Midnite Mine Superfund Site

Appendix S – Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments

Note: This Analytical Support and Verification Plan for Remediation of Surface Materials and Sediments has been prepared in general accordance with 90-percent level of completion in response to EPA comments to date. Minor edits to this plan are expected as the Midnite Mine Remedial Design is finalized. The final plan will be included with the Final Basis of Design Report. This plan is intended to satisfy the requirement for a Confirmation Sampling and Remedial Action Effectiveness Monitoring Plan as stipulated by the Consent Decree.

June 2015
July 31, 2014

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

SENES Consultants
8310 South Valley Highway, Suite 135
Englewood, Colorado, 80112
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 ............................................................................................... 15
  S.2.3 Onsite X-ray Florescence (XRF) Analysis .................................................................... 17
    Methodology ..................................................................................................................... 17
    S.2.3.1 Instrumentation ..................................................................................................... 19
    S.2.3.2 Calibration ........................................................................................................... 19
  S.2.4 Offsite Laboratory Analysis ......................................................................................... 19
    S.2.4.1 Offsite Laboratory Methods ................................................................................ 21
    S.2.4.2 Special Sample Handling/Analysis Protocols ...................................................... 22
S.3.0 REMEDIAL SUPPORT SURVEY PLAN ....................................................................... 23
  S.3.1 Gamma Scanning ....................................................................................................... 23
    S.3.1.1 Gamma Scanning for Excavation Support ............................................................ 23
    S.3.1.2 Gamma Surveys for Characterization of Class 2 Areas and Borrow Materials ...... 25
  S.3.2 In-situ XRF Surveys .................................................................................................. 26
    S.3.2.1 In-situ XRF Surveys for Excavation Support ......................................................... 26
    S.3.2.2 In-situ XRF Surveys for Characterization of Class 2 Areas .................................. 27
  S.3.3 Onsite Sample Analysis .............................................................................................. 27
    S.3.3.1 Onsite Sample Analysis for Excavation Support ................................................. 27
    S.3.3.2 Onsite Sample Analysis for Characterization of Class 2 Areas ......................... 29
  S.3.4 Decision Criteria for Remedial Support Surveys ......................................................... 29
    S.3.4.1 Surface Materials ................................................................................................. 29
    S.3.4.2 Sediments ............................................................................................................ 30
S.4.0 FINAL STATUS SURVEY PLAN .................................................................................... 32
  S.4.1 Final Status Survey Units ............................................................................................ 32
  S.4.2 Final Status Surveys for Surface Materials ................................................................. 39
    S.4.2.1 Final Status Gamma Survey ................................................................................ 39
    S.4.2.2 Final Status Surface Material Sampling ............................................................... 41
    S.4.2.3 Determination of Compliance for Surface Materials ........................................ 42
  S.4.3 Final Status Surveys for Sediments ............................................................................ 47
    S.4.3.1 XRF Surveys for Mine Drainages ....................................................................... 47
    S.4.3.2 Gamma Surveys for Mine Drainages ................................................................. 48
    S.4.3.3 Sediment Sampling .............................................................................................. 48
    S.4.3.4 Determination of Compliance for Sediments ...................................................... 49
S.5.0 DATA QUALITY ASSURANCE / QUALITY CONTROL (QA/QC) .................................. 54
S.6.0 REFERENCES .................................................................................................................. 57
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) ................................................................................... 3
Figure S-4 – Generalized mine waste removal, analytical assessment and decision diagram for remediation of surface materials (note: CL = cleanup level). ............................................................. 7
Figure S-5 – Generalized mine waste removal, analytical assessment and decision diagram for remediation of sediments (note: CL = cleanup level) ........................................................................ 8
Figure S-6 – Example backpack scanning system configuration. ................................................................. 10
Figure S-7 – Correlation plot soil sampling and gamma scanning design ................................................... 13
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 ........................................................................... 16
Figure S-10 – Class 1 zones delineated by remedial schedule .................................................................. 36
Figure S-11 – Class 1 and Class 2 survey units ........................................................................................ 38
Figure S-12 – In-situ XRF survey transects and laterally alternating direct soil sampling locations for onsite intrusive XRF analysis and offsite laboratory analysis ........................................ 48

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 .......... 21
Table S-5 – Analytical Parameters, Cleanup Levels, Analysis Methods and Detection Limits for Surface Materials ........................................................................................................................... 21
Table S-6 – Analytical Parameters, Cleanup Levels, Analysis Methods and Detection Limits for Sediments ...................................................................................................................................... 22
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 ........................................................................ 52
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

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>CERCLA</td>
<td>Comprehensive Environmental Response, Compensation, and Liability Act</td>
</tr>
<tr>
<td>cm</td>
<td>centimeter</td>
</tr>
<tr>
<td>COC</td>
<td>Constituents of Concern</td>
</tr>
<tr>
<td>DOE</td>
<td>US Department of Energy</td>
</tr>
<tr>
<td>DQO</td>
<td>Data Quality Objective</td>
</tr>
<tr>
<td>EPA</td>
<td>US Environmental Protection Agency</td>
</tr>
<tr>
<td>eU</td>
<td>equivalent uranium</td>
</tr>
<tr>
<td>g</td>
<td>gram</td>
</tr>
<tr>
<td>GIS</td>
<td>geographical information systems</td>
</tr>
<tr>
<td>GPS</td>
<td>global positioning system</td>
</tr>
<tr>
<td>HPGe</td>
<td>high-purity germanium</td>
</tr>
<tr>
<td>kg</td>
<td>kilogram</td>
</tr>
<tr>
<td>kV</td>
<td>kilovolt</td>
</tr>
<tr>
<td>MAA</td>
<td>Mining Affected Areas</td>
</tr>
<tr>
<td>MARSSIM</td>
<td>Multi-agency Radiation Site Survey and Investigation Manual</td>
</tr>
<tr>
<td>MCA</td>
<td>multi-channel analyzer</td>
</tr>
<tr>
<td>MDC</td>
<td>minimum detectable concentration</td>
</tr>
<tr>
<td>mg</td>
<td>milligram</td>
</tr>
<tr>
<td>NaI</td>
<td>sodium iodide</td>
</tr>
<tr>
<td>NRC</td>
<td>Nuclear Regulatory Commission</td>
</tr>
<tr>
<td>NPL</td>
<td>National Priorities List</td>
</tr>
<tr>
<td>NUREG</td>
<td>U.S. Nuclear Regulatory Commission regulation</td>
</tr>
<tr>
<td>pCi</td>
<td>picocuries</td>
</tr>
<tr>
<td>QA/QC</td>
<td>Quality Assurance / Quality Control</td>
</tr>
<tr>
<td>QAM</td>
<td>Quality Assurance Manager</td>
</tr>
<tr>
<td>QAPP</td>
<td>Quality Assurance Project Plan</td>
</tr>
<tr>
<td>RI/FS</td>
<td>Remedial Investigation/Feasibility Study</td>
</tr>
<tr>
<td>ROD</td>
<td>Record of Decision</td>
</tr>
<tr>
<td>ROI</td>
<td>regions of interest</td>
</tr>
<tr>
<td>SMI</td>
<td>Shepherd Miller, Inc.</td>
</tr>
<tr>
<td>Site</td>
<td>Midnite Mine Superfund Site</td>
</tr>
<tr>
<td>SOP</td>
<td>Standard Operating Procedure</td>
</tr>
<tr>
<td>UPL</td>
<td>upper prediction limit</td>
</tr>
<tr>
<td>µR/h</td>
<td>microroentgen per hour</td>
</tr>
<tr>
<td>URS</td>
<td>URS Corporation</td>
</tr>
<tr>
<td>VSP</td>
<td>Visual Sample Plan</td>
</tr>
</tbody>
</table>
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 sediments¹ 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)

