VP Soils Work Plan for the Colonie FUSRAP Site Vicinity Properties

By:

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ACRONYMS AND ABBREVIATIONS

AEC - Atomic Energy Commission
AHA - Activity Hazard Analysis
APP - Accident Prevention Plan and Activity Hazards Analysis
BMT - BMT Designers & Planners
CFR - Code of Federal Regulations
CISS - Colonie Interim (waste) Storage Site
cm - centimeter
cpm - count per minute
dpm - disintegration per minute
DOE - U. S. Department of Energy
DOT - U. S. Department of Transportation
FIDLER - Field Instrument for Detecting Low Energy Radiation
FUSRAP - Formerly Utilized Site Remedial Action Program
GET - General Employee Training in Radiation Protection
GM - Geiger Mueller Detector
GPS - Global Positioning System
IATA - International Air Transport Association
IDW - Investigation Derived Waste
IEM - Integrated Environmental Management, Inc.
LASP - Land Area Survey Program
LPM - Liters per Minute
m - meter
MARS - Multiple Award Radiological Services (Contract)
MDA - Minimum Detectable Activity
MDC - Minimum Detectable Concentration
mrem - Millirem
NaI(Tl) - Thallium-activated Sodium Iodide Detector
NIST - National Institute of Standards and Technology
NYSDEC - New York State Department of Environmental Conservation
NL - National Lead
NYSDOH - New York State Department of Health
NYSDOT - New York State Department of Transportation Construction Standards
pCi - picocurie
pCi/g - picocurie per gram
QA - Quality Assurance
QAPP - Quality Assurance Project Plan
QC - Quality Control
ROC - Radionuclide of Concern
RSO - Radiation Safety Officer
s - second
SSHP - Site-specific Safety and Health Plan
U - Uranium
U-238 - Uranium-238
USACE - U. S. Army Corps of Engineers
VP - Vicinity Properties

1 PROJECT DESCRIPTION

1.1 Introduction

Integrated Environmental Management, Inc. (IEM) was contracted by the U. S. Army Corps of Engineers (USACE) to provide radiological support under Contract No. W912DR-D-0022, "Multiple Award Radiological Services (MARS)." Delivery Order No. 0002 of that contract includes the acquisition of information sufficient to fill data gaps at two commercial properties in the vicinity of the Colonie FUSRAP site and confirm that the radiological status of those properties meets agreed-upon clean up criteria. This work plan, prepared in response to the scope of work for the Delivery Order, includes historical background, the objectives of the project, the project organization, a description of the planned field activities and additional supporting information (i.e., instrumentation, sample handing/analysis, waste management, quality assurance, site health and safety and reporting).

1.2 Facility Description

The Colonie FUSRAP Site, hereinafter referred to as "the Colonie Site" or "the Site," is located in the town of Colonie at 1130 Central Avenue, Albany New York. It consists of 11.2 acres surrounded by residential and commercial properties known as vicinity properties (VPs). A map showing the location of the Site and the two commercial VPs is presented in Figure 11.1. Figure 11.2 is an aerial photograph of the general area which shows the current physical conditions in the vicinity of the Site.

1.3 Facility History

Industrial operations at the Site began in 1923 when a facility was built for manufacturing wood products and toys. In 1937, National Lead (NL) purchased the facility for conducting electroplating operations. In 1958, the nuclear division of NL began producing items manufactured from uranium and thorium under a license issued by the Atomic Energy Commission (AEC) and New York State.

The New York State Supreme Court shut down the NL plant in 1984 due to environmental concerns, at which time ownership was transferred to the U. S. Department of Energy (DOE). In 1980, the DOE surveyed the VPs surrounding the NL plant and determined that uranium released into the air during former operations deposited on nearby residential and commercial properties and structures. They also found the preponderance of the deposition in the direction of the area's prevailing winds.

In October 1983, the DOE performed more detailed radiological investigations of the individual VPs, with the objective of locating where uranium concentrations exceeded the remedial action guidelines agreed upon by the State of New York and the DOE. The DOE surveys identified 56 VPs requiring remedial action. In 1984, 1985 and 1988, 53 of the VPs were remediated, Certification Dockets were prepared attesting to their radiological status, and all contaminated material from remediation activities was staged on-Site pending disposal. The remaining VPs, the Niagara Mohawk Substation VP, the Town of Colonie VP, and the CSX VP, were addressed by the USACE along with the main site. Remediation took place at the CSX and Town of Colonie VPs and no action was required at the Niagara Mohawk VP.

1.4 Contaminant Identification

Former operations at the Colonie Site involved the use of uranium. Therefore the radionuclide of concern (ROC) for this project is primarily Uranium-238 (U-238) with progeny not fully in secular equilibrium.

The previously approved remedial action guideline for the VPs impacted by former Site operations is 35 picocuries of U-238 per gram (pCi/g) with the following isotopic distribution: 83.2% U-238, 1.11% U-235 and 15.7% U-234 (Shaw, 2010). Therefore, the established site release criteria applicable to this investigation are:

- U-238 - 35 pCi/g
- U-235 - 0.5 pCi/g
- U-234 - 6.5 pCi/g

1.5 VP Background

The Baltimore District of the USACE, performed an independent review of the remedial activities at the aforementioned 53 VPs in order to verify data adequacy and confirm compliance with the remedial action guidelines as agreed upon in an Action Memorandum (USACE, 2010; Shaw, 2010). The USACE review identified data gaps for the VPs located at 50 Yardboro Avenue and at 1118 Central Avenue (see Figures 11.1 and 11.2 for their location with respect to the Site). The following subsections further describe the two VPs and their status.

1.5.1 Property at 50 Yardboro Avenue

The DOE Certification Docket for this property indicates detectable uranium exists on the back portion, extending to the remaining CSX Rail VP. Radioactivity allegedly reached this location via a storm water outfall that ran below the CSX Rail VP and discharged at the 50 Yardboro Avenue property boundary. Although the outfall no longer exists, the Certification Docket recommended further investigation of its former location during any future CSX VP removal actions. As a result, there is a data gap that must be closed before the 50 Yardboro Avenue property can be released. Appendix A contains recent (2010) photographs of the property and the general location of the former outfall.

1.5.2 Property at 1118 Central Avenue

This property is comprised of a building surrounded on either side by an asphalt surface (i.e., parking lots). The DOE's limited remedial action on accessible soil, followed by post remedial action samples, demonstrated compliance with the remedial action guidelines. However, the DOE's Certification Docket reported elevated gamma radiation readings over the asphalt surfaces. The source, believed to be natural radioactivity in asphalt base materials, was supported by analytical results demonstrating isotopic ratios that did not match those from former Colonie Site materials. However, these results were not presented in the Certification Docket, thus the data gap must be closed before the 1118 Central Avenue property can be released. Appendix A contains recent (2010) photographs of the property showing the building and the asphalt surfaces.

1.6 Project Objectives

The objective of the investigation of the 50 Yardboro Avenue VP is to assess residual radioactivity concentrations at the location of the former outfall and confirm whether it is suitable for release with respect to established site release criteria. The specific area of interest is the back portion of the property in the general vicinity of the remaining CSX Rail VP. The property itself hosts an active auto body shop.

For the 1118 Central Avenue VP, residual radioactivity in soil above the established site release criteria is not expected. Therefore, the objectives with respect to this VP are to confirm the DOE's finding that the source of elevated count rates over the asphalt surface that surrounds the building

on the property (an active restaurant operation) is natural radioactivity in bedding materials, and that the property is suitable for release for unrestricted use.

The provisions of this work plan will be implemented, the VPs will be subject to surveys and sampling as described herein, data will be reviewed/validated/analyzed, and a report of findings will be prepared. Included in the report will be a determination as to whether the 50 Yardboro Avenue VP is eligible for release, and a finding with respect to the source of the elevated count rates over the asphalt surfaces of the 1118 Central Avenue VP.

2 PROJECT ORGANIZATION

2.1 Key Project Personnel

The organization tasked with investigating the radiological status of the two VPs is shown in Figure 11.3. The following is a listing of key project personnel and a brief summary of their responsibilities:

- USACE Project Manager - James Moore, who serves as the USACE Project Manager, is responsible for the execution of the project objectives and management of the IEM Team.

- USACE Contracting Officer Representative - Sesh Lal, who Serves as the Contracting Officer Representative, is the liaison with the USACE Contracts Office and is responsible for facilitating contract-related issues.

- USACE Design Team Leader - Phyllis Della-Camera, who serves as the Design Team Leader, is the technical liaison with the Project Manager, responsible for direct management of the IEM Team, and for ensuring the quality of products/deliverables through technical review and oversight. She is also responsible for obtaining and providing design review and field support personnel for the project on an "as needed" basis.

- USACE Project Health Physicist - David Watters, who serves as the Project Health Physicist, provides technical input to the project team and radiation safety oversight.

- MARS Program Manager - Carol Berger, who serves as the MARS Program Manager, is responsible for ensuring project objectives are met, that expenditures are within the project budget, and that the quality of submittals meets applicable objectives.

- Project Manager - R. Alan Duff, who serves as Project Manager, is responsible for planning, coordinating, integrating, monitoring, and managing project activities. He is also responsible for managing the project budget and schedule, working with the Quality Assurance Manager to ensure procedural compliance for all tasks, and serves as the primary point of contact with the USACE.

- Quality Assurance Manager - Cathryn N. Chang, who serves as the Quality Assurance Manager for the project, is IEM's Quality Assurance Officer. She is responsible for the development and implementation of quality control measures in planning documents and during implementation, and for ensuring data quality objectives are met.

- Field Site Manager - Jennifer Gutierrez, who serves as Field Site Manager, is responsible for organization, scheduling, and implementation of field activities for the project. She is the primary point of contact for all on-site personnel, responsible for the activities of the field team/subcontractors, and the preparation and submittal of Daily Quality Control Reports. In addition, she ensures that all field activities are completed safely, efficiently, in compliance with applicable requirements, and in accordance with the provisions of this work plan and the Quality Assurance Project

Plan (QAPP). Ms. Gutierrez also serves as the Site Radiation Safety Officer (RSO) for all on-site activities (see below).

- Site Radiation Safety Officer - The Site RSO (Jennifer Gutierrez, see above) is responsible for ensuring the radiological safety of all field activities and has authority to direct such activities, to stop and restart work if necessary, and to take appropriate actions, as required, to address radiological emergency situations. She also ensures all survey and sampling activities are performed pursuant to the pertinent provisions of this plan and the QAPP.

- Site Quality Control Officer - The Site Quality Control Officer (Patrick Phillips, see below) is responsible for ensuring the provisions of the QAPP are implemented during acquisition of measurement data and sample collection.

- Site Safety and Health Officer - Patrick T. Phillips is responsible for ensuring the Accident Prevention Plan (APP) and site-specific health and safety plan is followed and that Site personnel are appropriately trained in its provisions. He has authority to issue stop work orders for any on-site task he believes to be unsafe. When so stopped, work shall not re-start until the Site RSO and the Project Manager approve the re-start.

The positions shown in Figure 11.3 and described above are not all-inclusive. Additional project personnel needed to meet the objectives will participate as needed and authorized by the Project Manager. The qualifications of all key personnel will be captured in the final project report.

2.2 Personnel Training

All personnel participating in the on-site portion of the investigation will receive General Employee Training in Radiation Protection (GET). Included in GET will be:

- The type and form of radioactive material present at the VPs;

- The location of the IEM Radiation Protection Program Plan;

- Individual and management responsibilities for radiation safety;

- Identification of radiation postings and barriers; and

- Radiation-related emergency procedures, industrial safety, and other items specified in the APP.

All on-site project participants will have successfully completed the 40-hour HAZWOPER training course (and current annual refresher training), to include three days of documented field experience under the direct supervision of a trained experienced supervisor. All senior project staff with on-site management responsibilities will be current on supervisory training as required in 29 CFR 1910.120(e)(4). At least two members of the on-site team will be first aid- and CPR-qualified.

2.3 Visitor Training

Visitors to the VPs during on-site activities will be instructed in the type and magnitude of the radiological and other hazards they might face. The daily report generated by the Field Site

Manager will contain signed visitor briefing forms that will also serve as a visitor log, described further in Section 8 and the APP (see Appendix B).

3 FIELD ACTIVITIES

3.1 Approach for the 50 Yardboro Avenue VP

Upon mobilizing to the Site, the work area at the 50 Yardboro Avenue VP will first be cleared for utilities by contacting *Dig Safely New York* for utility mark-out. Following utility clearance, the fencing along the northern perimeter and possibly along a portion of the western perimeter of the study area will be removed. Equipment and materials that may be in the way of the investigation, such as those evident in the Appendix A photographs, will be relocated. Every effort will be made to ensure investigation activities do not unduly damage or disrupt the property.

When the work area has been cleared, a material staging area as described in Section 3.2.1, below, will be set up and the investigation will follow a three-phased approach:

- Phase 1 - The first phase will involve the use of a Field Instrument for Detection of Low Energy Radiation (FIDLER), a hand-held radiation detection instrument, to confirm the outline of the study area by scanning, described further in Section 3.1.2, below. With the sensitive portion of the FIDLER held approximately two inches from the ground surface, this device is capable of detecting residual radioactivity in concentrations well-below the established site release criteria.

- Phase 2 - The second phase will utilize an ultra-compact excavator (i.e., Kubota Model K008-3 or equivalent) to remove surface soil from the delineated area in small lifts (approximately six to eight inches) to permit access to the soil below for surveying. The lifts will be placed directly into supersacks positioned at the material staging area and the area will then be re-surveyed to assess general contaminant levels with respect to the established site release criteria. This process will be repeated until the FIDLER scans indicate no residual radioactivity above the established site release criteria (see Section 3.1.2, below).

- Phase 3 - Once Phase 2 is complete, staged soil will be readied for eventual disposal as described in Section 6. All test equipment will be decontaminated, surveyed and released as described in Section 3.2.5, and the material staging area will be disassembled, surveyed and released. The third phase of the investigation will then be to perform a final status survey of the study area as described in Section 3.1.3, below. This will include a 100% walkover survey (using a FIDLER detector) and sample collection/analysis of the study area footprint and side walls, and the material staging area footprint. The entire area will make up a single MARSSIM Class 1 survey unit.

The survey and sampling frequency for the three phases of the 50 Yardboro Avenue VP investigation, including measurement action levels, are given in Table 10.1. When all measurement results have been received, validated and analyzed, and if they demonstrate the survey unit meets the established site release criteria to a reasonable degree of scientific certainty, the excavated area will be backfilled and the property restored as described in Section 3.2.4, below.

3.1.1 Preparation Activities

A reference (background) area will be designated on the opposite corner of the 50 Yardboro Avenue VP from the area of interest for use in acquiring background count rates for the hand-held instruments as part of the daily checks. Background information, in concert with calculated

instrument efficiencies, will be used to set the count rates and action levels for delineating the study area.

A land area contiguous to the study area of interest will be identified to serve as the material staging station. A pre-operational survey of this area using a FIDLER detector held in close proximity to the ground surface will be performed and documented before a liner/cover is installed and other contamination control features implemented. Caution tape will be posted around the perimeter of the work area (comprised of the study area, the material staging area and any material movement pathways) to deter unauthorized personnel access.

As soil lifts are removed to facilitate surveying, water from hand-held sprayers will be used to control fugitive dust. In addition, an air sampler will be deployed in the immediate vicinity of the excavation equipment, with a second sampler deployed farther away in a down-wind location. A F&J Model LV-1 or Model LV-14M Low Volume Air Sampler (or equivalent), with a typical flow range of 0.35 to 3.5 cubic feet per minute (10 to 100 liters per minute), will be used to draw air through a 47 mm glass fiber filter. For short duration activities, where a low volume sampler cannot draw sufficient air to produce a meaningful result, a high volume sampler, such as a F&J Model HV-1 sampler (50 LPM) or equivalent will be deployed.

The filters from the samplers will be collected at the end of each work shift and counted for gross alpha/beta activity using a Ludlum Model 2929 digital scaler with a 43-10-1 dual alpha/beta detector (or equivalent). Filters with detectable activity will be held and recounted once per day over several days in order to account for the radon progeny contribution. Those with count rates that remain elevated after one week will be forwarded to an off-site analytical laboratory for radionuclide identification, with results compared to the effluent release limits in 10 CFR 20, Appendix A, Table 2, Column 1. All analytical methods will have sufficient detection sensitivity to permit ready detection of the effluent release limits.

One-cubic-yard supersacks will be placed at the material staging area to hold soil removed from the study area. The supersacks will eventually be transferred to the Colonie Interim Storage Site (CISS) pending disposal. Equipment decontamination will be performed as described in Section 5.4, below.

3.1.2 Scanning Surveys and Soil Relocation

Scanning surveys using a FIDLER detector held approximately two inches above the ground surface will be performed to delineate the footprint of the study area (see Section 4 for additional instrument information and detection sensitivities for the scans). To ensure an element of conservatism in the process, the delineating count rate for the FIDLER detector will be determined as follows:

$$C_R = (G \times 0.75) \times 110 \approx 2{,}900\ cpm \quad (1)$$

where C_R = the delineating count rate (cpm) above background, G = the established site release criteria (e.g., 35 pCi/g of U-238) and 110 is a factor to convert G into instrument response (cpm per pCi/g) (Noce, 2000). However, geometry effects associated with scanning in an excavation depression could impact instrument response and/or background. If such effects are identified in the field, C_R will be updated as necessary. The footprint of the survey will extend one (1) foot beyond the measured C_R border.

After soil lifts are placed into the supersacks, the scans will be repeated and the survey unit boundary adjusted accordingly. Lifts and surveys will continue until the entire surface and side walls of the study area exhibit count rates that are below C_R.

3.1.3 Final Status Surveys

When the scans are complete (i.e., scanning survey count rates ≤ C_R), the material staging area will be disassembled and removed, and a final status survey of the work area (comprised of the study area, the material staging area and any material movement pathways), will be performed. A final status survey plan for the VPs has been previously approved, thus the VP survey will be performed in accordance with the provisions of that plan (IT, 2000).

IEM's Land Area Survey Program (LASP) will be used to provide 100% coverage of the survey unit, including the side walls of the excavation. The LASP uses a global positioning system for precise data acquisition, coupled to a radiation detector (i.e., FIDLER) and rate meter. Position, time and instrument read-outs are acquired once per second and stored for later processing.[1] This will be followed by collection and analysis of one surface soil sample (i.e., to a depth of six inches) at each of 15 systematic locations, plus one surface soil sample at the location that exhibits the highest count rate during the walkover survey, for a total of 16 samples from the survey unit.

The 15 equal-distant systematic sampling locations will be sited over the survey unit as specified in the previously-approved plan (IT, 2000). Figure 11.4 shows the projected location and size of the single survey unit, which is assumed to be comprised of approximately 7.2 square meters for the study area and approximately eight square meters for the material staging area. Figure 11.5 shows an exploded view of the survey unit (footprint plus side walls for the excavation and the material staging area footprint), 15 systematic sample collection locations and one biased location that will be positioned at the point of maximum measured count rate during the walkover survey.

The relative distance between each sampling location will remain as shown in Figure 11.5, although actual distances will be adjusted according to the final dimensions of the survey unit. In the unlikely event the footprint of the survey unit exceeds 2,000 square meters, it will be subdivided into two or more sampling areas, each with dimensions of no greater than 2,000 square meters. The 15 sampling locations will then be proportionally spaced as shown in Figure 11.5.

All samples will be forwarded to an off-site laboratory for analysis by gamma spectroscopy with a 24-hour turn around time on draft results to facilitate quick closure of the open excavation. All sample collection locations will be tied to GPS coordinates (State Plane NAD83 New York East coordinate system) for future re-location as required, recorded on a sample collection log, and photographs of the area will be taken as described in Section 3.4.2, below.[2]

3.1.4 Area Restoration

After the final status surveys are complete, the study area and its approaches will be backfilled and restored. Based on prior reconnaissance of this locale, the yard at 50 Yardboro Avenue is comprised of primarily compacted gravel. Therefore, crusher run/dense grade aggregate materials will be secured from a local road construction supply firm to create the subbase strata of backfill necessary to return the area to its pre-investigation condition. Following the placement and compaction of the backfill, a finish course of aggregate stone will be deposited and spread over the area to return the surface to an operable setting.

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[1] For more information on the LASP, see http://www.iem-inc.com/iemland.html.

[2] Individual data points can be located and re-located to as close as six inches, in most cases, after post-processing is complete.

New York State DOT construction standards (NYSDOT, Sections 203 and 305) apply to public vehicular rights-of-way. However, the yard at 50 Yardboro Avenue is not a right-of-way, therefore, the planned improvements, which will be consistent with the NYSDOT standards, will exceed typical restoration of common excavation tasks.

If, upon closer inspection the final location of the study area is not like the remainder of the gravel covered yard, modifications to the composition of the backfill materials will be made to match pre-construction condition (i.e., if the area is comprised of grass-covered soil, it will be returned to the same standard). In any case, an appropriate sub-base course of soil will be installed and compacted, then backfilled with soil from a local supplier, followed by compaction using either the excavation backhoe or a hand-operated compactor. The top six inches of fill will be a top soil mixture for re-seeding, which is then seeded and covered with a means of erosion control (i.e., jute matting). Once the yard surface is restored, a local fencing contractor will reinstall the sections of fence that were removed to accommodate the investigation.

3.2 Approach for the 1118 Central Avenue VP

From work performed previously by the DOE at the 1118 Central Avenue VP, residual contamination from former Site operations is not expected. Instead, the issues are whether the source of elevated count rates the DOE observed over the asphalt parking lots can be attributed to natural radioactivity in the bedding material, and if the property is eligible for release for unrestricrted use. To confirm these conclusions, a walkover survey will be performed to assess surface count rates, samples of asphalt bedding and underlying soil will be collected on a MARSSIM Class 1 sampling density, and results will be used to assess the release status of the survey unit pursuant to MARSSIM. The investigation of this VP will follow a two-phased approach:

- Phase 1 - A GPS-guided radiation survey of all accessible asphalt-covered surfaces of the property, using a FIDLER detector held approximately two inches from the surface, will be performed. Locations exhibiting elevated count rates with respect to the survey area as a whole will be marked for further investigation as described in Section 3.2.2, below.

- Phase 2 - Once the GPS-guided survey is complete, borehole studies will be performed as described in Section 3.2.3, below. Analytical results for isotopic uranium will be compared to the established site release criteria. These will include a combination of statistically-based and biased stationary measurements, collection of soil cores using a Geoprobe, scans of cores to facilitate sample (i.e., soil and asphalt/bedding) selection and "down hole" measurements as described in Section 3.2.3.2, below. The sampling frequency will be equivalent to a MARSSIM Class 1 survey unit as described in the previously-approved final status survey plan for the VPs (IT, 2000).

The survey and sampling frequency for the two phases of the 1118 Central Avenue VP investigation, including measurement action levels and instrument detection sensitivities, are shown in Tables 11.1 and 11.2. A Class 1 sampling density has been applied thus a correlation between the analytical results, the scanning results and the stationary measurements may be performed if so desired.

Once the borehole studies are complete, residual core cuttings will be readied for eventual disposal as described in Section 6. All test equipment will be decontaminated, surveyed and released as described in Section 3.3, below, and the material staging area will be disassembled, surveyed and

released. If the survey and borehole study results do not demonstrate the source of the elevated count rates over the asphalt surface of this VP to be the asphalt bedding, additional action may be taken as authorized by the USACE Project Manager.

3.2.1 Preparation Activities

Upon mobilization to the 1118 Central Avenue VP, a full utility clearance process, similar to that for the 50 Yardboro Avenue VP, will be conducted to ensure sub-surface activities do not conflict with utilities. Once the clearance process is completed, a soil core processing station will be set up and the team will initiate the surveys, boring operations and sample collection. For the daily instrument response checks, background data will be acquired for the survey instruments over a sidewalk immediately adjacent to the VP. At each boring location, caution tape will be erected around the work area to deter unauthorized personnel from entering the work area. Equipment decontamination will be performed as described in Section 3.3, below.

3.2.2 GPS-Guided Survey

IEM's LASP, coupled to a FIDLER detector, will be used to perform the walk-over scans and stationary measurements at the 1118 Central Avenue VP (see Section 4 for additional instrument details). The survey data will be acquired by walking over 100% of the accessible asphalt-covered area of the property with the sensitive portion of the detector held at a distance of approximately two inches above the ground surface. As the surveyor progresses, marks will be made on the ground using a long-handled paint wand (or equivalent) to facilitate tracking of paths. Locations that exhibit count rates that are readily distinguishable from the general area count rates will be marked for biased borehole studies as described in Section 3.2.3, below. When the surveys are complete, the data from the survey unit will be transferred directly into a computer-based Geographical Information System (GIS) that displays them in tabular form for statistical analysis, and in map form that will be laid over a photographic image of the property.

3.2.3 Borehole Studies

3.2.3.1 Borehole Siting and Installation

Borehole studies will be performed at systematically-placed locations using the triangular grid pattern specified in the previously-approved final status survey plan for the VPs. The distance between points (i.e., spacing) will be determined as follows (IT, 2000):

$$d = \sqrt{\frac{A}{0.866 \times n}} \qquad (2)$$

where d = the distance between borings (m), A = the investigation site area (m^2) , and n = the required number of borings (i.e., 15) (IT, 2000). The systematic measurement/survey pattern is shown in Figure 11.6, along with the proposed location of the soil core processing stations. Vehicles will access the study areas via the two driveways with routes that are subject to property owner approval and the amount/distribution of parked cars in the lot.

The location of maximum count rate from the GPS-guided survey will also be marked for borehole studies, thus a total of 16 boreholes will be sited. If a systematic point is located on an identified subterranean utility (i.e., electric line, gas line), it will be moved to the closest alternative location. The final location of each measurement/sampling point will be tied to GPS coordinates for future re-location as required, recorded on a sample collection log, and photographs of the area will be taken as described in Section 3.4.2, below.

At each of the 16 boring locations, a stationary measurement will be performed using a FIDLER detector positioned approximately two inches above the ground surface. Borings will then be advanced with a two-inch diameter Macro-Core® equipped with a removable acetate liner and core catcher.[3]

The boreholes will be advanced to a depth of four (4) feet using two cores. The first core will be advanced through asphalt and associated bedding material to a depth of approximately one foot or until natural soil or fill is encountered. The second core will be advanced from one foot to the terminal depth of four feet. A two-core approach will allow for better accuracy in segregating the asphalt bedding material, prevent smearing of residual activity through the length of the core itself, and maximize the overall recovery of soil and bedding from each boring location.

After each boring, the core cutting shoe and sampler (Macro-Core® assembly) of the Geoprobe will be cleaned with water and Alconox (power soap), then spray-rinsed with distilled water. Liquid waste will be captured in five-gallon plastic buckets with sealable lids and transferred to metal drums (or other approved containers) for consolidation, sampling and disposal (see Sections 6 and 7, below). Radiation surveys of accessible areas will be performed in order to release the equipment at the end of the project. Sample cuttings and other investigation-derived waste (IDW) from the boreholes will be containerized and prepared for eventual disposal as described in Section 6.

3.2.3.2 Core Scanning and "Down Hole" Measurements

Cores removed from the boreholes will be placed in a jig and scanned using a hand-held sodium iodide detector (Ludlum Model 44-10 or equivalent) positioned at a distance of approximately 0.5-inch from the core. That portion of the cores that exhibits the highest response will be packaged and forwarded to an off-site laboratory for analysis (see Section 3.2.3.3, below).

After each core is removed, a small-diameter (i.e., approximately one inch) sodium iodide detector (Ludlum Model 44-2 or equivalent) will be inserted into the hole and the count rate will be recorded every six inches of depth (i.e. eight measurements per hole). This qualitative information, when used in concert with the core scans and analytical results, will provide activity distribution information in the event future actions at this VP are necessary.

3.2.3.3 Analysis of Samples

The portion of each soil core that exhibits the highest count rate will be analyzed for the concentration of uranium isotopes in order to confirm isotopic ratios. The asphalt bedding portion of the cores will also be scanned and that portion exhibiting the highest response will forwarded for analysis by gamma spectroscopy. The bedding samples will be prepared (i.e., dried, ground), sealed into counting containers and held for 21 days to permit the short-lived uranium/radium progeny to return to secular equilibrium before analysis. The size of both soil and bedding samples sent to the laboratory will be approximately 500 grams. All analytical results will be compared to the established site release criteria in accordance with MARSSIM.

[3] This collection device allows for the collection of a complete soil core and subsequent screening and sampling at discrete depth intervals.

3.2.4 Area Restoration

When the borehole study is complete, each boring will be backfilled to grade with bentonite pellets.[4] Asphalt penetrations will then be filled with comparable (i.e., "in kind") paving materials, compacted to pavement level and allowed to set. Photographs depicting repairs made to the asphalt surface will also be taken as described in Section 3.4.2, below.

3.3 Equipment Decontamination and Release

Equipment used for the investigation of the 50 Yardboro Avenue and the 1118 Central Avenue VPs will remain within the designated work areas while in use. Before exiting the work area, they will be brushed off to remove soil to the greatest extent practical, then surveyed for residual gross activity in accessible areas, with results compared to industry-standard surface release criteria for uranium isotopes of 5,000/1,000 disintegrations per minute (dpm) per 100 square centimeters of total/removable alpha and beta radiation, with the limits applied independently (USNRC, 1974).[5] If not releasable, they will be decontaminated and re-surveyed or disposed of as radioactive waste.

The following equipment and materials will be present in the work area for physical decontamination of equipment in general and sample collection gear in particular:

- Disposable gloves;
- Laboratory-grade, non-phosphate detergent;
- Distilled water;
- Aluminum foil;
- Scrub brushes;
- Five-gallon plastic decon fluid wash/rinse buckets;
- Sealable plastic or metal containers for capture/transport of spent decon liquids;
- Metal spoons to facilitate transfer of solid samples into containers;
- "Drop cloth" to collect drips, splatter, and other errant decon fluids in/around the decon area; and
- Laboratory sample collection containers for required equipment blanks.

[4] Bentonite is a clay used in geotechnical applications because of its absorbent and swelling properties, making it useful for sealing holes and gaps.

[5] See Table 1 of USNRC Regulatory Guide No. 1.86, USNRC Policy and Guidance Directive No. FC 83-23, "Termination of Byproduct, Source and Special Nuclear Material Licenses", and NUREG-1757 ("Consolidated Decommissioning Guidance), Vol. 1, Section 15.11.1.1. These values apply to radioactive contamination deposited on but not incorporated into the interior of equipment.

Equipment decontamination will be performed as follows:

- Vigorously scrub equipment in laboratory-grade, non-phosphate detergent/water solution.
- Thoroughly rinse with approved potable (distilled) water.
- Air dry.
- Wrap in aluminum foil or polyethylene sheeting (if item is not intended for immediate reuse).

Care will be taken when choosing the decontamination area to avoid fugitive dust, fuel, oils, gasoline, organic solvents, or any potential airborne source of contamination. All wash/rinse water will be commingled into a single container, with an aliquot collected and forwarded to an off-site analytical laboratory for isotopic uranium analysis. It will then be disposed of as described in Section 6, below.

3.4 Field Records

Project data will be recorded in a field log (bound and with numbered pages), a Field Activity Daily Log form, or equivalent method of data and information recording. The contents of the logs will subsequently be transferred to an electronic format for inclusion in the project records. Field logs will be reviewed by the Project Manager at least weekly and after significant events. Each entry into a log will be legible, factual, detailed, complete and will be signed and dated by the individual making the entry. If a mistake is made, the error will have a single line drawn through it, with the initials of the person making the correction written next to the line. No erasures or "white out" use is permitted.

3.4.1 Electronic Records

Much of this investigation will rely on data collected and stored electronically. Electronic data are subject to damage and/or loss if not properly protected. Therefore, all electronic data will be downloaded from its collection device (e.g., laptop computers, data loggers, etc.), or scanned if hard copy, on a daily basis and forwarded to the IEM server where there exists multiple levels of redundant storage/backup.

3.4.2 Photographs

Photographs will be made to assist in documenting on-site activities and for future reference. The Project Manager will secure approval from the USACE to collect photos, as necessary, and the Field Site Manager will maintain a photo log.

3.5 Community Relations

All inquires that pertain to the on-site activities for this project will be directed to the USACE (New York District) Project Manager, Mr. Jim Moore, at (917) 790-8230.

4 INSTRUMENTATION

4.1 Instrument Type

The radiation detection instruments used for the various measurements at the 50 Yardboro Avenue VP and the 1118 Central Avenue VP were selected and will be operated according to the type of measurement being performed. The following is a listing of instrument types (or equivalent) that will be used:[6]

- Soil Surface Scans (quantitative) - Field Instrument for Detecting Low Energy Radiation (FIDLER) with a beryllium entrance window and coupled to an RS-232-equipped rate meter and GPS system (Trimble GeoXH handheld GPS unit with Trimble's Pathfinder software). The detector dimensions are 126.7 square centimeters in area and 0.5 centimeters thick.

- Asphalt Surface Scans and Stationary Measurements (quantitative) - Two-by-two-inch sodium iodide detector (Ludlum Model 44-10 or equivalent) coupled to an RS-232-equipped rate meter and GPS system.

- Core Scans (qualitative) - Two-by-two-inch sodium iodide detector (Ludlum Model 44-10 or equivalent) coupled to a Ludlum Model 2241 rate meter.

- Down-hole Measurements (qualitative) - One-by-one-inch sodium iodide detector (Ludlum Model 44-2 or equivalent) coupled to a Ludlum Model 2241 rate meter.

4.2 Instrument Calibration

The instruments used during the measurement campaign will be calibrated by a licensed commercial calibration service using National Institute of Standards and Technology (NIST) traceable sources and calibration equipment, and will include:

- High voltage;

- Discriminator threshold;

- Window width;

- Alarm operation verification;

- Scaler (timing) calibration verification.

The calibration of detectors will include:

- Operating voltage determination;

- Calibration constant determination; and

[6] Instrumentation used to determine the contamination status of people and equipment for release purposes is described in the APP that appears in Appendix B.

- Dead time correction determination.

Labels showing the instrument serial number, calibration date or calibration due date will be affixed to all instruments. A copy of relevant calibration records will be captured in the project report (see Section 9, below).

4.3 Response Checks

Instrument response checks will be conducted to assure constancy in instrument response, verify the detector is operating properly, that its response is similar to its calibrated response, and to demonstrate that measurement results are not the result of detector contamination or failure. Instrument response will be checked each day before the instrument is used and whenever instrument performance is questioned. If the instrument fails a response check, it will not be used until the problem is resolved.

4.4 Check Sources

All sealed radiation sources used for daily instrument response checks will be representative of the instrument's response to the identified radionuclides and traceable to NIST. These sources will include Cesium-137 and Technetium-99 for photon and beta radiation, respectively. The Site RSO will control the use and storage of these radiation sources and will sign them out when needed. A source sign-out log will show the location of all sources when removed from the designated source storage area or out of the custody of the Site RSO.

4.5 Minimum Detectable Activity

Minimum Detectable Activity (MDA) is defined as the smallest amount or concentration of radioactive material that will yield a net positive count with a 5% probability of falsely interpreting background responses as true activity. For scans, the following equation will be used in the field to determine detection sensitivity:

$$MDA = \frac{MDCR}{\sqrt{p}\ \epsilon_i\ \epsilon_s\ \frac{probe\ area}{100\ cm^2}} \quad (3)$$

where MDA = Minimum detectable activity (pCi/g), p = surveyor efficiency (generally assumed to be 0.5 based upon MARSSIM, Section 6.7.2), ϵ_i = instrument efficiency, ϵ_s = surface efficiency, and MDCR = minimum detectable count rate, determined as follows:

$$MDCR = (d' \times \sqrt{b_i}) \times \frac{60}{i} \quad (4)$$

where d' = a value selected from Table 6.5 of MARSSIM for selected "true positive" and "false positive" proportions and b_i = the number of background counts in scan interval, "i".

Nominal instrument responses and MDAs for soil scans using FIDLER detectors were developed in the previously-approved final status survey plan. These are incorporated by reference into this work plan (IT, 2000).[7] Values for other instruments and survey methods were developed using the aforementioned equations. Table 10.2 contains the MDA estimates for the instruments that will be used for the 50 Yardboro Avenue VP and the 1118 Central Avenue VP measurements. Appendix

[7] In IT, 2000, the Appendix is missing from project record, therefore quoted MDAs accepted at face value.

C contains a more detailed presentation of the calculation methods, input parameters and results. The actual MDA for the instruments used in the field will be determined as part of the daily instrument response checks (see Section 4.3, above).

5 SAMPLE HANDLING AND ANALYSIS

5.1 Sample Handling and Packaging

The Field Site Manager will arrange for delivery of coolers, labels, metal spoons and sample containers to the project site prior to conducting field activities. The QAPP in Appendix D contains requirements for container types, custody seals, preservatives, holding times and Chain-of-Custody procedures.

Soil, asphalt bedding material and other solid samples will be transferred to collection containers using metal spoons. Sealable plastic or metal containers to capture/transport liquids into collection containers. All items will be decontaminated as described in Section 3.3, above.

Each sample will be marked and logged with an unique sample identification, sample depth, the date and time sampled, and the initials of the individual who obtained the sample. Collection locations will be recorded on a survey map and/or by GPS coordinates. The individual who collected each sample will be responsible for sample custody from the time of collection until transfer to a commercial carrier or until properly transferred to another sample team member.

Sample containers shipped together as a group will be assigned a single Chain-of-Custody Record, which will be included in the shipping containers. A signed copy of the Chain-of-Custody Record will be retained by the Field Site Manager after the coolers are sealed, along with the shipment air bill. The laboratory will be instructed to attach a copy of the fully-executed Chain-of-Custody Record to the Certificates of Analysis.

Sample labels, field notebook information, and Chain-of-Custody forms will be checked for accuracy in sample identification and to verify that all the required information has been supplied by the Site Quality Control Officer. When the Field Site Manager is ready to offer samples for shipment to the laboratory, the laboratory point of contact will be notified of the pending shipment and the estimated arrival date. No chemical analytical samples will be held on-site for more than 24 hours unless specific prior approval is obtained from the IEM Team's Environmental Manager.

5.2 Sample Shipment

Samples will be packed and shipped by overnight carrier in accordance with DOT regulations, and International Air Transport Association (IATA) standards, as detailed in the most current edition of IATA Dangerous Goods Regulations for hazardous materials shipments. Samples that are not considered to be high hazard will be shipped as environmental samples in "Excepted Quantities" and handled as follows:

- Place samples in a cooler and surround them with vermiculite (or equivalent) packaging material for stability during transport. Samples will be shipped with ice packs to meet standard EPA sample preservation protocols and temperature requirements, as applicable.
- Place completed Chain-of-Custody form inside a re-sealable plastic bag, and attach the bag to the inside of the cooler lid.
- Secure the cooler lid with packing tape.

- Place signed and dated custody seals on two opposite sides of the lid and secure with clear tape.
- Tape the cooler drain plug closed so it will not open during transport.
- Place upward-pointing arrow labels on the four vertical sides of the cooler.
- Label the cooler with the laboratory address, name of laboratory contact, telephone number, and project identification.
- Close and latch the cooler. Strapping tape will be used to secure the lid by completely wrapping the cooler at a minimum of two locations.
- Label as "Excepted Quantity" as per IATA regulations (if applicable).[8]

Shipments of materials that exceed "limited quantity" levels for radioactivity are not anticipated for this project. Radiological shipments, if required, will be performed in accordance with DOT regulations. Only personnel trained in accordance with 49 CFR 172, Subpart H, are authorized to ship radioactive materials. Samples that do not meet the definition of a radioactive material (and are not a hazardous material) as detailed in 49 CFR 173.403 do not require any special handling and will be shipped without regard for their radiological constituents.

5.3 Data Verification and Validation

To meet the quality assurance objectives for the investigation, all data collected from the VPs will be subject to verification and quality review. The QAPP in Appendix D contains the data review, verification and validation process for ensuring the data set is complete, reasonable and useable.

[8] At least one person on the field team will have completed the IATA dangerous goods shipping class.

6 WASTE MANAGEMENT

Excavated soil, core residuals, bedding material, asphalt and other items not submitted for laboratory analysis will be placed into one cubic yard supersacks. Other IDW (e.g., towels, trash, used PPE, etc.) will be sealed in plastic bags and placed into metal 55-gallon drums or supersacks as necessary. Decon fluids generated during site activities will be collected in 5-gallon plastic buckets, consolidated into a single container such as a 30-gallon drum and solidified. Waste packages will then be weighed and moved to the CISS for storage pending eventual disposal.

All containers will be weighed with a pre-calibrated portable scale staged on-site or other means as appropriate (i.e. obtain truck tare weight at a certified scale, load container onto truck and have the truck weighed again at a certified scale when loaded). Waste shipments over public roads (i.e., from the VPs to the CISS) will be treated as DOT hazardous shipments, thus only trained, qualified radioactive materials shippers and a properly licensed, trained, and insured transporters will execute the transfers.

Aliquots of material placed into each waste container will be collected, homogenized and a single composite sample (i.e., approximately 500 grams) submitted to an off-site laboratory for uranium isotopes analysis and TCLP metals (i.e., lead) for waste profiling purposes. The waste staged at the CISS will be shipped for disposal once characterization results are obtained and the waste profile completed. At this time, disposal at the U. S. Ecology facility in Idaho or the EnergySolutions facility in Utah is anticipated, with final selection dependent on characterization results.

7 QUALITY ASSURANCE

Quality assurance programs are designed to ensure that all quality and regulatory requirements applicable to a particular effort are identified and satisfied. Therefore, VP investigation all activities affecting quality will be controlled by this work plan and the QAPP that appears in Appendix D.

8 SITE HEALTH AND SAFETY

Safety and health procedures/practices for personnel involved in the project, including training requirements, have been established in an APP (see Appendix B). Included in the APP is APP is an Activity Hazard Analysis (AHA) for potential hazards that might be encountered during the performance of this project. Appendix S of the APP is a site-specific safety and heath plan (SSHP) that was developed to facilitate implementation of the APP in the field.

9 REPORTS

9.1 Routine Reporting

While on-site, the Field Site Manager will prepare a Daily Contractor Quality Control Report that includes daily Tailgate Safety Briefing forms. The Field Site Manager will also prepare a Weekly Contractor Quality Control Report. Reports will be submitted to the Project Manager, USACE Project Manager and the USACE Design Team Leader, with copies maintained in the project file.

9.2 Final Report

Once the on-site work is complete and all analytical results have been received and validated (see Appendix D for more on the validation process), an investigation report will be prepared that summarizes the investigation in its entirety. The contents of the report will include the following:

- Introduction (objective and scope);
- Project organization and personnel qualifications;
- Description of the Site;
- Radionuclides of concern (ROCs);
- Description of the on-site activities;
- Description of sampling procedures;
- Data compilation and summary, including quality control data and validation report;
- A comparison of data to the established site release criteria and an evaluation of the nature and extent of residual uranium within the property boundaries;
- Recommendations for follow-up action, if necessary; and
- Description of waste management activities, including shipping manifests and Certificates of Disposal.

10 TABLES

10.1 - Survey and Sampling Schedule

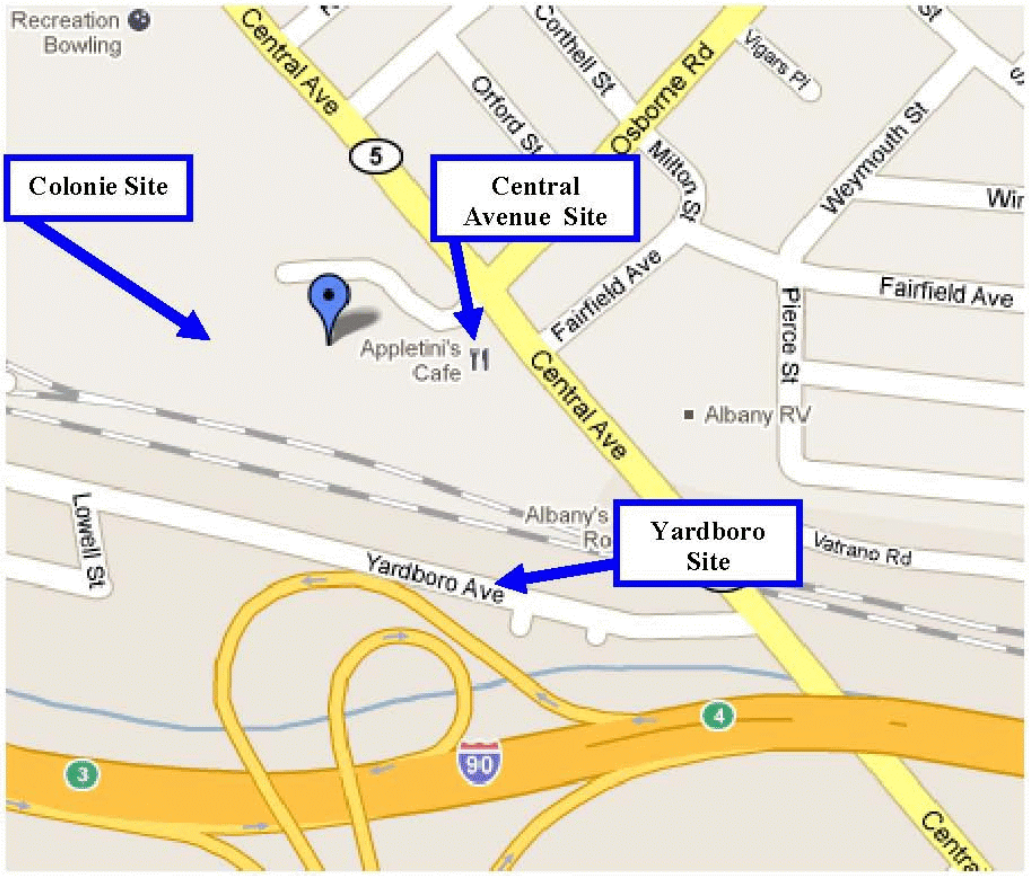
Collection Locations	Surveys Required	Number of Samples	Sampling Distribution	Depth	Analyses	MDA (pCi/g)	Action Level
50 Yardboro Survey unit	Walkover gamma survey (FIDLER)	--	100%	2-in. from ground surface	--	13	2,850 cpm (75% of equivalent established site release criteria)
	Co-location of measurement data (GPS)	--	100%	--	--	Post-processing accuracy to 6 in.	--
	Soil sample collection	15 (plus 1 biased, 1 QC dup for a total of 17)	Systematic (triangular grid)	0 to 6-in.	Gamma spec (24 hour turn around)	1.5	U-238 - 35 pCi/g U-235 - 0.5 pCi/g U-234 - 6.5 pCi/g
1118 Central Ave.	Walkover gamma survey (NaI(Tl))	100%	100%	2-in. from ground surface	--	266 (1-min count through 1-ft asphalt/bedding)	None (count rate determination only)
	Co-location of measurement data (GPS)	--	100%	--	--	Post-processing accuracy to 6 in.	--
	Stationary measurements (NaI(Tl))	15 (plus 1 biased for a total of 16)	Systematic (triangular grid)	2-in. from ground surface	--	87 (See Section 3.3)	None (count rate determination only)
	Soil sample collection	15 (plus 1 biased, 1 QC dup for a total of 17)	Systematic (triangular grid)	1 to 4 ft at each sample location	Iso-U (30-day turn around)	0.1 pCi/g	U-238 - 35 pCi/g U-235 - 0.5 pCi/g U-234 - 6.5 pCi/g
	Asphalt bedding material collection	15 (plus 1 biased, 1 QC dup for a total of 17)		0 to 1 ft at each sample location	Gamma spec (21-day ingrowth; 30-day turn around)	1.5 pCi/g	TBD
	Down-hole measurements	15 (plus 1 biased for a total of 16)	Every 6 inches (8 measurements per sample location)	0 to 4 ft at each sample location	--	n.a.	n.a.
	Scan of Geoprobe cores to identify highest reading for sample collection (NaI(Tl))	--	100%	1/2-in. from core surface. Scan over 0 to 1 ft for asphalt bedding; 1 to 4 ft for soil	--	n.a.	Segments with highest scan rates to be collected for analysis

10.2 - Instrumentation

Detector Type	Purpose	Type	Probe Dimensions	Primary Radiations Detected	Reference Source for Efficiency Determination	MDC (pCi/g ROCs) *see Appendix C*	
						Scanning	Stationary
FIDLER	Soil Scans	Quantitative	126.6 cm^2	< 200 keV Photon	Cs-137	13	4.1
NaI(Tl) Model 44-10 or equivalent	Asphalt Scans	Quantitative	2" x 2"	> 100 keV Photon	Cs-137	266	87
NaI(Tl) Model 44-2 or equivalent	Down-hole Measurement	Qualitative (distribution)	1" x 1"	> 100 keV Photon	Cs-137	n.a.	n.a.
NaI(Tl) Model 44-10 or equivalent	Core Scans	Qualitative (sample selection)	2" x 2"	> 100 keV	Cs-137	n.a.	n.a.

11 FIGURES

11.1 - Colonie Site Vicinity Map

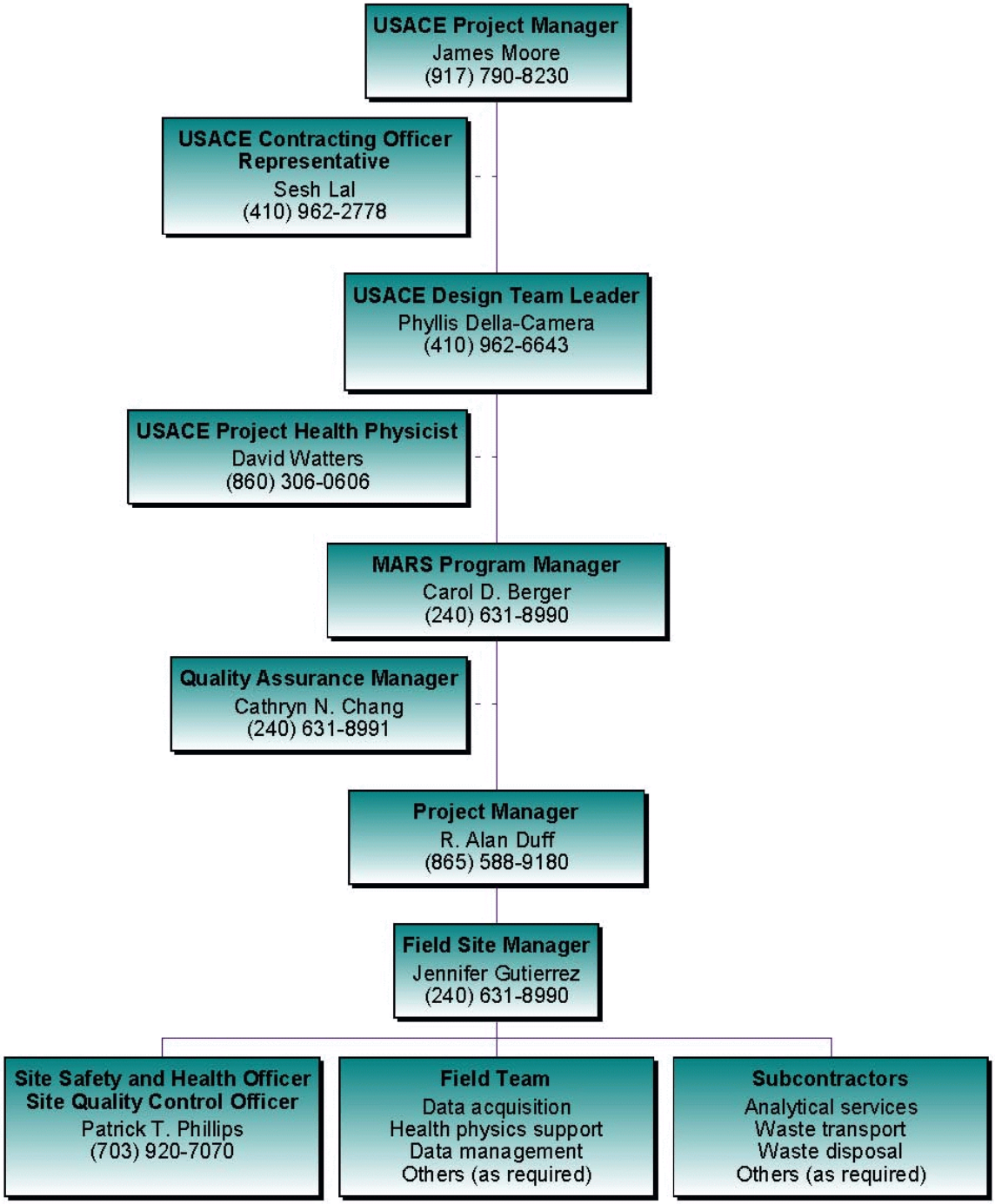


The Colonie Site
N42.692414, W73.802756
Site Vicinity Map
Source: Google, 2011.

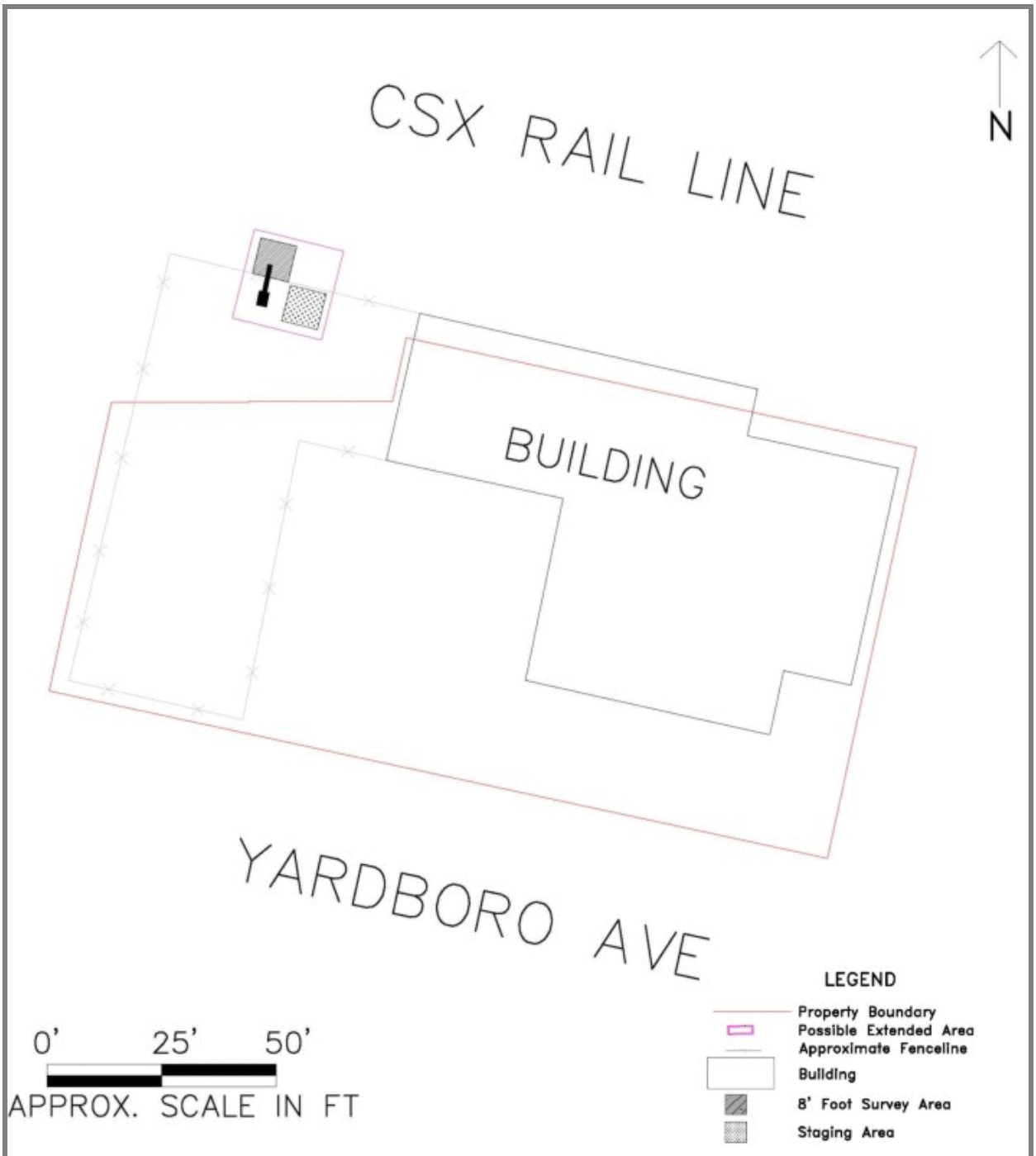
11.2 - Colonie Site Aerial Photograph

The Colonie Site
N42.692414, W73.802756
Aerial Photograph
Source: New York GIS, 2011.

