	Metals	NELAP	1/24/2001
Barium	EPA 6020	Metals	NELAP	1/24/2001
Benzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Beryllium	EPA 6010	Metals	NELAP	7/7/2009
Beryllium	EPA 6020	Metals	NELAP	1/24/2001
Boron	EPA 6010	Metals	NELAP	1/24/2001
Boron	EPA 6020	Metals	NELAP	1/12/2012
Bromobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	1/24/2001
Cadmium	EPA 6010	Metals	NELAP	7/7/2009
Cadmium	EPA 6020	Metals	NELAP	1/24/2001
Calcium	EPA 6010	Metals	NELAP	1/24/2001
Calcium	EPA 6020	Metals	NELAP	1/12/2012
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	1/24/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	1/24/2001
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	1/24/2001
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/22/2002
Chromium	EPA 6010	Metals	NELAP	7/7/2009
Chromium	EPA 6020	Metals	NELAP	1/24/2001
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	1/24/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Cobalt	EPA 6010	Metals	NELAP	7/7/2009
Cobalt	EPA 6020	Metals	NELAP	1/24/2001
Copper	EPA 6010	Metals	NELAP	7/7/2009
Copper	EPA 6020	Metals	NELAP	1/24/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Dibromomethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	1/24/2001

Laboratory Scope of Accreditation

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E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diethyl ether	EPA 8260	Volatile Organics	NELAP	1/24/2001
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	6/24/2014
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	1/24/2001
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Extractable organic halides (EOX)	EPA 9023	General Chemistry	NELAP	1/24/2001
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	1/24/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Ignitability	EPA 1010	General Chemistry	NELAP	1/24/2001
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/22/2002
Iron	EPA 6010	Metals	NELAP	1/24/2001
Iron	EPA 6020	Metals	NELAP	1/12/2012
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Lead	EPA 6010	Metals	NELAP	7/7/2009
Lead	EPA 6020	Metals	NELAP	1/24/2001
Magnesium	EPA 6010	Metals	NELAP	1/24/2001
Magnesium	EPA 6020	Metals	NELAP	1/12/2012
Manganese	EPA 6010	Metals	NELAP	1/24/2001
Manganese	EPA 6020	Metals	NELAP	7/7/2009
Mercury	EPA 7471	Metals	NELAP	1/24/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	1/24/2001
Molybdenum	EPA 6010	Metals	NELAP	1/24/2001
Molybdenum	EPA 6020	Metals	NELAP	7/7/2009
Naphthalene	EPA 8260	Volatile Organics	NELAP	1/24/2001
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Nickel	EPA 6010	Metals	NELAP	7/7/2009
Nickel	EPA 6020	Metals	NELAP	1/24/2001
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	1/24/2001
p-Dioxane	EPA 8260	Volatile Organics	NELAP	1/24/2001
pH	EPA 9045	General Chemistry	NELAP	1/24/2001
Phosphorus, total	EPA 6010	Metals	NELAP	5/12/2009
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	1/24/2001

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E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Potassium	EPA 6010	Metals	NELAP	1/24/2001
Potassium	EPA 6020	Metals	NELAP	1/12/2012
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	1/24/2001
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Selenium	EPA 6010	Metals	NELAP	1/24/2001
Selenium	EPA 6020	Metals	NELAP	7/7/2009
Selenium	EPA 7742	Metals	NELAP	1/12/2012
Silicon	EPA 6010	Metals	NELAP	1/24/2001
Silver	EPA 6010	Metals	NELAP	7/7/2009
Silver	EPA 6020	Metals	NELAP	7/7/2009
Sodium	EPA 6010	Metals	NELAP	1/24/2001
Sodium	EPA 6020	Metals	NELAP	1/12/2012
Strontium	EPA 6010	Metals	NELAP	1/24/2001
Strontium	EPA 6020	Metals	NELAP	7/7/2009
Styrene	EPA 8260	Volatile Organics	NELAP	1/24/2001
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Thallium	EPA 6010	Metals	NELAP	7/7/2009
Thallium	EPA 6020	Metals	NELAP	1/24/2001
Tin	EPA 6010	Metals	NELAP	7/7/2009
Tin	EPA 6020	Metals	NELAP	7/7/2009
Titanium	EPA 6010	Metals	NELAP	7/7/2009
Titanium	EPA 6020	Metals	NELAP	7/7/2009
Toluene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	1/24/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	1/24/2001
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	1/24/2001
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	1/24/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	1/24/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	1/24/2001
Uranium	EPA 6020	Metals	NELAP	7/7/2009
Vanadium	EPA 6010	Metals	NELAP	1/24/2001
Vanadium	EPA 6020	Metals	NELAP	7/7/2009
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	1/24/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	1/24/2001
Xylene (total)	EPA 8260	Volatile Organics	NELAP	1/24/2001

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E87641
Energy Laboratories Inc.
2393 Salt Creek Hwy
Casper, WY 82602

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Zinc	EPA 6010	Metals	NELAP	7/7/2009
Zinc	EPA 6020	Metals	NELAP	1/24/2001

Attachment S-3

Determination of Bedrock During Remedial Excavation

Midnite Mine Superfund Site
100 Percent Design

Attachment S-3 – Determination of Bedrock during Remedial Excavation

Note: This attachment provides a memo from MWH Americas Inc. concerning how bedrock will be determined if encountered during remedial excavations. This memo was previously provided to EPA and discussed in a technical meeting on July 23, 2013, and is included as a relevant attachment to the (100 percent) Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments (Appendix S). This attachment has not changed from the 60 percent design submittal. Appendix S and its supporting attachments are intended to satisfy the requirement for a Preliminary Confirmation Sampling and Remedial Action Effectiveness Monitoring Plan as stipulated by the Consent Decree.

June 2015

Prepared for:

Dawn Mining Company
PO Box 250
Ford, Washington 990413

and

Newmont USA Limited
6363 South Fiddler's Green Circle
Greenwood Village, Colorado 80111
Prepared By:

Prepared by:

MWH Americas, Inc.
3665 JFK Parkway, Suite 206
Fort Collins, Colorado 80525

MWH

BUILDING A BETTER WORLD

TECHNICAL MEMORANDUM

TO: *Lou Miller (WME)* **DATE:** *July 12, 2013*

FROM: *Tom Kelley (MWH)* **REFERENCE:** *1011322.*

SUBJECT: *Defining Rock Excavation during RA Construction at the Midnite Mine Superfund Site*

This memorandum summarizes our review of potential methods for defining bedrock for the purposes of classification of excavation during soil cleanup at the Midnite Mine Superfund Site (Site). Remedial actions at the Site will include excavation of waste rock and contaminated soil, and placement of these materials in waste containment areas within Pit 3 and Pit 4 at the Site. The *Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments* included as Appendix S of the Preliminary (30 Percent) Basis of Design Report (30% BODR) (MWH, 2012) indicates that excavations of mine-impacted soils will continue until either soil cleanup criteria is achieved or until "bedrock" is encountered.

Previous construction and geotechnical investigations at the site indicate that in the eastern portion of the site, soils are underlain by the metamorphic Togo formation bedrock, and that the bedrock contact should generally be easily identifiable during excavation. However in the western portion of the site, the stratigraphic profile consists of topsoil and colluvium/alluvium of variable thickness overlying quartz-monzonite bedrock that is variably altered and has weathered to a soil-like material (residuum) in some places, with the degree of weathering gradually decreasing with depth. It has been proposed that material not meeting soil cleanup criteria that has been weathered to residuum and can reasonably be excavated without resorting to ripping or blasting will be removed and placed in the waste containment areas.

Concern has been expressed by EPA that, given the gradual transition from residual soil-like bedrock to harder bedrock in some areas of the Site, that there could be discrepancies in the interpretation as to what constitutes "bedrock" during soil cleanup activities. At the request of the EPA, a review has been made of potential methods for defining or specifying when unexcavatable soil, or "bedrock excavation," has been reached. This memorandum summarizes the results of this review and includes a proposed procedure for defining the depth when "bedrock excavation" has been reached at the Site and soil-cleanup excavation will cease.

Quantitative Test Methods

EPA has specifically asked whether any quantitative test methods can be used to determine the point where "bedrock excavation" requiring ripping and/or blasting has been reached. A review of available construction and geotechnical-engineering related literature indicates that there is no standard test method for defining the point bedrock excavation begins. Although numerous tests have been proposed over the years to define "bedrock" for excavation purposes from the "Plow Test" (Gillette, 1916) to the "Drop Bar Test" (Charlie, et al, 1993) none have been found to be entirely satisfactory and have not gained acceptance in construction practice. As a result, the current standard of practice is to define bedrock excavation by observation of the excavation in progress and obtaining concurrence of stakeholders involved in the construction (typically an Engineer or other Owner's representative, and a

Contractor's representative) in the field. Example specifications for earthworks where rock excavation may be encountered are included in the following section.

Typical Rock Excavation Specifications

Typical earthwork specifications where bedrock excavation may be encountered often distinguish bedrock excavation based upon field observations by the Contractor and Owner's CQA representatives. For example, the USACE Unified Facilities Guide Specifications divides excavations into common earth excavations and bedrock excavation and includes the following procedures for identifying rock excavation (USACE, 2008):

> "Submit notification of encountering rock in the project. Include rock excavation with blasting, excavating, grading, disposing of material classified as rock, and the satisfactory removal and disposal of boulders 1/2 cubic meter yard or more in volume; solid rock; rock material that is in ledges, bedded deposits, and unstratified masses, which cannot be removed without systematic drilling and blasting; firmly cemented conglomerate deposits possessing the characteristics of solid rock impossible to remove without systematic drilling and blasting; and hard materials (see Definitions). Include the removal of any concrete or masonry structures, except pavements, exceeding 1/2 cubic meter yard in volume that may be encountered in the work in this classification.
>
> If at any time during excavation, including excavation from borrow areas, the Contractor encounters material that may be classified as rock excavation, uncover such material and notify the Contracting Officer."

Likewise, the NRCS Standard Specifications for Construction Contracts (NRCS, 2009) defines rock excavation as:

> "…the excavation of all hard, compacted, or cemented materials that require blasting or the use of ripping and excavating equipment larger than defined for common excavation. The excavation and removal of isolated boulders or rock fragments larger than 1 cubic yard encountered in materials otherwise conforming to the definition of common excavation shall be classified as rock excavation. The presence of isolated boulders or rock fragments larger than 1 cubic yard is not in itself sufficient cause to change the classification of the surrounding material."

and places authority for determining the point at which rock is reached with the filed engineer at the time of excavation.

Recommendations for Defining Rock Excavation During Soil Cleanup

It is recommended that the current standard of construction practice, where the occurrence of bedrock is determined by direct observation of the excavation in progress be used during soil cleanup at the Site. It is proposed that it will be the responsibility of the RA Construction Contractor to notify the supervising Engineer and obtain concurrence that bedrock has been reached prior to terminating soil cleanup excavations.

It is also proposed that in the event a disagreement occurs between the stakeholders (Contractor, Owner, Engineer, Agencies, etc.) over whether bedrock has been reached at a specific location, that observation of field performance of the specified excavation equipment be used to determine the occurrence of bedrock. Specifically, it is recommended that:

"In the event of a dispute over whether bedrock has been reached, direct observation of excavator "bucket refusal" will be taken as an indication that bedrock has been reached, and the excavation will be terminated. The point of "bucket refusal" will be defined as the depth where the teeth (Caterpillar K-Series Extra Duty Teeth) on a 30-inch-wide bucket mounted on a track-mounted Caterpillar 320 hydraulic excavator cannot fully penetrate into the formation when the leading end (closest to the bucket) of the tracks off have been lifted off the ground. "Bucket refusal" on boulders will not be taken as an indication that bedrock has been reached."

References

Charlie, W.A., Ross, C.A., Skinner, M.M. and Burleigh, J.B., 1993. Evaluation of the Drop Bar Test for Concrete and Rock Quality. Geotechnical Testing Journal, Vol. 16, Issue 3, September.

Gillette, H.P. 1916. *Handbook of Rock Excavation, Methods and Cost.* McGraw-Hill Book Company, New York.

MWH, Americas, Inc. (MWH), 2012. *Midnite Mine Superfund Site Preliminary (30 Percent) Basis of Design Report.* Prepared on behalf of Newmont USA Limited and Dawn Mining Company for the U.S. EPA Region 10. December 19.

Natural Resources Conservation Service (NRCS) 2009. National Engineering *Handbook.* United States Department of Agriculture, 210-VI-NEH. January.

U.S. Army Corp of Engineers (USACE), 2008. Unified Facilities Guide Specifications. USACE/NAVFAC/AFCESA/NASA UFGS-31-00-00. August.

Midnite Mine Superfund Site

100 Percent Design

Appendix T – Water Source Identification and Development Plan

Revised October 2015

Prepared for:

Dawn Mining Company
PO Box 250
Ford, Washington 990413

and

Newmont USA Limited
6363 South Fiddler's Green Circle
Greenwood Village, Colorado 80111

Prepared By:

Worthington Miller Environmental, LLC
1027 W Horsetooth Road, Suite 200
Fort Collins, Colorado 80526

TABLE OF CONTENTS

T1.0 INTRODUCTION 1
T2.0 REGULATORY REQUIREMENTS AND USES 1
T3.0 WATER SOURCES 3
T4.0 WATER NEEDS, STORAGE, AND DISTRIBUTION 3
T5.0 REFERENCES 8

LIST OF TABLES

Table T-1. Summary of Water Sources, Standards, Use Conditions, Areas, and Sources
Table T-2. Quarterly Water Requirements (acre-feet)

LIST OF FIGURES

Figure 1. Location of Mancamp Well

LIST OF ACRONYMS

CFR	Code of Federal Regulations
Companies	Dawn Mining Company LLC/Newmont USA Limited
Dawn	Dawn Mining Company LLC
HDPE	high-density polyethylene
MCL	maximum contaminant level
MM	Midnite Mine
Newmont	Newmont USA Limited
OSHA	Occupational Safety and Health Administration
RA	remedial action
RCW	Revised Code of Washington
RO	Reverse Osmosis
SVOC	semivolatile organic compound
TDS	Total Dissolved Solids
VOC	volatile organic compound
WRIA	Water Resource Inventory Areas
WTP	water treatment plant

T1.0 INTRODUCTION

The Midnite Mine (MM) remediation project will use both potable and non-potable water to meet necessary construction and operational needs. Current water sources are assumed to be from the MM water treatment plant and from the mancamp well (Figure 1). On May 29, 2015, as part of joint mediation efforts, the Tribe and the Dawn/Newmont (Companies) entered into a non-binding terms of agreement, which sets forth the framework for detailed definitive agreements that will provide for water use from these sources. The parties are proceeding to prepare the necessary lease to implement these terms. The Tribe confirms the parties' entered into a confidential non-binding terms of agreement as described in the companies' 100% design. The parties are proceeding to prepare the necessary leases and agreements to implement those terms, subject to all necessary approvals.

T2.0 REGULATORY REQUIREMENTS AND USES

Non-potable water is water that meets NPDES discharge standards and will be sourced from the treated effluent from the water treatment plant (WTP) and used in contaminated areas. This water is not intended for drinking or human contact such as washing, cooking, laundry (29 CFR 1910.141(b)(2)(iii)). The majority of the non-potable water will be used on-site for construction water (i.e., dust control) in contaminated areas.

Potable water is water that meets the Safe Drinking Water Act (42 U.S.C. §300f et seq.) standards and will be used at the construction support facility for activities such as showers, toilets, laundry, decontamination, etc. Potable water will also be used for construction-related purposes (i.e., dust control) in uncontaminated or remediated areas that are deemed to be clean. Potable water will be obtained from either the mancamp well that was drilled on-site, or by providing additional treatment to a portion of the WTP effluent. The additional treatment will occur at the existing or new WTP, whichever one is active during the various phases of remediation (Figure 1). Potable non-human consumption water supplies will meet Safe Drinking Water Act standards for organic and inorganic constituents and will be appropriately disinfected. All water used specifically for human consumption (drinking) will be purchased by the contractor from a local vendor and supplied to the site for employees, contractors, and subcontractors. Table T-1 provides a summary of water quality, use conditions, areas, and sources.

Table T-1. Summary of Water Sources, Standards, Use Conditions, Areas, and Sources

Water Quality	Standard	Use Conditions	Use Areas	Water Sources
Potable	Safe Drinking Water Act standards	Drinking water	Unrestricted Area	Contractor provided drinking water purchased from local vendors
Potable	Safe Drinking Water Act standards	Construction Support Facility water includes non-consumptive human uses such as showers, toilets, laundry, hand washing, not drinking water, and decontamination	Construction Support Facility	Treated mancamp well water or water treatment plant water with additional treatment
Potable	Safe Drinking Water Act standards	Dust control in uncontaminated or remediated areas	Any area not listed as mine waste	Treated mancamp well water or water treatment plant water with additional treatment
Potable	Safe Drinking Water Act standards	Dust control for clean cover over the Waste Containment Area and clean topsoil	Any uncontaminated area such as the Rhoads Borrow area, area where waste is covered with uncontaminated material or clean areas once remediated	Treated mancamp well water or water treatment plant water with additional treatment
Potable	Safe Drinking Water Act standards	Fire suppression water (10,000 gallons) stored in a temporary HDPE tank	All areas	Treated mancamp well water or water treatment plant water with additional treatment
Non-potable	NPDES standard	Dust control in contaminated areas of mine waste, prior to surface clean-up	Areas contaminated with mine waste and sediment identified in the BODR	Water Treatment Plant Effluent water or untreated Man Camp well water

Cross contamination of potable and non-potable water will be avoided with separate plumbing and all non-potable water fixtures would be demarcated and signed in accordance with local and state requirements (29 CFR 1619.1). For facilities with 20 to 200 employees, OSHA requires 1 toilet and 1 urinal for every 40 employees (29 CFR 1926.51(c)(1)). Sizing of the septic system and leach fields will comply with the Northeast Tri-County District requirements for on-site sewer treatment systems and disposal programs.

Water needed for dust control could range from one to eight gallons per cubic yard of material excavated and transported, depending upon the season. The effectiveness of water for dust control is relatively short lived and can last anywhere from ½ to 12 hours (Bolander and Yamada, 1999). Actual water application rates and water demand will be dependent upon the size and type of construction equipment, characteristics of the soil particles, area of exposed soil, ambient weather temperature, and relative humidity. Dust control water usage may be augmented by the use of organic chemical dust suppressant as Ligno-sulfonate. Estimated potable water use for the construction support facility will vary as the construction employee work force numbers fluctuate; however, the current estimate of potable water usage is 55 gallons per day per person for consumptive potable water, but does not include drinking water. Water storage facilities will be required to store both potable and non-potable water to allow capacity for peak use times. It is estimated that approximately 350 acre feet of water would be needed for the life of the project of which five to ten percent would be potable.

T3.0 WATER SOURCES

It is anticipated that construction water will be obtained from both the WTP and the mancamp well. Based on an investigation (WME, 2015) the well at the mancamp produces approximately 6,000 gallons per day using a pump. The well's water quality sample was tested for VOCs, SVOCs, radionuclides, metals, total coliform, fecal coliform and E. coli (WME, 2015). The sample was also below MCLs for all constituents except uranium. The sample tested at 0.0344 mg/L total uranium, and the MCL for uranium is 0.030 mg/L (WME, 2015). The water would be treated for uranium using a custom designed system that integrates negatively charged ion exchange resin, from a local contractor who has extensive experience in treating groundwater wells in the area, unless further well development and water use activities demonstrate the well routinely produces water quality below the uranium MCL. Local vendors indicate that they have extensive experience treating water in the area with uranium concentrations up to 200 mg/l to levels well below the MCL. The water would be stored in a tank for potable use. The resin would be flushed once a week and the resin would be reused. The backwash water used to flush the resin will be treated at the WTP. Once established, the water supply would be tested annually. The resin would be replaced every 15 to 20 years, depending upon its continued effectiveness. Spent resin will be sampled, analyzed and disposed of in accordance with State and Federal regulations.

The WTP effluent will be used for non-potable water that will meet NPDES discharge standards and be used only in contaminated areas. The WTP effluent that receives additional treatment to Drinking Water Act standards will be deemed potable water that will be used at the Construction Support Facility and for construction in uncontaminated or remediated areas.

T4.0 WATER NEEDS, STORAGE, AND DISTRIBUTION

The water needs for construction have been estimated based on the construction schedule the amount of water necessary for dust control, water use to support the construction support zone

and WTP. That estimate is shown In Table T-2. It is assumed that treated water from the water treatment plant could be used for dust control on contaminated surfaces. Water for dust control on un-contaminated surfaces and for use in the construction support zone would meet drinking water standards (potable water). Make up water for the water treatment plant has historically been obtained from Pit 4. It is assumed that water from Pit 4 would continue to supply this water. Water use and supply will be re-evaluated following the first and second phases of construction to determine if water supplies are adequate. Additional water will be obtained if determined to be necessary.