<table>
<thead>
<tr>
<th>Contaminant of Concern</th>
<th>Cleanup Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uranium (total)²</td>
<td>43 mg/kg</td>
</tr>
<tr>
<td>Lead-210</td>
<td>7.5 pCi/g</td>
</tr>
<tr>
<td>Radium-226</td>
<td>4.7 pCi/g</td>
</tr>
</tbody>
</table>

²Note: Table 8-3 lists the units for Uranium as mg/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)

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

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).

²Throughout this document, “uranium” refers the natural (total) form unless an isotopic form (e.g. U-238) is specified.
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 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, analytical assessment and decision criteria, are depicted in

---

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.
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:

- **Attachment S-1 – Technical Basis**
- **Attachment S-2 – Quality Assurance Project Plan (QAPP)**
  - 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 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.
**REMEDIAL SUPPORT SURVEYS**

**Step 1: Excavate impacted surface materials.**

**Analytical guidance approach:**
- Gamma scans during excavation
- Verification sampling/onsite Ra-226 analysis
- 10% offsite confirmatory analysis

**Decision criteria:**
- Gamma readings ≤ gamma cutoff value
- Onsite Ra-226 analysis ≤ ROD CL
- Offsite analysis ≤ all ROD CLs

- Does scanning & soil analysis indicate ROD CLs are met?
  - Yes: Stop excavation
  - No: Repeat Step 1 for areas not sufficiently remediated

- Does scanning & soil analysis indicate ROD CLs are met?
  - Yes: Stop excavation
  - No: Was bedrock encountered?
    - Yes: Waste removal complete
    - No: Did verification scanning/sampling indicate cleanup levels met?
      - Yes: Waste removal complete
      - No: Re-survey areas of additional excavation, document new analytical evidence of compliance in subject areas

**FINAL STATUS SURVEYS**

**Step 2: Conduct final status survey.**

**Analytical verification approach:**
- Recorded GPS-based gamma survey
- Onsite Ra-226 analysis (100% of samples)
- Offsite analysis (all COCs, 33% of samples)
- Data analysis / statistical testing

**Decision criteria:**
- 95% Gamma readings ≤ gamma cutoff value
- 95% Scan-based Ra-226 estimates ≤ ROD CL
- Stat test on sample analysis results indicates 95% probability of ≥ 95% rate of compliance
- No single sample result > twice ROD CL

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

**REMEDIAL SUPPORT SURVEYS**

- **Step 1: Excavate impacted sediments.**
  - **Analytical guidance approach:**
    - In-situ XRF measurements during excavation
    - Intrusive verification XRF analysis onsite
    - 20% offsite confirmatory analysis

- **Decision criteria:**
  - In-situ XRF data ≤ in-situ XRF cutoff values
  - Intrusive XRF analyses ≤ ROD CLs
  - Offsite analysis ≤ ROD CLs

- **Remedial Support Surveys Flowchart:**
  - Do sediment analyses indicate ROD CLs are met?
    - Yes: Waste removal complete
    - No: Repeat Step 1 for areas not sufficiently remediated
      - Was bedrock encountered?
        - Yes: Waste removal complete
        - No: Do sediment analyses indicate ROD CLs are met?
          - Yes: Waste removal complete
          - No: Stop excavation

- **Final Status Surveys Flowchart:**
  - **Step 2: Conduct final status survey.**
    - **Analytical verification approach:**
      - Radiological COCs: same as surface materials
      - In-situ XRF field survey
      - Intrusive XRF analysis (100% of samples)
      - Offsite analysis (all COCs, 33% of samples)
      - Data analysis / statistical testing

    - **Decision criteria:**
      - Radiological criteria same as surface materials
      - 95% in-situ XRF data ≤ in-situ cutoff values
      - Stat test on sample analysis results indicates 95% probability of ≥ 95% rate of compliance
      - No single sample result > twice ROD CL

  - Stop excavation
    - Re-survey areas of additional excavation, document new analytical evidence of compliance in subject areas
      - Did verification surveys/sampling indicate ROD CLs met?
        - Yes: Waste removal complete
        - No: Stop excavation
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.

![Example backpack scanning system configuration.](image)

**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

\(^4\) 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.
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 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 m² 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).

---

\(^5\) 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, ore rocks cannot be detected in-situ 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.
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-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 (α = 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.
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

---

^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.
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 Fluorescence (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.