11.3 - Project Organization

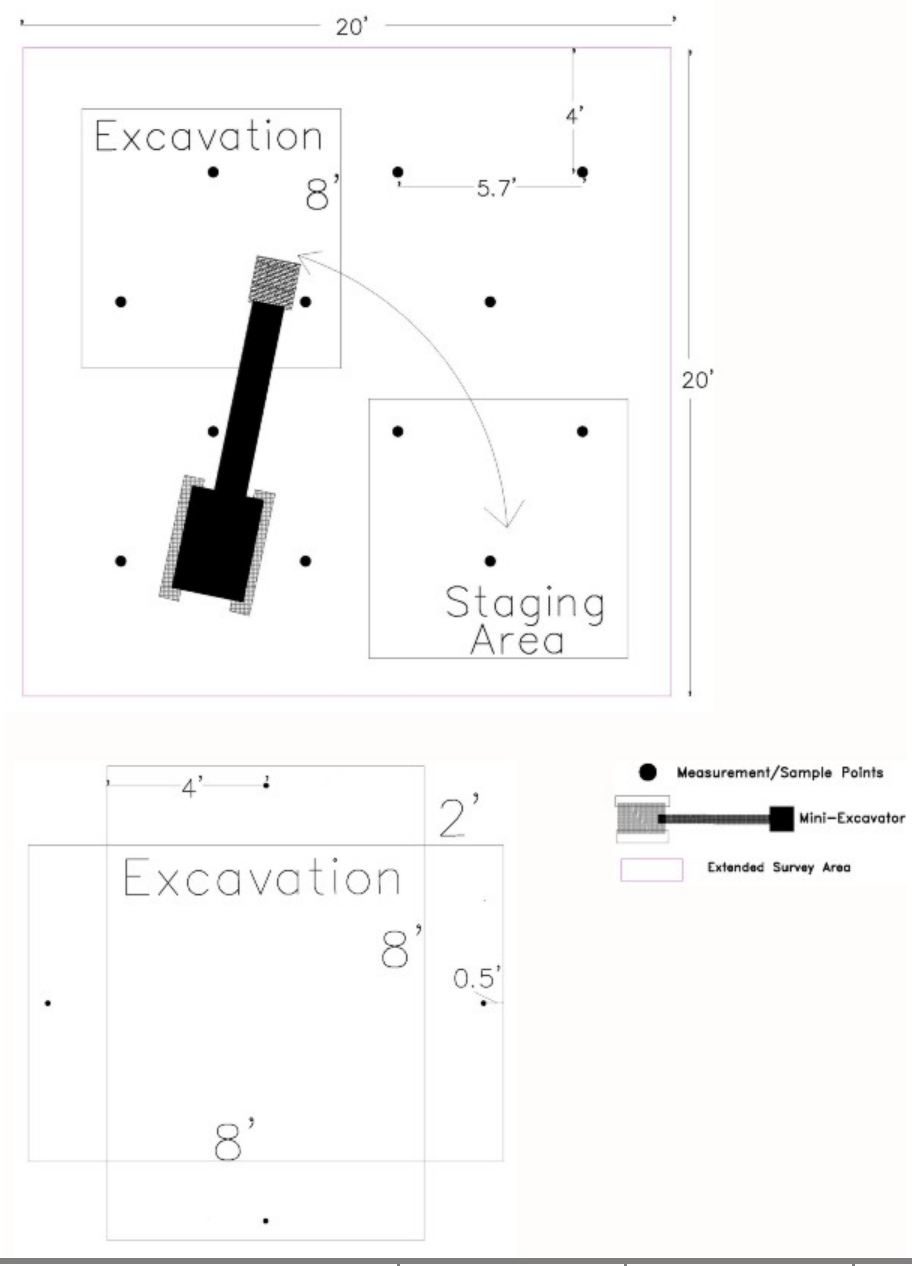


11.4 - 50 Yardboro Avenue (Potential) Study Area



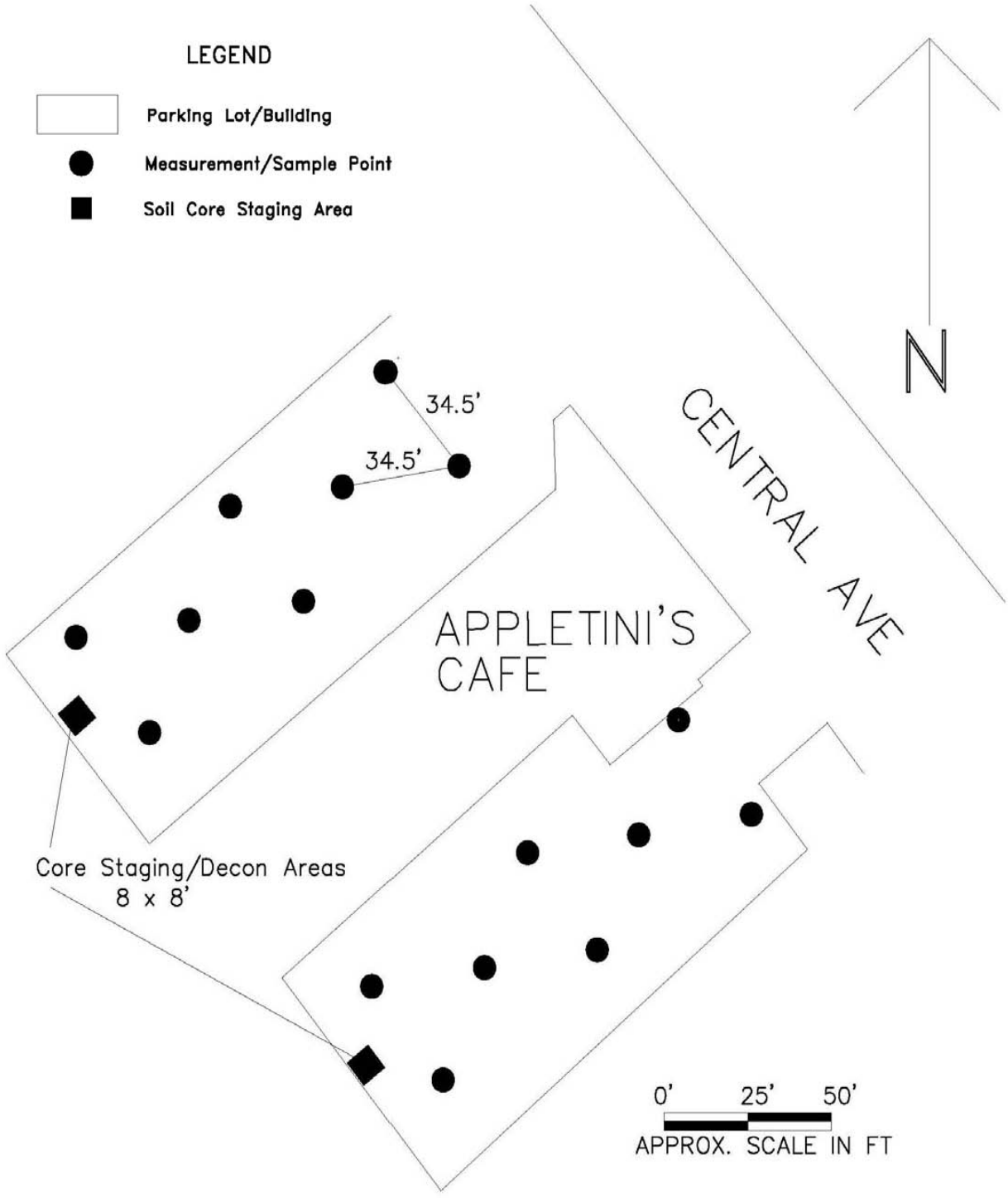
IEM	Integrated Environmental Management, Inc. 6700 Baum Drive, Suite 19 Knoxville, Tennessee 37919 (865) 588-9180	Figure: 11.4	Date: April 7, 2011	Project No.: 2008012.202
Colonie FUSRAP Site Viciniity Properties		**50 Yardboro Avenue (Potential) Study Area**		

11.5 - 50 Yardboro Avenue Sampling Locations



	Integrated Environmental Management, Inc. 6700 Baum Drive, Suite 19 Knoxville, Tennessee 37919 (865) 588-9180	Figure: 11.5	Date: April 6, 2011	Project No.: 2008012.202
Colonie FUSRAP Site Viciniity Properties		**50 Yardboro Avenue Sampling Locations**		

11.6 - 1118 Central Avenue Sampling Locations



	Integrated Environmental Management, Inc. 6700 Baum Drive, Suite 19 Knoxville, Tennessee 37919 (865) 588-9180	Figure: 11.6	Date: April 6, 2011	Project No.: 2008012.202
Colonie FUSRAP Site Viciniity Properties		**1118 Central Avenue**		

REFERENCES

DOE, 1981 — U.S. Department of Energy, Radioactive Decay Data Tables, DOE/TIC-11026, 1981.

Grove, 2005 — Grove Software, Inc., MicroShield, Version 7.00, 2005.

IT, 2000 — The IT Group, "Final Status Survey Plan; Colonie FUSRAP Site", Rev. 1, March 2000.

Noce, 2000 — Memo by Tony Noce, Shaw E&I, to Tony Sheeran, Shaw E&I, Correlation Data Summary to Date, April 14, 2000.

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USNRC, 1998 — U.S. Nuclear Regulatory Commission, Minimum Detectable Concentrations With Typical Radiation Survey Instruments for Various Contaminants and Field Conditions, NUREG 1507, Section 6.8.2, June, 1998.

APPENDICES

A - Recent (2010) Site Photographs

PHOTOGRAPHS

Fenced yard at 50 Yardboro Avenue (view from approximate location of former Colonie Site outfall)

50 Yardboro Avenue (view of property from CSX rail line)

1118 Central Avenue (aerial view) showing the asphalt-covered surfaces to the north and south of the restaurant building.

1118 Central Avenue (view of the north parking lot from the north west)

1118 Central Avenue (south east corner of the north lot)

1118 Central Avenue (south east corner of the north lot, overlooking Colonie Site)

1118 Central Avenue (south lot, overlooking utility manholes)

B - Accident Prevention Plan and Activity Hazards Analysis

Document delivered under separate cover

C - Instrumentation Performance

D.1 Introduction

Radiation surveys will be performed to confirm that the radiological status of the VPs in light of the remedial action guidelines. This appendix contains the approach and assumptions used to develop the instrument detection limits given in Table 10.2 of this report.

D.2 Radionuclides of Concern

Former operations at the Colonie site involved the use of uranium isotopes. Therefore, the radionuclides of concern (ROCs) are Uranium-238 (U-238) with progeny not likely to be in secular equilibrium. The following are the isotopic distributions, including progeny ingrowth based upon the established release criterion of 35 pCi/g of U-238:

Isotope	Isotopic distribution	Parent	Concentration (µCi/cm3)[9]
Uranium 238 (U-238)	83.20%	--	1.585x10-6
Thorium 234	83.20%	U-238	1.585x10-6
Protactinium 234 metastable	83.20%	U-238	1.585x10-6
Protactinium 234	0.11%[10]	U-238	2.063x10-9
Uranium 235	1.11%	--	2.098x10-8
Thorium 231	1.11%	U-235	2.098x10-8
Uranium 234	15.70%	--	2.992x10-7

The radiations emitted by these isotopes during decay are as follows (DOE, 1981):[11]

Isotope	Half- life	Radiation Decay						
		Alpha (Kev)	Yield	Beta (Kev)		Yield	Gamma (Kev)	Yield
				Avg	Max			
U-238	4.46x109 yr	4,147	23.0%					
		4,196	77.0%					
Th-234	24.1 d			24.9	96.2	18.5%	63.3	3.8%
				50.6	188.6	72.5%	92.4	2.7%
							92.8	2.7%

[9] The concentration is calculated for the unit activity of 1 pCi/gram of the ROC. The density of the soil is assumed to be 1.6 grams/cm^3.

[10] The branching ratio for Pa234m to Pa234 is 0.13%, 83.2%*0.13% = 0.11%

[11] This list represents the major radiation emissions. Radiations with a yield of less than 1% were not listed.

Isotope	Half- life	Radiation Decay						
		Alpha (Kev)	Yield	Beta (Kev)		Yield	Gamma (Kev)	Yield
				Avg	Max			
Pa-234m	1.2 m			825.4	2,281	98.6%	1,001	0.60%
Pa-234[12]	6.7 hr			121.9	424	4.0%	94.6	15.7%
				128.6	445	2.0%	98.4	25.4%
				141	484	24.0%	131.2	20.4%
				141.2	514	11.0%	152.7	6.8%
				150.8	514	4.2%	569.5	11.0%
				198.1	654	16.0%	699	4.7%
				217.6	711	3.8%	796.3	3.9%
				364.3	1,115	7.7%	883.2	24.2%
				390	1,183	10.0%	926	11.2%
				410.7	1,238	6.2%	949	20.2%
							980.5	5.0%
							1,394	3.1%
							1,694.6	1.2%
U-235	7.04x105 yr	4,364	11.0%				13	31.0%
		4,396	55.0%				143.8	10.5%
							185.7	54.0%
Th-231	25.5 hr			55.4	205.5	15.4%	13	71.0%
				79.6	287.6	49.2%	25.6	14.7%
				84.8	304.8	35.2%	84.2	6.4%
U-234	2.44x105 yr	4,723	27.4%				13	10.5%
		4,776	72.4%					

D.3 Instrument Descriptions

The following subsections contain descriptions of the instruments used for the various surveys, including parameters used as input to determinations of instrument sensitivity (i.e., minimum detectable concentration or MDC).

[12] The branching ratio for Pa234m to Pa234 is 0.13%.

D.3.1 Surface Soil

Surface soil will be scanned with a Field Instrument to Detect Low Energy Radiation, called a FIDLER detector (equivalent to a Thermo Scientific Model G5) coupled to a Ludlum Model 2221 rate meter. The FIDLER is a thin-crystal (i.e., 1.6 mm) scintillation detector with a diameter of 127 square centimeters. While in use for surface soil scanning, a lower limit discriminator will be set to reduce the background below 20 keV. Daily efficiency determinations will be performed using a Cs-137 sealed source.

D.3.1.1 Instrument Response

The FIDLER response to the ROCs was evaluated empirically by Shaw E&I and determined to be approximately 110 cpm per pCi/g of the ROCs (Noce, 2000). Specifically, 3,800 cpm above background was shown to be equivalent to a soil concentration of 35 pCi/gram and 11,800 cpm equivalent to an average of 100 pCi/gram. The detector background was reportedly 9,200 cpm.

D.3.1.2 Scanning MDC

The scanning MDC for this instrument, using the approach described in NUREG -1575, Section 6.7 is equivalent to 13 pCi/g.[13] For this determination it was assumed that the scan rate was approximately 0.5 meters per second (m/s) with the detector held approximately two (2) inches from the ground surface. The following are the parameters used as input to the calculation:

Parameter	Value	Reference
Nominal Background	9,200 cpm	Shaw E&I, 2000
Scan Rate	0.5 m/sec	
Survey interval	1 second	
True Positive Proportion	0.05	
False Positive Proportion	0.60	
Index of Sensitivity (d')	1.38	NUREG-1575, Table 6.5.
Background counts in the interval (bi)	153.3 counts	$b_i = 9200 cpm/60 \text{sec/min}$
Minimum Detectable counts	17.1	$s_i = 1.38 * \sqrt{153.3}$
Minimum Detectable Count Rate (MDCR)	1,026 cpm	$MDCR = (17.1) * 60$
Surveyor Efficiency	0.5	NUREG-1575, Section 6.7.2
MDCR Surveyor	1,450 cpm	$MDCR_{surveyor} = 1026 cpm/\sqrt{0.5}$
Count rate vs Activity in Soil	110 cpm/pCi/gram	Shaw E&I, 2000
MDC scanning	13 pCi/gram	$MDC = 1450 cpm/110 cpm/pCi/g$

[13] U.S. Nuclear Regulatory Commission, *Multi-Agency Radiation Survey and Site Investigation Manual*, NUREG-1575, Revision 1, August, 2000.

D.3.1.3 Stationary MDC

The stationary MDC for the FIDLER, using the approach described in NUREG-1575, Section 6.7 is 4.1 picocuries per gram based upon the following input parameters:

Parameter	Value	Reference
Nominal Background	9,200 cpm	Shaw E&I, 2000
Count time	1 minute	
Probability of Type I error, α	0.05	
Probability of Type II error, β	0.05	
Poisson probability sum for α and β errors	1.645	NUREG-1575, Table I.1
Detection limit, Ld	449 counts	$L_d = 3 + 4.65\sqrt{9200}$
Count rate vs Activity in Soil	110 cpm/pCi/gram	Shaw E&I, 2000
MDC stationary	4.1 pCi/gram	$MDC = 449 cpm / 110 cpm/pCi/g$

D.3.2 Asphalt

Asphalt surfaces are assumed to be comprised of a one-foot thick layer of gravel bedding material and an asphalt covering over soil. The effective density of the asphalt and gravel layer was assumed to be 1.5 grams per cubic centimeter. Asphalt-covered surfaces will be scanned using a sodium iodide NaI(Tl) detector, equivalent to a Ludlum Model 44-10. The sensitive portion of the detector is a crystal of sodium iodide with a diameter of two inches and a thickness of two inches. This detector is suitable for measuring gamma ray photons with energies ranging from 100 to 2,000 keV. Daily efficiency determinations will be performed using a Cesium-137 sealed source.

D.3.2.1 Instrument Response

The sodium iodide detector responds to gamma radiation only, and the ROCs exhibit an average energy of 78 keV with predominant energies at 100 keV and 1,000 keV. A unit concentration of the of one (1) pCi/gram of U-238 was used as input to the MicroShield computer code in order to determine the photon exposure rates by energy (summary report appears at the end of this appendix) (Grove, 2005). The relative concentration of each isotope is as shown in the table in Section 2, above. This analysis demonstrates that the asphalt/gravel layer attenuates the photons emitted by the ROCs by a factor of 35.[14]

D.3.2.2 Scanning MDC

The radionuclides of interest in this case are U-238 plus progeny. However, the sodium iodide detector is typically calibrated with Cesium-137 photons, with a gamma energy of 667 keV. Therefore, the detector response must be corrected for gamma energy.

[14] MicroShield was used to model the effects of one foot of asphalt and gravel over the surface of soil. (See MicroShield summary report for run [Colonie12.ms6] at the end of this appendix.)

As shown in NUREG-1507, the detector response is related to the fluence rate at specific gamma energies according to the probability of interaction for each energy as follows (USNRC, 1998):

$$Fluence\ Rate = \frac{1\ \mu R/hr}{(E_\gamma)\ (\mu_{en})}$$

where Eγ = the photon energy and (μ_{en}) = the energy absorption coefficient. The probability of interaction (P) for the specific gamma energies is calculated as follows:

$$P = 1 - e^{-(\mu_{en})_{NaI}\ (x)\ (\rho)}$$

where x = the absorber thickness and ρ = the absorber density. The relative response for each photon energy in the detector is thus determined by multiplying the fluence rate by the probability of interaction.

The manufacturer of the sodium iodide detector provides a relative response for a 2x2 NaI detector for Cs-137 gammas of 900 cpm/μR/hr.[15] This value is corrected for photons of other energies as follows:

$$Detector\ Response = \frac{900 cpm}{\mu R/hr} \times \frac{Response_{photon}}{Response_{Cs-137}}$$

The following table shows the relevant parameters for a sodium iodide detector held at a distance of 10 centimeters above an asphalt-covered surface, with mass energy absorption coefficients for air, μ_{en}, interpolated from published data:[16]

Energy (keV)	(u^{en}/r)air (cm2/g)	Fluence to Exposure Rate	(m/r) NaI, cm2/g	Prob. of Interaction	RDR	Detector Resp. (cpm per μR/hr)	MicroShield exposure rate mR/hr at measurement distance over asphalt (w/ buildup) Colonie12.ms6	cpm/μR/hr (weighted)	Detector Response Percentage
15	1.29	0.0517	47.4	1.00	0.0517	20233	3.949E-30	1.620E-19	0.0%
20	0.516	0.0969	22.3	1.00	0.0969	37937		0.000E+00	0.0%
30	0.147	0.2268	7.45	1.00	0.2268	88779	2.077E-32	3.739E-21	0.0%
40	0.064	0.3906	19.3	1.00	0.3906	152935	6.142E-24	1.905E-12	0.0%
50	0.0384	0.5208	10.7	1.00	0.5208	203913	2.884E-17	1.192E-05	0.0%

[15] http://www.ludlums.com/index.php?page=shop.product_details&flypage=flypage_ludlum.tpl&product_id=164&category_id=71&keyword=44-10&option=com_virtuemart&Itemid=116.

[16] National Institutes of Standards and Technology, http://physics.nist.gov/PhysRefData/XrayMassCoef/tab4.html

Energy (keV)	(u^{en}/r)air (cm2/g)	Fluence to Exposure Rate	(m/r) NaI, cm2/g	Prob. of Interaction	RDR	Detector Resp. (cpm per µR/hr)	MicroShield exposure rate mR/hr at measurement distance over asphalt (w/ buildup) Colonie12.ms6	cpm/µR/hr (weighted)	Detector Response Percentage
60	0.0292	0.5708	6.62	1.00	0.5708	223467	2.535E-13	1.149E-01	0.0%
80	0.0236	0.5297	3.12	1.00	0.5297	207370	5.745E-12	2.416E+00	0.0%
100	0.0231	0.4329	1.72	1.00	0.4329	169486	1.076E-09	3.698E+02	3.4%
150	0.0251	0.2656	0.625	1.00	0.2656	103987	4.929E-10	1.039E+02	1.0%
200	0.0268	0.1866	0.334	1.00	0.1862	72899	5.534E-09	8.180E+02	7.5%
300	0.0288	0.1157	0.167	0.96	0.1106	43300	2.250E-10	1.976E+01	0.2%
400	0.0296	0.0845	0.117	0.89	0.0749	29334	4.060E-10	2.415E+01	0.2%
500	0.0297	0.0673	0.0955	0.83	0.0560	21921	1.028E-09	4.569E+01	0.4%
600	0.0296	0.0563	0.0826	0.79	0.0442	17319	6.300E-09	2.212E+02	2.0%
800	0.0289	0.0433	0.0676	0.72	0.0310	12132	7.346E-08	1.807E+03	16.6%
1,000	0.0280	0.0357	0.0586	0.66	0.0237	9293	3.873E-07	7.298E+03	66.9%
1,500	0.0255	0.0261	0.0469	0.58	0.0152	5966	1.431E-08	1.731E+02	1.6%
2,000	0.0234	0.0214	0.0413	0.54	0.0115	4492	3.018E-09	2.749E+01	0.3%
						Totals:	4.932E-07	1.091E+04	100%

The following additional assumptions were used as input to the scan MDC calculation, plus the results:

Parameter	Value	Reference
Height of measurement	2" above ground	
Scan Rate	0.5 m/sec	
Survey interval	1 second	
True Positive Proportion	0.05	
False Positive Proportion	0.60	
Index of Sensitivity (d')	1.38	NUREG-1575, Table 6.5.

Parameter	Value	Reference
Background counts in the interval (bi)	150 counts	Background assumed to be 10 µR/hr
Minimum Detectable Count Rate (MDCR)	1,014 cpm	
Surveyor Efficiency	0.5	NUREG-1575, Section 6.7.2
MDCR Surveyor	1,434 cpm	$MDCR_{surveyor} = 1{,}014 cpm/\sqrt{0.5}$
Minimum Detectable Exposure Rate (MDER)	1.59 µR/hr	
MDC scanning asphalt	266 pCi/gram	see MicroShield [Colonie12.ms6]

D.3.2.3 Stationary MDC

The stationary MDC , using the approach described in NUREG-1575, Section 6.7 is 87 picocuries per gram when the detector is located approximately two (2) inches above the asphalt surface and counts integrated for one minute.[17] The following are the parameters used as input to the calculations:

Parameter	Value	Reference
Nominal Background	10,000 cpm	Ludlum Model 44-10 Specification
Count time	1 minute	
Probability of Type I error, α	0.05	
Probability of Type II error, β	0.05	
Poisson probability sum for α and β errors	1.645	NUREG-1575, Table I.1
Detection limit, Ld	468 counts	$L_d = 3 + 4.65\sqrt{10000}$
Count rate to Exposure rate	1.091x10+4 cpm/µR/hr	See relevant parameters table (above)
Minimum Detectable Exposure rate (MDER)	0.043 µR/hr	$MDER = 468\ cpm/56500\ cpm/uR/hr$
Exposure rate from 1 pCi/g ROC in soil over an asphalt surface	4.932x10-4 µR/hr/pCi/gram	Microshield [Colonie12.ms6]
MDC stationary asphalt	87 pCi/gram	

D.3.3 Soil Core Scanning

Soil cores are assumed to be two (2) inches in diameter with a density equivalent of soil, or 1.6 grams per cubic centimeter. The length of the soil core will be scanned using a sodium iodide NaI(Tl) detector, equivalent to a Ludlum Model 44-10. The sensitive portion of the detector is a

[17] For a background of 10,000 cpm, a stationary count time of 26 minutes is required in order to achieve a detection limit of 35 pCi/gram.

crystal of sodium iodide with a diameter of two inches and a thickness of two inches, which is suitable for measuring gamma ray photons with energies ranging from 100 to 2,000 keV. Daily efficiency determinations will be performed using a Cesium-137 sealed source. However, cores will be scanned only to identify the location of maximum count rate for sample collection purposes.

D.3.4 Down-hole Stationary Measurements

The interior of each soil boring will be surveyed using a sodium iodide NaI(Tl) detector, equivalent to a Ludlum Model 44-2. The active area of the detector is a sodium iodide crystal with a diameter of one inch and a thickness of one inch. It is capable of recording gamma energies between 100 and 2,000 keV. Daily efficiency determinations will be performed using a Cesium-137 sealed source. However, down-hole measurements will be used to identify relative count rates at depth.

MicroShield 7.00
Integrated Environmental Management, Inc. (05-MSD-7.00-1014)

Date	By	Checked

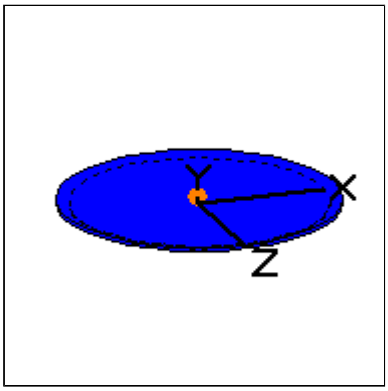
Filename	Run Date	Run Time	Duration
Colonie 12.ms6	March 29, 2011	4:50:57 PM	00:00:00

Project Info	
Case Title	Colonie VPs
Description	Effects of 12 inches of asphalt over soil 0.13% Pa-234
Geometry	8 - Cylinder Volume - End Shields

Source Dimensions	
Height	15.0 cm (5.9 in)
Radius	1.0e+3 cm (32 ft 9.7 in)

Dose Points			
A	X	Y	Z
#1	0.0 cm (0.0 in)	55.48 cm (1 ft 9.8 in)	0.0 cm (0.0 in)

Shields			
Shield N	Dimension	Material	Density
Source	4.71e+07 cm³	Concrete	1.6
Shield 1	30.48 cm	Concrete	1.5
Air Gap		Air	0.00122



Source Input: Grouping Method - Standard Indices
Number of Groups: 25
Lower Energy Cutoff: 0.015
Photons < 0.015: Included
Library: Grove

Nuclide	Curies	Becquerels	µCi/cm³	Bq/cm³
Pa-234	9.7217e-008	3.5970e+003	2.0630e-009	7.6331e-005
Pa-234m	7.4880e-005	2.7706e+006	1.5890e-006	5.8793e-002
Th-231	9.8866e-007	3.6580e+004	2.0980e-008	7.7626e-004
Th-234	7.4880e-005	2.7706e+006	1.5890e-006	5.8793e-002
U-234	1.4099e-005	5.2168e+005	2.9920e-007	1.1070e-002
U-235	9.8866e-007	3.6580e+004	2.0980e-008	7.7626e-004
U-238	7.4880e-005	2.7706e+006	1.5890e-006	5.8793e-002

Buildup: The material reference is Source Integration Parameters	
Radial	20
Circumferential	10
Y Direction (axial)	10

Results

Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm²/sec	Fluence Rate MeV/cm²/sec	Exposure Rate mR/hr	Exposure Rate mR/hr

		No Buildup	With Buildup	No Buildup	With Buildup
0.015	6.198e+05	8.884e-169	4.604e-29	7.620e-170	3.949e-30
0.03	5.360e+03	3.626e-31	2.096e-30	3.594e-33	2.077e-32
0.04	4.403e+00	3.815e-22	1.389e-21	1.687e-24	6.142e-24
0.05	6.156e+02	1.706e-15	1.068e-14	4.544e-18	2.844e-17
0.06	1.086e+05	3.834e-11	3.793e-10	7.616e-14	7.535e-13
0.08	8.228e+03	2.095e-10	3.631e-09	3.315e-13	5.745e-12
0.1	1.736e+05	2.899e-08	7.033e-07	4.435e-11	1.076e-09
0.15	6.831e+03	9.738e-09	2.993e-07	1.604e-11	4.929e-10
0.2	2.333e+04	1.054e-07	3.136e-06	1.861e-10	5.534e-09
0.3	2.612e+02	5.110e-09	1.186e-07	9.694e-12	2.250e-10
0.4	2.209e+02	1.165e-08	2.084e-07	2.270e-11	4.060e-10
0.5	3.280e+02	3.666e-08	5.240e-07	7.195e-11	1.028e-09
0.6	1.340e+03	2.731e-07	3.227e-06	5.330e-10	6.300e-09
0.8	8.543e+03	4.397e-06	3.862e-05	8.363e-09	7.346e-08
1.0	2.889e+04	2.993e-05	2.101e-04	5.517e-08	3.873e-07
1.5	5.034e+02	1.759e-06	8.505e-06	2.960e-09	1.431e-08
2.0	6.494e+01	5.044e-07	1.952e-06	7.799e-10	3.018e-09
Totals	**9.865e+05**	**3.706e-05**	**2.674e-04**	**6.816e-08**	**4.932e-07**

D - Quality Assurance Project Plan

Quality Assurance Project Plan
Soil Sampling of Vicinity Properties
at the Colonie FUSRAP Site

TABLE OF CONTENTS

1 INTRODUCTION AND BACKGROUND

1.1 Facility Description

The Colonie FUSRAP Site, hereinafter referred to as "the Colonie Site" or "the Site," is located in the Town of Colonie at 1130 Central Avenue, Albany New York. It consists of 11.2 acres surrounded by residential and commercial properties known as vicinity properties (VPs). Figure 5.1 is an aerial photograph showing the location of the site.

1.2 Facility History

Industrial operations at the Site began in 1923 when a facility was built for manufacturing wood products and toys. In 1937, National Lead (NL) purchased the facility for conducting electroplating operations. In 1958, the nuclear division of NL began producing items manufactured from uranium and thorium under a license issued by the Atomic Energy Commission (AEC) and New York State.

The New York State Supreme Court shut down the NL plant in 1984 due to environmental concerns, at which time ownership was transferred to the U. S. Department of Energy (DOE). In 1980, the DOE surveyed the VPs surrounding the NL plant and determined that uranium released into the air during former operations deposited on nearby residential and commercial properties and structures. They also found the preponderance of the deposition in the direction of the area's prevailing winds.

In October 1983, the DOE performed more detailed radiological investigations of the individual VPs, with the objective of locating where uranium concentrations exceeded the remedial action guidelines agreed upon by the State of New York and the DOE. The DOE surveys identified 56 VPs requiring remedial action. In 1984, 1985 and 1988, 53 of the VPs were remediated, Certification Dockets were prepared attesting to their radiological status, and all contaminated material from remediation activities was staged at an on-Site facility called the Colonie Interim Storage Site (CISS) pending disposal. The remaining VPs, the Niagara Mohawk Substation VP, the Town of Colonie VP, and the CSX VP, were addressed by the USACE along with the main site, with remediation taking place at the CSX and Town of Colonie VP. N, and no action required at the Niagara Mohawk VP.

1.3 VP Background

The Baltimore District of the USACE, performed an independent review of the remedial activities at the aforementioned 53 VPs in order to verify data adequacy and confirm compliance with the remedial action guidelines as agreed upon in an Action Memorandum.[1],[2] The USACE review identified data gaps for the VPs located at 50 Yardboro Avenue and at 1118 Central Avenue. The following subsections further describe the two VPs and their status.

1.3.1 Property at 50 Yardboro Avenue

The DOE Certification Docket for this property indicates detectable uranium exists on the back portion, extending to the remaining CSX Rail VP. Radioactivity allegedly reached this location via a storm water outfall that ran below the CSX Rail VP and discharged at the 50 Yardboro Avenue property boundary. Although the outfall no longer exists, the Certification Docket recommended further investigation of its former location during any future CSX VP removal actions. As a result, there is a data gap that must be closed before the 50 Yardboro Avenue property can be released.

1.3.2 Property at 1118 Central Avenue

This property is comprised of a building surrounded on either side by an asphalt surface (i.e., parking lots). The DOE's limited remedial action on accessible soil, followed by post remedial action samples, demonstrated compliance with the

[1]U. S. Army Corps of Engineers, "Technical Memorandum; Vicinity Property Assessment; Colonie FUSRAP Site; Colonie, New York", August 12, 2010.

[2]Shaw Environmental, Inc., "Final Post Remedial Action Report; Colonie FUSRAP Site; Formerly Utilized Sites Remedial Action Program; Colonie, New York", prepared for the U. S. Army Corps of Engineers, January 12, 2010.

remedial action guidelines. However, the DOE's Certification Docket reported elevated gamma radiation readings over the asphalt surfaces. The source, believed to be natural radioactivity in asphalt base materials, was supported by analytical results demonstrating isotopic ratios that did not match those from former Colonie Site materials. However, these results were not presented in the Certification Docket, thus the data gap must be closed before the 1118 Central Avenue property can be released.

1.4 Scope of Work

The objective of the investigation of the 50 Yardboro Avenue VP is to assess residual radioactivity concentrations at the location of the former outfall and confirm whether it is suitable for release with respect to established site release criteria. The specific area of interest is the back portion of the property in the general vicinity of the remaining CSX Rail VP. The property itself hosts an active auto body shop.

For the 1118 Central Avenue VP, residual radioactivity in soil above the established site release criteria is not expected. Therefore, the objectives with respect to this VP are to confirm the DOE's finding that the source of elevated count rates over the asphalt surface that surrounds the building on the property (an active restaurant operation) is natural radioactivity in bedding materials, and that the property is suitable for release for unrestricted use.

The provisions of this work plan will be implemented, the VPs will be subject to surveys and sampling as described herein, data will be reviewed/validated/analyzed, and a report of findings will be prepared. Included in the report will be a determination as to whether the 50 Yardboro Avenue VP is eligible for release, and a finding with respect to the source of the elevated count rates over the asphalt surfaces of the 1118 Central Avenue VP.

1.5 Contaminant Identification

Former operations at the Colonie Site involved the use of uranium. Therefore the radionuclide of concern (ROC) for this project is primarily Uranium-238 (U-238) with progeny not fully in secular equilibrium.

The previously approved remedial action guideline for the VPs impacted by former Site operations is 35 picocuries of U-238 per gram (pCi/g) with the following isotopic distribution: 83.2% U-238, 1.11% U-235 and 15.7% U-234.[3] Therefore, the established site release criteria applicable to this investigation are:

- U-238 - 35 pCi/g
- U-235 - 0.5 pCi/g
- U-234 - 6.5 pCi/g

1.4 Project Organization

The organization tasked with investigating radiological issues at the VPs is shown in Figure 5.2. The following is a listing of key project personnel and a brief summary of their responsibilities:

- USACE Project Manager - James Moore, who serves as the USACE Project Manager, is responsible for the execution of the project objectives and management of the IEM Team.
- USACE Contracting Officer Representative - Sesh Lal, who Serves as the Contracting Officer Representative, is the liaison with the USACE Contracts Office and is responsible for facilitating contract-related issues.

[3]Shaw Environmental, Inc., "Final Post Remedial Action Report; Colonie FUSRAP Site; Formerly Utilized Sites Remedial Action Program; Colonie, New York", Prepared for the U. S. Army Corps of Engineers, New York District, January 12, 2010.

- USACE Design Team Leader - Phyllis Della-Camera, who serves as the Design Team Leader, is the technical liaison with the Project Manager, responsible for direct management of the IEM Team, and for ensuring the quality of products/deliverables through technical review and oversight. She is also responsible for obtaining and providing design review and field support personnel for the project on an "as needed" basis.

- USACE Project Health Physicist - David Watters, who serves as the Project Health Physicist, provides technical input to the project team and radiation safety oversight.

- MARS Program Manager - Carol Berger, who serves as the MARS Program Manager, is responsible for ensuring project objectives are met, that expenditures are within the project budget, and that the quality of submittals meets applicable objectives.

- Project Manager - R. Alan Duff, who serves as Project Manager, is responsible for planning, coordinating, integrating, monitoring, and managing project activities. He is also responsible for managing the project budget and schedule, working with the Quality Assurance Manager to ensure procedural compliance for all tasks, and serves as the primary point of contact with the USACE.

- Quality Assurance Manager - Cathryn N. Chang, who serves as the Quality Assurance Manager for the project, is IEM's Quality Assurance Officer. She is responsible for the development and implementation of quality control measures in planning documents and during implementation, and for ensuring data quality objectives are met.

- Field Site Manager - Jennifer Gutierrez, who serves as Field Site Manager, is responsible for organization, scheduling, and implementation of field activities for the project. She is the primary point of contact for all on-site personnel, responsible for the activities of the field team/subcontractors, and the preparation and submittal of Daily Quality Control Reports. In addition, she ensures that all field activities are completed safely, efficiently, in compliance with applicable requirements, and in accordance with the provisions of this work plan and the Quality Assurance Project Plan (QAPP). Ms. Gutierrez also serves as the Site Radiation Safety Officer (RSO) for all on-site activities (see below).

- Site Radiation Safety Officer - The Site RSO (Jennifer Gutierrez, see above) is responsible for ensuring the radiological safety of all field activities and has authority to direct such activities, to stop and restart work if necessary, and to take appropriate actions, as required, to address radiological emergency situations. She also ensures all survey and sampling activities are performed pursuant to the pertinent provisions of this plan and the QAPP.

- Site Quality Control Officer - The Site Quality Control Officer (Patrick Phillips, see below) is responsible for ensuring the provisions of the QAPP are implemented during acquisition of measurement data and sample collection.

- Site Safety and Health Officer - Patrick T. Phillips is responsible for ensuring the Accident Prevention Plan (APP) and site-specific health and safety plan is followed and that Site personnel are appropriately trained in its provisions. He has authority to issue stop work orders for any on-site task he believes to be unsafe. When so stopped, work shall not re-start until the Site RSO and the Project Manager approve the re-start.

2 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are part of a seven-step process that defines a format for problem solving by identifying the steps needed to make a decision based on the input of data and information. The following are the DQO process steps:

- State the problem;
- Identify the decision;
- Identify inputs to the decision;
- Define the study boundaries;
- Develop the decision rule;
- Specify tolerable limits on decisions, and
- Optimize the design.

A summary of the DQOs for the investigation of the VPs appear on Worksheet No. 11 in Appendix 6.1. Data acquired during this investigation will be reviewed as shown in Chapter 4, below, to ensure they provide applicable and defensible support for the DQOs.

3 UFP -QAPP WORKBOOK

One aspect of the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) is a series of worksheets to ensure the QAPP was prepared in accordance with Part 1 of the UFP-QAPP (the UFP-QAPP Manual) and Section 6 (Part B) of Quality Systems for Environmental Data and Technology Programs - Requirements with guidance for use, ANSI/ASQ E4 (February 2004). Each worksheet, included herein as Appendix 6.1, addresses the specific requirements outlined in the UFP-QAPP.

In the worksheets, there are references to the project organization chart, the project schedule, meeting minutes, an example Chain of Custody form, and a collection of standard operating procedures. These appear herein as Figure 5.2, Figure 5.3, Appendix 6.2, Appendix 6.3 and Appendix 6.4, respectively.

4 ASSESSMENT OF DATA USABILITY

4.1 Quantitative Measures

Measurement data will be evaluated to ensure the Quality Assurance (QA) objectives for the project are met and that quantitative measures of data quality meet those outlined in the QAPP. The routine QC procedures conducted at the off-site analytical laboratory are based on established in the published methods and published in analytical SOPs. The laboratory is responsible for following those procedures and for operating equipment such within acceptable levels of statistical control for the following tasks:

- Instrument maintenance and calibration;
- Internal quality control sample analyses (i.e., method blanks, surrogate spikes, matrix spikes/matrix spike duplicates, analytical spikes, laboratory duplicates).

Data assessment procedures that will be performed for at the Colonie FUSRAP site will include the following:

- Initial review of analytical and field data for completeness and accuracy and for the required frequency of QC samples;
- Evaluation of blank results to identify systematic contamination;
- Statistical calculations for accuracy and precision using the appropriate quality control sample results;
- Estimates of completeness, in terms of the percent of valid unqualified data; and
- Assigning data qualifier flags to the data as necessary to reflect limitations identified by the process.

Qualified data will be presented in the project report with data flags captured in the Certificates of Analysis and results data base. The following is a summary of the calculations used for data validation criteria and acceptance:

4.1.1 Instrument Response Linearity (Calibration)

Acceptance criteria for instrument response linearity checks are based upon the correlation coefficient, R^2, of the best-fit line for the calibration data points. The correlation coefficient reflects the linearity of response to the calibration standards and is calculated as follows:

$$R^2 = \frac{[\Sigma(x_i - x_{ave})(y_i - y_{ave}]^2}{\Sigma(x_i - x_{ave})^2 \Sigma(y_i - y_{ave})^2}$$

where x = the calibration concentration and y = the instrument response.

4.1.2 Precision

The degree of agreement between the numerical values of a set of duplicate samples performed in an identical fashion constitutes the precision of the measurement. Control limits for control sample analyses, acceptability limits for replicate analyses, and response factor agreement criteria specified for calibration and internal QC checks are based upon precision, in terms of the coefficient of variation (CV) or the RPD. The standard deviation (S) of a sample set is calculated as:

$$S = \sqrt{\frac{\Sigma(x - x_{ave})^2}{n - 1}}$$

where x = the individual measurement result and n = the number of measurements. The CV as a percentage is then calculated as:

$$CV = \frac{S}{x_{ave}} \times 100$$

The RPD, which permits comparison of two analytical values in terms of precision only (i.e., no estimate of accuracy) is calculated as follows:

$$RPD = \frac{|M - m|}{\frac{M + m}{2}} \times 100$$

where M = the first measurement value and m = the second measurement value. For duplicate measurements, CV relates to RPD as follows:

$$CV = \frac{RPD}{\sqrt{2}}$$

For radiological samples, it is possible to have net negative results, thus resulting in the RPD implying agreement between the two samples. Instead, the method used for variability testing is the "z-score", which expresses the divergence of a measurement result from the most probable value as the number of standard deviations. The larger the value of "z", the less probable the result is due to chance. A normally-distributed measurement result, "x", is thus standardized by subtracting the mean and dividing by the standard deviation of the data set as follows:

$$z = \frac{x - \mu}{\sigma}$$

where x = the individual data point, μ = the mean of the entire data set and σ = the data set standard deviation. The calculated z-scores are compared to a performance criterion of ± 2.57, thus duplicate analyses with results that are within this range leads to the conclusion that the two results are of the same normally-distributed population and that the standard deviations represent the actual standard deviation of the measured population.

4.1.3 Accuracy

Accuracy is the degree of agreement of a measurement, x, with an accepted reference or true value, T.[4] It is a measure of the bias in a system due to personnel, instruments or method factors, and is evaluated from reference samples and percent recoveries. The accuracy of data collected using field instruments is difficult to quantify. However, it can be qualitatively maximized by following approved procedures, manufacturer's instructions and calibration guidance.

Two sample types are used to evaluate accuracy; the laboratory blank spike and the matrix spike, with accuracy expressed as the percent recovery of an analyte that has been added to the control samples or to a standard matrix (e.g., an inert soil sample) at a known concentration prior to analysis. The accuracy is thus given in terms of relative error (RE), which reflects the degree to which the measured value agrees with the actual value (generally within $\pm$ 30%). The Percent RE is calculated as follows:

[4]Accuracy is typically expressed as either the difference between the two values (x-T) as a percentage of the true value (100(x-T)/T), or as a ratio (x/T).

$$Percent\ RE = \frac{Measured - Actual}{Actual} \times 100$$

Similarly, the percent spike recovery is determined as follows:

$$Percent\ Spike\ Recovery = \frac{(Measured + spike) - Measured}{spike} \times 100$$

4.1.4 Control Limits
Control limits for central tendency and variability are generated by the laboratory to statistically monitor system performance. Because they vary from system to system and matrix to matrix, control limits are not provided herein.

4.1.5 Completeness
Completeness is a measure of the degree to which the amount of sample data collected meets the scope and a measure of the relative number of analytical data points that meet the acceptance criteria, including accuracy, precision, and any other criteria required by the specific analytical method used. It is defined as a comparison of the actual numbers of valid data points and expected numbers of points expressed as a percentage.

For this project, a completeness goal of 90% has been set. The ability to meet or exceed this completeness objective depends on the nature of samples submitted for analysis. If data cannot be reported without qualifications, project completion goals may still be met if the qualified data, i.e., data of known quality even if not perfect, are suitable for the specified project goals.

Completeness is calculated after the QC data have been evaluated, and the results applied to the measurement data. In addition to results identified as being outside of the QC limits established for the method, broken or spilled samples, or samples that could not be analyzed for any other reason are included in the assessment of completeness. The percentage of valid results is reported as completeness as follows:

$$Percent\ Completeness = \frac{T - (I + NC)}{T} \times 100$$

where T = Total number of expected measurements for a method and matrix; I = Number of invalidated results for a method and matrix; and NC = Number of results not collected (e.g., bottles broken etc.) for a specific and a matrix.

4.1.6 Detection Limits
The Minimum Detectable Concentration, or MDC, describes the ability of a particular analytical method to measure a specific radionuclide or radiation, and it is a function of instrument sensitivity, sample geometry, target analyte, and count time. the MDC is an *a priori* value that describes the lowest radionuclide concentration that a given analytical system can detect with 95% confidence. The following is the equation used to determine MDA:

$$MDA = \frac{4.66 \times \sqrt{C_B} + 2.71}{2.22 \times V \times Eff \times A \times T_s}$$

2.71 = Statistical Factor (95% Confidence Level); 4.66 = Confidence Factor (95% Confidence Level); C_B = Background counts; 2.22 = dpm to pCi conversion factor; Eff = Detector efficiency; V = Sample volume/weight; A = Abundance; and T_S = Sample count time. For methodologies that involve an extraction step (i.e., Isotopic Uranium analysis), the chemical yield, Y, is included as an additional variable in the denominator and is determined as follows:

$$Y = \frac{C_T}{T \times Eff \times D_T}$$

where C_T = Total Counts in the Tracer Peak; T = Count Time (in minutes); Eff = Detector Efficiency and D_T = Activity of Tracer (in dpm) added to each sample.

4.1.7 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of the sampled area. It is a qualitative parameter that relies on proper design of the sampling program and is best satisfied by ensuring sampling locations are properly selected and a sufficient number of samples are collected. The sample collection locations at the VPs is patterned after a previously-approved final status survey plan. For both VPs, the sample collection density follows a MARSSIM Class 1 frequency.

4.1.8 Comparability

Comparability, a qualitative parameter that expresses the confidence with which one data set can be compared to another, is influenced by sampling and analytical procedures. By providing specific protocols for collecting and analyzing samples, data sets should be comparable regardless of who obtains the sample or who performs the analysis.

4.2 Data Review, Verification, Reduction and Validation

4.2.1 Field and Technical Data

The field and technical (non-laboratory) data collected at the site can be generally characterized as either "objective" or "subjective" data. Objective data (e.g., radiological field screening results) include direct measurements of field data such as field screening/analytical parameters and water level measurements. Subjective data include descriptions and observations such as descriptions of sampling locations and conditions, and physical descriptions of the samples.

Field data collected during the field activities will be evaluated for usability by conducting a Quality review, which will consist of checking the procedures used and comparing the data to previous measurements. Field QC samples will be evaluated to ensure that field measurements and sampling protocols have been observed and followed. Checks will include:

- Use of pre-approved procedures;
- Calibration method and frequency;
- QC batch number;
- Date and time sampled;
- Preservation;
- Samplers;
- Laboratory;
- Chain of custody forms; and
- Date shipped.

A quality review of data obtained from field measurements will be performed by field personnel. The validity of data will be determined by checking calibration procedures used in the field and by comparing the data to previous

measurements, if any, at the specific site. Large variations (greater than 50%) will be examined for possible re-collection of data or assignment to a lower level of validity.

4.2.2 Data Reduction
Field data will be exported from its data collection devices, as appropriate and imported to appropriate data base management systems. Original field forms will be filed as hard copies for later review and verification of electronic copies of such data. Subjective data will be filed as hard copies for later review and incorporation into technical reports, as appropriate, and be formatted into a usable medium, such as a computer database program. The database will allow for the generation of summary tables, graphs, and figures while maintaining the integrity and accountability of the original data.

4.2.3 Data Verification
Data verification is the process of evaluating the completeness, correctness, and conformance/compliance of the data set against the method, procedural and contract requirements. The goal is to ensure and document that the data are what they purport to be and that the results reflect what was actually done for this project. When deficiencies in the data are identified, they will be documented for the end user's review and, where possible, resolved by corrective action

Data verification, which will apply to activities in the field (see Section 4.2.1, above) as well as in the laboratory, will be performed by the Project Manager. The off-site analytical laboratory will also review their data and capture findings in the applicable case narratives. A final review will be performed by the Contracts Manager (in concert with the Project Manager) to confirm the completeness of the data package before authorizing payment for work.

Sampling protocols, analytical methods, and project-specific planning documents (i.e., work plans and this QAPP) will provide the specifications for the data collection effort. Data verification will thus serve as an evaluation as to how closely these documents and procedures were followed as data are generated, thus the planning documents will be readily available to all personnel involved in the review process. Any deviations from the requirements of these documents will be noted in the data review summary. The following records will be used as input to the data review process:

- Sample Collection - Daily field logs, drilling logs, sample collection logs, Chain of Custody forms, shipping papers and survey results.
- Sample Receipt - Chain of Custody forms, receiver's copy of air bills, internal laboratory receipt forms, internal laboratory custody forms, and laboratory refrigerator or freezer logs, as applicable.
- Sample Preparation - Analytical services requests, internal laboratory receipt forms, internal laboratory custody forms, laboratory refrigerator or freezer logs, as applicable, preparation logs or bench notes and manufacturer's certificates for standards or solutions.
- Sample Analysis - Analytical services requests, internal laboratory receipt forms, internal laboratory custody forms, laboratory refrigerator or freezer logs (as applicable), manufacturer's certificates for standards or solutions, instrument logs or bench notes, instrument readouts (raw data), calculation worksheets and quality control results.
- Records Review - Internal laboratory checklists.

There will be two outputs of the data verification process: The verified data and the data verification records. Verified data will be those that have been checked for such items as transcription errors, correct application of dilution factors, appropriate reporting of dry weight versus wet weight, correct application of conversion factors, etc. They may also include laboratory qualifiers, if assigned. Any changes to the draft analytical results will be so noted in the case narrative.

Included in the data verification records will be a statement certifying that the data have been verified. It will also include a narrative that identifies technical non-compliance issues or shortcomings of the data produced during

field or laboratory activities. If the data review identified any cases of non-compliance issues, the narrative will identify the records involved and indicate what corrective action was taken in response. Also included in the data verification records will be the laboratory data package and other documentation, as applicable (i.e., checklists, hand-written notes or tables), along with supporting documentation for any laboratory qualifiers that are assigned.

4.2.4 Data Validation

Data validation is an analyte- and sample-specific process that extends the aforementioned review of data (i.e., verified data set) beyond method, procedural, or contractual compliance in order to determine the analytical quality of the data. Data validation criteria will be based upon the data quality objectives given herein, including a determination, where possible, of the reasons for any failure to meet method, procedural, or contractual requirements, and an evaluation of the impact of such failure on the overall data set. The validation process will apply to field as well as off-site laboratory activities and will include the following:

- Inspection of the reviewed data and both field and analytical laboratory data verification records;
- Review of the verified data to determine the analytical quality of the data set; and
- Preparation of a data validation report and, where applicable, qualified data

The goals of the data validation process are to evaluate whether the data quality goals established herein have been achieved, to ensure all project requirements have been met, to determine the impact on data quality of those that were not met, and to document the findings from the data validation.

Data validation will be performed by the Quality Assurance Manager, who functions independently from the field and analytical activities being validated. All planning documents and data review results will be made available to the validator prior to the start of the process. Since one of the goals of the data validation process is to evaluate whether the data quality objectives established herein have been achieved, certain data quality attributes (i.e., precision, bias, sensitivity, accuracy, completeness) will be defined and measured. Inputs to data validation may thus include all project plans, field activity records and analytical laboratory records that may or may not have been included in the data packages, as well as the reviewed data themselves. The following are the data validation steps:

- Identifying the project needs for records;
- Obtaining the records that were produced during data verification; and
- Validating the appropriate records to determine the quality of data and whether or not project needs were met by performing data validation as requested.

Identifying the project needs will begin with a review of the planning documents to confirm the objective of the analysis performed and the project-specific needs to be met. The data validator will then outline all of the planning document needs in order to understand what documents and records should be reviewed during data validation.

Obtaining verified data and the data verification records, including field records or analytical data packages, will be important to ensure that the data validator has a complete set of information to perform the data validation. The data validator will account for all records that are needed by the planning documents and will so note if documentation is missing as this may render the process incomplete.

During the validation process, the Quality Assurance Manager will ensure that all samples collected and the data generated for those samples are fully supported by documentation that will assist in the defense of decision-making. As necessary, data qualifiers may be assigned in order to identify potential deficiencies or concerns about the quality of the data.

Using the reviewed data and verification records, field activities will be validated as follows:

- Evaluate the field records for consistency,
- Review specific QC information,
- Summarize deviations and determine impact on data quality,
- Summarize samples collected, and
- Prepare a field data validation report, which will be captured in the final project report.

Validation of analytical laboratory data will be performed as follows:

- Assemble planning documents and data to be validated and review data verification records to determine method, procedural, and contractual required QC compliance/non-compliance;
- Review verified, reported sample results collectively for the data set as a whole, including laboratory qualifiers;
- Summarize data and QC deficiencies and evaluate the impact on overall data quality;
- Assign data validation qualifiers as necessary; and
- Prepare an analytical data validation report, which will be captured in the final project report.

The following are the outputs of the data validation process:

- Validated Data - This will be a set of data that has been validated before being passed on to the Project Manager as a basis for decision-making. They should be the same as the reviewed and verified data with the addition of any qualifiers that may have been assigned. Any corrections or changes noted during the review of verified data will be reflected in the validated data.
- Data Validation Report - The data validation report will document the results of the field and analytical data validation process and serve as the primary means of communication between the validator and the end users of the data. It will include a discussion of the objectives for sampling and analysis activities and a summary of the needs that the validator observed from the planning documents. It will also emphasize any deficiencies encountered and describe their impact on overall data quality. If data qualifiers are assigned, a summary of their definitions, assignments and reasons for assignment will be included in the report, along with the validated data set. Any updates or corrections made to the original verified data set will also be explained.

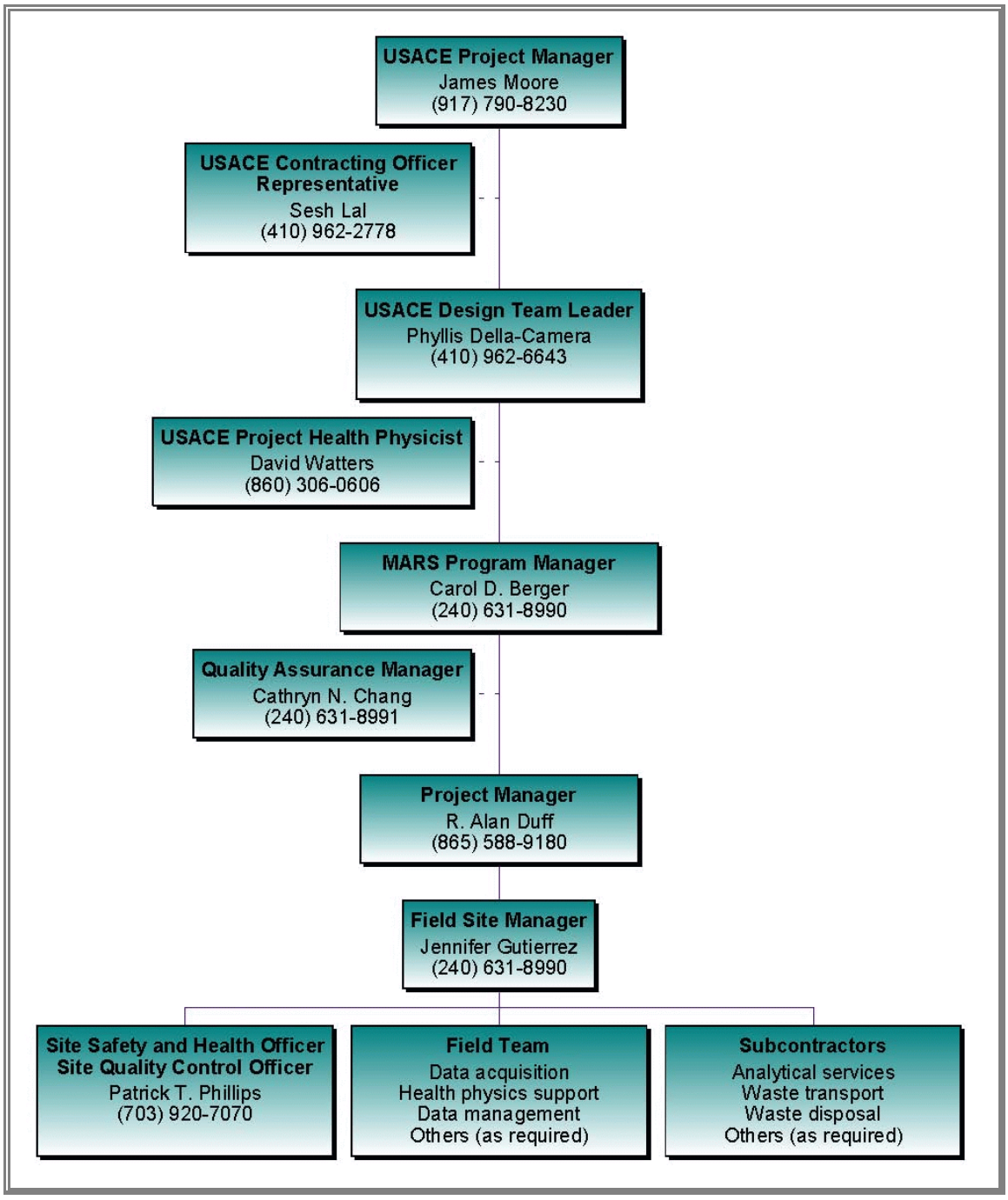
If the Quality Assurance Manager is asked to review specific information during data validation, clarify information in the data validation report, or review additional information in the form of hard-copy or electronic records, the validation report will address this request (i.e., details such as the question that was asked, how it was resolved, and the person who requested the information). Any items or observations that seem out of the ordinary during the validation process, from start to finished, will also be noted in the validation report.