Water for construction will be piped from the water treatment plant to selected areas where it will be stored in temporary storage tanks or ponds. The potable water, necessary for use in uncontaminated or remediated areas will be kept in a separate system. The potable water will receive additional treatment at the WTP and then piped to the construction support facilities for use. Details of the potable and non-potable water storage and distribution systems will be determined by the contractor as required in section 3.4, 01510 – Temporary Utilities of the Technical Specifications in Appendix K in the BODR.

Water for fire suppression needs will be provided by the selected RA Contractor, as described in the specifications. A temporary HDPE tank will be provided to store at least 10,000 gallons of clean water for fire suppression during the construction activities. Should a fire start during construction, it is assumed that the Spokane Tribal firefighting crews can be relied on to provide additional fire suppression support.

Table T-2. Quarterly Water Requirements (acre-feet)

Year/ Qtr	Quarterly On-Site (WTP Effluent) Water Usage (Non-Potable)							Quarterly Off-Site (Polished WTP Effluent) Water Usage (Potable)								Quarterly Construction Support Facility Potable Water Usage		
	Mine Waste EW Quantity (yd^3)	Earthworks Dust Suppression (gal)	Crusher Operation (days)	Crusher Dust Suppression (gal)	Vehicle Decontaminatio n (gal)	Total On-site Water Usage (gal)	Total On-site Water Usage (acre-feet)	Access Road Dust Suppression (gal) assuming paved, assume 160,000 min/qtr	Cover Soil EW Quantity (yd^3)	Cover Soil Dust Suppression (gal)	Compacted Fill Quantity (yd^3)	Compacted Fill Dust Suppression (gal)	Compacted Fill - Compaction Water (gal)	Total Off-site Water Usage (gal)	Total Off-site Water Usage (acre-feet)	Personnel Onsite	Total Potable Water Usage (gal)	Total Potable Water Usage (acre-feet)
2016	140,000						3.3								2.9			0.5
Q1	-	-	-	-	65,000	65,000	0.20	200,000	-	-	-	-	-	200,000	0.61	20	19,500	0.06
Q2	-	-	-	-	65,000	65,000	0.20	200,000	-	-	11,082	55,410	86,440	341,850	1.05	20	19,500	0.06
Q3	80,000	640,000	-	-	65,000	705,000	2.16	200,000	-	-	-	-	-	200,000	0.61	60	58,500	0.18
Q4	60,000	60,000	32	115,200	65,000	240,200	0.74	200,000	-	-	-	-	-	200,000	0.61	60	58,500	0.18
2017							7.7	-							2.5			0.7
Q1	57,500	57,500	65	234,000	65,000	356,500	1.09	200,000	-	-	-	-	-	200,000	0.61	60	58,500	0.18
Q2	76,500	382,500	-	-	65,000	447,500	1.37	200,000	-	-	-	-	-	200,000	0.61	60	58,500	0.18
Q3	142,200	1,137,600	-	-	65,000	1,202,600	3.69	200,000	-	-	-	-	-	200,000	0.61	60	58,500	0.18
Q4	434,900	434,900	-	-	65,000	499,900	1.53	200,000	-	-	-	-	-	200,000	0.61	60	58,500	0.18
2018							56.4	-							7.6			1.0
Q1	245,000	245,000	-	-	65,000	310,000	0.95	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q2	658,200	3,291,000	-	-	65,000	3,356,000	10.30	200,000	271,000	1,355,000	-	-	-	1,555,000	4.77	80	78,000	0.24
Q3	1,716,900	13,735,200	-	-	65,000	13,800,200	42.35	200,000	40,000	320,000	-	-	-	520,000	1.60	80	78,000	0.24
Q4	741,900	741,900	32	115,200	65,000	922,100	2.83	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
2019							19.2	-							2.5			1.0
Q1	340,000	340,000	65	234,000	65,000	639,000	1.96	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q2	573,500	2,867,500	32	115,200	65,000	3,047,700	9.35	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q3	292,900	2,343,200	-	-	65,000	2,408,200	7.39	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q4	88,300	88,300	-	-	65,000	153,300	0.47	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24

Table T-2,
Pg 1 of 3

Table T-2. Quarterly Water Requirements (acre-feet) , continued

Year/ Qtr	Quarterly On-Site (WTP Effluent) Water Usage (Non-Potable)							Quarterly Off-Site (Polished WTP Effluent) Water Usage (Potable)								Quarterly Construction Support Facility Potable Water Usage		
	Mine Waste EW Quantity (yd^3)	Earthworks Dust Suppression (gal)	Crusher Operation (days)	Crusher Dust Suppression (gal)	Vehicle Decontaminatio n (gal)	Total On-site Water Usage (gal)	Total On-site Water Usage (acre-feet)	Access Road Dust Suppression (gal) assuming paved, assume 160,000 min/qtr	Cover Soil EW Quantity (yd^3)	Cover Soil Dust Suppression (gal)	Compacted Fill Quantity (yd^3)	Compacted Fill Dust Suppression (gal)	Compacted Fill - Compaction Water (gal)	Total Off-site Water Usage (gal)	Total Off-site Water Usage (acre-feet)	Personnel Onsite	Total Potable Water Usage (gal)	Total Potable Water Usage (acre-feet)
2020							34.3	-							2.5			1.0
Q1	105,800	105,800	-	-	65,000	170,800	0.52	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q2	511,500	2,557,500	-	-	65,000	2,622,500	8.05	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q3	974,000	7,792,000	-	-	65,000	7,857,000	24.11	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q4	464,000	464,000	-	-	65,000	529,000	1.62	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
2021							57.3											1.0
Q1	1,228,286	1,228,286	-	-	65,000	1,293,286	3.97	200,000								80	78,000	0.24
Q2	1,228,286	6,141,429	-	-	65,000	6,206,429	19.05	200,000								80	78,000	0.24
Q3	1,228,286	9,826,286	-	-	65,000	9,891,286	30.36	200,000								80	78,000	0.24
Q4	1,228,286	1,228,286	-	-	65,000	1,293,286	3.97	200,000								80	78,000	0.24
2022							55.7											1.0
Q1	1,228,286	1,228,286	-	-	65,000	1,293,286	3.97	200,000								80	78,000	0.24
Q2	1,228,286	6,141,429	-	-	65,000	6,206,429	19.05	200,000								80	78,000	0.24
Q3	1,268,286	10,146,286	-	-	65,000	10,211,286	31.34	200,000								80	78,000	0.24
Q4	385,667	385,667	-	-	65,000	450,667	1.38	200,000								80	78,000	0.24
2023							29.0											1.0
Q1	345,667	345,667	-	-	65,000	410,667	1.26	200,000								80	78,000	0.24
Q2	845,667	4,228,333	-	-	65,000	4,293,333	13.18	200,000								80	78,000	0.24
Q3	514,000	4,112,000	-	-	65,000	4,177,000	12.82	200,000								80	78,000	0.24
Q4	514,000	514,000	-	-	65,000	579,000	1.78	200,000								80	78,000	0.24

Table T-2,
Pg 2 of 3

Table T-2. Quarterly Water Requirements (acre-feet) , continued

Year/ Qtr	Quarterly On-Site (WTP Effluent) Water Usage (Non-Potable)							Quarterly Off-Site (Polished WTP Effluent) Water Usage (Potable)								Quarterly Construction Support Facility Potable Water Usage		
	Mine Waste EW Quantity (yd^3)	Earthworks Dust Suppression (gal)	Crusher Operation (days)	Crusher Dust Suppression (gal)	Vehicle Decontaminatio n (gal)	Total On-site Water Usage (gal)	Total On-site Water Usage (acre-feet)	Access Road Dust Suppression (gal) assuming paved, assume 160,000 min/qtr	Cover Soil EW Quantity (yd^3)	Cover Soil Dust Suppression (gal)	Compacted Fill Quantity (yd^3)	Compacted Fill Dust Suppression (gal)	Compacted Fill - Compaction Water (gal)	Total Off-site Water Usage (gal)	Total Off-site Water Usage (acre-feet)	Personnel Onsite	Total Potable Water Usage (gal)	Total Potable Water Usage (acre-feet)
2024							5.2	-							7.6			1.0
Q1	500,000	500,000	-	-	65,000	565,000	1.73	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q2	50,000	250,000	-	-	65,000	315,000	0.97	200,000	-	-	129,732	648,660	1,011,910	1,860,570	5.71	80	78,000	0.24
Q3	87,000	696,000	-	-	65,000	761,000	2.34	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
Q4		-	-	-	65,000	65,000	0.20	200,000	-	-	-	-	-	200,000	0.61	80	78,000	0.24
2025							7.9	-							12.5			0.5
Q1		-	-	-	65,000	65,000	0.20	200,000	-	-	-	-	-	200,000	0.61	40	39,000	0.12
Q2	460,000	2,300,000	-	-	65,000	2,365,000	7.26	200,000	654,000	3,270,000	-	-	-	3,470,000	10.65	40	39,000	0.12
Q3	-	-	-	-	65,000	65,000	0.20	200,000	-	-	-	-	-	200,000	0.61	40	39,000	0.12
Q4	-	-	-	-	65,000	65,000	0.20	200,000	-	-	-	-	-	200,000	0.61	40	39,000	0.12

Table T-2,
Pg 3 of 3

Assuming
1 gal/yd^3 Average Annual Dust Suppression Water Usage During Q1 & 4
5 gal/yd^3 Average Annual Dust Suppression Water Usage During Q2
8 gal/yd^3 Average Annual Dust Suppression Water Usage During Q3
65 work days per quarter
3 gpm crusher dust suppression water application rate
1,000 gpd used for vehicle contamination each work day during quarter
15 gpd/per person potable water consumption, primarily incidental laundry and showers, does not include drinking water
8 gal/yd^3 may be required for moisture conditioning of compacted fill, assuming 2% moisture may be added to fill materials during placement
0 gal/day (no dust suppression watering) required on access road due to paved surface

Notes:
1. Schedule is based on current project assumptions and are subject to change based on input from selected contractor.
2. These estimates will be updated following the first and second phases of construction.
3. Water requirements for equipment decontamination and compaction during backfill operations were relatively insignificant and were not broken out from the overall dust control and construction support facility needs.

T5.0 REFERENCES

Bolander, Peter and Alan Yamada. 1999. Dust Palliative Selection and Application Guide. Prepared for the US Forest Service (USFS). Accessed at http://www.ecy.wa.gov/programs/air/pdfs/Dust_Palliative.pdf on 11/21/2012.

Worthington Miller Environmental (WME). 2015. Man Camp Water Supply Well, Data Evaluation Report.

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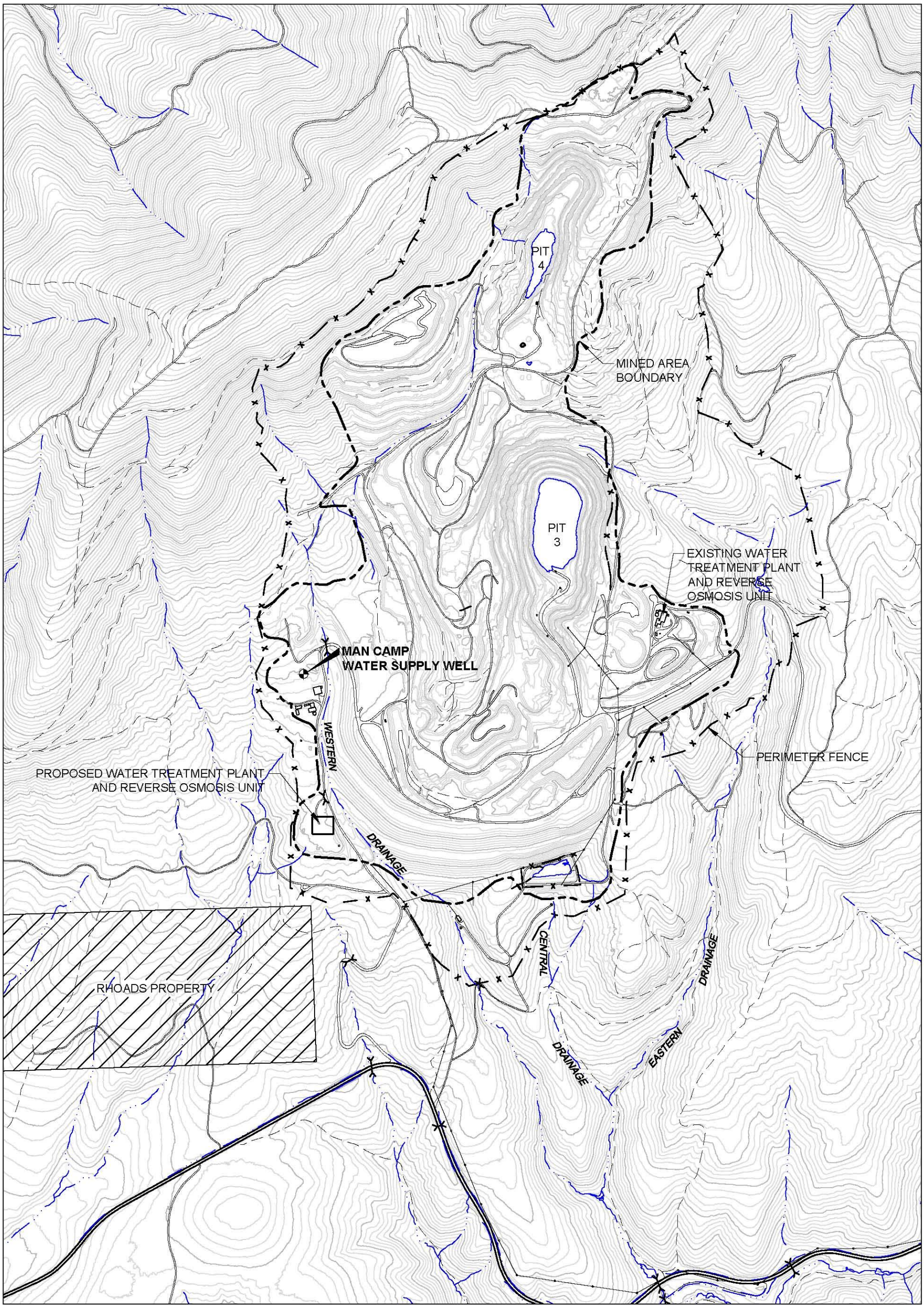


WORTHINGTON MILLER ENVIRONMENTAL, LLC.

FIGURE 1
MAN CAMP WATER SUPPLY WELL LOCATION

Date:	SEPTEMBER 2015
Project:	MIDNITE
File:	FIG-MANCAMPWELL-JK

Midnite Mine Superfund Site
100 Percent Design

Appendix U – Remedial Action Construction Quality Assurance Plan

Revised October 2015

Prepared for:

Newmont USA Limited
6363 South Fiddler's Green Circle
Greenwood Village, Colorado 80111

and

Dawn Mining Company
PO Box 250
Ford, Washington 990413

Prepared By:

MWH Americas, Inc.
2890 E. Cottonwood Parkway, Suite 300
Salt Lake City, UT 84121

TABLE OF CONTENTS

U1.0 INTRODUCTION 1

U1.1 BACKGROUND 1
U1.2 KEY QUALITY PROGRAM TERMS 2
U1.3 CONSTRUCTION QUALITY PROGRAM OVERVIEW 2
U1.4 CQA/CQC PROCESS 3

U2.0 RESPONSIBILITIES AND AUTHORITIES 4

U2.1 RESPONSIBILITIES AND AUTHORITIES OF ORGANIZATIONS 5
U2.1.1 Environmental Protection Agency 5
U2.1.2 Spokane Tribe of Indians 5
U2.1.3 Newmont USA, Limited/Dawn Mining Company 5
U2.1.4 Supervising Contractor 5
U2.1.5 Project Designer 6
U2.1.6 Construction Management 6
U2.1.7 Construction Contractor (CC) 6
U2.2 RESPONSIBILITIES OF KEY PERSONNEL 7
U2.2.1 Construction Management Quality Assurance Personnel 7
U2.2.2 Construction Contractor's Quality Control Personnel 9

U3.0 PROJECT MEETINGS 10

U3.1 PRE-CONSTRUCTION MEETINGS 10
U3.2 PROGRESS MEETINGS 10

U4.0 INSPECTION AND VERIFICATION ACTIVITIES 11

U4.1 FOUR-PHASE INSPECTION PROGRAM 11
U4.1.1 Preparatory Inspection 11
U4.1.2 Initial Phase Inspection 12
U4.1.3 Follow-Up Phase Inspection 12
U4.1.4 Completion Inspection 13
U4.2 COMPLIANCE WITH TECHNICAL SPECIFICATIONS 13
U4.2.1 CQC Testing 13
U4.2.2 CQA Monitoring 14
U4.2.3 Construction Acceptance Criteria 14
U4.3 COMPLIANCE WITH HANDLING, STORAGE, PACKAGING, PRESERVATION, AND DELIVERY REQUIREMENTS 14
U4.4 MATERIAL IDENTIFICATION AND Management 15

U5.0 CONSTRUCTION DEFICIENCIES 15

U5.1 DEFICIENCY DEFINITION 15
U5.2 CC CQC DEFICIENCY IDENTIFICATION AND CONTROL 15
U5.2.1 Minor Deficiencies 15
U5.2.2 Nonconformances 16

U5.3 CC CQC DEFICIENCY CORRECTION 16
U5.4 PREVENTATIVE ACTIONS 17
U6.0 DOCUMENTATION.....17
U6.1 DAILY RECORDKEEPING 17
U6.2 DAILY CONSTRUCTION REPORT 18
U6.3 RECORD DRAWINGS.....18
U6.4 CONTROL OF QUALITY RECORDS.....19
U7.0 CHANGE MANAGEMENT 19
U7.1 Engineering Change Notices 19
U7.2 CQAP CHANGES 19
U7.3 CC QUALITY CONTROL SYSTEM CHANGES 20
U8.0 REFERENCES.....20

TABLE

Table U-1 Construction Quality Program Summary

FIGURE

Figure U-1 Remedial Action Organizational Chart

ATTACHMENT

Attachment U-1 Example Forms

LIST OF ACRONYMS

CC	Construction Contractor
CD	Consent Decree
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CM	Construction Management
Company	Newmont USA, Limited/Dawn Mining Company, LLC
CQA	Construction Quality Assurance
CQAO	Construction Quality Assurance Officer
CQAP	Construction Quality Assurance Plan
CQC	Construction Quality Control
ECN	Engineering Change Notice
EPA	U.S. Environmental Protection Agency
FE	Field Engineer
FI	Field Inspector
NNC	Notice of Non-Compliance
RA	Remedial Action
RAWP	Remedial Action Work Plan
RD	Remedial Design
RFI	Request for Information
ROD	Record of Decision
Site	Midnite Mine Superfund Site
SOW	Scope of Work
QA	Quality Assurance
QC	Quality Control
QCM	Quality Control Manager
Tribe	Spokane Tribe of Indians
TERO	Tribal Employment Rights Ordinance
USACE	United States Army Corps of Engineers
WME	Worthington Miller Environmental

U1.0 INTRODUCTION

This Construction Quality Assurance Plan (CQAP) describes the site-specific components of the quality assurance (QA) program to ensure, to the extent practicable, that the completed remedial action (RA) at the Midnite Mine Superfund Site (Site) meets design criteria, plans, and specifications. This CQAP presents the Construction Quality Assurance (CQA) components that are common to the RAs. This CQAP will be supplemented with task-specific CQA information that will be included in the Remedial Action Work Plan (RAWP). Examples of task-specific CQA information that will be included in the RAWP include identification of key personnel and construction contractors (CCs) and defining the task-specific inspection and testing requirements. Together, this CQAP and the CQA information included in the RAWP identify the methods and procedures that will be used by the Construction Management staff to obtain independent, documented confirmation that the construction standards required by the contract documents have been met.

This CQAP has been prepared in accordance with the *Technical Guidance Document – Construction Quality Assurance for Hazardous Waste Land Disposal Facilities* (U.S. Environmental Protection Agency [EPA], 1987) and the CQAP requirements outlined in the *Consent Decree Statement of Work* (CD SOW; EPA, 2011), and includes the following information:

- Responsibility and Authority – The responsibility and authority of organizations and key personnel involved in the RA.
- Construction Quality Assurance Officer (CQAO) Qualifications.
- Inspection Activities – The observations and tests to assure that the RA meets design criteria.
- Sampling Strategies – The sampling activities, acceptance and rejection criteria, and methods for assuring that corrective measures are implemented.
- Documentation – Reporting requirements for CQA activities.