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

<table>
<thead>
<tr>
<th>Onsite Analysis:</th>
<th>Gamma Spectroscopy</th>
<th>Intrusive XRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface Materials</td>
<td>100%</td>
<td>Judgmental</td>
</tr>
<tr>
<td>Sediment</td>
<td>100%</td>
<td>100%</td>
</tr>
<tr>
<td>Remedial Support Samples</td>
<td>% Sent Offsite*</td>
<td>% Sent Offsite*</td>
</tr>
<tr>
<td>Surface Materials</td>
<td>10%</td>
<td>5%</td>
</tr>
</tbody>
</table>
## Final Status Survey Samples

<table>
<thead>
<tr>
<th></th>
<th>5%</th>
<th>10%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface Materials</td>
<td>33%</td>
<td>33%</td>
</tr>
<tr>
<td>Sediment</td>
<td>33%</td>
<td>33%</td>
</tr>
</tbody>
</table>

*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

<table>
<thead>
<tr>
<th>Analytical Objective</th>
<th>Offsite Laboratory Analysis Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Remedial Support Samples</td>
<td>Radiological(^1,3)</td>
</tr>
<tr>
<td>Surface Materials</td>
<td>Metals(^2,3)</td>
</tr>
<tr>
<td>Ra-226, Pb-210, U</td>
<td></td>
</tr>
<tr>
<td>Sediment</td>
<td></td>
</tr>
<tr>
<td>Ra-226, Pb-210, U</td>
<td>U, Cr, Mn, Se, V</td>
</tr>
<tr>
<td>Final Status Survey Samples</td>
<td>Radiological(^1,3)</td>
</tr>
<tr>
<td>Surface Materials</td>
<td>Metals(^2,3)</td>
</tr>
<tr>
<td>Ra-226, Pb-210, U</td>
<td></td>
</tr>
<tr>
<td>Sediment</td>
<td></td>
</tr>
<tr>
<td>Ra-226, Pb-210, U, U-238, U-234, U</td>
<td>U, Cr, Mn, Se, V</td>
</tr>
</tbody>
</table>

\(^1\)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

<table>
<thead>
<tr>
<th>ROD Parameter</th>
<th>Cleanup Level</th>
<th>Proposed Method</th>
<th>Method Detection Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uranium (total)</td>
<td>43 mg/kg</td>
<td>EPA 3050/EPA 6020</td>
<td>0.05 mg/kg</td>
</tr>
<tr>
<td>Lead-210</td>
<td>7.5 pCi/g</td>
<td>EPA 3050/EPA 909.0M</td>
<td>0.1 pCi/g</td>
</tr>
<tr>
<td>Radium-226</td>
<td>4.7 pCi/g</td>
<td>EPA 901.1 (soil, full Rn ingrowth)</td>
<td>0.4 pCi/g</td>
</tr>
</tbody>
</table>
Table S-6 – Analytical Parameters, Cleanup Levels, Analysis Methods and Detection Limits for Sediments

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

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 proscriptively 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

\(^7\) 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.
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 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 or as needed basis, but not less than once every other week during periods of active excavation. Four to five random grab samples of borrow materials will also be collected on a weekly basis at least once every other week 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 m² 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 XRF 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:

**Compliance Evaluation Criteria**

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 proscriptively 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.

---

\(^8\) 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.
• **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 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 **prospectively** 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

\(^{9}\) 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 76 in Section S.3.1.1.
excavation and re-surveys, to help 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 2 percent of sampling locations (at least 2 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.

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:

Compliance Evaluation Criteria

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 remediared.

### 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 2 percent of sampling locations (at least 2 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

<table>
<thead>
<tr>
<th>ROD cleanup level for U (mg/kg)</th>
<th>ROD cleanup level (pCi/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>92.3</td>
<td>-</td>
</tr>
<tr>
<td>ROD cleanup level for U expressed in terms of activity concentration (pCi/g)</td>
<td>62.5</td>
</tr>
<tr>
<td>Equivalent U-238 activity concentration calculated based on disequilibrium in impacted sediments (pCi/g)*</td>
<td>27.4</td>
</tr>
<tr>
<td>Equivalent U-234 activity concentration calculated based on disequilibrium in impacted sediments (pCi/g)*</td>
<td>33.8</td>
</tr>
</tbody>
</table>

*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.

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 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:

Program QA Summary:

- 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


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


Midnite Mine Superfund Site

1090 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). It has been prepared in general accordence with 90-percent level of completion in response to related EPA comments to date. Minor edits to this Technical Basis document may occur as the Midnite Mine Remedial Design progresses through the final design stage. The final plan will be included with the Final Basis of Design Report. 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.

July 31, 2014

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:

SENES Consultants
8340 South Valley Highway, Suite 135
Englewood, Colorado 80112

Environmental Resources Group (ERG)
1358809 Washington St NE, Suite 150
Albuquerque, New Mexico 8011287113
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).

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

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.

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.

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).

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

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).

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.
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 m² 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](image)

**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 ($\approx 11-20$ 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 (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 m² 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 m² 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.

<table>
<thead>
<tr>
<th>Hot Spot Diameter (m)</th>
<th>2x2 NaI Detector Height (m)</th>
<th>Scan Speed (m/s)</th>
<th>Calculated Scan MDC for Ra-226 (pCi/g)</th>
<th>Calculation Source and Methodology</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>0.25</td>
<td>0.5</td>
<td>2.8</td>
<td>MARSSIM (NUREG 1507/1575 method)</td>
</tr>
<tr>
<td>1.5</td>
<td>1.0</td>
<td>1.0</td>
<td>6.2</td>
<td>SENES (NUREG 1507/1575 method)</td>
</tr>
<tr>
<td>3.0</td>
<td></td>
<td>1.0</td>
<td>2.1</td>
<td>SENES (NUREG 1507/1575 method)</td>
</tr>
</tbody>
</table>

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 m² 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. Such real-world data are consistent with the calculated scan MDC values provided in Table 1. 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 $\alpha = 0.05$, a statistical bound commonly set by regulatory agencies in terms of limiting decision errors on compliance with cleanup criteria.

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.
• 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 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 – Left: Linear regression curves for 87 paired HPGe/Nal results for Ra-226, where the Nal 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 Nal-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).](image)

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
- \( \lambda \) = 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 “C₀” 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-226⁶. 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 programmed 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 rule8 (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 (\(\beta\)) 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 \(\alpha\) 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 \(\alpha\) (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 (\(\beta\)) is not actually controlled because the LBGR was set at zero (\(\beta\) 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 \(\beta\) 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 (\(\alpha\)) is unaffected should there be an insufficient number of samples to also limit the probability of Type II errors to the value specified for \(\beta\) (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


Attachment S-2

Quality Assurance Project Plan for Remedial Action Analytical Support and Verification (QAPP)
Midnite Mine Superfund Site

9100 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). This attachment has not changed between the 60 and 90 percent design stages. Minor edits to this QAPP may occur as the Midnite Mine Remedial Design progresses through the final design stage. The final plan will be included with the Final Basis of Design Report. 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.