5 FIGURES

5.1 Aerial Photograph of the Site and Vicinity Properties

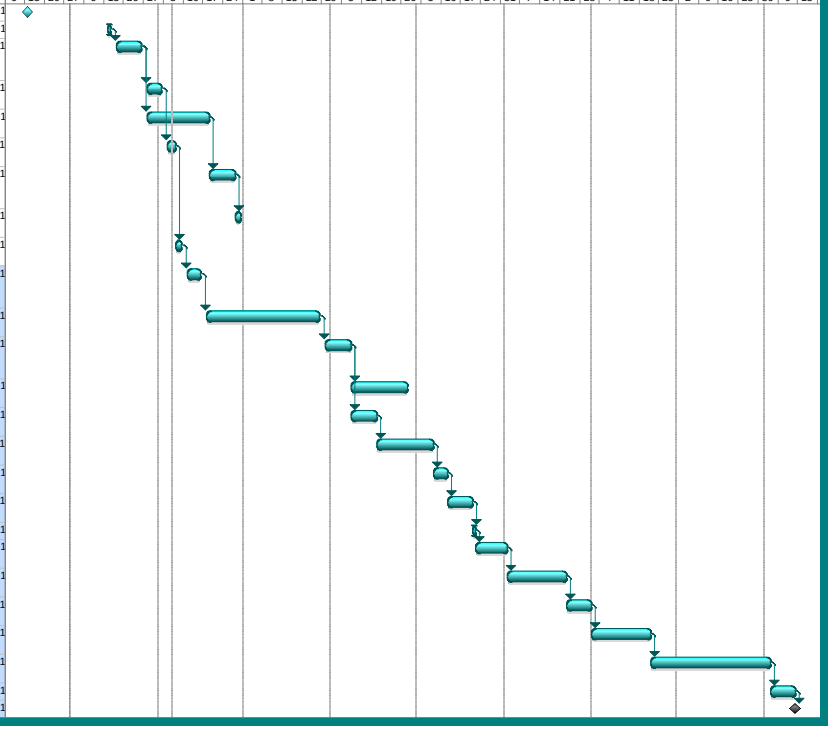
The Colonie Site
N42.692414, W73.802756
Aerial Photograph
Source: New York GIS, 2011.

5.2 Project Organization



5.3 Project Schedule

ID	Task Name	Duration	Start	Finish
1	Contract Award	0 days	Mon 2/14/11	Mon 2/14/11
2	Kickoff Meeting	1 day	Mon 3/14/11	Mon 3/14/11
3	Delivery of Draft VP Soils and Dust Sampling Work Plan and Draft APP and SSHP	7 days	Thu 3/17/11	Fri 3/25/11
4	USACE Technical Review of Draft Work Plan	5 days	Mon 3/28/11	Fri 4/1/11
5	USACE HSE Review of Draft APP and SSHP	16 days	Mon 3/28/11	Mon 4/18/11
6	Comment Resolution and Delivery of Draft Final VP Soils and Dust Sampling	3 days	Mon 4/4/11	Wed 4/6/11
7	Comment Resolution and Delivery of Draft Final APP and SSHP	7 days	Tue 4/19/11	Wed 4/27/11
8	USACE Technical Review of Draft APP and SSHP	2 days	Thu 4/28/11	Fri 4/29/11
9	USACE HSE Review of Draft Work Plan	2 days	Thu 4/7/11	Fri 4/8/11
10	Comment Resolution and Delivery of Final VP Soils and Dust Sampling Work Plan	5 days	Mon 4/11/11	Fri 4/15/11
11	NYSDEC and NYSDOH Review of Draft Final Work Plan	30 days	Mon 4/18/11	Fri 5/27/11
12	Publication of Final VP Soils and Dust Sampling Work Plan and Final APP and SSHP	7 days	Mon 5/30/11	Tue 6/7/11
13	Performance of Tasks 5A to 5E (Optional)	14 days	Wed 6/8/11	Mon 6/27/11
14	Performance of Tasks 3 and 4	7 days	Wed 6/8/11	Thu 6/16/11
15	Receipt and Verification of Analytical Data	14 days	Fri 6/17/11	Wed 7/6/11
16	Performance of Tasks 4A to 4H (Optional)	3 days	Thu 7/7/11	Mon 7/11/11
17	Receipt and Verification of Analytical Data	7 days	Tue 7/12/11	Wed 7/20/11
18	Performance of Task 8	1 day	Thu 7/21/11	Thu 7/21/11
19	Delivery of Draft VP Soils and Dust Sampling Reports	7 days	Fri 7/22/11	Mon 8/1/11
20	USACE Technical Review of Draft Reports	15 days	Tue 8/2/11	Mon 8/22/11
21	Comment Resolution and Delivery of Draft Final VP Soils and Dust Sampling	7 days	Tue 8/23/11	Wed 8/31/11
22	USACE Technical Review of Draft Final Reports	15 days	Thu 9/1/11	Wed 9/21/11
23	NYSDEC and NYSDOH Review of Draft Reports	30 days	Thu 9/22/11	Wed 11/2/11
24	Publication of Final VP Soils and Dust S	7 days	Thu 11/3/11	Fri 11/11/11
25	Project Close-out	0 days	Fri 11/11/11	Fri 11/11/11



6 APPENDICES

6.1 - UFP-QAPP Worksheets

Intergovernmental Data Quality Task Force

Workbook for Uniform Federal Policy for Quality Assurance Project Plans

Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs

Part 2A: UFP-QAPP Workbook

This workbook supplements Part 1 of the UFP-QAPP, the UFP-QAPP Manual. Proper completion of these worksheets requires knowledge of the QAPP elements explained in the Manual.

Final
Version 1
March 2005

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WORKBOOK FOR UNIFORM FEDERAL POLICY FOR QUALITY ASSURANCE PROJECT PLANS

INTRODUCTION

This *Workbook for Uniform Federal Policy for Quality Assurance Project Plans* is Part 2A of the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP). It provides examples of worksheets to assist with the preparation of QAPPs in accordance with Part 1 of the UFP-QAPP (the UFP-QAPP Manual) and Section 6 (Part B) of *Quality Systems for Environmental Data and Technology Programs - Requirements with guidance for use*, ANSI/ASQ E4 (February 2004). This Workbook may be used by the lead organization and its contractors to assist with the preparation of QAPPs for environmental data gathering activities.

Each worksheet addresses specific requirements of the UFP-QAPP. Both the UFP-QAPP Manual and the Workbook are intended to be comprehensive and are not intended to be program-specific. Since the content and level of detail in a specific QAPP will vary by program, by the work being performed, and by the intended use of the data, specific worksheets may not be applicable to all projects.

The ultimate success of an environmental program or project depends on the quality of the environmental data collected and used in decision-making, and this may depend significantly on the adequacy of the QAPP and its effective implementation. It is recommended that the individual worksheets included in this Workbook be taken to the project scoping and planning sessions. The use of the worksheets will aid in identifying the critical project information that will ensure that the right type, quality, and quantity of data are collected to meet all of the project's quality objectives. Though the format of each worksheet is not mandatory, the information required on the worksheets must still be presented in the QAPP, as appropriate to the project. In addition, QAPP preparers are encouraged to develop additional tables, as appropriate to the project. Sufficient written discussion in text format should accompany all tables. Certain sections, by their nature, will require more written discussion than others. In particular, Section 3.1.1 should provide an in-depth explanation of the sampling design rationale, and Section 5.2 should describe the procedures and criteria that will be used for data review.

QAPP_Worksheet_#1
(UFP-QAPP Section 2.1)
Title and Approval Page

Site Name/Project Name: Colonie FUSRAP Site Vicinity Properties
Site Location: Colonie, New York

Document Title: Quality Assurance Project Plan for the Colonie FUSRAP Site Investigation

Lead Organization: U. S. Army Corps of Engineers (USACE)

Preparer's Name and Organizational Affiliation: Carol D. Berger, Integrated Environmental Management, Inc.

Preparer's Address, Telephone Number, and E-mail Address: 975 Russell Avenue, Suite A, Gaithersburg, Maryland 20879, (240) 631-8990, cdberger@iem-inc.com

Preparation Date (Day/Month/Year): March 18, 2011

Investigative Organization's Project Manager/Date:_____
Signature
Printed Name/Organization: R. Alan Duff/Integrated Environmental Management, Inc.

Investigative Organization's Project QA Officer/Date:_____
Signature
Printed Name/Organization: Cathryn Chang/Integrated Environmental Management, Inc.

Lead Organization's Project Manager/Date:_____
Signature
Printed Name/Organization: James Moore/USACE

Approval Signatures/Date: _____
Signature
Printed Name/Title: Phyllis Della-Camera/USACE Design Team Leader

Approval Authority:

Other Approval Signatures/Date: _____
Signature
Printed Name/Title: David Watters/USACE Project Health Physicist

Document_Control_Numbering_System : N.A.

QAPP_Worksheet_#2
(UFP-QAPP Section 2.2.4)
QAPP Identifying Information

Site Name/Project Name: Colonie FUSRAP Site Vicinity Properties
Site Location: Colonie, New York
Site Number/Code: n.a.
Operable Unit: n.a.
Contractor Name: Integrated Environmental Management, Inc.
Contractor Number: W912DR-08-D-022

Contract Title: Soil and Dust Sampling at Vicinity Properties
Work Assignment Number: 0002

Title: VP Soils Investigation
Revision Number: 000
Revision Date: March 22, 2011
Page 3 of 56

1. Identify regulatory program: CERCLA
2. Identify approval entity: New York State Department of Environmental Conservation (NYSDEC)
3. The QAPP is (select one): ☐Generic ☒Project Specific
4. List dates of scoping sessions that were held: February 14, 2011, March 14, 2011
5. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
n.a.	

6. List organizational partners (stakeholders) and connection with lead organization:
7. List data users:
   USACE, NYSDEC, IEM
8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Type "NA" for the QAPP elements that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	Page 2 of 2
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	2 Page headers
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	Worksheet 3 Worksheet 4
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2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	Worksheet 9 Worksheet 9 Worksheet 10 and QAPP Ch. 1 QAPP Fig. 5.1
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	Worksheet 1 Worksheet 12

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	Worksheet 13
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	Worksheet 14 Worksheet 15 QAPP Fig. 5.3
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QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Required Documents
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal	Worksheet 16 Worksheet 27 QAPP App. 6.3
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(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	Worksheet 34 Worksheet 35 Worksheet 36 Worksheet 37
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining		Worksheet 13 and 37

QAPP_Worksheet_#3

(UFP-QAPP Manual Section 2.3.1)

List those entities to whom copies of the approved QAPP, subsequent QAPP revisions, addenda, and amendments will be sent.

☐ Worksheet Not Applicable (State Reason)

Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
John E. Abunaw	Radiological Sites Section	NYSDEC	518-402-8578			
James Moore	USACE Project Manager	USACE, New York District	917-790-8230		James.T.Moore@usace.army.mil	
Phyllis Della-Camera	USACE Design Team Leader	USACE, Baltimore District	410-962-6643		Phyllis.A.Della-Camera@usace.army.mil	
David Watters	USACE Project Health Physicist	USACE, Baltimore District	860-306-0606		David.J.Watters@usace.army.mil	
Alan Duff	IEM Project Manager	Integrated Environmental Management, Inc.	865-588-9180	865-588-9661	raduff@iem-inc.com	
Jennifer Gutierrez	Field Site Manager	Integrated Environmental Management, Inc.	240-631-8990	240-631-8991	jlgutierrez@iem-inc.com	
Patrick Phillips	Site QCO	BMT	703-720-7090		pphillips@dandp.com	
Carol Berger	IEM Program Manager	Integrated Environmental Management, Inc.	240-631-8990	240-631-8991	cdberger@iem-inc.com	
Cathryn N. Chang	IEM Quality Assurance Manager	Integrated Environmental Management, Inc.	240-631-8992	240-631-8991	cnchang@iem-inc.com	

QAPP Worksheet #4 (UFP-QAPP Manual Section 2.3.2)

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable sections of the QAPP and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

☐ Worksheet Not Applicable (State Reason)

Project Personnel Sign-Off Sheet

Organization: U. S. Army Corps of Engineers

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
James Moore	Project Manager	917-780-8230		
Phyllis Della Camera	Design Team Leader	410-962-6643		
David Watters	Project Health Physicist	860-306-0606		

Project Personnel Sign-Off Sheet

Organization: New York State

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
John E. Abunaw	NYSDEC Radiological sites Section	518-402-8578		

Project Personnel Sign-Off Sheet

Organization: Integrated Environmental Management, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Carol D. Berger	MARS Program Manager	240-631-8990		
R. Alan Duff	Project Manager	865-588-9180		
Jennifer L. Gutierrez	Field Site Manager	240-631-8990		
Cathryn N. Chang	Quality Assurance Manager	240-631-8992		
Patrick L. Phillips	Site Safety and Health Officer; Site Quality Control Officer	703-720-7070		

QAPP_Worksheet_#5

(UFP-QAPP Manual Section 2.4.1)

Identify reporting relationships between all organizations involved in the project, including the lead organization and all contractor and subcontractor organizations. Identify the organizations providing field sampling, on-site and off-site analysis, and data review services, including the names and telephone numbers of all project managers, project team members, and/or project contacts for each organization.

☐ Worksheet Not Applicable (State Reason)

See Organization Chart in QAPP (Figure 5.2)

QAPP_Worksheet_#6

(UFP-QAPP Manual Section 2.4.2)

Describe the communication pathways and modes of communication that will be used during the project, after the QAPP has been approved. Describe the procedures for soliciting and/or obtaining approval between project personnel, between different contractors, and between samplers and laboratory staff. Describe the procedure that will be followed when any project activity originally documented in an approved QAPP requires real-time modifications to achieve project goals or a QAPP amendment is required. Describe the procedures for stopping work and identify who is responsible.

☐ Worksheet Not Applicable (State Reason)

Communication Pathways

Communication_Drivers	Responsible_Entity	Name	Phone Number	Procedure_(Timing,_Pathways,_etc.)
Point of Contact with NYSDEC	USACE Project Manager	James Moore	917-790-8230	Mr. Moore will serve as the point of contact and information exchange between regulators and the project team.
Project management	USACE Design Team Leader	Phyllis Della-Camera	410-962-6643	Ms. Della-Camera will serve as the technical liaison with the IEM Team and providing design review.
Technical oversight	USACE Project Health Physicist	David Watters	860-306-0606	Mr. Watters provides technical input to the IEM Team and radiological oversight.
Contract management	USACE Contracting Officer's Representative	Sesh Lal	410-962-2778	Mr. Lal will serve as the contracting officer's representative for the project and serve as a liaison to the Contracting Officer on contractual matters.
Project management	IEM Project Manager	R. Alan Duff	865-588-9180	Mr. Duff will serve as the point of contact and information exchange between the project team and Mr. Moore and will authorize field changes to the QAPP.
Daily field progress reports and data submissions	IEM Field Site Manager	Jennifer Guttierez	865-588-9180	Ms.. Gutierrez will ensure daily progress reports are forwarded to Mr. Moore, and that data acquired are forwarded to data management staff (lkselby@iem-inc.com)
Reporting Laboratory data quality issues	Laboratory Manager	Ron Eidson	918-251-2515	All QA/QC issues associated with field samples will be reported to Ron Eidson and Cathryn Chang upon identification but no later than 2 days from identification

Communication Pathways

Communication_Drivers	Responsible_Entity	Name	Phone Number	Procedure_(Timing,_Pathways,_etc.)
Field and analytical corrective actions	IEM Quality Assurance Manager	Cathryn N. Chang	240-631-8992	Ms. Chang will determine the need for corrective action for field and analytical issues and authorize field changes to the QAPP.
Release of analytical data	IEM Quality Assurance Manager	Cathryn N. Chang	240-631-8992	No analytical data shall be released for use until verification and validation is complete and Ms. Chang has approved the release.
QAPP Amendments	USACE Project Manager	James Moore	917-790-8230	Any major changes to the QAPP must be authorized by Ms. Chang, Ms. Berger and Mr. Duff, then approved by Mr. Moore before implementation.

QAPP Worksheet #7

(UFP-QAPP Manual Section 2.4.3)

Identify project personnel associated with each organization, contractor, and subcontractor participating in responsible roles. Include data users, decision-makers, project managers, QA officers, project contacts for organizations involved in the project, project health and safety officers, geotechnical engineers and hydrogeologists, field operation personnel, analytical services, and data reviewers. Identify project team members with an asterisk (*). Attach resume to this worksheet or note the location of the resumes.

☐ Worksheet Not Applicable (State Reason)

Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
John E. Abunaw	Radiological Sites Section	NYSDEC	Regulatory authority for public protection	
Kent Johnson	Remedial Bureau E	NYSDEC	Regulatory authority for public protection	
James Moore	USACE Project Manager	USACE, Baltimore District	Oversees project and responds to regulators	
Carol D. Berger	MARS Program Manager	Integrated Environmental Management, Inc.	Single point of contact for coordination with USACE; directs and coordinates project team; manages implementation of policy and procedures; ensures corrective action taken if performance criteria not met; holds periodic status meetings; provides resources necessary to ensure successful completion of project.	CHP; BS, Physics; MS Rad.Physics; MS HP; 34 yrs exp.
R. Alan Duff	IEM Project Manager	Integrated Environmental Management, Inc.	Point of contact for activies; directs and manages project activities; interfaces with USACE technical managers; integrates project activiteis and efforts; communicates information across the program; ensures QC program is implemented and effective; performs data validation; establishes proejct organization and management procedurs; notifies project team of non-conformances..	NRRPT; DOT; 30 yrs exp.
Jennifer Guttierez	IEM Field Site Manager and Site RSO	Integrated Environmental Management, Inc.	Manages all field activities and personnel associated with sampling and analysis; ensures requirements for labels, logs, presergvation and sampling are met; coordinates with laboratory on delivery of supplies and samples; coordinates with subcontractors to ensure compliance; evaluates waste disposal alternatives; ensures	ANSI 3.1; ANSI18.1; DOT; 11 yrs exp.

Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
			radiological instruments and test equipment are properly calibrated and response checked; ensures provisions of RSP-001 are followed and that field personnel are trained in same.	
Cathryn N. Chang	IEM Quality Assurance Manager	Integrated Environmental Management, Inc.	Plans, implements and tracks QA/QC activites, including audits; maintains communication with analytical laboratory on QC issues; interfaces with USACE regarding the QA program; prepares this QAPP; approves validated data; confirms compliance with DQOs; reviews purchase orders, plans and reports for compliance with contract documents; forwards approved POs, plans and reports to the project manager for approval; performs data validation; manages data review; ensures data review process is supported by DQOs.	BS, Business Administration, IEM Quality Assurance Officer (ISO 9001:2008); 7 years experience
Patrick Phillips	IEM Site Safety & Health Officer and Site Quality Control Officer	BMT Designers & Planners	Ensures provisions of APP and QAPP are followed and that field team members are appropriately trained in relvant provisions; ensures instruments and test equipment are properly calibrated and response-checked; confirms sample collection and shipping logs.	BS, Biology; 7 years experience.
Ron Eidson	Laboratory Manager	Outreach Laboratories	Manages generation of analytical data	Degree in chemistry; 25+ years experience
Bill Thomas	Project CHP/CIH	Integrated Environmental Management, Inc.	Ensures APP complies with regulatory and general safety requirements; approves modifications to the APP as necessary to adjust for on-site changes; evaluates and authorizes changes to the APP; ensures sufficient trained supervisory personnel are available to support the field work.	CHP; CIH; BS HP; MS IH; 32 yrs exp.
Leslie K. Selby	Data Management Coordinator	Integrated Environmental Management, Inc.	Manages data collected during field activities; manages data integrity; coordinates with laboratory; evaluates measurement and laboratory data to ensure QA/QC processes are met.	12 yrs experience

QAPP Worksheet #8

(UFP-QAPP Manual Section 2.4.4)

Provide the following information for those projects requiring personnel with specialized training. Attach training records and/or certificates to the QAPP or note their location.

☐ Worksheet Not Applicable (State Reason)

Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
On-site Work	OSHA 1910 HAZWOPER	Corporate	See Records	All on-site personnel	Field Site Manager, IEM; Site Health & Safety Officer, BMT; Field team members	Integrated Environmental Management, Inc. (Copy of certification will be maintained by the SSHO and made available for inspection at the project site)
On-site Work	Confined space training	Corporate	See records	All on-site personnel	Field Site Manager, IEM; Site Health & Safety Officer, BMT; Field team members	Integrated Environmental Management, Inc. (Copy of certification will be maintained by the SSHO and made available for inspection at the project site)
On-site work	OSHA 1910 HAZWOPER Supervisor	Corporate	See records	At least one on-site team member	Field Site Manager, IEM	Integrated Environmental Management, Inc. (Copy of certification will be maintained by the SSHO and made available for inspection at the project site)
On-site work	CPR and First Aid	Corporate	See records	At least one on-site team member	Site Health & Safety Officer, BMT	Integrated Environmental Management, Inc. (Copy of certification will be maintained by the SSHO and made available for inspection at the project site)
On-site work	49 CFR 172 Transportation Training	Corporate	See records	Shipper of all radioactive materials from the work areas over public roads	Radioactive Materials Shipper, IEM	Integrated Environmental Management, Inc.

Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
						(Copy of certification will be maintained by the SSHO and made available for inspection at the project site)

QAPP Worksheet #9

(UFP-QAPP Manual Section 2.5.1)

Complete this worksheet for each project scoping session held. Identify project team members who are responsible for planning the project.

☐ Worksheet Not Applicable (State Reason)

Project Scoping Session Participants Sheet

Project Name: VP Soils and Dust Sampling **Projected Date(s) of Sampling:** May 25-June 2, 2011 **Project Manager:** R. Alan Duff	**Site Name:** Colonie FUSRAP Site Vicinity Properties **Site Location:** Colonie, New York

Date of Session: February 14, 2011
Scoping Session Purpose: Project kickoff

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
James Moore	USACE Project Manager	USACE New York	917-790-8230	James.T.Moore@usace.army.mil	Management lead.
Phyllis Della Camera	USACE Design Team Leader	USACE Baltimore	410-962-6643	Phyllis.A.Della-Camera@usace.army.mil	Technical liaison with the Project Manager; responsible for direct management of the IEM Team; ensures the quality of products/deliverables through technical review and oversight; obtaining and providing design review and field support personnel for the project in support of the Project Manager on an "as needed" basis.
Hans Honerlah	USACE Oversight	USACE Baltimore	410-962-9184	Hans.B.Honerlah@nab02.usace.army.mil	TBD
David Watters	USACE Project Health Physicist	USACE Baltimore		David.J.Watters@usace.army.mil	Provides technical and radiological input to the Design Team Leader and technical support to the project team.
Alan Duff	Project Manager	IEM	865-588-9180	raduff@iem-inc.com	Directs development and approves project deliverables; defines Team resource needs; assigns staff and selects resources; prepares Work Plan; negotiates task scope and budget; tracks project performance relative to schedule and budget
Carol Berger	MARS Program Manager	IEM	240-631-8990	cdberger@iem-inc.com	Directs corporate and IEM Team resources; delegates authority
Steven Baker	Environmental Manager	BMT	703-720-7070	sbaker@dandp.com	Provides general environmental (non-radiological) support to the Project Manager.
Patrick Phillips	SSHO	BMT	703-720-7070	pphillips@dandp.com	Recommends stop work orders to the Project Manager in the event of unsafe tasks; requires rework or replacement of deficient items, procedures or work.

Project Scoping Session Participants Sheet

Project Name: VP Soils and Dust Sampling **Projected Date(s) of Sampling:** May 25-June 2, 2011 **Project Manager:** R. Alan Duff	**Site Name:** Colonie FUSRAP Site Vicinity Properties **Site Location:** Colonie, New York

Date of Session: March 14, 2011
Scoping Session Purpose: Technical and scope of work review

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Phyllis Della Camera	USACE Design Team Leader	USACE Baltimore	410-962-6643	Phyllis.A.Della-Camera@usace.army.mil	Technical liaison with the Project Manager; responsible for direct management of the IEM Team; ensures the quality of products/deliverables through technical review and oversight; obtaining and providing design review and field support personnel for the project in support of the Project Manager on an "as needed" basis.
David Watters	USACE Project Health Physicist	USACE Baltimore		David.J.Watters@usace.army.mil	Provides technical and radiological input to the Design Team Leader and technical support to the project team.
Megan Garrett	USACE Technical Representative	USACE Baltimore	410-962-6813	Megan.G.Garrett@usace.army.mil	Provides technical input to the project team.
Alan Warminski	USACE Technical Representative	USACE Baltimore	410-962-7677		Provides technical input to the project team
Debbie McKinldy	USACE Technical Representative	USACE Baltimore			Provides technical input to the project team
Alan Duff	Project Manager	IEM	865-588-9180	raduff@iem-inc.com	Directs development and approves project deliverables; defines Team resource needs; assigns staff and selects resources; prepares Work Plan; negotiates task scope and budget; tracks project performance relative to schedule and budget
Carol Berger	MARS Program Manager	IEM	240-631-8990	cdberger@iem-inc.com	Directs corporate and IEM Team resources; delegates authority
Leslie Selby	Data Manager	IEM	240-631-8991	lkselby@iem-inc.com	Provides data management and administrative support to the project.

Comments/Decisions: See Appendix 6.2 of the QAPP

Action Items: See Appendix 6.2 of the QAPP

Consensus Decisions: See Appendix 6.2 of the QAPP

QAPP Worksheet #10

(UFP-QAPP Manual Section 2.5.2)

Clearly define the problem and the environmental questions that should be answered for the current investigation and develop the project decision "If…, then…" statements in the QAPP, linking data results with possible actions. The prompts below are meant to help the project team define the problem. They are not comprehensive.

☐ Worksheet Not Applicable (State Reason)

Problem Definition

The problem to be addressed by the project: Former industrial operations at the Colonie FUSRAP site resulted in the release of uranium to vicinity properties (VPs). Some of these properties were remediated and radiological surveys were performed to show whether the properties met the agreed-upon remedial action guidelines. An independent review of the remedial actions and the survey results revealed data gaps at two of the VPs (50 Yardboro Avenue and 1118 Central Avenue). 50 Yardboro Avenue: The objective is to assess residual radioactivity concentrations and confirm whether it is suitable for release with respect to the pre-approved remedial action guidelines. The specific area of interest is the former outfall, located on the back portion of the property in the general vicinity of the remaining CSX Rail VP. The property itself hosts an active auto body shop. 1118 Central Avenue: Residual radioactivity in soil above the remedial action guidelines in soil is not expected. Therefore, the objective is to confirm the DOE's finding that the source of the elevated count rates observed over the asphalt surface that surrounds the building on the property (an active restaurant operation) is natural radioactivity present in bedding materials and that the site is eligible for release for unrestricted use.
The environmental questions being asked: 50 Yardboro Avenue: Is there residual uranium at the site area of interest above the remedial action guidelines. 1118 Central Avenue: Is the source of elevated count rates over the asphalt surface of the property associated with natural radioactivity in asphalt bedding materials and may the property be released for unrestricted use.
Observations from any site reconnaissance reports: At the 50 Yardboro site, the Certification Docket indicates elevated uranium in the back portion that extends to the remaining CSX Rail VP. At the 1118 Central Avenue site, elevated gamma radiation readings from below asphalt surfaces were attributed to the presence of natural radioactivity in the asphalt base materials, although no data are available to support this conclusion.
A synopsis of secondary data or information from site reports: Historical information prepared as part of the former remediation actions, including data reports, final status survey plans, sampling results and technical basis documents, will be used as input to the reporting process. Soil analytical data will be compared to pre-approved remediation goals established for the 50 Yardboro Avenue site and pre-approved PRGs for the 1118 Central Avenue site. See Worksheet #13 for reference listing.
The possible classes of contaminants and the affected matrices: Uranium isotopes in soil. For the asphalt bedding materials, the presence of natural radioactivity may be used to rule out uranium isotopes as the source of elevated gamma readings.
The rationale for inclusion of chemical and nonchemical analyses: With the exception of TCLP (metals) for waste characterization, only radiological analysis being performed.
Information concerning various environmental indicators: None
Project decision conditions ("If..., then..." statements): 50 Yardboro Avenue: If measurement data are indicative of uranium isotope concentrations above the remedial action guidelines, soil layers will be removed until such time as a surface that meets the guidelines is present. A final status of survey of that area will be performed at that time. 1118 Central Avenue: If the source of elevated count rates is associated with uranium isotopes in soil from former Colonie Site operations, follow-up actions will be based upon measurement findings.

QAPP Worksheet #11

(UFP-QAPP Manual Section 2.6.1)

Use this worksheet to develop project quality objectives (PQOs) in terms of type, quantity, andquality of data determined using a systematic planning process. Provide a detailed discussion of PQOs in the QAPP. List PQOs in the form of qualitative and quantitative statements. These statements should answer questions such as those listed below. These questions are examples only, however; they are neither inclusive nor appropriate for all projects.

☑ Worksheet Not Applicable (State Reason) Project specific DQO's inserted below.

Data Quality Objectives for VP Soil Sampling

DQO Step	Response
State the problem	There is a potential for residual uranium in soil in excess of pre-approved remedial action guidelines at two of the VPs.
Identify the decision	Mean of the sample distribution exceeds the remedial action guideline (H_o) is rejected
Identify inputs to the decisions	Input data consists of the remedial action guideline of 35 pCi/g of U-238.
Define the study boundaries	Measurement areas where the residual levels are less than 35 pCi/g of U-238 above the reference area concentration.
Develop the decision rule	If the UCL is below the remedial action guidelines, then H_o is rejected and the area meets the remediation objective.
Specify tolerable limits on decision errors	A decision error occurs when H_o is rejected when it is true or fails to reject H_o when it is false. These errors are commonly referred to as false positive or "Type I" error and false negative or "Type II" error, respectfully. The measure of the size or the level of significance of the "Type I" error is designated as alpha (α) while the equivalent measure of the level of significance of the "Type II" error is designated as beta (β). The choice for α and β values is 0.05 each, which results in a 5% probability of incorrectly concluding that activity is present when it is not and accepting a 5% probability of incorrectly concluding that no activity is present when in fact it is present.
Optimize the design	To the extent practical, the design for collecting data has been optimized to achieve the stated DQOs (i.e., survey units designated MARSSIM Class 1). The scope of work and data collection process has been designed to provide near real-time data during implementation of field activities (i.e., hand-held instrument results). These data will be used to modify and expand the scope of field activities, as needed, to ensure the DQOs are met.

QAPP_Worksheet_#12

(UFP-QAPP Manual Section 2.6.2)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQIs), measurement performance criteria (MPC), and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for a specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

☐ Worksheet Not Applicable (State Reason)

Measurement Performance Criteria Table 12.a

Matrix	Soil
Analytical_Group	Isotopic U
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance_Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
RSP-026	LANL	Precision	RPD less than 50% for overall precision	Field Duplicates	S A
		Precision	RPD less than 20% for laboratory precision	Laboratory Duplicates	A
		Accuracy/Bias	% Recovery 70-130%	Internal Standards	A
		Sensitivity	Less than quantitation limit	MDL Verification	S A

Measurement Performance Criteria Table 12.b

Matrix	Soil
Analytical_Group	Photon Emitters
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Quality Indicators (DQIs)**	**Measurement Performance_Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
RSP-026	HASL	Precision	RPD less than 50% for overall precision	Field Duplicates	S A
		Precision	RPD less than 20% for laboratory precision	Laboratory Duplicates	A
		Accuracy/Bias	% Recovery 70-130%	Internal Standards	A
		Sensitivity	Less than quantitation limit	MDL Verification	S A

Measurement Performance Criteria Table 12.c

Matrix	Asphalt Bedding Material
Analytical_Group	Photon Emitters
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Quality Indicators (DQIs)**	**Measurement Performance_Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
RSP-026	HASL	Precision	RPD less than 50% for overall precision	Field Duplicates	S A
		Precision	RPD less than 20% for laboratory precision	Laboratory Duplicates	A
		Accuracy/Bias	% Recovery 70-130%	Internal Standards	A
		Sensitivity	Less than quantitation limit	MDL Verification	S A

Measurement Performance Criteria Table 12.d

Matrix	Liquid (equipment blanks)
Analytical_Group	Isotopic U
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Quality Indicators (DQIs)**	**Measurement Performance_Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
RSP-026 and decontamination requirements in Geoprobe MK3140	LANL	Precision	RPD less than 50% for overall precision	Field Duplicates	S A
		Precision	RPD less than 20% for laboratory precision	Laboratory Duplicates	A
		Accuracy/Bias	% Recovery 70-130%	Internal Standards	A
		Sensitivity	Less than quantitation limit	MDL Verification Equipment Blanks	S A

Measurement Performance Criteria Table 12.e

Matrix	Waste Profile
Analytical_Group	Metals
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance_Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
RSP-026	1311/6010A	Precision	RPD less than 50% for overall precision	Field Duplicates	S A
		Precision	RPD less than 35% for laboratory precision	Laboratory Duplicates	A
		Accuracy/Bias	% Recovery 70-130%	Internal Standards	A
		Completeness	90%	Data completeness check	S A

QAPP Worksheet #13

(UFP-QAPP Manual Section 2.7)

Identify all secondary data and information that will be used for the project and their originating sources. Specify how the secondary data will be used and the limitations on their use.

☐ Worksheet Not Applicable (State Reason)

Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Technical Memorandum, Vicinity Property Assessment Colonie FUSRAP Site Colonie, New York	U. S. Army Corps of Engineers, August 12, 2010		Input to report as a source of background information	None
"Final Status Survey Plan; Colonie FUSRAP Site"	IT Group, March, 2000		Input to survey design	Technical basis for scan MDCs not provided; capabilities in report will be taken at face value.

QAPP_Worksheet_#14

(UFP-QAPP Manual Section 2.8.1)

Provide a brief overview of the listed project activities.

☐ Worksheet Not Applicable (State Reason)

Summary of Project Tasks

Sampling Tasks: Collection of 17 (15 plus 1 biased and 1 dup) soil samples using standardized collection methods and GPS co-location at 50 Yardboro Ave. site; Collection of 17 (15 plus 1 biased and 1 dup) soil core segments (pre-screened) and 17 (15 plus 1 biased and 1 dup) asphalt bedding samples using standardized collection methods and GPS co-location at 1118 Central Ave. site.. Collection of one (composite) soil sample for waste profiling. Collection of two (composite) liquid samples from containerized decon fluids and equipment blanks.
Analysis Tasks: Analysis of soil samples by isotopic uranium; asphalt bedding samples by gamma spectroscopy after 21-day ingrowth; waste profile by isotopic uranium and TCLP metals (lead); liquid samples by isotopic uranium.
Quality Control Tasks: Field and lab QC samples listed in Worksheet 12, 20, 24 and 28.
Secondary Data: See Worksheet 13
Data Management Tasks: Analytical data to be captured in spreadsheets after validation; spreadsheets controlled at IEM Gaithersburg (server).
Documentation and Records: GPS locations for all samples will be recorded using a State Plane NAD83 New York East coordinate system; records of each sample collected will be maintained in a Sample Collection Log; field notebooks of all collection ativities will be maintained for field measurements; COCs and airbills will be prepared and retained for each sample; copy of final QAPP will be maintained as part of IEM's project files; hard copies of all documents will be scanned and maintained at IEM Gaithersburg (server).
Assessment/Audit Tasks: Audits and assessments performed in the field; review of field, lab and data validation SOPs will be performed at the start of work; audit of records by the QAM with summary of findings captured in the project report.
Data Review Tasks: Field Site Manager will confirm samples in shipping containers match COC listing and work plan requirements; analytical laboratories will verify that all data are complete for samples received. Data will be validated by IEM as described in the QAPP. A data validation report will be prepared and summarized in the project report. validated data and all related field logs/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for decision-making. Data limitations will be determined and data will be compared to Data Quality Objectives and required Action Levels. Corrective action will be initiated, as necessary. Final data will be maintained in a database (redundant backup) with applicable qualifiers, tables, charts and graphs included. Certificates of Disposal will be reviewed to ensure waste mass/volume is consistent with that recorded on the shipping documentation.

QAPP_Worksheet_#15

(UFP-QAPP Manual Section 2.8.1)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Finally, list the published and achievable detection and quantitation limits for each analyte.

☐ Worksheet Not Applicable (State Reason)

Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: U-238

Concentration Level: Low

Analyte	CAS Number	Project_Action Limit (applicable units)	Project Quantitation_Limit (applicable units)	Analytical_Method		Achievable_Laboratory_Limits	
				MDLs	Method QLs	MDLs	QLs
U-238	24678-82-8	35 pCi/g	3.5 pCi/g	1.5 pCi/g	3.5 pCi/g	1.5 pCi/g	3.5 pCi/g
U-234	139966-29-5	6.5 pCi/g	0.7 pCi/g	1.5 pCi/g	0.7 pCi/g	1.5 pCi/g	0.7 pCi/g
U-235	15117-96-1	0.5 pCi/g	0.5 pCi/g	1.5 pCi/g	0.5 pCi/g	1.5 pCi/g`	0.5 pCi/g
TCLP Metals (lead)	7439-92-1	5.0 mg/L	0.5 mg/L	300 ug/L	0.5 mg/L	300 ug/L	0.5 mg/L

QAPP_Worksheet_#16

(UFP-QAPP Manual Section 2.8.2)

List all project activities as well as the QA assessments that will be performed during the course of the project. Include the anticipated start and completion dates.

☐ Worksheet Not Applicable (State Reason)

See QAPP and Project Schedule (Fig. 5.3 of QAPP).

QAPP Worksheet #17

(UFP-QAPP Section 3.1.1)

Describe the project sampling approach. Provide the rationale for selecting sample locations and matrices for each analytical group and concentration level.

☐ Worksheet Not Applicable (State Reason)

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach): Sampling design in the survey areas, patterned after the approved design for other vicinity properties (see Worksheet #13 for references), is consistent with MARSSIM recommendations. Grid systems are triangular to facilitate placement once final area dimensions are determined, as applicable.
Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]: 50 Yardboro Avenue: A total of 15 surface soil samples, plus one biased sample will be collected from a depth of 0 to 6 inches using a sample spoon. The minimum size of all solid samples is 500 grams. Samples will be double-bagged and labeled prior to shipment. Sample collection equipment will be decontaminated prior to changing sampling locations. Soil samples will be analyzed for photon emitters (via gamma spectroscopy). Concentration levels will be less than 35 pCi/g of U-238. Field duplicates will comprise approximately 5% (but no less than one) of each sample set. Equipment blanks and field duplicates will be analyzed for uranium isotopes. 1118 Central Avenue: A total of 15 soil core segments (plus one biased segment) and 15 asphalt bedding segments (plus one biased segment) will be collected. The minimum size of all solid samples is 500 grams. Samples will be double-bagged and labeled prior to shipment. Sample collection equipment will be decontaminated prior to changing sampling locations. Soil and asphalt bedding samples will be analyzed for uranium isotopes (alpha spectroscopy) and photon emitters after progeny ingrowth, respectively. Expected concentration levels will be less than 35 pCi/g of U-238 for soil samples. Asphalt bedding samples will reflect the presence of natural radioactivity as well as estimated U-238 and U-235 concentrations.

QAPP_Worksheet_#18

(UFP-QAPP Manual Section 3.1.1)

List all site locations that will be sampled and include sample/ID number, if available. (Provide a range of sampling locations or ID numbers if a site has a large number.) Specify matrix and, if applicable, depth at which samples will be taken. Only a short reference for the sampling location rationale is necessary for the table. The text of the QAPP should clearly identify the detailed rationale associated with each reference. Complete all required information, using additional worksheets if necessary.

☐ Worksheet Not Applicable (State Reason)

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth (See Worksheet #17)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Sampling Location
50 Yardboro	Soil	6 inches	Photon emitters	Low	15+1 biased and 1 dup	RSP-026	Final status survey requirements (Class 1 sampling and survey frequency).
1118 Central Geoprobe	Soil	1-4 ft bgs	Uranium isotopes	Low	15+1 biased and 1 dup	Geoprobe, MK3140	Partial final status survey requirements (Class 1 sampling frequency)
1118 Central Geoprobe	Asphalt bedding	0-1 ft bgs	Photon emitters	Low	15+1 biased and 1 dup	RSP-026, Geoprobe, MK3140	Partial final status survey requirements (Class 1 sampling frequency)
Waste Profile	Soil	Composite	Metals	Low	1	RSP-026	Waste profiling
Decon/equip. blanks	Liquids	Composite	Uranium isotopes	Low	2	Geoprobe	Confirmation of decontamination effectiveness

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet_#21).

QAPP_Worksheet_#19

(UFP-QAPP Manual Section 3.1.1)

For each matrix, analytical group, and concentration level, list the analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time.

☐ Worksheet Not Applicable (State Reason)

Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical_and Preparation Method/SOP Reference [1]	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding_Time (preparation/ analysis)
Soil	Uranium isotopes	Low	LANL	500 g	Baggie	Homogenize	n.a.
Soil	Photon emitters	Low	HASL	500 g	Baggie	Homogenize	n.a.
Asphalt Bedding	Photon Emitters	Low	HASL	500 g	Baggie	Homogenize	n.a.
Soil	TCLP Metals	Low	1311/6010A	8 oz	8-oz glass jar	Ice at 4 degrees	6 months
Surface Water	Uranium Isotopes	Low	LANL	1 liter	8-oz glass jar	n.a.	n.a.

1 See Worksheet #23

QAPP_Worksheet_#20

(UFP-QAPP Manual Section 3.1.1)

Summarize by matrix, analytical group, and concentration level the number of field QC samples that will be collected and sent to the laboratory.

☐ Worksheet Not Applicable (State Reason)

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Field Duplicate Pairs	Inorganic No. of MS	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab
Soil	Isotopic Urani	Low	LANL	16	1	0	0	0	0	17
Soil	Photon emitte	Low	HASL	16	1	0	0	0	0	17
Aggregate (asphalt bedding)	Photon emitte	Low	HASL	16	1	0	0	0	0	17
Soil	TCLP Metals	Low	1311/6010A	1	0	0	0	0	0	1
Liquid (equipment blanks)	Isotopic Urani	Low	LANL	0	0	0	0	2	0	2

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet_#23).

QAPP Worksheet #21

(UFP-QAPP Manual Section 3.1.2)

List all SOPs associated with project sampling including, but not limited to, sample collection, sample preservation, equipment cleaning and decontamination, equipment testing, inspection and maintenance, supply inspection and acceptance, and sample handling and custody. Include copies of the SOPs as attachments or reference all in the QAPP. Sequentially number sampling SOP references in the Reference Number column. The reference number can be used throughout the QAPP to refer to a specific SOP.

☐ Worksheet Not Applicable (State Reason)

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
RSP-026	RSP-026, Sample Collection, Rev. 1	IEM	n.a.	☒	Modifed as necessary to meet work plan requirements
RSP-008	RSP-008, Instrumentation, Rev. 6	IEM	n.a.	☐	
RSP-035	RSP-035, Operation of the Trimble Backpack Gamma Survey System, Rev. 0	IEM	n.a.	☐	
Geoprobe	Geoprobe Systems 54DT Track Mounted Rig Owner's Manual	BMT	Geoprobe	☐	
MK3140	Technical Bulletin No. MK3140, dual tube soil sample collection	BMT	Geoprobe	☐	

Copies of each procedure appear in Appendix 6.4 of the QAPP

QAPP Worksheet #22

(UFP-QAPP Manual Section 3.1.2.4)

Identify all field equipment and instruments (other than analytical instrumentation) that require calibration, maintenance, testing, or inspection and provide the SOP reference number for each type of equipment. In addition, document the frequency of activity, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Hand-held radiation survey instruments (FIDLER, NaI, pancake GM)	ANSI N323; high voltage, discriminator, window, scaler; NIST-traceable sources	Manufacturer's instructions.	n.a.	Background and response to NIST-traceable source, physical damage, current calibration	Daily prior to use	Within 2-sigma of the calibration value as determined via post-calibration 10-point testing.	Remove from service	Field Site Manager	RSP-008
GPS	Calibration not required	Manufacturer's instructions	10 static positional readings	Performance and functionality	Daily prior to use	Positional error >1m will be removed from service	Remove from service	Field Site Manager	RSP-035
Geoprobe	n.a.	daily	daily	Physical damage, performance and functionality	Daily prior to use	Go/No-go	Remove from service, on-site repair, off-site repair	Field Site Manager	Geoprobe
Portable scale (waste container weights)	Pre-calibrated by vendor	Vendor's instructions	Daily using a NIST-traceable mass.	Physical damage, performance and functionality	Daily prior to use	Go/No-go	Remove from service	Field Site Manager	Vendor's instructions

[1]Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

QAPP_Worksheet_#23

(UFP-QAPP Manual Section 3.2.1)

List all SOPs that will be used to perform on-site or off-site analysis. Indicate whether the procedure produces screening or definitive data. Sequentially number analytical SOP reference in the Reference Number column. Include copies of the SOPs as attachments or reference in the QAPP. The reference number can be used throughout the QAPP to refer to a specific SOP.

☐ Worksheet Not Applicable (State Reason)

Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive_or Screening_Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
LANL	LANL ER290	Definitive Isotopic Uranium	Isotopic Uranium	Alpha spectrometer	Outreach Laboratory	☒
HASL	HASL 300/901/ASTM D3972	Definitive Gamma spectroscopy	Photon Emitters	Gamma spectrometer	Outreach Laboratory	☒
1311/6010A	SW846 1311/6010A	Definitive TCLP	Metals (Lead)	ICP-AES	Outreach Laboratory	☐

QAPP_Worksheet_#24

(UFP-QAPP Manual Section 3.2.2)

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Alpha Spectrometer	Internal	monthly; NIST-traceable sources	+/- 2-sigma	Repair or tag out	Outreach Laboratory	LANL
Gamma spectrometer	Internal	annual; NIST-traceable sources	10%	Repair or tag out	Outreach Laboratory	HASL

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet_#23).

QAPP_Worksheet_#25

(UFP-QAPP Manual Section 3.2.3)

Identify all analytical instruments that require maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Alpha spectrometer	Daily checks (background; efficiency, FWHM)	Daily	FWHM/Eff	Daily prior to use	+/- 2-sigma	Repair or tag out	Outreach Laboratory	LANL
Gamma spectrometer	Daily checks (background, efficiency, FWHM)	Daily	FWHM/Eff	Daily prior to use	+/- 2-sigma	Repair or tag out	Outreach Laboratory	HASL

[1]Specify the appropriate reference letter or number from Analytical SOP References table (Worksheet_#23).

QAPP Worksheet #26

(UFP-QAPP Manual Appendix A)

Use this worksheet to identify components of the project-specific sample handling system. Record personnel, and their organizational affiliations, who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal. Indicate the number of days field samples and their extracts/digestates will be archived prior to disposal.

☐ Worksheet Not Applicable (State Reason)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Site Manager/IEM
Sample Packaging (Personnel/Organization): Field Team Members/IEM and BMT
Coordination of Shipment (Personnel/Organization): Field Site Manager/IEM
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Outreach Laboratory
Sample Custody and Storage (Personnel/Organization): Outreach Laboratory
Sample Preparation (Personnel/Organization): Outreach Laboratory
Sample Determinative Analysis (Personnel/Organization): Ron Eidson/Outreach Laboratory
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Less than 5
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 21
Biological Sample Storage (No. of days from sample collection): n.a.
SAMPLE DISPOSAL
Personnel/Organization: Outreach Laboratory
Number of Days from Analysis: 30 days from issue of Certificate of Analysis.

QAPP Worksheet #27

(UFP-QAPP Manual Section 3.3.3)

Describe the procedures that will be used to maintain sample custody and integrity. Include examples of chain-of-custody forms, traffic reports, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms. Attach or reference applicable SOPs.

☐ Worksheet Not Applicable (State Reason)

Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Field sampling equipment will be decontaminated before use and after each sample location as described in the work plan. Non-expendible sampling equipment will be wiped for removable radiological contamination to determine the potential for cross-contamination. Decon water and other fluids generated during decontamination will be containerized and managed according to the requirements listed in the work plan.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): The off-site laboratory will have a QA designee who will be responsible for assuring that the QA/QC requirements outlined in the work specifications, the corporate quality policy, and its associated operating procedures, including the chain of custody process, are strictly followed. The QA designee will be responsible for review of data, and alerting the Project Manager of the need for corrective action (when necessary).
Sample Identification Procedures: Sample labels shall be required for properly identifying samples. All samples shall have the label affixed to the sample container prior to its transport to the laboratory. Information maintained on each sample will include, but is not limited to, the following: IEM Project Code - an assigned IEM client/task number and site name; Location/Station Number - a unique identifier assigned to a sampling point by the sampling team; Unique Sample Identification Number - a unique number assigned to each sample, including field control samples; Sampling personnel - name of the person collecting the sample; Preservative - whether a preservative is used and the type of preservative; Analysis - the type of analysis requested; Date/Time - the date and time the sample was taken; and Type of Sample - the type of sample (e.g., grab or composite). containers. The sample label will include the IEM Project Code, the Location/Station Number, the Unique Sample ID and the Date/Time of collection. The field collection system will be set up to allow the Field Site Manager to generate field sample identifiers prior to sample collection.
Chain-of-custody Procedures: Chain-of-custody (COC) procedures shall be followed in order to provide documentation of the handling of each sample from the time it is collected until it is destroyed. The COC record shall serve as a legal record of possession of the sample and shall be initiated with the acquisition of the sample. The COC record shall remain with the sample at all times and bear the name of the person assuming responsibility for the samples.To simplify the COC record and eliminate potential legal challenges, as few people as possible should handle the sample between collection and disposal. A sample is considered to be under custody if one or more of the following criteria are met: The sample is in the sampler's possession; The sample is in the sampler's view after taking possession; The sample was in the sampler's possession and was then locked up to prevent tampering; or The sample is in a designated secure area.

A copy of the Chain of Custody form appears in Appendix 6.3 of the QAPP

QAPP_Worksheet_#28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

QC Samples Table 28.a

Matrix	Soil
Analytical Group	Photon emitters
Concentration Level	Low
Sampling SOP	RSP-026
Analytical Method/ SOP Reference	HASL
Sampler's Name	Field Team Member
Field Sampling Organization	IEM
Analytical Organization	Outreach
No. of Sample Locations	15

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data_Quality Indicator_(DQI)	Measurement_Performance Criteria
Field Duplicates	1 per batch	RPD less than 50%	If sufficient vol, re-analyze; qualify data as needed	Jennifer Gutierrez	Precision	No act. greater than QL
Laboratory Duplicates	5%	RPD less than 20%	If sufficient vol, re-analyze; quality data as needed	Ron Eidson	Precision	No act. greater than QL
Internal Standards	5%	RPD less than 20%	Re-analyze; qualify data as needed	Ron Eidson	Precision	No Act greater than QL

QC Samples Table 28.b

Matrix	Soil
Analytical Group	Isotopic Uranium
Concentration Level	Low
Sampling SOP	RSP-026
Analytical Method/ SOP Reference	LANL
Sampler's Name	Field Team Member
Field Sampling Organization	IEM
Analytical Organization	Outreach
No. of Sample Locations	15

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicates	1 per batch	RPD less than 50%	If sufficient vol, re-analyze; qualify data as needed	Jennifer Gutierrez	Precision	No act. greater than QL
Laboratory Duplicates	5%	RPD less than 20%	If sufficient vol, re-analyze; quality data as needed	Ron Eidson	Precision	No act. greater than QL
Matrix spike	5%	Recovery 75 to 125%	Re-analyze; qualify data as needed	Ron Eidson	Accuracy/Bias	No Act greater than QL

QC Samples Table 28.c

Matrix	Liquids (Equipment Blanks)
Analytical Group	Isotopic Uranium
Concentration Level	Low
Sampling SOP	RSP-026
Analytical Method/ SOP Reference	LANL
Sampler's Name	Field Team Member
Field Sampling Organization	IEM
Analytical Organization	Outreach
No. of Sample Locations	6

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data_Quality Indicator_(DQI)	Measurement_Performance Criteria
Field Duplicates	1 per batch	RPD less than 50%	If sufficient vol, re-analyze; qualify data as needed	Jennifer Gutierrez	Precision	No act. greater than QL
Laboratory Duplicates	5%	RPD less than 20%	If sufficient vol, re-analyze; quality data as needed	Ron Eidson	Precision	No act. greater than QL
Matrix spike	5%	Recovery 75 to 125%	Re-analyze; qualify data as needed	Ron Eidson	Accuracy/Bias	No Act greater than QL
Equipment Blanks	1 per container	Less than 3.5 pCi/l	Investigate	Alan Duff	Precision	Pass <3.5 pCi/l; Fail >3.5 pCi/l

QC Samples Table 28.d

Matrix	Asphalt Bedding
Analytical Group	Photon emitters
Concentration Level	Low
Sampling SOP	RSP-026
Analytical Method/ SOP Reference	HASL
Sampler's Name	Field Team Member
Field Sampling Organization	IEM
Analytical Organization	Outreach
No. of Sample Locations	16

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicates	1 per batch	RPD less than 50%	If sufficient vol, re-analyze; qualify data as needed	Jennifer Gutierrez	Precision	No act. greater than QL
Laboratory Duplicates	5%	RPD less than 20%	If sufficient vol, re-analyze; quality data as needed	Ron Eidson	Precision	No act. greater than QL
Internal Standards	5%	RPD less than 20%	Re-analyze; qualify data as needed	Ron Eidson	Accuracy/Bias	No Act greater than QL

QC Samples Table 28.e

Matrix	Soil
Analytical Group	Metals
Concentration Level	Low
Sampling SOP	RSP-026
Analytical Method/ SOP Reference	ISW846 1311/6010A
Sampler's Name	Field Team Member
Field Sampling Organization	IEM
Analytical Organization	Outreach
No. of Sample Locations	1

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Duplicates	5%	RPD less than 20%	If sufficient vol, re-analyze; quality data as needed	Ron Eidson	Precision	No act. greater than QL
Internal Standards	5%	RPD < 20%	Re-analyze; qualify data as needed	Ron Eidson	Accuracy/Bias	No Act greater than QL

QAPP_Worksheet_#29

(UFP-QAPP Manual Section 3.5.1)

Identify the documents and records that will be generated for all aspects of the project including, but not limited to, sample collection and field measurement, on-site and off-site analysis, and data assessment.

☐ Worksheet Not Applicable (State Reason)

Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field notes Chain of Custody Forms Airbills Field Activity Daily Logs CAQ Forms	Sample collection logs Chain of Custody Forms Instrument daily check records Daily QC reports and FADLs Field screening data forms	Instrument daily check sheets Sample screening forms CAQ forms Sample receipt, custody and tracking records Standard traceability records Equipment calibration documents Sample preparation/run logs Reported results for standards, QC checks and QC samples Field sample results Sample residual disposal records	Data validation report CAQ forms Communication logs	VP Soils Work Plan APP QAPP UFP-QAPP Worksheets Certificates of Disposal

QAPP Worksheet #30

(UFP-QAPP Manual Section 3.5.2.3)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify all laboratories or organizations that will provide analytical services for the project, including on-site screening, on-site definitive, and off-site laboratory analytical work. If applicable, identify the subcontractor laboratories and backup laboratory or organization that will be used if the primary laboratory or organizations cannot be used.