U1.1 BACKGROUND

The Site is in the Remedial Design/Remedial Action (RD/RA) stage of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) process.

The RD provides the engineering plans and Technical Specifications for implementing the Selected Remedy, which is defined in the *Midnite Mine Superfund Site Record of Decision* (ROD; EPA, 2006) and stipulated in an RD/RA (Remedial Action) CD lodged by the United States District Court on 17 January 2012.

This CQAP supports the RAs and will be updated as necessary to incorporate major changes to the project team or CQA procedures. Newmont USA, Limited and Dawn Mining Company, LLC (Company) collectively are the responsible party for the planned RAs.

U1.2 KEY QUALITY PROGRAM TERMS

Two related, but independent processes that are associated with the construction quality program are CQA and Construction Quality Control (CQC), defined as follows:

Construction Quality Assurance (CQA) - Refers to a planned system of activities that provide documentation that the project is constructed as specified in contract documents, and that the materials used in construction are manufactured according to specification. CQA is the process of planning or managing for quality, and is intended to *prevent defects* in the finished product. CQA includes inspections and audits of materials and workmanship necessary to determine and document that the end product complies with the quality established by the contract. The Construction Management (CM) staff will perform CQA activities on behalf of the Company.

Construction Quality Control (CQC) – Refers to the process by which the characteristics of the constructed product are compared with applicable standards; and the action taken when nonconformance is detected. CQC is the process for verifying predefined requirements for quality, and is intended to *identify defects* in the product during and prior to construction completion. CQC includes surveying, sampling, and testing to directly monitor the quality of all furnished, constructed, and installed components. CQC activities are the responsibility of the CC in order to demonstrate that the work product complies with the Technical Specifications.

U1.3 CONSTRUCTION QUALITY PROGRAM OVERVIEW

Table U-1 presents a summary of the construction quality program. Specific details regarding how the construction quality program will be implemented is presented below.

The Company will contract a Prime CC to perform the construction activities and CM staff to supervise the CC. The approach to managing the quality of the RA includes a combination of CQA performed by the CM staff and CQC performed by the CC. The CC is responsible for

constructing the work in accordance with the contract documents, which incorporate the design plans and Technical Specifications.

The CC CQC is the systematic implementation of a program of surveys, sampling, and tests to attain the required construction standards and to preclude and/or resolve problems related to noncompliance. In accordance with the Technical Specifications, the CC will establish a quality control (QC) system to perform sufficient inspection and tests of all items of work, including that of their subcontractors, to ensure conformance to the functional performance of the project. The CC's QC system shall be established for all construction except where the contract documents provide for specific compliance tests by testing laboratories employed by the Company. The CC's QC system shall specifically include all testing required by the various sections of the Technical Specifications.

Independent of the CC's efforts, the CM staff will provide CQA through monitoring of test results and scheduled inspections to verify the effectiveness of the CC's CQC program, and assure that the quality and contract requirements are achieved and documented.

U1.4 CQA/CQC PROCESS

The two primary CQA/CQC processes that will be implemented during the RA include:

Four-Phase Inspection Program. The CM staff will implement a four-phase inspection program modeled after the U.S. Army Corps of Engineers (USACE) Construction Quality Management regulations (ER 1180-1-6; USACE, 1995). The primary purpose of the program is to require that the CM and CC staff jointly plan, schedule, and review the work as it progresses, identify and correct deficiencies, and confirm that the completed work meets the requirements of the contract documents. The four program phases include 1) Preparatory, 2) Initial, 3) Follow-up, and 4) Completion. The CM staff will facilitate (and the CC staff will participate in) the process for each major work element. The major work elements (or "definable features of work") will be identified in the RAWP.

Compliance with Technical Specifications. The CC staff will perform (and the CM staff will monitor) surveys, inspections, sampling, and testing, as required by the Technical Specifications. The specific CQA/CQC requirements of the Technical Specifications will be summarized in the RAWP.

Additional details regarding the inspection and testing activities to be performed by the CM and CC staff during RA are presented in Section U4.0.

Table U-1 – Construction Quality Program Summary

	Construction Quality Assurance (CQA)	Construction Quality Control (CQC)
Performed by	Construction Management (CM) – Company representative	Construction Contractor (CC) contracted by the Company
What	Planning and oversight conducted to provide documentation that the RA is constructed as specified in contract documents.	Surveying, sampling, and testing performed to compare the constructed product with predefined standards
Primary Goals	• Prevent nonconformance through planning • Monitor quality during construction	• Identify nonconformance via testing • Correct nonconformance
Roles [a]	• Construction Quality Assurance Officer (CQAO)/Field Engineer (FE)/Field Inspector (FI)	• Quality Control Manager (QCM)/QC Technician(s))
Four-Phase Inspection Program		
Preparatory	• Plan, schedule, facilitate and document • Review pertinent parts of the plans and Technical Specifications before work begins.	• Participate • Confirm understanding of SOW and commitment to follow pre-established quality procedures.
Initial	• Plan, schedule, facilitate and document • Inspect initial work products to identify nonconformance early during construction.	• Participate • Correct nonconformance before work progresses too far.
Follow-up	• Plan, schedule, facilitate and document • Routine inspections to ensure compliance with pre-established quality criteria.	• Participate • Demonstrate that acceptable level of quality in workmanship is maintained.
Completion	• Plan, schedule, facilitate, and document • Verify that completed work meets contractual requirements.	• Participate • Resolve nonconformance items • Demonstrate that completed work meets contractual requirements.
Compliance with Technical Specifications [b]		
Identify required verification tests and inspections	• Identify and summarize required verification tests and inspections for inclusion in the RAWP • Review and approve CC's QC system	Establish a QC system in accordance with the Technical Specifications
Testing/Monitoring	Perform monitoring and verification of CC's inspections and testing during construction	Perform inspection and tests during construction

[a] Roles may be combined for uncomplicated tasks and/or smaller construction projects.
[b] The Technical Specifications are the explicit requirements to be satisfied by the CC and are included in the contract documents.

U2.0 RESPONSIBILITIES AND AUTHORITIES

This section presents the responsibilities and authorities of organizations and key personnel involved in the RA, the structure of the CQA/CQC organization, and the minimum training and experience of the CQA/CQC personnel. The overall RA organizational structure and key

CQA/CQC personnel are shown on Figure U-1. The identities and roles and responsibilities of key personnel will be further defined in the CQA section of the RAWP.

U2.1 RESPONSIBILITIES AND AUTHORITIES OF ORGANIZATIONS

U2.1.1 Environmental Protection Agency

The EPA is the lead agency governing the remediation of the Midnite Mine Superfund Site. The EPA issued the ROD and CD, and is responsible for approving all plans and reports related to implementing the Selected Remedy, including this CQAP. The EPA Remedial Project Manager is Ms. Karen Keeley. The EPA has contracted CH2M Hill as their oversight contractor. The CH2M Hill point of contact is Ms. Kira Sykes.

U2.1.2 Spokane Tribe of Indians

The Site is located on lands owned by the Federal Government and held in trust for the Spokane Tribe of Indians (Tribe) and individual Tribe members. Mr. Randy Connolly is the Tribe Superfund Coordinator. The Tribe has access to contract technical support from AESE, Inc. The AESE, Inc. point of contact is Dr. F. E. Kirschner. In its capacity as a support agency under CERCLA, the Tribe has the opportunity to review each submittal and provide comment to the EPA.

U2.1.3 Newmont USA, Limited/Dawn Mining Company

The Company has responsibility for procuring consultants and contractors to perform the RA work, including budgeting and securing the necessary funds, and assuring that the requirements of the CD are met. The Company is responsible for executing all administrative aspects of the contract with the CC such as contract approval, claims, change orders, amendments, pay applications, and materials through the CM team (described below). The CM team will assist and advise the Company with contract related matters. The Company Project Coordinator is Mr. Nick Cotts and the Alternate Project Coordinator is Mr. William Lyle.

U2.1.4 Supervising Contractor

Mr. Louis Miller, P.E. of Worthington Miller Environmental (WME) is the Supervising Contractor procured by the Company to implement the Selected Remedy and supervise site management activities. As the Supervising Contractor, Mr. Miller will direct and supervise the RA, and is responsible on behalf of the Company for assuring the overall quality of the RA.

U2.1.5 Project Designer

The Project Designer is MWH Americas, Inc. (MWH), a licensed design firm retained by the Company to provide design and engineering services in connection with the RA. The Project Designer reports directly to the Supervising Contractor and is responsible to both the Supervising Contractor and the Company. The Project Designer will identify in the RAWP the tests and inspections required to demonstrate that the RA is performed as specified in the design. The MWH Project Manager is Mr. Vance Drain, P.G., and the MWH Engineering Manager is Mr. Clint Strachan, P.E.

U2.1.6 Construction Management

The CM includes qualified personnel retained by the Company to provide professional construction management and related services in connection with the RA, including CQA. The CM is responsible for implementation of this CQAP (see Section U2.2.1). The CM will be independent from the CC (described below). The CM will oversee the CC on behalf of the Company (including review of the CC's proposed means and methods) and serve as the primary point of contact with the CCs. The CM provides CQA and monitors the day-by-day CQC activities performed by the CCs to verify compliance with the contract documents, design plans, and Technical Specifications. The CM also will manage, coordinate, and administer all CQA activities and requirements. The CM will be identified in the RAWP.

U2.1.7 Construction Contractor (CC)

The Company will retain the CC to provide the labor, materials, and equipment required to construct the project in accordance with the contract documents. The CC will operate in compliance with the Tribal Employment Rights Ordinance (TERO). The CC is responsible for scheduling, coordinating, and planning the construction work (e.g., the means and methods). The CC is responsible for the quality of their constructed work product as well as the necessary inspections and tests required to ensure that their work complies with the contract documents. They exercise authority over their workforce, including CQC personnel (described below in Section U2.2.2), subcontractors and their CQC support services. The CC and CQC staff will be identified in the RAWP. As described above, the CC will establish a QC system in accordance with the Technical Specifications.

U2.2 RESPONSIBILITIES OF KEY PERSONNEL

The CQA/CQC roles and responsibilities for CM and CC personnel are described below in Section U2.2.1 and Section U2.2.2, respectively. These may vary based on nomenclature used by the actual CM and CCs contracted by the Company to implement the RAs. The CQA/CQC staff will be on Site as needed during the RA based on the nature, volume, or complexity of the tasks being performed at any given time, and the CQA/CQC requirements associated with those tasks. A single qualified person may perform multiple CQA/CQC roles, as approved by the CM CQAO. This will allow the flexibility for staff only to be on Site as necessary when routine or uncomplicated RA tasks are being performed. For example, a single person may assume the roles of Construction Quality Assurance Officer (CQAO), Field Engineer (FE) and Field Inspector (FI) as discussed below. Likewise, because of the long duration of the RA, multiple qualified persons might fill a CQA/CQC staffing role. These details and personnel responsibility will be included in the CQA section of the RAWP.

U2.2.1 Construction Management Quality Assurance Personnel

Construction Quality Assurance Officer (CQAO). The CQAO will coordinate the field implementation of this CQAP, including designating and delegating appropriate CM tasking to provide CQA oversight of the RA at any given time. The CQAO will have responsibility for assembling, tracking, and storing all CQA/CQC related documentation. The primary duty of the CQAO is to confirm and document that the RA is implemented in accordance with the contract documents.

The CQAO will have authority to institute actions necessary for the successful implementation of the CQA/CQC program to ensure compliance with the contract plans and Technical Specifications (including stop-work authority). The CQAO coordinates activities to ensure that FE activities (discussed below), inspection staff, and testing firms as well as CC CQC staff carry out the requirements of this CQAP and the CC's QC system.

The CQAO will track and report nonconformances to CC management and CC CQC staff. The CQAO has authority to obtain direct access to CC CQC files. Other CQAO responsibilities will include:

- Reviewing CC reports, tests, and inspection results
- Facilitating the implementation of the four-phase inspection program (Section U4.1) and participating in the required inspections

- Ensuring that CQA personnel conducting inspections are adequately trained and understand assignment limits and time frames.
- Review and comply with the RA design plans and Technical Specifications.

The CQAO will be an individual with sufficient combined experience, as deemed adequate by the Company and the Supervising Contractor in one or more of the following positions: Project Superintendent, QC Manager, Project Manager, Project Engineer, or Construction Manager on similar size and type construction contracts.

The CQAO may assume the role of the FE described below.

Field Engineer (FE). The FE's primary responsibility is to administer the CC's contract. This includes ensuring the RA is performed in accordance with the design plans and Technical Specifications. The FE reviews CQC testing documentation with the CC, engineers, and inspectors. The FE also reviews design plans and Technical Specifications for assigned project components and estimates the type and number of CQC tests that should be accomplished for each specification section.

Specific responsibilities of the FE include:

- Coordinate requests for information (RFIs) and facilitating design clarifications with the Project Designer when necessary and distribute them to CQA/CQC team members and construction staff
- Assist in facilitating process change(s) to eliminate nonconformance trends
- Maintain, control, and supervise the RAWP and required submittals between the CC, subcontractors, suppliers, and the Project Designer.

The FE may assume the role of the FI described below.

Field Inspector (FI). CM staff may include one or more FIs to support the CQAO and FE. The FE/FI will monitor the day-to-day activities of the CC. This includes ensuring that CC complies with the design plans and Technical Specifications, applicable building codes, good workmanship, and the CQC requirements of the contract. As part of this effort, FI will:

- Conduct independent inspections to verify the quality of the work
- Participate in the four-phase inspection program (refer to Section U4.1)
- Review test and inspection reports

- Ensure that the required documentation is generated and submitted to the CQAO.

The FI must be alert to detecting, recording, and reporting any deviation from the design and contract documents, including calling any deficient item to the attention of the FE, the CC superintendent, and/or other representative. The FI must keep accurate and detailed records of the CC's performance and progress, delivery of materials, and other pertinent matters, including the daily inspection report.

U2.2.2 Construction Contractor's Quality Control Personnel

Quality Control Manager (QCM). The QCM is responsible for daily on-site implementation of the CC's QC system and coordinating CQC activities with the CM team. The QCM is responsible for:

- Ensuring all tests and inspections are performed in accordance with the Technical Specifications
- Reviewing CQC reports, tests, and inspection results to determine compliance with design plans and Technical Specifications, and other contractual documents
- Participating in the four-phase inspection program (refer to Section U4.1)
- Documenting all CQC activities, and supplying this documentation to the CM team
- Rectifying nonconformances in a timely fashion
- Ensuring that CC and subcontractor CQC personnel conducting inspections are adequately trained and understand assignment limits and time frames.

QC Technicians. CC staff may include QC Technicians to support the QCM. The QCM may assume the role of the QC Technician, which includes the following functions:

- Inspect materials, construction, and equipment for conformance with the Technical Specifications
- Perform CQC tests, as required by the Technical Specifications.

CQA/CQC staff will be on Site as needed during the RA activities based on the nature, volume, or complexity of the tasks being performed at any given time and the CQA/CQC requirements associated with those tasks. A single qualified person may perform multiple CQA roles as approved by the CQAO. This will allow the flexibility for staff only to be on-site as necessary. Staffing details will be included in the CQA section of the RAWP.

U3.0 PROJECT MEETINGS

U3.1 PRE-CONSTRUCTION MEETINGS

The CQAO will plan and participate in a pre-construction meeting prior to initiating RA work components. The purpose of pre-construction meetings is to resolve uncertainties following award of the construction contract, but prior to the start of construction. At a minimum, the meeting will be attended in person or via telephone by the Company (or Company's representative), the Supervising Contractor, the Project Designer, the CQAO, FE, and CC project manager or superintendents and QCM.

Topics covered in this meeting will include, but will not be limited to, the following:

- Responsibilities of each organization
- Lines of authority and communication for each organization
- Relevant CQA/CQC documents and supporting information
- Procedures and/or protocols for observations, inspections, and tests
- Procedures and/or protocols for handling construction deficiencies
- Submittal requirements
- RFIs and design change process
- Methods for distributing and storing documents and reports
- Work area security and health and safety protocols
- Project schedule

The CQAO will document the meeting and distribute minutes to all parties identified in Section U2.0 within five working days of the meeting.

U3.2 PROGRESS MEETINGS

The FE will plan and participate in weekly progress meetings held at the Site during the RA. Additional progress meetings can be scheduled by either the CM or CC staff. The FE will document these meetings in daily field reports. At a minimum, the progress meetings will be attended by the FE, the CC QCM, and superintendent. Topics covered in the progress meetings will include:

- Previous activities and accomplishments
- Planned work locations and activities
- Personnel and equipment assignments
- Any new test data
- Resolution of previously identified problems
- Any potential construction problems, deficiencies, or nonconformances
- Changes or delays to the construction schedule
- Claims, change orders, and similar items

U4.0 INSPECTION AND VERIFICATION ACTIVITIES

U4.1 FOUR-PHASE INSPECTION PROGRAM

The CQAO will implement a four-phase inspection program for each definable feature of the work (the definable features of work will be identified in the RAWP). The CQAO will plan, schedule, facilitate, and document the inspections, and the CC QCM will participate in all phases. The four phases of inspection are defined below:

U4.1.1 Preparatory Inspection

The CQAO and QCM will perform preparatory inspections prior to beginning any work on any definable feature of work. During the preparatory phase inspections, the CM CQAO and CC QCM generally will:

- Review applicable sections of the contract documents, including the design drawings, RAWP, and Technical Specifications
- Review the Health and Safety Plan and the appropriate activity hazard analysis to ensure that safety requirements are planned
- Verify that all materials and/or equipment requiring submittal and/or testing under that definable feature and are ready to install have been tested, submitted, and approved
- Examine work area to ensure integral preceding work is in compliance so that subject work can proceed

- Physically examine materials that have arrived on the project site for compliance with submittals and contract requirements, if they have not previously been inspected
- Ensure CC is coordinating work processes with CC's subcontractors
- Check to ensure that the portion of the plan for the work to be performed has been approved/accepted by the EPA

The CQAO will document the preparatory phase inspections and associated observations.

U4.1.2 Initial Phase Inspection

The CM CQAO and CC QCM will perform an initial inspection as soon as a representative portion of the particular feature of work has been accomplished. Initial phase inspections generally will include:

- Review preparatory inspection notes
- Check preliminary work to ensure that it is in compliance with the contract documents
- Establish the level of workmanship required and verify that it meets minimum acceptable workmanship standards
- Resolve differences/conflicts
- Review the Health and Safety Plan and the appropriate activity hazard analysis to ensure that safety requirements are met
- Ensure that inspections and testing are being performed

The CQAO will document the initial phase inspections and associated observations.

U4.1.3 Follow-Up Phase Inspection

The CM FE and CC QCM will perform follow-up inspections daily to ensure:

- The work is in compliance with contract requirements
- An acceptable level of quality in workmanship is maintained
- That inspections, field measurements, and testing are being performed
- All "rework" items are being corrected
- Any deficiencies are corrected prior to the start of additional tasks, which may be affected by the deficient work

The CQAO will document the follow-up phase inspection activities and associated observations.

U4.1.4 Completion Inspection

The CM CQAO and CC QCM will perform a completion inspection of the work when major elements of the work are completed. The completion inspection will include:

- Develop a list of items that do not conform to the approved design plans and Technical Specifications
- Include the nonconformance list in the CC CQC documentation. Include the estimated date by which the deficiencies will be corrected
- Perform a second completion inspection after nonconformance items have been resolved and the CM CQAO has been notified by the CC QCM

The CQAO inspection documentation shall identify inspections conducted, results of inspections, location and nature of defects found, causes for rejection, and remedial or corrective action taken or proposed. When deficiencies are discovered during the four-phase or other inspection processes, focused inspections shall be considered by the CQAO. When material, performed work, or installation is found on the basis of a focused inspection to be deficient and/or does not meet the project design plans or Technical Specifications, the CQAO will assure deficiency correction is implemented, as discussed in Section U5.0.

U4.2 COMPLIANCE WITH TECHNICAL SPECIFICATIONS

U4.2.1 CQC Testing

The CC will establish a QC system to perform sufficient inspection and tests of all items of work, including that of their subcontractors, to ensure conformance to the functional performance of the project. The CC's QC system shall be established for all construction except where the contract documents provide for specific compliance tests by testing laboratories employed by the Owner. The CC's QC system shall specifically include all testing required by the various sections of the Specifications. The CQAO will review and approve the CC's QC system prior to beginning RA activities.

The CC shall be responsible for establishing a system of daily test reports that will record all CQC test results. Test results from each day's work period shall be submitted to the FE prior to the start of the next day's work period. The CC's responsible technician and the CC QCM shall

sign the daily test reports. The FE will review test results on a daily basis and identify any non-conforming test results for discussion with the CC regarding potential corrective action.