July 31, 2014 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:

Worthington Miller Environmental, LLC
201 Linden Street Suite 3011027 W
Horsetooth Rd, Suite 200
Fort Collins, Colorado 805246

SENES Consultants Environmental Resource Group (ERG)
8310 South Valley Highway, Suite 1358809
Washington St. NE, Suite 105
EnglewoodAlbuquerque, ColoradoNew Mexico, 8014287113
Approval Sheet

Issued by:

Project Quality Assurance Manager: ________________________
Jill Richards, Worthington Miller Environmental, LLC

Prepared for:

Supervising Contractor: ________________________
Louis Miller, Worthington Miller Environmental, LLC

Alternate Project Coordinator: ________________________
Bill Lyle, Newmont Mining Cooperation

EPA Remedial Project Manager: ________________________
Ellen Hale, Karen Keeley, US EPA

EPA Quality Assurance Manager: ________________________
Gina Grepo-Grove, US EPA
## Distribution List

<table>
<thead>
<tr>
<th>QAPP Recipient Name</th>
<th>Organization</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ellen Hale, Karen Keeley</td>
<td>U.S. Environmental Protection Agency</td>
</tr>
<tr>
<td>Gina Grepo-Grove</td>
<td>U.S. Environmental Protection Agency</td>
</tr>
<tr>
<td>Randy Connolly</td>
<td>Spokane Tribe of Indians</td>
</tr>
<tr>
<td>Bill Lyle</td>
<td>Newmont Mining Cooperation.</td>
</tr>
<tr>
<td>Louis Miller</td>
<td>Worthington Miller Environmental, LLC</td>
</tr>
<tr>
<td>Jill Richards</td>
<td>Worthington Miller Environmental, LLC</td>
</tr>
<tr>
<td>Laboratory Project Managers</td>
<td>Laboratory Project Manager</td>
</tr>
</tbody>
</table>
Table of Contents

A. Project Management and Data Quality Objectives .......................................................... 11
   A.1 Project Organization ................................................................................................. 11
   A.2 Problem Definition/Background ........................................................................... 66
      A.2.1 Purpose ............................................................................................................. 66
      A.2.2 Problem Statement ......................................................................................... 66
      A.2.3 Background ..................................................................................................... 66
   A.3 Project Description and Schedule ........................................................................... 66
      A.3.1 Description of Work to be Performed ............................................................. 66
      A.3.2 Schedule of Activities ..................................................................................... 66
   A.4 Data Quality Objectives (DQOs) ........................................................................... 77
      A.4.1 Measurement Performance Criteria ............................................................... 1010
   A.5 Special Training Requirements/Certification ......................................................... 1515
   A.6 Documentation and Records ................................................................................. 1616
      A.6.1 Field Operation Records ................................................................................. 1616
      A.6.2 Laboratory Records ......................................................................................... 1616

B. Measurement Data Acquisition .................................................................................. 1818
   B.1 Field Surveys and Sampling Design ...................................................................... 1818
   B.2 Onsite Analytical Methods Requirements .............................................................. 1818
      B.2.1 Onsite Sampling Handling and Custody ......................................................... 1818
   B.3 Offsite Analytical Methods Requirements ............................................................. 2222
      B.3.1 Offsite Sample Handling and Custody ............................................................. 2323
      B.3.2 Special Sample Handling/Analysis Protocols for the Offsite Laboratory .......... 2323
   B.4 Quality Control Requirements .............................................................................. 2929
      B.4.1 Field QC Procedures ..................................................................................... 2929
      B.4.2 Laboratory QC Procedures .......................................................................... 3333
   B.5 Instrument/Equipment Testing, Inspection and Maintenance Requirements .......... 3535
      B.5.1 Field Instruments and Equipment ................................................................. 3535
      B.5.2 Laboratory Instruments ............................................................................... 3535
      B.5.3 Acceptance Requirements for Supplies and Consumables ............................. 3535
   B.6 Instrument Calibration and Frequency .................................................................... 3535
      B.6.1 Field Calibration Procedures ....................................................................... 3535
      B.6.2 Laboratory Calibration Procedures ............................................................... 3636
   B.7 Data Acquisition Requirements ............................................................................. 3636
   B.8 Data Management .................................................................................................. 3737

C. Assessment/Oversight ................................................................................................. 3838
   C.1 Assessment and Response Actions ........................................................................ 3838
C.1.1 Field Measurement Data ................................................................. 3838
C.1.2 Laboratory Data ................................................................................. 3838
C.1.3 Performance and System Audits......................................................... 3838
C.1.4 Corrective Actions.................................................................................. 3939
C.2 Quality Assurance Reports to Management............................................. 4040
   C.2.1 Data Validation Reports ................................................................. 4141

D. Data Validation and Usability ................................................................. 4141
   D.1 Data Review, Validation and Verification Requirements...................... 4141
   D.2 Validation and Verification Methods................................................... 4141
      D.2.1 Validation/Verification of Field and Onsite Analysis Data .............. 4141
      D.2.2 Validation/Verification of Offsite Laboratory Data ......................... 4242

E. References................................................................................................ 4444

List of Tables

Table 1: Data Quality Objectives (DQO) Process Summary ...................................... 88
Table 2: Analytical Acceptance/Performance Criteria ........................................... 1313
Table 3: Sample Containers, Preservation, and Holding Time Requirements for Solid Samples .... 1949
Table 4: Soil and Sediment Laboratory Parameters, Analysis Methods, and Method Detection Limits ........................................................................................................................... 2525
Table 5: Analytical Method QC Specifications ....................................................... 2626
Table 5: Analytical Method QC Specifications (Cont.) ............................................ 2727
Table 5: Analytical Method QC Specifications (Cont.) ............................................ 2828

List of Figures

Figure 1: Project Organizational Chart.................................................................. 55
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. ........ 2929
Figure 4: Example instrument background quality control chart for multiple NaI detectors. .......................................................... 3030
Figure 5: Example field strip control chart for multiple NaI detectors. ......................... 3131
Figure 6: Example QC chart for soils lab measurements. ........................................... 3232

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

Attachment S2 – Quality Assurance Project Plan
6100 Percent Design

vi

December 2013 June 2015
Revision 0
List of Acronyms

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

EPA Quality Assurance Manager (Gina Grepo-Grove, EPA)

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 Offsite Laboratory Project Manager will work directly with the Laboratory QA Officer 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), offsite laboratory analysis.