☐ Worksheet Not Applicable (State Reason)

Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number
Soil and water	Isotopic Uranium	Low	All water samples and 1118 Central Avenue soil samples	LANL	30 calendar days	Outreach Laboratory Ron Eidson 311 North Aspen Broken Arrow, OK 74012 918-251-2515	General Engineering Laboratory 2040 Savage Road Charleston, SC 29417 843-556-8171
Soil	Photon Emitters	Low	50 Yardboro Avenue soil samples and 1118 Central Avenue asphalt bedding samples	HASL	24 hours for soil and 30 calendar days for asphalt bedding (to permit progeny ingrowth)	Outreach Laboratory Ron Eidson 311 North Aspen Broken Arrow, OK 74012 918-251-2515	General Engineering Laboratory 2040 Savage Road Charleston, SC 29417 843-556-8171
Soil (Waste Profile)	Metals	Low	Site	SW846 1311/6010A	30 calendar days	Outreach Laboratory Ron Eidson 311 North Aspen Broken Arrow, OK 74012 918-251-2515	General Engineering Laboratory 2040 Savage Road Charleston, SC 29417 843-556-8171

QAPP Worksheet #31

(UFP-QAPP Manual Section 4.1.1)

Identify the type, frequency, and responsible parties of planned assessment activities that will be preformed for the project.

☐ Worksheet Not Applicable (State Reason)

Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Readiness review	1 time at start-up	Int.	IEM	Alan Duff, PM, IEM	Jennifer Gutierrez, FSM, IEM	Jennifer Gutierrez, FSM, IEM	Cathryn N. Chang, QAM, IEM
Field Activities	1 time at startup	Int.	IEM	Cathryn Chang, QAM, IEM	R. Alan Duff, PM, IEM	R. Alan Duff, PM, IEM	Carol D. Berger, MARS Program Manager, IEM
Field records, sample numbering and accountability	Daily while on-site	Int.	IEM	Patrick Phillips, Site QCO, BMT	Jennifer Gutierrez, FSM, IEM	Jennifer Gutierrez, FSM, IEM	Cathryn N. Chang, QAM, IEM
Confirmation of Lab certification status	1 time prior to sample shipment	Ext.	USACE ELAP certification to be reviewed and confirmed applicable	Cathryn Chang, QAM, IEM	Ron Eidson, Director, Outreach Laboratory	Ron Eidson, Director, Outreach Laboratory	Carol D. Berger, MARS Program Manager, IEM

QAPP_Worksheet_#32

(UFP-QAPP Manual Section 4.1.2)

For each type of assessment describe procedures for handling QAPP and project deviations encountered during the planned project assessments.

☐ Worksheet Not Applicable (State Reason)

Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Field Activities	Daily QC report	R. Alan Duff, PM, IEM	24 hours after audit	e-mail	Cathryn Chang, QAM, IEM	24 hours after notification
Field records, sample numbering and accountability	Daily QC report	Jennifer Gutierrez, FSM, IEM R. Alan Duff, PM, IEM	Immediately	e-mail	Cathryn Chang, QAM, IEM	24 hours after notification
Confirmation of Lab ELAP certification status	Summary Report	Ron Eidson, Director, Outreach Laboratory	48 hours after review	e-mail	Cathryn Chang, QAM, IEM	5 days after notification
Laboratory activities	Laboratory deficiency report	Ron Eidson, Director, Outreach Laboratory	Immediately upon identification	Case narrative (with Certificates of Analysis)	Cathryn Chang, QAM, IEM	24 hours after notification

QAPP_Worksheet_#33

(UFP-QAPP Manual Section 4.2)

Identify the frequency and type of planned QA Management Reports, the projected delivery date, the personnel responsible for report preparation, and the report recipients.

☐ Worksheet Not Applicable (State Reason)

QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Field Activities	Daily QC reports	Daily while on site	Jennifer Gutierrez, FSM, IEM	J. Moore, USACE Project Manager P. Della-Camera, USACE Design Center Manager A. Duff, Project Manager, IEM J. Guttierez, Field Site Manager, IEM
Confirmation of Lab certification status	1 time at startup	Prior to mobilization	Cathryn Chang, QAM, IEM	A. Duff, Project Manager, IEM J. Guttierez, Field Site Manager, IEM
Sample receipt log	With each sample receipt by lab	Immediately upon notification	Ron Eidson, Director, Outreach Laboratory	Cathryn Chang, QAM, IEM

QAPP Worksheet #34

(UFP-QAPP Manual Section 5.2.1)

Describe the processes that will be followed to verify project data. Verification inputs include items such as those listed in Table 9 of the UFP-QAPP Manual (Section 5.1). Describe how each item will be verified, when the activity will occur, and what documentation is necessary, and identify the persons responsible. *Internal* or *external* is in relation to the data generator.

☐ Worksheet Not Applicable (State Reason)

Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain of custody and shipping forms	Form will be internally reviewed upon completion and verified against field logs, laboratory report and QAPP. Review will be conducted with completion of each report.	Int.	Jennifer Gutierrez, IEM
Field Report	Field reports will be verified with field log books to ensure correct reporting of information. Review will be conducted with completion of each report.	Int.	Jennifer Gutierrez, IEM
Field and Lab data and QC report	A summary of all QC samples and results will be verified for measurement performance criteria, completeness and 10% verified to field and laboratory data reports from vendors. A report shall be prepared within 30 days of receipt.	Ext.	Ron Eidson, Outreach Cathryn Chang, IEM
Laboratory data	Lab data packages will be used to verify the reported results in the project specific sit area report and against the QAPP criteria.	Ing.	Ron Eidson, Outreach Cathryn Chang, IEM

QAPP Worksheet #35

(UFP-QAPP Manual Section 5.2.2)

Describe the processes that will be followed to validate project data. Validation inputs include items such as those listed in Table 9 of the UFP-QAPP Manual (Section 5.1). Describe how each item will be validated, when the activity will occur, and what documentation is necessary and identify the person responsible. Differentiate between steps IIa and IIb of validation.

☐ Worksheet Not Applicable (State Reason)

Validation (Steps IIa and IIb) Process Table

Step Iia / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Methods	Records support implementation of the procedure – sampling and analysis	Alan Duff, IEM
IIa	Chain of custody	Examine traceability of data from sample collection to generation of project reported data.	Alan Duff, IEM
IIb	Deviations	Determine impacts of any deviations from methods	Project Team
IIb	Project Quantitation Limit	Limits achieved as outlined in the QAPP and that the laboratory successfully analyzed standard at the specified limits.	Cathryn Chang, IEM
IIb	Field and Lab data and QC report	A summary of all QC samples and results will be verified for measurement performance criteria, completeness and 10% verified to field and laboratory data reports from vendors. A summary of findings will be captured in the project report.	Cathryn Chang, IEM

QAPP_Worksheet_#36

(UFP-QAPP Manual Section 5.2.2)

Identify the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data.

☐ Worksheet Not Applicable (State Reason)

Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa	Soil	Uranium Isotopes	Low	Section 4.2.4 of QAPP	Cathryn Chang, IEM
IIa	Asphalt Bedding	Photon Emitters	Low	Section 4.2.4 of QAPP	Cathryn Chang, IEM

QAPP Worksheet #37

(UFP-QAPP Manual Section 5.2.3)

Describe the procedures/methods/activities that will be used to determine whether data are of the right type, quality, and quantity to support environmental decision-making for the project. Describe how data quality issues will be addressed and how limitations of the use of the data will be handled.

☐ Worksheet Not Applicable (State Reason)

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
Determine if any detectable activity is present. If no detectable activity and MDC's below QL, all data are useable.

If detectable activity present and the verification and validation are acceptable then the data are usable.

If verification and validation are not acceptable then take corrective action (determine cause, data impact, evaluate impact and document rationale for resample or use of the data.)

If all results are non detectable and the detection amount is < ½ the applicable criterion, and all QC is acceptable then further analysis not is necessary. The survey area is assumed to meet the applicable criteria and no further action is necessary.

If statistically-positive values are detected, then perform a data assessment to determine if any statistical anomalies exist in the data set. Using the output from the statistical review, the project team must determine the usability of the data to make the final decision and provide the rationale in the final report.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
Determine if quality control data are within specifications though validation process IIb

Identify the personnel responsible for performing the usability assessment:
Alan Duff (IEM Project Manager), Cathryn Chang (IEM Quality Assurance Manager) and Carol Berger (IEM MARS Program Manager)

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

A usability summary describing the rationale for the data used and presentation of any data limitations will be captured in the project report.

6.2 - Meeting Minutes

ISO 9001:2008
Integrated Environmental Management, Inc.
Certificate No. 08.002.1

IEM
Integrated Environmental Management, Inc.

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MEETING MINUTES

Date: February 14, 2011
Start Time: 2:00 p.m. EST
End Time: 2:35 p.m. EST
Location: Call-in number
Topic: Colonie FUSRAP Site kickoff teleconference

Attendees:
R. Alan Duff - IEM Project Manager
Carol D. Berger - IEM Program Manager
Steven Baker - IEM Environmental Manager
Patrick Phillips - IEM SSHO
Phyllis Della Camera - USACE Design Center Manager
James Moore - USACE Project Manager
David Watters - USACE Technical Lead
Hans Honerlah - USACE Program Manager

Topics:
The participants introduced themselves and lines of communication for the project were set. Jim provided a brief summary of the scope of work and asked that he be informed before communicating with regulators or members of the public. Jim is also to be copied on all project correspondence. The IEM Team concurred. Jim is working on site access issues and he believes access issues should be resolved by the time the project plans are completed.

Contract information was discussed and Carol relayed what appear to be errors in the Line Item titles. Phyllis confirmed the errors and will discuss same with the contracts officer to see what corrections need to be made.

The contents of the APP were discussed, with reference to the USACE's checklist for APPs as being a possible table of contents. Phyllis noted that all sections named in the checklist should be incorporated into the project APP, with "not applicable" notations inserted as appropriate. She cautioned that we need to follow the checklist closely, without neglecting any of the requirements. She also suggested we send questions to her on it as they arise, rather than waiting until the last minute. Any questions on plans will be directed to Phyllis and she will forward them to the appropriate parties.

David offered to send templates to assist in the preparation of the APP.

Jim offered to send electronic files that comprise the historical archive for the site.

The proposed schedule for the project was discussed, and it appears the June 30, 2010 deadline for completion is no longer in effect. Carol will send out the schedule as it appeared in the IEM Team's proposal, adjusted only for the contract award date. If any modifications are required, they should be returned to Carol who will send out a revised schedule shortly thereafter. The official start date for the project is the award date, February 14, 2011.

Invoicing on the project should be via an Engineering Form 93, with backup information, deliverables, and the monthly report. An electronic copy should be sent to Phyllis each month, but the signed original should be forwarded to her as well. A copy of the signed original (hard copy) should go to the COR, Sesh Lal.

Alan asked for a copy of the site FSSP (to assist with determinations of sampling density) and the Lloyd study for comparison to air sampling data. Hans offered to send the draft PRG document as long as the IEM Team restricts distribution to team members only.

Carol solicited either a template Certificate of Insurance (COI) or the required wording that should be added to our COI. Phyllis said to get that information from the contracts officer (S. Evertte), but that if we don't receive it by February 16, 2011 we should let her know.

In closing, Carol stated that contact information for the IEM team would be sent to USACE participants.

Action Items:

1. Carol to send project schedule and contact information for IEM Team members to all call participants.
2. Jim to send a DVD with the electronic historical files to Carol (who will post on the IEM web site for other team members to access).
3. Dave will send APP templates to Carol, who will post on the IEM web site.
4. Hans will provide copies of the site Final Status Survey Plan and the Lloyd study to assist in preparation of plans.

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MEETING MINUTES

Date: March 14, 2011
Start Time: 9:00 a.m. EST
End Time: 12:00 p.m. EST
Location: USACE Baltimore Office/Call-in number
Topic: Colonie FUSRAP Scoping Session/Teleconference at Baltimore USACE Office

Attendees:
R. Alan Duff - IEM Project Manager (via phone)
Carol D. Berger - IEM MARS Program Manager
Leslie Selby - IEM Data Manager and Project Administration
Phyllis Della Camera - USACE Design Team Manager
David Watters - USACE Project Health Physicist (via phone)
Megan Garrett - USACE Technical Representative
Alan Warminski - USACE Technical Representative
Debbie McKinley - USACE Technical Representative

Topics:
The participants introduced themselves and lines of communication for the meeting were set. Phyllis turned over the meeting over to David to discuss preliminary comments on the work plans. David stated that the team never had a technical kickoff for the project, thus the reason why there appear to be a number of misconceptions about its scope. He then provided project background information and described the goals for the activities that are currently planned for the site. His general comment on the draft work plans was to "tell the story" of the site and integrate information from the CSM. The following are project directives given during the conference, presented in no particular order:

- David asked that IEM use a Fiddler detector vs. a 2x2 NaI for the walkover surveys. He also asked that IEM perform a GPS/GIS gamma walkover survey at both of the VPs.
- David recommended that IEM distinguish between instruments for H&S and instruments for area surveys in the plans, perhaps moving discussion on the H&S instruments to the APP.
- David asked that IEM conduct soil screening of cores with a GM pancake detector instead of a sodium iodide detector, based on his prior experience.
- David asked that IEM provide information on equipment decontamination in the work plans, more information on how IEM will handle soil during the project (i.e. direct loaded into a container, staging areas, etc.), and a site layout map showing the work area and surrounding location.
- David asked that IEM address soil backfill in the plans (i.e. type of soil to be used, compaction, grass seed, etc.)
- David asked that IEM collect samples of the asphalt base materials at the 1118 Central Avenue location to determine if that material has activity due to natural materials vs. DU from past site operations.
- David asked IEM to identify the instrument to be used for down-hole measurements in light of the small diameter cores. (IEM stated that a one-by-one-inch sodium iodide detector would be used.)

- David asked that IEM delete the ranking chart that appears in the draft plans and visual observation to select sampling locations. He also asked that IEM analyze both the respirable (filter) and non-respirable (separated solids) portions of each sample.

- David asked that comparisons to the Lloyd sampling effort should be removed. Instead, all dust sampling results should be compared to the PRG's.

- David confirmed that dust samples be analyzed by ICP-MS (i.e., isotopic uranium) rather than ICP (total uranium).

- David was skeptical of sending radioactive wastes from FUSRAP sites to a commercial processor as planned by IEM, stating that the normal procedure is to ship direct to either the Idaho or Utah disposal sites. He asked that the certificate of disposal be provided in the final report.

Action Items:

1. IEM will modify the draft plans based on the comments from the meeting.
2. IEM will include photographs of the two VPs and maps showing where samples will be collected, even though in the case of the 50 Yardboro VP, the dimensions of the survey area are as yet unknown. (The size assumed in the IEM proposal will be used for the time being.)
3. Excavated materials will not be returned to the excavation in spite of analytical results. Instead, all IDW, including excavated soil, will be staged at the Colonie Site pending disposal.
4. The aforementioned directives will be implemented.
5. Minor action items that appear in IEM's notes to be addressed/implemented.

6.3 - Chain of Custody Form

INTEGRATED ENVIRONMENTAL MANAGEMENT, INC.

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

Reference No. _____

Page 1 of _____

(1) Client Name: Integrated Environmental Management, Inc.	**(7) Samples Shipment Date**	**(5) Bill to:**
(2) Sample Team Leader	**(8) Lab Destination**	
(3) Task No.	**(9) Lab Contact**	
(4) Project Manager	**(12) Technical Contact/Phone**	**(10) Report to:**
(6) Purchase Order No.	**(13) Carrier/Waybill No.**	
(11) Required Report Date		

ONE CONTAINER PER LINE

(14) Sample Number	(15) Sample Description/Type	(16) Date/Time Collected	(17) Container Type	(18) Sample Volume	(19) Preservative	(20) Requested Testing Program

(23) Special Instructions

(24) Possible Hazard Identification (circle): **Non-hazard Flammable Skin Irritant Poison B Unknown**	**(25) Sample Disposal (circle):** **Return to Client Disposal by Lab Archive _____ months**
(26) Turnaround Time Required (circle): **Normal Rush**	**(27) QC Level (circle):** **I II III Project Specific _____**
(28) Relinquished by: (signature, date, time):	**Received by: (signature, date, time)**
Relinquished by: (signature, date, time):	**Received by: (signature, date, time)**
Relinquished by: (signature, date, time):	**Received by: (signature, date, time)**

(See Reverse for Instructions)

INSTRUCTIONS FOR COMPLETING THIS FORM

1. Client Name: Record the name of the client/site location.

2. Sample Team Leader: List the name of the team taking these samples.

3. Task No.: Indicate the IEM task number, if applicable.

4. Project Manager: Record the project manager's name.

6. Purchase Order No.: Non-IEM personnel should use this space to record the purchase order number authorizing the analysis of these samples. IEM and IEM subcontractors should leave this space blank if a project number has been given for billing.

7. Samples Shipment Date: Indicate the date these samples are shipped to the laboratory.

8. Lab Destination: Indicate the laboratory designated for sample shipment. Do not list more than one lab on this form. Be certain before sending samples that the laboratory you are designating is aware of the shipment and is capable of accepting these sample types and has available capacity.

9. Lab Contact: Give the name of the laboratory contact (typically the lab's project manager).

10. Report to: Give the name, address and phone number of the person to receive the data report for these samples.

11. Required Report Date: Record the date which you and the laboratory contact have determined the results will be reported (include verbal or final report as appropriate).

12. Technical Contact/Phone: Indicate the name of the person to be contacted in case of any questions regarding these samples and the phone number where the contact may be reached the day the samples arrive in the laboratory.

13. Carrier/Waybill Number: If you are sending the samples by a commercial carrier such as Airborne or Federal Express, record the courier company name and the waybill or air bill number under which these samples will be shipped (Example - Fed-Ex/#513631771).

14. Sample Number: List the complete, unique identification number of each sample. These numbers must correspond with the identification numbers on the sample containers and the field sample collection document(s).

15. Sample Description/Type: Provide a short physical description of the sample and the sample type such as soil, sediment, sludge, water, wipe, air, concentrated waste or bulk.

16. Date/Time Collected: Record date and exact time each sample was collected. Use a 24-hour clock; i.e., 1645 not 4:45 p.m.

17. Container Type: Indicate the volume, color and type of the sample container used (Example - 1 gallon amber glass, 1 liter clear plastic, 40 milliliter clear glass).

18. Sample Volume: Estimate the amount of sample in the container. For air samples, indicate the volume of air sampled.

19. Preservative: Indicate what type of preservative, if any, has been used for the samples (Example - ice to 4ºC nitric acid, hydrochloric acid).

20. Requested Testing Program: List the analyses to be performed on each sample <u>by method number or quotation number</u>.

23. Special Instructions: Use this space to record any special instructions to the lab regarding the processing of these samples.

24. Possible Hazard Identification: Indicate all hazard classes associated with the sample(s).

25. Sample Disposal: Indicate how the samples should be disposed of following analysis. The lab may charge for packing, additional archiving and disposal.

26. Turnaround Time Required: Check "Normal" or "Rush" as determined by the Technical Contact and the Lab Contact. Rush samples are subject to a surcharge.

27. QC Level: These should be specific to the analytical laboratory and should not be confused with USEPA Analytical Levels. Project Specific should reference a quotation number or other specifications that have been submitted to the laboratory before beginning work.

28 Signatures: When releasing custody of these samples, use the "Relinquished By" space to sign your full legal name, date and time of release. After verifying that all samples are present, the person receiving the samples must sign the "Received By" space to take custody of the samples.

6.4 - Procedure Compendium

TABLE OF CONTENTS

1 PURPOSE

This Radiation Safety Procedure (RSP) describes the requirements for calibration and use of radiation survey instruments and for performing radiological surveillance by Integrated Environmental Management, Inc. (IEM) employees and project personnel.

2 SCOPE

This RSP applies to all radiological instrumentation used by IEM employees or project personnel for radiation protection purposes. Instruments that are not used for radiation protection purposes are exempt from the requirements of this procedure.

3 REFERENCES

3.1 American National Standards Institute, "Radiation Protection Instrumentation Test and Calibration," N323-1978, 1978.

3.2 MARSSIM - U. S. Nuclear Regulatory Commission, NUREG-1575, “Multi-Agency Radiation Survey and Site Investigation Manual”, December, 1997.

3.3 NUREG-1507, “Minimum Detectable Concentrations with Typical Radiation Survey Instruments for Various Contaminants and Field Conditions”, December, 1997.

3.4 Instrument instruction manuals published by the instrument manufacturers.

3.5 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-001, "Radiation Protection Program Plan".

3.6 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-004, “Radiation Protection Records".

3.7 Integrated Environmental Management, Inc. Quality Policy Statement No. QPS-005, “Corrective Actions”.

4 DEFINITIONS

The definition of terms used in this RSP that may not be commonly understood shall be found in RSP-002, “Definitions”.

5 PROCEDURE

5.1 Responsibilities

5.1.1 The President shall supply adequate resources to ensure compliance with this procedure.

5.1.2 The Radiation Safety Officer (RSO) shall:

5.1.2.1 Assure the adequacy of the radiation survey instrumentation program.

5.1.2.2 Ensure current and proper calibration of all radiation detection instruments in the active inventory.

5.1.2.3 Maintain instrument calibration certificates on file for all radiation detection instruments in the active inventory.

5.1.2.4 Assure that all Health Physics Technicians are properly trained in the provisions of this procedure.

5.1.2.5 Verify compliance with this procedure during planned and periodic audits of the radiation protection program.

5.1.3 Health Physics Technicians shall:

5.1.3.1 Verify that only calibrated radiation detection instruments are used.

5.1.3.2 Follow this procedure when performing radiological surveillance activities.

5.1.3.3 Periodically review this procedure.

5.2 Radiation Survey Instruments

5.2.1 Instrumentation used by Health Physics Technicians shall:

5.2.1.1 Be of sufficient sensitivity and accuracy to assess the radiation exposure rates from radioactive materials which may be found at the project location;

5.2.1.2 Detect the presence of radioactivity on tools, equipment, clothing, materials, and personnel at all levels which may be found at a client site;

5.2.1.3 Be of sufficient quantity to support on-going or planned operations.

Note: MARSSIM and NUREG-1507 describe the different methods for determining instrument sensitivity.

5.2.2 The basis for selection of instruments for use by IEM shall include:

5.2.2.1 Type of radiation to be measured.

5.2.2.2 Sensitivity required.

5.2.2.3 Purpose of the survey.

5.2.3 Instruments maintained in the active inventory should be evaluated, tested, and documentation obtained, as appropriate, for the following:

5.2.3.1 Physical construction

5.2.3.2 Effect of shock, sound, vibration, electric transients, RF energy, magnetic fields and high humidity

5.2.3.3 Extent of switching transients, capacitance effects, geotropism and static charge effects

5.2.3.4 Power supply, including stability and battery life

5.2.3.5 Range, sensitivity, linearity, detection limit, and response to overload conditions

5.2.3.6 Accuracy and reproducibility precision

5.2.3.7 Energy dependence

5.2.3.8 Angular dependence

5.2.3.9 Response to ionizing radiation other than those being measured

5.2.3.10 Temperature and pressure dependence on measurements

Note: These tests are normally performed by the manufacturer and credit may be taken for the manufacturer's evaluation and testing. If credit is taken for the manufacturer's testing, a copy of the test results, in the form of instrumentation manuals or specification sheets, should be maintained

along with instrument records. These results should be requested in the purchase order for each new instrument.

5.3 Instrument Calibration

5.3.1 Instruments shall be calibrated as noted in the Attachments 8.1 through 8.13 and following significant repairs to the rate meter and/or detector.

Note: Cable and battery changes may not necessitate re-calibration, depending upon whether such action induces response changes. Response checks must be documented when these changes are made (see Attachment 8.15).

5.3.2 Instruments not listed in Attachments 8.1 through 8.13 should be calibrated as recommended by the vendor until such time as an instrument-specific attachment is prepared for this RSP.

5.3.3 Each ratemeter should be calibrated with a specific detector, designated by the detector serial number.

Note: The use of a ratemeter with a different detector may constitute the use of an un-calibrated meter.

5.3.4 A contractor shall provide calibration services using radiation sources which are traceable to the National Institute of Standards and Technology (NIST).

5.3.5 Instruments shall be calibrated according to the guidelines of ANSI-N323-1978, "Radiation Protection Instrumentation Test and Calibration".

5.3.6 A Request for Instrument Calibration Sheet (see Attachment 8.14) should accompany each instrument sent for calibration.

5.3.7 The Certificate of Calibration shall cite the applicable standard used, certify that radiation sources are traceable to NIST, and provide both pre- and post-calibration responses.

5.3.8 The contractor shall be the manufacturer of the instrument or an individual/firm that has been pre-qualified by the RSO.

5.3.9 Calibration schedules should be staggered to maintain at least one calibrated contamination survey meter, one calibrated ambient exposure rate instrument, one calibrated high-range exposure rate instrument, and one calibrated stationary smear counter available at all times.

5.3.10 If the Certificate of Calibration indicates an out-of-tolerance condition for a pre-calibration response, the following shall be performed:

5.3.10.1 A memo justifying the acceptability of the out-of-tolerance condition shall be written and attached to the Certificate; or

5.3.10.2 The use records for that instrument over the previous calibration period shall be evaluated for continued acceptability and corrections made pursuant to QPS-005, as necessary.

5.4 Reference Source Response

5.4.1 The response of each instrument to a reference source placed in a repeatable position shall be determined and recorded on the calibration label before the instrument is placed into active use and after each calibration.

5.4.2 With the instrument switch position noted, the reference source and detector shall be placed in the reference position and the instrument shall be allowed to stabilize.

5.4.3 A minimum of ten (10) data points shall be acquired at the beginning of an instrument's deployment and documented on an Instrument Response Check form (Attachment 8.15).

Note: For instruments with both a scaler and rate meter mode, data shall be acquired for both modes.

5.4.4 Control levels shall be determined as follows:

5.4.4.1 The arithmetic mean, the standard deviation, and the 2σ and the 3σ values from the 10 data points shall be determined.

5.4.4.2 The mean measurement $\pm$ the 2σ value, and the mean measurement $\pm$ the 3σ value should be recorded on a label affixed to the instrument, if space is available, or on Attachment 8.15 (or equivalent).

5.5 Pre-operational Checks

5.5.1 Each instrument shall be labeled with a unique identifier (e.g., serial number of detector and rate meter) to enable traceability to surveys and records.

5.5.2 Prior to each use, or daily when kept in use, each instrument shall be checked for the following, as applicable:

5.5.2.1 Battery function.

5.5.2.2 High voltage.

5.5.2.3 Response to a reference source.

5.5.2.3.1 If any response exceeds the mean measurement $\pm$ the 3σ value, that instrument shall be removed from service.

5.5.2.3.2 If any two consecutive daily responses exceed the mean measurement $\pm$ the 2σ value, the instrument may be removed from service pending an evaluation of its operational status.

5.5.2.4 Reset Button function.

5.5.2.5 Audible response function.

5.5.2.6 Physical damage.

5.5.2.7 Current calibration sticker.

5.5.2.8 Response to background radiation.

5.5.2.8.1 Response to background radiation should be determined at a reproducible location that is in the vicinity of but not near known radiation sources or radiation-producing machines.

5.5.2.8.2 Three readings should be obtained at the start of each work shift, and three readings obtained at the end of each work shift.

5.5.3 The results of the daily check should be recorded on the Exposure Rate Survey Instrument Data Sheet (Attachment 8.16) or the Contamination Survey Instrument Data Sheet (Attachment 8.17).

5.5.4 Instruments failing any pre-operational check shall be taken out of service, segregated from other instruments, tagged as "out of service", and repaired and calibrated prior to use.

6 EXEMPTION PROVISIONS

Variances and exceptions to the requirements of this RSP shall be permitted pursuant to the written authorization of the RSO and the President.

7 DOCUMENTATION

7.1 All records pertinent to this procedure shall be maintained pursuant to RSP-004.

7.2 The following records shall be maintained:

7.2.1 Instrument calibration and maintenance records.

7.2.2 Manufacturer instruction manuals for each type of rate meter and detector.

7.3 All forms associated with the implementation of this procedure shall be completed in their entirety (i.e., all boxes and fields shall have an entry of data, the initials "NA" for "not applicable", or an indicator of "no further entries).

8 ATTACHMENTS

8.1 Ludlum Model 44-9 Alpha, Beta, Gamma Detector

8.2 Ludlum Model 12 Count Ratemeter

8.3 Ludlum Model 2241 Scaler/Ratemeter & Model 44-10 High Energy Gamma Detector

8.4 Ludlum Model 2929 Dual Channel Scaler & Model M43-10-1 Alpha/Beta Sample Counter

8.5 Ludlum Model 2224 Scaler/Ratemeter & Model M43-89 Alpha/Beta Scintillator

8.6 Bicron Micro Rem Survey Meter

8.7 Mini-Buck Gas Flow Calibrator Model M-30

8.8 F&J Specialty Products, Inc. Model LV-1, Regulated Low Volume Air Sampler

8.9 MSA ESCORT™ Pump

8.10 Ludlum Model 44-110 Tritium Detector

8.11 Ludlum Model 19 MicroR Meter

8.12 Ludlum Model 2224 Scaler/Ratemeter & Model 239-1F Floor Monitor

8.13 Ludlum Model 9 Ion Chamber

8.14 Request for Instrument Calibration Sheet

8.15 Instrument Response Check form

8.16 Exposure Rate Survey Instrument Data Sheet

8.17 Contamination Survey Instrument Data Sheet

ATTACHMENT 8.1

Instrument Name:
Ludlum Model 44-9 Alpha, Beta, Gamma Detector

General Description:
The Model 44-9 GM (Pancake) Detector detects Alpha, Beta, and Gamma emitting radiation. Its size and shape provide easy handling for surveying or personnel monitoring. The detector is energy dependent, over responding by a factor of six in the 60 keV - 100 keV range when normalized to Cs-137.

The thin mica window is protected by a 79% open stainless steel screen. The GM tube can easily be removed for replacement if necessary.

The GM detector operates between 850 - 1000 volts. The tube manufacture recommends operation at approximately 900V. The recommended instrument input sensitivity is approximately 30 mV or higher to prevent the detector from double pulsing.

The Model 44-9 should operate with any Ludlum instruments or equivalent instruments that provide 900 VDC and input sensitivity of approximately 30 mV or higher.

Typical Uses:
Alpha, Beta, Gamma contamination surveys, Personnel frisking.

Required Maintenance:
Consult Model 44-9 Alpha, Beta, Gamma Detector Instruction Manual.

Operation Procedure:
Consult Model 44-9 Alpha, Beta, Gamma Detector Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

ATTACHMENT 8.2

Instrument Name:
Ludlum Model 12 Count Ratemeter

General Description:
The Ludlum Model 12 Count Ratemeter provides the required electronic circuitry for radiation monitoring with proportional, scintillation and GM detectors.

Typical Uses:
Contamination and radiation surveys.

Required Maintenance:
Consult Model 12 Count Ratemeter Instruction Manual.

Operation Procedure:
Consult Model 12 Count Ratemeter Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

Check Source:
Dependent on probe being used with instrument.

* **ATTACHMENT 8.3**

Instrument Name:
Ludlum Model 2241 Scaler/Ratemeter & Model 44-10 High Energy Gamma Detector

General Description:
The Model 2241 is a portable microprocessor-based digital scaler/ratemeter designed for use with scintillation, GM, and proportional type detectors for measurement of ionizing radiation. The data is presented on a four digit (six digit in the scaler mode) Liquid Crystal Display (LCD) with a moving decimal point. A three-position switch labeled OFF/RATEMETER/SCALER selects the desired operating mode for the instrument. Programmable display units (ratemeter mode only) are represented in R/hr, Sv/hr, cpm, or cps with multipliers of micro (μ) or milli (m) for R/hr and Sv/hr and kilo (k) for cpm or cps. The display units are auto-ranging enabling the readout to display a broad range of radiation activity.

The Ludlum Model 44-10 utilizes a two-inch by two-inch NaI(Tl) crystal for high energy gamma detection (approximately 60 keV to 2 MeV range). The detector also provides high sensitivity for surveying and pulse height discrimination for single channel or multichannel applications.

Typical Uses:
Gamma Surveys where a detachable probe is required. Gamma surveys when time integration is required. Surveys where radionuclide identification is desirable.

Required Maintenance:
Consult Model 2241 Scaler/Ratemeter Instruction Manual.
Consult Model 44-10 High Energy Gamma Detector Instruction Manual.

Operation Procedure:
Consult Model 2241 Scaler/Ratemeter Instruction Manual.
Consult Model 44-10 High Energy Gamma Detector Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

Check Source:
Cs-137 Button Source[1]

[1]Another source (e.g., Am-241, Co-60) may be selected if it is more consistent with the radiations to be detected.

ATTACHMENT 8.4

Instrument Name:
Ludlum Model 2929 Dual Channel Scaler & Model M43-10-1 Alpha/Beta Sample Counter

General Description:
The Model 2929 is a Dual Channel Scaler designed for use with “Phoswich” and/or proportional detectors. A pulse height analyzer is employed to provide information to the two independent counters. The Model 2929 has adjustable count time periods ranging from 0.1 to 990 minutes and has a click per event audio for each of the two channels. The unit may also be operated with two printing calculators (Ludlum Model 264) via data connectors located on the rear chassis.

The Model 43-10-1 is a Alpha/Beta Sample Counter capable of holding up to a two inch diameter filter or planchet. The sample drawer, when fully closed strikes a micro switch to allow HV to be applied to the photo multiplier tube (PMT). The sample drawer is locked in the closed position by rotation of the side lever mounted on the side of the instrument towards the rear. To discriminate alpha and beta radiations simultaneously requires the counting instrument to have separate power supplies or threshold controls for each channel. The Ludlum Model 2929, Model 2223, or Model 2224 instruments provide the necessary circuitry for simultaneous alpha/beta discrimination.

Typical Uses:
Counting air samples, contamination swabs and smears.

Required Maintenance:
Consult Model 2929 Dual Channel Scaler Instruction Manual.
Consult Model M43-10-1 Alpha/Beta Sample Counter Instruction Manual.

Operational Procedure:
Consult Model 2929 Dual Channel Scaler Instruction Manual.
Consult Model M43-10-1 Alpha/Beta Sample Counter Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

Check Source:
Th-230 for Alpha, Sr-90 or Tc-99 for Beta.

ATTACHMENT 8.5

Instrument Name:
Ludlum Model 2224 Scaler/Ratemeter & Models 43-89 and 43-93 Alpha/Beta Scintillators

General Description:
The Model 2224 is a portable microprocessor based radiation survey instrument used to measure and discriminate low level alpha/beta radiation when used with an alpha/beta scintillation or proportional detector. The data is displayed by an analog ratemeter and six-digit LCD counter. The ratemeter dial indicates 0 - 500 cpm with four linear multipliers of X1 - X1000 producing an overall range of 0 - 500K cpm. The LCD is used to display the counts accumulated during the preset count time. There are four count times available via internal switches. These count times are 6 seconds, 30 seconds, 60 seconds, and 120 seconds. The ratemeter and LCD can display alpha only, beta only or alpha and beta by selecting the corresponding toggle switch selection.

The Models 43-89 and 43-93 detectors have an active area of 100 cm^2 used for detecting alpha and/or beta radiation. To discriminate alpha and beta radiations simultaneously requires the counting instrument to have either separate power supplies or window/threshold controls for each channel. The Model 2224 instrument provides the necessary circuitry for simultaneous alpha/beta discrimination.

Typical Uses:
Contamination surveys, building/area release surveys, and personnel frisking.

Required Maintenance:
Consult Model 2224 Scaler/Ratemeter Instruction Manual.
Consult Model 43-89 Alpha/Beta Scintillator Instruction Manual.
Consult Model 43-93 Alpha/Beta Scintillator Instruction Manual.

Operation Procedure:
Consult Model 2224 Scaler/Ratemeter Instruction Manual.
Consult Model 43-89 Alpha/Beta Scintillator Instruction Manual.
Consult Model 43-93 Alpha/Beta Scintillator Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

Check Source:
Th-230 for Alpha, Sr-90 or Tc-99 for Beta

ATTACHMENT 8.6

Instrument Name:
Bicron Micro Rem Survey Meter

General Description:
The Bicron Micro Rem model is a lightweight, portable survey meter for applications where accurate dose rate measurements of low gamma radiation levels are required. The Bicron Micro Rem model reads dose rate directly, eliminating the need for conversation from mR/hr.

Typical Uses:
Area radiation surveys, and radiation release surveys.

Required Maintenance:
Consult Bicron Micro Rem Survey Meter Instruction Manual.

Operation Procedure:
Consult Bicron Micro Rem Survey Meter Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

Check Source:
Cs-137 button source.

ATTACHMENT 8.7

Instrument Name:
Mini-Buck Gas Flow Calibrator Model M-30

General Description:
The Mini-Buck Calibrator® utilizes the principle of measuring the flow rate of gases over a fixed volume per unit of time. The Mini-Buck Calibrator® Model M-30 has a flow rate range of 100 cc/min to 30.00 liters/min.

Typical Uses:
Flow calibration of air sampling pumps.

Required Maintenance:
Consult the Mini-Buck Gas Flow Calibrator Model M-30 Instruction Manual.

Operation Procedure:
Consult the Mini-Buck Gas Flow Calibrator Model M-30 Instruction Manual.

Calibration:
Annually by the manufacturer

ATTACHMENT 8.8

Instrument Name:
F & J Speciality Products, Inc. Model LV-1, Regulated Low Volume Air Sampler

General Description:
The Model LV-1 is a 100 to 120V AC low volume air sampler consisting of an oil-less, carbon vane vacuum pump with a combination filter holder, flow meter, a vacuum gauge, a constant air flow regulator for use where a nearly consistent air flow is desirable.

Typical Uses:
Area air sampling.

Required Maintenance:
Consult the F & J Speciality Products, Inc. Model LV-1, Regulated Low Volume Air Sampler Instruction Manual.

Operation Procedure:
Consult the F & J Speciality Products, Inc. Model LV-1, Regulated Low Volume Air Sampler Instruction Manual.

Calibration:
Annually by the manufacturer.

ATTACHMENT 8.9

Instrument Name:
MSA ESCORT™ Pump

General Description:
The ESCORT™ Pump contains a diaphragm pump mechanism driven by an acentric on a motor shaft. A rechargeable battery pack powers the motor. The motor speed is varied by turning the flow control. The pump flow ranges from 0.5 to 3.4 LPM on a integral flowmeter, that is graduated in 0.2 LPM divisions. The elapsed time readout utilizes a quartz controlled timer with a capacity to read out to 9999 minutes in one-minute step increments. The ESCORT pump has an operation time of a minimum of eight-hours at 2.5 LPM with a 15-inch water column pressure drop depending on the sample device loading.

Typical Uses:

Required Maintenance:
Consult the MSA ESCORT™ Pump Instruction Manual.

Operation Procedure:
Consult the MSA ESCORT™ Pump Instruction Manual.

Calibration:
Prior to each use with a mini-Buck Flow Calibrator.

ATTACHMENT 8.10

Instrument Name:
Ludlum Model 44-110 Tritium Detector

General Description:
The Model 11-110 is a windowless, gas flow proportional detector. The Model 44-110 is used with a count-rate instrument such as a Ludlum Model 12. The Model 44-110 works using a count gas referred to as P-10, which is a gas combination of 90% Argon and 10% Methane.

Typical Uses:
Tritium surface contamination surveys.

Required Maintenance:
Consult the Ludlum Model 44-110 Tritium Detector Instruction Manual.

Operation Procedure:
Consult the Ludlum Model 44-110 Tritium Detector Instruction Manual.

Calibration:
Annually by the manufacturer

Check Source:
Not used.

ATTACHMENT 8.11

Instrument Name:
Ludlum Model 19 MicroR Survey Meter

General Description:
The Ludlum MicroR Meter is a lightweight, portable survey meter for applications where accurate exposure rate measurements of low gamma radiation levels are required. The Ludlum MicroR Meter reads exposure rate.

Typical Uses:
Area radiation surveys, and radiation release surveys.

Required Maintenance:
Consult Ludlum MicroR Survey Meter Instruction Manual.

Operation Procedure:
Consult Ludlum MicroR Survey Meter Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

Check Source:
Cs-137 button source.

ATTACHMENT 8.12

Instrument Name:
Ludlum Model 2224 Scaler/Ratemeter & Model 239-1F Floor Monitor

General Description:
The Model 2224 is a portable microprocessor based radiation survey instrument used to measure and discriminate low level alpha/beta radiation when used with an alpha/beta scintillation or proportional detector. The data is displayed by an analog ratemeter and six-digit LCD counter. The ratemeter dial indicates 0 - 500 cpm with four linear multipliers of X1 - X1000 producing an overall range of 0 - 500K cpm. The LCD is used to display the counts accumulated during the preset count time. There are four count times available via internal switches. These count times are 6 seconds, 30 seconds, 60 seconds, and 120 seconds. The ratemeter and LCD can display alpha only, beta only or alpha and beta by selecting the corresponding toggle switch selection.

The Model 239-1F Floor Monitor is a gas proportional floor monitor detector mounted on a roll-around cart. The detector has an active area of 582 cm^2. The instrument features a flow system, quick disconnects, a gas bottle mount, and a means to adjust the height of the detector from the floor for optimum performance. A P-10 gas bottle and regulator are required for operation of the floor monitor.

Typical Uses:
Building/area release surveys

Required Maintenance:
Consult Model 2224 Scaler/Ratemeter Instruction Manual.
Consult Model 239-1F Floor Monitor Instruction Manual.

Operation Procedure:
Consult Model 2224 Scaler/Ratemeter Instruction Manual.
Consult Model 239-1F Floor Monitor Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

Check Source:
Th-230 for Alpha, Sr-90 or Tc-99 for Beta

ATTACHMENT 8.13

Instrument Name:
Ludlum Model 9 Ion Chamber Survey Meter

General Description:
The Ludlum Model 9 Ion Chamber is a self-contained, portable instrument used in the detection of beta-gamma radiation. The unit is provided with a sliding window for beta-gamma discrimination. The data is displayed by an analog meter. The dial indicates 0 - 5 mR/hr with four linear multipliers of X1 - X 1000 producing an overall range of 0 - 5,000 mR/hr. With the sliding window open, beta-gamma radiation is measured. With the sliding window closed, gamma radiation is measured.

Typical Uses:
General purpose beta-gamma radiation survey.

Required Maintenance:
Consult Model 9 Ion Chamber Instruction Manual.

Operation Procedure:
Consult Model 9 Ion Chamber Instruction Manual.

Calibration:
Annually or as otherwise recommended by manufacturer.

Check Source:
Cs-137

ATTACHMENT 8.14

REQUEST FOR INSTRUMENT CALIBRATION SHEET

(*Available in PDF on the server*)

Contact Person:	Phone No.	Date:
Purchase Order No:	Call for Purchase Order: ☐ Yes ☐ No	Call with Estimate: ☐ Yes ☐ No
Bill to:	Ship to:	
Instrument Model Number:	Instrument Serial Number:	
Probe Model Number:	Probe Serial Number:	

INSTRUMENT RETURNED FOR CALIBRATION		
Calibration Type: ☐ Point source ☐ Surface Contamination ☐ Pipe (internal) source ☐ Other (specify):	Calibration Frequency: ☐ Quarterly ☐ Six months ☐ Annual ☐ Other (specify):	Calibration Geometry: ☐ 2-π ☐ 4-π ☐ Other (specify):
Calibration Source: ☐ Cesium-137 ☐ Thorium-230 ☐ Technetium-99 ☐ Other (specify):	Units of Intended Use ☐ R/hr, µR/hr ☐ rem/hr, µrem/hr ☐ dpm ☐ Other (specify):	Calibration factor required: ☐ No ☐ Yes (as checked below): ☐ dpm per cpm ☐ µR per cpm ☐ Other (specify)

INSTRUMENT RETURNED FOR REPAIR	
Symptoms:	Special Instructions: *Please be sure the "signature required" box is checked on the return shipment manifest.* *Certificate of Calibration must cite applicable standard (i.e., ANSI N323, ANSI/NCSL Z540) and certify that sources used are traceable to NIST.* *Both pre- and post-calibration instrument responses shall be shown on the Certificate of Calibration*.

ATTACHMENT 8.15

INSTRUMENT RESPONSE CHECK

(*Available in PDF and spreadsheet form on the server*)

Location:	**Meter Model No.:**	**Probe Model No.:**
Check Source No.:	**Meter Serial No.:**	**Probe Serial No.:**
Scaler Count Time (Min):	**Response Switch:**	**Name:**

Meas. Number	**Radiation Type (check): ☐ Alpha ☐ Beta ☐ Beta/Gamma**				**Radiation Type (check): ☐ Alpha ☐ Beta ☐ Beta/Gamma**			
	Background (counts per minute)	**$(x - x_{ave})^2$**	**Check Source (counts per minute)**	**$(x - x_{ave})^2$**	**Background (total counts)**	**$(x - x_{ave})^2$**	**Check Source (total counts)**	**$(x - x_{ave})^2$**
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
Sum $a = (x-x_{ave})^2$								
Mean $b = (x_{ave})$								
σ^2 $c = a \div [n - 1]$								
σ $d = c^{½}$								
2σ $e = 2 \times d$								
3σ $f = 3 \times d$								

Acceptable Check Source Ranges			**Notes/Calculations:**
Range	**Alpha**	**Beta**	
2σ	to	to	
3σ	to	to	

ATTACHMENT 8.16

EXPOSURE RATE SURVEY INSTRUMENT DATA SHEET

(*Available in PDF on the server*)

Project No.	**Detector**		**Meter**		
Site Location/Background Location:	Type:	Serial No.	Type:	Serial No:	Operating Voltage:

Check Source Number	**Radionuclide:**	**Calibration Activity and Date:**

Date	**Units**	**Start of Shift Background**				**End of Shift Background**				**Daily Response (µR/hr)**	**Initials**
		1	**2**	**3**	**Avg.**	**1**	**2**	**3**	**Ave.**		

ATTACHMENT 8.17

CONTAMINATION SURVEY INSTRUMENT DATA SHEET

(*Available in PDF on the server*)

Project No:			**Detector**			**Meter**		
Site Location/Background Location:			**Type:**	**Serial No.**	**Probe Area (cm^2)**	**Type:**	**Serial No:**	**Operating Voltage:**
Check Source No:			**Check Source No:**			**Check Source No:**		
Radionuclide:	**Activity:**	**Date:**	**Radionuclide:**	**Activity:**	**Date:**	**Radionuclide:**	**Activity:**	**Date:**

Date	Start of Shift Background (cpm for a _____ minute count)								End of Shift Background (cpm for a _____ minute count)								Daily Source Check (α)		Daily Source Check (β)		MDA** - Scaler Mode (dpm)		Bat. OK	HV OK	Initials
	Alpha				Beta				Alpha				Beta				Source (cpm)	Eff.	Source (cpm)	Eff.	α	β			
	1	2	3	Av.	1	2	3	Av.	1	2	3	Av.	1	2	3	Av.									

** $MDA = \frac{2.71 + 4.65\sqrt{BKG_{avg} \times t}}{t \times E \times \frac{A}{100}}$, where MDA = the activity level (dpm/100 cm^2), BKG_{avg} = the background count rate for this measurement type (cpm), t = the sample measurement duration (min), E = the 4-π instrument efficiency, and A = the physical probe area (cm^2).

TABLE OF CONTENTS

1 PURPOSE

This Radiation Safety Procedure (RSP) describes the methodologies and requirements for radiation surveys performed by Integrated Environmental Management, Inc. (IEM) employees and project personnel.

2 SCOPE

This RSP applies to all radiological surveys conducted by IEM employees and project personnel for radiation protection purposes. Surveillance activities performed for other than radiological protection reasons are exempt from the provisions of this RSP.

3 REFERENCES

3.1 U.S. NRC Regulatory Guide 8.10, "Operating Philosophy for Maintaining Occupational Radiation Exposures As Low As is Reasonably Achievable".

3.2 U.S. NRC Regulatory Guide 8.21, "Health Physics Surveys for Byproduct Material at NRC-Licensed Processing and Manufacturing Plants".

3.3 MARSSIM - U. S. Nuclear Regulatory Commission, “Multi-Agency Radiation Survey and Site Investigation Manual”, NUREG-1575, 1997.

3.4 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-001, "Radiation Protection Program Plan".

3.5 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-004, “Radiation Protection Records".

3.6 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-006, “Training and Qualifications of Radiation Protection Personnel”.

3.7 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-008, “Instrumentation”.

3.8 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-019, “Smear Counting”.

3.9 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-022, “Air Sampling”.

4 DEFINITIONS

The definition of terms used in this RSP that may not be commonly understood shall be found in RSP-002, “Definitions”.

5 PROCEDURE

5.1 Responsibilities

5.1.1 The President shall supply adequate resources to ensure compliance with this procedure.

5.1.2 The Radiation Safety Officer (RSO) shall:

5.1.2.1 Assure the adequacy of the radiation survey program.

5.1.2.2 Assure that all radiological surveillance is performed pursuant to this procedure.

5.1.2.3 Assure that all Health Physics Technicians are properly trained in the provisions of this procedure.

5.1.2.4 Verify compliance with this procedure during planned and periodic audits of the radiation protection program.

5.1.3 Health Physics Technicians shall:

5.1.3.1 Follow this procedure when performing radiological surveillance activities.

5.1.3.2 Periodically review this procedure.

5.2 Survey Program

5.2.1 Radiation surveys shall be performed, as necessary, to evaluate:

5.2.1.1 The magnitude of radiation exposures to personnel performing routine operations, maintenance, and/or research and development.

5.2.1.2 Fixed and removable contamination on equipment and materials to be released from the project location.

5.2.1.3 The radiological status of the project location with respect to applicable federal, state or license requirements.

5.2.1.4 Radiological conditions in the event of non-routine circumstances (e.g., spills, decontamination efforts, special activities).

5.2.2 Radiation surveys for official purposes shall be performed by Health Physics Technicians who are qualified in accordance with RSP-006.

5.2.3 All official radiation surveys shall be documented on a Radiological Survey Form (Attachment 1, or equivalent).

5.3 Surveillance Planning

5.3.1 Each survey shall be planned in regard to:

5.3.1.1 Specific radiation types

5.3.1.2 Predetermined radiation levels

5.3.1.3 Locations where the radiation is expected

5.3.1.4 Special conditions for the survey (e.g., special locations)

5.3.1.5 The minimum detection sensitivity for each survey pursuant to MARSSIM, Section 6

5.3.2 Survey plans should be documented in a work plan or project plan.

5.4 Survey Methods for Determining Ambient Gamma (General Area) Exposure Rates

5.4.1 Surveys shall be performed with a portable radiation survey instrument that is sensitive to gamma radiation (e.g., sodium iodide detector, microR meter, microrem meter, ionization chamber).

5.4.2 The instrument shall be turned on and permitted to stabilize for a minimum of 30 seconds before proceeding further.

5.4.3 Pre-operational checks as described in RSP-008 shall be completed before proceeding further.

5.4.4 Surveys shall be conducted by walking slowly over the area of interest with the detector held at a height of approximately one meter above the ground (waist high).

5.4.4.1 An increase in the audible response or in the needle/indicator movement may indicate the presence of radioactivity.

5.4.4.2 The instrument shall be held stationary in the locations where the increased response is noted.

5.4.5 Readings shall be recorded *as read* on a Radiological Survey Form (Attachment 1 or equivalent).

Note: Do not record "background" or censor the "as read" measurement in any way.

Note: Carefully evaluate the position of the range selector switch when observing the meter reading.

5.4.5.1 Any comments and notations that may be necessary for interpretation of results should be recorded on a Radiological Survey Form (Attachment 1 or equivalent).

5.4.5.2 Extraneous remarks or comments shall not be recorded on a Radiological Survey Form (Attachment 1 or equivalent).

5.4.5.3 The individual performing the survey shall sign and date the completed Radiological Survey Form.

5.5 Survey Methods for Determining Contact Exposure Rates on Equipment Surfaces

5.5.1 Surveys shall be performed with a portable radiation survey instrument that is sensitive to gamma radiation (e.g., sodium iodide detector, microR meter, microrem meter).

5.5.2 The instrument shall be turned on and permitted to stabilize for a minimum of 30 seconds before proceeding further.

5.5.3 Pre-operational checks as described in RSP-008 shall be completed before proceeding further.

5.5.4 Surveys shall be conducted by holding the instrument stationary with the detector end of the instrument approximately 0.25 inch from the surface of the item being evaluated.

5.5.5 Readings shall be recorded *as read* on the Radiological Survey Form.

Note: Do not record "background" or censor the "as read" measurement in any way.

Note: Carefully evaluate the position of the range selector switch when observing the meter reading.

5.5.5.1 Any comments and notations that may be necessary for interpretation of the results should be recorded on the Radiological Survey Form.

5.5.5.2 Extraneous remarks or comments shall not be recorded on the Radiological Survey Form.

5.5.5.3 The individual performing the survey shall sign and date the completed Radiological Survey Form.

5.6 Survey Methods for Determining the Extent of Total Contamination on Surfaces

5.6.1 Total (fixed plus removable) contamination shall be measured by direct survey with portable radiation survey instruments sensitive to beta/gamma radiation (e.g., pancake Geiger-Mueller detector, or equivalent) or alpha radiation (e.g., alpha scintillation detector), or equivalent).

5.6.2 The instrument shall be turned on and permitted to stabilize for a minimum of 30 seconds) before proceeding further.

5.6.3 Pre-operational checks as described in Procedure RSP-008 shall be completed before proceeding further.

5.6.4 Surveys shall be conducted by moving the detector at a pre-determined rate and within 0.25 inch of the surface.

Note: Scan speeds necessary to achieve the detection objective should be determined pursuant to MARSSIM and documented. Similarly, any adjustments to instrument response to account for surface coatings should be documented.

5.6.4.1 An increase in the audible response or in the needle/indicator movement may indicate the presence of radioactivity.

5.6.4.2 The detector shall be held stationary over the areas where the increased response was noted.

5.6.4.3 Any changes in the surface structure that may affect instrument response shall be documented on the Radiological Survey Form.

5.6.5 Survey points with the highest count rates shall be identified and recorded *as read* on the Radiological Survey Form, along with an estimate of the physical dimensions of the area with elevated readings.

Note: Do not record "background" or censor the "as read" measurement in any way.

5.6.5.1 Any comments and notations that may be necessary for interpretation of the results should be recorded on the Radiological Survey Form.

5.6.5.2 Extraneous remarks or comments shall not be recorded on the Radiological Survey Form.

5.6.5.3 The individual performing the survey shall sign and date the completed Radiological Survey Form.

Note: Carefully evaluate the position of the range selector switch when observing the meter reading.

5.7 Survey Methods for Determining the Extent of Loose Contamination on Surfaces

5.7.1 Loose contamination shall be measured with numbered dry disc smears wiped over a surface area of at least 100 cm^2, with the unnumbered side in contact with the surface.

Note: If the item/area to be smeared is less than 100 cm^2, the smear results shall be recorded as "per smear", with the size of the smeared area noted on the Radiological Survey Form.

Note: Smears for ^{3}H and ^{14}C shall be collected using polyfoam media and counted by the methodology of liquid scintillation.

5.7.1.1 A filter paper disc (unnumbered side down) shall be placed on the surface to be smeared.

5.7.1.2 The disc shall be moved over an "S"-shaped area using moderate pressure, covering approximately 100 square centimeters (16 square inches), or about 16 inches in length, or the entire surface, if it is less than 100 cm^2 in area.

5.7.1.3 The disc smear shall be placed in a sample holder such that individual smears are separated from each other to prevent cross contamination.

5.7.1.4 A sample number that uniquely identifies the smear shall be documented on the sample holder and on the Radiological Survey Form.

5.7.2 Each smear may be submitted to an analytical laboratory for determination of gross alpha and/or gross beta activity (disintegrations per minute) or may be counted in-house pursuant to RSP-019.

5.8 Survey Methods for Determining Airborne Radioactivity

Airborne radioactivity shall be determined using an air pump connected to a filter cartridge in accordance with RSP-022.

5.9 Analysis of Samples by an Analytical Laboratory

5.9.1 A chain-of-custody record (Attachment 2) shall be initiated by the individual collecting or overseeing the collection of samples.

5.9.2 A copy of the chain-of-custody form shall accompany the samples throughout transportation and analyses; any break in custody or evidence of tampering shall be documented.

5.9.3 Sample custody shall be assigned to one individual at a time in order to prevent confusion of responsibility.

Note: Custody is maintained when (1) the sample is under direct surveillance by the assigned individual, (2) the sample is maintained in a tamper-free or tamper-evident container, or (3) the sample is within a controlled-access facility.

5.9.4 Samples that are submitted to a radioanalytical laboratory shall be accompanied by a "Request for Analysis" form used by the laboratory.

Note: Prior to shipment of the samples, the Health Physics Technician should contact the laboratory to ensure all necessary pre-shipment steps are accomplished.

5.9.5 The samples shall be packaged and shipped to the laboratory by overnight carrier in order to demonstrate chain of custody.

Note: The "Request for Analysis" form and the chain of custody form shall accompany the shipment.

5.9.6 The radioanalytical laboratory shall have written procedures that document the laboratory's analytical capabilities for the request analysis and a QA/QC program which assures the validity of the analytical results.

6 EXEMPTION PROVISIONS

Variances and exceptions to the requirements of this RSP shall be permitted pursuant to the written authorization of the RSO and the President.