U4.2.2 CQA Monitoring

The CQAO and FE will perform ongoing CQA monitoring and oversight of CC's inspections and testing. The FE will use the inspection-and-test summary tables (included in the RAWP) in the field as a guide and checklist to document completion of the CQC activities for each task. In this manner, the inspections and tests required to measure compliance with the relevant portions of the RAWP and Technical Specifications are established and carried out.

The CQAO will verify the adequacy and effectiveness of the CC's CQC program through review of the FE's inspection documentation and review of the CQC test results. The CQA inspection frequency will be at the discretion of the CQAO based on results of CQC tests, evaluation of daily reports, and audits of the CQC program. Should information become available that indicates a potential problem, the CQAO will review in detail all pertinent information and require additional verification testing or re-work, if necessary.

U4.2.3 Construction Acceptance Criteria

The CQAO will document that the completed work meets the construction acceptance criteria for materials qualifications, inspection, and testing as established by Technical Specifications and as summarized in the inspection-and-test summary tables (included in the RAWP or as modified based on claims, change orders, amendments, etc.) Criteria for materials and equipment have been set by the Project Designer in accordance with the applicable codes and standards, and by manufacturers' recommendations. The CQAO will assemble necessary CC submittals to document conformance with acceptance criteria as detailed in the inspection-and-test summary tables (included in the RAWP).

U4.3 COMPLIANCE WITH HANDLING, STORAGE, PACKAGING, PRESERVATION, AND DELIVERY REQUIREMENTS

The CM will inspect the CC activities to ensure technical compliance in identification, handling, storage, packaging, preservation, and delivery of materials, parts, assemblies, and end products with either the Technical Specifications, manufacturers' recommendations, or generally accepted practices. Related quality records and documents will be maintained and controlled in accordance with the procedures provided in Section U6.0 of this CQAP.

U4.4 MATERIAL IDENTIFICATION AND MANAGEMENT

CM will monitor the CC to ensure that material identification and management requirements are met. Products and materials shall be traced from receipt through installation. Documentation such as project control checklists, material receipts, sample and test documentation, and reports will ensure that the applicable material/item is received and installed. Project Technical Specifications and/or procedures define product identification and management requirements, which generally include the following:

- Construction materials or equipment intended for project use are identified and segregated until inspection confirms that they conform to technical and quality requirements.
- Materials or equipment are traceable to documents attesting to their conformance with technical requirements that are stated in Technical Specifications or design drawings.

U5.0 CONSTRUCTION DEFICIENCIES

This section provides procedures for tracking construction deficiencies (noncompliance) from identification through acceptable corrective action. It defines the controls and related responsibilities and authorities for dealing with noncompliant products or services.

U5.1 DEFICIENCY DEFINITION

A deficiency occurs when a material, performed work, or installation does not meet the plans and/or Technical Specifications and/or intended use for the project. A deficiency can be identified by any CC or CM personnel.

U5.2 CC CQC DEFICIENCY IDENTIFICATION AND CONTROL

When material, performed work, or installation is found deficient, the CQAO (or designee) shall ensure that the non-conforming material, work, or installation is identified and controlled to prevent unintended use or delivery. The CQAO will notify the CC of any noncompliance with any of the contract requirements. The CC shall, after receipt of such notice, immediately take corrective action.

U5.2.1 Minor Deficiencies

Minor deficiencies are items that do not require significant rework or repair work to correct, and will not result in significant deviations from required construction standard(s) if corrected

immediately. Minor deficiencies noted by the CQAO or FE during testing or inspection are verbally reported to the CC's representative and noted on the Daily Construction Report form (see Attachment U-1).

Control and disposition of such deficiencies shall be by the originator of the Daily Construction Report form and the CC's supervisor responsible for the work; and do not require formal action by the CC's CQC System Manager or the CM. Ideally, such minor deficiencies can be corrected on the spot by agreement with the CC's supervisor. Failure by the CC to correct a minor deficiency after having been put on notice will result in a nonconformance (discussed below) if it is not corrected within five working days of notification.

U5.2.2 Nonconformances

Nonconformances are major deviations from the contract requirement and/or accepted construction standard which must be formally documented for corrective action by the CQAO or a testing group.

Nonconformances shall be formally documented by the CQAO on a Notice of Non-Compliance form (NNC; see example contained in Attachment U-1). The NNC is a formal notification to the CC that work does not meet the design plans or the Technical Specifications for the project. Nonconformance reports will be included on a nonconformance log and tracked through verification that the nonconformance has been corrected.

U5.3 CC CQC DEFICIENCY CORRECTION

When material, performed work, or installation is found to be deficient and/or does not meet the project Technical Specifications, the CQAO will assure deficiency correction is implemented. The CQAO shall ensure that the non-conforming material, work or installation is identified and controlled to prevent unintended use or delivery. The non-conforming material or work product shall be tagged and segregated by the CC, when practical, from conforming material or items to preclude their inadvertent use. The CQAO is responsible for documenting the nonconformance in an NNC.

The CC will implement corrective actions to remedy work that is not in accordance with the design drawings and Technical Specifications. The corrective actions will include removal and replacement of deficient work using methods approved by the CQAO. Removal must be done in a manner that does not disturb existing work and that meets CQA/CQC criteria; otherwise, the disturbed work must be removed and replaced. Replacement must be done in accordance

with the corresponding design plans, RAWP, and Technical Specifications. Replacement will be subjected to the same scope of CQA/CQC inspection and testing as the original work. If the replacement work is not in accordance with the design drawings and Technical Specifications, the replacement work will be removed, replaced, re-inspected, and re-tested.

U5.4 PREVENTATIVE ACTIONS

The CC and CQA/CQC team shall take preventative actions as necessary to eliminate the causes of potential deficiencies so as to prevent their occurrence. The CQAO will monitor, inspect, and audit processes used to prevent erroneous information or construction products from being passed to the Company. The CQAO has the authority to implement, verify, and review the project's preventative and corrective action effectiveness. He/she is empowered to improve the project's work processes to eliminate the causes of potential nonconformities.

U6.0 DOCUMENTATION

The CC's CQC documentation must cover all aspects of CQC program activities, and includes Daily Inspection Reports and Daily Test Reports. The CC will document the CQC activities pursuant to the contract documents. The CQAO and FE will document ongoing CQA oversight.

U6.1 DAILY RECORDKEEPING

Both the CC and CM personnel will manage project documents through a secure document filing and storage system. Sufficient records shall be prepared and maintained as work is performed to furnish documentary evidence of the quality of construction and laboratory analysis and of activities affecting quality. Each CC QC technician shall maintain a daily log of all inspections performed for both CC and subcontractor operations in a written report form (i.e., field logbook, Daily Construction Report form) acceptable to the CQAO. Typical information to be documented by the CC includes:

- Description or title of the inspection activity
- Location of the inspection activity or location from which the sample was obtained
- Recorded observation or test data
- Results of the inspection activity
- Personnel involved in the inspection activity
- Signature of the inspector

The CQC documentation shall be signed by the responsible QC Technician and the CC QCM. The CC QCM will provide to the FE at least one copy of each daily inspection and test report on the work day following the day of record.

U6.2 DAILY CONSTRUCTION REPORT

The FE will prepare and sign a daily construction report. The report will include a summary of the CC's daily construction activities. Supporting inspection data sheets will be attached to the daily report where needed. An example Daily Construction Report form is included in Attachment U-1.

The daily construction report typically will include the following information:

- Date, project name, location, and other identification
- Description of weather conditions
- Personnel, subcontractors and visitors
- Equipment used
- Summary of construction activities
- CQA and CQC activities (including inspections and test results)
- Discussions with Company and CCs
- Potential causes for delays or change orders
- Photo log

The daily construction reports will be filed on a daily basis in the project CQA/CQC files and will be maintained by the CQAO as part of the permanent project record.

U6.3 RECORD DRAWINGS

The CC will be responsible for red-lining design drawings in the field as preparation for Record Drawings. The red-lined design drawings will document actual field conditions upon completion of the work. Where there was a change to a specified material, dimension, location, or other feature, the red-lined design drawings will indicate the work performed.

The FE working with the CC will be responsible for assuring that red-line design drawings are maintained daily throughout the construction process. These red-lined design drawings will be used to update the design drawings to Record Drawings at the completion of the work.

The CC will submit draft red-line design drawings to the CQAO for review and will prepare final red-line design drawings based on CM-staff comments. The CQAO will provide the red-lined design drawings to the Project Designer who will incorporate the red lines and issue the final Record Drawings to the Company.

U6.4 CONTROL OF QUALITY RECORDS

The CC will provide an electronic or paper copy (suitable for scanning) of CQC documentation associated with the work to the CQAO within five working days of the generation of such documents. The CC shall maintain a fire-resistant file box at the project site for the duration of the field activities. The CC's documentation shall contain all inspection reports, test records, contract documents, project, and daily field reports. The CQAO verifies CQA record accuracy and maintains copies of all quality-related documentation in the permanent project files at the CQAO's home office.

U7.0 CHANGE MANAGEMENT

U7.1 ENGINEERING CHANGE NOTICES

Any changes required to approved Final Designs shall be documented with an Engineering Change Notice (ECN). ECNs shall be prepared and submitted to EPA with weekly reports, unless otherwise directed by EPA. The Company shall specifically identify in the weekly construction reports ECNs which constitute a significant deviation from the approved RAWP or which may affect the project schedule. Such ECNs require EPA review and approval. ECNs shall not impact achievement of the performance standards. ECNs may be required as a result of the Adaptive Management activities described in the RAWP.

U7.2 CQAP CHANGES

The Company, Supervising Contractor, or the CQAO may initiate revisions to this CQAP. The CQAP may be revised when it becomes apparent that the CQAP procedures or controls are inadequate to support work being produced in conformance with the specified quality requirements, or are deemed to be more excessive than required to support work being produced in conformance with the specified quality requirements. Changes to CQA procedures necessitating modification to this CQAP will be submitted for EPA review and approval. Updates to CQAP staffing will be made by the Company with notification to EPA without submission of a fully revised CQAP.

U7.3 CC QUALITY CONTROL SYSTEM CHANGES

The CC QC system may require revisions as necessary to correct unsatisfactory performance. At any time, the CQAO, Company, or Supervising Contractor, may require the CC to make changes to their QC system, including personnel changes, as necessary to obtain the quality specified. Moreover, the CC may initiate system changes to correct CQC process problems, and is required to notify the CQAO in writing of any desired changes; all changes are subject to the Company and CQAO acceptance.

U8.0 REFERENCES

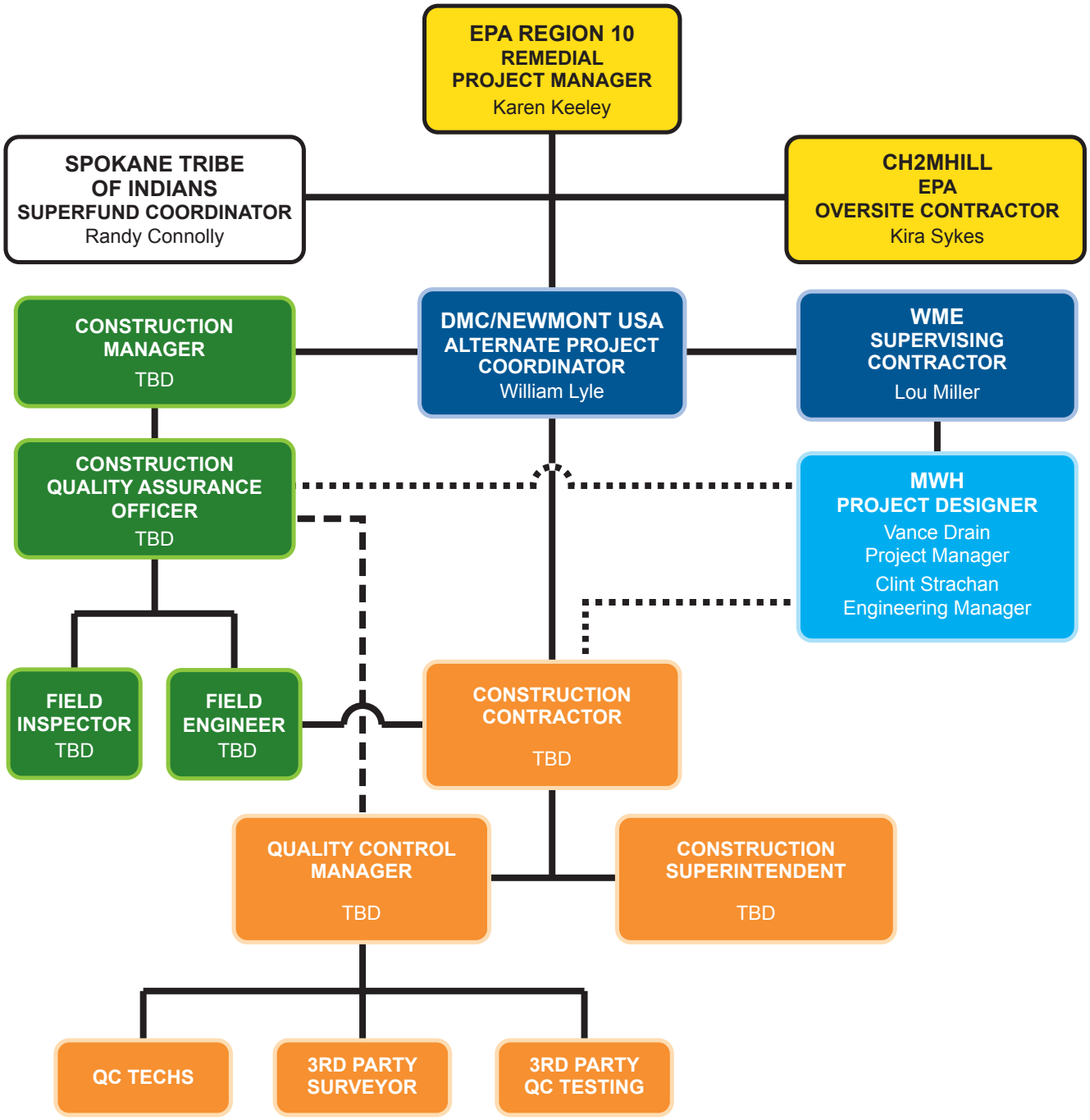
U.S. Army Corps of Engineers (USACE), 1995. Construction Quality Management. Regulation No. ER 1180-1-6. September 30.

U.S. Environmental Protection Agency (EPA), 1987. Technical Guidance Document – Construction Quality Assurance for Hazardous Waste Land Disposal Facilities. Hazardous Waste Engineering Research Laboratory, Cincinnati, Ohio. EPA/530-(S)SW-86-031. February.

U.S. Environmental Protection Agency (EPA), 2006. Midnite Mine Superfund Site Spokane Indian Reservation, Washington Record of Decision. Prepared by the Office of Environmental Cleanup, EPA Region 10. September.

U.S. Environmental Protection Agency (EPA), 2011. Consent Decree Statement of Work for the Remedial Action for the Midnite Mine Superfund Site, Spokane Indian Reservation, Washington. Civil Action No. CV-05-020-JLQ. United States of America, Plaintiff v. Dawn Mining Company, LLC and Newmont USA Limited, Defendants. August.

FIGURE



Administrative and functional interface

Direct communication and coordination related to construction QA/QC

Direct communication and coordination related to technical issues

MWH

Midnite Mine

FIGURE U-1

REMEDIAL ACTION ORGANIZATIONAL CHART

DRAWN BY D. Severson

4 June 2015

Ussic1s01\ifo\Industrial Projects\MIDNITE MINE\Deliverables_Working Documents\Basis of Design Rpt\Appendix U - CQAP\60% Submittal\FIGURES\Fig U-1_Midnite Mine_Remedial Action Org Chart_June2015

Attachment U-1

Example Forms

Daily Construction Report

Notice of Non-Compliance Form

Daily Construction Report

Date _____

PROJECT: Midnite Mine Superfund Site

JOB NO: _____

CLIENT: Newmont USA and Dawn Mining LLC

CONTRACTOR: _____

PROJECT MANAGER: _____

Weather		
Temp. °F		Report No.
Wind		

Personnel, Subcontractors, and Visitors			
Time	Name	Representing	Remarks

Equipment Used:
Construction Activities:
Quality Assurance/Quality Control Activities (including field calibrations):
Health and Safety Levels and Activities:
Preparatory Control Issues:
Initial and Follow-up Control Activities:
Summary of Inspections and Test Results:
Conversations with Client:
Conversations with Contractors:
Potential Causes for Delays or Change Orders:

Daily Construction Report (continued)

Photos:

Distribution:
1. Proj. Mgr.
2. Field Office
3. File
4. Client

Page 1 of X Pages

By _____ Title _____

NOTICE OF NON-COMPLIANCE (NNC)

Date: _____ NNC No. _____ 1

To: _____ 2

Company Name: From: _____ 3

Address CQAO: _____ 4

City, State, Zip Project no: _____ 5

Phone Project name: _____ 6

Date Deficiencies Corrected and 7

NNC Closed: _____ 8

(See line 18 below)

Deficiencies and Corrective Actions: 9

Outstanding Items: 10

Attachments: 11

CQAO Signature: _____ Date: _____ 12

SUBCONTRACTOR'S COMPLIANCE

Corrective Action: 13

Outstanding Items: 14

Attachments: 15

Date Deficiencies Corrected: _____ 16

Subcontractor's Signature: _____ 17

Name: _____ 18

To be completed by CQAO Only:

Date Deficiencies Corrected and

NNC Closed: _____ CQAO Signature: _____ 18

(Write this date on line 8 above)

Midnite Mine Superfund Site
100 Percent Design

Appendix V – Procurement Plan

Revised October 2015

Prepared for:

Dawn Mining Company
PO Box 250
Ford, Washington 990413

and

Newmont USA Limited
6363 South Fiddler's Green Circle
Greenwood Village, Colorado 80111

Prepared By:

MWH Americas, Inc.
2890 E. Cottonwood Parkway, Suite 300
Salt Lake City, UT 84121

TABLE OF CONTENTS

V1.0 INTRODUCTION 1

V2.0 CONTRACTING STRATEGY 1

V2.1 BIDDER'S INTEREST CAMPAIGN AND PRE-QUALIFICATION OF BIDDERS ..3

V2.2 REQUEST FOR PROPOSALS 4

V3.0 GENERAL CONTRACT REQUIREMENTS 6

V4.0 DESCRIPTION OF MIDNITE REQUESTS FOR PROPOSAL 6

V4.1 MINE WASTE EXCAVATION AND CONTAINMENT 7

V4.2 WATER COLLECTION AND TREATMENT 8

V4.3 TREATED EFFLUENT WATER CONVEYANCE LINE 8

V4.4 GROUNDWATER CONTROLS 8

V4.5 CONSTRUCTION MANAGEMENT 9

V5.0 CERTIFICATIONS, QUALIFICATIONS, AND TRAINING REQUIREMENTS 9

V6.0 TRIBAL EMPLOYMENT RIGHTS ORDINANCE 10

ATTACHMENTS

Attachment V-1 Spokane Tribe of Indians TERO Compliance Plan

Attachment V-2 Newmont USA Limited Procurement Policies

LIST OF ACRONYMS

BMP	Best Management Practice
BODR	Basis of Design Report
CM	Construction Manager
CQAP	Construction Quality Assurance Plan
DMC	Dawn Mining Company, LLC
EHS	Environmental Health and Safety
EMR	Worker's Compensation Experienced Modification Rating
EPA	U.S. Environmental Protection Agency
FFP	Firm-Fixed Price
FUP	Fixed Unit Price
HSWRP	Hillside Waste Rock Pile
MSHA	Mine Safety and Health Administration
Newmont	Newmont USA Limited
O&M	Operation and Maintenance
OSHA	Occupational Safety and Health Administration
QA	Quality Assurance
RA	Remedial Action
RAWP	Remedial Action Work Plan
RD	Remedial Design
RFI	Request for Information
RFP	Request for Proposal
Site	Midnite Mine Superfund Site
SOW	Scope of Work
SWPPP	Stormwater Pollution Prevention Plan
TERO	Spokane Tribe of Indians Tribal Employment Rights Ordinance
T&M	Time & Material
WTP	Water Treatment Plant

V1.0 INTRODUCTION

This Procurement Plan presents the process for procuring goods and services related to the remedial actions (RAs) at the Midnite Mine Superfund Site (Site). This Procurement Plan is an appendix to the *Midnite Mine Basis of Design Report* (BODR), which presents the background and supporting information relevant to the Site and the planned RAs. The BODR also contains the engineering plans and specifications that will be used to solicit bids for products and services.