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.

### 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.
### 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.

#### Decision Criteria Guidelines

- Nearly 100% of gamma readings$^*$ $\leq 1$.

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 vertically and horizontally until remedial support survey data provide sufficient evidence to support a decision that respective locations are ready for final status surveys.

#### Possible Outcomes:

1. The data indicate that surface materials meet cleanup levels and final status surveys 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.

2. 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 spectrometry, 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.

3. Excavation has progressed to bedrock and no further excavation can proceed.

**Decision Criteria Guidelines for Evaluation of Sediments (Section S.3.4.2 of Appendix S):**

Decision criteria guidelines for evaluation of remedial support survey data for sediments include the following:

- Nearly 100% of gamma readings$^*$ $\leq 1$.

On a macro scale, the initial study area for remedial support surveys in mine drainage will include all areas in the vicinity of the drains with the potential to exceed the cleanup levels for sediments. Based on existing data, about 2 linear 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.

### REMEDIAL SUPPORT SURVEYS FOR SEDIMENTS

The ROD requires mine drainage materials 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 additional survey data are required.

#### Decision Criteria Guidelines

- Nearly 100% of gamma readings$^*$ $\leq 1$.

#### Possible Outcomes:

1. The data indicate that sediments meet cleanup levels and final status surveys can accommodate considerable radiological disequilibrium in these parameters. Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that non-radiological parameters (manganese) particularly manganese, exceed cleanup levels to the greatest extent in sediments.

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 Criteria Guidelines for Evaluation of Sediments (Section S.3.4.2 of Appendix S):**

Decision criteria guidelines for evaluation of remedial support survey data for sediments include the following:

- Nearly 100% of in-situ XRF Mn readings$^*$ $\leq$ Mn cutoff value.
- Nearly 100% of onsite XRF Mn readings$^*$ $\leq$ Mn cutoff value.

On a macro scale, the initial study area for remedial support surveys in mine drainage will include all areas in the vicinity of the drains with the potential to exceed the cleanup levels for sediments. Based on existing data, about 2 linear 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.

#### Possible Outcomes:

1. The data indicate that sediments meet cleanup levels and final status surveys can accommodate considerable radiological disequilibrium in these parameters. Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that non-radiological parameters (manganese) particularly manganese, exceed cleanup levels to the greatest extent in sediments.

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 Criteria Guidelines for Evaluation of Sediments (Section S.3.4.2 of Appendix S):**

Decision criteria guidelines for evaluation of remedial support survey data for sediments include the following:

- Nearly 100% of in-situ XRF Mn readings$^*$ $\leq$ Mn cutoff value.

#### Possible Outcomes:

1. The data indicate that sediments meet cleanup levels and final status surveys can accommodate considerable radiological disequilibrium in these parameters. Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that non-radiological parameters (manganese) particularly manganese, exceed cleanup levels to the greatest extent in sediments.

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 Criteria Guidelines for Evaluation of Sediments (Section S.3.4.2 of Appendix S):**

Decision criteria guidelines for evaluation of remedial support survey data for sediments include the following:

- Nearly 100% of in-situ XRF Mn readings$^*$ $\leq$ Mn cutoff value.

#### Possible Outcomes:

1. The data indicate that sediments meet cleanup levels and final status surveys can accommodate considerable radiological disequilibrium in these parameters. Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that non-radiological parameters (manganese) particularly manganese, exceed cleanup levels to the greatest extent in sediments.

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 Criteria Guidelines for Evaluation of Sediments (Section S.3.4.2 of Appendix S):**

Decision criteria guidelines for evaluation of remedial support survey data for sediments include the following:

- Nearly 100% of in-situ XRF Mn readings$^*$ $\leq$ Mn cutoff value.

#### Possible Outcomes:

1. The data indicate that sediments meet cleanup levels and final status surveys can accommodate considerable radiological disequilibrium in these parameters. Data obtained from pre-design investigations (Miller Geotechnical, 2011) indicate that non-radiological parameters (manganese) particularly manganese, exceed cleanup levels to the greatest extent in sediments.

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.
The study area for final status surveys for surface material will include defined Class 4: Define the Boundaries and Specify Limits. These areas will be defined 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.

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.

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., \( \alpha = 0.05 \)).

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**

Results against the estimation uncertainty for a given instrument over time, and precision between 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 on 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.
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 onsite 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

<table>
<thead>
<tr>
<th>Sample Media</th>
<th>Analyte</th>
<th>Accuracy Measures and Control Limits¹</th>
<th>Precision Measures and Control Limits¹</th>
<th>Completeness Goal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soil and Sediment Samples</td>
<td>Total chromium (Cr), total manganese (Mn), total selenium (Se), total uranium (U), total vanadium (V); Ra-226², Pb-210, U-234, and U-238</td>
<td>Offsite lab analysis: LCS Recovery: 80-100%, MS/MSD Recovery: 70-130%</td>
<td>Offsite lab analysis: Analytical Duplicate RPD: &lt;35%, MS/MSD RPD: &lt;35%, Radiochemical Analysis RER: &lt;2.0</td>
<td>Laboratory: 95%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Onsite lab data:</td>
<td>Field duplicate RPD: &lt;50%</td>
<td>Gamma and XRF: 100%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1) QC measurements on site-specific calibration standards within established control limits.</td>
<td>XRF measurements: &lt;20% RSD</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2) 90% of field sample results within limits of 90% prediction intervals on calibration curves.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
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 compositing soil samples across a gamma/Ra-226 correlation plot accurately reflect the true average Ra-226 concentration across the plot.

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).
• 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 types of media to be surveyed in-situ in the field or to be directly sampled for laboratory analysis (by gamma spectroscopy, intrusive XRF analysis, and chemical analysis) include surface materials (soil/rock) and sediments.

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 been 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**

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

*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. Table 3 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.