7 DOCUMENTATION

7.1 All records pertinent to this procedure shall be maintained pursuant to RSP-004.

7.2 The following records shall be maintained:

7.2.1 Radiological Survey Forms

7.2.2 Smear Counting Results

7.2.3 Chain-of-Custody Forms

7.2.4 Project logs and work plans

7.2.5 Field notes and records.

8 ATTACHMENTS

8.1 Attachment 1 - Radiological Survey Form

8.2 Attachment 2 - Analysis Request and Chain-of-Custody Form

ATTACHMENT 1

INTEGRATED ENVIRONMENTAL MANAGEMENT, INC.

RADIOLOGICAL SURVEY FORM

(Full-page version on the server)

Survey Number_____ Page _____ of _____

Instrument/SN:	Calibration Due:	Site Name:	Date:	Time:
Instrument/SN	Calibration Due:	Location:		
Instrument/SN	Calibration Due:	Purpose:		
Survey Performed By (Signature):		Survey Checked By (Signature):		
☐ Battery OK ☐ HV OK ☐ Source Check OK		Grid Dimensions: _____ x _____ ☐ meters ☐ inches ☐ feet ☐ centimeters		

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	W	X	Y	Z
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Notes:

ATTACHMENT 2

INTEGRATED ENVIRONMENTAL MANAGEMENT, INC.

ANALYSIS REQUEST AND CHAIN-OF-CUSTODY RECORD

Reference No. _____

(Full-page version on the server)

Page 1 of _____

(1) Integrated Environmental Management, Inc.	(7) Samples Shipment Date	(5) Bill to:
(2) Sample Team Leader	(8) Lab Destination	
(3) Task No.	(9) Lab Contact	
(4) Project Manager	(12) Technical Contact/Phone	(10) Report to:
(6) Purchase Order No.	(13) Carrier/Waybill No.	
(11) Required Report Date		

ONE SAMPLE PER LINE

(14) Sample Number	(15) Sample Description/Type	(16) Date/Time Collected	(17) Container Type	(18) Sample Volume	(19) Preservative	(20) Requested Testing Program

(23) Special Instructions

(24) Possible Hazard Identification Non-hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown ☐	(25) Sample Disposal Return to Client ☐ Disposal by Lab ☐ Archive _____ months
(26 Relinquished by: (signature, date, time):	Received by: (signature, date, time)
Relinquished by: (signature, date, time):	Received by: (signature, date, time)
Relinquished by: (signature, date, time):	Received by: (signature, date, time)

ATTACHMENT 2 (cont.)
INSTRUCTIONS FOR COMPLETING THIS FORM

1. Integrated Environmental Management, Inc.
2. **Sample Team Leader**: List the name of the team taking these samples.
3. **Task No.**: Indicate the **IEM** task number, if applicable.
4. **Project Manager**: Record the project manager's name.
6. **Purchase Order No.**: Non-**IEM** personnel should use this space to record the purchase order number authorizing the analysis of these samples. **IEM** and **IEM** subcontractors should leave this space blank if a project number has been given for billing.
7. **Samples Shipment Date**: Indicate the date these samples are shipped to the laboratory.
8. **Lab Destination**: Indicate the laboratory designated for sample shipment. Do not list more than one lab on this form. Be certain before sending samples that the laboratory you are designating is aware of the shipment and is capable of accepting these sample types and has available capacity.
9. **Lab Contact**: Give the name of the laboratory contact (typically the lab's project manager).
10. **Report to**: Give the name, address and phone number of the person to receive the data report for these samples.
11. **Required Report Date**: Record the date which you and the laboratory contact have determined the results will be reported (include verbal or final report as appropriate).
12. **Technical Contact/Phone**: Indicate the name of the person to be contacted in case of any questions regarding these samples and the phone number where the contact may be reached the day the samples arrive in the laboratory.
13. **Carrier/Waybill Number**: If you are sending the samples by a commercial carrier such as Airborne or Federal Express, record the courier company name and the waybill or airbill number under which these samples will be shipped (Example - Fed-Ex/#513631771).
14. **Sample Number**: List the complete, unique identification number of each sample. These numbers must correspond with the identification numbers on the sample containers and the field sample collection document(s).
15. **Sample Description/Type**: Provide a short physical description of the sample and the sample type such as soil, sediment, sludge, water, wipe, air, concentrated waste or bulk.
16. **Date/Time Collected**: Record date and exact time each sample was collected. Use a 24-hour clock; i.e., 1645 not 4:45 p.m.
17. **Container Type**: Indicate the volume, color and type of the sample container used (Example - 1 gallon amber glass, 1 liter clear plastic, 40 milliliter clear glass).
18. **Sample Volume**: Estimate the amount of sample in the container. For air samples, indicate the volume of air sampled.
19. **Preservative**: Indicate what type of preservative, if any, has been used for the samples (Example - ice to 4^{o}C nitric acid, hydrochloric acid).
20. **Requested Testing Program**: List the analyses to be performed on each sample by method number or quotation number.
23. **Special Instructions**: Use this space to record any special instructions to the lab regarding the processing of these samples.
24. **Possible Hazard Identification**: Indicate all hazard classes associated with the sample(s).
25. **Sample Disposal**: Indicate how the samples should be disposed of following analysis. The lab may charge for packing, additional archiving and disposal.

26 **Signatures**: When releasing custody of these samples, use the "Relinquished By" space to sign your full legal name, date and time of release. After verifying that all samples are present, the person receiving the samples must sign the "Received By" space to take custody of the samples.

Integrated Environmental Management, Inc.

SAMPLE COLLECTION	Procedure: RSP-026	Revision No.: 001
	Page: 1 of 16	Date: September 28, 2006
	Approved by (RSO):	
	Approved by (President):	

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1 PURPOSE

This Radiation Safety Procedure (RSP) provides general guidance in the collection and analysis of environmental media for the presence of radioactivity. Detailed step-by-step instructions for collecting individual sample types is incorporated in project-specific work plans rather than in this RSP.

2 SCOPE

This RSP applies to the collection of samples by Integrated Environmental Management, Inc. (IEM) employees on behalf of its clients. Samples collected for other purposes are exempt from the requirements of this RSP.

3 REFERENCES

3.1 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-001, "Radiation Protection Program Plan".

3.2 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-002, “Definitions”.

3.3 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-009, “Contamination Control”.

3.4 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-011, "Radiological Areas and Posting".

3.5 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-021, “Final Status Surveys”.

3.4 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-015, “Packaging and Transportation of Radioactive Material”.

3.6 Integrated Environmental Management, Inc. Radiation Safety Procedure No. RSP-018, “Surveillance”.

3.7 MARSSIM - U. S. Nuclear Regulatory Commission, NUREG-1575, “Multi-Agency Radiation Survey and Site Investigation Manual - MARSSIM”.

3.8 MARLAP - U. S. Nuclear Regulatory Commission, NUREG-1576, “Multi-Agency Radiological Laboratory Analytical Protocols Manual”.

3.9 USEPA Manual - U.S. Environmental Protection Agency, Region IV, “Standard Operating Procedures and Quality Assurance Manual”, Appendix B.

3.10 USEPA, Characterization of Hazardous Waste sites - A Methods Manual, Volume 2, EPA 600/4-84/076.

3.11 USEPA, Soil Sampling Quality Assurance User's Guide, Second Edition, EPA 600/8-89/046.

3.12 USEPA, Preparation of Soil Sampling Protocols: Sample Techniques and Strategies, EPA 600/R-92/128.

4 DEFINITIONS

The definition of terms used in this RSP that may not be commonly understood shall be found in RSP-002.

5 PROCEDURE

5.1 Responsibilities

5.1.1 The President shall supply adequate resources to ensure implementation of this procedure.

5.1.2 The Radiation Safety Officer (RSO) shall:

5.1.2.1 Incorporate radiation protection procedures, as necessary, in planning the sample collection program.

5.1.2.2 Assure that Health Physics Technicians are trained in the operational and radiation safety aspects of this RSP.

5.1.3 Health Physics Technicians shall:

5.1.3.1 Have a clear understanding of the methodologies implemented in each project-specific Work Plan (WP), and the required sample collection techniques for executing the WP.

5.1.3.2 Have adequate training in any applied sample collection techniques required by a WP.

5.1.3.3 Adhere to the provisions of this RSP unless directed otherwise in a WP.

5.2 Radiological Monitoring Considerations

5.2.1 Radiological monitoring shall be considered:

5.2.1.1 During any sample collection activity or operation associated with sample collection known or suspected to impact on the health and safety of personnel.

5.2.1.2 During any sample collection activity which would require the transportation of radioactive materials under the requirements of the United States Department of Transportation (USDOT).

5.2.1.3 When shipment of radioactive samples could impact on the radiological materials licensing status of the analytical laboratory.

5.2.1.4 On a project-specific basis and shall include a determination of posting requirements.

5.2.1.5 Exposure rate and contamination controls should be implemented as necessary (see RSP-018 and RSP-009).

Note: Special postings are typically discussed in Tailgate Safety Meetings.

5.2.2 Posting for sample media/sampling locations, when necessary, should be in accordance with RSP-011.

5.3 General Considerations in Designing a Soil Sampling Program

5.3.1 All sampling strategies shall be predetermined prior to the start of sampling activities.

5.3.2 These strategies should include, but are not limited to:

5.3.2.1 Types of samples

5.3.2.2 Number of samples

Note: The number and types of samples should be as defined in the applicable sampling or decommissioning plan, or as approved by the RSO.

5.3.2.3 Sampling locations

5.3.2.4 Sampling frequency

5.3.2.5 Sampling depth and area of collection

5.3.2.6 Sampling methods (e.g., surface, subsurface)

5.3.2.7 Sample preparation

5.3.2.8 Analytical protocols

5.3.2.9 Quality Assurance and Quality Control requirements

5.3.2.10 Data quality objectives

5.3.3 The number and location of samples collected shall be representative of the site conditions so that a reasonably sound assessment of contamination levels can be made.

Note: The number of samples collected should be kept to a practical minimum.

5.3.4 If sampling is being performed as part of a final status survey, the number of samples shall satisfy the requirements of MARSSIM as described in RSP-021.

5.3.5 The number of samples necessary to assess contaminant concentrations and distributions within a given area should take into account:

5.3.5.1 The size of the area;

5.3.5.2 Radionuclide variability;

5.3.5.3 Action levels or release criteria for individual radionuclides;

5.3.5.4 The detection capability of the preferred analytical method;

5.3.5.5 Whether sufficiently-sensitive survey methods are used to support the site assessment;

5.3.5.6 The criteria for hot spot acceptance; and

5.3.5.7 Physical and other individual site characteristics and considerations.

5.3.6 To reduce analytical costs, sample compositing should be considered within specific areas in order to smooth out minor spatial variations of contaminants.

Note: The ability to assess the spatial variability of the contaminants and to detect hot spots within the area being evaluated is lost when composite sampling is performed. When making a decision to composite samples,

the importance of these factors to the overall objectives of the sampling program should be considered.

5.3.7 Sampling should employ a systematic grid system utilizing site boundaries and/or permanent markers, or other means as appropriate.

5.3.8 Samples should be collected at pre-determined locations.

5.3.9 Each sample collected should be analyzed individually unless compositing is performed.

5.3.10 The sample container should be clearly labeled as described in section 5.8, below.

5.3.11 Appropriate information should be recorded and the chain-of-custody form and field sample collection logs (see Attachment 8.1) completed.

5.3.12 Samples should be prepared for shipment as described in Section 5.9, below.

5.3.13 Sampling equipment should be cleaned and decontaminated prior to reuse or storage.

5.4 Water Sampling Considerations

5.4.1 Water sampling may be performed in order estimate exposures to the public, demonstrate compliance with appropriate regulations and guidance, and to determine whether migration of radioactive material is occurring.

Note: Water sampling is a complex topic, especially for groundwater sampling. Specific procedures must be utilized which will be dependent upon the objectives of the sampling program.

5.4.2 Prior to sample collection, sampling personnel should document the type of sample (e.g., surface, groundwater, potable, etc.). and the sampling approach employed (e.g., grab sampling, continuous sampling, etc.).

5.4.3 When collecting surface water samples, sampling should proceed from downstream locations to upstream locations so that any disturbance associated with sample collection does not affect the quality of subsequent samples.

5.4.4 If sediment samples are collected at the same locations as water samples, the water samples shall be collected first.

5.4.5 The collection container (e.g., plastic, glass) type shall be determined based on the radionuclide and other considerations as advised by the analytical laboratory.

Note: For radiological analyses, plastic containers are generally preferred. Glass containers are preferred for H-3 and Carbon-14 analysis.

5.4.6 Containers should be of a size appropriate to the volume required by the laboratory for the performance of the specified analytical method.

5.4.7 Samples collected from surface waters (rivers, streams, or creeks) should be well-mixed and as homogeneous as possible prior to placement into the collection container.

5.4.8 The collection container should be clearly labeled as described in section 5.8, below.

5.4.9 Collection information should be recorded on the chain-of-custody form and field sample collection logs.

5.4.10 Samples should be prepared for shipment as described in Section 5.9, below.

5.4.11 Collection equipment should be cleaned and decontaminated prior to reuse or storage.

5.4.12 If well sampling is performed, the following should be considered in developing and documenting the approach and documenting the sample collection:

5.4.12.1 Depth of the well

5.4.12.2 Depth and volume of water

5.4.12.3 Amount of water (number of well volumes) to purge

5.4.12.4 Acceptable recovery of well prior to sampling

5.4.12.5 Method of removing water for sampling (e.g. bailer, pump, etc)

5.5 Sample Preparation

The method for preparing samples for analysis in advance of shipment shall be determined in concert with the analytical laboratory and shall be based on the size of individual samples, the total number of samples being handled, physical characteristics of the sample (e.g., soil, water), etc.

5.6 Analytical Requirements

5.6.1 Radiological and chemical analytical requirements shall be clearly defined, evaluated for applicability to project needs, and based upon the required level of quality for those data prior to the start of the sampling effort.

5.6.2 Analytical requirements shall take into account site history, current site operations, and surrounding operations related to or near the site location.

5.6.3 The required level of quality control (QC) may be modified (more or less stringent) based upon project-specific requirements.

5.6.4 Analytical results shall be reported in appropriate and applicable units (e.g., pCi/g, pCi/l, etc.)

5.6.5 Specifications for performance of the analyses to be sent to the analytical laboratory shall include, but are not limited to the following:

5.6.5.1 The proposed date of sample shipment.

5.6.5.2 The analytical method to be used.

5.6.5.3 The minimum detection limits.

5.6.5.4 The proposed size or volume of each sample.

5.6.5.5 Method-specific analytical considerations.

Note: For example, for samples analyzed by gamma spectroscopy, the following considerations should be included in the specifications: "The concentration of all positively-identified radionuclide(s) shall be reported in units of picocuries per gram. Actual values, along with the error and the Minimum Detectable Activity (MDA), shall be reported. Do not report <MDA, < [value], not detected, or any other censored value.

5.6.5.6 Method-specific sample preparation provisions.

Note: For example, if the measurement method requires samples to be dried and ground prior to chemical extraction or counting, the specifications should include a requirement for "percent moisture" determination in advance of sample preparation.

5.6.5.7 Draft report delivery date (turn-around time).

5.6.5.8 Final report delivery date.

5.6.5.9 Disposition of residual sample material.

Note: In some cases, the cost of sample disposal is not included in the analytical cost. If the disposal cost is high, consideration should be given to returning the sample to the collection site.

5.6.6 The draft analytical report should be reviewed for conformance with specifications prior to authorizing the laboratory to issue the final report.

Note: The laboratory should be cautioned to make *no* changes to the approved draft other than to remove draft markings.

5.7 Sample Documentation and Custody Requirements

5.7.1 Chain-of-custody (COC) procedures shall be followed in order to provide documentation of the handling of each sample from the time it is collected until it is destroyed.

5.7.2 The COC record (Attachment 8.2) shall serve as a legal record of possession of the sample and shall be initiated with the acquisition of the sample.

5.7.3 The COC record shall remain with the sample at all times and bear the name of the person assuming responsibility for the samples.

5.7.4 To simplify the COC record and eliminate potential legal challenges, as few people as possible should handle the sample between collection and disposal.

5.7.5 A sample is considered to be under custody if one or more of the following criteria are met:

5.7.5.1 The sample is in the sampler's possession;

5.7.5.2 The sample is in the sampler's view after taking possession;

Note: The COC from the analytical laboratory may be used in lieu of Attachment 8.2 as approved by the RSO.

5.7.5.3 The sample was in the sampler's possession and was then locked up to prevent tampering; or

5.7.5.4 The sample is in a designated secure area.

5.7.6 In addition to the COC record, the sample may also contain a COC seal.

Note: The COC seal is an adhesive seal placed in areas such that if a sealed container is opened, the seal is broken. The COC seal ensures that no sample tampering occurs between the field and laboratory analysis.

5.7.7 A Sample Collection Log (see Attachment 8.1) should be completed for each sample/group of samples.

5.8 Sample Labeling and Numbering

5.8.1 Sample labels shall be required for properly identifying samples.

5.8.2 All samples shall have the label affixed to the sample container prior to its transport to the laboratory.

5.8.3 Information on sample labels should include, but is not limited to, the following:

5.8.3.1 IEM Project Code - an assigned IEM client/task number and site name.

5.8.3.2 Location/Station Number - a unique identifier assigned to a sampling point by the sampling team.

5.8.3.3 Unique Sample Identification Number - a unique number assigned to each sample, including field control samples.

5.8.3.4 Sampling personnel - name of the person collecting the sample.

5.8.3.5 Preservative - whether a preservative is used and the type of preservative.

5.8.3.6 Analysis - the type of analysis requested.

5.8.3.7 Date/Time - the date and time the sample was taken.

5.8.3.8 Type of Sample - the type of sample (e.g., grab or composite).

5.8.4 A sample numbering system shall be used to identify each sample collected and submitted for analysis.

Note: It is acceptable to write the sample number directly onto the sample container or lid with a permanent marker.

5.8.4.1 The sample identification numbers for each sampling effort should be used on sample labels, sample tracking matrix forms, chain-of-custody forms, field logs, and all other applicable documentation.

5.8.4.2 A listing of all sample identification numbers should be recorded in the field logs and placed into project files.

5.8.4.3 Sample numbers should change when the media or location changes.

5.8.4.4 Sample numbers should not change because different analyses are requested.

Note: For example, water samples collected at the same location, date, and time for several different analyses would be considered a sample set and all have the same sample number, although the various sample aliquots would be collected in different containers.

5.9 Packaging and Shipping

Note: This section shall apply to environmental samples, and radioactive waste and radiological samples.

5.9.1 Packages shall be shipped in accordance with RSP-015.

Note: Environmental samples are defined as those samples collected from environmental matrices such as soil, groundwater, or sediments.

5.9.2 The following considerations in sample labeling, packaging, and shipping should be taken into account:

5.9.2.1 Identifying the sample container with a sample label (see section 5.8, above).

5.9.2.2 Ensuring all sample containers are securely sealed to prevent leakage during transport.

5.9.2.3 Using a cooler or other packaging device as a shipping container.

5.9.2.4 Positioning the sample containers upright in the shipping container with the objective of avoiding sample-to-sample contact during shipment.

Note: An absorbent in the cooler may be required.

5.9.2.5 Placing a copy of the COC form (including designation of the overnight carrier and air bill number) going to the analytical laboratory inside a plastic bag (or equivalent) of the shipping container.

5.9.2.6 Closing and taping the shipping container with strapping tape (or equivalent).

5.9.2.7 Transferring the shipping container to the overnight carrier.

5.9.2.8 Notifying the laboratory that the samples have been sent and their anticipated arrival time.

5.10 Equipment Decontamination

5.10.1 Field sampling equipment shall be decontaminated in order to prevent cross-contamination of project samples.

5.10.2 All sampling equipment that has come in contact with a potentially contaminated media shall be cleaned prior to the subsequent use of that device.

Note: Another approach to minimizing the potential for cross-contamination may be to dedicate or use disposable sample equipment.

5.10.3 In advance of the sampling campaign, the media sampled and the type of sampling equipment selected should be evaluated to determine an acceptable decontamination strategy.

5.10.4 All sampling equipment that has been used for radiological materials or radioactive waste sampling should be surveyed by a Health Physics Technician prior to re-use.

Note: Example sampling equipment decontamination procedures can be found in the USEPA Manual.

6 EXEMPTION PROVISIONS

Variances and exceptions to the requirements of this RSP shall be permitted pursuant to the written authorization of the RSO.

Note: Specifications within the work plan that deviate from this RSP are covered by this exemption provision.

7 DOCUMENTATION

All records pertinent to this procedure shall be maintained pursuant to RSP-004.

8 ATTACHMENTS

8.1 Sample Collection Log

8.2 Analysis Request and Chain-Of-Custody Record

ATTACHMENT 8.1

INTEGRATED ENVIRONMENTAL MANAGEMENT, INC.

SAMPLE COLLECTION LOG

(Full page version on the server)

Project Number_____

Sample Number:	Site Name:	Date:	Time:	Page :
Preservative:		Grid Dimensions: _____ x _____ Units (circle): meters inches feet centimeters		

Sample No.	Sampled by (initials)	Date/Time	Sample Description (media, depth)	Sample Type (grab, composite)	Container Type	Sample Location

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	W	X	Y	Z
1																										
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ATTACHMENT 8.2

INTEGRATED ENVIRONMENTAL MANAGEMENT, INC.
ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD
(Full page version on the server)

Reference No. _____
Page 1 of _____

(1) Client Name: Integrated Environmental Management, Inc.	(7) Samples Shipment Date	(5) Bill to:
(2) Sample Team Leader	(8) Lab Destination	
(3) Task No.	(9) Lab Contact	
(4) Project Manager	(12) Technical Contact/Phone	(10) Report to:
(6) Purchase Order No.	(13) Carrier/Waybill No.	
(11) Required Report Date		

ONE CONTAINER PER LINE

(14) Sample Number	(15) Sample Description/Type	(16) Date/Time Collected	(17) Container Type	(18) Sample Volume	(19) Preservative	(20) Requested Testing Program

(23) Special Instructions	
(24) Possible Hazard Identification (circle): Non-hazard Flammable Skin Irritant Poison B Unknown	(25) Sample Disposal (circle): Return to Client Disposal by Lab Archive _____ months
(26) Turnaround Time Required (circle): Normal Rush	(27) QC Level (circle): I II III Project Specific _____
(28) Relinquished by: (signature, date, time):	Received by: (signature, date, time)
Relinquished by: (signature, date, time):	Received by: (signature, date, time)
Relinquished by: (signature, date, time):	Received by: (signature, date, time)

(See Reverse for Instructions)

INSTRUCTIONS FOR COMPLETING THIS FORM

1. **Client Name**: Record the name of the client/site location.

2. **Sample Team Leader**: List the name of the team taking these samples.

3. **Task No.**: Indicate the **IEM** task number, if applicable.

4. **Project Manager**: Record the project manager's name.

6. **Purchase Order No.**: Non-**IEM** personnel should use this space to record the purchase order number authorizing the analysis of these samples. **IEM** and **IEM** subcontractors should leave this space blank if a project number has been given for billing.

7. **Samples Shipment Date**: Indicate the date these samples are shipped to the laboratory.

8. **Lab Destination**: Indicate the laboratory designated for sample shipment. Do not list more than one lab on this form. Be certain before sending samples that the laboratory you are designating is aware of the shipment and is capable of accepting these sample types and has available capacity.

9. **Lab Contact**: Give the name of the laboratory contact (typically the lab's project manager).

10. **Report to**: Give the name, address and phone number of the person to receive the data report for these samples.

11. **Required Report Date**: Record the date which you and the laboratory contact have determined the results will be reported (include verbal or final report as appropriate).

12. **Technical Contact/Phone**: Indicate the name of the person to be contacted in case of any questions regarding these samples and the phone number where the contact may be reached the day the samples arrive in the laboratory.

13. **Carrier/Waybill Number**: If you are sending the samples by a commercial carrier such as Airborne or Federal Express, record the courier company name and the waybill or air bill number under which these samples will be shipped (Example - Fed-Ex/#513631771).

14. **Sample Number**: List the complete, unique identification number of each sample. These numbers must correspond with the identification numbers on the sample containers and the field sample collection document(s).

15. **Sample Description/Type**: Provide a short physical description of the sample and the sample type such as soil, sediment, sludge, water, wipe, air, concentrated waste or bulk.

16. **Date/Time Collected**: Record date and exact time each sample was collected. Use a 24-hour clock; i.e., 1645 not 4:45 p.m.

17. **Container Type**: Indicate the volume, color and type of the sample container used (Example - 1 gallon amber glass, 1 liter clear plastic, 40 milliliter clear glass).

18. **Sample Volume**: Estimate the amount of sample in the container. For air samples, indicate the volume of air sampled.

19. **Preservative**: Indicate what type of preservative, if any, has been used for the samples (Example - ice to 4^{o}C nitric acid, hydrochloric acid).

20. **Requested Testing Program**: List the analyses to be performed on each sample by method number or quotation number.

23. **Special Instructions**: Use this space to record any special instructions to the lab regarding the processing of these samples.

24. **Possible Hazard Identification**: Indicate all hazard classes associated with the sample(s).

25. **Sample Disposal**: Indicate how the samples should be disposed of following analysis. The lab may charge for packing, additional archiving and disposal.

26. **Turnaround Time Required**: Check "Normal" or "Rush" as determined by the Technical Contact and the Lab Contact. Rush samples are subject to a surcharge.

27. **QC Level**: These should be specific to the analytical laboratory and should not be confused with USEPA Analytical Levels. Project Specific should reference a quotation number or other specifications that have been submitted to the laboratory before beginning work.

28 **Signatures**: When releasing custody of these samples, use the "Relinquished By" space to sign your full legal name, date and time of release. After verifying that all samples are present, the person receiving the samples must sign the "Received By" space to take custody of the samples.

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1 PURPOSE

In order to implement field projects pursuant to a client contract, Integrated Environmental Management, Inc. (IEM) must plan and implement the plans, then produce deliverables on time and within budget. The purpose of this Standard Operating Procedure (SOP) is to establish the approach for managing field projects such that cost and schedule demands can be met.

2 SCOPE

This SOP applies to the performance of all IEM field projects with a duration of greater than two days and with more than four IEM personnel (employees or subcontractors) on-site. Projects of lesser duration and staffing are exempt from the provisions of this SOP.

3 REFERENCES

3.1 Integrated Environmental Management, Inc., Radiation Safety Procedure No. RSP-006, “Training and Qualifications of Radiation Protection Personnel”.

3.2 Integrated Environmental Management, Inc., Radiation Safety Procedure No. RSP-018, “Surveillance”.

3.3 Integrated Environmental Management, Inc., Radiation Safety Procedure No. RSP-020, “Daily Tailgate Safety Training”.

3.4 Integrated Environmental Management, Inc., Standard Operating Procedure No. SOP-002, “Client-Related Records”.

3.5 Integrated Environmental Management, Inc., Standard Operating Procedure No. SOP-009, “Subcontracting and Purchases”.

3.6 Integrated Environmental Management, Inc., Standard Operating Procedure No. SOP-011, “Project Management”.

3.7 Integrated Environmental Management, Inc., Quality Policy Statement No. QPS-004, “Control of Nonconforming Items, Activities and Products”.

4 DEFINITIONS

4.1 Company Officer - Either the Secretary or the President.

4.2 Contracts Manager - An IEM employee who is responsible for preparing, monitoring and securing payment of purchase orders pursuant to SOP-009.

4.3 FADL - Field Activity Daily Log (see Attachment 8.1).

4.4 Field Project - A series of activities involving radioactive materials, the conduct of surveys, radiological sampling, etc., performed at a client-designated site designed to achieve a specific objective.

4.5 Field Team Member - IEM employee or subcontractor who has been given actions to carry out in the project plan(s).

4.6 May - The word *may* is used to denote permission.

4.7 President - Senior official of the corporation.

4.8 Project - A series of activities designed to achieve a specific objective within a set budget and time frame.

4.9 Project Manager - IEM employee responsible for achieving the field project's overall objectives and leading the field team. It is possible for the same employee to serve as Project Manager and Program Manager for a particular project.

4.10 Program Manager - IEM employee that serves as the primary client contact, initiates field projects and serves as the most senior project team member. The same employee may serve as both Program Manager and Project Manager for any given project.

4.11 Shall - The word *shall* is to be understood as a requirement.

4.12 Should - The word *should* is to be understood as a recommendation.

4.13 Subcontractor - Provider of materials, products or services under contract to IEM that are needed to carry out the project.

5 PROCEDURE

5.1 Responsibilities

5.1.1 The President shall:

5.1.1.1 Enter into a contract with the client to perform the field project.

5.1.1.2 Provide the resources necessary to implement this SOP.

5.1.1.3. Assign the program manager, as applicable, and the project manager.

5.1.1.4 Confirm project plans are designed to maximize the safety of all participants, including employees, contractors and site visitors.

5.1.1.5 Hold the program manager and project manager accountable for the safety of participants for the duration of the project.

5.1.2 The Program Manager shall:

5.1.2.1 Secure client- and regulator-approved plans, as applicable.

5.1.2.2 Assist the Project Manager in preparations for the field project.

5.1.2.3 Secure updates on the field project status, health safety issues and other project information weekly or bi-weekly.

5.1.2.4 Communicate field project information to the President as needed.

5.1.2.5 Update the client contact on the field project status, health and safety, and other project information weekly, bi-weekly or as required by contract provisions.

5.1.2.6 Forward purchase requisitions (with specifications) for all purchased or subcontracted items to the Contracts Manager pursuant to SOP-009.

5.1.2.7 Approve all project plans to ensure they will result in all objectives being met and the safety of all participants being maximized.

5.1.3 The Project Manager shall:

5.1.3.1 Serve as the main point of contact for the field project.

5.1.3.2 Select a Project HP to manage data acquisition pursuant to Section 5.3, below.

5.1.3.3 Execute the field project pursuant to the plans (see Section 5.1.2.1, above).

5.1.3.4 Ensure the safety of all project personnel, contractors and visitors for the duration of the on-site effort.

5.1.3.5 Confirm daily safety training is provided pursuant to RSP-020 to all project personnel.

5.1.3.6 Apply for reciprocal recognition of License No. MD-31-281-01, if required, at least five (5) days before the first on-site date.

Note: The RSO or ARSO will do the filing with the applicable regulatory agency based upon the site address, contact information, telephone number, etc., provided by the Project Manager.

5.1.3.7 Manage all field personnel, including IEM employees and subcontractors.

5.1.3.8 Ensure all IEM equipment, material and data at the job site remain in the control of an IEM employee at all times.

Note: For example, subcontractors who are given an IEM computer for data acquisition or other purposes shall not be permitted to remove that computer from the job site or an IEM office. Likewise, subcontractors who use their own computers for data acquisition or other project-related purposes shall turn over all data and information before leaving the job site for the day. The Project Manager should make mass storage devices (i.e., CDs, thumb drives) available for this purpose.

5.1.3.9 Communicate field project information to the Program Manager, team members, subcontractors and other interested parties, as necessary.

5.1.3.10 Ensure all data and information acquired at the job site are secured pursuant to Section 5.3 and 5.5, below.

5.1.3.11 Capture all deviations in the project plans pursuant to Section 5.4.1, below.

5.1.3.12 Update the Program Manager on the status of the field project at the required frequency.

Note: E-mail communications on field project status should be retained in the electronic project file.

5.1.3.13 Perform all other project management tasks required in SOP-011.

‘ 5.1.3.14 Complete a FADL once each day while on site.

5.1.3.15 Maintain all records and reports associated with the field project pursuant to SOP-002.

5.2 Project Planning and Preparation

5.2.1 The following project plans should be prepared and approved by the client, IEM and other stakeholders (see SOP-011, Section 5.2), as applicable:

Note: The following plans may be stand-alone documents or compiled into a single document or a planning package, depending upon contract requirements.

5.2.1.1 Work plan or sampling/analysis plan.

5.2.1.2 Health and safety plan.

5.2.1.3 Waste management plan.

Note: If no waste handling or disposition is associated with the field project, the work plan should clearly state, "No client-generated or project-derived waste will be generated for this project" (or equivalent language).

5.2.1.4 Quality Assurance plan

5.2.1.5 Scoping, Characterization, Remediation or Final Status Survey plans (as applicable).

Note: If not performed during the field project, the work plan should clearly state, "No radiation surveys for scoping, characterization, remediation or final status will be performed for this project"(or equivalent language).

5.2.1.6 Other project related documents as described in the Scope of Work or IEM proposal.

5.2.2 The Project Manager shall:

5.2.2.1 Carefully review the Project Plans prior to issuing subcontracts, selecting field team personnel and preparing for the field project.

5.2.2.2 Maintain a full and complete copy of the Project Plans on-site for the duration of the field project.

5.2.2.3 Confirm with the RSO that applicable licensing/permitting is in place and maintain a full and complete copy of IEM License No. MD-31-281-01, RSP-001 and, if so required, the applicable reciprocity license on-site for the duration of the project.

5.2.2.4 Confirm with the Program Manager or President that Project Plans address the steps necessary to meet safety objectives.

5.2.2.5 Determine the type/amount of forms, materials, equipment and personnel needed to implement the project plans and secure same.

5.2.3 Radiation surveyors, sample collection personnel and others responsible for data acquisition shall:

5.2.3.1 Be trained in IEM's data acquisition and reporting procedures and the project plans pursuant to RSP-006.

5.2.3.2 Terminate data acquisition activities, as necessary, at least one hour before the close of each work day in order to transcribe field notes.

5.2.3.3 Turn over all data and related documentation to the Project Manager (or designee) at the end of each work day.

Note: These data may include field notes, transcribed survey records, collection logs, survey data on electronic storage media, etc.

5.2.4 Purchase requisitions shall be issued by the Project Manager for all subcontracted services pursuant to SOP-009.

5.2.5 Spreadsheets for capturing radiation survey data shall be prepared by the radiation surveyors and reviewed by the Project Manager on a planned and periodic basis.

NOTE: All records, hard copies and electronic copies shall be copied (backed up) on the IEM server as described in Section 5.5 of this SOP.

5.2.6 A means of ensuring telephone and internet communications at the field site should be instituted.

Note: Cellular telephones, wireless cards for computers and 3G-type telephones may be used to ensure adequate and necessary communications.

5.3 Radiation Survey Data Management

5.3.1 Each radiation survey to be performed shall be entered into a Radiation Survey Log (see Attachment 8.2) by the Project Manager (or designee), who will then assign the performance of each survey to a radiation surveyor.

5.3.2 The radiation surveyor shall record all initial radiation survey data (field notes) onto IEM survey forms (see RSP-018), IEM graph paper, in an approved log book or on a form approved in advance by the Project Manager.

Note: Data captured in a data logger are exempt from this requirement.

5.3.3 On a daily basis:

5.3.3.1 All field survey notes for scoping, characterization or final status survey purposes shall be transcribed onto IEM survey forms (see RSP-018) or forms specified in the project plans.

Note: Field survey notes for other purposes (i.e., equipment/material release surveys, waste shipments, remediation surveys) are exempt from this requirement.

5.3.3.2 All field notes and transcribed survey forms shall be turned over to the Project Manager (or designee).

5.3.3.3 The Project Manager shall:

5.3.3.3.1 Review/approve the survey forms;

5.3.3.3.2 Forward all field notes and survey forms to a designated data handler for capture in spreadsheets.

Note: If a data handler is not an assigned position on the field team, the field notes and survey forms should be forwarded to the IEM Maryland office for capture.

5.3.4 The updated spreadsheets shall be forwarded to the Project Manager.

5.3.5 The Project Manager (or designee) shall review the updated survey sheets for compliance with project plan requirements on the following, as applicable:

5.3.5.1 Measurement type;

5.3.5.2 Scan speed;

5.3.5.3 Detection limits;

Note: The minimum detectable activity (MDA) of the measurement method was used to determine the number of measurements needed. If the MDA is higher than assumed in the project plan, the number of measurements to be acquired during the project may change.

5.3.5.4 Measurement background;

5.3.5.5 Data set standard deviation;

Note: The data set standard deviation, σ, is used as input to the σ/Δ values used to determine the number of measurements needed. If the σ assumption used in the work plan is found to be in error, the number of measurements to be acquired during the project may change.

5.3.5.6 Consistency with assumed radiological conditions in the survey unit of interest; and

5.3.5.7 Other data parameters specified in the project plans.

5.3.6 The originals of the field notes and survey forms shall be retained as specified in SOP-002.

5.4 Deviations and Exceptions from Project Plans

5.4.1 All deviations from requirements specified in the project plans shall be noted in the field copy of the plan.

5.4.2 All changes in the scope of the project shall be recorded on the applicable FADL.

5.4.3 Scope changes shall be addressed pursuant to Section 5.4 of SOP-002.

5.5 Information Preservation

5.5.1 All data and information generated during the field project shall be maintained pursuant to SOP-002.

5.5.2 All field records and survey forms shall remain in the custody of an IEM employee at the end of each work day.

5.5.3 The following shall be converted to electronic format (scanned) and placed onto the IEM server pursuant to SOP-002 and at the minimum frequency shown:

5.5.3.1 Field notes (weekly);

5.5.3.2 Completed and approved survey forms (immediately after data capture is complete);

5.5.3.3 Completed FADLs (weekly);

5.5.3.4 Contract Change Orders (weekly);

5.5.3.5 Photographs from the field site (weekly);

5.5.3.6 Radiation Survey Logs (project completion);

5.5.3.7 Instrument daily check forms (weekly);

5.5.3.8 Instrument calibration records (project completion);

5.5.3.9 Electronic or hard copies of related correspondence (weekly).

6 EXEMPTION PROVISIONS

Variances and exceptions to the requirements of this SOP shall be permitted pursuant to the authorization of the President or the Secretary.

7 DOCUMENTATION

7.1 The Project Manager shall ensure the project file contains a copy of all records generated by all field team members.

7.2 The Project Manager shall ensure all project records are converted to electronic format and backed up on the IEM server pursuant to SOP-002.

8 ATTACHMENTS

8.1 Field Activity Daily Log

8.2 Radiation Survey Log

ATTACHMENT 8.1

FIELD ACTIVITY DAILY LOG

[*An electronic copy of this form can be found on the IEM server]*

Facility:	
Date:	Job/Task Number:
Client Name:	
Address of Work Site:	
Description of Work	
Arrived on site at (insert date and time):	Departed site at (insert date and time):

DESCRIPTION OF DAILY ACTIVITIES AND EVENTS

Unusual Occurrences (list):
Client or Regulator Activity Requests or Special Orders (list):

Changes in the Project Scope (list):	
Important Decisions:	
Important Telephone Calls and Interactions:	
Weather Conditions:	
Visitors on Site (list): ☐ Visitor log attached	
Attachments: ☐ H&S Report ☐ Tailgate Safety Training Form ☐ Additional pages ☐ Other (specify)	
Name (print):	Signature:
Distribution:	

ATTACHMENT 8.2

RADIATION SURVEY LOG

[*An electronic copy of this form can be found on the IEM server]*

Facility/Site:			IEM Job/Task No.		Responsible HP (Name):		Responsible HP (Signature):	
Survey No.	**Location (SU/OU)**	**Purpose**	**Assigned To:**	**Date Assigned:**	**Confirmations (check when complete)**			
					Completed	**Reviewed**	**Captured (Spreadsheet)**	**Captured (Server)**

Designation: D5438 – 05

Standard Practice for Collection of Floor Dust for Chemical Analysis[1]

This standard is issued under the fixed designation D5438; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a procedure for the collection of a sample of dust from carpets and bare floors that can be analyzed for lead, pesticides, or other chemical compounds and elements.

1.2 This practice is applicable to a variety of carpeted and bare floor surfaces. It has been tested for level loop and plush pile carpets and bare wood floors, specifically.

1.3 This practice is not intended for the collection and evaluation of dust for the presence of asbestos fibers.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D422 Test Method for Particle-Size Analysis of Soils

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

E1 Specification for ASTM Liquid-in-Glass Thermometers

E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

F608 Test Method for Evaluation of Carpet Embedded Dirt Removal Effectiveness of Household/Commercial Vacuum Cleaners

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D1356.

3.1.1 *carpet-embedded dust*—soil and other particulate matter, approximately 5-µm equivalent aerodynamic diameter and larger, embedded in carpet pile and normally removable by household vacuum cleaners.

3.1.2 *surface dust*—soil and other particulate matter, approximately 5-µm equivalent aerodynamic diameter and larger, adhering to floor surfaces and normally removable by household vacuum cleaners.

4. Summary of Practice

4.1 The sampling method described in this practice is taken from work published in Roberts, et al (**1-3**),[3] and Stamper, et al (**4**).

4.2 Particulate matter is withdrawn from the carpet or bare floor by means of vacuum-induced suction which draws through a sampling nozzle at a specific velocity and flow rate, and the particles are separated mechanically by a cyclone. The cyclone is designed to efficiently separate and collect particles approximately 5-µm mean aerodynamic diameter and larger. However, much smaller particles are also collected at unknown efficiencies. The sampling system allows for height, air flow, and suction adjustments to reproduce systematically a specific air velocity for the removal of particulate matter from carpeted and bare floor surfaces, so that these sampling conditions can be repeated.

NOTE 1—Side-by-side comparison of the HVS3 and a conventional upright vacuum cleaner revealed that both collected particles down to at least 0.2 µm and that the HVS3 was more efficient at collecting particles smaller than 20 µm than conventional vacuum cleaners (**5**). If desired, a fine-particle filter may be added downstream of the cyclone to collect 99.9 % of particles above 0.2 µm aerodynamic mean diameter.

4.3 The particulate matter in the air stream is collected in a catch bottle attached to the bottom of the collection cyclone. This catch bottle shall be capped for storage of the sample and transported to the laboratory for analysis.

5. Significance and Use

5.1 This practice may be used to collect dust from carpeted or bare floor surfaces for gravimetric or chemical analysis. The collected sample is substantially unmodified by the sampling procedure.

[1] This practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

Current edition approved March 1, 2005. Published March 2005. Originally approved in 1993. Last previous edition approved in 2000 as D5438 - 00. DOI: 10.1520/D5438-05.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

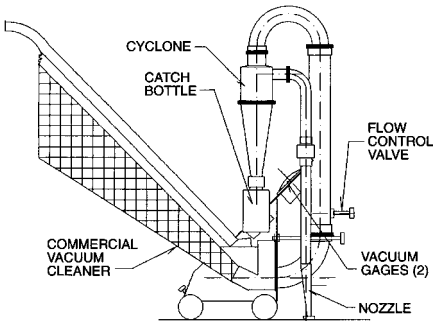


FIG. 1 Floor Dust Sampler Using a Commercial Vacuum Cleaner as the Suction Source

5.2 This practice provides for a reproducible dust removal rate from level loop and plush carpets, as well as bare floors. It has the ability to achieve relatively constant removal efficiency at different loadings of surface dust.

5.3 This practice also provides for the efficient capture of semivolatile organic chemicals associated with the dust. The test system can be fitted with special canisters downstream of the cyclone for the capture of specific semivolatile organic chemicals that may volatilize from the dust particles during collection.

5.4 This practice does not describe procedures for evaluation of the safety of floor surfaces or the potential human exposure to carpet dust. It is the user's responsibility to evaluate the data collected by this practice and make such determinations in the light of other available information.

6. Interferences

6.1 There are no known interferences to the determination of dust loadings covered by this practice.

7. Apparatus

7.1 *Sampling Apparatus*, which may be acquired commercially[4] (as shown in Fig. 1) or constructed as follows:

7.1.1 The dimensions of the sampling apparatus (nozzle size, cyclone diameter, cyclone inlet diameter, etc.) are interdependent. The flow rate must produce a sufficient velocity both at the sampled surface and in the cyclone. The cyclone must have a cut diameter of 5 µm at the same velocity that will provide a horizontal velocity of 40 cm/s at 10 mm from the nozzle in the carpet material, or 5 mm from the nozzle on bare floors. The fundamental principles of this device have been discussed in detail in Roberts, et al (**1-3**).

7.1.2 *Nozzle*—The edges and corners of the sampling nozzle shall be rounded to prevent catching the carpet material. The nozzle must be constructed to allow for sufficient suction to separate loose particles from the carpet or bare floor and carry them to the cyclone. It must have an adjustment mechanism to establish the nozzle lip parallel to the surface and to achieve the proper suction velocity and pressure drop across the nozzle. A nozzle 12.4 cm long and 1 cm wide, with a 13-mm flange and tapered to the nozzle tubing at no more than 30°, will yield the appropriate velocities when operated as specified in Section 11.

7.1.3 *Gaskets*—Gaskets in joints should be of a material appropriate to avoid sample contamination.

7.1.4 *Cyclone*—The cyclone shall be of a specific size such that a given air flow allows for separation of the particles 5-µm mean aerodynamic diameter and larger. The cyclone must be made of aluminum or stainless steel, and the catch bottle must be made of clear glass or fluorinated ethylene propylene (FEP) to avoid contamination and allow the operator to see the sample.

7.1.5 *Flow Control System*—The flow control system shall allow for substantial volume adjustment. The suction source must be capable of drawing 12 L/s (26.5 CFM) through the system with no restrictions other than the nozzle, cyclone, and flow control system connected. An upright commercial vacuum cleaner with a 7 amp or greater motor capable of pulling a vacuum of 6.5 kPa may be used for this purpose.

7.1.6 *Flow Measuring and Suction Gages*— Two vacuum gages are required— one with a range of 0 to 3.7 kPa (0-15 in. water) is used for setting flow rate and another with a range of 0 to 2.5 kPa (0-10 in. water) is used to set the pressure drop across the vacuum nozzle.

7.1.7 Optional filter holder assembly with appropriate fine particle filter, such as a 25-cm micro-quartz-fibre, binderless, acid-washed filter.[5]

7.2 *Other Equipment*:

7.2.1 *Stopwatch*.

7.2.2 *Masking Tape and Marking Pen*, for outlining sections for sampling.

7.2.3 *Clean Aluminum Foil and Clean Glass or FEP Jars*, for the collection and storage of samples.

7.2.4 *Thermometer* (see Specification E1).

7.2.5 *Relative Humidity Meter* (see Test Method E337).

7.2.6 *Shaker Sieve*, as specified in Test Method D422, with 100 mesh-screen above the pan to separate the fine dust below 150 µm.

7.2.7 *Analytical Balance*, sensitive to at least 0.1 mg and having a weighing range from 0.1 mg to 1000 g.

[4] The sampling device used in the development and performance evaluation of this test method was manufactured by CS-3, Inc., P.O. Box 1461, Sandpoint, ID 83864, which is the sole source of supply of the sampler known to the committee at this time. If you are aware of alternative suppliers, please provide this information to the Committee on Standards, ASTM Headquarters, 100 Barr Harbor Dr., West Conshohocken, PA 19428. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

[5] A filter holder for circular 25–cm particle filters and flow control valve assembly which replaces the normal flow control assembly is available from the manufacturer of the floor vacuum device.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available (**6**).

8.2 Methanol is required for sampling train cleaning after sample collection.

9. Sampling Strategy

9.1 The overall sampling strategy should be designed to address the goals of the study. Users should consider factors such as foot traffic volume, types of activities, proximity to potential sources, etc. The sampling strategy should be described in the sampling report so it can be taken into consideration when readers are comparing loadings or concentrations, or both, to those obtained from other studies. The ideal sampling location(s) for the beginning of the test procedure are an area that conforms with the protocol for the user's overall sampling strategy. For example, when sampling in a home for child exposure assessment, protocol may require the selection of a carpeted area for sampling where small children play or are likely to play.

10. Pretest Preparation and Calibration

10.1 *Calibration*—The sampling system described in this practice does not have any calibrated flow devices other than the cyclone and the Magnehelic gages. The cyclone used for the separation of the particles must be designed to give proper separation at varying flow rates throughout the sampling range of the system. The pressure gages and any other devices (that is, temperature gage) used for testing purposes should be calibrated against a primary standard.

10.1.1 *Pressure Gages*—Pressure gages shall be calibrated against an inclined manometer or other primary standard prior to any field test. One means of checking a Magnehelic gage is to set a flow rate through the sampling system with a manometer and then switch to the Magnehelic gage. If the difference in the readings is more than 3 %, the gage is leaking or is in need of repair or calibration. This should be done at two different flow rates when checking the gage.

10.1.2 The cyclone flow measurement is calibrated with a laminar flow element, spirometer, or roots meter. See the appendix for cyclone calibration with a laminar flow element.

10.2 *Pretest Preparation*:

10.2.1 Each catch bottle to be used shall be clean and inspected for any contamination. The bottles should be marked with masking tape and a marking pen for identification of the test site, time, and date.

10.2.2 The sampling train shall be inspected to ensure that it has been cleaned and assembled properly.

10.2.3 The sampling train shall be leak-checked prior to sampling. This can be accomplished by placing a mailing envelope or a piece of cardboard beneath the nozzle and switching on the suction source. The flow Magnehelic gage should read 5 Pa (0.02 in. H_2O) or less to ensure that the system is leak free. If any leakage is detected, the system shall be inspected for the cause and corrected before use.

11. Sampling

11.1 *Sampling a Carpeted Floor*:

11.1.1 *Pre-Test Survey*—Immediately prior to testing, complete a data form recording all requested information and sketch the area to be sampled. (See Fig. 2 for a sample data form.)

11.1.2 Select a sampling area in accordance with the established protocol for your sampling campaign. This should be determined prior to testing.

11.1.3 A typical sampling procedure may use measuring tapes placed on the carpet so that they are parallel to each other and on either side of the portion of carpet to be sampled (Fig. 3). The measuring tapes should be between 0.5 and 1.5-m apart and extended as far as practical. They should be taped to the carpet with masking tape every 30 cm.

11.1.4 Place the sampler in one corner of the sampling area and adjust the flow rate and pressure drop according to the type of carpet (see 11.1.8). The two factors that affect the efficiency of the sampling system are the flow rate and pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and distance between the surface and the nozzle flange.

11.1.5 Clean the wheels and nozzle lip with a clean laboratory tissue immediately before sampling. Begin sampling by moving the nozzle between the ends of the two measuring tapes. The sampler is then moved back and forth four times on the first strip, moving the sampler at approximately 0.5 m/s. (The widths of the strips are defined by the width of the sampling nozzle.) Effective nozzle width is 13 cm for the CS_3 sampler. Move in a straight line between the numbers on the measuring tape. Angle over to the second strip on the next pass gradually, and repeat four double passes. After sampling approximately 0.5 m^2, determine the amount of collected material in the bottom of the catch bottle. As a rough estimate, the collection of dust to a depth of 6 mm [0.25 in.] in a 55-mm diameter catch bottle corresponds to approximately 6 to 8 g. If there is less than 6 mm of dust, sample an additional 0.5 m^2 next to the area already sampled. Hair, carpet fibers, and other large objects should be excluded from the sample when estimating the quantity collected.

11.1.6 Continue sampling in the area laid out until an adequate sample is collected. Switch off the vacuum. The catch bottle can now be removed, labeled, and capped for storage and analysis. Record the dimensions of the sampled area on the data sheet.

11.1.7 If the rug area to be sampled is very dirty, or has not been cleaned frequently, care must be taken to avoid filling up the cyclone catch bottle on the first sample area. If it is suspected that this will be the case, start with a 0.25-m^2 sampling area. Then take a second and a third area as before, until the catch bottle is 75 % full.

11.1.8 Adjust the flow rate and nozzle pressure drop to values that approximate those given in Table 1. Use the same flow rate and pressure drop on multilevel and shag carpets as that used for plush carpets.

11.2 *Sampling a Bare Floor*:

SAMPLE DATA SHEET

Operator _____ Date _____ Sample Ident. #: ____

Sampling site _____

Type of Carpet: Plush ___ Level Loop ___ Multilevel ___ Shag ___
Type of Vacuum: Upright ___ Canister ___ Other _____

Last Vacuumed _____ Temp. _____ Humidity _____%

Comments: _____
_____

Location of Area Sampled: _____ Area _____m^2

Sketch of Area Sampled:

Leak Check: Yes__ No__; 20 second cleaning @ end: Yes __ No __

Total Sample Time:__minutes __seconds Flow ▲P ____ Nozzle ▲P ___

Bottle final Wt: _____g Tare Wt: _____g Net Wt: _____g

Pan & Sample Wt: _____g Pan Tare Wt: _____g Net Wt: _____g

Total Dust: _____ grams/m^2
Fine Dust: _____ grams/m^2

Cyclone Sample #: _____

Lab Sample #: _____

FIG. 2 Sample Data Sheet for Sampling for Floor Dust

11.2.1 *Pre-Test Survey*—Immediately prior to testing, complete a data form recording all requested information and sketch the area to be sampled. (See Fig. 2 for sample data form.)

11.2.2 Select a sampling area that is as large as possible and in accordance with the established protocol for your sampling campaign. This should be determined prior to testing. Divide the area into parallel areas 0.5 to 1.5 m apart.

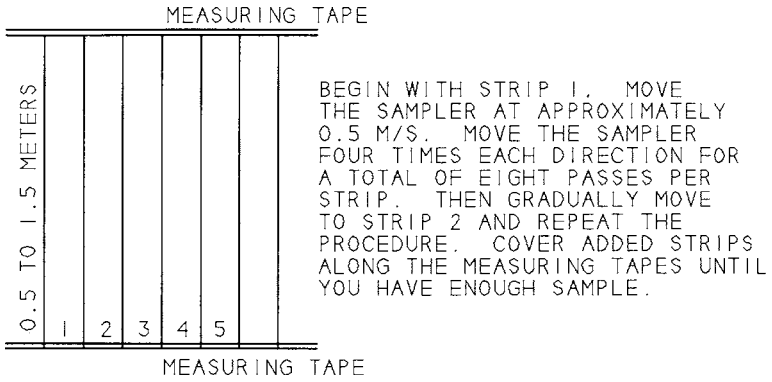


FIG. 3 Example of a Typical Sampling Procedure

TABLE 1 Approximate Values for Flow Rate and Nozzle Pressure Drop

Carpet Type	Flow Rate	Nozzle Pressure Drop
Plush	9.5 L/s (20 CFM)	2.2 kPa (9 in. H_2O)
Level loop	7.6 L/s (16 CFM)	2.5 kPa (10 in. H_2O)

11.2.3 A typical sampling procedure may utilize measuring tapes placed on the floor so that they are parallel to each other and on either side of the portion of floor to be sampled (Fig. 3). The measuring tapes should be between 0.5 and 1.5 m apart and extended as far as practical. They should be taped to the floor every 30 cm with masking tape.

11.2.4 Place the sampler in one corner of the sampling area. Set the height of the nozzle above the floor at approximately 1 mm (a U.S. penny under the nozzle lip will hold it at this height) and adjust the flow rate (see 11.2.7). The two factors that affect the efficiency of the sampling system are the flow rate and the pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and the distance between the surface and nozzle flange.

11.2.5 Clean the wheels and nozzle lip immediately before sampling with a clean laboratory tissue. Begin sampling by moving the nozzle between the ends of the two tapes. The sampler is then moved back and forth two times on the first strip, moving the sampler at approximately 0.5 m/s. (The width of the strips are defined by the width of the sampling nozzle. For the CS_3 sampler, effective nozzle width is 13 cm. Move in a straight line between the numbers on the measuring tape. Gradually angle over to the second strip on the next pass and repeat two double passes. After sampling approximately 10 m^2, check the amount of collected material in the bottom of the catch bottle. As a rough estimate, the collection of dust to a depth of 6 mm [0.25 in.] in a 55 mm diameter catch bottle corresponds to approximately 6 to 8 g. If there is less than 6 mm of dust, sample additional areas as available. It may not be possible to obtain 6 g of dust from a clean or small bare floor.

11.2.6 Continue sampling in the area laid out until an adequate sample is collected. Switch off the vacuum. The catch bottle can now be removed, capped, and labeled for storage and analysis. Record the dimensions of the sampled area on the data sheet.

11.2.7 Adjust the flow rate to a flow of 9.5 L/s (20 CFM).

12. Sample Analysis

12.1 After collection of the sample in the catch bottle, the sample may be left in the same bottle or transferred to another container for transport to the laboratory. The procedure for sample handling is different for metals and organic chemicals. Samples for organic analysis should be maintained at 4°C to the extent possible. (Samples should not be frozen before sieving, as this could alter the particle size distribution.) Storage at ambient temperature is appropriate for samples that will be analyzed only for metals, but cooling the sample is also acceptable.

12.2 If the sample will be analyzed for pesticides or other organic chemicals, transfer the dust from the cyclone catch bottle onto the middle of a piece of aluminum foil that has been cleaned by washing with pesticide-free methanol or hexane. Fold the foil into a small package carefully, keeping the dust in the middle. Place the foil pouch in a clean glass jar. Cover the jar opening with another piece of precleaned foil and secure the lid to the jar. Seal the seam of the lid to the jar with polytetrafluoroethylene tape. Place the sample jar in an ice chest to keep it cool during transport to the laboratory. Label the jar for reference.

12.3 If the sample will be analyzed for metals, it can be transferred from the catch bottle to a new polyethylene "zipper" seal sample bag. Seal the zipper, and tape the seal with any marking tape that will adhere well to the polyethylene bag. Label the sample for reference.

TABLE 2 Sampling Efficiency Using Modified Laboratory Test Method F608[A]

Parameters	Carpet Type	
	Plush	Level Loop
Flow rate (L/s)	9.4	7.6
Delta P (kPa)[B]	2.3	2.5
Mean % of mass collected in cyclone	69.5	66.8
Standard deviation	1.2	2.8
Number of tests	3	3

[A] Carpet dust loading was 15.9 g/m^2.
[B] Pressure drop at nozzle.