V2.0 CONTRACTING STRATEGY

There are a number of important principles that apply to the procurement of goods and services for the RA at the Site. Bidders must demonstrate a history of similar project experience, exemplary safety performance, effective management controls, and skilled workmanship as prerequisites in order to be added to the list of Bidders. Bidders will be equitably treated in a manner consistent with high ethical standards.

It is anticipated that the Selected Remedy will be bid in phases conforming to the construction phases discussed in Appendix D and other key design appendices. Under this scenario, Newmont will first solicit bids for the Phase 1 work activities in accordance with DMC/Newmont's Procurement Policies (included in Attachment V-2). The bidders will be provided the Midnite Mine Remedial Action Work Plan (RAWP), final design documents and specifications, and asked to provide costs for the initial phase of work.

The major Phase 1 work activities include:

1. Construction of the Site Access Road
2. Clearing, grading, and installation of the Construction Support Zone facilities
3. Initial BMP placement as outlined by the Construction Stormwater Pollution Protection Plan (SWPPP)
4. Initial processing of the Hillside Waste Rock Pile (HSWRP)
5. Dewatering of Pit 4
6. Pit 4 scaling and other rockfall measures
7. Pit 4 sediment cleanup/dewatering and sump construction

8. Construction and conveyance of water from the Pit 4 dewatering wells
9. Polishing of water as necessary to be used for dust suppression in uncontaminated areas
10. Backfilling of Pit 4
11. Hauling and placement of borrow material, and revegetation of borrow area once Pit 4 is completely backfilled (at the end of Phase 1)
12. Capping and revegetation of the Pit 4 cover

DMC/Newmont will evaluate the selected RA (or Prime/General) Contractor's performance toward the end of the Phase 1 construction period and, based on that evaluation, either re-solicit bids or continue with the selected RA Contractor into Phase 2 of the RA.

The selected RA Contractor will perform the construction activities either by self-performing the construction tasks or by subcontracting specific portions of the Phase 1 earthworks to specialty contractors (e.g., the crushing and screening portion of the HSWRP). The selected RA Contractor will be required to have all subcontracts comply with the contracting terms and conditions, certifications, qualifications and training requirements, etc. that are included in the prime contract with DMC/Newmont.

The predominant means by which contracts will be awarded include the following:

- Comparable project management and work experience
- Technical approach and project scheduling

The general types of contracts considered for use, in order of preference for this project, are "Firm-Fixed Price" (FFP), "Fixed Unit Price" (FUP), and "Time & Material" (T&M). Where assigned-agreements (sole source) are necessary, the price, terms, and conditions will be negotiated to the mutual benefit of all parties. Relevant prime contract terms and conditions will flow down to subcontracts.

A Construction Manager (CM) contract will be awarded to a qualified firm that is independent from the RA Contractor, or DMC/Newmont may provide a qualified CM. The CM will oversee the RA Contractor and perform quality assurance (QA) duties in accordance with the Construction Quality Assurance Plan (CQAP).

V2.1 BIDDER'S INTEREST CAMPAIGN AND PRE-QUALIFICATION OF BIDDERS

DMC/Newmont will identify a list of Tribal and non-Tribal RA contractors. These contractors will receive a Request for Information (RFI). Responses to this RFI will be used as a screening tool to identify a smaller group of pre-qualified contractors that will receive the request for proposal (RFP) to perform the RA. An evaluation system will be established to determine the acceptable level of qualifications to bid as a Prime Contractor or perform in a support role to the Prime Contractor.

Candidate RA contractors will be notified and pre-qualified as follows:

1. DMC/Newmont (or their designated representative) will contact via certified-mail, and/or email, all Spokane TERO-listed contractors and non-tribal contractors and request the firms respond to a RFI questionnaire that includes necessary qualifications and experience as listed immediately below.
2. The RFI will seek responses to the following topics. The prospective RA contractors:
    - Native American business status (for compliance with TERO).
    - Technical capabilities and core competencies. Experience and performance on similar large construction, Superfund or Tribal projects, including client references.
    - Safety record, including Occupational Safety and Health Administration (OSHA) 300 Logs, OSHA Incident and Severity Rate, and Worker's Compensation Experienced Modification Ratings (EMR).
    - Construction safety plans, policies, and procedures and their compliance with applicable laws and regulations.
    - Workplace Drug-Free policy and procedures.
    - Workplace discrimination policies and procedures.
    - Financial stability and resources necessary for the successful completion of the project. Resources evaluated include those related to personnel, equipment, and operating revenue.
    - Bank and credit references.
    - Payment history of subcontractors and suppliers.

- Bond and insurance capacity.
- Business and professional licensing and registration, as applicable to bidder's trade specialty.

3. DMC/Newmont will screen the prospective Contractor's RFI responses. The Tribal TERO representatives also will be given the opportunity to review and provide input on the responses. Based on these evaluations, DMC/Newmont will select a list of prospective Tribal and non-Tribal contractors that will receive a Request for Proposal (RFP) as discussed in Section V2.2 below.

V2.2 REQUEST FOR PROPOSALS

For the Midnite RA project, the relevant design elements have been progressed to the 100% level to allow for a clear scope of work (SOW) prior to seeking bids. Following the RFI process discussed in Section V2.1, DMC/Newmont will assemble the Phase 1 procurement package (also known as the RFP). The Phase 1 RFP package will contain the following information for the prospective Contractors to consider and respond to:

- A description of the Phase 1 activities that will be necessary to complete (i.e., a SOW) along with pertinent 100% design drawings, specifications and the RAWP. The prospective Contractors will be required to detail their technical approach in performing the Phase 1 SOW and include a list of any exceptions they may have to the RFP documents.
- The RFP will state how bids will be evaluated, and what constitutes a responsive bid. Refer to sections 9 (Receipt and Opening of Bids), 10 (Bid Evaluation Procedures) and 11 (Recommendations and Purchase Order Award) in Attachment V-2 for general discussions of each of these topics as they relate to the contractor's proposal and DMC/Newmont's response to their submittal.
- A requirement to present other similar projects that the contractors have executed and references to determine how the contractor performed. In addition, the contractors will have to discuss in detail the size of their company, their current work load, and how they plan to accomplish the Phase 1 work based on their other commitments.
- Resumes of key project personnel and their degrees / training
- Information on all key subcontractors that are proposed.

- List of equipment that will be used for the project and whether it is leased or owned.
- Provide details on the contractor's and key subcontractors Health and Safety Program including:
  a. A requirement to identify the company's Environmental Health and Safety (EHS) Representative / Competent Person responsible for this project
  b. A list Interstate Experience Modification Rating (EMR) for each of the last 3 years. Include a separate verification letter of the EMR data from the Insurance Carrier or Broker.
  c. Provide the following injury information for the past 3 calendar years:

YEAR	FATALITIES	# OF OSHA RECORDABLE INJURIES	OSHA RECORDABLE RATE	LOST TIME RATE

  d. Any OSHA or MSHA "Serious or Willful" Citations in the past 3 years? Yes/No
  e. A description of work-related fatalities in the past 5 years. If any, provide the year of occurrence, circumstances and description of any post-fatality changes in Health and Safety Program or practices.
  f. Attach a copy of the TABLE OF CONTENTS of the current EHS Management System Process.
  g. All of the above information and documentation for all proposed subcontractors.
- Licenses: List all applicable licenses the contractors have or will acquire (including descriptions, numbers and expiration dates) to lawfully conduct the Work.
- Bonding capacity of the company.
- A proposed Construction Schedule given the Phase 1 SOW activities.
- An estimate of labor, equipment, and other direct costs that will be incurred in completing the Phase 1 SOW. DMC/Newmont will provide a bid schedule for the contractors to prepare their estimates. Any assumptions the contractors use in preparing pricing should also be included with their cost estimate.
- Any exceptions the prospective contractors might have to the DMC/Newmont's contract terms and conditions.

The DMC/Newmont RFP also will clearly identify the contractors' proposal due date, cutoff for submission of questions, site walk information, the anticipated date of award, etc. DMC/Newmont will keep a record of prospective RA contractors' questions on the RFP during the proposal preparation phase so that all the perspective contractors can review DMC/Newmont's responses during preparation of their cost proposal. DMC/Newmont realizes there is some redundancy between the information that the prospective RA Contractors will provide during the RFI and RFP process but this information is considered critical in initial and final selection of the RA contractor.

V3.0 GENERAL CONTRACT REQUIREMENTS

The RA Contractor(s) and CM will be required to perform work in accordance with the EPA-approved Final RD and specifications, the RAWP and the CQAP. The RAWP and the CQAP describe how each element/component of the Selected Remedy will be addressed during the construction activities, identify specific tasks necessary to complete the Remedy, and provide an overall management and quality control/assurance strategy for completion of all such tasks. The RAWP will include a project schedule for each major activity and submission of deliverables required during construction of the Remedy.

The RA Contractor and CM will participate in a pre-construction conference prior to each construction season, as well as regular meetings with EPA to discuss the Remedy construction as it progresses. The RA Contractor and CM will provide full and complete access to EPA (or their designated representatives) for periodic scheduled inspections intended to assure that the construction activities are proceeding or have been completed in substantial compliance with the approved Final RD and RAWP/CQAP. Should specific work activities be determined non-compliant, DMC/Newmont will require the RA Contractor(s) to take necessary steps to correct deficiencies and/or bring the construction into compliance with the approved Final RD and RAWP/CQAP.

V4.0 DESCRIPTION OF MIDNITE REQUESTS FOR PROPOSAL

Work (or RFP) packages will be developed that align with the following RA Work Elements:

- Mine Waste Excavation and Containment – broken into phases 1, 2, and 3 of mine waste excavation and containment including backfilling, capping, revegetation and all the associated construction activities that support completion of these construction phases

- Water Collection and Treatment – permanent pumps, influent lines, and WTP construction
- Treated Water Conveyance Line – effluent pipeline construction
- Groundwater Controls – dewatering trenches in the Far Eastern, Central and Western Drainages
- Construction Management – quality assurance throughout the RA.

Because the RAWP, design documents, and technical specifications are not specifically broken out per these delivery elements, there will be important clarifications required within for example, the Early Works/Phase 1 SOW to clearly identify what work elements are part of each RFP, and what the interface work elements are with other phases of construction.

At the RFP level, the work will be broken down into primary bid items to manage the detailed costs of subcontract placement, administration, field oversight, and execution. The individual RFP packages will include the scope items discussed below. One or more of these RFPs may be combined depending on the capabilities and performance of the selected RA Contractor in the Phase 1 Mine Waste Excavation and Containment procurement. For example, the selected Phase 1 RA Contractor might be able to implement the RFP discussed in V4.4 below. In that case, the contractor would be asked to provide anticipated costs for the section V4.4 RFP without going through the entire bid process with other contractors.

V4.1 MINE WASTE EXCAVATION AND CONTAINMENT

The Mine Waste Containment RFPs will be broken into three phases (including tasks to be completed in each phase) to follow the approach as described in Appendix D of the BODR. Overall, the Mine Waste Excavation and Containment will include the following SOW items:

- Health and Safety Program (includes preparing the Site Safety and Health Plan to cover all activities performed by the contractor)
- Mobilization/construction support zone facility layout and staging areas
- Surface water and sediment controls
- Storage ponds and water conveyance lines (including temporary conveyance lines from the groundwater controls)

- Construction of permanent influent lines and pumps. Phase 1 will include construction of the permanent influent lines from the Pit 4 cover to the new WTP and Phase 3 will include construction of the permanent influent lines from the Pit 3 cover to the new WTP.
- Demolition of existing Water Treatment Plant (WTP)
- Mine waste excavation
- Mine waste containment including backfilling and capping
- Site revegetation

V4.2 WATER COLLECTION AND TREATMENT

The Water Collection and Treatment Work Package will include the following SOW items:

- Health and Safety Program (includes preparing the Site Safety and Health Plan to cover all activities performed by the contractor)
- Mobilization/facility layout and staging areas
- Construction of new Water Treatment Plant
- Prepare technical and O&M manuals

V4.3 TREATED EFFLUENT WATER CONVEYANCE LINE

The Treated Water (Effluent) Conveyance Line RFP will include the following SOW items:

- Health and Safety Program (includes preparing the Site Safety and Health Plan to cover all activities performed by the contractor)
- Mobilization/facility layout and staging areas
- Construction of effluent conveyance line from new WTP to Lake Roosevelt
- Fabrication and installation of the diffuser in Lake Roosevelt

V4.4 GROUNDWATER CONTROLS

The Groundwater Controls RFP will include the following SOW items:

- Health and Safety Program (includes preparing the Site Safety and Health Plan to cover all activities performed by the contractor)

- Mobilization/facility layout and staging areas
- Construction of groundwater extraction trenches and low-permeability barriers
- Installation of new wells/piezometers for performance monitoring

V4.5 CONSTRUCTION MANAGEMENT

The CM Work Package will include the following SOW items:

- Construction oversight
- Construction QA in accordance with the CQAP.

The Construction Management QA functions will be conducted by a contractor/consultant that is independent from the Construction Contractor.

V5.0 CERTIFICATIONS, QUALIFICATIONS, AND TRAINING REQUIREMENTS

The following certifications, qualifications and training are the minimum requirements that will be required to perform the Midnite Mine RA construction activities. These requirements will be included in the RAWP and RFPs. As listed in Section V2.1 requirements will include:

- Resumes of selected project personnel and their degrees / training
- Details on the contractor's Health and Safety Program including:
  a. A requirement to identify the company's EHS Representative / Competent Person responsible for this project.
  b. A list Interstate EMR for each of the last 3 years. Include a separate verification letter of the EMR data from your Insurance Carrier or Broker.
  c. Provide the following injury information for the past 3 calendar years:

YEAR	FATALITIES	# OF OSHA RECORDABLE INJURIES	OSHA RECORDABLE RATE	LOST TIME RATE

  d. Any OSHA or MSHA "Serious or Willful" Citations in the past 3 years? Yes/No
  e. A description of work-related fatalities in the past 5 years. If any, provide the year of occurrence, circumstances and description of any post-fatality changes in Health and Safety Program or practices.

f. Attach a copy of the TABLE OF CONTENTS of the current EHS Management System Process.

g. All of the above information and documentation for all proposed subcontractors.

- Licenses: List all applicable licenses the contractors have or will acquire (including descriptions, numbers and expiration dates) to lawfully conduct the Work.

V6.0 TRIBAL EMPLOYMENT RIGHTS ORDINANCE

The Site is located on the Spokane Tribe of Indians Reservation; therefore, all RA work will be conducted in accordance with the Spokane Tribe of Indians TERO. These requirements include submitting to the Spokane TERO Office a Compliance Plan documenting how the RA Contractor (and all lower-tier subcontractors) will meet their obligations and responsibilities under TERO before commencing work on the Spokane Indian Reservation. A copy of the Spokane TERO Office Compliance Plan is included as Attachment V-1 to this Procurement Plan.

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Attachment V-1

Spokane Tribe of Indians TERO Compliance Plan

The Spokane Tribe of Indians does hereby enact the following:

SPOKANE TRIBAL EMPLOYMENT RIGHTS ORDINANCE

SECTION 1.0 NAME

This Ordinance shall be known as the "Spokane Tribal Employment Rights Ordinance".

1.1 PURPOSE
The purpose of this Ordinance is to assist in and require the fair employment of Indians on or near the Spokane Indian Reservation and to prevent discrimination against Indians in the employment practices of Reservation employers through the creation of the Tribal Employment Rights Commission and the establishment of rules, regulations and policies governing its responsibility and authority to insure Reservation-wide compliance with this Ordinance.

SECTION 2.0 DEFINITION OF IMPORTANT TERMS AND WORDS

2.1 AGENT(S)
Any employee or individual authorized to act on behalf of the Commission.

2.2 BUSINESS FOR PROFIT
Shall mean any business enterprise or operation which is not considered a non-profit or not-for-profit organization by the IRS.

2.3 CHAIRPERSON
Shall mean the Chairperson of the Spokane Tribal Employment Rights Commission

2.4 COMISSION
Shall mean the Spokane Tribal Employment Rights Commission.

2.5 COMMISSIONER
Shall mean a Commissioner of the Spokane Tribal Employment Rights Commission.

2.6 CORE CREW
Shall mean key personnel required by the employer who are regular, permanent employees and are in a supervisory or other key position; only the first and thereafter every seventh employee may be employed as key personnel and/or core crew by an employer.

2.7 COUNCIL
Shall mean the Business Council of the Spokane Tribe of Indians.

2.8 DIRECT FEDERAL CONTRACTS
A direct Federal Contract is a contract let by the Federal agency directly to a prime contractor.

2.9 DIRECTOR
Shall mean the Director of the Spokane Tribal Employment Rights Program.

2.10 EEOC
Shall mean the Equal Employment Opportunity Commission of the United States.

2.11 EMPLOYER
Shall mean any person, company, contractor, subcontractor or other entity located or engaged in work on the Spokane Reservation. However, the Spokane Tribe, the State of Washington and the Federal Government along with any subdivision are excluded from the definition of employer and not subject to the Tribal Employment Rights Ordinance.

2.12 ENGAGED IN WORK ON THE RESERVATION
An employer is "engaged in work on the Reservation" if during any portion of conducting business for a business enterprise or working on a specific project, contract or subcontract, he or any of his employees spend a majority of his (their) time performing work within the exterior boundaries of the Reservation on a continuing basis.

2.13 FEDERALLY FUNDED CONTRACTS
A Federally funded contract is one in which the Federal Government has contracted or granted funds to an entity which, in turn, lets the prime contract.

2.14 FOR THE BENEFIT OF INDIANS
Work to be performed under a Federal or Federally funded contract is for the benefit of Indians if the benefits provided to Indians are in addition to or incidental to any benefits which might occur to the general public.

2.15 HRC
Shall mean the Human Rights Commission of the State of Washington.

2.16 INDIAN

Shall mean any person recognized as an Indian by the United States pursuant to its trust responsibilities to American Indians.

2.17 INDIAN CONTRACTOR OR SUBCONTRACTOR
Contractor or Sub-Contractor that is 51% or more Indian owned and controlled.

2.18 INDIAN OWNED BUSINESS
Shall mean a business entity of which at least 51% is actively owned, operated and managed by Indians.

2.19 INDIAN PREFERENCE APPLICANT
Shall mean any person recognized as one of the following in the stated order, except any order may be followed on Federal and State projects:

a) Enrolled member of the Spokane Tribe of Indians.
b) Child or an enrolled member of the Spokane Tribe of Indians.
c) Spouse of an enrolled member of the Spokane Tribe of Indians.
d) Enrolled member of a recognize tribe of Indians.

2.20 LOCAL INDIAN OR LOCAL INDIAN OWNED BUSINESS
Is any Indian Preference applicant or Indian owned business having social and economic ties to the Spokane Tribe of Indians with primary place of residence or business on the Spokane Reservation which is owned by a Spokane Tribal member living on or near the Reservation.

2.21 LOCATED NEAR THE RESERVATION
Near shall mean any employer located within daily commuting distance of the Reservation.

2.22 LOCATED ON THE RESERVATION
An employer is located on the Reservation if during any p0ortion of a business enterprise or specific project, contract or sub-contract, he maintains a temporary or permanent office or facility within the exterior boundaries of the Reservation.

2.23 OFCCP
Shall mean the Office of Federal Contract Compliance Programs of the United States.

2.24 PRIME CONTRACTOR
A prime contractor is the prime construction, forestry or mining contractor responsible for construction or mining projects.

2.25 RESERVATION
Shall mean the Spokane Indian Reservation, Washington and includes all land, Indian and non-Indian, within the exterior boundaries of the Spokane Indian Reservation and any trust lands under jurisdiction of the Spokane Tribe, wherever they are located.

2.26 SECRETARY
Shall mean the Secretary of the Interior or his duly authorized representatives.

2.27 SUBCONTRACT
A subcontract is any contract let by a prime contractor to its subcontractors for supplies or work on prime contracts regardless of tier.

2.28 TRIBAL MEMBER
Shall mean any person who is a duly enrolled member of the Spokane Tribe of Indians.

2.29 TRIBE
Shall mean the Spokane Tribe of Indians.

SECTION 3.0 SPOKANE TRIBAL EMPLOYMENT RIGHTS COMMISSION

3.1 APPOINTMENT
The Spokane Tribal Employment Rights Commission shall be comprised of five members and two alternates appointed by the Spokane Tribal Business Council.

3.2 TERMS OF OFFICE
The members of the Commission shall be appointed for a term of three years. To ensure continuity, the Tribal Council shall appoint two members plus two alternates for a three-year term, two members for a two-year term and one for a one-year term. The Council shall fill each vacancy by appointing a new member or reinstating the member whose term is expiring. An alternate shall replace any member who is removed or for some other reason cannot fulfill his term. An alternate shall also sit in place of any member of the commission absent whose presence is required to create a quorum.