<table>
<thead>
<tr>
<th>Laboratory</th>
<th>Proposed Analyses Capabilities</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACZ Laboratories Inc.</td>
<td>All metals and general chemistry</td>
</tr>
<tr>
<td>7773 Downhill Drive</td>
<td></td>
</tr>
<tr>
<td>Steamboat Springs, CO 80487</td>
<td></td>
</tr>
<tr>
<td>800-334-5493</td>
<td></td>
</tr>
</tbody>
</table>
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

<table>
<thead>
<tr>
<th>Sample Media</th>
<th>Analyte</th>
<th>Analytical Method</th>
<th>Target Method Detection Limit (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soil</td>
<td>Uranium, total</td>
<td>EPA 3050/EPA 6020</td>
<td>0.5 mg/Kg</td>
</tr>
<tr>
<td></td>
<td>Lead 210</td>
<td>EPA 3050/EPA 909.0</td>
<td>5.0 pCi/g</td>
</tr>
<tr>
<td></td>
<td>Radium 226</td>
<td>EPA 901.1</td>
<td>1.0 pCi/g</td>
</tr>
<tr>
<td></td>
<td>Gamma Emitting Radionuclides</td>
<td>EPA 901.1</td>
<td>&lt;1.0 pCi/g</td>
</tr>
<tr>
<td></td>
<td>Radium 226 (onsite)</td>
<td>NaI-based Gamma Spectroscopy</td>
<td>1.0 pCi/g</td>
</tr>
<tr>
<td></td>
<td>XRF Metals</td>
<td>EPA Method 6200</td>
<td>TBD*</td>
</tr>
<tr>
<td>Sediment</td>
<td>Chromium, total</td>
<td>EPA 3050/EPA 6020</td>
<td>0.5 mg/Kg</td>
</tr>
<tr>
<td></td>
<td>Manganese, total</td>
<td>EPA 3050/EPA 6020</td>
<td>0.5 mg/Kg</td>
</tr>
<tr>
<td></td>
<td>Selenium, total</td>
<td>EPA 3050/EPA 6020</td>
<td>0.5 mg/Kg</td>
</tr>
<tr>
<td></td>
<td>Uranium, total</td>
<td>EPA 3050/EPA 6020</td>
<td>0.5 mg/Kg</td>
</tr>
<tr>
<td></td>
<td>Vanadium, total</td>
<td>EPA 3050/EPA 6020</td>
<td>0.5 mg/Kg</td>
</tr>
<tr>
<td></td>
<td>Lead 210</td>
<td>EPA 3050/EPA 909.0</td>
<td>5.0 pCi/g</td>
</tr>
<tr>
<td></td>
<td>Radium 226</td>
<td>EPA 901.1</td>
<td>1.0 pCi/g</td>
</tr>
<tr>
<td></td>
<td>Uranium 234</td>
<td>EPA 3050/EPA 908.0</td>
<td>0.4 pCi/g</td>
</tr>
<tr>
<td></td>
<td>Uranium 238</td>
<td>EPA 3050/EPA 908.0</td>
<td>0.4 pCi/g</td>
</tr>
<tr>
<td></td>
<td>Gamma Emitting Radionuclides</td>
<td>EPA 901.1</td>
<td>&lt;1.0 pCi/g</td>
</tr>
<tr>
<td></td>
<td>XRF Metals</td>
<td>EPA Method 6200</td>
<td>TBD*</td>
</tr>
</tbody>
</table>