12.4 Sieve the samples for 5 min in a shaker in accordance with Test Method D422, with a 100-mesh screen above the pan, to determine the weight of fine dust below 150-µm mean diameter.

12.5 Alternative methods for the storage, shipment, and preparation of samples for analysis may be required for some analytes and should be prescribed for specific sampling protocols. The FEP catch-bottle may be used for storage and shipping.

13. Sampler Cleaning

13.1 After the sample bottle is removed, open the flow control valve to maximum flow, tip the sampler back so that the nozzle is approximately 5 cm [2 in.] off the floor, and switch the vacuum on. Place a hand covered by a rubber glove over the bottom of the cyclone and alternate closing and opening the cyclone for 10 s to free any loose material adhering to the walls of the cyclone and tubing. It is not necessary to catch this small amount of dust, as it is usually much less than 1 % of the collected sample.

13.2 Remove the sampler to a well-ventilated cleaning area free of dust. Remove the cyclone and elbow at the top of nozzle tubing from the sampler. Use a 50-cm long by 3-cm diameter [20 by 1.25-in.] brush to clean the nozzle, and clean all related items up to and including the cyclone and catch bottle with reagent grade methanol. This wash can be analyzed at the discretion of the operator. The total amount of dust removed in the air and wet cleaning is usually much less than 1 % of the collected dust. The air and wet cleaning is performed to prevent contamination from passing from one sample to another.

14. Data Analysis

14.1 Weigh the sieved dust sample with an analytical balance accurate to 0.1 mg.

14.2 Calculate the dust weight by subtracting the weight of the pan sample from the final weight in accordance with Method D422.

14.3 Calculate the loading for dust per square metre (g/m^2) by dividing the final dust weight by the area sampled (expressed in m^2).

14.4 When the analysis results are received from the laboratory, it is possible to calculate the loading of lead, pesticides, or other analytes per square metre of carpet or bare floor area ($\mu g/m^2$) in the same way.

14.5 The concentration of any element or chemical associated with the dust may be determined by analysis.

15. Precision and Bias [6]

15.1 Tests for dust collection efficiency have been performed using Test Method F608 modified by passing it through a 100-mesh sieve (**1,2**). The results are given in Table 2.

15.2 Tests performed with a fine particle filter downstream of the cyclone showed that 99 % or more of the collected test dust was retained in the cyclone catch bottle (**1,2**).

15.3 Tests performed as in 15.2, but with test dust containing lead, showed that 99 % or more of the lead collected was retained in the cyclone catch bottle (**1,2**).

15.4 Tests performed as in 15.2, but with test dust fortified with pesticides, showed that 97 % or more of the pesticides collected were retained in the cyclone catch bottle. The pesticides tested were chlordane, aldrin, chlorpyrifos, heptachlor, and diazinon.

15.5 Tests were conducted on conditioned carpets, as described in Test Method F608.

16. Keywords

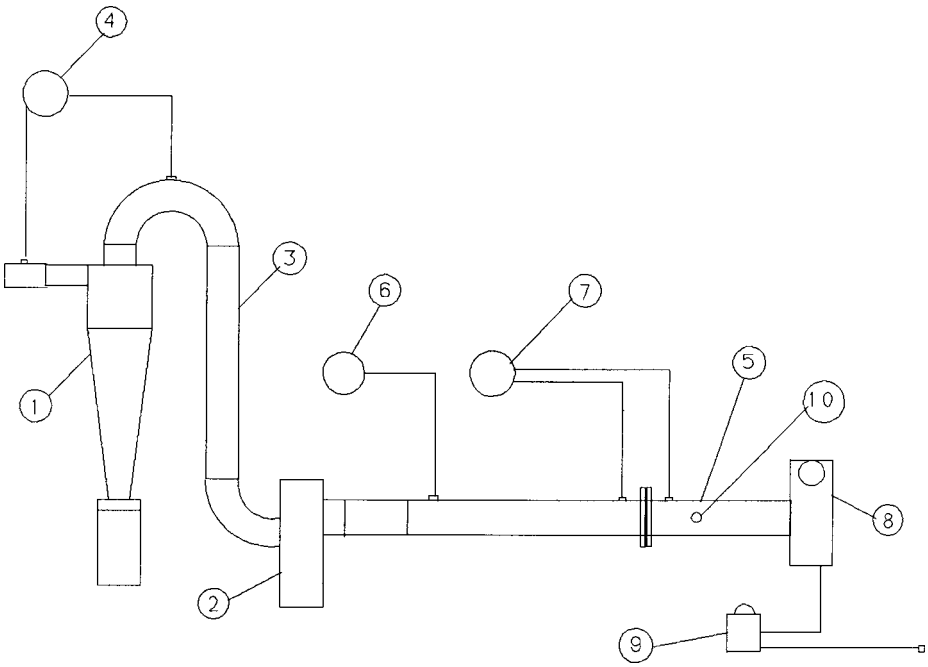
16.1 carpet; cyclone; dust; floors; metals; organic chemicals; particle size; particulate matter; vacuum

[6] Supporting data have been filed at ASTM Headquarters. Request D22-1010.

APPENDIX

(Nonmandatory Information)

X1. CALIBRATION OF CYCLONE USING A LAMINAR-FLOW ELEMENT



1. Cyclone
2. Suction/blower
3. Flow control valve
4. 0 to 2.5 kPa vacuum gage
5. Laminar flow element
6. ±0.25 kPa vacuum gage
7. 0 to 1 kPa vacuum gage
8. Suction/blower
9. Variable autotransformer
10. Temperature gage

FIG. X1.1 Calibration Using a Laminar Flow Element

X1.1 Assemble the necessary components (see Fig. X1.1).

X1.1.1 *Cyclone*.

X1.1.2 *Suction/Blower*.

X1.1.3 *Flow Control Valve*, 1 to 2.5 kPa (0 to 10 in.).

X1.1.4 *Magnehelic Gage*, 1 to 2.5 kPa (0 to 10 in.)

X1.1.5 *Laminar Flow Element* (with manufacturer's certified calibration), with pressure gages and dial thermometer.

X1.1.6 *Suction/Blower*, with power transformer; leak check the system by plugging the inlet to the cyclone and observing the pressure gage.

X1.1.7 Activate Blowers 2 and 8.

X1.1.8 Open the flow control valve on Flow Control Valve 3 so that 2.0 kPa (8.0 in. H_2O) registers on Pressure Gage 4. Then adjust Variable Autotransformer 9 so that 0.0 kPa (0.0 in. H_2O) registers on Pressure Gage 6. Some adjusting of the flow control valve will be necessary.

X1.1.9 Check Pressure Gage 7 for the gas flow reading and record the flow.

X1.1.10 Adjust the flow through the cyclone to 2.5 kPa (10.0 in. H_2O), and repeat the procedure. This action should provide a gas flow rate through the cyclone. This should be between 7.1 and 8.5 L/s (15 to 18 CFM).

REFERENCES

(1) Roberts, J. W., Budd, W. T., Ruby, M. G., Stamper, V. R., Camann, D. E., Fortmann, R. C., Sheldon, L. S., and Lewis, R. G., "A Small High Volume Surface Sampler HVS3 for Pesticides, and Other Toxic Substances in House Dust," *Paper No. 91-150.2*, 84th Annual Meeting, Air & Waste Management Association, Vancouver, British Columbia, June 16-21, 1991.

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(3) Roberts, J. W., Han, W., and Ruby, M. G., "Evaluation of Dust Samplers for Bare Floors and Upholstery," Battelle Subcontract No. 46534(g21733808)-00 03EQ, EPA Prime Contract No. 68-00-0007, U.S. EPA, AREAL, Office of Research and Development, 1993.

(4) Stamper, V. R., Roberts, J. W., and Ruby, M. G., "Development of a High Volume Small Surface Sampler for Pesticide and Toxics in House Dust," *Research Triangle Institute Report No. RTI/171-01/02F*, Research Triangle Park, NC, June 1990. Included in supporting data, which are on file at ASTM Headquarters. Request D22-1010.

(5) Lewis, R. G., Fortune, C. R., Willis, R. D., Camann, D. E., and Antley, J. T., "Distribution of Pesticides and Polycyclic Aromatic Hydrocarbons in House Dust as a Function of Particle Size," *Environ. Health Perspect.*, Vol. 107, 1999, pp. 721–726.

(6) *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

Geoprobe® Operational Program Summary

The following are safety and operational aspects relative to the operation of a Geoprobe® direct-push unit (Model 54DT track-mounted unit). Only specifically qualified personnel will be permitted to operate this hydraulically powered system, and operators will observe the following safety checks and operational practices when working with the direct push system:

- Refer to the Kubota Diesel Engine Operator's Manual for all engine-related safety instructions before operating the Geoprobe® track-mounted machine (cover page and table of contents attached).
- Heed all CAUTION, WARNING, and DANGER decals posted on the machine.
- Operators should wear OSHA-approved steel-toed shoes and keep feet clear of probe foot.
- Operators should wear OSHA-approved safety glasses at all times during the operation of this machine.
- Operators must wear hearing protection. OSHA-approved hearing protection for sound levels exceeding 85 dbs is recommended.
- The Emergency Kill switch button on the control panel will immediately shut off the engine when pushed. Familiarize yourself with the location of this button before operating the machine.
- Ensure that everyone is clear of all moving parts before starting the engine.
- Check that both outriggers are fully raised before attempting to drive the unit.
- Do not drive the machine with the probe cylinder or winch mast extended. This practice could result in equipment damage and/or personal injury from contact with overhead objects such as power lines.
- The unit should only be driven using the remote control box. The steering levers located on the machine are for positioning only. Do not attempt to drive using the levers on the machine as this requires the operator to walk too close to the tracks while the vehicle is in motion.
- Do not attempt to drive the unit on slopes of more than 30 degrees. Always drive straight up or down steep grades. Avoid side slopes whenever possible. Continuous operation should be limited to slopes of less than 20°° to avoid engine damage.
- When maneuvering the unit in close quarters, lower engine speed to provide more precise control of the track assemblies.
- A track-mounted machine is generally transported on a trailer. Use special caution when loading the unit with wet ramps as it is significantly easier for the tracks to slip under such conditions.
- When operating the unit on sloped surfaces, always position the unit parallel with the slope. This provides the greatest degree of stability and will limit shifting during probing or augering operations. Position the track-mounted machine with the control panel up slope whenever possible so the machine will roll away from the operator if it becomes unstable and moves unexpectedly.
- Do not extend the outriggers such that the tracks are raised off of the ground more than one or two inches. Raising the tracks several inches off of the ground surface decreases the stability of the machine and provides no operational advantage.
- Designate one person to operate the machine while probing or augering. This will avoid injuries from having someone unexpectedly engage the machine controls while another person is working near moving parts.
- Operators must stand to the control side of the machine, clear of the probe foot and derrick, while operating the controls. Never reach across the probe assembly to manipulate the machine controls.
- Never place your hands on top of the tool string while raising or lowering the GH60 hammer.
- Never move the probe assembly (swing, extend, fold, etc.) or operate the tracks while anyone is in physical contact with the tool string.

- Use caution when probing on loose or soft surfaces. Reduced weight on the tracks may allow the unit to shift or slide under such conditions.
- Limit the rate at which the GH60 hammer is lowered while advancing the tool string to avoid raising the probe foot more than approximately 6 inches off of the ground surface.
- Never raise the machine foot more than a few inches from the ground surface with the probe cylinder and/or winch mast fully extended. If the foot must be raised significantly, first lower the hammer and winch.
- Always place the machine foot firmly on the ground when pulling tools from the subsurface.
- In the event of a problem, the operator should release all control levers. The spring-loaded levers will automatically return to the neutral position and machine operation will cease.
- Rotating parts can cause serious injuries. Shut off the engine before attempting to clean or service the unit.
- Do not make modifications or add attachments to this machine which are not approved by Geoprobe Systems®.
- Do not wear loose clothing while operating this machine. Severe injury will result if clothing becomes entangled in moving parts.
- Avoid hydraulic fluid leaks. Pressurized fluid may be injected into the skin resulting in serious bodily injury. In the event of an accident, seek medical attention immediately.

Prior to any intrusive activities, areas that will be sampled via direct push techniques will be surveyed and declared free of utility obstructions. Utility infrastructure that can/could pose a hazard to Geoprobe® operators include electrical, gas, water, communications, steam, and sewer lines. This utility marking/clearance certification will be issued by the local public utility's delineation service (e.g., "Miss Utility") and a private, specialty contractor. The public utility's clearance service must engaged in support of this activity out of legal necessity.

OWNER'S MANUAL

Geoprobe Systems

A DIVISION OF KEJR ENGINEERING

5400 Model Geoprobe®

OPERATOR'S MANUAL

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ISOTOPIC URANIUM
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ISOTOPIC URANIUM

1.0 PURPOSE

The purpose of this SOP is to provide a standardized method for the analysis of uranium in water, soil, urine, air filters, and vegetation samples.

2.0 DETECTION LIMITS

The typical detection limit for a 500 ml sample aliquot counted for 500 minutes should be about 0.2 pCi/l.

3.0 SCOPE & APPLICATION

3.1 This method covers the measurement of isotopic uranium in drinking water and other matrices such as soils, air particulates and vegetation samples. Most drinking water sources, especially ground water sources, contain soluble carbonates and bicarbonates, which form complexes and keep uranium in solution.

3.2 Uranium isotopic abundances vary depending upon the source. Uranium isotopes in drinking water may be present in ratios that differ from those in the minerals from which the uranium entered the aqueous phase. The predominant natural alpha emitting isotopes of uranium are Uranium-234, Uranium-235 and Uranium-238.

4.0 SUMMARY

4.1 This procedure is a modification of ASTM D3972-97. The following are modifications:

4.1.1 Ammonium Iodide or Sodium Iodide used instead of hydriodic Acid,
4.1.2 Neodymium Fluoride precipitation instead of electroplating,
4.1.3 Section added for the digestion of urine, soils and other solids.

4.2 The sample is acidified by the addition of hydrochloric acid and boiled to eliminate carbonate and bicarbonate. Uranium is then co-precipitated with ferric hydroxide and separated from the sample. The ferric hydroxide is then dissolved in 9N HCl, and the uranium is separated from other radionuclides in an ion exchange column. The column is then rinsed with 9N HCl and NH_4I or NaI, washed with 9N HCl, and the uranium is finally eluted with 0.1N HCl.

4.3 The eluate is evaporated and the salts converted to chlorides prior to microprecipitation as a neodymium fluoride complex. The sample is counted by alpha spectroscopy for Uranium-238, Uranium-235 and Uranium-234 at 4.2 MeV,

4.4 MeV and 4.7-4.8 MeV respectively with U-232 at 5.27 - 5.32 MeV as a tracer.

4.4 Uranium chemical recoveries are determined on each sample through the use of U-232 as a tracer. QC standards and/or matrix spikes are taken through the entire procedure. The U-232 tracer is added to a measured aliquot or weighed volume of sample.

5.0 DEFINITIONS

5.1 Accuracy – The closeness of agreement between an observed value and an accepted value.

5.2 Batch – A group of samples which behave similarly with respect to the sampling or the testing procedures employed and which are processed as a unit.

5.3 Bias – the deviation due to matrix effects of the measured value from a known spiked amount.

5.4 Method Detection Limit – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

5.5 Precision – The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses.

5.6 MAPEP – The United States Department of Energy Mixed Analyte *Performance Evaluation Program.*

6.0 INTERFERENCES

6.1 The only potentially interfering alpha-emitting radionuclide is Protactinium-231 at 5.0 MeV. However, Protactinium-231 has a low natural abundance and, therefore, the interference is normally minor.

6.2 Since uranium is a naturally occurring radionuclide, reagents must be checked for uranium contamination by analyzing a reagent blank along with the samples.

6.3 If you are uncertain as to the extent of the matrix interferences within a particular sample, a separate aliquot of that sample must be spiked with a known amount of NIST or equivalent traceable standard and carried through the entire procedure to monitor the recovery.

7.0 SAFETY PRECAUTIONS

7.1 Hazardous Chemicals/Equipment

All work is to be performed in a fume hood with the following chemicals, 1) nitric acid, concentrated, and 2) hydrofluoric acid.

7.2 Radiological Hazards

When working with radioactive materials that are capable of being volatilized or airborne, perform activities in a fume hood.

7.3 General Laboratory Safety

Refer to the Laboratory Chemical Hygiene Plan for general laboratory safety.

8.0 EQUIPMENT & SUPPLIES

8.1 Canberra Genie 2000 Alpha Spectroscopy System or equivalent.

8.2 Automatic Pipette.

8.3 Electric hot plate.

8.4 Ion exchange column: approximately 13mm (I.D.) X 150 mm long with a 100 mL reservoir.

8.5 Stainless steel and/or aluminum counting planchets, 25.4mm in diameter by 1.59mm deep or equivalent.

8.6 Gelman filter apparatus, 47 mm, and 25 mm.

8.7 Membrane filters, 0.45µ or equivalent, 47 mm. Membrane filters 0.1µ, or equivalent, 25 mm.

8.8 Exhaust/Fume hood. If perchloric acid is being used, perchloric hood is required.

8.9 200, 500, and 1000 ml beakers (Glass and Teflon)

8.10 Filters, Fisherbrand Q5 or equivalent.

9.0 REAGENTS

All chemicals shall be ACS reagent grade or equivalent where applicable.

9.1 Ammonium hydroxide, concentrated and 6N (mix 2 volumes 15N NH_4OH with 3 volumes of carbonate-free deionized water).

9.2 Anion exchange resin - Strongly basic, styrene, quaternary ammonium salt, 4% cross-linked, 100-200 mesh, chloride form (such as Dowex 1 x 8, or equivalent).

9.3 Ferric chloride carrier, 20 mg Fe^{+3}/mL: Dissolve 9.6 g of $FeCl_3$. $6H_2O$ in 100 mL of 0.5N HCl.

9.4 Ammonium Iodide in 9N HCl solution: Dissolve 25 grams NH_4I in 1000 ml 9N HCl. (Make up immediately before use)

9.5 Hydrochloric acid, 12 N: HCl (conc.) spec. grav. 1.19, 37.2%.

9.6 Hydrochloric acid, 9N (mix 3 volumes 12N HCl (conc.) with 1 volume of deionized water).

9.7 Hydrochloric acid 6N (mix 1 volume 12N HCl (conc.) with 1 volume of deionized water).

9.8 Hydrochloric acid 0.1N (mix 1 volume 0.5N HCl with 4 volumes of deionized water).

9.9 Nitric Acid, 16N: HNO_3 (conc.), spec. grav. 1.42, 70.4%.

9.10 Uranium-232 tracer solution, NIST traceable or equivalent.

9.11 Deionized water, 10 μ mho/cm.

9.12 Uranium 238 standard, NIST traceable or equivalent.

9.13 Titanium IV chloride 20%.

9.14 Neodymium carrier $Nd(NO_3)$ $6H_2O$ (0.2625 g/400 ml).

9.15 Elmers glue or equivalent.

9.16 Hydrogen Peroxide 30-35%.

9.17 NaI in 9N HCl. Dissolve 25g NaI in 1000ml of 9N HCl. (Must be filtered prior to use)

9.18 80% Ethanol and 20% water.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 The sample should be collected, usually by the client, in accordance with accepted practices for obtaining the best representative sample.

10.2 Although carbonate ions in a water sample will help to keep uranium in solution, the addition of extra carbonate or bicarbonate ions to the sample as a preservative is not recommended because an increased carbonate concentration in the sample may cause some precipitation. Therefore, it is recommended that samples be preserved with HNO_3 to pH < 2 at the time of collection.

10.3 If sample is received unpreserved, preserve with HNO_3 to pH <2 and hold for 16 hours prior to analysis.

11.0 QUALITY CONTROL

11.1 Samples shall be stored or handled in such a manner that they will not be contaminated by, nor will they contaminate other samples or standards.

11.2 To monitor analytical performance, each set of samples processed by this method shall contain the following quality control samples:

11.2.1 Laboratory Control Standard (LCS) – A control sample of known composition. Aqueous and control samples are analyzed using the same preparation, reagents, and analytical methods as used for the samples. Frequency: one per batch.

11.2.2 Method Blank (MB) – The preparation blank shall be a volume / weight of deionized water of the same volume / weight as the sample that is taken through the entire procedure. Frequency: one per batch.

11.2.3 Duplicate – A second aliquot (same volume / weight / count time) of the sample that is taken through the exact same procedure. Frequency: one per batch.

11.2.4 Matrix Spike (MS) & Matrix Spike Duplicate (MSD) – An aliquot of a sample fortified (spiked) with known quantities of analyte and subjected to the entire analytical procedure. Frequency: one per batch.

11.3 Chemical Recovery – The Chemical Recovery for U (isotopic) will be conducted using U-232 as a chemical tracer. Frequency: Each sample will be spiked with a U-232 tracer.

12.0 CALIBRATION

All counting equipment used for analysis shall be properly calibrated and response checked prior to use. Refer to the appropriate manufacturer instructions and Outreach SOP for Alpha Spec Calibration (RAD_03) for further details.

13.0 PROCEDURE

13.1 SAMPLE PREPARATION - WATERS

Determine which fraction (dissolved, suspended, or total) of the sample is to be analyzed. The client in the contract or sales order should specify this.

13.1.1 DISSOLVED is that fraction which has passed through a 0.45 µ filter. Water samples, which have been pre-filtered in the field, need no further preparation.

13.1.1.1 Aliquot 100 to 1000 mL of sample, or enough to meet the required detection limit, into an appropriately sized beaker. Add Uranium-232 tracer if applicable.

13.1.1.2 Dilute to approximately 700 mL, if necessary, add 10mL of concentrated hydrochloric acid and proceed to step 13.4.1.

13.1.2 SUSPENDED is that fraction retained on a 0.45 µ filter.

13.1.2.1 Suspended fractions are prepared from the "total unacidified" fraction by shaking an unfiltered sample, aliquoting the desired amount of sample needed to meet the required detection limit (if the sample volume and suspended load permits) and filtering the sample using a 0.45 µ membrane filter.

13.1.2.2 The filter containing the suspended fraction is placed in a medium-sized beaker with the addition of 10 mL of HNO_3 and 10 mL of HF and placed on a hotplate. Uranium-232 tracer is added if applicable.

13.1.2.3 After digesting to near dryness, remove and allow to cool. Do not bake as this could cause poor chemical recoveries.

13.1.2.4 Add 5 mL of HCl, rinse the sides of the beaker and bring the volume to approximately 50 mL with DI H_2O. Transfer to an appropriately sized beaker (1000 mL or larger).

13.1.2.5 Heat for approximately 5 minutes.

13.1.2.6 If necessary, filter the digest through a 0.45 µ filter to remove any undissolved residue. (Note that some samples may require that the solid material should be completely dissolved. Refer to any special instructions from the client

for additional information.)

13.1.2.7 Revolume the filtrate to 700 mL with deionized water and proceed to step 13.4.1.

13.1.3 TOTAL analyses are prepared by performing the suspended fraction preparation step in 13.1.2 and then adding this fraction back into the dissolved fraction. If necessary, two separate analyses can be performed (dissolved and suspended) and the final results added together to arrive at a "total" analysis. Proceed to step 13.4.1. Each fraction must have tracer added unless the fractions are to be analyzed as one sample.

13.2 SAMPLE PREPARATION – SOILS, VEGETATION & MISC SOLIDS

13.2.1 All solids shall be dried and homogenized prior to aliquoting.

NOTE: Vegetation requires ashing prior to digestion at 500^{o}C for 16 hours.

13.2.2 Weigh a representative 0.500 to 1.000 grams (or enough to meet the required detection limit) of dry, homogenized material into a 250 mL Teflon beaker and record the weight on the worksheet. Add Uranium-232 tracer.

13.2.3 Add 20 mL of concentrated nitric acid (HNO_3), 10 ML of hydrofluoric acid (HF), 5mL 18N Sulfuric Acid (H_2SO_4). Place on a hotplate and cook to near dryness.

13.2.4 Remove the sample and allow it to cool.

13.2.5 Add 10 ml of concentrated HCl and place the sample back on the hotplate and heat to just under boiling to dissolve the residue (5-10 minutes).

13.2.6 Once the residue has dissolved, remove the sample from the heat and allow it to cool.

13.2.7 Rinse the sides of the beaker with deionized water.

13.2.8 Transfer to a 400 mL glass beaker and evaporate to dryness.

13.2.9 Place on a hot plate capable of 500^{o}C and evaporate till H_2SO_4 no longer fumes. Cool. Add 10ml HNO_3 and 10ml H_2O_2 and evaporate to dryness.

NOTE: If obvious organic material remains the H_2O_2 should be added in 1 ml portions.

13.2.10 Add 20 mL 9M HCl to dissolve and proceed to Ion Exchange Procedure.

13.3 SAMPLE PREPARATION - URINES

13.3.1 Shake the sample vigorously and aliquot a representative sample of up to 700mL, depending upon sample size, of urine or enough to meet the required detection limit, into an appropriately sized beaker (>/=1000 mL). Add Uranium-232 tracer if applicable.

13.3.2 Dilute to 700 mL with deionized water, if necessary, and add 10mL of concentrated hydrochloric acid and proceed with steps 13.4.1.

13.4 CHEMICAL SEPARATION OF URANIUM

13.4.1 Add 1 mL ferric chloride carrier.

13.4.2 Mix the sample completely and use pH paper to check the hydrogen-ion concentration. If the pH is > 1, add 12N HCl until the pH is less than 1.

13.4.3 Cover the beaker with a watch glass and heat the sample to steaming hot (approximately 20 minutes).

13.4.4 The pH must be checked again after boiling and if it is > 1, 12N HCl must be added to bring the pH back to 1.

13.4.5 While the sample is still hot and covered, gently add, while stirring, concentrated NH_4OH or 6N NH_4OH to the sample from a polyethylene squeeze bottle.

13.4.6 Add NH_4OH until turbidity persists while boiling continues and then add an additional 10 mL. Add phenolthalene to ensure alkalinity.

13.4.7 Continue to heat the sample for 10 minutes more and then set it aside and stir for at least 30 minutes to cool. After 30 minutes, remove stir bar and allow to settle.

13.4.8 After the sample has settled sufficiently, decant and discard as much of the supernatant as possible.

13.4.9 Slurry the remaining precipitate, transfer to a labeled C-tube with deionized water and centrifuge for 5 to 10 minutes. The sample can be filtered through a 0.45 um membrane filter instead of centrifuging, but it is very slow.

13.4.10 Discard the supernatant. Repeat step 13.4.9 until all of the precipitate has been transferred to the C-tube.

13.4.11 Rinse the sides of the beaker with 9N HCl to dissolve all residue. Add this to the C-tube. Dissolve the precipitate in approximately 5-10 mL of 9N HCl. The more solids the more 9M HCI needs to be added.

13.5 ION EXCHANGE COLUMN PREPARATION

13.5.1 Mix AG 1x 8 anion exchange resin with DI and fill column at least to the 7 ml mark.

13.5.2 Charge with 30 mL of 9N HCl and then drain solution to the top of the resin.

13.6 ION EXCHANGE PROCEDURE

NOTE: Soils or high solid samples filter through a Q5 filter before loading sample.

13.6.1 Pass each sample solution from 13.4.11 through its respective resin column at a flow rate not to exceed 5 mL per minute.

13.6.2 After all of the sample solution has passed through the column, eluate the iron (and plutonium if present) with 50 mL of NH_4I solution.

NOTE: If NaI is used instead of NH_4I, filter prior to adding to column.

NOTE: The eluate from this step can be analyzed for Pu.

13.6.3 Wash the column with an additional 50 mL of 9N HCl.

13.6.4 Elute the uranium with 50 mL of 0.1N HCl into a glass beaker.

13.6.5 Add 10ml HNO_3 acid and evaporate the eluate to dryness. Rinse side of beaker with 10 mL conc. HNO_3 and add 5 ml H_2O_2 and evaporate to dryness. Repeat as necessary until the residue is white or colorless.

13.6.6 Add 5mL HCI and evaporate to dryness.

13.7 MOUNTING PROCEDURES

13.7.1 Microprecipitation

13.7.1.1 Add 5 ml concentrated HCl.

13.7.1.2 Add 45 ml DI water and warm slightly to dissolve any salts.

13.7.1.3 Add 1 ml Neodymium carrier and mix.

13.7.1.4 Add 1 ml Titanium (IV) chloride, mix and allow to sit for 5 minutes.

13.7.1.5 Add 1 mL concentrated HF carefully.

13.7.1.6 Mix the samples for 1 – 2 minutes.

13.7.1.7 Let the samples sit for 20 – 30 minutes.

13.7.1.8 Mount the precipitate on a 25mm/0.1 μ filter membrane. Rinse once with deionized water and ethanol to dry.

13.7.1.9 Stick to a labeled planchet with glue. Let dry 30 minutes.

13.7.1.10 Place in a plastic holder and submit for counting.

13.8 COUNTING BY ALPHA SPECTROSCOPY

13.8.1 Place sample disc in counting chamber by venting chamber, open chamber door and insert sample then close chamber and reapply vacuum.

13.8.2 Load "Alpha Analysis" from menu.

13.8.3 Select the "File" menu, select "Open", Select "Detectors", and choose the detectors being used. This has to be done only once for each detector.

13.8.4 Select the “MCA” menu and select “Acquire Setup”. Enter in the desired count-time. Select OK when complete.

13.8.5 Select Edit and enter the Sample id.

13.8.6 Select the "MCA" menu again and select "Acquire Start".

13.8.7 After complete, save data as YXXPPPPSS.cnf

Y	=	Analysis Code Example (“R” = Radium)
XX	=	detector number
PPP	=	Run Number
SS	=	Batch sample number (example MB = 01)

14.0 CALCULATIONS

$$\text{ACTIVITY} = \frac{(CPM_{sample} - CPM_{bkgd})}{}$$

$$R*V*E*D*2.22$$

$$ERROR = \frac{1.96*SQRT\left(\frac{CPM_{sample}}{T_{sample}} + \frac{CPM_{bkgd}}{T_{bkgd}}\right)}{R*V*E*D*2.22}$$

$$MDR = \frac{4.65*SQRT(CPM_{bkgd}) + 2.71}{T}$$

$$MDA/MDC = \frac{MDR}{R*V*E*D*2.22}.$$

T = Count time

R = Chemical or Tracer recovery

V = Sample Volume or Weight

E = Efficiency

D = Decay or Ingrowth Factor

2.22 = Conversion from dpm to pCi

15.0 METHOD PERFORMANCE

This method has been validated by the ASTM.

16.0 POLLUTION PREVENTION

Refer to Laboratory Chemical Hygiene Plan, SOP #GEN_22, for laboratory pollution control.

17.0 DATA ASSESSMENT & ACCEPTANCE CRITERIA

Calibration/QC Sample	Frequency	Acceptance Criteria	Corrective Action
Instrument Calibration Standard	Daily	90 –110 %	Recount twice, if not in control limits place detector on hold for maintenance
Instrument Calibration Blank	Daily	Control chart ± 3 sigma	Recount twice, if not in control limits, place detector on hold for maintenance
Laboratory Control Standard (LCS)	5%	80 – 120 %	Recount/Reanalyze LCS and all associated samples
Preparation Blank (PB)	5%	(result – error) ≤ SDL	Recount/Reanalyze PB and all associated samples
Duplicate	5%	< 20 RPD	Recount/Reanalyze duplicate, if not in control limits, flag and report in case narrative
Matrix Spike/MSD	5%	75 – 125% (W) 65 – 135 % (S)	Recount/Reanalyze spike, if not in control limits, flag and report in case narrative
Tracer Recovery	U-232	25-150%	Recount/Reanalyze sample, if not in control limits, flag and report in case narrative

18.0 WASTE MANAGEMENT

This procedure generates acid, possible radioactive waste from sample preparation and radioactive waste from counting the planchet. Refer to GEN-19, GEN-20 and GEN-23 for the disposal of the appropriate waste stream.

19.0 REFERENCES

19.1 ASTM, Method D3972-97." October 1997.

19.2 US EPA, "Prescribed Procedures of Measurement of Radioactivity in Drinking Water, Method 908.0." EPA-60014-80-032, August 1980.

19.3 Bishop, C. T., et.al. "Radiometric Method for the Determination of Uranium in Water," EPA 600/7-79-093, EMSL-LV, April 1979.

19.4 Edwards, K. W., "Isotopic Analysis of Uranium in Natural of Waters by Alpha Spectrometry," Radiochemical Analysis of Water, Geological Survey Water - Supply Paper 1697-F, U.S. Government Printing Office, Washington, D.C., 1968.

19.5 DOE HASL 300, U-02-RC.

19.6 EPA U-00-07

19.7 DoD Quality systems Manual Version 4.1, 04/22/09

GAMMA SPECTROMETRY
TABLE OF CONTENTS

SAMPLE ANALYSIS BY GAMMA SPECTROMETRY

1.0 PURPOSE

This method describes the use of gamma spectroscopy for the measurement of gamma photons emitted from radionuclides without separating them from the sample matrix. The method can be applied to soil, water, air filters, etc. providing the sample can be condensed or reduced in size such that it can be placed in a calibrated geometry for counting.

2.0 DETECTION LIMIT

The detection limits for Cs-134 and Cs-137, which are 10 and 20 respectively, are met with this procedure. Many other isotopes can be identified with this method and the detection limits are established by the customer or regulatory agency that governs the specific project.

3.0 SCOPE AND APPLICATION

3.1 Two types of gamma detectors are currently used, sodium iodide crystal, NaI, and high purity germanium detector, HPGe. Because of its energy resolution advantage and the availability of large active volume detectors, a HPGe detection system is used for measuring gamma emitting radionuclides.

3.2 The method is applicable for analyzing aqueous, non-aqueous, and solids samples that contain radionuclides emitting gamma photons with energies ranging from about 55 to 2000 keV.

4.0 SUMMARY OF METHOD

Solid samples are mixed as well as possible in their as-received containers. Liquid samples are shaken immediately before being transferred to the counting container. No other sample processing is done except to transfer to a suitable container (or plate) for counting.

A portion of the as-received or dried and ground sample is placed in a container such as a poly-bottle or marinelli beaker for which a calibration exists. An intrinsic germanium detector, interfaced with a 8192-channel multichannel analyzer controlled by Canberra Genie 2000 software is used to acquire and analyze the gamma spectrum. Data is then corrected for background.

5.0 DEFINITIONS

5.1 Accuracy – The closeness of agreement between an observed value and an accepted value.

5.2 Batch – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit.

5.3 Bias – the deviation due to matrix effects of the measured value from a known spiked amount.

5.4 Matrix Duplicate – An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

5.5 Matrix – The component or substrate which contains the analyte of interest; ie: surface water, drinking water, or soil.

5.6 Method Detection Limit – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

5.7 Precision – The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses.

5.8 MAPEP – The United States Department of Energy *Mixed Analyte Performance Evaluation Program.*

6.0 INTERFERENCES

6.1 Interferences are minimal in this method due to the high resolution of the intrinsic germanium detector.

6.2 Naturally occurring radionuclides found in building components may result in the background peaks not truly in the sample. These are corrected during the data reduction process.

7.0 SAFETY PRECAUTIONS

7.1 Hazardous Chemicals/Equipment

All work is to be performed in a fume hood with the following chemicals, 1) nitric acid, concentrated, and 2) hydrofluoric acid.

7.2 Radiological Hazards
When working with radioactive materials that are capable of being volatilized or airborne, perform activities in a fume hood.

7.3 General Laboratory Safety

Refer to the Laboratory Chemical Hygiene Plan for general laboratory safety.

8.0 EQUIPMENT AND SUPPLIES

8.1 Marinelli beakers: 0.5, 1.0, 2.0 and 4.0-L.

8.2 Graduated cylinders: 0.5, 1.0 and 2.0-L.

8.3 Poly-bottles and poly-containers: 160 & 500 mL.

8.4 Petri dishes: 50mm and 100mm plastic disposable

8.5 Spatula.

8.6 Electric or duct tape.

8.7 Analytical balance.

8.8 High purity germanium detector with shield.

8.9 Spectroscopy amplifier.

8.10 High voltage supply.

8.11 Multichannel analyzer: 4096 channels, equipped with Computer with printer and Genie 2000 application software.

8.12 90 mm / 0.45 μm membrane filter

8.13 Fleaker Filtration System

8.14 Pestle and mortar

8.15 12 mesh sieve

8.16 Liquid Nitrogen – Nitrogen Service

9.0 REAGENTS AND STANDARDS

NOTE: All chemicals used for reagents are ACS or equivalent.

9.1 Deionized Water.

9.2 Sand.

9.3 Calibration Standards – NIST or equivalent

	Source #	Geometry	Wt/Vol
9.3.1	763-96-4	**B1 -** 160ml bottle	244g
9.3.2	3617-C2	**C2 -**500ml container	300g
9.3.3	763-86-5	**C1** -500ml container	718g
9.3.4	M1020499	**M1**-1L marinelli	1 L
9.3.5	M2020499	**M2**-2L Marinelli	2 L
9.3.6	3617-P1	**P1** (50 mm petri dish)	21.5g
9.3.7	3617-P2	**P2** (100 mm petri dish)	81.9g
9.3.8	3617-P3	**P3** (100 mm petri dish)	66g
9.3.9	3454 BLI	**BLI** (160 ml bottle)	163g (Pb210)
9.3.10	4949L	**BI129** (160ml bottle)	100 ml (I129)
9.3.11	081604F1	**Fl** (47mm filter)	filter
9.3.12	081604P50	**P1** (50mm Petri dish)	39g

10.0 SAMPLE COLLECTION AND PRESERVATION

10.1 Samples should be collected and stored in plastic rather than glass to prevent loss due to breakage during transportation and handling.

10.2 Samples should be preserved at the time of collection with nitric acid to the pH of 2 or less. If samples are to be collected without preservation, they should be brought to the laboratory within 5 days and then preserved and held in the original container for a minimum of 16 hours before analysis or transfer of the sample. Samples do not require refrigeration.

Note: If soluble or insoluble analysis is requested do not preserve.

10.3 Care should be taken during shipment of samples to avoid a contaminating spillage or cross contamination of multiple samples.

11.0 QUALITY CONTROL

11.1 Sample Quality Control

11.1.1 Prepare a blank, a lab control standard, and a duplicate for each batch of up to twenty like samples.

11.1.2 Blanks and lab control samples are prepared using sand for solid matrices and reagent water for aqueous matrices.

11.2 Instrument Quality Control

11.2.1 Count a background check for 1800 seconds or longer on each detector each day it is in use or with each sample batch.

11.2.2 Count a 60,000 second blank monthly, at a minimum, for each matrix analyzed to be used for background correction.

11.2.3 Count the appropriate check source for sample geometry for a minimum of 300 seconds on each detector each day the detector is in use.

B1 - 59, 662, 1173 and 1332
C1 - 59, 662, 1173 and 1332
P1-P3 – 77, 242, 352, 609, 1120 and 1765
C2- 46.5, 77, 242, 352, 609, 1120 and 1765
M1-M2-P50 – 81, 356, 662, 1173 and 1332

ALL ENERGY LINES MUST PASS

11.3 Corrective Action

11.3.1 Nonconformances may occur at any level. Some may be corrected immediately and documented using normal laboratory document procedures. Out-of-control nonconformances shall be documented by the use of a Corrective Action Report. With the use of a CAR, findings can be tracked and used to detect future trends.

11.3.2 Work shall be stopped when out-of-control deficiencies occur until the problem is alleviated, ie., instrumentation malfuntion, QC out of control limits, failure to perform demonstration of capabilities, etc.

12.0 CALIBRATION

Note: Calibration is performed on each spectrometer system at least annually, at a minimum, on each geometry in which measurements will be made.

12.1 Determine which calibration standard needs to be used and place in appropriate detector.
12.2 Acquire spectrum following steps 13.3.

12.3 Save Calibration with the following format:

XXZZMMDD.MCA

where XX = the detector number
ZZ = a geometry description (e.g., MI is a 1 L marinelli beaker).
MM = Month
DD = Day

12.4 Select from the **Calibration** Menu.

12.5 Select **Full Energy Calibration** from the Calibration Menu and select appropriate geometry. (KeV ≈ 0.5 per channel, Full width half max, FWHM, < 3.0 for Co-60 at 1173 KeV).

12.6 Select **Full Efficiency** from the Calibration Menu and select appropriate geometry.

12.7 Save the sample data using the following format:

C:\CALIBRATIONS\DET*\ZZMMDDYY.s0

where ZZ = a geometry description (e.g., M1 is a 1 L marinelli beaker).
MM = Month
DD = Day
YY = Year

12.8 Repeat for each detector and calibration source.

13.0 PROCEDURE

Note: If a detector has been out of service (e.g., thermocycle), do not apply bias across the detector unless it has been sufficiently cooled to liquid nitrogen temperatures overnight.

13.1 Water Sample Preparation

13.1.1 Measure 0.1, 0.5, 1.0 or 2.0 L of a sample and transfer into the appropriate sized marinelli beaker or 160 bottle for 0.1L. Aliquot depends on sample available, but 1.0 or 2.0 L is preferable.

Note: If soluble and insoluble are requested filter the sample through a 90 mm/0.45 μm pore size filter. Transfer filtrate to an appropriate sized marinelli beaker and save filter for analysis.

13.1.2 Place a lid on the container and seal it with tape.

13.1.3 Transfer the container to the counting room for analysis.

13.1.4 Record the sample number on the lid and record the sample number and volume in the gamma spec log book prior to counting the sample.

13.2 Soil or Sludge Sample Preparation

Note: If sample does not require drying and grinding, skip to step 13.2.2.

SOP WET_24 also outlines solid prep procedures.

13.2.1 Dry sample at 100^{o}C for at least 16 hours. Grind the sample to pass through a 12 mesh sieve.

Note: Grind all samples in an exhaust hood.

13.2.2 Place well mixed sample into a tared 160 ml bottle or 500 ml container. Fill the container full with sample depending on the volume of sample available and cap the bottle or container and seal with tape.

Note: If there is not enough sample to fill a 160ml bottle, a petri dish (P1-P3) may be used.

13.2.3 Weigh the filled sample bottle subtracting the bottles tare weight.

13.2.4 Record the weight of the sample, the sample number on the bottle, sample date and prep date in oven drying process log book.

13.2.5 Transfer the sample to the counting room for analysis.

Note: Solid samples should be compacted as well as possible to eliminate channels and air pockets.

13.2.6 Place sample in a plastic bag prior to counting to prevent contamination on the detector.

13.3 Data Acquisition

13.3.1 Record the detector number, sample identification number, volume, count date and geometry in the gamma spec log book.

13.3.2 Place the samples in the appropriate detector cavities and close the shield lids.

13.3.3 Preset the appropriate counting time using the **MCA-Aquire setup** keys. Liquid samples are typically counted for 500 minutes (30,000 sec) and solid samples are typically counted for 100 minutes (6000 sec). Counting times may be lengthened or shortened depending on required minimum detectable activities (MDA).

13.3.4 Clear any previous spectra on the screen by selecting the clear button on the screen. (Be sure previous spectra have been stored prior to erasing.)

13.3.5 Acquire the sample spectra by selecting the **Start** button on the screen. Select **Edit** from the top menu and enter sample information. Enter data in the appropriate fields.

13.3.6 Save the acquired spectra individually. This is accomplished by selecting **File** then **Save**. Save the sample using the following format:

The filename has the following format:

XXZZZZSS.CNF

where XX = the detector number. ie., Detector 1 – 01
ZZZZ = the unique batch number assigned to the samples.
SS=sample sub id

13.3.7 Detectors may be operated independently by following steps 13.3.1 through 13.3.5.

13.4 Spectral Data Reduction

13.4.1 Select **Analyze** from the top menu.

13.4.2 Select from the Macro for detector analysis being performed on.

13.4.3 Select from File Menu-**Print Listing**.

13.4.4 Save the sample data using the following format:

The filename has the following format:

XXZZZZSS.CNF

where XX = the detector number. ie., Detector 1 – 01
ZZZZ = the unique batch number assigned to the samples.
SS=sample sub id

13.5 Data Collection

Document the data in a bound lab notebook for each set of analyses performed. Entries must be made at the time of analysis and include the following:

- description of activity being documented (e.g., "gamma Spec Analysis") and procedure being followed.

- Date analysis started and analyst(s) initial(s).
- count length.

- Outreach batch number and sample aliquot. Identify any lab quality control samples (LCSs).

13.6 Data Reduction and Reporting for Gamma Spec

Using the Canberra Genie 2000, report gamma spectroscopy scan data as follows:

Report each nuclide that the data system has identified and quantified to two significant figures along with the counting uncertainty rounded to two significant figures with the following provisos:

- Report only those nuclides positively identified and quantified by the Canberra program.

In evaluating the identification made by the software, consider the following:

- Agreement of results for multiple photon peaks of a given nuclide.

- Possible identifications of any unidentified peaks.

- Possibility that identified nuclides may actually be low abundance peaks of other nuclides found in the sample that have not been included in the libraries.

Document and support changes to automated nuclide identifications on the instrument printout.

- Do not report a lower order of magnitude (decimal place) in the uncertainty than will be reported in the activity.

  Examples:

  176 +/- 32.1 pCi/L is reported as 176 +/- 32
  176 +/- 8.7 pCi/L is reported as 176 +/- 9

- If the counting uncertainty is greater than the reported activity, do not report the nuclide as found.

- If no nuclides are identified by the data system, report "ND" in the results column of the LIMS data entry screen and "ND = Not detected" in the Value Text field of the data entry screen.

13.7 Data Reduction and Reporting for Specific Target Nuclides

If a specific nuclide is listed as an analyte in the LIMS and it was identified and quantified by the data system, report it as described above. If the specific nuclide is not identified and quantified by the data system, report the minimum detectable activity (MDA) for those nuclides as calculated by the data system. Report MDA values to one significant figure.

Qualify results for soil samples as follows: "Samples were oven-dried at 103^{o}C prior to analysis. Nuclides volatile at this temperature are, therefore, excluded from analysis."

14.0 CALCULATIONS

The Genie 2000 software calculates the data using programs provided by Canberra and takes into account many variables. If Outreach calculates manually these are the equations used.

$$\text{ACTIVITY} = \frac{(CPM_{sample} - CPM_{bkgd})}{R*V*E*D*2.22}$$

$$\text{ERROR} = \frac{1.96*SQRT\left(\frac{CPM_{sample}}{T_{sample}} + \frac{CPM_{bkgd}}{T_{bkgd}}\right)}{R*V*E*D*2.22}$$

$$\text{MDR} = 4.65*SQRT(CPM_{bkgd}) + \frac{2.71}{T}$$

$$\text{MDA/MDC} = \frac{MDR}{R*V*E*D*2.22}.$$

T	=	Count time
R	=	Chemical or Tracer recovery
V	=	Sample Volume or Weight
E	=	Efficiency
2.22	=	Conversion from dpm to pCi

D = Radionuclide decay factor $e^{-\Lambda t}$

15.0 METHOD PERFORMANCE

15.1 Method performance is established by the use of demonstration of capability documentation. Performance evaluations by way of *QAP* and/or *MAPEP* are performed to track ongoing competency.

15.2 Method Detection Limits may be established but will vary due to sample matrices as well as concentration of particular isotopes found in the sample(s).

16.0 POLLUTION CONTROL

Refer to the Laboratory Chemical Hygiene Plan, SOP# GEN_22, for laboratory pollution control.

17.0 DATA ACCEPTANCE CRITERIA

Calibration/QC Sample	Frequency	Acceptance Criteria	
Instrument Calibration Standard	Daily	Control chart ± 3 sigma	Recount twice, if not in control limits place detector on hold for maintenance
Instrument Calibration Blank	Daily	Control chart ± 3 sigma	Recount twice, if not in control limits, place detector on hold for maintenance
Laboratory Control Standard (LCS)	5%	80 – 120% or acceptance range by vender.	Reanalyze LCS and all associated samples
Duplicate	5%	≤ 1.0 RER < 20 RPD	If not in control limits, flag and report in case narrative

18.0 EQUIPMENT/INSTRUMENT MAINTENANCE & TROUBLESHOOTING

Maintenance is outlined in SOP GEN 17.

19.0 WASTE MANAGEMENT

Refer to SOP #GEN_19 (Hazardous Material Management) and SOP #GEN_20 (Waste Disposal) for laboratory waste management.

20.0 REFERENCES

20.1 Canberra Genie 2000 Software Operating Instructions.

20.2 Environmental Protection Agency (EPA) Method 901.1.

20.3 Los Alamos National Laboratory (LANL) Method WR140-5.

20.4 DOE HASL 300, 4.5.2.3 Ga-01-R.

20.5 ANSI N42.14-1991, Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides, American National Standards Institute, 1991.

20.6 DoD Quality Systems Manual Version 4.1, 04/22/09

21.0 ATTACHMENTS

21.1 Gamma Spec Run Log

21.2 Gamma Spec Data Checklist

ORGANIC SAMPLE PREP
TABLE OF CONTENTS

ORGANIC SAMPLE PREPARATION

1.0 PURPOSE

The method is used for the extraction, derivitization, clean-up and concentration of Total Petroleum Hydrocarbons (TPH), Diesel Range Organics (DRO), Herbicides, PCB's, Pesticides and Semivolatile Organic Compounds.

2.0 DETECTION LIMITS

Detection limits are found in the SOP for each analysis. The sample size used for extraction directly effects the detection limit.

3.0 SCOPE AND APPLICATION

This procedure is based on EPA 3510C, 3550B, 3620B, 3665A and 8151A methods. This method is designed to provide direction for preparation of water, sediment, and soil samples for extractable organic analyses.

4.0 SUMMARY OF METHOD

DIESEL RANGE ORGANICS (DRO)

4.1 A measured volume of aqueous sample or a measured weight of soil/sediment sample is extracted with methylene chloride. The methylene chloride extract is concentrated to a volume of 1.0 ml using a KD, turbo vap and/or nitrogen blowdown system.

HERBICIDES

4.2 Method 8151A provides extraction, derivatization and gas chromatographic conditions for the analysis of chlorinated acid herbicides. Water samples are extracted with diethyl ether and then esterified with diazomethane. Soil and waste samples are extracted and esterified with diazomethane. If herbicide esters are to be determined using this method, hydrolysis conditions for the esters in water and soil extracts are described.

PESTICIDES (8081a) and POLYCHLORINATED BIPHENYLS (PCB) 8082

4.3 A measured volume of aqueous sample or a measured weight of soil/sediment sample is extracted with methylene chloride. Aqueous samples are extracted at a neutral pH of $\geq$ 5 and $\leq$ 9 pH. The methylene chloride extract is concentrated to a volume of 20 ml and then converted to hexane by the addition of 50ml hexane and

concentrated to a volume of 1.0 ml using a KD/Turbo Vap and nitrogen blowdown system. Pesticides are cleaned up py passing through a florsil column.

SEMI-VOLATILE ORGANICS (8270C)

4.4 A measured volume of aqueous sample or a measured weight of soil/sediment sample is extracted with methylene chloride. Aqueous samples are extracted initially at a basic pH of $\geq$ 11 for base/neutral extractables and further extracted at $\leq$ 2 pH for acid extractables. The methylene chloride extract is concentrated to a volume of 1.0 ml.

5.0 DEFINITIONS

5.1 Accuracy – The closeness of agreement between an observed value and an acceptance reference value.

5.2 Batch – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

5.3 Bias – The deviation due to matrix effects of the measured value from a known spiked amount. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

5.4 Laboratory Control Sample (LCS) – A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

5.5 Matrix Spike (MS) – An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

5.6 Matrix Spike Duplicate (MSD) – Intralaboratory split sample spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. It is used to document the precision and bias of a method in a given sample matrix.

5.7 Method Blank (MB) – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

6.0 INTERFERENCES

6.1 Phthalate esters introduced during sample preparation can pose a major problem in organics determination. The phthalate esters are commonly found in plastics; therefore plastics are to be completely avoided in this procedure.

6.2 Unclean glassware causes interferences. Avoid it by:

- Detergent washing with hot water
- Multiple rinses with tap water followed by multiple rinses with deionized water
- Rinsing with methanol
- Baking of glassware in a 130°C oven

6.3 Matrix interferences may occur, especially without proper cleanup of the sample extract.

6.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCBs and organochlorine pesticides.

6.5 Organic acids, especially chlorinated acids, cause the most direct interferences with the herbicide determination. Phenols, including chlorophenols, may also interfere.

6.6 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware and glass wool must be acid rinsed, and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this possibility.

7.0 SAFETY PRECAUTIONS

7.1 Hazardous Chemicals/Equipment

All work shall be performed under a fume hood when working with the hazardous chemicals.

7.2 Radiological Hazards

When radioactive materials are capable of being volatilized or airborne, perform all work under a fume hood.

7.3 General Laboratory Safety

Refer to the Chemical Hygiene Plan, SOP#GEN_22 for general laboratory safety.

8.0 EQUIPMENT & SUPPLIES

8.1 Kuderna-Danish (K-D) Evaporative Concentrators with evaporative flasks, concentrator tips, and Snyder columns.

8.2 Boiling chips or beads

8.3 Water Bath - Capable of temperature control of 95 +/- 5^{o}C and kept in a fume hood.

8.4 Various syringes.

8.5 Various vials, 20ml, 1ml.

8.6 Fisher Q5 filter paper or equivalent.

8.7 Volumetric flasks, 10ml -1000 ml.

8.8 Analytical balance – capable of weighing 0.001 grams; Mettler PM 420, Sartorius b120s or equivalent.

8.9 Glass beakers – 50-600 ml.

8.10 Glass funnels – small or large.

8.11 Separatory funnels – 500-2L.

8.12 Sonicator ¾" probe with cabinet.

8.13 Centrifuge.

8.14 Centrifuge tubes – 50 ml glass disposable.

8.15 Pipets – pasture, glass disposable.

8.16 Beakers, 250 ml and 500 ml Pyrex.

8.17 pH paper.

8.18 Silica gel.

8.19 Florosil Columns – 500mg/4ml.

8.20 Filters – FisherBrand Q5, Whatman 41 or equivalent.

8.21 Turbo Vap.

8.22 Turbo Vap Tubes.

9.0 REAGENTS

All Reagents are ACS grade or equivalent.

9.1 Pesticide Grade Methylene chloride, CH_2Cl_2.

9.2 Pesticide Grade Hexane, C_6H_{14}.

9.3 Pesticide Grade 2-Propanol, $CH_3CH(OH)CH_3$.

9.4 ACS Grade Sulfuric acid, H_2SO_4. Mix 1 part H_2SO_4 and 1 part DI water.

9.5 Lauric Acid – reagent grade. Weight 10.0 g of lauric acid in a 500 ml volumetric flask. Add 50 ml hexane to the flask to dissolve the lauric acid, then dilute to 500 ml with hexane.

9.6 Potassium permanganate, $KMnO_4$, 5 % aqueous solution (w/v).

9.7 Acetone, HPLC grade.

9.8 Methanol, Double Distilled.

9.9 Diethylether, Pesticide grade.

9.10 KOH, 37% - Weigh 37g KOH pellets and mix with DI water to a final volume of 100 ml.

9.11 Diazald.

9.12 Silicic Acid.

9.13 K_2CO_3.

9.14 Carbitol.

9.15 Iso Octane.

9.16 Herbicide Standards, prepared mix from Ultra Scientific.

9.17 Anhydrous sodium sulfate, dried at 400^{o}C or pre-extracted with methlene chloride.

9.18 Organic free reagent water, ASTM Type II, (DI water) or better.

9.19 PCB Surrogate standards: 2,4,5,6-Tetrachlor-m-xylene and Decachloribiphenyl. Prepared mix, 200 ug/ml ISM-320-1 Ultra Scientific or equivalent.

9.20 Aroclors 1016 and 1260 are used for the matrix spike for PCB's. Prepared mix from Ultra or equivalent.

9.21 Internal Standards - 1,4-Dichlorobenzene-d_4, Napthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, Perylene-d_{12}. An internal standard solution can be purchased at a concentration of 4000 ng/ul in methylene chloride. A 10 uL portion of this solution should be added to each 1 ml of sample extract. This will give a concentration of 40 ng/uL of each constituent when 1ul is injected. (Ultra Scientific # US-108N or equivalent)

9.22 BNA surrogate spiking solution is prepared from stock standards received from commercial suppliers. Base-Neutral compounds are prepared at a concentration level of 100 ug/ml. Acid compounds are prepared at a concentration level of 200 ug/ml. (Ultra Scientific # ISM-333X or equivalent).

9.23 BNA Matrix Spiking Solutions are prepared using commercially available certified stock standards. Prepare at a concentration of 100 ug/ml for Base Neutral compounds and 200 ug/ml for Acid compounds. This standard can also be purchased from Ultra ready to use. (Ultra Scientific # CLP-351X or equivalent).

9.24 Semivolatile Tuning Standard, containing Decafluorotriphenylphosphine (DFTPP), Benzidine, Pentachlorophenol, and 4,4'-DDT. Prepare from stock standard at a concentration of 50 ug/ml. (Ultra Scientific # GCM-150-1 or equivalent).