3.3 REMOVAL FROM OFFICE
A Commissioner may be removed by the Tribal Council only for good cause shown after notice and hearing by the Council.

3.4 VACANCY AND INTERIM APPOINTMENT

If a Commissioner shall die, resign, by incapacitated or be removed from office, a vacancy on the Commission shall be created automatically, and the unexpired term shall be filled by the Alternate and in ***such a*** case where 2 or more vacancies are created, Council members shall be appointed to serve the unexpired terms.

3.5 CHAIRPERSON

The Chairperson of the Commission shall be elected by the members of the Commission on an annual basis. The chairperson shall preside at all formal and informal meetings and hearings of the full Commission.

3.6 DUTIES OF THE COMMISSION

The Commission or its authoriz3ed representatives shall administer the Employment Rights Program of the Spokane Tribe of Indians in Accordance with this Ordinance and shall adopt by-laws under which it shall operate internally.

3.7 POWERS OF THE COMMISSION

The Commission or its authorized representatives shall have the power:

a) To make recommendations to the Tribal Council when hiring and firing Commission employees and to pay salaries pursuant to a salary schedule established by the Council.

b) To establish rules and regulations governing all activities of the Commission and the Director consistent with this Ordinance and as approved by the Tribal Council.

c) To make recommendations to the Tribal Council on amendments to the Ordinance after public participation and comments are reviewed by the Commission in accordance with the procedures established by the Commission.

d) To expend funds appropriated by the Council for the Spokane Tribal Employment Rights Program and funds collected from employers as provided herein.

e) To obtain funding from Federal, State or other sources to supplement Council appropriations.

f) To establish numerical hiring goals and timetables specifying the minimum number of Indians an employer must hire by craft or skill level.

g) To require employers to establish or participate in job training programs as the Commission or the Director deems

necessary to increase the pool of Indians eligible for employment on the Reservation.

h) To establish and administer the Tribal Hiring Hall and require employers to use the Hiring Hall.

i) To prohibit employers from using job qualifications criteria or personnel requirements that may bar Indians from employment unless such criteria or requirements are bona fide and necessary occupational qualifications for employment. Commission regulations may adopt EEOC guidelines or may adopt additional requirements to eliminate employment barriers unique to Indians and the Reservation.

j) To enter into agreements with the unions to insure union compliance with this Ordinance.

k) To require employers to give preference to Tribal and other Indian-owned businesses in the award of contracts and subcontracts.

l) To establish counseling programs to assist Indians in obtaining and retaining employment.

m) To hold hearings and to subpoena witnesses and documents in accordance with this Ordinance.

n) To require employers to submit reports and take all action deemed necessary by the Commission or the Director for fair and vigorous implementation of this Ordinance.

o) To enter into cooperative agreements with Federal Employment Rights agencies such as EEOC and OFCCP to eliminate discrimination against Indians both on and off the Reservation as well as to enter into agreements with the Council for the same purposes.

p) To take such other actions consistent with this Ordinance as are necessary to achieve the purpose and objectives of the Spokane Employment Rights Program established in this Ordinance.

q) The Commission including any of its agents, employees or delegates, shall retain all rights and privileges of Sovereign Immunity of the Spokane Tribe of Indians.

3.8 DIRECTIVE TO THE COMMISSION

a) In establishing and maintaining the numerical goals provided for in Paragraph (g) and the requirements for training programs as provided for in the first sentence of Section 4, Paragraph 4.4, The Commission, the Director and staff, whenever practical, shall consult with the affected employers so as to consider their input and recommendations.

b) The Commission may hold such formal and informal meetings, and regulate the times and procedures thereof as it may deem necessary in order to carry out effectively its duties and powers under this Ordinance.

c) The Commission shall attempt whenever possible to administer this Ordinance and execute its powers hereunder by a consensus approach. If a consensus cannot be3 achieved, the affirmative vote of at least three Commissioners shall be required to render effective any decision or action of the Commission, and the Chairperson shall be entitled to vote on any decision or action.

d) All written agreements or plans, directives, orders, complaints, and appeals which the Commission is authorized or required to issue or file hereunder shall bear the signature of at least two Commissioners.

3.9 APPOINTMENT AND REMOVAL OF EMPLOYMENT RIGHTS DIRECTOR

The Council shall appoint the Director to serve on a full-time basis as the staff director for the Commission. The Director may be removed by the Council for good cause shown after notice and hearing by the Council. The Council may consider Commission recommendations concerning appointment and removal of the Director.

3.10 DUTIES AND POWERS OF THE DIRECTOR

a) Subject to the supervision and direction of the Commission, the Director shall exercise all those duties and powers under this Ordinance which may be delegated to the Director by the Commission, provided, however that any decision which the Commission is authorized or required to make hereunder, including the issuance or filing or the written materials listed in Section 3.8 hereof, shall not be so delegated. Routine or administrative correspondence not involving any decision making powers of the Commission may be delegated by it to the Director.

b) As soon as practicable after their appointment, the initial Commissioners shall define by rule or regulation the specific powers and duties hereunder which the Director shall exercise. Pending the appointment of the initial

Commissioners, the Council may act as it deems necessary to exercise the duties and powers conferred by this Ordinance upon the Commission, including delegation of interim authority by the Council to the Director.

SECTION 4.0 SPOKANE EMPLOYMENT RIGHTS PROGRAM

4.1 COVERAGE AND SCOPE

a) All employers are required to give preference to local Indians in hiring, promotion, training and all other aspects of employment, contracting or subcontracting, must comply with this ordinance and the Rules, Regulations and orders of the Commission. The foregoing shall apply to all facilities of an employer including a subcontractor located or engaged in work on the Reservation.

b) The foregoing requirements shall apply only to an employer located or engaged in work while on the Reservation. When an employer has previously agreed in a contract, lease or other document to give preference to Indians, this Ordinance and authorized Commission Rules, Regulations, and directives shall define the specific minimum obligations of the employer pursuant to such written agreement.

4.2 CONTRACTORS AND SUBCONTRACTORS

The Indian Preference requirements contained in this Ordinance shall be binding on all contractors and sub-contractors of employers, regardless of tier, and shall be deemed part of all contract and sub-contract specifications. The employer shall have the initial and primary responsibility for insuring all contractors and sub-contractors comply with these requirements. The employer as well as any of his contractors or sub-contractors shall be subject to penalties provided herein for any violation of this Ordinance if the contractor or sub-contractor fails to comply

4.3 MINIMUM NUMERICAL GOALS AND TIMETABLES FOR INDIAN EMPLOYEMENT

a) The Commission or the Director may establish the minimum number of Indians each employer must employ on his work force during any year that he or any of his employees are engaged in work on the Reservation. Numerical goals may be set for each craft, skill, job classification, etc., used by the employer and shall include, but not be limited to administrative supervisory and professional categories. The goals shall be expressed in terms of man hours of Indian employment as a percentage of the total man hours worked by the employer’s work force in the job classification involved.

b) For both new and existing employers, the goal shall be reviewed by the Commission at least annually and shall be revised as necessary to reflect changes in number of Indians available or changes in employer hiring plans.

c) Each employer shall submit a monthly report to the Director indicating the number of Indians in his work force, how close he is to meeting his goals, all persons hired or fired during the last month, the job positions involved, and other information required by the Commission.

4.4 TRAINING

Employers may be required by the Commission to participate in training programs to assist Indians to become qualified in the various job classifications used by the employer. The Commission shall set the ratio of Indian trainees to fully qualified workers after consultation with the Director and employers.

4.5 TRAINING ADMINISTRATIVE FEE

Every building trade's employer that is a prime contractor on the Reservation of $10,000.00 or more shall pay a training fee of 3% (three percent) of the total contract dollar amount. This revenue shall be used for Journeyman upgrade training and/or Apprenticeship Trainee.

4.6 JOB QUALIFICATION AND PERSONNEL REQUIREMENTS

Employers are prohibited from using job qualification criteria or personnel requirements which bar Indians from employment unless such criteria or requirements are required by business necessity and are bona fide occupational qualifications for employment as listed in the Dictionary of Occupational Titles established by the Commission, or unless they are unemployable for other valid reasons.

a) Employers who employ more than two employees may designate key personnel who are regular, permanent employees in a supervisory or other key position. Only the first and thereafter every seventh employee may be employed as key personnel and core crew by an employer.

b) At no time shall core crew and/or key personnel displace Indian Preference Employee(s) and/or potential Indian Preference Employee(s) by performing work outside their normal classification.

4.7 TRIBAL HIRING HALL

The Commission will cause to be established and administer a Tribal Hiring Hall to assist the Commission and employers in placing Indians in job positions. An employer may recruit and hire workers from whatever process he chooses, as long as he

complies with this Ordinance, and the Federal Equal opportunity Act, in hiring practices, Indian job preference regulations and agreements pertaining to his operation.

4.8 COUNSEILNG AND SUPPORT PROGRAMS

The Commission shall cause to be established counseling and other support programs to assist Indians to obtain and retain employment:

a) Every employer shall be required to cooperate with the Commission or the Director regarding such counseling and support programs.

b) Every employer must provide opportunity to any employee to enter any such program for counseling prior to termination unless the employee was terminated for good cause a defined herein.

c) The Commission is authorized to develop a Local Indian Business Support Program to promote, facilitate and encourage the success of local Indian owned Businesses. It is authorized to develop and set criteria allowing for the reimbursement of certain administrative fees to said businesses with contracts of $200,000 or less. The Commission shall develop and adhere to specific standards providing for equal treatment of those seeking such reimbursements.

4.9 LAYOFFS AND/OR TERMINATIONS

a) In making any layoffs and reductions in force, all employers shall maintain required ratios of Indian Preference Employees. Furthermore, no Employer shall terminate or layoff any Indian Preference person who is employed pursuant to this Ordinance without good cause for such termination. Good cause for termination shall be defined as: "Failure to adequately perform the job, failure to follow orders, any illegal or fraudulent act pertaining to employment or any act which puts the employer or other employees in an unsafe position in the course of employment."

b) The employer in determining layoffs shall apply the layoffs in a reasonable and fair fashion. Furthermore layoffs should only occur when there is not adequate work to keep all employees on the job or when the employer has a cash flow shortage necessitating reduction in the work force.

4.10 PROMOTION

Every employer shall in accordance with required ratios give Indians preferential consideration for all promotion opportunities and shall encourage Indians to seek such opportunities.

4.11 SUMMER STUDENTS

Employers shall give Indian students preferential consideration for summer student employment. The Employer shall make every effort to promote after-school, summer and vacation employment for Indian students.

4.12 INDIAN PREFERENCE REQUIREMENTS IN AWARDING PRIME CONTRACTS AND SUB-CONRACTS

a) Employers shall give preference in the award of contracts to tribally owned and "Local Indian-owned" businesses. These rules and regulations apply to all construction, forestry and mining that will take place on or near the Spokane Indian Reservation. A contract or sub-contract taking place "on or near the Spokane Indian Reservation" means 50% of the unfinished products or by-products comes from within Reservation boundaries. The Indian contract preference requirements set out herein shall apply to:

   i. All contracts and sub-contracts which are not Direct Federal or Federally funded contracts; and

   ii. All sub-contracts or Direct Federal contracts which are for the benefit of Indians and which are covered by Section 7(b) of the Indian Self Determination Act, P.L. 93-638, 25 U.S.C. 45(e) (b).

In granting such preference, the following order shall be adhered to:

   i. Local Indian-owned Businesses.

   ii. Other Indian-owned Businesses.

b) Any Business or Indian-owned Business may bid as a supplier, prime contractor or subcontractor on the Reservation, but shall not submit bids for a sub-contract or to be supplier if bidding on the prime contract and shall sign a non—collusive statement for each contract.

c) The Tribal employment Rights Office shall maintain a list of tribally-owned, locally Indian-owned and Indian-owned businesses which shall be supplied to the employers for their use. On all contracts of $75,000 or over, employers shall provide an approved INDIAN CONTRACTING UTILIZATION PLAN which

shall conform to the Indian Preference Contracting/sub-contracting rules and regulations contained in this Ordinance and must provide documentation to:

i. How Indian and local Indian Preference Bids were solicited; and

ii. Who was awarded the contract; and

iii. Why each contractor was awarded said contract.

d) On all contracts $75,000 or over, no employer may commence work on the Spokane Reservation until it has submitted a Commission-approved INDIAN CONTRACTING UTILIZATION PLAN setting forth how the employer intends to meet the requirements when awarding prime contracts and sub-contracts.

e) Employers on or near the Spokane Reservation shall comply with these requirements when awarding prime contracts and sub-contracts.

i. If the entity asking for bids has reason to believe that two or more qualified local Indian firms will bid, then the Invitation for bids shall be restricted to qualified local Indian-owned enterprises and Indian Organization.

ii. If there are less than two local Indian bidders then, the Invitation for Bids shall be open competition to Indian and non-Indian firms alike.

f) In the case of (i) above, the Lowest Qualified Local Indian Preference bidder shall be awarded the Contract or Sub-contract, if within budgetary limits of the project or within the fixed percentage as outlined below. In the case of (ii) above, the Lowest Indian bidder shall be awarded if:

i. The lowest responsible Indian Preference bidder is within budgetary limits established for the specific project or component of the project, and;

ii. The lowest responsible Indian Preference bidder is not more than a fixed percentage higher than the total bid price of the lowest responsive bid from any qualified bidder. The fixed percentage is determined as follows:

When the lowest responsive bid is At LEAST	but LESS than	PERCENTAGE OF BID OR DOLLAR AMOUNT, WHICH EVER IS LOWEST
$000,000	$100,000	10% or $ 9,000.
$100,000	$200,000	9% or $16,000.
$200,000	$300,000	8% or $21,000.
$300,000	$400,000	7% or $24,000.
$400,000	$500,000	6% or $25,000.
$500,000	$1 million	5% or $40,000.
$1 million	$2 million	4% or $60,000.
$2 million	$4 million	3% or $80,000.
$4 million	$7 million	2% or $105,000.
$7 million or more responsive responsive		1% of lowest bid, w/no $ amt

g) Any contractor or sub-contractor failing to provide an Indian Contracting Utilization Plan shall be deemed a violation of this Ordinance and subject to the sanctions as provided for in Section 7.

4.13 BURDEN OR PROOF

In any hearing before the Commission where the issue is employer compliance with any of the requirements or provisions of the foregoing subsections of Section 4, the burden of proof shall be on the employer to show compliance within said subsection.

SECTION 5.0 COMPLIANCE PLANS

Every employer of two or more employees and every contractor or sub-contractor obtaining a contract or subcontract of $10,000 or more dollars on the Reservation shall establish and abide by a hiring and employment Compliance Plan approved by the Director. Such plan shall set forth how the employer will meet the goal of 100% Indian employment in each job classification and other obligations set out herein. Final

approval of the commission shall be obtained by the Director within 30 days of his initial approval.

No employer as identified above may commence work unless it has submitted a Commission-approved Compliance Plan setting forth how the employer intends to meet the Compliance Provision hereunder.

5.1 COMPLIANCE PLAN FOR NEW EMPLOYERS

a) Employers who shall locate or engage in work on-Reservation after the effective date of this Ordinance, Compliance plans shall establish hiring and employment goals for the entire work force of that employer.

b) Each new employer shall meet with the Director as soon as possible prior to actually beginning work and shall furnish the Director with a precise list of the number and kinds of employees he expects to employ. They shall conclude a Compliance Plan after considering any special factors or circumstances the employer wishes to present.

c) Every plan shall establish the goal of 100% Indian employment in each job classification if:

   i. Qualified Indians are reasonably available when the Plan is adopted to fill all job positions anticipated by the new employer in the job classification; or

   ii. Qualified Indians can reasonably be expected to become available from training programs or other sources to fill all such positions during the one year period of the Plan.

d) If qualified Indians are not available, then Plans may provide for goals of less than 100% Indian employment in affected job classifications.

5.2 COMPLIANCE PLANS FOR EXISTING EMPLOYERS

a) Employers who have located or engaged in work on-Reservation prior to the effective date of this Ordinance, Compliance Plans shall establish hiring and employment goals for new employment goals for new employees expected to be employed during the one-year period of any Plan.

b) After due notice, each existing employer shall meet with the Director, as required, and shall furnish a reasonably specific list of the number and kinds of new employees he expects to employ in the ensuing year. The Director and the employer shall conclude a Compliance Plan after considering

any special factors or circumstances the employer wishes to present.

c) Compliance Plans for existing employers shall establish the goal of 100% Indian employment for new hiring in each job classification if qualified Indians can reasonably be expected to become available to fill such new positions during the duration of the Plan. If qualified Indians are not thus available, then Plans may provide for goals of less than 100% Indian employment in affected job classifications.

5.3 REVISION OF COMPLIANCE PLANS; MONTHLY EMPLOYER REPORTS

a) Each employer who has agreed to a Compliance Plan shall submit a monthly report to the Director, or other report as otherwise directed by the Commission, on a form provided indicating:

i. The number of Indians in his work force;

ii. How close he is to meeting his goals;

iii. The number of persons hired or terminated during the month and affected job positions; and

iv. Any other information specified by the Commission and reasonably necessary in monitoring the employer's efforts to abide by the Compliance Plan.

b) The Director shall meet at least annually with each new and existing employer to review the goals of every Compliance Plan. Any Plan may be revised at any time, after meetings between the Director and employer, when necessary to reflect changes in the number of qualified Indians available or changes in employer hiring practices.

5.4 FINAL APPROVAL OF COMPLIANCE PLANS

Each Compliance Plan or revision thereof shall be approved in writing by at least two members of the Commission prior to said Plan being effective.

5.5 NON-COMPLIANCE OR VIOLATIONS

If any employer fails or refuses to comply with the Compliance Plan requirements herein or with the terms of this Compliance Plan as writing, such non-compliance shall be deemed a violation of this Ordinance and subject to sanctions provided for ion Section 7 of this Ordinance.

5.6 COMPLIANCE BY UNIONS

Every union with a collective bargaining agreement with an employer must file a written agreement stating the union will comply with this Ordinance and the rules, regulations and orders of the Commission. Until such agreement is filed with the Director and approved by the Commission, the employers thereunder may not commence work on the Reservation.

a) Contents of Union Agreements
Every union agreement with an employer must be field with the Director and must provide:

i. Indian Preference
The union will give preference to Indians in job referrals regardless of which union referral list they are on.

ii. Cooperation with the Commission
The union will cooperate with the Director in all aspects and assist in the compliance and enforcement of this ordinance and related regulations and agreements.

iii. Registration
The union will establish a mechanism allowing Indians to register for job referral lists by telephone or mail.

iv. Training Programs
The union will establish journeyman upgrade and advance apprenticeship programs.

v. Temporary Work Permits
The union will grant temporary work permits to Indians who do not with to join the union.

b) Memorandum of Agreement
The Director will provide a memorandum of agreement for use by all unions who have collective bargaining agreements with any employer.

c) Recognition of Unions
Nothing herein or any activity by the Commission or the Director authorized hereby shall constitute official Tribal recognition of any union or Tribal endorsement of any union activities on the Reservation.

d) Burden of Proof
In any hearing before the Commission where the issue is compliance by an employer with any of the requirements and provisions of the foregoing subsections of Section 5, the burden of proof shall be on the employer rather than on the employee or other complainant to show compliance with said provisions.

SECTION 6.0 COMPLAINTS AND HEARINGS

6.1 NOTICE

If a hearing is requested by the Commission, the Director, an individual, an employer, or a union pursuant to this section, the Commission shall cause a written notice of hearing to be served upon all concerned parties stating the nature of the hearing and the evidence to be presented. The notice shall advise such parties of the violation, their required presence or the presence of a representative on their behalf, their right to be present at the hearing, to present testimony of witnesses or other evidence, and to be advised by counsel at their own expense.

6.2 COMPLAINTS

a) Complaints Regarding Violations

Any individual, employer, entity or organization that believes any covered employer or its agents has violated or is in violation of this Ordinance or any Rules or Regulations issued pursuant to it may file a complaint with the Director, a Commissioner, or the Commission's authorized agent. The complaint shall be in writing and shall provide such information as is necessary to enable the Director to carry out an investigation. The Director shall investigate every complaint filed. If upon investigation, he has reason to believe a violation has occurred, he shall proceed pursuant to the provisions of Section 7. Within 20 days after receipt of the complaint, and on a regular basis thereafter, the Director shall provide the complaining party with a written report on the status of the complaint.

b) Time and Place of Filing Complaints

Complaints filed under subsection (a) above shall be filed by the charging party with the Commission or its authorized agent within seven working days after the alleged unlawful violation(s) occurred or seven working days after the charging party learned of the alleged unlawful violation(s), but no complaint shall be accepted after 180 days from the date of alleged violation or occurrence. The complaint shall include the date, place and circumstances of the alleged unlawful violation(s) and shall be served upon the person(s) against whom the charge(s) are made within 10 days thereafter.

c) Complaints Regarding Administrative Acts of TERO Director

Any individual employer, employee or other party may challenge any action of the Commission or the Director by filing a written complaint with the Director, the Commission, or its authorized agent within 5 days from said action and

requesting a hearing no later than 10 days from the date said complaint was filed

d) Service of Complaints
Service may be made by registered mail or hand delivery with receipt.