*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

<table>
<thead>
<tr>
<th>Method</th>
<th>QC Check</th>
<th>Minimum Frequency</th>
<th>Acceptance Criteria</th>
<th>Corrective Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>SW6020 (ICP-MS Metals)</td>
<td>MS tuning sample</td>
<td>Prior to initial calibration and calibration verification</td>
<td>SW6020 paragraph 5.8</td>
<td>Retune instrument then reanalyze tuning solution</td>
</tr>
<tr>
<td></td>
<td>Initial calibration (minimum 1 standard and a blank)</td>
<td>Daily initial calibration prior to sample analysis</td>
<td>If more than one standard is used, correlation coefficient must be ≥ 0.995; If applicable, correct problem and repeat initial calibration</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Calibration blank</td>
<td>Before beginning a sample run, after every 10 samples and at end of the analysis sequence</td>
<td>No analytes detected ≥ RL</td>
<td>Correct problem then analyze calibration blank and previous 10 samples</td>
</tr>
<tr>
<td></td>
<td>Initial calibration verification (Second source standard)</td>
<td>After initial calibration before beginning a sample run – at a concentration other than used for calibration</td>
<td>All analytes within ±10% of expected value</td>
<td>Correct problem then repeat initial calibration</td>
</tr>
<tr>
<td></td>
<td>Continuing calibration verification</td>
<td>After every 10 samples and at the end of the analysis sequence</td>
<td>All analytes within ±10% of expected value</td>
<td>Correct problem then repeat calibration and reanalyze all samples since last successful calibration</td>
</tr>
<tr>
<td></td>
<td>Low level calibration check standard (at or below RL)</td>
<td>Once per analytical batch prior to sample analysis unless multi-point (3+) calibration with low std at or below RL is performed</td>
<td>All analyte(s) with ± 50% of expected value</td>
<td>Correct problem then reanalyze</td>
</tr>
<tr>
<td></td>
<td>Linear range calibration (High) check standard</td>
<td>Every three months</td>
<td>Analyze ± 10% of expected value</td>
<td>Correct problem then reanalyze or re-set linear range</td>
</tr>
<tr>
<td>M6020: Method blank; M200.8: Lab reagent blank (LRB)</td>
<td>Initial calibration verification (Second source standard)</td>
<td>After initial calibration before beginning a sample run – at a concentration other than used for calibration</td>
<td>All analytes within ±10% of expected value</td>
<td>Correct problem then repeat calibration</td>
</tr>
<tr>
<td></td>
<td>Interference check solutions (ICS-A and ICS-AB)</td>
<td>At the beginning and end of an analytical run or once during an 12 hour period, whichever is more frequent</td>
<td>ICS-A: All non-spiked analytes &lt; RL unless they are a verified trace impurity from one of the spiked analytes; ICS-AB: Within ±20% of true value</td>
<td>Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples</td>
</tr>
<tr>
<td>M6020: Lab Control Sample (LCS); M200.8: Lab fortified blank (LFB)</td>
<td>Dilution test</td>
<td>Each matrix in a analytical batch (only applicable for analytes with concentrations &gt;100X MDL)</td>
<td>Field test (1+4) dilution must agree within ±10% of the original determination</td>
<td>Perform post digestion spike addition</td>
</tr>
<tr>
<td></td>
<td>Post digestion spike addition</td>
<td>When dilution test fails, or if an analyte’s concentration for all samples in a batch is less than 100X MDL</td>
<td>Recovery within 75-125% of expected results</td>
<td>Dilute the sample; reanalyze post digestion spike addition</td>
</tr>
<tr>
<td>M6020: Matrix Spike (MS)/MSD; M200.8: Lab fortified matrix (LFM)</td>
<td>Internal Standards (ISs)</td>
<td>Every sample</td>
<td>IS intensity within ±50-120% of intensity of the IS in the initial calibration</td>
<td>Perform corrective action as described in method SW6020, section 8.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>QC acceptance criteria, to be reported by lab</td>
<td>Recalculate results; locate and fix problem with system and then re-run demonstration for those analytes that did not meet criteria</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>QC acceptance criteria, to be provided by lab</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>QC acceptance criteria, to be provided by lab</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>QC acceptance criteria, to be provided by lab</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>QC acceptance criteria, to be provided by lab</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>QC acceptance criteria, to be provided by lab</td>
<td>None</td>
</tr>
<tr>
<td>Method</td>
<td>QC Check</td>
<td>Minimum Frequency</td>
<td>Acceptance Criteria</td>
<td>Corrective Action</td>
</tr>
<tr>
<td>--------</td>
<td>-----------</td>
<td>-------------------</td>
<td>--------------------</td>
<td>-------------------</td>
</tr>
<tr>
<td>SW6010 (ICP Metals)</td>
<td>Initial calibration (minimum 1 standard and a blank)</td>
<td>Daily initial calibration prior to sample analysis</td>
<td>If more than one standard is used, correlation coefficient must be ≤ 0.995</td>
<td>If applicable, correct problem and repeat initial calibration</td>
</tr>
<tr>
<td></td>
<td>Initial calibration verification (second source)</td>
<td>Daily after initial calibration</td>
<td>All analytes within ±10% of expected value</td>
<td>Correct problem then repeat initial calibration</td>
</tr>
<tr>
<td></td>
<td>Calibration verification (Instrument Check Standard – ICB, CCB)</td>
<td>After every 10 samples and at the end of the analysis sequence</td>
<td>All analyte(s) within ±10% of expected value and RSD of replicate integrations &lt;5%</td>
<td>Repeat calibration and reanalyze all samples since last successful calibration</td>
</tr>
<tr>
<td></td>
<td>Calibration blank</td>
<td>After every calibration verification</td>
<td>No analytes detected ≥ RL</td>
<td>Correct problem then analyze calibration blank and previous 10 samples</td>
</tr>
<tr>
<td></td>
<td>Low level calibration check standard (at or below RL)</td>
<td>Once per analytical batch prior to sample analysis unless multi-point (≥3) calibration with low std at or below RL is performed</td>
<td>All analyte(s) with ≥ 50% of expected value</td>
<td>Correct problem then reanalyze</td>
</tr>
<tr>
<td></td>
<td>Linear range calibration (high) check standard</td>
<td>Every three months</td>
<td>Analyte within ± 10% of expected value</td>
<td>Correct problem then reanalyze or re-set linear range</td>
</tr>
<tr>
<td></td>
<td>M6010: Method blank; M200.7: Lab reagent blank (LRB)</td>
<td>One per analytical batch</td>
<td>No analytes detected ≥ RL</td>
<td>Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank</td>
</tr>
<tr>
<td></td>
<td>Interference check solution (ICS) (All begin at or below RL)</td>
<td>At the beginning of an analytical run</td>
<td>Within ±20% of expected value</td>
<td>Terminate analysis; correct problem; reanalyze ICS; reanalyze all affected samples</td>
</tr>
<tr>
<td></td>
<td>M6010: Lab Control Sample (LCS); M200.7: Lab fortified blank (LFB)</td>
<td>One per analytical batch</td>
<td>QC acceptance to be provided by lab criteria</td>
<td>Correct problem then reanalyze; if still out, reprop and reanalyze the LCS and all samples in the affected batch</td>
</tr>
<tr>
<td></td>
<td>Serial Dilution test</td>
<td>Each new sample matrix, at least once per analytical batch (only applicable for analyte with concentrations &gt;50X MDL)</td>
<td>Fivefold (1+4) dilution must agree within ±10% of the original determination</td>
<td>Perform post digestion spike addition</td>
</tr>
<tr>
<td></td>
<td>Post digestion spike addition</td>
<td>When dilution test fails, or if an analyte’s concentration for all samples in a batch is less than 50X MDL</td>
<td>Recovery within 75-125% of expected results</td>
<td>Check for instrumental problem then reanalyze post digestion spike addition if appropriate</td>
</tr>
<tr>
<td></td>
<td>M6010: Matrix Spike (MS/MSD); M200.7: Lab fortified matrix (LFM)</td>
<td>One MS/MSD or LFM/LFMD per every 20 project samples/ matrix</td>
<td>QC acceptance criteria, to be provided by lab</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td>Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample</td>
<td>Once per analyst</td>
<td>QC acceptance criteria, to be provided by lab</td>
<td>Recalculate results; locate and fix problem with system and then rerun demonstration for those analyses that did not meet criteria</td>
</tr>
<tr>
<td></td>
<td>MDL study</td>
<td>Once per 12 month period</td>
<td>Detection limits established shall be ≤ ½ the RLs</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td>Results reported between MDL and RL</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
</tbody>
</table>
## Table 5: Analytical Method QC Specifications (Cont.)

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

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).

![Field Strip Gamma Reading Chart](image)

**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. A field duplicate sample will be collected at about 2% of sampling locations (e.g. about 2 field duplicates for every 75 samples). All field duplicates will be analyzed onsite, and at least 50% of field duplicates will also be analyzed offsite. For final status survey samples, 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. A field duplicate sample will be collected at about 2% of sampling locations (e.g. about 2 field duplicates for every 75 samples). All field duplicates will be analyzed onsite, and at least 50% of field duplicates will also be analyzed offsite. For final status survey samples, 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:

\[
RPD = \frac{\text{sample} - \text{duplicate values}}{\frac{\text{sample} + \text{duplicate values}}{2}} \times 100\%
\]

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:

\[
RER = \frac{|S_x - Dup|}{\sqrt{(S_x error)^2 + (Dup error)^2}}
\]

Where: 
- \(S_x\) = sample concentration in pCi/L
- \(S_x error\) = 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 25 field samples. If less than 25 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
- 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 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 (also detailed in Table 5):
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)

- Tracer or carrier data
- Counter control source
- Instrument background

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

Internal Field Audit Responsibilities

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 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 also 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 and run logs will be reviewed 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 (2004) for inorganic parameters. EPA (2004) 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. Radiological data will be validated according to MARLAP (2004). 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 2004) for:

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

The laboratory will provide a QC summary suitable for this level of review (described in Section B.8). 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 (2004) will be applied to the data:

<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>J</td>
<td>The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.</td>
</tr>
<tr>
<td>J+</td>
<td>The result is an estimated quantity, but the result may be biased high.</td>
</tr>
<tr>
<td>J-</td>
<td>The result is an estimated quantity, but the result may be biased low.</td>
</tr>
<tr>
<td>R</td>
<td>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.</td>
</tr>
<tr>
<td>U</td>
<td>Analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.</td>
</tr>
<tr>
<td>UJ</td>
<td>Analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.</td>
</tr>
</tbody>
</table>
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


Appendix 1
Standard Operating Procedures
<table>
<thead>
<tr>
<th>MIDNITE MINE SUPERFUND SITE</th>
</tr>
</thead>
<tbody>
<tr>
<td>REMEDIAL ACTION PROJECT</td>
</tr>
<tr>
<td>STANDARD OPERATING PROCEDURE</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>AS-SOP 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>REVISION: 01</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>DECONTAMINATION FOR</th>
</tr>
</thead>
<tbody>
<tr>
<td>FIELD SAMPLING</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>DATE:</th>
</tr>
</thead>
<tbody>
<tr>
<td>December 15, 2013</td>
</tr>
<tr>
<td>June 16, 2015</td>
</tr>
</tbody>
</table>
## 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


<table>
<thead>
<tr>
<th>MIDNITE MINE SUPERFUND SITE</th>
</tr>
</thead>
<tbody>
<tr>
<td>REMEDIAL ACTION PROJECT</td>
</tr>
<tr>
<td>STANDARD OPERATING PROCEDURE</td>
</tr>
<tr>
<td>AS-SOP 2</td>
</tr>
<tr>
<td>REVISION: 01</td>
</tr>
<tr>
<td>SURFACE MATERIAL AND</td>
</tr>
<tr>
<td>SEDIMENT SAMPLING</td>
</tr>
<tr>
<td>DATE: December 15, 2013</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>
# 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 m²) 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


<table>
<thead>
<tr>
<th>MIDNITE MINE SUPERFUND SITE REMEDIAL ACTION PROJECT STANDARD OPERATING PROCEDURE</th>
<th>AS-SOP 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAMPLE PROSSESSING</td>
<td>REVISION: 01</td>
</tr>
<tr>
<td>DATE:  December 16, 2013</td>
<td>December 16, 2013</td>
</tr>
<tr>
<td></td>
<td>June 16, 2015</td>
</tr>
</tbody>
</table>
## 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)
- Attachment S2 – Quality Assurance Project Plan (QAPP) (WME/SENES, 2013)
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/weighting 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


<table>
<thead>
<tr>
<th>MIDNITE MINE SUPERFUND SITE</th>
</tr>
</thead>
<tbody>
<tr>
<td>REMEDIAL ACTION PROJECT</td>
</tr>
<tr>
<td>STANDARD OPERATING PROCEDURE</td>
</tr>
<tr>
<td>AS-SOP 4</td>
</tr>
<tr>
<td>REVISION: 10</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>ONSITE GAMMA SPECTROSCOPY</th>
</tr>
</thead>
<tbody>
<tr>
<td>DATE: December 16, 2013</td>
</tr>
<tr>
<td>June 16, 2015</td>
</tr>
</tbody>
</table>
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)
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 to 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


| MIDNITE MINE SUPERFUND SITE | AS-SOP 5 |
| REMEDIAL ACTION PROJECT | REVISION: 01 |
| STANDARD OPERATING PROCEDURE | |
| FIELD PORTABLE X-RAY | DATE: December 15, |
| FLUORESCENCE PROCEDURES | 2013 |
| | 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

        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 GOLD+ (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.

<table>
<thead>
<tr>
<th></th>
<th>Whole body exposure: 5,000 mrem/yr</th>
<th>Extremities: 50,000 mrem/yr</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1,250 mrem/quarter</td>
<td>18,750 mrem/quarter</td>
</tr>
</tbody>
</table>

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 In-Situ XRF Measurement

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

<table>
<thead>
<tr>
<th>General Sample Supplies:</th>
<th>XRF Equipment and Supplies (obtained from the XRF manufacturer):</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless steel bowls</td>
<td>XRF instrument and mini lab kit</td>
</tr>
<tr>
<td>Stainless steel trowel or spoon</td>
<td>Sieves (60-mesh/0.2 mm)</td>
</tr>
<tr>
<td>Disposable plastic spoons</td>
<td>Mortar and pestle (ceramic)</td>
</tr>
<tr>
<td>Paper towels</td>
<td>Polyethylene sample cups, collar, and bottom</td>
</tr>
<tr>
<td>Toaster oven</td>
<td>X-ray window film (Mylar, or similar)</td>
</tr>
<tr>
<td>Deionized water</td>
<td></td>
</tr>
<tr>
<td>Alconox detergent (or similar)</td>
<td></td>
</tr>
<tr>
<td>Scrub brushes</td>
<td></td>
</tr>
<tr>
<td>Sample bags/containers</td>
<td></td>
</tr>
<tr>
<td>Field logbook</td>
<td></td>
</tr>
</tbody>
</table>

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

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:

\[
RSD = \left( \frac{SD}{\text{Mean conc.}} \right) \times 100
\]

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


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

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.
<table>
<thead>
<tr>
<th>MIDNITE MINE SUPERFUND SITE REMEDIAL ACTION PROJECT STANDARD OPERATING PROCEDURE</th>
<th>AS-SOP 6</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>REVISION: 01</td>
</tr>
<tr>
<td>GAMMA SURVEYS</td>
<td>DATE: December 15, 2013June 16, 2015</td>
</tr>
</tbody>
</table>
## 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 ...................................................................................................................... 32  
   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  

AS-SOP 6: Gamma Surveys  
Preliminary (63% Percent) Design  

December 2013 | June 2015  
Revision 10
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)
  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 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).

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 Nal 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


Appendix 2
Corrective Action Report Form
Appendix 3
Approved Laboratory Quality Assurance Plans

Due to their size, the Laboratory Quality Assurance Plans are included in the electronic submittal only.
Appendix 4
Laboratory Certification

Due to its size, the Laboratory Certification is included in the electronic submittal only.
Attachment S-3

Determination of Bedrock During Remedial Excavation