9.25 Anhydrous Sodium sulfate, NA_2SO_4 – acid washed and pre-extracted ether.

9.26 10N NaOH: Dissolve 40 grams NaOH in organic free reagent water and dilute to 100 ml.

9.27 6N NaOH – Dissolve 24g NaOH in 0orgajic free reagent water

9.28 Methanol, CH_3OH – Pesticide grade.

9.29 Acetone, CH_3COCH_3 – Pesticide grade.

9.30 Phosphate Buffer (0.1 M), pH 2.5. Dissolve 12 g NaH_2PO_4 in Deionized water and dilute to 1 L. Add H_3PO_4 to adjust pH to 2.5.

9.31 Acidified Na_2SO_4 – Weigh 100g of Na_2SO_4 and mix with di-ethyl ether until slurry. Add H_2SO_4 until color indicating pH paper reads <2.

9.32 Sodium Hydroxide- Weigh out 20 g of NaOH (pellets, reagent grade) in a 500 ml volumetric flask. Dissolve in organic-free reagent water and dilute to 50 ml to make a 1N solution. Dilute 25 ml of the 1 N NaOH to 500 ml with water in a second 500 ml volumetric flask, yielding a 0.05N solution. The NaOH solution must be standardized against lauric acid as follows:

9.32.1 Weigh 100-200 mg of lauric acid to the nearest 1 mg in a 125 ml Erlenmeyer flask. Add 50 ml of ethanol to the flask and swirl to dissolve the lauric acid.

9.32.2 Add 3 drops of phenolphthalein indicator to the flask and titrate with the 0.05 N NaOH solution to a permanent endpoint (i.e., the indicator color does not disappear when the solution is allowed to stand for 1 minute).

9.32.3 Calculate the "strength" of the NaOH solution as the mg of lauric acid neutralized per ml of NaOH solution.

10.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

10.1 Containers used to collect samples for determination of semi-volatiles are purchased and certified from vendor to be organic free. (QEC or equivalent).

10.2 Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination.

10.3 Samples and extracts must be shipped and stored under refrigeration of 0-4º C.

10.4 Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

11.0 QUALITY CONTROL

11.1 With every batch of samples a MB, LCS, MS and MSD will be prepared and analyzed. The control limits will be determined by the use of control charts.

11.2 Nonconformances may occur at any level. Some may be corrected immediately and documented using normal laboratory document procedures. Out-of-control nonconformances shall be documented by the use of a Corrective Action Report, usually issued by the QAO. With the use of a CAR, findings can be tracked and used to detect future trends.

11.3 Work shall be stopped when out-of-control deficiencies occur until the problem is alleviated, (i.e.: instrumentation malfunction, QC out of control limits, failure to perform demonstrations of capabilities, etc.).

12.0 PROCEDURE

12.1 DRO WATER EXTRACTION

12.1.1 Add 1000 ml of sample to a separatory funnel containing 60 ml of methylene chloride. If 1000 ml is not used, record volume used.

12.1.2 Add 10uL of surrogate to sample and shake vigorously for two minutes, venting the pressure as needed.

12.1.3 Allow the layers to separate and drain the methylene chloride through approximately 10g of sodium sulfate and into a Kuderna-Danish apparatus or Turbo Vap tube.

12.1.4 Repeat the procedure two more times.

12.1.5 Concentrate the methylene chloride until an apparent volume of 10 ml's has been reached.

12.1.6 Use nitrogen blow down to reduce the volume to less than one mL, and accurately bring up to one mL using a syringe.

12.1.7 Transfer sample to vial and label with analysis run number, sample ID, prep date and initials.

12.1.8 Place vial in sample extracts refrigerator.

12.2 DRO SOLID EXTRACTION

12.2.1 Place 5.0g of the sample in a 20ml scintillation vial.

12.2.2 Add 5g $NaSO_4$ and shake.

12.2.3 Add 10ul of surrogate.

12.2.4 Add 10.0 ml of methylene chloride and shake.

12.2.5 Sonicate for 3 minutes and let separate.

12.2.6 Filter the extract and place in a concentration tube.

12.2.7 Repeat steps 12.2.4 through 12.2.6.

12.2.8 Concentrate sample to 1ml using nitrogen blowdown system.

12.2.9 Transfer sample to vial and label with analysis run number, sample ID, prep date and initials.

12.2.10 Place vial in sample extracts refrigerator.

12.3 HERBICIDES WATER EXTRACTION

12.3.1 Using a 1-liter graduated cylinder, measure up to 1-liter (nominal) of sample.

12.3.2 Record the sample volume to the nearest 5-ml, and transfer it to the separatory funnel or 600 mL beaker.

NOTE: If high concentrations are anticipated, a smaller volume may be used.

12.3.3 Add 25-g of sodium chloride for every 100 ml of sample and shake or stir to dissolve the salt.

Hydrolysis

12.3.4 Add 17-ml of 6N NaOH to the sample and shake or stir to adjust the pH above 12.

12.3.5 Let the sample stand at room temperature for 2 hours. If not in a seperatory funnel, transfer to one.

12.3.6 Extract the sample with 3 separate 60-ml portions of methylene chloride. (Discard the lower methylene chloride phase).

12.3.7 Use sulfuric acid (1:1) and pH indicating paper to adjust pH to <2.

12.3.8 Add 120-ml of diethyl ether to the separatory funnel and extract the sample by shaking the funnel for 2 minutes with frequent venting to release excess pressure.

NOTE: Pressure could be very extreme so be very careful venting.

12.3.9 Allow the organic layer to separate from the water layer for a minimum of 10 minutes.

NOTE: If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must centrifuge the sample to complete the phase separation.

12.3.10 Drain the lower aqueous phase into original sample container.

12.3.11 Collect the ether extract in a 600mL beaker containing 10g of acidified Na_2SO_4.

12.3.12 Repeat the extraction with 2 60-ml portions of diethyl ether, combining all portions.

12.3.13 Cover beaker with aluminum foil and allow the extract to stay in contact with the acidified sodium sulfate for at least two hours to ensure complete drying.

Sample Concentration

12.3.14 Transfer the ether extract, through a Q5 filter paper containing 10g of acidified Na_2SO_4 into a 250 mL Turbo Vap tube or 500 mL KD flask equipped with a 10-ml concentrator tube.

12.3.15 Use a glass rod to crush caked Na_2SO_4 during the transfer.

12.3.16 Rinse the beaker and funnel with 30-ml of diethyl ether to complete the quantitative transfer.

12.3.17 Add one or two boiling chips to the tube or flask. If using the KD attach a three ball Snyder column.

KD PREP

12.3.18 Pre-wet the Snyder column by adding about 1 mL of diethyl ether to the top.

12.3.19 Place the apparatus in a hot water bath (60°-65°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor.

12.3.20 Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 15-20 minutes

12.3.21 When the apparent volume of liquid reaches 5-ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

12.3.22 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2-ml of diethyl ether. (A 1-ml syringe is recommended for this operation). Proceed to 12.3.23.

Turbo Vap Prep

12.3.23 Place Turbo Vap tube in Turbo Vap. Set temperature to 60°C.

12.3.24 When sample has evaporated to approximately 5-10 mL, remove tube.

12.3.25 Allow to cool and transfer to a concentrator tube.

12.3.26 Use nitrogen blow down to reduce the volume to an apparent volume of 1-ml.

12.3.27 Rinse the walls of the concentrator tube with diethyl ether.

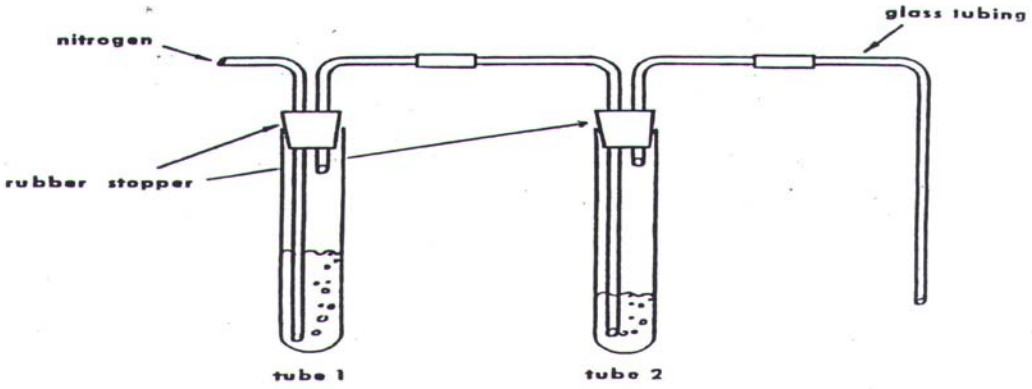
12.3.28 Add to the extract with 1 ml iso octane and 0.5 ml methanol.

12.3.29 Dilute to a final volume of 4 ml with diethyl ether.

Esterification – Diazomethane Derivatization

NOTE: Diazomethane is a carcinogen and can explode under certain conditions.

12.3.30 Assemble the diazomethane bubbler.



12.3.31 Add diethyl ether to the first tube.

12.3.32 Add 1 ml of diethyl ether, 1 ml of carbitol, 1.5 ml of 37% KOH, and 0.1-0.2 g of Diazald to the second tube.

12.3.33 Immediately place the exit tube into the sample extract.

12.3.34 Apply nitrogen flow at a rate of 10 mL per minute and bubble diazomethane through the extract for ten minutes, or until the yellow color of diazomethane persists.

12.3.35 Remove the extract, seal it and let stand for 20 minutes in the fume hood.

12.3.36 Destroy any unreacted diazomethane by adding 0.1-0.2 g of silicic acid to the extract.

12.3.37 Let stand until the evolution of nitrogen gas has stopped.

12.3.38 Adjust the sample extract volume to 10 ml with hexane.

12.3.39 Transfer sample to 25 mL scintillation vial and label with analysis run number, sample ID, prep date and initials.

12.3.40 Place vial in sample extracts refrigerator.

12.4 HERBICIDES SOLID EXTRACTION

12.4.1 To a 400-ml, thick walled beaker or 16 oz sample jar, add 30-g (dry weight) of the well-mixed sample.

12.4.2 Add 85 ml of phosphate buffer and 100 ul of surrogate. Adjust the pH to 2 with concentrated HCl and monitor the pH for 15 minutes with occasional stirring. If necessary, add additional HCl until the pH remains at 2.

12.4.3 Add 10g sodium sulfate and mix so the sample is free flowing.

12.4.4 Add 100-ml acetone/methylene chloride (1:1) to the beaker and sonicate for 3 minutes.

12.4.5 Decant and filter through a Q5 filter containing 10-g of acidified sodium sulfate into an Erlenmeyer containing an additional 10-g of acidified sodium sulfate.

12.4.6 Repeat the sonication 2 more times using 100ml of methylene chloride combining all three organic extracts.

12.4.7 The extract must dry over the sodium sulfate for at least two hours.

12.4.8 Quantitatively transfer the extract into a K-D and reduce the volume to around 5ml and allow cooling.

Hydrolysis

Use this step only if herbicide esters in addition to herbicide acids are to be determined.

12.4.9 Add 30-ml of organic-free reagent water, 5-ml of 37% KOH and one or two clean boiling chips to the KD flask.

12.4.10 Place a three ball Snyder column on the flask, evaporate the diethyl ether on a water bath, and continue to heat for a total of 90 minutes.

12.4.11 Transfer the aqueous solution to a separatory funnel and extract three times with 100 ml of methylene chloride. Discard the methylene chloride phase.

12.4.12 Adjust the pH to <2 with cold 1:1 sulfuric acid and extract once with 40-ml diethyl ether and then twice with 20-ml. Collect in 250-500 mL Erlenmyer Flask.

12.4.13 Dry over acidified sodium sulfate for at least 2 hours.

12.4.14 K-D down to around 5-mls. Concentrate using steps 12.3.25.

12.5 PCB WATER EXTRACTION

12.5.1 Add between 250mL and 1L of sample into a 1 or 2-liter separatory funnel. Record the volume of sample used on the extraction log.

12.5.2 Add 5mL of surrogate standard solution to all samples, method blanks, laboratory control samples (LCS), and 1 ppb to matrix spikes.

12.5.3 Add 5ul of matrix spike solution to the LCS, matrix spike and the matrix spike duplicate samples.

12.5.4 Add 60 ml methylene chloride to the separatory funnel and extract the sample by shaking the funnel for two minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes.

NOTE: If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must centrifuge the sample to complete the phase separation.

12.5.5 Drain Methylene Chloride into a 600 mL beaker containing 10g of pre-extracted $NaSO_4$.

12.5.6 Perform two more serial extractions with 60 ml of methylene chloride.

12.5.7 Dry the extracts over pre-extracted $NaSO_4$ for 30 minutes

Concentration

12.5.8 Add 1-2 clean boiling chips and attach a 3-ball Snyder column. Wet the column with 1-2 ml of hexane and place assembly into a 85°C water bath and concentrate to an apparent volume of 20 ml.

12.5.9 In all aqueous extractions and in soil/sediment extractions where methylene chloride-acetone is used, exchange the 20 ml extract to hexane by adding 50 ml hexane to the K-D apparatus. Adjust volume to 10 ml in the concentrator tube. Transfer to a labeled scintillation vial.

12.5.10 Filter through a #40 filter containing 10g Na_2SO_4 into a Kd apparatus.

Extraction Clean Up

12.5.11 See PCB Solid Extraction Clean Up Section, Section 12.6.11-12.6.22.

12.6 PCB SOLID EXTRACTION

12.6.1 Weigh between 5-20 g of sample to the nearest 0.1 gram into a 20ul scintillation vial and add 5 g of pre-extracted anhydrous sodium sulfate. Mix well with a glass rod.

NOTE: The sample should be free-flowing.

12.6.2 Add 5ul surrogate to the blank, LCS, samples, and matrix spikes.

12.6.3 Add 5ul matrix spike to the LCS and the matrix spike and matrix spike duplicate.

12.6.4 Immediately add 5 ml hexane and 5 ml acetone.

NOTE: Hexane-acetone (1:1) may be more affective as an extraction solvent for organochlorine pesticides in some environmental and waste matrices. Hexane-acetone (1:1) generally reduces the amount of interferences that are extracted and improve signal-to-noise.

12.6.5 Sonicate the samples using a 1/2" probe held about 1/2" below the liquid surface but above the sediment level. Use full power and a 50% pulse for 3 minutes.

12.6.6 Decant and filter the solvent layer through a glass funnel with filter paper filled with approximately 3-5 grams of pre-extracted sodium sulfate. Collect the extract in a 50 ml centrifuge tube or Turbo Vap tube.

12.6.7 Repeat this procedure two more times with 10 ml of hexane-acetone (1:1). Decant off the solvent after each sonication, pouring the entire sample into the funnel the last time.

12.6.8 Rinse the beaker and soil/sediment with approxiamtely 10 mls more extraction solvent. Proceed to "Concentration" Section.

Concentration

12.6.9 Add 1-2 clean boiling chips and attach a 3-ball Snyder column. Wet the column with 1-2 ml of hexane and place assembly into a 85°C water bath and concentrate to an apparent volume of 20 ml.

12.6.10 In all aqueous extractions and in soil/sediment extractions where methylene chloride-acetone is used, exchange the 20 ml extract to hexane by adding 50 ml hexane to the K-D apparatus. Adjust volume to 10 ml in the concentrator tube. Transfer to a labeled scintillation vial. Proceed to Extract Cleanup.

Extract CleanUp - Sulfuric acid: Method 3665A

12.6.11 Transfer 2 ml of hexane extract into a vial. Add 5ml of 1:1 H_2SO_4 solution.

12.6.12 Cap vial and vortex for 1 min.

12.6.13 Allow the layers to separate for 1 min. Examine the hexane layer. If it is colorless, proceed to 12.6.16.

12.6.14 If the hexane layer is colored, discard the 1:1 H_2SO_4 layer and repeat cleanup using another 5 ml aliquot of 1:1 H_2SO_4 solution.

Note: If an emulsion persists, centrifuge to separate and discard 1:1 $H2SO_4$ layer and repeat cleanup using another 5ml aliquot of 1:1 H_2SO_4.

12.6.15 Transfer the hexane layer to a clean vial. Add 1 ml hexane to the 1:1 H_2SO_4 layer, cap and shake. This is done to ensure quantitative transfer of the PCBs.

12.6.16 Combine the second hexane layer with the first. Proceed with Permanganate Cleanup or Final Preparation.

Extract CleanUp - Permanganate cleanup

12.6.17 Transfer 2 ml of hexane extract into a vial. Add 5 ml of 5% $KMnO_4$ solution.

12.6.18 Cap vial and vortex for 1 min.

12.6.19 Allow the layers to separate for 1 min. Examine the hexane layer. If it is colorless, proceed to Final Preparation step.

12.6.20 If the hexane layer is colored, discard the 5% $KMnO_4$ layer and repeat cleanup using another 5 ml aliquot of 5% $KMnO_4$ solution.

Note: If an emulsion persists, centrifuge to separate and discard 5% $KMnO_4$ layer and repeat cleanup using another 5ml aliquot of 5% $KMnO_4$.

12.6.21 Transfer the hexane layer to a clean vial. Add 2 ml hexane to the 5% $KMnO_4$ layer, cap and shake. This is done to ensure quantitative transfer of the PCBs.

12.6.22 Combine the second hexane layer with the first. Proceed with final preparation.

Final preparation:

12.6.23 Reduce the volume of the combined extract layers to the original volume using the nitrogen evaporator.

12.6.24 Transfer the extract to an autosampler vial. The extract is now ready for analysis.

12.7 PESTICIDES WATER EXTRACTION

12.7.1 Add between 250mL and 1L of sample into a 1 or 2-liter separatory funnel. Record the volume of sample used on the extraction log.

12.7.2 Add 5ul of surrogate standard solution to all samples, method blanks, laboratory control samples (LCS), and 1 ppb to matrix spikes.

12.7.3 Add 5ul of matrix spike solution to the LCS, matrix spike and the matrix spike duplicate samples.

12.7.4 Add 60 ml methylene chloride to the separatory funnel and extract the sample by shaking the funnel for two minutes, with periodic venting to

release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes.

NOTE: If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation or other physical means.

12.7.5 Drain Methylene Chloride into a 600 mL beaker containing 10g of pre-extracted $NaSO_4$. Perform two more serial extractions with 60 ml of methylene chloride. Dry the extracts over pre-extracted $NaSO_4$ for 30 minutes.

Concentration

12.7.6 Filter through a Q5 filter containing 10g Na_2SO_4 into a Kd apparatus or turbo vap tube.

12.7.7 Add 1-2 clean boiling chips and attach a 3-ball Snyder column. If using a KD, wet the three ball snyder column with 1-2 ml of hexane. Place assembly into Turbo Vap or 85°C water bath and concentrate to an apparent volume of 20 ml.

12.7.8 In all aqueous extractions and in soil/sediment extractions where methylene chloride-acetone is used, exchange the 20 ml extract to hexane by adding 50 ml hexane to the Turbo Vap tube or K-D apparatus. Adjust volume to 10 ml in the concentrator tube. Transfer to a labeled scintillation vial. Proceed to clean up.

Extraction Clean Up

12.7.9 See Florisil Clean Up Section 12.8.12.

12.8 PESTICIDE SOLID EXTRACTION

12.8.1 Weigh between 5-20 g of sample to the nearest 0.1 gram into a 20ul scintillation vial or appropriate sized sample jar and add 5-20 g of pre-extracted anhydrous sodium sulfate. Mix well with a glass rod.

NOTE: The sample should be free-flowing.

12.8.2 Add 5 ul surrogate to the blank, LCS, samples, and matrix spikes.

12.8.3 Add 5 ul matrix spike to the LCS and the matrix spike and matrix spike duplicate.

12.8.4 Immediately add 10 ml hexane-acetone, per 5 grams of sample.

NOTE: Hexane-acetone (1:1) may be more affective as an extraction solvent for organochlorine pesticides in some environmental and waste matrices. Hexane-acetone (1:1) generally reduces the amount of interferences that are extracted and improve signal-to-noise.

12.8.5 Sonicate the samples using a 1/2" probe held about 1/2" below the liquid surface but above the sediment level. Use full power and a 50% pulse for 3 minutes.

12.8.6 Decant and filter the solvent layer through a glass funnel with filter paper filled with approximately 3-5 grams of pre-extracted sodium sulfate. Collect the extract in a turbovap tube.

12.8.7 Repeat this procedure two more times with 10 ml or appropriate amount of hexane-acetone (1:1). Decant off the solvent after each sonication, pouring the entire sample into the funnel the last time.

12.8.8 Rinse the beaker and soil/sediment with approxiamtely 10 mls more extraction solvent. Proceed to “Concentration” Section.

Concentration

12.8.9 Filter through a Q5 filter containing 10g Na_2SO_4 into a Kd apparatus or turbo vap tube.

12.8.10 Add 1-2 clean boiling chips and attach a 3-ball Snyder column. If using a KD, wet the three ball snyder column with 1-2 ml of hexane. Place assembly into Turbo Vap or 85°C water bath and concentrate to an apparent volume of 20 ml.

12.8.11 In all aqueous extractions and in soil/sediment extractions where methylene chloride-acetone is used, exchange the 20 ml extract to hexane by adding 50 ml hexane to the Turbo Vap tube or K-D apparatus. Adjust volume to 10 ml in the concentrator tube. Transfer to a labeled scintillation vial.

Extract Cleanup - Florisil Cleanup Method 3620B

12.8.12 Florisil cleanup is required for all extracts including all associated QC samples. Cleanup significantly reduces matrix interferences caused by polar compounds.

12.8.13 Deactiviation of Florisil - To prepare for use, place 100 +/- g of Florisil into a 500 ml beaker and heat to 140^{o} C for approximately 16 hours. After heating, transfer to a 500 ml reagent bottle. Tightly seal and cool to room temperature. When cool, add 3 +/- 0.1 ml of organic-free reagent water. Mix thoroughly by shaking or rolling for 10 minutes and let stand for at least 2 hours. Keep the bottle sealed tightly.

12.8.14 Activation of Florisil – for all cleanups other than phthalate esters. It is advisable to treat both Florisil A and Florisil PR prior to use to drive off any moisture adsorbed during storage and handling. Heat the Florisil in a glass container loosely covered with aluminum foil in an oven at 130^{o} C overnight. Cool the Florisil in a dessicator before use.

12.8.15 Florisil from different batches or sources may vary in adsorptive capacity. To standardize the amount of Florisil which is used, use the lauric acid value described below. The procedure determines the adsorption from a hexane solution of lauric acid (mg) per g of Florisil.

12.8.15.1 Weigh 2.000 g of Florisil in a 25 ml glass-stoppered Erlenmeyer flask. Cover loosely with aluminum foil and heat overnight at 130^{o} C. Stopper flask and cool to room temperature.

12.8.15.2 Add 20.0 ml of the lauric acid solution to the flask, stopper, and shake occasionally for 15 minutes.

12.8.15.3 Let the Florisil settle and using a volumetric pipette, transfer 10.0 ml of supernatant liquid into a 125 ml Erlenmeyer flask. Avoid inclusion of any Florisil.

12.8.15.4 Add 60 ml of ethanol and 3 drops of the phenolphthalein indicator solution to the flask.

12.8.15.5 Titrate the solution in the flask with the 0.05N NaOH solution until a permanent end point is reached (i.e., the indicator color does not disappear when the solution is allowed to stand for 1 minute).

12.8.15.6 The lauric acid value is calculated as follows:

Lauric acid value = 200 – (titration vol in ml of NaOH) (strength of NaOH)

Where the strength of the NaOH is measured in Section 9.32 as the mg of lauric acid neutralized per ml of NaOH solution.

12.8.15.7 Use the following equation to obtain an equivalent quantity of any batch of Florisil

$$\frac{\text{Lauric acid value}}{110} \times 20\ \text{g} = \text{required weight of Florisil}$$

12.8.16 Assemble the florisil cartridge and add 1 g of activated Florisil and condition with hexane. Pour the extract into the florisil cartridge and collect the extract in a 20 ml labeled scintillation vial. Perform the following washes:

1. Add 3 ml hexane to cartridge and collect.
2. Add 5 ml methylene chloride/hexane(26/74) mix to cartridge and collect.
3. Add 5 ml acetone/hexane(10/90) mix to cartridge and collect.

12.8.17 Concentrate the extract via a nitrogen blowdown apparatus to 1 ml for GC analysis.

12.9 SEMI VOA WATER EXTRACTION

12.9.1 Make sure enough sample is retained for pH determination.

12.9.2 Measure 250 to 1000 ml of aqueous sample into either a 1 L separatory funnel. (See Note 2.) Record volume on the extraction data log.

12.9.3 All solutions are pH adjusted to >11 (with 10N sodium hydroxide), checking the pH with pH paper.

NOTE: If pesticides are to be determined by this method, it is recommended that the sample first be extracted under neutral conditions prior to adding NaOH to prevent decomposition of target analytes.

12.9.4 Add 0.5 ml BNA surrogate spiking solution to all samples including QC samples.

12.9.5 Add 0.5 ml of BNA matrix standard spiking solution to the LCS, matrix spike and matrix duplicate samples.

12.9.6 Record the volume and I.D. of spiking solutions in the Organic Prep logbook at this time. To ensure spiking solutions are added, mark extraction vessels with a check mark for each spiking solution added.

12.9.7 The inital extraction is performed using a 60 ml portion of methylene chloride where the funnel is shaken for two minutes (with periodic venting) and is allowed to rest for a minimum of 10 minutes to allow the two phases to separate.

12.9.8 Perform this serial extraction two more times, collecting the extracts in a labeled 600 ml beaker containing 10g Na_2SO_4.

NOTE: If an emulsion interface between the layers forms and cannot be broken by the use of physical means (i.e., stirring, filtration of the emulsion through glass wool, centrifugation or other physical methods) the sample should be extracted in a continuous L-L extractor or smaller sample sized used.

12.9.9 Adjust the pH of the aqueous phase to less than 2 using (1:1) sulfuric acid.

12.9.10 Again serially extract three times with 60 ml portions of methylene chloride, filtering and collecting the combined extracts in the 600 ml beaker used for collecting BN. Proceed to Concentration, Section 12.10.13.

12.10 SEMIVOLATILE SOLID EXTRACTION (LOW LEVEL)

12.10.1 If the pH is less than 4.5, report it to the supervisor. At this time, a moisture determination should be performed since concentrations of individual analytes will be reported relative to the dry weight of sediment. Also at this time, the pH should be determined.

12.10.2 Weigh 30 grams of the sample to the nearest 0.1 gram into one 16 oz sample jar and add 60 grams of pre-extracted anhydrous sodium sulfate, mixing well with a glass rod.

NOTE: Sample should be free-flowing.

12.10.3 Add 0.5 ul BNA surrogate spiking solution to all samples including QC samples.

12.10.4 Add 0.5 ul of BNA matrix standard spiking solution to the matrix spike and matrix spike duplicate samples.

12.10.5 Immediately add 100 ml of 1:1 methylene chloride-acetone.

12.10.6 Record the volume and spiking I.D. in the Organic Prep logbook at this time.

12.10.7 Sonicate the samples one at a time using the 1/2" probe held 1/2" below the liquid surface but 1/2" above the sediment level. Use full power and 50% pulse for 3 minutes.

12.10.8 Decant and filter the extracts through a filter paper in a glass funnel containing a filter with 3-5 grams Na_2SO_4, collecting the extracts in a 600 ml beaker.

12.10.9 Repeat the extraction two more times with two more 100 ml portions of the 1:1 methylene chloride-acetone.

12.10.10 Decant the solvent after each sonication, pouring the entire sample into the funnel the last time.

12.10.11 Rinse the vial and soil/sediment with about 30 ml more extraction solution.

12.10.12 Collect all extract portions in a 600 ml beaker. Proceed to Concentration.

Concentration

12.10.13 Add 1-2 clean boiling chips to a Turbo Vap tube or the 500 ml KD evaporative flask and attach a 3-ball Snyder column. Pre-wet it with about 1 ml of methylene chloride.

12.10.14 Put the concentration in a Turbo Vap or hot water bath (about 75°C) so that the concentrator tube is partially immersed in the water and the entire lower rounded surface is bathed with hot vapor. If using KD, the balls of the column should actively chatter and the chambers should not flood with condensed solvent.

12.10.15 When the apparent volume of liquid reaches 10 ml, remove from water bath and allow the apparatus to drain and cool for at least 10 minutes.

12.10.16 Remove the column and rinse the flask and lower joint into the concentrator tube with 1-2 ml of methylene chloride. If using a turbo vap transfer to a 10 mL concentrator tube.

12.10.17 Place the concentrator tube on the nitrogen blowdown apparatus and evaporate the solvent volume to just below 1 ml with rinsing down of the

inside wall of the tube several times with about 1 ml of methylene chloride.

12.10.18 The final volume is brought to 1 ml with methylene chloride.

NOTE: During evaporation, the tube must never be allowed to become dry. When volume of solvent extract is reduced below 1 ml, semi-volatile analytes may be lost.

12.10.19 Transfer these 1 ml extracts to the appropriate vials labeled for base neutral/acid fractions. Also include on the label the date and beginning amount of sample and the final volume.

12.10.20 All data is to be recorded in the Organic Prep logbook.

12.11 SEMIVOLATILE SOLID EXTRACTION (MEDIUM/HIGH LEVEL)

12.11.1 At this time, a moisture determination should be performed since concentrations of individual analytes will be reported relative to the dry weight of sediment. Also at this time, the pH should be determined.

12.11.2 In a fume hood, open the sample container and mix the sample, discarding any foreign objects such as sticks, leaves, and rocks.

12.11.3 Transfer 5 grams (record to the nearest 0.1 g) of sample to a 20 ml vial.

12.11.4 Add 0.5 ml of BNA surrogate spiking standard to each vial.

12.11.5 Add 0.5 ml of matrix standard spiking solution to the LCS and MS/MSD sample vials.

12.11.6 Add 5 grams pre-extracted sodium sulfate to each vial.

12.11.7 Prepare a blank using reagents used in sample extraction.

12.11.8 Immediately add 10 ml methylene chloride to the blank and sample(s).

12.11.9 Add 10 ml methylene chloride to the matrix and matrix duplicate.

12.11.10 Disrupt the sample by ultrasonic probe for 3 minutes at power output setting at 5 and 50% pulse.

12.11.11 Concentrate to 1 ml on the nitrogen blowdown apparatus.

12.11.12 Transfer the 1 ml to a sample vial. Label this with Outreach Laboratory Lab #, volume/weight, initials and date.

12.12 PERCENT MOISTURE

Refer to Outreach SOP for Loss on Drying WET-03 .

13.0 METHOD PERFORMANCE

These methods have been valadated by the USEPA.

14.0 POLLUTION CONTROL

Refer to the Laboratory Chemical Hygiene Plan, SOP# GEN-22 for laboratory pollution control.

15.0 WASTE MANAGEMENT

Refer to SOP#GEN_19 (Hazardous Material Management) and SOP#GEN_20 (Waste Disposal) for laboratory waste maangement.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Methods 3500B/3510C/3520C/3550B/8151A.

16.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), CleanUp Methods 3620B, 3660B, 3665A,8151A.

16.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Derivitization Method, 8151A.

METALS SAMPLE PREPARATION
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METALS SAMPLE PREPARATION

1.0 SCOPE AND APPLICATION

Samples that are to be analyzed by Inductively Coupled Argon Plasma must be digested prior to analysis. This standard operating procedure is applicable to the digestion of soils, solids, waters, wastewaters, and leachates. This procedure is also applicable to the digestion of cryolite and like matrices by fusion.

2.0 SUMMARY

2.1 For water samples, 50ml of sample is digested using Nitric acid and/or Hydrochloric acid, and the final volume is 50ml.

2.2 For solid samples, 1.0g is digested using Nitric acid and/or Hydrochloric acid and/or hydrogen peroxide. The final volume is 100ml.

2.3 For cryolite or like samples in a wet matrix, percent solids must be evaluated and the wet weight increased appropriately. Potassium pyrosulfate is added in conjunction with furnace baking and the sample is then further digested using 1:1 Hydrochloric acid.

2.4 For cryolite or like samples in a dry matrix, weigh 0.5 g of sample. Potassium pyrosulfate is added in conjunction with furnace baking and the sample is then further digested using 1:1 Hydrochloric acid.

3.0 DEFINITIONS

3.1 Accuracy – The closeness of agreement between an observed value and an accepted reference value.

3.2 Batch – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

3.3 Laboratory Control Sample – A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

3.4 Matrix Spike – An aliquot of sample spiked with a known concentration of target analytes(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

3.5 Matrix Spike Duplicates – Intralaboratory split samples spiked with identical concentrations of target analyte(s).

3.6 Method Blank – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.0 INTERFERENCES

4.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge.

4.2 Various interferences are possible and the appropriate analytical method discusses the potential interferences.

5.0 SAFETY PRECAUTIONS

5.1 Hazardous Chemicals/Equipment

All work shall be performed under a fume hood when working with the hazardous chemicals.

5.2 Radiological Hazards

When radioactive materials are capable of being volatilized or airborne, perform all work under a fume hood.

5.3 General Laboratory Safety

Refer to the Laboratory Chemical Hygiene Plan, SOP #GEN_22, for general laboratory safety.

6.0 EQUIPMENT & SUPPLIES

6.1 Analytical Balance; Mettler PC 4400 or equivalent.

6.2 Hot plate; Presto 0703205 or equivalent.

6.3 Porcelain crucibles; Coors 60105 or equivalent.

6.4 Watch Glasses; Pyrex 3 inch fluted or equivalent.

6.5 Muffle Furnace; Thermolyne 1400 or equivalent.

6.6 Automatic pipette; Rainin EPD2 or equivalent.

6.7 200 ml and 100 ml glass beakers; Pyrex 1040 or equivalent.

6.8 Volumetric flasks; Kimax 10 ml or equivalent.

6.9 Snap cap vials (100 ml); Fisher 0334175G or equivalent.

6.10 Disposal plastic beakers; 100 ml.

6.11 Volumetric flasks; 100 ml and 50 ml class "A" or equivalent.

6.12 Graduated cylinder; EXAX 20040 or equivalent class “A” , calibrated to 50 mls.

6.13 50 ml disposable centrifuge tubes, or equivalent.

7.0 REAGENTS

7.1 Spiking Solutions, made from ICP Stock Standard Solutions, N.I.S.T. Traceable.
QC26 –CPI international –100 mg/l
QC4 – SCP Science – 100 mg/l
Individual Standards – SCP Science and CPI
Interference Check Standard – SCP Science.

7.2 Nitric acid conc. and 8N HNO_3, ACS Grade.

7.3 Hydrochloric acid conc. and 6 N HCl, ACS Grade.

7.4 30% Hydrogen peroxide, ACS Grade.

7.5 Distilled water

7.6 Potassium pyrosulfate; Fisher P281-500 or equivalent.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 All samples are to be collected in a prewashed container.

8.2 Aqueous samples to be analyzed for Total metals are to be preserved with nitric acid to a pH of < 2, and should be analyzed within 6 months of collection. Aqueous samples to be analyzed for dissolved metals are to be filtered on site and preserved with nitric acid to a pH of < 2, and should be analyzed within 6 months of collection. Aqueous samples to be analyzed for Suspended metals are to be filtered on site, and should be analyzed within 6 months of collection. Solid samples are not to be preserved and are to be analyzed within 6 months of collection.

8.3 All samples should be shipped and stored under refrigeration of 0-4ºC.

9.0 QUALITY CONTROL

9.1 A Preparatory Method Blank (MB) is digested with every batch of samples digested to ensure that no contamination has occurred from the digestion process.

9.2 With every batch of samples a Laboratory Control Sample (LCS) at 1.0 mg/l is digested and analyzed . The control limits for the LCS are 80-120% recovery. For 200.7 the recovery requirements are 85-115.

9.3 With every batch of samples a Matrix Spike Sample (MS) and a Matrix Spike Duplicate (MSD) is digested and analyzed. The control limits are determined by control charts maintained by the laboratory.

9.4 Nonconformances may occur at any level. Some may be corrected immediately and documented using normal laboratory document procedures. Out-of-control nonconformances shall be documented by the use of a Corrective Action Report, usually issued by the QAO. With the use of a CAR, findings can be tracked and used to detect future trends.

9.5 Work shall be stopped when out-of-control deficiencies occur until the problem is alleviated, ie., instrumentation malfunction, QC out of control limits, failure to perform demonstrations of capabilities, etc.

10.0 PROCEDURE

All sample preparation information is to be recorded in the Metals Sample Prep Log, including initial volume or weight, spike added, final volume, hot plate temperature, and the method number along with the date and the analyst's initials.

10.1 Water Sample Digestion for ICP Analysis (EPA SW 846 3010A)

10.1.1 Measure a 50 ml aliquot of sample in a Class A graduated cylinder and transfer the sample into a 100 ml beaker. If silica is requested, use 100 ml disposable beaker. For LCS and MS/MSD, add 1.0 mL of 100 mg/l QC26 Standard use for ICV and appropriate amounts of individual standards to make a final concentration of 1.0 mg/l.

10.1.2 Add 1.5 ml of concentrated nitric acid, cover and place on the hot plate set at 95^{o}C being careful not to boil the sample.

10.1.3 When the sample reaches a volume of around 20ml, add another 1.5 ml of concentrated nitric acid and cover and reflux until the digestion is complete.

10.1.4 Uncover and let the sample volume go down to around 10ml. **DO NOT LET GO DRY!!!**

10.1.5 Add 5 ml of 1:1 Hydrochoric acid or 2.5 ml concentrated Hydrochloric acid and cover and reflux for 15 minutes.

10.1.6 Cool and filter if needed, add 0.05 ml Yittrium Standard. and bring the sample back up to 50ml in a class “A” volumetric flask.

10.1.7 Transfer the sample to a 50 ml centrifuge tube for storage.

10.2 Soil Sample Digestion for ICP Analysis (EPA SW 846 3050B section 7.5)

10.2.1 Transfer approximately 1.00g of sample into 200 ml glass beaker. If silica is requested, use 100 ml disposable beaker. For LCS and MS/MSD, add 1.0mL of 100 mg/l QC26 Standard used for ICV and appropriate amounts of individual standards to make a final concentration of 1.0 mg/l.

10.2.2 Add 2.5 ml conc. nitric acid and 10 ml conc. HCl, cover and place on the hot plate at 95°C and reflux 15 minutes.

10.2.3 Cool and filter through Q5 filter paper in to 100ml in a class "A" volumetric flask.

10.2.4 Add 0.1 ml Yttrium Standard and dilute the sample up to 100ml in a class "A" volumetric flasks.

10.2.5 Transfer sample to a 5 oz. plastic bottle for storage prior to analysis.

10.3 Metals digestion by Fusion

10.3.1 Weigh 0.5 grams +/- 0.1 mg of dry sample into a porcelain crucible. If sample is wet, run percent solids and increase the wet weight appropriately. . For LCS and MS/MSD, add 1.0mL of 100 mg/l QC26 Standard used for ICV and appropriate amounts of individual standards to make a final concentration of 1.0 mg/l.

10.3.2 Weigh in 6.0 grams potassium pyrosulfate. Run a blank and spikes.

10.3.3 Heat on a hotplate, if any moisture is present, making certain no splattering takes place.

10.3.4 Place in a muffle furnace at 700ºC for 1 hour.

10.3.5 Remove from heat and allow the melt to solidify.

10.3.6 Add 50 ml of 1:1 Hydrochloric acid to a 250 ml beaker and place the crucible in the beaker. Stir and heat until the melt has dissolved.

10.3.7 Add 0.1 ml Yittrium Standard. Cool and dilute to 100 ml with distilled water in a class "A" volumetric flasks. Transfer sample to a 5 oz. plastic bottle for storage prior to analysis.

10.3.8 Agitate the vial vigorously to ensure homogeneity. Let the solids settle. This is the first dilution and can be kept for the analysis of impurities such as Fe, V, Mg, Si, and Ca.

10.3.9 Transfer a 1 ml aliquot into a 100 ml snap cap vial. Adjust aliquot size for lower Al and Na results.

10.3.10 Add 20 mls of 1:1 Hydrochloric acid and dilute to volume with distilled water.

10.3.11 Agitate vigorously to ensure homogeneity.

10.3.12 Analyze metals by ICP.

11.0 METHOD PERFORMANCE

This method has been validated by the USEPA.

12.0 POLLUTION CONTROL

Refer to the Laboratory Chemical Hygiene Plan, SOP #GEN_22, for laboratory pollution control.

13.0 WASTE MANAGEMENT

Refer to SOP #GEN_19 (Hazardous Material Management) and SOP #GEN_20 (Waste Disposal) for laboratory waste management.

14.0 REFERENCES

14.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 3010A.

14.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 3050B.

14.3 Fansteel Inc. Standard Operating Procedure for Cryolite and Related Materials % Determination of Sodium and Aluminum by DCP, Revision 2, August 9, 1999, Initial DEH.

TCLP EXTRACTION – EPA SW 846 1311
SPLP EXTRACTION - EPA SW 846 1312

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TCLP EXTRACTION - EPA SW 846 1311
SPLP EXTRACTION - EPA SW 846 1312

1.0 PURPOSE

The purpose of this procedure is to simulate the leaching which might occur in a landfill containing the particular waste (sample(s)).

2.0 SCOPE & APPLICATION

The TCLP (Toxicity Characteristic Leaching Procedure) and the SPLP (Synthetic Precipitation Leaching Procedure) are designed to simulate the leaching that might occur in a sanitary landfill containing the particular waste. The TCLP and SPLP are suitable for determining the mobility of both organic and inorganic compounds present in liquid, solid, and multiphase wastes.

3.0 SUMMARY

3.1 For liquid samples (i.e., those containing less than 0.5% dry solid material), the waste after filtration through a 0.6 to 0.8 um glass fiber filter, is defined as the TCLP extract or the SPLP extract.

3.2 For wastes containing than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an acidic fluid equal to 20 times the weight of the solid phase. The extraction fluid chosen for a particular waste is a function of the alkalinity of that waste. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 um glass fiber filter.

3.3 If compatible, (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

4.0 DEFINITIONS

4.1 Accuracy – The closeness of agreement between an observed value and an accepted value.

4.2 Batch – A group of samples that behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, the number of samples analyzed as a group is 20 samples or less.

4.3 Bias – the deviation due to matrix effects of the measured value from a known spiked amount.

4.4 CCB – Continuing Calibration Blank – A blank ran during or at the end of analysis.

4.5 CCV – Continuing Calibration Verification standard – A standard ran during or at the end analysis.

4.6 ICB – Initial Calibration Blank – A blank ran right after making a calibration curve.

4.7 ICS – Initial Calibration Standard – A standard ran right after making a calibration curve.

4.8 LCS – Laboratory control Sample – A known matrix spiked with representative of the target analyte. This is used to document laboratory performance.

4.9 Matrix – The component or substrate which contains the analyte of interest, i.e.: surface water, drinking water, soil, etc.

4.10 MD or DU - Matrix Duplicate – An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

4.11 MS - Matrix Spike – Aliquot of sample spiked with a known concentration of target analyte. The spiking occurs prior to preparation of analysis. A matrix spike is used to document the bias of a method in a given matrix.

4.12 MSD - Matrix Spike Duplicate – A second matrix spike used to document precision in a give matrix.

4.13 MB – Method Blank – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is ran through the complete sample preparation procedures and analytical process. The method blank is used to document contamination resulting from analytical process.

4.14 Precision – The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses.

4.15 Solid portion of waste – the portion of the sample not passing through a 0.6 to 0.8 um glass fiber filter.

5.0 INTERFERENCES

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

6.0 SAFETY PRECAUTIONS

6.1 Hazardous Chemicals/Equipment

All work shall be performed under a fume hood when working with the hazardous chemicals.

6.2 Radiological Hazards

When radioactive materials are capable of being volatilized or airborne, perform all work under a fume hood.

6.3 General Laboratory Safety

Refer to the Laboratory Chemical Hygiene Plan, SOP#GEN_22 for general laboratory safety.

7.0 EQUIPMENT & SUPPLIES

7.1 Rotary Extractor - Capable of rotating extraction vessels in and end-over-end fashion at 30 $\pm$ 2 rpm.

7.2 Extraction Vessel - 2 liter borosilicate glass having Teflon lined lid.

7.3 Extraction Vessel – 2 liter plastic container with a plastic lid.

7.4 Zero-Headspace Extraction Vessel (ZHE) - made of 316 stainless steel, viton o-rings, and having a minimum volume of 500 ml.

7.5 Filter Holder - Buchner funnel capable of holding a 110 mm diameter filter and with a minimum volume of 300 ml. (Alternatively, a pressure filter holder may be used).

7.6 Vacuum Flask - 1200 ml, glass; Fleaker Pyrex 5900 or equivalent

7.7 Vacuum Pump - Precision Model PV35, or equivalent.

7.8 Top-loader Balance - Capable 0.01 g, mettler PC 4400 or equivalent.

7.9 pH Meter; Cole-Parmer 835 or equivalent.

7.10 Combination pH electrode, Orion 915600 or equivalent.

7.11 Magnetic stirrer – Corning PC-351 or equivalent

7.12 Magnetic stir bar

7.13 VOA Vials - 40 ml with Teflon-lined lids.

7.14 Amber Glass Bottles - 1000 ml, narrow mouth with Teflon-lined lids.

7.15 Aluminum Weigh Boats - For % moisture determination.

7.16 Water Bath - Set to 50°C.

7.17 Glass Fiber Filters - 90 to 110 mm diameter and 0.6 - 0.8 um pore size.

7.18 200 ml glass beakers and watch glasses - for initial pH determination.

7.19 Plastic sample containers of various sizes.

7.20 1 liter volumetric flask, class A.

8.0 REAGENTS

All reagents are ACS grade or better.

8.1 DI Water - ASTM Type II; <10 umho/cm.

8.2 Glacial acetic acid, CH_3CH_2OOH

8.3 Hydrochloric acid, concentrated, HCl

8.4 Nitric Acid, concentrated, HNO_3

8.5 Sulfuric Acid, concentrated, H_2SO_4

8.6 Sodium Hydroxide, pellets, NaOH

8.7 Hydrochloric Acid - 1N: Dilute 83 ml of concentrated hydrochloric acid with DI water to 1 liter in a volumetric flask.

8.8 Nitric Acid - 1N: Dilute 28 ml of concentrated nitric acid with DI water to 1 liter in a volumetric flask

8.9 Sulfuric acid/nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of nitric acid. If preferred, a more dilute H_2SO_4/HNO_3 acid mixture may be prepared making it easier to adjust the pH of SPLP extraction fluids #1 and #2.

8.10 Sodium Hydroxide - 1N: Dissolve 40 g of sodium hydroxide pellets in DI water and dilute to 1 liter with DI water in a 1 liter volumetric flask.

8.11 Sodium Hydroxide – 10 N: Dissolve 400 g of sodium hydroxide pellets in DI water. When cooled to room temperature, dilute with DI water to 1 liter in a volumetric flask.

8.12 Compressed Nitrogen; Air Gas UN1066 or equivalent.

8.13 TCLP Extraction Fluid #1 - Add 5.7 ml of glacial acetic acid to each 500 ml increment of DI water. Add 64.3 ml 1N sodium hydroxide per each increment, and dilute to 1000 ml with DI water per increment. Adjust the pH to 4.93 $\pm$ 0.05 with glacial acetic acid or sodium hydroxide. Check pH on the day of use. A twenty-liter batch of Extraction Fluid #1 is made by diluting 114ml of glacial acetic acid, and 129 ml of 10N sodium hydroxide to 20 liters with DI water. Each batch is identified by fluid type and date prepared.

8.14 TCLP Extraction Fluid #2 - Add 5.7 ml of glacial acetic acid to each 994.3 ml increment of DI water. Adjust the pH to 2.88 + 0.5 with glacial acetic acid. Check pH on day of use. A twenty-liter batch of Extraction Fluid #2 is made by diluting 114 ml of glacial acetic acid to 20 liters with DI water. Each batch is identified by fluid type and date prepared. Check pH on the day of use.

8.15 SPLP Extraction Fluid #1 – Add 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to DI until the pH is 4.20 ± 0.05. The fluid is used for samples from a site that is from a site **east** of the Mississippi River.

NOTE: If the pH of the DI water is < 5, it should not be used for making the SPLP Extraction fluids # 1 and # 2.

NOTE: SPLP Extraction Fluids # 1 and # 2 are unbuffered and the exact pH may not be attained.

8.16 SPLP Extraction Fluid #2 – Add 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to DI until the pH is 5.00 ± 0.05. The fluid is used for samples from a site that is from a site **west** of the Mississippi River.

8.17 SPLP Extraction Fluid #3 - DI water that has also been passed through a carbon filter bed.

8.18 Analytical standard shall be prepared according to the appropriate analytical method.

9.0 SAMPLE HANDLING & PRESERVATION

9.1 Soil samples should be collected and stored in airtight glass containers with Teflon-lined lids. Containers should have minimal or no headspace after filling with the sample. A minimum of 100 grams of sample should be collected. The samples should be kept at 4°C or lower except during preparation. Soil samples should be extracted within three days of receipt or within holding times for each test method. Refer to CFR 55 #61 for maximum holding times to extraction.

9.2 Aqueous samples should be collected in airtight glass containers with Teflon-lined lids. Containers should have no headspace after filing with the sample. A minimum of one liter of sample should be collected. The samples should be kept between 35 and 40°F except during sample preparation. Samples should be extracted within 3 days of receipt.

9.3 Samples are shipped refrigerated where applicable and with great care given to prevent loss of sample. Where radiological samples are shipped, specific regulations must be adhered to as well as safety precautions as to the packing and shipping of the samples.

10.0 QUALITY CONTROL

10.1 A minimum of one blank (using same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction batch. (A blank is to be performed on each new batch of extraction fluid).

11.0 PROCEDURE

11.1 Preliminary Investigations

Determination of solid/liquid content.

11.1.1 If the waste obviously contains no liquid portion, proceed with the extraction without further solids determination.

11.1.2 Weigh a minimum of 90 to 100 gram well-mixed portion into a pressure filter container or vacuum filter that has a preweighed (to 0.1 mg) glass fiber filter in place. If there is limited sample available to analyze, record this in the prep log in the comment section for the sample being prepped.

11.1.3 Gradually apply vacuum until air moves through the filter, or no additional liquid passes through the filter for 2 minutes.

11.1.4 Weigh the filter and residue.

11.1.5 Determine the percent solids by subtracting the filter's tare weight from the gross weight.

11.1.5.1 If the solid portion is $\geq$ 0.5% or 0.5g of the total weight, extraction must be performed on the solid phase. Particle size reduction will be required if the solid particles are greater that 1 cm in their narrowest dimensions. If particle size is required, crush, cut, or grind the waste until the size requirements are met. The solids must then be extracted and the extract must be combined with any liquid filtrate generated in the previous step. The method of extraction will depend on the type of analysis required. The remainder of this section will address the different types of extractions separately.

11.1.5.2 If the solid portion is < 0.5% of the total, the filtered liquid is considered to be the TCLP extract and no further action is taken with the solids portion. Filter sufficient volume of the sample to provide the laboratory with enough liquid to perform the final analysis. The volume needed will depend on the analyses required.

11.2 TCLP Extraction for semi volatile, pesticides, herbicides, and metals analyses

11.2.1 After particle size reduction if required, weigh 5 grams of the solid into a 125 ml container. Add 96.5 ml of DI water, and mix for 5 minutes.

Measure the pH and record mixing time and pH in TCLP Extraction logbook.

11.2.2 If pH is <5, proceed to the extraction step using extraction fluid #1.

11.2.3 If pH is >5, add 3.5 ml 1N HC1, slurry briefly, cover and heat at 50^{o} C for 10 minutes. Allow the solution to cool and measure the pH.

11.2.3.1 If pH is <5, proceed to the extraction step using extraction fluid #1.

11.2.3.2 If pH is >5, proceed to the extraction step using extraction fluid #2.

11.2.4 The extraction step should provide enough extract such that, when combined with any initial liquid filtrate (if compatible), there will be sufficient volume for all analytical fractions. The following are the desired volumes for the various fractions: semi volatile - 1000 ml, pesticides/herbicides - 1000 ml, metals - 250 ml. The ratio of extraction fluid to solids in the extraction step is 20:1.

11.2.4.1 Weigh into a 2 liter glass jar (a plastic 2 liter jar may be used if TCLP metals only are to be analyzed) enough of the solid portion of the waste to meet the requirements above. If the waste is 100% solids, use 90 grams and add 1800 milliliters extraction fluid as determined above. If the sample is less than 100% solids calculate the sample volume required by the following equation.

$$\frac{100}{S} = EW$$

S -% solids of the sample
EW-sample weight required to obtain 100 g solids.

Alternitively if insufficent sample volume remains the extraction fluid volume can be adjusted from 2000 ml to the correct volume by multiplying the amount of solids by 20.

NOTE: Remember the liquid portion of sample has to be added to the final extract.

11.2.4.2 Cap the jar tightly and tape the lid shut. Place in the rotary extractor and rotate for 18 $\pm$ 2 hours.

11.2.4.3 Following extraction, filter the extract through a 110 mm glass fiber filter by use of Buchner funnel with vacuum filtration. Alternatively pressure filtration may be used if the solids are likely to plug the filter rapidly.

11.2.4.4 Measure the pH of the filtered extract and record.

11.2.4.5 If compatible, combine this filtered extract with the initial filtrate and split into the various fractions. If initial filtrate and extract are not compatible (not miscible) they must be kept separate and analyzed separately with the results being combined mathematically. The semi volatile portion is collected in a 1 liter amber glass jar with Teflon-lined lid. A 500 ml amber glass jar with Teflon-lined lid is used for pesticides/herbicides. The portion for metals analysis is collected in a 250 ml plastic container. To the metals portion, add concentrated nitric acid to a pH of <2. Store at 4°C until analysis.

11.3 TCLP Extraction for Volatiles

The extraction step should provide enough extract such that, when combined with any initial liquid filtrate (if compatible), there will be sufficient volume for the volatiles analysis. Approximately 80 ml (two VOA vials) is normally provided. The extraction fluid to solids ratio is 20:1. A maximum of 25 grams of solids may be used in this extraction since the extractor has an internal volume of 500 ml.

11.3.1 Assemble the ZHE extraction vessel but do not put the top on. Extraction fluid #1 may be used to wet the Viton o-rings. Make certain the 90 mm glass fiber filter and screens are in place in the vessel top. Weigh into the assembled bottom portion enough sample such that there will be maximum of 25 grams of the solids portion of the waste. As quickly as possible, attach the top of the vessel and secure each fastener. Exposure of the sample or extract to the atmosphere should be kept at the minimum possible. Use cold sample if, at all possible, to minimize loss of volatiles. If the sample contains a liquid portion, filter the sample as discussed in section 11.2 under preliminary investigations using the ZHE apparatus as the pressure filter. Retain the filtrate for combination with the extract (if compatible). Determine the weight of sample remaining in the ZHE extractor and introduce the appropriate amount of extraction fluid #1 into

the closed system containing the solids. Fluid introduction may be accomplished by using a pump or other method capable of overcoming the pressure required to retract the piston.

11.3.2 After filling with extraction fluid and the ZHE device in the vertical position, gently apply gas pressure beneath the piston to force all headspace out of the ZHE device. At the first appearance of liquid from the device, discontinue pressure and close the outlet valve. Repressurize the ZHE with 5 - 10 psi and check all fittings for tightness. Rotate for 18 +/- 2 hours.

11.3.3 After the 18 hour agitation period, ascertain that the pressure has been maintained. The extraction fluid is then filtered and collected in the appropriate container. Filtration should be accomplished as quickly as possible to limit any exposure to the atmosphere, but should not be so rapid as to produce "fizzing" of the liquid which may result in loss of volatiles. Extract may be collected in a gas-tight syringe and transferred to 40 ml VOA vial. If the initial liquid filtrate was produce, the extraction fluid should be transferred directly into this filtrate, if compatible. A separate container must be used if the liquids are not compatible. Store the extracts/filtrates at 4°C until analysis.

11.4 SPLP Extraction for semi volatile, pesticides, herbicides, and metals analyses

NOTE: Determination of appropriate extraction fluid:

For soils: sample is from a site east of the Mississipi River, extraction fluid #1 is used; sample is from a site west of the Mississippi River, extraction fluid #2 is used.

For wastes and wastewater: extractionfluid # 1 is used.

For cyanide-containging wastes and/or soils: extraction fluid #3 is used.

11.4.1 The extraction step should provide enough extract such that, when combined with any initial liquid filtrate (if compatible), there will be sufficient volume for all analytical fractions. The following are the desired volumes for the various fractions: semi volatile - 1000 ml, pesticides/herbicides - 1000 ml, metals - 250 ml. The ratio of extraction fluid to solids in the extraction step is 20:1.

11.4.1.1 Weigh into a 2 liter glass jar (a plastic 2 liter jar may be used if TCLP metals only are to be analyzed) enough of the solid

portion of the waste to meet the requirements above. If the waste is 100% solids, use 90 grams and add 1800 milliliters of the appropriate SPLP extraction fluid. If the sample is less than 100% solids calculate the sample volume required by the following equation.

$$\frac{100}{S} = EW$$

S -% solids of the sample
EW-sample weight required to abtain 100 g solids.

Alternitively if insufficent sample volume remains the extraction fluid volume can be adjusted from 2000 ml to the correct volume by multiplying the amount of solids by 20.