6.3 INVESTIGATIONS
On its own initiative or pursuant to a complaint, the Commission or Director shall make such public or private investigation within the Spokane Reservation as the Director or the Commission deems appropriate and necessary to determine whether any provision of this Ordinance or any rules, regulations or orders hereunder have been violated.

a) The Director or his duly authorized delegate may enter, during business hours, the place of business or employment of any employer he deems necessary to monitor compliance with the requirements of this Ordinance or any Rule or orders hereunder.

b) The Director or his delegate shall bear and show official identification adequate to identify them as the authorized agents of the Commission to make such investigation.

6.4 HEARING PROCEDURE
Hearing shall be governed by the following rules of procedure:

a) All parties may present testimony of witnesses and other evidence and may be represented by counsel at their own expense.

b) The Commission may have the advice and assistance of counsel provided by the Tribe.

c) The chairperson of the Commission, or the Vice-Chairperson, shall preside and the Commission shall proceed to ascertain the facts in a reasonable and orderly fashion.

d) The hearing may be adjourned, postponed and continued at the discretion of the Commission.

e) At the final close of the hearing, the Commission may take immediate action or take the matter under advisement.

f) The Commission shall notify all parties in writing 30 days after the hearing of its decision in the matter.

g) If any party fails to appear for a hearing then it may be ruled as forfeiture by default.

6.5 POWER TO REQUIRE TESTIMONY AND PRODUCTION OF RECORDS

For the purpose of investigations or hearings which, in the opinion of the Director or the Commission are necessary and proper for the enforcement of this Ordinance, the Commission or the Director, or any designee may administer oaths or affirmation, subpoena witnesses, take evidence, and require, by citation, the production of books, papers, contracts, agreements or other documents, records or information which the Director or the Commission deems relevant or material to the inquiry.

6.6 PRE-HEARING PROCEDURES

a) Review of TERO Files

The Respondent (the employer or entity against whom a charge has been filed) shall have the right to review the case file of the Director by scheduling an appointment with him for that purpose during regular working hours at any point after receiving notice of a hearing. The Director shall remove any portion of the file to protect what is deemed confidential.

b) List of Witnesses

Ten days prior to the hearing (or as soon as possible if the hearing is to be held within 10 days after notice), the Respondent and the Director shall submit to the Commission Chairperson a list of witnesses each intends to call at the hearing, the approximate length of their testimony, and the general substance of said testimony. It shall indicate any witnesses that must be subpoenaed. The Commission shall then cause necessary subpoenas to be issued.

c) Expert Witnesses

Any party who intends to present testimony of an expert on their behalf shall within 15 days of hearing provide the Commission and the opposing party the name, qualifications, and the substance of said expert testimony. Failure to do so shall disqualify such testimony from hearing.

d) Pre-hearing Interview of Witnesses

The Respondent and the Director shall have the right to interview the witnesses of the other party prior to hearing. The director’s witnesses shall be interviewed in the presence of the Director or his delegate. The Respondent’s witness shall be interviewed under such reasonable conditions as are established by the Respondent. Either party may appeal to the Chairperson of the Commission if cooperation is not

forthcoming on this matter. The Chairperson is empowered to require such steps as are necessary to resolve the problem.

e) Production of Documents and Things
The Parties shall provide each other with a list of documents, papers and tangible evidence intended to be introduced at hearing, including a statement regarding the substance and relevance of each. Said documents, papers and tangible evidence shall be made available to one another forthwith, no later than 10 days prior to the hearing or within two days if the hearing date is noticed less than 10 days before the hearing. Failure to produce such list and the documents listed within the time prescribed may render said documents inadmissible at hearing on motion of opposing party in the Commission discretion.

f) Postponements
Any request for a postponement of the hearing must be submitted in writing to the Chairperson of the Commission no fewer than three days prior to the hearing. However, if the Director and Respondent mutually submit a request for a postponement because there is a possibility of settling the matter, the request for a postponement may be submitted at any time prior to hearing.

6.7 CONDUCT OF THE HEARING

a) Presiding Official
As presiding official, the Chairperson or a designated Commissioner of the Tribal Employment Rights Commission will control the proceedings. He or she will take whatever action is necessary to insure an equitable, orderly, and expeditious hearing. Parties will abide by the presiding official's rulings. The presiding official has the authority to:

i. Administer oaths or affirmations;
ii. Regulate the course of the hearing;
iii. Rule on offers of proof;
iv. Limit the number of witnesses when testimony would be unduly repetitious; and
v. Exclude any person from the hearing for conduct or misbehavior that obstructs the hearing.

b) Director
The director may represent the Tribal Employment Rights Commission on all charges filed by it, even if the charge was initiated by a complaint filed by a private individual.

c) Respondent
The Respondent shall be preset for the entire hearing and he or his representative (other than an attorney) shall represent him during the proceeding.

d) Attorneys
Either party may have an attorney present as an advisor. However, the attorney may not make any presentations, cross-examine witnesses or address the Commission.

e) Recording of the Hearing
All hearings shall be recorded in full and the Commission shall retain the tape(s) for no less than one year after the hearing. The Respondent shall also be permitted tape the hearing upon request.

f) Prohibition Against Reprisals
All parties shall have a right to testify on their own behalf, without fear of reprisal.

g) Starting Time
The hearing shall be opened promptly at the time specified by the Commission.

h) Opening Statements
Both parties will be afforded the opportunity to present opening statements with respect to what they intend to prove at the hearing.

i) Order of Proceeding
The complainant or his representative will present his case first.

j) Examination and Cross Examination of Witnesses
Both parties may subpoena and examine friendly and hostile witnesses. Both parties may examine and cross examine witnesses. However, no harassment or efforts to intimidate witnesses shall be permitted. The commission members may examine witnesses at any point in their testimony. The testimony of all witnesses shall be under oath or affirmation.

k) Irrelevant Testimony
Parties may object to clearly irrelevant material, but technical objections to testimony as used in a court of law will not be entertained. The Commission shall prohibit any testimony that it deems clearly irrelevant in order to keep control of the hearing.

l) Written Testimony
Evidence or exhibits may be presented. Written testimony will be admitted into evidence during the hearing only when a witness cannot appear in person. When a party wishes to use the written testimony of a witness who cannot appear, the party must submit, in advance of the hearing, a written explanation for the nonappearance of the witness to the Tribal Employment Rights Commission. If the Commission is satisfied with the explanation, the party will obtain the testimony by means of deposition. When, for reasons satisfactory to the Tribal Employment Rights Commission, a deposition cannot be used, an affidavit or a sworn statement from the witness may be used. A signed but unsworn, statement will not be admitted in evidence.

m) Closing Statement
Closing statements for each party will be permitted. The Complainant shall proceed first.

n) Audience
The hearing shall be open to the public. However, the Commission may remove any person who disrupts the hearing or behaves in an inappropriate manner.

6.8 THE DECISION
The decision shall be in writing and issued within 30 days after the hearing. The decision shall consist of the following parts, in the following order.

a) The facts as found to be true by the Commission;

b) The finding of violation or no violation on each charge along with the legal and factual basis for the finding;

c) The orders and or sanctions imposed, if any;

d) Information on rights to appeal;

e) Information on the authority of the Commission to act if the party fails to comply with its orders or fails to appeal; and

f) The injunctive or bonding requirements, if any, that the Commission will seek from the court pending the completion of the appeal if an appeal is filed.

6.9 THE RECORD
The Commission shall include in the record copies of all documents and descriptions of information used in arriving at the decision

and shall maintain and store said record for a period of one year from the date of the decision.

6.10 FINALITY OF THE DECISION - APPEAL

Decisions of the Commission shall be effective immediately and final when the time for filing a Notice of Appeal has expired. Appeal may be taken by an aggrieved party, to the Spokane Tribal Court as provided in Section 8.0.

The Commission's decision is final and shall be in writing. It shall be served on the charged party by registered mail or in person no later than 30 days after the close of the hearing. Should the party fail to comply immediately with the Commission's orders, the Commission shall request the Tribal Court, and the Tribal Court shall grant, such injunctive relief as necessary to preserve the rights of the beneficiaries of this Ordinance, pending the party's appeal or expiration of the time for appeal.

SECTION 7.0 ENFORCEMENT AND PENALTIES FOR VIOLATIONS

7.1 ENFORCEMENT AND PENALTIES FOR VIOLATIONS

The Director is authorized to police and enforce this Ordinance and the Rules and Regulations of the Commission. He may issue citations to violators and initiate investigations for the purpose of settling disputed violations or formally enforcing this Ordinance and the Rules and Regulations of the Commission as follows:

a) Informal Settlement

If upon investigation initiated by the filing of a complaint pursuant to Section 6.2 hereof or on his own initiative, the Director has reason to believe a violation has occurred; the Director shall issue written notice of violation to the alleged violator. The Director and the employer shall seek to achieve an informal settlement of the alleged violation immediately. If no settlement can reasonably be made, he shall issue a formal Citation and Notice of Non-Compliance.

b) Formal Citation and Notice of Non-Compliance

The formal Citation and Notice of Non-Compliance shall set out the nature of the alleged violation and the steps that must be taken to come into compliance. The violator shall be provided no more than five days to comply with said Notice of Non-Compliance or request in writing a hearing before the Commission on the matter. This time may be shortened in the Director's discretion should he feel irreparable harm will occur should the violation continue.

If the violator refuses to comply with requirements within the time imposed by the Director and has not requested a hearing before the Commission on the matter pursuant to Section 6.2(c) the Director may seek to impose one or more penalties below upon said violator. After being fully advised on the matter, the Commission may issue an order imposing such penalties it deems necessary.

Said order shall constitute a final decision of the Commission and can be appealed only pursuant to Section 8.0 below.

7.2 ENFORCEMENT

Any employer, contractor, sub-contractor or union who is found in violation of this Ordinance or Rules, Regulations or Orders of the Commission or the Director shall be subject to the following penalties for such violations:

7.3 PENALTIES

a) Denial of right to commence or continue business inside the Reservation.

b) Suspension of operations inside the Reservation.

c) Payment of back pay and damages to compensate any injured party.

d) An order to summarily remove employees hired in violation of this Ordinance or rules, regulations or orders of the Commission or the Director.

e) Imposition of monetary civil penalties.

f) Prohibition from engaging in future operations on the reservation.

g) An order requiring changes in procedures and policies necessary to eliminate the violation(s).

h) An order requiring employment, promotion and training of Indians injured by the violation.

i) An order making any other provision deemed by the Commission necessary to alleviate, eliminate, and compensate for any violation(s).

j) Imposition of a one-time 10% penalty fee on all amounts due on monetary judgments ordered by the Commission if not paid by the employer within 30 days of the initial judgment. If

said judgments are to paid within 60 days of the initial billing, the employer will be assessed a one-time 30% penalty fee in addition to the 10% referred to above. After 60 days, interest will accrue at the rate of 1% per month on any unpaid balance.

k) If enforcement and collection action becomes necessary for any Commission order, the Respondent shall pay all attorney fees and costs incurred in such proceeding.

The maximum penalty which may be imposed is $5000.00 for each violation. Each day during which a violation exists shall constitute a separate violation.

SECTION 8.0 APPEALS

8.1 RIGHT TO APPEAL

Any party to a hearing shall have the right to appeal any decision of the Commission to the Spokane Tribal Court by filing a Notice of Appeal with the said Court within 20 days of the Final Decision of the Commission.

8.2 SCOPE OF REVIEW

The Tribal Court shall uphold the decision of the Commission unless it is demonstrated that the decision of the Commission is arbitrary, capricious, unsupported by the weight of the evidence or in excess of the authority of the commission.

8.3 METHOD OF APPEAL

The appeal shall be taken by serving a written Notice of Appeal with the Tribal Court, with a copy to the Director within 20 days after the date of the entry of the order. The Notice of Appeal shall:

a) Set forth the order from which appeal is taken;

b) Specify the grounds upon which reversal or modification or order is sought; and

c) Be signed by appellant.

8.4 ABATEMENT OF ORDER ON APPEAL

Except as provided below, the order of the Commission shall not abate pending the outcome of the appeal in Tribal Court. If the order of the Commission is reversed or modified, the Court shall by its mandate specifically direct the Commission as to further action in the matter, including making and entering any order or orders in connection therewith and the limitations or conditions to be contained therein.

8.5 BOND ON APPEAL

The Director may petition and, for good cause shown, the Court may order the party requesting a hearing to post a bond sufficient to cover monetary damages that the Commission previously assessed against the party or some other amount to assure the party's compliance with other sanctions or remedial actions imposed by the Commission's order if that order is upheld by the Court.

8.6 SECURITY FOR COMPLIANCE

If the Commission, in its discretion, has reason to believe a party will remove itself or its property from the jurisdiction of the Commission or the Tribal Court during any stage of an enforcement action, the Commission may petition the Tribal Court pursuant to the rules and procedures of the Court to attach such property as necessary to ensure payment of any fees or damages owed or to secure compliance or for such other relief as is necessary and appropriate to protect the rights of the Commission and other affected parties.

SECTION 9.0 CONFISCATION AND SALE

9.1 PURPOSE OF PROCEDURE

a) If a party as has failed to pay monetary damages imposed on it or failed to otherwise comply with an order of the Commission within 21 days after its decision and no appeal has been filed, the Commission may petition the court to order the Tribal police to confiscate, and hold for sale, such property of the party as is necessary to ensure payment of said monetary damages or to otherwise achieve compliance.

b) Said petition shall be accompanied by a list of property belonging to the party which the Commission ahs reason to believe is within the jurisdiction of the Tribal Court, the value of which approximates the amount of monetary damages at issue. If the Court finds the petition to be valid, it shall order the Tribal police to confiscate and hold said property. The Tribal police shall deliver in person or by certified mail a notice to the party informing it of the confiscation and of it right to redeem said property by complying with the order outstanding against it.

c) If, 30 days after confiscation, the party has not come into compliance, the Court shall order the police to sell said property, the proceeds of which shall be used to pay any outstanding fees and damages and to pay any costs incurred by the Court and police in the confiscation and sale.

d) Any sale made pursuant hereto shall be made in a commercially reasonable manner.

SECTION 11.0 COMMISSION FUNDS/FEE WAIVERS

a) All fees collected pursuant to this Chapter shall be deposited into the Tribe's General Fund.

b) The Tribe reserves the right to waive any fees required under this Chapter for contracts under which the Tribe is a party.

SECTION 12.0 EMPLOYMENT ADMINSITRATIVE FEE

The Director shall assess employers on Employment Administrative Fee to proved revenue for the operation of the Tribal Employment Rights Office as follows:

12.1 CONTRACTS

Every prime contractor obtaining a contract of $10,000.00 or more shall pay an employment Administrative Fee equal to 2% (two percent) of the total amount of each contract on the Reservation.

12.2 OTHER EMPLOYERS

Every employer, other than the contractor with 20 or more employees shall pay an Employment Administrative fee of 2% (two percent) of the Employers annual Payroll. This fee shall be paid in quarterly installments and shall not be required of Education, Health, or Nonprofit Employers.

12.3 ACCOUNTING

Employment Administrative Fees shall be paid to the Spokane Tribe of Indians and placed in a Special Account for use by the Tribal Employment Rights Office. The Tribal Employment Rights Office is authorized to establish such rules and regulations as are necessary to assure fair and timely fee collection process. Any employer or contractor who fails to pay the required Employment Administrative Fee shall be subject to sanctions provided for in Section 7 of this Ordinance.

SECTION 13.0 EQUAL EMPLOYMENT OPPORTUNITES

No employer shall discriminate against any Indian Preference employee of applicant for employment because of color, religious, sex, national origin or age and must assure advertisements include equal opportunity for access.

SECTION 14.0 WAGE AND HOUR STANDARDS

14.1 EQUAL PAY
Every employer shall be required to provide equal pay to Indian Preference Employees performing work similar or comparable to other employees.

14.2 RATE OF PAY
Every employer shall be required to pay whichever rate of pay is highest when Federal, State, or Tribal wage, rates and guidelines are used.

14.3 FEDERAL, STATE, AND LOCAL AGENCIES
The Commission or the Director may use Federal, State, or Tribal agencies in resolving a discrepancy concerning wages and hours worked.

Any employer who is in violation to the above paragraphs shall be subject to the penalties outlined in Section 7.

SECTION 15.0 HEALTH, WELFARE, AND PENSION FUND BENEFITS

Payroll deductions shall be permissible for health, welfare, and pension funds benefits, but any payroll deduction constituting a contribution on behalf of the person employed to any fund established by the employer, employees or both, for the purpose of providing medical or hospital care, pensions, annuities, retirement, death benefits, compensation for injuries, illness, accidents, sickness or disability, or which are applied to payment of insurance to provide any of the foregoing, including unemployment benefits, vacation pay, savings accounts, or similar payments for the benefit of employees, their families and dependents shall be permissible, PROVIDED HOWEVER, the following standards must be met:

a) The deduction is not otherwise prohibited by law;

b) It is either:

   i. Voluntarily consented to by the employee in writing and in advance of the period in which the work is to be done and

such consent is not a condition either for obtaining or for the continuation of employment, or

ii. Provided for in a bona fide collective bargaining agreement between the contractor or sub-contractor and representatives of its employees;

c) No profit or other benefit is otherwise obtained directly or indirectly, by the contractor or subcontractor or any affiliated person in the form of commission, dividend, or otherwise;

d) The deduction shall serve the convenience and best interest of the employee.

SECTION 16.0 SAFETY,HEALTH AND ENVIRONMENTAL PROTECTION

Contracts of $75,000 or greater, and any project or contract work the Commission determines may have an impact on the environment, shall provide a written plan with the Director providing for the protection of health and environment of employees and other persons and prevent damage to property, materials, equipment and the physical environment of the Spokane Indian Reservation. Any employer contractor who fails to provide such plan or failure to follow or carry out such plan shall be deemed a violation of this Ordinance and is subject to penalties outlined in Section 7.

SECTION 17.0 RETALIATION OR REPRISAL

Any harassment, discrimination or threat against any person or business which has filed a charge, opposed any unlawful employment practice, or testified, assisted or participated in any manner in an investigation, proceeding or hearing involving an unlawful employment practice is a violation of the Spokane Employment Rights Ordinance and said violation shall be subject to the penalties outlined in Section 7.

SECTION 18.0 QUALIFIED PRIME CONTRACTORS, SUBCONTRACTORS OR SPECIALTY CONTRACTOR LICENSES

All prime contractors, qualified subcontractors or specialty contractors in the building trades must acquire a Spokane Tribal Contractors license to operate within the boundaries of the Spokane Reservation. The applicant is required to provide proof of an operational record with a minimum of two successful consecutive years of contracting or subcontracting in a specified component or field or the applicant must provide proof that they are journeymen employees of a subcontracting firm and worked as a journeymen for a minimum of five years in a specified trade before a license may be issued. A fee of $100.00* (One hundred Dollars) will be charged for said license. (*Subject to change without notice)

The application must be renewed on a yearly basis, expiring one year from the day said license was purchased, and failure to renew or obtain a Spokane Tribal Contractor License shall be a violation of this Ordinance and is subject to sanctions outlined in Section 7

SECTION 19.0 PUBLICATION OF ORDINANCE

The Commission or the Director shall post a public notice for all amendments to this Ordinance. It shall be the employers' obligation to make a written request for a copy of said Ordinance or amendments. All bid announcements issued by any Tribal, Federal, State or other private or public entity shall contain a statement that the successful bidder will be obligated to comply with this Ordinance and all rules, regulations and orders of the Commission.

19.1 LICENSING AND BUSINESS PERMITS

All Tribal agencies responsible for issuing business permits for Reservation activities or otherwise engaged in activities involving contact with prospective employers on the Reservation shall be responsible for advising such prospective employers of their obligations under this Ordinance and all rules, regulations and orders of the Commission.

Copies of the Ordinance are available for Tribal members from the TERO Office upon written request.

SECTION 20.0 EFFECTIVE DATE

This Ordinance shall be effective from the date of its approval by the Spokane Tribal Business Council.

SECTION 21.0 RULES AND REGULATIONS

The Commission may from time to time adopt detailed rules, regulations, policies and guidelines consistent with and necessary for full implementation of this Ordinance.

SECTION 22.0 SEVERABILITY

If any provision of this Ordinance or its application to any person or circumstances is held invalid, the remainder of the Ordinance, or the application of the provision to other persons or circumstances is not affected.

Attachment V-2

Newmont USA Limited Procurement Policies

NEWMONT USA LIMITED

PROCUREMENT POLICIES

TABLE OF CONTENTS

1.0 INTRODUCTION 1
1.1 General 1
1.2 Governing Procurement Policy 1

2.0 PROCUREMENT PLANNING 1
2.1 Preliminary Activities 1
2.1.1 SAP Procurement Software Tool 1
2.1.2 Procurement Planning 1
2.1.3 Project Team Participation 2
2.2 Planning Approach 2

3.0 SERVICE AND SUPPLY SOURCING PROCEDURES 3
3.1 RFx Tender Package 3
3.2 Competition 3
3.3 Sole-Source Procurement 4
3.4 Bidders List & Lead Discipline PO Strategy 5

4.0 PROCUREMENT PROCEDURE FOR SUPPLIES/EQUIPMENT 5
4.1 Corporate Procurement Procedures 5
4.2 Terms and Conditions - Equipment and Supplies 5

5.0 SYSTEMS 5
5.1 SAP Modules 5

6.0 TERMS AND CONDITIONS FOR SERVICES 6
6.1 General Terms and Conditions of Purchase 6
6.2 Bidder Exceptions to the General Terms and Conditions of Purchase 6

7.0 PROCUREMENT OF SERVICES 6
7.1 Roles and Responsibilities 6

8.0 PURCHASING 7
8.1 Request for Quotation 7
8.2 Method for Issuing RFx 7
8.3 Activities during RFx Period 8

9.0 RECEIPT AND OPENING OF BIDS 8
9.1 Bid/Proposal Submission 8

10.0 BID EVALUATION PROCEDURES 9
10.1 Objective 9
10.2 Initial Bid/Proposal Evaluation 9
10.3 Bid/Proposal Evaluation Guidelines 9
10.3.1 Evaluation Team 10
10.3.2 Evaluation Criteria 10
10.3.3 General Criteria for Rejection 11

10.3.4 Non-Competitive Bid/Proposal 11
10.4 Bid/Proposal Review Meeting..... 11
10.5 Finalize Evaluation of Bids/Proposals 12

11.0 RECOMMENDATION & PURCHASE ORDER AWARD 12
11.1 Recommendation for Award 12
11.2 Award of Purchase Order/ Contract 12

12.0 EXPEDITING AND INSPECTION 12
12.1 Expediting 13

13.0 PROCUREMENT CLOSE OUT 13

1.0 INTRODUCTION

1.1 General

This document defines the Newmont USA Limited (Newmont's) Procurement Policies (Policies) for the Midnite Mine Project hereinafter referred to as the "Project." These policies serve to identify the majority of the procurement requirements for the Project in terms of listing goods, materials and services to be procured, and the methods and procedures that will be followed.

These policies, after incorporation of the design drawings, specifications, and Remedial Action Work Plan/Construction Quality Assurance Plan (RAWP/CQAP), ultimately will become the procurement plan at the 90% design stage. In addition, the discussion below does not reflect the Spokane Tribal Employment Rights Ordinance (TERO), which will be incorporated in the procurement plan prior to the procurement of contractors and suppliers for the Midnite Mine RA.

1.2 Governing Procurement Policy

Newmont's policy is to perform procurement services in an ethical and professional manner by applying sound business practices. This includes utilizing sources of services and supplies which provide maximum value for each acquisition taking into consideration: safety, schedule, reliability, quality, warranty, after-sales-services, cost, and applicable laws and regulations.

2.0 PROCUREMENT PLANNING

2.1 Preliminary Activities

Newmont will develop a comprehensive list of materials, goods and services that need to be procured. This is referred to as the "Procurement List". The items to be procured will be separated into logical procurement packages hereinafter referred to as "Request for Quotations" or "RFx's".

2.1.1 SAP Procurement Software Tool

SAP is the automated system used by Newmont to effectively plan, schedule and monitor procurement activities for each RFx package.

2.1.2 Procurement Planning

Under the leadership of the Newmont's Global Supply Management ("GSM") Representative, project solicitations will contain RFx packages that are large dollar, complex, or items/services that are high

risk as well as transactional procurement items. Procurement planning and execution will be conducted and controlled by Newmont's GSM group for the purpose of ensuring an efficient, well thought out plan is developed for each specific procurement package (e.g., the WTP might be procured separately from the project earthworks as the skill set of the contractors bidding this work are very different). The planning will address: deliverables needed, scope of procurement, vendor SAP requirements, vendor representative requirements, risk assessment, shipping terms, bidder's list and other critical information to prepare any required RFx packages.

A critical component of the planning is the pre-procurement scheduling necessary to ensure that the plan supports in a timely manner the Project activities. The strategy will integrate the RFx tender schedule and the equipment/material delivery schedules to the Project schedules to avoid delays.

The level of procurement planning will depend on the size and complexity of the tender package.

2.1.3 Project Team Participation

For procurement planning to succeed uniquely qualified Newmont Managers should be included in the pre-procurement planning process. The Newmont GSM Representative for the Project will be in contact with specific Newmont Project team members to obtain their input and to introduce a higher level of planning and scheduling. The objective is to efficiently and effectively carry out a planning process that includes the necessary Newmont Managers (or their appointed representatives) who will identify critical path needs/items, supply risks, and other factors critical to the procurement process.

2.2 Planning Approach

For each individual RFx package, the Newmont GSM Representative will work collaboratively with other Newmont organizations/representatives to develop a plan that addresses all key procurement elements to ensure that equipment, materials, and services are delivered to the Site on time. The following points address certain aspects of the procurement planning process:

- The Newmont GSM Representative will be responsible for ensuring the procurement planning process is timely so services/supplies meet project deadlines.

- A detailed procurement schedule will be prepared that gives the necessary time to effectively (1) prepare a strategy for procurement, (2) prepare bidders list(s) for various services/supplies, (3) prepare tender documents, and (4) provide ample time for bidding and negotiating, management approval, purchase order (PO) preparation and (5) give full consideration long lead time items and their delivery.

- The Newmont GSM Representative, working with the Site-specific Managers, will identify potential risks that may affect any aspect of the procurement process.

The Newmont GSM Representative will ensure that labor rates for the supply of services at site are sought as part of the RFx process.

3.0 SERVICE AND SUPPLY SOURCING PROCEDURES

Newmont's GSM group will conduct competitive procurement activities through a full and open competition process among bidders. A transparent environment of fairness will be displayed to potential bidders as part of the bidding process. Procurement activities will be conducted by Newmont's GSM group based on the procedures established herein.

3.1 RFx Tender Package

The Newmont GSM Representative will develop the RFx packages using at a minimum the following Project approved documents comprising a:

1) Tender Invitation Letter,
2) Instruction to Bidders,
3) A Form of Proposal for bidders to insert prices,
4) Specifications and other technical documents (if required),
5) Newmont's standard terms and conditions.

3.2 Competition

A part of the planning process will be for Newmont's GSM group to solicit quotations from qualified, reputable, and dependable vendors and contractors that are capable of providing equipment, service and product warranty's, performance guaranty's (as necessary), after-sales-services, replacement parts and other requirements as deemed necessary. Unless otherwise instructed, a minimum of 3 and maximum of 6 bidders will be solicited.

3.3 Sole-Source Procurement

Newmont may determine that during the procurement planning process that there are compelling reasons and a sound justification to make a sole source award to a particular vendor. In that case, a Sole Source Justification that states the reasoning and rationale for such a decision will be formally documented within SAP.

The following are guidelines for authorizing a sole source acquisition:

- When only one vendor, supplier, manufacturer or contractor is identified as being capable (under any circumstances) of supplying the necessary goods or services.
- Procuring or contracting with government agencies, power and water utilities and municipalities or an enterprise that is a recognized monopoly.
- When a vendor, manufacturer, supplier or contractor has exclusive rights to a commodity that is highly specialized, unique or expert in nature and it can be clearly demonstrated that other vendors, manufacturers, suppliers or contractors do not have the same level of product expertise.
- When equipment or technical items are of a proprietary nature resulting in availability from just one source, and there are compelling reasons to require this proprietary item.
- In the event of an unforeseeable emergency that for the purposes of safety or protection of life or property requires immediate delivery of equipment, spare parts, goods or services (thus voiding the time to bid/proposal).
- Where there is a compelling economic reason to maintain equipment commonality throughout the Project site.
- Acquisition of equipment, spare parts or services to maintain a vendor warranty.
- Procuring or contracting to fulfil a community or tribal relations obligation for Newmont.

When sole sourcing is required, Newmont's GSM Representative nevertheless will prepare and issue an RFx package to the selected sole source vendor. The RFx

received by this vendor or contractor will be the basis for proceeding with sole-source negotiation and award.

3.4 Bidders List & Lead Discipline PO Strategy

Bidder's lists will be developed for all material and equipment purchases exceeding US$50,000 and services exceeding US$100,000. Worldwide sourcing will be used to locate the most responsible, reliable and economically beneficial suppliers and contractors as well as types of equipment, services and commodities.

A sufficient number of bidders will be used to ensure an efficient competition. Where prior qualifications of bidders are not documented, suppliers and contractors will be requested, to submit qualification questionnaires and, if required, will be surveyed through visits.

The Project Bidders Lists is a confidential Newmont document.

4.0 PROCUREMENT PROCEDURE FOR SUPPLIES/EQUIPMENT

4.1 Corporate Procurement Procedures

Newmont's Corporate Procurement Procedures will be utilized by Newmont for the Midnite Mine Project.

4.2 Terms and Conditions - Equipment and Supplies

Newmont will use standard "Purchase Terms & Conditions" for purchase of off-the-shelf and low value goods (under US$50,000). Standard "Service Terms and Conditions" will be utilized for contracting of low-value and low-risk services (under US$100,000).

5.0 SYSTEMS

Newmont's SAP procurement system shall be used for procurement and contracting purposes.

5.1 SAP Modules

The following SAP modules will be used for the purpose of monitoring and managing procurement activities:

- E-Sourcing (Contracting and Contracts Planning Module)
  - Enterprise Sourcing (Procurement Planning and RFX module)
  - Contract Request Form

- Contract Management (Contract Creation/Issuance to Vendor)

- ECC
  - ME51N – Create Purchase Requisition
  - ME54N – Release Purchase Requisition
  - ME21N – Create PO
  - ME29N – Release PO
  - ME31N – Create Outline Agreement
  - ME35K – Release Outline Agreement
  - XK01 – Create Vendor Master Record

6.0 TERMS AND CONDITIONS FOR SERVICES

6.1 General Terms and Conditions of Purchase

For services, Newmont's Service Terms and Conditions, Services Agreement or Master Services Agreement (dependent upon the estimated dollar value, type of service and risk profile) shall be issued with all RFP enquiries.

6.2 Bidder Exceptions to the General Terms and Conditions of Purchase

Newmont will make its best effort to resolve bidder's exceptions in the most favorable manner for the Project in the event a bidder provides exceptions or deviations to the Newmont's terms and conditions as part of its bid/proposal process. As a general rule, all exceptions shall be negotiated and finalized prior to making a formal award.

Vendors Terms & Conditions are not accepted to Newmont.

7.0 PROCUREMENT OF SERVICES

7.1 Roles and Responsibilities

Newmont GSM staff will be responsible for managing the complete tendering process comprising the following activities for each RFx/PO/contract:

- Assume the lead in developing each RFx package working with the Project team and other organizations to keep the RFx tender and award process on schedule.

- Prepare and issue pre-qualification letters, if necessary.

- Prepare RFx packages.

- Issue RFx enquiries and any additional details required during the tendering period.
- Act as sole means of communication and information sharing between the Project and the bidders.
- Receive quotations and conduct bid/proposal opening.
- Issue bid/proposal clarification letters and receive responses.
- Perform commercial review of bids/proposals received, prepare bid/proposal tabulations and conduct commercial bid/proposal evaluations (as required).
- Act as coordinator in joining the commercial evaluation and technical evaluation to arrive at a final recommendation for award decision.
- Prepare letters of recommendation.
- Conduct negotiations and PO/contract awards.
- After approval, award the PO/contract and obtain signed PO/contract back from vendor or contractor.
- Conduct expediting and inspection of materials and equipment.

8.0 PURCHASING

8.1 Request for Quotation

The Newmont GSM Representative will be responsible for the preparation of the RFx and will ensure that the entire package is complete and in order prior to issuance. The requisitioning process will be closely coordinated with the Project schedule. Upon receipt of an approved requisition, the Newmont GSM Representative will prepare and issue an RFx to the approved bidders.

8.2 Method for Issuing RFx

All RFx's will be issued electronically via Newmont's SAP system in accordance with the conditions set forth in the RFx. All files and documents will be issued in PDF format apart from Pricing Schedules which shall be in Microsoft Word to facilitate RFx tender preparation.

8.3 Activities during RFx Period

During the RFx period, the Newmont GSM Representative will:

- Make every effort to maintain and control the integrity of the bidding process.
- Ensure bidders confirm their intention to bid/proposal.
- Answer bidders' queries in a timely manner and equally share information equally amongst bidders.
- Prepare and issue RFx Amendments and all other information and documents forwarded to bidders during the tender period.

9.0 RECEIPT AND OPENING OF BIDS

This section sets out the procedure for receiving and opening bids/proposals.

9.1 Bid/Proposal Submission

(a) The RFx Instructions to Bidders will clearly state (1) the form in which the bids/proposals shall be submitted, (2) to whom they shall be addressed and (3) the latest time for submission.

(b) Bids/Proposals shall be submitted in electronic form as indicated in the respective RFx Documents.

Bids/Proposals shall be submitted to the named Newmont GSM Representative as shown on the Request for Quotation/Request for Proposal Letter.

- Bids/Proposals must contain all required price and delivery information and any other required information and a copy of the signed Bid/Proposal Form/Proposal.
- The formal bid/proposal must be received by the Newmont GSM Representative no later than the nominated bid/proposal date and closing time.
- Non-compliance with these procedures may be cause for rejection of the bid/proposal.

Bids/Proposals received after the closing date/ time will not be accepted and shall be returned to the bidder with an explanation of why it will not be considered.

Faxed bids/proposals will not be accepted. Verbal or informal bids/proposals will not be accepted.

10.0 BID EVALUATION PROCEDURES

10.1 Objective

The objective of bid evaluation is to identify and eventually recommend for award the bid/proposal/bidder which is determined to be offering goods and services that meet the minimum acceptable requirements set forth in the RFx for the best overall commercial offer and that fully complies with the RFx requirements.

10.2 Initial Bid/Proposal Evaluation

After receiving the bids/proposals, the Newmont GSM Representative shall:

- Acknowledge receipt of each bid/proposal by email within 48 hours.
- Check the contents of each bid/proposal for completeness of submission and expedite from bidders items that they have failed to submit.
- Determine that electronic copies have been received on time, in the correct format, and the original bid/proposal signed by the bidder.
- Identify the appropriate technical Newmont personnel to review and evaluate the bids/proposals
- Control the exchange of documents and information between evaluators.
- Maintain close coordination with technical evaluators.

10.3 Bid/Proposal Evaluation Guidelines

A bid/proposal evaluation procedure with specific criteria (commercial and technical) will be established by the Newmont GSM Representative and the technical evaluators prior to the receipt of bids/proposals (typically during the RFx development stage) to determine the basis for contract award.

10.3.1 Evaluation Team

The Newmont GSM Representative will assume responsibility as the lead evaluator. The GSM representative will control the overall evaluation process and be responsible for all components of the RFx and bids/proposals. The lead evaluator will be the sole individual to communicate with bidders. The lead evaluator will prepare spread sheets and prepare the final letter of recommendation for award in cooperation with the technical evaluator for submission to the selected Newmont project team member.

Depending on the size and complexity of the RFx package there may be more than one technical evaluator. An engineer or other technical person will be appointed as the lead technical evaluator. The lead technical evaluator shall develop with assistance of the GSM representative, the appropriate technical evaluation criteria prior to receipt of bids/proposals (typically during the RFx development stage). Bidder responses to these technical criteria will be evaluated as a means of reaching a technical recommendation.

The lead evaluator and lead technical evaluator will meet to discuss the technical and commercial merits of the various bids/proposals and reach a joint recommendation for award.

10.3.2 Evaluation Criteria

The evaluation of bids/proposals will be on a point system or some other acceptable method to systematically/uniformly score the technical bids/proposals. All bids/proposals will be evaluated with respect to best value which includes some or all of the following criteria:

The best overall and most favorable bid/proposal in terms of:

- Technically conforms to the SOW
- Lowest cost that is supported by sound and rational cost and pricing data
- Demonstrated ability to meet delivery schedule
- Warranty
- After-sales-services
- Agreeing to KPI’s
- other unique project-specific factors

The criteria provided above shall be modified by the evaluators to accommodate the respective bid/proposal evaluation.

10.3.3 General Criteria for Rejection

The following is the general criteria that will be used for rejection of non-responsive bids/proposals.
Technical:

Bid/proposal fails to meet the minimum acceptable technical requirements set forth in the RFx

Commercial:

- Received bid/proposal late
- Received grossly incomplete, irregular, unrealistic, or significantly not prepared in accordance with bid/proposal instructions (or)
- Does not have the financial or technical capabilities to execute the SOW or services.

10.3.4 Non-Competitive Bid/Proposal

During the commercial evaluation where it is clearly determined that a bid/proposal is not in contention, and that there are clearly a minimum of three other bidders that are competitive and responsive to the RFx, Newmont may declare this bid/proposal to be "Commercially Non-Competitive".
During the technical review process where it is clearly determined that a bidder has gross technical deficiencies (that would be difficult to reconcile through negotiation), and that there are clearly other bidders that are technically responsive to the RFx, Newmont may declare this bid/proposal to be "Technically Non-Competitive". Newmont will outline compelling reasons to make such a bid/proposal non-competitive as part of its evaluation.

The purpose of this clause is to save time and to accelerate the evaluation process by not having to spend time on bids/proposals that are recognizably not viable.

10.4 Bid/Proposal Review Meeting

For larger procurement packages, the Newmont GSM Representative may consider a bid/proposal review meeting or teleconference with the potential successful bidder(s) (or if prices are competitive - two favored bidders). Such meetings or teleconferences shall be limited to discussions and resolution of any potentially unclear issues, affirmation of cost and pricing, discussing any possible change in the SOW, and agreeing to any late adjustments to price, as applicable. If a technical deficiency or commercial errors or omissions are apparent in the

respective bidder's quotation/proposal this may be discussed and possibly reconciled. Anything agreed upon in the meeting or over the telephone shall be affirmed in writing by the bidder. In addition, bidder's exceptions and/or deviations should be discussed to eliminate miscommunication.

At this stage, bidders will be aware that their bids/proposals are being considered favorably and recommendations of award will be forthcoming.

10.5 Finalize Evaluation of Bids/Proposals

Upon completion of evaluating bids/proposals the lead GSR and technical evaluators will meet to rank their bids/proposals based on the evaluation criteria and point system established during the RFx development period and make a joint decision for recommending award.

11.0 RECOMMENDATION & PURCHASE ORDER AWARD

11.1 Recommendation for Award

Upon finalizing the commercial and technical evaluations, a recommendation for award letter will be prepared by the Newmont GSM Representative. The recommendation for award letter will reflect what has been agreed to in terms of price, technical and commercial requirements, shipping and delivery terms, a negotiating strategy, and other specifics. Depending on the solicitation, some of these items may or may not be included in the award letter.

11.2 Award of Purchase Order/ Contract

The Newmont GSM Representative will prepare and issue the PO/contract based on the terms, pricing schedules, and final conditions that were negotiated with the successful bidder.

The Newmont GSM Representative will ensure that all particulars are incorporated into the PO and that the vendor appropriately signs and dates the order.

12.0 EXPEDITING AND INSPECTION

An inspection and expediting plan for procured commodities will be prepared by the Procurement Department based on the project schedule and the equipment criticality rating. POs will be expedited by visits to shop, e-mail and/or telephone as required.

12.1 Expediting

The degree of expediting of POs shall be dictated by the criticality (vendor data and delivery) and the potential risk of project delay. Critical path items regularly shall be expedited while expediting of non-critical items shall be at the discretion of the Newmont GSM Representative and Project Manager.

13.0 PROCUREMENT CLOSE OUT

The Newmont GSM Group will be responsible for all aspects of closeout related to departmental activities. The Newmont GSM Group will formally close out all POs/contracts ensuring there are no outstanding claims by vendors. Contractors or potential liens held by third parties against Newmont as a result of outstanding procurement issues.