NOTE: Remember the liquid portion of sample has to be added to the final extract.

11.4.1.2 Cap the jar tightly and tape the lid shut. Place in the rotary extractor and rotate for 18 $\pm$ 2 hours.

11.4.1.3 Following extraction, filter the extract through a 110 mm glass fiber filter by use of Buchner funnel with vacuum filtration. Alternatively pressure filtration may be used if the solids are likely to plug the filter rapidly.

11.4.1.4 Measure the pH of the filtered extract and record.

11.4.1.5 If compatible, combine this filtered extract with the initial filtrate and split into the various fractions. If initial filtrate and extract are not compatible (not miscible) they must be kept separate and analyzed separately with the results being combined mathematically. The semi volatile portion is collected in a 1 liter amber glass jar with Teflon-lined lid. A 500 ml amber glass jar with Teflon-lined lid is used for pesticides/herbicides. The portion for metals analysis is collected in a 250 ml plastic container. To the metals portion, add concentrated nitric acid to a pH of <2. Store at 4°C until analysis.

11.5 SPLP Extraction for Volatiles

The extraction step should provide enough extract such that, when combined with any initial liquid filtrate (if compatible), there will be sufficient volume for the volatiles analysis. Approximately 80 ml (two VOA vials) is normally provided. The extraction fluid to solids ratio is 20:1. A maximum of 25 grams of solids may be used in this extraction since the extractor has an internal volume of 500 ml.

11.5.1 Assemble the ZHE extraction vessel but do not put the top on. Extraction fluid #3 may be used to wet the Viton o-rings. Make certain the 90 mm glass fiber filter and screens are in place in the vessel top. Weigh into the assembled bottom portion enough sample such that there will be maximum of 25 grams of the solids portion of the waste. As quickly as possible, attach the top of the vessel and secure each fastener. Exposure of the sample or extract to the atmosphere should be kept at the minimum possible. Use cold sample if, at all possible, to minimize loss of volatiles. If the sample contains a liquid portion, filter the sample as discussed in section 11.2 under preliminary investigations using the ZHE apparatus as the pressure filter. Retain the filtrate for combination with the extract (if compatible). Determine the weight of sample remaining in the ZHE extractor and introduce the appropriate amount of extraction fluid #1 into the closed system containing the solids. Fluid introduction may be accomplished by using a pump or other method capable of overcoming the pressure required to retract the piston.

11.5.2 After filling with SPLP extraction fluid #3 and the ZHE device in the vertical position, gently apply gas pressure beneath the piston to force all headspace out of the ZHE device. At the first appearance of liquid from the device, discontinue pressure and close the outlet valve. Repressurize the ZHE with 5 - 10 psi and check all fittings for tightness. Rotate for 18 +/- 2 hours.

11.5.3 After the 18 hour agitation period, ascertain that the pressure has been maintained. The extraction fluid is then filtered and collected in the appropriate container. Filtration should be accomplished as quickly as possible to limit any exposure to the atmosphere, but should not be so rapid as to produce “fizzing” of the liquid which may result in loss of volatiles. Extract may be collected in a gas-tight syringe and transferred to 40 ml VOA vial. If the initial liquid filtrate was produce, the extraction fluid should be transferred directly into this filtrate, if compatible. A separate container must be used if the liquids are not compatible. Store the extracts/filtrates at 4°C until analysis.

12.0 CALCULATIONS

Percent solids (%) = $\frac{(A - B) * 100\%}{C}$

Where:

A = weight of dried glass fiber filter after filtering sample (g)
B = tarred weight of glass fiber filter (g)
C = Sample volume (ml)

13.0 METHOD PERFORMANCE

This method has been validated by EPA.

14.0 POLLUTION PREVENTION

Refer to Laboratory Chemical Hygiene Plan, SOP # GEN_22 for laboratory pollution control.

15.0 WASTE MANAGEMENT

Refer to SOP #GEN-19 (Hazardous Material Management) and SOP # GEN_20 (Waste Disposal) for laboratory waste management.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 1311, SEPA.

16.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 1312

16.3 CFR 55 #61,

17.0 MISCELLANEOUS / ATTACHMENTS

17.1 Sample Maximum Hold Times (days)

Volatiles:

From Field collection to TCLP extraction – 14
From TCLP extraction to Prep extraction – N/A
From Prep extraction to Analysis – 14

Semi-volatiles:

From Field collection to TCLP extraction – 14
From TCLP extraction to Prep extraction – 7
From Prep extraction to Analysis – 40

Mercury:

From Field collection to TCLP extraction – 28
From TCLP extraction to Prep extraction – N/A
From Prep extraction to Analysis – 28

Metals, except mercury:

From Field collection to TCLP extraction – 180
From TCLP extraction to Prep extraction – N/A
From Prep extraction to Analysis – 180

17.2 Attachments follow:

17.2.1 Volatile Analytes (EPA SW 846 – Table I)

17.2.2 EPA SW 846 - Figure 2. Zero-Headspace Extractor (ZHE)

17.2.3 TCLP diagram (EPA SW 846)

METALS ANALYSIS BY IRIS ICAP
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METALS ANALYSIS BY IRIS ICAP

1.0 PURPOSE

The purpose of this procedure is to provide a standardized method for the analysis of metals in water, wastewater, soil, solids, leachates, and vegetation samples.

2.0 DETECTION LIMITS

Minimum Detection Limit (MDL) studies are performed annually or after major instrumentation maintenance. The most recent MDL studies are located in Attachment 1.

3.0 SCOPE AND APPLICATION

3.1 This method is used for the determination of dissolved, suspended or total metals in prepared solutions on a variety of matrices and is based upon the EPA SW 846 6010B method. Attachment 2 lists elements for which this method applies with current wavelengths recommended. Elements and wavelengths will be added as ICP methods are developed or as needed.

4.0 SUMMARY

4.1 Prior to analysis the samples must be digested using the appropriate Sample Preperation Methods. When analyzing groundwater for dissolved metals, acid digestion is not necessary if samples were filtered and preserved prior to analysis.

4.2 This method describes the technique for the determination of trace metals in a prerared solution using a Thermo Jarell Ash IRIS Intrepid II XSP. The ICP uses Echelle optics and a Charge Injection Device (CID) solid-state detector to provide complete and continous wavelength coverage over the typicle wavelength range. The High-Resolution (HR) version of the IRIS can measure either axially or radially for wavelenghts 160 to 800 nm.

5.0 DEFINITIONS

5.1 Accuracy – The closeness of agreement between an observed value and an accepted reference value.

5.2 Batch – A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC

purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

5.3 Laboratory Control Sample – A 1.0 mg/l or prepurchased standard with compound(s) representative of the target analytes. This is used to document laboratory performance.

5.4 Matrix Spike – An aliquot of sample spiked with 1.0 mg/l concentration of target analytes(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

5.5 Matrix Spike Duplicates – Intralaboratory split samples spiked with identical concentrations of target analyte(s).

5.6 Method Blank – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

6.0 INTERFERENCES

6.1 Spectral Interferences

6.1.1 Glassware Contaminants

6.1.2 Overlap of a spectral line from another element compensated by interelement correction by instrument.

6.1.3 Unresolved overlap of molecular band spectra may require selection of an alternate wavelength.

6.1.4 Background contribution from continuous or recombination phenomena.

6.1.5 Background contribution from stray light from the line emission of high concentration elements.

Note: The two listed above can usually be compensated by a background correction adjacent to the analyte line.

6.2 Physical Interferences

Affects associated with the sample nebulization and transport processes. Dilution may be necessary.

7.0 SAFETY PRECAUTIONS

7.1 Hazardous Chemicals/Equipment

All work shall be performed under a fume hood when working with the hazardous chemicals.

7.2 Radiological Hazards

When radioactive materials are capable of being volatilized or airborne, perform all work under a fume hood.

7.3 General Laboratory Safety

Refer to the Laboratory Chemical Hygiene Plan, SOP #GEN_22, for general laboratory safety.

7.4 Precautions shold be taken when diluting concentrated acids.

8.0 EQUIPMENT AND SUPPLIES

8.1 TJA IRIS Intrepid II ICP with autosampler

8.2 Volumetric flasks (various sizes)

8.3 15 ml plastic centrifuge tubes

8.4 Auto-pipets and tips – EDP 100, EDP 2500 plus, oxford 1-5ml, and oxford 5-10ml.

8.5 Auto sampler tubing

8.6 50 ml plastic centrifuge tubes

8.7 150 ml plastic bottles or equivalent

8.8 Optifix repipetor – 10 ml

9.0 REAGENTS

9.1 Nitric Acid, conc; ACS grade

9.2 Nitric Acid, (1+1); add 500 ml conc. HNO_3 to 400 ml DI Water and dilute to 1 L.

9.3 Hydrochloric Acid, conc; ACS grade

9.4 Hydrochloric Acid, (1+1); add 500 ml conc. HCl to 400 ml DI Water and dilute to 1 L.

9.5 ICP Standards diluted using DI Water and 6 ml Nitric Acid and 5 ml Conc. HCl. Quality Control Standard 4 w/26 elements (100 mg/l N.I.S.T. Traceable). The following standards are to be prepared:

METALS
ICP STANDARDS
PREPARATION

Standard Number	mL Added	STD Conc mg/l	Final Volume mL	Yttrium(IS) mL	Final Concentration mg/L	SiO_2 mg/L
W1	0.020	5	100	0.10	0.001	0.002
W2	0.040	5	100	0.10	0.002	0.004
W3	0.100	5	100	0.10	0.005	0.011
W4	0.200	5	100	0.10	0.010	0.021
W5	0.100	100	100	0.10	0.100	0.214
W6	0.500	100	100	0.10	0.500	1.07
W7	1.00	100	100	0.10	1.00	2.14
W8	2.00	100	100	0.10	2.00	4.28
W9	5.00	100	100	0.10	5.00	10.70
W10	10.00	100	100	0.10	10.0	21.40

Acid Volumes					
	Water	HNO_3	6.0 ml	HCl	5.0 ml
	Soil	HNO_3	2.5 ml	HCl	10.0 ml

9.6 Calibration blanks - dilute 6 ml of Conc. HNO3, 5 ml Conc. HCl and 0.1 ml of Yttrium internal standard to 100ml with Distilled Water. This blank is used in establishing the analytical curve and to flush the system between standards and sample.

9.7 Initial Calibration Verification - A standard made from a different stock solution as that used for the calibration standards and analyzed immediately after calibration to validate calibration. Concentration of the calibration check standard is 1.0 mg/l at the mid-point of the calibration curve.

9.8 Distilled Water

9.9 Liquid Nitrogen with gas valve: Nitrogen Service Co.

9.10 Liquid Argon with gas valve; Victor Gas.

10.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

All aqueous samples are to be collected in prewashed containers. Aqueous samples for Total Metals must be acidified to a pH of < 2 with Nitric acid. Aqueous samples for Dissolved Metals must be filtered on site with a 0.45 micron filter and acidified to a pH of < 2 with Nitric acid. Aqueous samples for Suspended Metals must be filtered on site. Soil/sediment samples are to be collected in an 8 oz. soil jar or like container. The maximum holding time for metals is 6 months. Samples should be shipped and stored under refrigeration at 0-4°C.

11.0 QUALITY CONTROL

11.1 Refer to Section 17 for normal QC requirements.

11.2 Corrective Action

11.2.1 Nonconformances may occur at any level. Some may be corrected immediately and documented using normal laboratory document procedures. Out-of-control nonconformances shall be documented by the use of a Corrective Action Report, usually issued by the QAO. With the use of a CAR, findings can be tracked and used for future trends.

11.2.2 Work shall be stopped when out-of-control deficiencies occur until the problem is alleviated, ie., instrumentation malfunction, QC out of control limits, failure to perform demonstrations of capabilities, etc.

11.3 Refer to section 8.8 of Quality Assurance Manual (QAM) for LOD and LOQ criteria.

11.4 When analyzing new or unusal matrixes the following tests need to be performed prior to reporting data:

11.4.1 Dilution Test: If sample is > 10 times the LOD, dilute 1:5 and the result should be within 10% of the orginal result. If the result is > 10% flag and report in the case narratinve that the sample has possible matix interferences.

11.4.2 Post Digestion Spike: Add 0.1ml of a Q26 standard to 10 ml sample and analyze. The spike recovery should be between 75% and 125% recovery. If the spike recovery is not within limits, report in case narrative that the sample has possible matrix interferences.

12.0 CALIBRATION

12.1 Setting Plasma Conditions

12.1.1 Choose "Plasma Control" from the Setup drop-down menu. Set the Plasma at the following setting:

Flush Pump rate (rpm) :	130
Analysis Pump rate(rpm):	130
Relaxation Time (sec):	5
Pump Tubing Type:	Tygon-Orange
Nebulizer Flow(LPM):	20.6-39 (variable)
Auxiliary Gas	0.5
RF Power	1150
Rinse	90 sec.

Note: These settings are adjustable for optimum performance

12.2 Analytical Preferences

12.2.1 Choose "Analytical preferences" from the Setup drop-down menu. Set the preferences at the following setting:

Repeats:	3
Delay time:	0
Sample Flush Time :	60
CID Max integration time (sec)	
Low Wavelengths:	30
High Wavelengths:	30

Note: These may adjusted to lower detection limit or optimizing performance.

12.3 Element Mapping

12.3.1 Select “Instrument” then “Map editor..” .

12.3.2 Select the element that needs calibrated and select “Edit”.

12.3.3 Add the selected wavelengths and place autosampler tube in a single element standard 10 mg/l or greater. Select “Calibrate” and the instrument automatically calibrates each wavelength. Wavelengths are selected based on 6010B recommended wavelengths and wavelengths that the most intensity and least possible interferences. The wavelengths are identified as thre example below:

As – wavelength 193.7
AsAlt – wavelength 189.6

A full list of elements and wavelengths are in Attachment 2.

12.4 Annually verify interelement and background correction factors for each wavelength. See Attachement 3. The ICS and consists of two solutions: Solution A consists of the interferants and solution AB consists of the analytes mixed with the interferants. An ICS analysis consists of analyzing both solutions consecutively for all wavelengths used. Results for the analyses of solution AB must fall within the control limits of $\pm$ 20% of the true value for the each element at the each wavelength. The results of the ICS must be below 3 times the MDL or LOD.

12.5 Standardization (performed daily)

12.5.1 From the Analysis Main Menu select “6010B_200.7Y” from the Methods drop-down menu.

12.5.2 From the Analysis Main Menu select “Standardize…” from the Setup drop-down menu. The standards that are to be analyzed at the following concentrations:

<u>Standard calibration standards</u>
Blank
S1-0.2 mg/l

S2-0.5 mg/l
S3-1.0 mg/l
S4-2.0 mg/l
S5-5.0 mg/l

Trace calibration standards
Blank
T1-0.020 mg/l
T2-0.050 mg/l
T3-0.100 mg/l
S1-0.200 mg/l
S2-0.500 mg/l

12.5.3 Standard Analysis

12.5.3.1 Select the autosample Icon from the tool bar. Select the standards rack and select clear all tubes. Place the mouse curser in the 1st tube of the standards rack and select add all standards.

12.5.3.2 Using the mouse click on the first blank and select start run from current sample from menu.

13.0 PROCEDURE

13.1 Start-Up

Check that the argon tank main valve is open and the pressure is set to at least 70 psi. Check that the liquid nitrogen tank main valve is open and the pressure is set to at least 70 psi. After nitrogen has been flowing for 1 hour across the lens for the camera the ICP power can be turned on. Allow for the optics to warm to 90 ^{o}C before starting any analysis.

Check all tubing, drain lines, deionized water and CID temperature. Press the ingnite button to light the torch. Allow the torch to stablize for at least 30 minutes.

Load method files to be used.

Setup autosampler table with sample information. Load the autosampler with standards, ICS A, ICS AB, ICV, ICB, samples, CCV's and CCB's. Using the mouse click on the first sample and select start run from current sample from menu.

After analysis is complete make sure that the ICAP sample introduction system has been thoroughly rinsed by aspirating deionized water for at least 10 minutes.

13.2 Shutdown

Extinquish plasma by selecting shutdown from the menu.

Release the pressure on the pump winding.

If it is going to be over 24hrs before the ICAP is used again shut off the argon gas supply.

14.0 CALCULATIONS

14.1 Water Samples

Result in mg/L = Concentration from Curve x Dilution Factor

14.2 Soil/Sediment Samples

$$\text{Result in mg/kg} = \frac{(\text{CC} \times \text{Vf})}{(\text{Wt})} \times \text{DF} \times \text{DWF}$$

where,

CC	=	Concentration from curve in ug/l
Vf	=	Final volume in liters (usually 0.l)
Wt	=	Initial weight of sample in grams (usually 1.00g)
DF	=	Any Dilution Factor used
DWF	=	Dry Weight Factor to account for percent moisture

15.0 METHOD PERFORMANCE

This method has been validated by the USEPA.

16.0 POLLUTION PREVENTION

Refer to Laboratory Chemical Hygiene Plan, SOP #GEN_22 for laboratory pollution control.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

After analyst has reviewed the data the Metals Supervisor, QA Officer, or Lab Director will review the data for the above mentioned items to ensure no errors have been made.

17.1 ICP ACCEPTANCE CRITERIA

Replicates RSD < 30%

17.2 CALIBRATION AND QC ACCEPTANCE CRITERIA

Calibration/ QC Samp	Frequency	Acceptance Criteria	Corrective Action
Calibration	Daily	Minimum of 5 levels plus on ($R^2 < 0.995$)	Corrective action per instrument manu recommendations and recalibrate.
Initial Calibration Verification (ICV)	1.0 mg/l independent standard prior to analysis.	90 – 110% Recovery	Recalibrate
Initial Calibration Blank (ICB)	Analyze after each ICV	3 * IDL	Recalibrate
Continuing Calibration Verification (CCV)	1.0 mg/l standard after every 10 samples and at the end of the run.	90 – 110% Recovery	Recalibrate
Continuing Calibration Blank (CCB)	Analyze after each ICV or CCV	<3*IDL	Recalibrate
Method Blank	5%	<3*MDL or <1/10 of sample concentration	Rerun. If not in control limits, flag and explain in case narrative.
Laboratory Control Standard (LCS)	5%	80 – 120% Recovery	Rerun. If not in control limits, flag and explain in case narrative.
Matrix Spike (MS)	5%	75 – 125% Recovery	If not in control limits, flag and explain in case narrative.
Matrix Spike Duplicate (MSD)	5%	75 – 125% Recovery ≤ 20% RPD	If not in control limits, flag and explain in case narrative.
Interference Check Blank (ICS A)	Daily	<3*MDL	Recalibrate
Interference Check Standard (ICS AB)	Daily	80 – 120% Recovery	Recalibrate

18.0 WASTE MANAGEMENT

Refer to SOP #GEN_19 (Hazardous Waste Management) and SOP #GEN_20 (Waste Disposal) for laboratory waste management.

19.0 REFERENCES

19.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), EPA, Method 6010B.

19.2 Methods for Chemical Analysis of Water and Wastes, EPA, Method 200.7.

20.0 ATTACHMENTS

20.1 Attachment 1 – MDL Studies

20.2 Attachment 2 – Current Elements & Wavelengths

20.3 Attachment 3 – Interelement Correction Factors.

ATTACHMENT 1

MDL STUDIES

ATTACHMENT 2

WAVELENGTH

ELEMENT	Primary	LDR	Alternate	LDR

		mg/L		mg/L
Aluminum	396.152	50	237.312	10
Antimony	206.833	10	195.039	10
Arsenic	193.606	10	189.042	10
Barium	455.403	10	233.527	10
Beryllium	313.042	5	313.107	10
Boron	249.678	100	249.773	50
Cadmium	226.502	10	214.438	10
Calcium	317.933	20	315.887	20
Chromium	267.716	20	276.654	20
Cobalt	228.616	50	237.862	50
Copper	324.754	50	224.700	50
Iron	259.940	50	238.208	20
Lead	220.353(152)	100	220.353(153)	100
Magnesium	279.553	10	279.079	50
Manganese	257.610	20	259.373	20
Molybdenum	202.030	10	203.844	10
Nickel	231.604	50	221.647	50
Potassium	766.491	100		
Selenium	196.026	20	203.985	10
Silver	328.068	20	338.289	5
Sodium	588.995	10	589.582	20
Titanium	336.121	20	337.280	20
Thallium	190.864(175)	100	190.864(176)	10
Vanadium	292.402	50	309.311	20
Zinc	213.856	10	206.200	10
Yttrium	224.306(UV)		324.228(VIS)	

ATTACHMENT 3

Interelement Correction Factors

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

ACID DIGESTION OF

SEDIMENTS, SLUDGES, AND SOILS

(GL-MA-E-009 REVISION 20)

APPLICABLE TO METHODS:
EPA SW-846 3050B Modified
16 CFR part 1303 Modified

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

2.0 METHOD CODE

2.1 EPA SW-846 3050B Modified

2.2 16 CFR Part 1303 Modified

3.0 MEHTOD OBJECTIVE/PURPOSE

To describe the manner in which sediments, sludges, and soils for Inductively Coupled Plasma (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) analysis are digested by EPA SW-846 Method 3050B Modified. Samples digested by this procedure are applicable for analysis by SW-846 methods 6010B and 6020. Included in this standard operating procedure is guidance for paint and jewelry preparation.

4.0 METHOD SUMMARY

A representative portion of sample is digested with nitric acid and hydrogen peroxide. This digestate is then refluxed with hydrochloric acid for ICP analysis only. Samples prepared by this method may be analyzed for all the listed metals. Other metals may be analyzed if they pass control standard criteria:

Aluminum	Chromium	Lead	Thallium
Antimony	Copper	Sulfur	Vanadium
Arsenic	Iron	Sodium	Zinc
Barium	Potassium	Selenium	Silver
Beryllium	Manganese	Strontium	Titanium
Calcium	Nickel	Tin (ICP Only)	Phosphorus
Cadmium	Cobalt	Thorium (ICP-MS Only)	
Uranium (Isotopes 233, 234, 235, 236, and 238 via ICP-MS only)	Molybdenum	Lithium (ICP-MS Only)	

This method is not a “total” digestion technique for most samples. It is a very strong acid digestion that will dissolve all elements that could become “environmentally available” by design; elements bound in silicate structures (boron, silicon, silica) are not normally dissolved by this procedure as they are not usually mobile in the environment.

5.0 APPLICABLE MATRICES

5.1 Soils

5.2 Sludges

5.3 Sediments

5.4 Solid debris/powders

5.5 Heavy oils

5.6 Filters

5.7 Paints

5.8 Metal jewelry

6.0 HOLD TIME

Holding time is 180 days from the time and date of collection until the start of analysis unless otherwise specified by contract.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

Solid samples are not preserved but must be stored at 0°- 6° C.

8.0 INTERFERENCES

There are rarely any interferences with this digestion. If any are encountered, consult the group leader or quality officer before continuing.

9.0 PERFORMANCE CHARACTERISTICS

Method detection limits (MDLs) and method detection limit verification (MDLVs) are performed annually.

10.0 DEFINITIONS

10.1 Blank: Type I water that has been taken through the digestion process. The blank is used to determine the amount of background contamination.

10.2 Laboratory Control Sample (LCS): A certified reference material that has been taken through the digestion process. The LCS is used to determine digestion accuracy and to determine if the digestion process is in control.

10.3 Laboratory Control Sample Duplicate (LCS DUP): A duplicate of the LCS. The LCS DUP is used to determine reproducibility and to indicate precision.

10.4 Matrix Spike (MS): A sample that has added to it a known amount of solution containing known concentrations of analytes. The MS is used to determine the presence or absence of interferences and matrix effects in the digested sample.

10.5 Matrix Spike Duplicate (MSD): A duplicate of the MS. The MSD indicates reproducibility.

10.6 Sample Duplicate (DUP): A duplicate of a sample. The DUP indicates reproducibility.

10.7 AlphaLIMS: The Laboratory Information Management System used at GEL.

10.8 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

11.0 ANALYST VERIFICATION

Before a technician/analyst is allowed to analyze samples without supervision, he or she is trained by qualified personnel and is required to successfully analyze a proficiency sample. Training records are maintained as quality records (Refer to GL-QS-E-008).

12.0 DOCUMENTATION OF DATA

Sample preparation data are recorded in AlphaLIMS.

13.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

WARNING

CONCENTRATED HYDROCHLORIC ACID AND NITRIC ACID ARE EXTREMELY CORROSIVE AND CAN CAUSE SEVERE BURNS TO THE SKIN.

CONCENTRATED 30% HYDROGEN PEROXIDE IS A VIOLENT OXIDIZER. KEEP AWAY FROM OPEN FLAMES, AND RINSE WITH WATER IF SKIN CONTACT OCCURS.

13.1 Wear eye protection with side shields while performing procedures in the lab.

13.2 Treat all chemicals and samples as potential health hazards, and reduce exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals. A reference file of Material Safety Data Sheets (MSDS) and individual client sample MSDSs are also maintained.

13.3 Personal protective equipment

13.3.1 Disposable gloves are worn and changed frequently when working with acids, glassware, or samples. Dirty gloves pose a contamination hazard to the samples. Gloves that have holes can be dangerous to the wearer by allowing acids and toxic metals to come in contact with skin.

13.3.2 Hood doors are pulled down partially while digesting samples. Acidified samples can splash and pop as they are being heated.

13.3.3 To protect clothes and skin from exposure to corrosive material, wear a lab jacket.

13.4 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

13.4.1 Wear plastic apron over lab coat when working with radioactive samples.

13.4.2 Protect counter tops with counter paper, or work from radioactive sample handling trays.

13.4.3 Prohibit admittance to immediate work area.

13.4.4 Post signs indicating radioactive samples are in the area.

13.4.5 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

13.4.6 Segregate radioactive wastes. Radioactive waste containers are obtained from the Waste Management.

13.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

13.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

13.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

13.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

13.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician submits the list of samples needed to the sample custodian group. The sample custodian removes the appropriate sample from the cooler and scans it using the barcode scanner to the appropriate area of the lab. The analyst then takes custody of the samples and scans them to the sample batch. The samples are now ready to be prepared or analyzed.

14.2 Analysts/technicians are responsible for retrieving their own samples when the sample custodian is unavailable.

15.0 INSTRUMENT/EQUIPMENT/GLASSWARE

15.1 Equipment

15.1.1 Air displacement pipettes

15.1.1.1 5-10 mL with disposable tips

15.1.1.2 0.5-5 mL with disposable tips

15.1.1.3 100-1000 µL with disposable tips

15.1.1.4 10-100 µL with disposable tips

15.1.2 Environmental Express hot blocks or equivalent

15.1.3 Analytical balance capable of reading to three decimal places

15.1.4 Certified disposable 50 mL digestion tubes (polypropylene)

15.1.5 Ribbed disposable watch glasses (polypropylene)

15.1.6 Water resistant lab markers

15.1.7 Styrofoam trays to handle up to 25 digestion tubes

15.1.8 500 mL Nalgene squirt bottle

15.1.9 Teflon chips

15.1.10 1-inch white laboratory tape

15.1.11 Borosilicate beakers (various sizes)

15.1.12 Borosilicate watch glasses (various sizes)

15.1.13 Disposable razor blade or scalpel

15.1.14 Metal cutters

16.0 REAGENTS

16.1 Nitric acid (HNO_3), concentrated high purity grade 70% nitric acid

16.2 Hydrochloric acid (HCl), concentrated high purity grade 37% hydrochloric acid

16.3 Hydrogen peroxide (H_2O_2), concentrated 30% hydrogen peroxide

16.4 Type I water, DI water that conforms to the following specifications:

Electrical resisitivity, minimum	16.67
Total organic carbon, maximum, µg/L	100
Sodium, maximum, µg/L	1

Chlorides, maximum, µg/L	1
Total Silica, maximum, µg/L	3

Type I water is dispersed with the metals prep lab by the "Milli Q" water system.

16.5 Multi-element spiking solutions are purchased from NIST-traceable vendors.

17.0 PREPARATION OF SAMPLES

A batch consists of samples of the same matrix and quality control (QC) samples that are digested together. Each of the quality control samples listed in steps 10.1 through 10.6 must be included in each batch at the frequency listed or as per client request. The blank, LCS, and/or LCS DUP are digested at a frequency of one in 20 or per batch, whichever is more frequent. The MS, MSD, and/or DUP are digested at a frequency of one in 20 or per batch, whichever is more frequent, or per specified client/program requirements.

17.1 Glassware preparation:

17.1.1 Glassware that has been cleaned according to GL-LB-E-003 for Glassware Preparation is soaked in a water and acid mixture for at least 30 minutes.

17.1.2 After soaking, the glassware is rinsed with copious quantities of Type I water and then inverted over clean, absorbent paper or onto a rack for drying.

17.2 Label the Teflon beakers or centrifuge tube with the sample numbers in the batch. If centrifuge tube is to be used for measuring initial and final volumes it must be calibrated before usage. Refer to GL-LB-E-026 for centrifuge tube testing procedure.

17.3 Refer to GL-LB-E-029 for subsampling instructions. Mix the sample to achieve homogeneity. Weigh approximately 0.5 g of sample. Transfer the weighed sample to the appropriately labeled Teflon beaker or centrifuge tube.

17.3.1 Sample aliquots should not be taken from the top of an unmixed sample because large particles tend to rise in solid matrixes and heavy materials tend to sink in liquid matrixes.

17.3.2 Powdered samples may be homogenized by gently rocking the sample side to side. Then a representative aliquot may be taken from the center of the powder.

17.3.3 Other matrixes must be stirred, turned or mixed before sampling.

17.4 Quality control samples are prepared prior to digestion.

17.4.1 The beaker or tube to be used for the blank, MS, MSD, and/or DUP, LCS, and/or LCS DUP is labeled.

17.4.2 Weigh approximately 0.5 g of sample and transfer to the MS, MSD, and/or DUP beaker or tube.

The MS, MSD, LCS, and/or LCS DUP are spiked with known amounts of spiking solution.

17.4.3 Select a LCS. The LCS is purchased for an outside vendor and comes with a certificate of certified values and recovery ranges. The LCS is logged into the AlphaLIMS system for traceability and for the use of nominal calculations. For analytes not certified in the Solid Reference Material

(SRM), a series of 20 preparations and analyses are conducted and an average concentration is determined. A value of 3 times the standard deviation is used as control limits for the LCS recovery for these analytes. Mix the LCS to achieve homogeneity. Weigh approximately 0.5 g of the sample and transfer to the LCS and/or LCS DUP Teflon beaker or centrifuge tube. For non-soil solid samples, a liquid LCS is used in combination with approximately 0.5 g of Teflon chips.

17.4.4 The blank beaker or tube is labeled and no water, spike, or sample is added to it. Approximately 0.5 g of Teflon chips is used.

17.5 If the samples are being prepared for ICP-MS analysis:

17.5.1 Add 2.5 mL nitric acid and Type I DI water to the samples and quality control samples.

17.5.2 Gently swirl the sample and acid mixture.

17.5.3 Cover the sample with a watch glass and heat the sample on a hot plate/block to 95° ± 5° C. Reflux the sample for 10 to 15 minutes.

17.5.4 Remove the sample from the hot plate or block and allow the sample to cool.

17.5.5 Add 2.5 mL of concentrated nitric acid, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated indicating oxidation of the sample by nitric acid, repeat step 17.5.5 over and over until no brown fumes are given off by the sample.

17.5.6 Using a ribbed watch glass or vapor recovery system, allow the solution to evaporate to approximately 0.5 mL without boiling, or heat for 2 hours.

17.5.6.1 Remove the sample from the hot plate or block and allow the sample to cool.

17.5.6.2 Add 1.5 mL of hydrogen peroxide and 1.0 mL of Type I water. Return the sample to the hot plate or block and allow the peroxide reaction to occur. Continue to add hydrogen peroxide to the sample until the effervescence subsides. Do not add more than 5 mL hydrogen peroxide.

17.5.6.3 Cover the sample with a ribbed watchglass, heating the acid-peroxide digestate until the volume is reduced to approximately 2.5 mL, or heat at 95° ± 5° C without boiling for 2 hours.

17.5.6.4 Do not allow the sample to evaporate to dryness.

17.5.6.5 Remove the sample from step 17.5.6.3 from the hot plate or block.

17.5.6.6 Allow the sample to cool.

17.5.6.7 Dilute the sample to 50 mL with Type I water.

17.5.6.8 Cap and shake the sample.

17.5.6.9 Filter each sample with a 2.0 µm pore size plunger type filter (PTF grade) or allow to settle overnight.

17.5.6.10 Organize the samples in a storage container, and label the container with the batch number of the sample group.

17.6 If the samples are being prepared ICP analysis:

17.6.1 Add 1.25 mL nitric acid and 10 mL hydrochloric acid to the samples and quality control samples.

17.6.2 Gently swirl to mix.

17.6.3 Cover the sample with a watch glass and heat the sample on a hotplate/block to 95° ± 5° C. Reflux the sample for 30 minutes.

17.6.4 Remove the sample from the hotplate/block and allow to cool.

17.6.5 Dilute the sample to 50 mL with Type I water.

17.6.6 Cap and shake the sample.

17.6.7 Filter each sample with 2.0 µm pore size plunger type filter (PTF grade) or allow to sit overnight.

17.6.8 Organize the samples in a storage container, and label the container with the batch number of the sample group.

17.7 If the sample contains particulate material that could clog the nebulizer, you may filter or centrifuge the sample if necessary.

17.8 Be advised that filtration is a common cause of contamination. If a sample is filtered, any QC associated with the sample must also be filtered. Additionally, if any sample in the batch is filtered the method blank and laboratory control sample must also be filtered.

17.9 Filters may be prepared via this method. If the filters are small enough to fit inside the 50 mL digestion tubes, they can be treated as any solid prep materials. If the filters are too big to undergo adequate digestion using the 50 mL digestion tube, a borosilicate beaker will need to be used. All reagents and standards will need to be adjusted for any extra volumes needed. All filter analyses should be discussed and the process verified with the group/team leader prior to digestion. The group leader or project manager may have to contact the client to get the full description of what is required.

17.10 If testing paint for the U.S. Consumer Product Safety Commission's (CPSC) Product Testing Program, follow the preparation instructions below:

17.10.1 For testing of wet paint, apply a think coating to a glass slide and dry completely prior to testing by heating in an oven at a recommended 105° C until weight is stable.

17.10.2 For products coated with paint or a similar surface coating, remove and digest the coating separately from the substrate material for lead content. Care should be taken to remove as little of the substrate as possible. The scraped paint should be finely divided to help in digesting.

17.10.3 Scrape and weigh approximately 5 to 20 mg of paint from the product. If it is not possible to collect this much paint, it may be necessary to combine more than one unit of such product to collect sufficient paint.

17.10.4 Prepare samples and QC using procedure in section 17.6.

17.11 If testing metal jewelry for the U.S. Consumer Product Safety Commission's (CPSC) Product Testing Program, follow the preparation instructions below:

17.11.1 If the children's metal jewelry is coated with paint or a similar surface coating, the coating shall be removed and analyzed separately from the base metal for lead content. Care should be taken to remove as little of the substrate metal as possible. Ideally, the sample should contain only identical items, not a mix of several different items. An item such as a bracelet may be broken into its component parts such as bead, hook, pendant, with those component parts individually analyzed.

17.11.2 Weigh approximately 5 to 20 mg of the paint or coating from the jewelry item. If it is not possible to collect this much paint or coating, it may be necessary to combine more than one unit of such product to collect sufficient sample.

17.11.3 Prepare paint or coating and QC using procedure in section 17.6.

17.11.4 Weigh out a 30 to 50 mg piece of children's metal jewelry. Children's metal jewelry items generally weigh several grams, and an aliquot piece (with no paint or similar surface coating) will have to be clipped from items using metal cutters. Samples should be cut into several small pieces or ground to increase the rate of dissolution. If used, grinding apparatus must be thoroughly cleaned to prevent cross-contamination.

17.11.5 Prepare children's metal jewelry and QC using the procedure in section 17.6.

18.0 PREPARATION OF STANDARDS

Documentation of standards and their preparation is maintained in AlphaLIMS in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

Hot plates/blocks are allowed to come to the proper temperature before digestions are started. The temperatures are monitored before and after a daily digestion session.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Frequency of QC

20.1.1 A matrix spike (MS) and a matrix spike duplicate (MSD) or a sample duplicate (DUP) and a matrix spike are prepped for every batch of $\leq$ 20 samples

20.1.2 A method blank (MB) and a laboratory control standard (LCS) are prepped for every batch of $\leq$ 20 samples. A laboratory control standard duplicate (LCSD) is prepared if matrix QC is unavailable or upon client request.

20.2 Makeup of QC Samples

20.2.1 Sample duplicate (DUP) is a separate aliquot taken through the prep process exactly the same as the original sample.

20.2.2 Matrix spike and/or matrix spike duplicate is a separate aliquot of the sample to which appropriate spike volumes and solutions are added. The ID numbers and volumes of the spikes are recorded in the prep logbook.

20.2.3 The method blank (MB) is a reagent blank taken through the same prep process as the samples. Teflon chips are used to approximate matrix weights of 0.5 g.

20.2.4 The laboratory control standard (LCS) is a standard performed two different ways. For DOE-ALB clients, a purchased SRM is used at approximately 0.5 g and is taken through the same process as the samples. For all other clients, Teflon chips weighted to approximately 0.5 g are used. The chips and acid solution is spiked with the appropriate spike volumes and solutions. The ID number and volumes of the spikes are recorded in the prep logbook.

20.3 Handling Out-Of-Control Situations

If sample reactions cause popping or splattering of the digestate, discontinue the prep and contact team leader or group leader.

21.0 RUN SEQUENCE

Not applicable

22.0 PROCEDURE

Refer to section 17.0, Preparation of Samples

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

Before turning off the hotplate/blocks at the end of the day, a final monitoring temperature is recorded for each plate/block that was utilized.

24.0 METHOD VARIATION

24.1 This procedure deviates from method 3050B in that sample volumes are half the method recommendations.

24.2 The ICP procedure references a modified 3050B section 7.5 procedure. The modification eliminates the use of the Whatman 41 filters, thus eliminating contamination of common minerals.

25.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

25.1 Upon completion of batch preparation, digestion data shall be entered into the AlphaLIMS Prep Logbook (refer to Appendix 1) following the guidelines in GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.

25.2 Data to be entered into the electronic logbook include analyst name, prep data and time, initial volume or weight with units, and final volume with units.

25.3 Standards and reagents may also be entered into the logbook and fall under the guidelines of GL-LB-E-015 for Control of Laboratory Standards and GL-LB-E-007 for Laboratory Standards Documentation.

25.4 Upon entry of prep data, obtain a printout of the logbook. The analyst listed on the logbook should sign and date the page near their printed initials. The logbook page is kept with the samples with which it is associated.

25.5 The entry of correct prep data is peer reviewed (correct dates, times, weights, volumes, SOP/revision, spikes, spike amounts, and reagent information, etc.) Once data are reviewed, the batch is statused to DONE in AlphaLIMS, the logbook is signed and dated by the reviewer, and the batch is ready for analysis. A copy of the prep logbook sheet is kept in the metals prep lab and is bound and given a control number when sufficient numbers of sheets are collected.

26.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 LABORATORY WASTE

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

28.0 REFERENCES

28.1 Test Method for Evaluating Solid Waste; Laboratory Manual Physical/ Chemical Methods, Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils," Revision 2, December 1996.

28.2 1992 Annual Book of ASTM Standards, Standard D1193-91, "Standard Specification for Reagent Water."

28.3 16 CFR Part 1303

29.0 HISTORY

Revision 20: Added clarification to storage temperature in section 7.0.

APPENDIX 1: SAMPLE PREP LOGBOOK

(For illustrative purposes only)

Prep LogBook

Analyst:	AJM			Type	Sample Id	Lot Id	Spike Amount	Spike Units
Batch:	10607			LCS	1000023482	5385	.25	mL
Prep Date:	07-FEB-2000 14:00			LCS	1000023482	5386	.25	mL
Lab SOP:	GL-MA-E-013			MS	1000023455	5385	.25	mL
				MS	1000023455	5386	.25	mL
				MSD	1000023454	5385	.25	mL
				MSD	1000023454	5386	.25	mL

Type	Sample Id	Parent Sample	Method	Initial Wt.	Final Volume	Prep Factor	Comments	Matrix
MB	1000023451		200.2/200.7 Full List For QC	50mL	50mL	1		Water
SAMPLE	214126001		200.2/200.7 Selenium	50mL	50mL	1		Waste Water
LCS	1000023482		200.2/200.7 Full List For QC	50mL	50mL	1		Water
SAMPLE	214126002		200.2/200.7 Selenium	50mL	50mL	1		Waste Water
SAMPLE	214126003		200.2/200.7 Selenium	50mL	50mL	1		Waste Water
SAMPLE	214126004		200.2/200.7 Selenium	50mL	50mL	1		Waste Water
SDILT	1000023483	214126004	200.2/200.7 Full List For QC	50mL	50mL	1		Water
MSD	1000023454	214126004	200.2/200.7 Full List For QC	50mL	50mL	1		Water
MS	1000023455	214126004	200.2/200.7 Full List For QC	50mL	50mL	1		Water

General Engineering Laboratories Page#_____

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

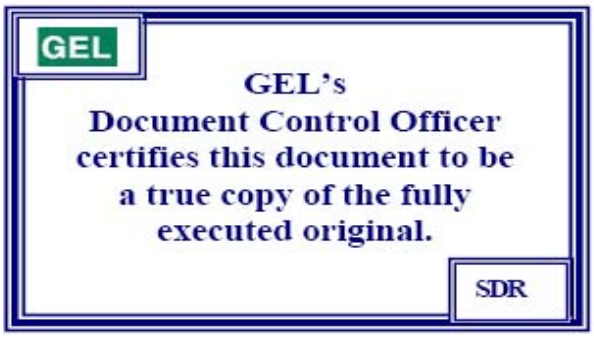
FOR

DETERMINATION OF METALS BY ICP-MS

APPLICABLE TO METHODS:
EPA Method 200.8
EPA SW-846 Method 6020 and 6020A

(GL-MA-E-014 REVISION 22)

PROPRIETARY INFORMATION



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1.0 STANDARD OPERATING PROCEDURE FOR DETERMINATION OF METALS BY ICP-MS

2.0 METHOD CODES

2.1 EPA SW-846 Method 6020

2.2 EPA SW-846 Method 6020A

2.3 EPA Method 200.8

2.4 ASTM D4698-92 Total Dissolution

3.0 METHOD OBJECTIVE AND PURPOSE

This standard operating procedure (SOP) describes the determination of metals using a Perkin Elmer ELAN ICP-MS Model 6100 Spectrometer or a Perkin Elmer ICP-MS Model 9000 Spectrometer. Prior to analysis, samples must be digested using appropriate sample preparation methods (such as Methods 3005, 3010, 3050, or 200.2) and other applicable requests.

4.0 METHOD APPLICABILITY AND METHOD SUMMARY

4.1 Analytes: Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Tl, U, ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U, isotopic U, V, and Zn. As well as client specific elements such as Os, Au, Pt, Pd, Cs, B, P, Sc, Ti, Ge, Sr, Zr, Hg and Th and others as needed.

4.2 Applicable Matrices: These methods are applicable to the determinations of any of the analytes listed above for various matrices including waters, oils, soils, sludges, biological tissues, Toxicity Characteristic Leaching Procedure (TCLP) extracts and other more unusual types of sample which are generally classified as a miscellaneous matrix.

4.3 General Method Summary: After the samples are prepared in accordance with the sample preparation SOP, they are analyzed by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) as follows:

4.3.1 The instrument is calibrated with a minimum of two calibration points for each element to be analyzed. The points consist of a calibration blank solution to define the lower calibration point and at least one standard calibration solution at the analyte concentrations to define the higher calibration point(s). A correlation coefficient of 0.995 or better (0.998 or better for SW-846 Method 6020A) is required for each analyte if multiple standards are used or the instrument is recalibrated for the analyte of interest.

4.3.2 Prepared client samples and numerous check standards and quality control samples, identified in Section 22.1 are then analyzed. The check standards and quality control samples are used to determine the quality and acceptability of the analytical data.

4.3.3 Continuing Calibration Verification standards (CCV) and Continuing Calibration Blanks (CCB) are analyzed a minimum of every 10 samples to ensure that the instrument is continuing to perform correctly. For 6020A, low level continuing calibration verification standards are analyzed in conjunction with the CCVs and CCBs.

4.4 Method Codes: Analyses must conform to SW-846 Method 6020, SW-846 Method 6020A, EPA Method 200.8, and/or customer contract specifications.

4.5 Radiochemistry conversion calculations for the uranium isotopes are included in Appendix 4.

5.0 METHOD SCOPE AND PERFORMANCE CHARACTERISTICS

5.1 Calibration Range: The range of concentrations between the calibration blank, typically 0, and that of the highest calibration standard for each analyte. Calibration standards vary according to method and equipment. A minimum of two, a blank and value standard, are required.

5.2 Linear Dynamic Range (LDR) standards are analyzed with each calibration. For ICP-MS analyses, the upper limit of the linear range for each analyte must be determined by observing the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range.

5.3 The linear calibration range that may be used for the analysis of samples should be judged by the analyst from the resulting data. The instrument is calibrated. The target LR should be prepared and analyzed. The sample results must fall within ± 10% of the target value. This LR value is entered into the instrument's software. Any hits below this value will be valid. Hits at or above this value will be flagged by the system and must be diluted to fall within the linear dynamic range.

5.4 Instrument Detection Limits (IDLs) in µg/L are determined by calculating the average of the standard deviation of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined at least every three months.

5.5 Method Detection Limit (MDL) studies for each analyte are performed and/or verified at least annually. These studies are conducted and calculated in accordance with SW-846, Chapter 1, paragraph 5.0, and GL-LB-E-001 for the Determination of Method Detection Limits. The relevant quantitation limits are established based on the most current MDL study. The current MDLs are maintained and can be found in the AlphaLIMS database.

5.6 Method Precision: To assure analytical precision of methods used, Laboratory Control Samples (LCS) are analyzed with each batch. LCS duplicates are analyzed with each batch when requested.

5.7 Method Bias (Accuracy) is determined by calculating recoveries of LCS of a similar matrix.

5.8 If uncertainty and total propagated uncertainty measurements are needed, they may be determined using GL-QS-E-014 for Quality Assurance Measurement Calculations and Processes.

6.0 DEFINITIONS

6.1 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

6.2 Analysis Date/Time: The date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

6.3 Analytical Sample: Any solution of media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification (ICV), initial calibration blank (ICB), continuing calibration verification (CCV), and continuing calibration blank (CCB).

6.4 Calibration Standard (CAL): A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.5 Continuing Calibration Blank (CCB): An aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

6.6 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added. The CCV is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during the sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentrations.

6.7 Contract Required Detection Limit (CRDL): Minimum level of detection acceptable under the client project requirements or CLP Statement of Work.

6.8 Control Limits: A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

6.9 Correlation Coefficient: A number (r) that indicates the degree of dependence between two variables (concentration-absorbance). The more dependent they are, the closer the value to one. Determined on the basis of the least squares line.

6.10 Data Qualifiers: The following qualifiers should be used in order to identify analytical situations that might need additional information stated in narrative before the release of the data.

U - Non-Detect. Below the Instrument or Method Detection Limit (depending upon specific project requirements)

B - Sample concentration value is between the MDL (or IDL) and the CRDL or analyte was detected in the Method Blank (Client Specific)

J - Sample concentration is between the MDL (or IDL) and the CRDL-client specific qualifier.

Blank - Concentration value is above the CRDL

* - An RPD value in the duplicate sample is out of criteria

N - A percent recovery value in the spike sample is out of criteria

E - A percent difference in the serial dilution sample is out of criteria because of the presence interference.

6.11 Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

6.12 Initial Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination.

6.13 Initial Calibration Verification (ICV): A solution of method analytes of known concentrations. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

6.14 Instrument Performance Check Solution (IPC): A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

6.15 Interferants: Substances that affect the analysis for the element of interest.

6.16 Internal Standard: Pure analyte(s) added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes.

6.17 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

6.18 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

6.19 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.20 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified measured and reported with 99% confidence that the analyte concentration is greater than zero.

6.21 Serial Dilution: The dilution of a sample by a known factor. When corrected by the dilution factor, the diluted sample should agree with the original undiluted sample within the specified limits. Serial dilution may reflect the influence of interferants.

6.22 Spike (Matrix Spike or Post Spike): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or PS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS or PS corrected for background concentrations.

6.23 Limit of Detection (LOD): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reliably detected.GEL has established LOD = 2 x MDL.

6.24 Limit of Quantitation (LOQ): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reported with a specific level of confidence. The LOQ is set at or above the concentration of the lowest initial calibration standard. The laboratory must empirically demonstrate precision and bias at the LOQ. The LOQ and associated precision and bias must meet client requirements and must be reported. GEL uses the following guidance (LOD < LOQ):

When LOD < PQL, PQL = LOQ

When LOD > PQL, LOQ is raised to next lowest calibration standard.

6.25 Practical Quantitation Limit (PQL): The PQL is typically at or above the lowest point on an acceptable initial calibration curve. It may also be determined by multiplying the MDL by approximately 2 to 10. Concentrations of a target analyte determined to be greater than its PQL are defined as quantitative results. This limit is not used in DoD ELAP reporting.

6.26 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS and PSD.

6.27 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

6.28 10% Frequency: A frequency specification during an analytical sequence allowing for not more than 10 analytical samples between required calibration verification measurement, as specified by the EPA methodology or CLP Statement of Work.

7.0 INTERFERENCES TO THE METHOD

7.1 Chemical interferences are minimal in ICP-MS Spectroscopy because the extremely high energy of the plasma breaks nearly all the chemical bonds. However, ICP-MS analysis is subject to the following three types of interferences:

7.1.1 Physical interferences are those physical properties of a sample solution that prevent their introduction to the plasma with efficiency equal to that of the calibration standards. This type of interference can be corrected via the bias correction calculation in SW-846, Chapter 1, paragraph 5.0, through the use of an internal standard in accordance with the instrument operating manual or by diluting the sample in reagent blank solution until the percent recovery falls with method guidelines.

7.1.2 Isobaric elemental interferences are caused by isotopes of different elements that form singly or doubly charged ions of the same nominal mass-to-charge ratio and that cannot be resolved by the mass spectrometer. If analytical isotopes are selected that may have an isobaric interference, then all data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest.

7.1.3 Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom that have the same nominal mass-to-charge ratio as the isotope of interest, and that cannot be resolved by the mass

spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified and are listed in Method 200.8, Table 2 together with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes or sample prep procedures, appropriate corrections must be made to the data.

8.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

8.1 PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

8.2 WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

8.3 Sample digestates are not extremely volatile or spontaneously combustible, but they are normally acidic and should be handled with care. Small spills may generally be wiped up with paper towels that can be disposed of in the trash. Larger spills may require the use of a mop, and the mop head may have to be disposed of as potentially hazardous waste in accordance with the Laboratory Waste Management Plan (GL-LB-G-001). If the spilled digestates begin any obvious fuming or reacting, pour a generous amount of the acid neutralizer, which is located in each lab, onto the spill before attempting to clean it up.

8.3.1 Gloves should be worn to avoid skin contact with digestate during clean-up.

8.3.2 Eye protection is required when handling samples and an eyewash station is located in each analysis lab.

8.3.3 Do not persist in cleaning up a spill in the presence of strong fumes. Move out of the area, try to isolate the area and notify your supervisor immediately.

8.4 Handling radioactive samples requires the use of gloves, a lab coat or an apron in addition to eye protection. Refer to GL-RAD-S-004 for Radioactive Material Handling

8.5 These instruments use high voltage electricity and therefore, should be shut completely down any time electronic components may be exposed to personnel or any liquids.

8.6 Wear eye protection with side shields while performing procedures in the lab.

8.7 All chemicals and samples should be treated as potential health hazards, and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

8.8 Personal protective equipment

8.8.1 Gloves are required when handling the chemicals in this procedure.

8.8.2 Work under a hood when using concentrated acids.

8.9 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

8.9.1 Wear a lab coat when working with radioactive samples.

8.9.2 Prohibit admittance to immediate work area.

8.9.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

8.9.4 Post signs indicating radioactive samples are in the area.

8.9.5 Take swipes of the counter tops upon completion of work. Deliver those swipes to the nearest swipe count box.

8.9.6 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

8.10 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.10.1 Segregate solid wastes from liquid wastes in the satellite area containers.

8.10.2 Segregate oil wastes from water-soluble wastes in the satellite area containers

8.11 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

8.12 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

8.13 Fire escape routes are posted in the lab; all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

9.0 CAUTION WARNINGS

9.1 Because they can be health hazards, the exhaust gases from the plasma and vacuum systems must be eliminated through the laboratory's ventilation duct, which is attached to the instrument's exhaust vent. If inadequate ventilation occurs, pump-fluid vapor, ozone, and other toxic products of combustion can accumulate in the laboratory and cause bodily harm. Hydrofluoric acid (HF) fumes, if inhaled, extensively burn lung tissue. Ensure that the exhaust system established at installation continues to operate effectively.

9.2 Prepare sample and transfer acids using a hood to avoid fumes.

9.3 Store and prepare sample away from the instrument to minimize corrosion.

9.4 Clean up any spills quickly.

9.5 The drain vessel contains the spray chamber's effluent, which can be toxic. Corrosion of the vessel and connecting tube can result in leaks that damage the instrument or cause bodily harm.

9.5.1 Use the capped plastic drain vessel that was provided with the instrument. Never use glass.

9.5.2 Place the drain vessel on the instrument table below the peristaltic pump where the container is easy to check.

9.5.3 Check the drain vessel frequently. Empty it before it is three-fourths full.

9.5.4 Check the tubing and vessel for deterioration. If the tubing becomes brittle or cracked, replace it. Organic solvents cause more rapid deterioration than aqueous solutions.

9.6 The torch and interface remain hot after the plasma is turned off. Do not touch the torch box or interface cones for 10 minutes after the plasma has been shut off.

9.7 High voltages and radio frequencies are potential hazards of the ICP-MS. Shut the instrument down completely before removing any of the outside panels (to clean air filters, replace a fuse, etc.).

9.8 When changing the rotary pump oil, remember that the pump oil may be hot. The oil can cause a burn if allowed to contact the skin.

10.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTS

10.1 Apparatus and Equipment

10.1.1 Replacement special glass parts for the ICP-MS such as quartz torch bodies, injector tips and spray chambers may be ordered from a qualified vendor through the GEL Purchasing Agent and Inorganic Group Leader.

10.1.2 Replacement ICP-MS interface parts such as sampling cones, skimmer cones and ion-optics can be purchased from a qualified vendor through the GEL Purchasing Agent and Inorganic Group Leader.

10.1.3 Consumable materials such as tubing are often attainable from various scientific product companies. The GEL Purchasing Agent can help to find the best prices. These items may also be ordered from the instrument manufacturer if necessary. All orders must be placed through the GEL Purchasing Agent.

10.2 Reagents, Chemicals, and Standards

10.2.1 Reagents: Refer to Reagent Logbook

10.2.2 Standards: Refer to GL-LB-E-007 for Laboratory Standards Documentation and GL-LB-E-015 for Control of Laboratory Standards.

10.2.3 Other Chemicals: Additional compounds, surfactants, oils, cleaning agents, etc., may be routinely ordered through the GEL Purchasing Agent.

10.3 Instrumentation

10.3.1 Perkin Elmer ICPMS ELAN Model 6100 with IBM compatible PC, Monitor, Printer.

10.3.2 Perkin Elmer ICP-MS ELAN Model 9000 with IBM compatible PC Monitor, Printer.

10.3.3 CETAC Model ASX-500 Autosampler (PE 6100)

10.3.4 CETAC Model ASX-510 Autosampler with accessory autodiluter (PE 9000)

10.3.5 Neslab CFT75 recirculating bath provides cooling to the ICP-MS (PE 6100/ PE 9000)

10.3.6 CETAC ASXpress Rapid Sample Introduction System

11.0 SAMPLE HANDLING AND PRESERVATION REQUIREMENTS

11.1 Aqueous samples should be preserved with nitric acid to a pH of < 2 prior to receipt by the analyst. Solid samples should be kept at 0° ≤ 6° C prior to digestion.

11.2 Refer to GL-SR-E-001 for Sample Receipt, Login and Storage.

12.0 SAMPLE PREPARATION TECHNIQUES

12.1 All samples except drinking water with Turbidity < 1 NTU and samples specifically exempted by contract, are prepared in accordance with the following SOPs:

12.1.1 GL-MA-E-006 for Acid Digestion of Total Recoverable or Dissolved Metals in Surface and Groundwater Samples for Analysis by ICP or ICP-MS (USEPA SW-846 Method 3005A)

12.1.2 GL-MA-E-008 for Acid Digestion of Total Metals in Aqueous Samples and Extracts for Analysis by ICP or ICP-MS (USEPA SW-846 Method 3010A)

12.1.3 GL-MA-E-009 for Acid Digestion of Sediments, Sludges and Soils (USEPA SW-846 Method 3050B)

12.1.4 GL-MA-E-021 for Total Digestion of Sediment Samples for Analysis by ICP or ICP-MS (ASTM D4698-92)

12.2 Additional filtration may be required to prevent clogging of sample introduction system.

12.3 All sample preparation records are kept in the digestion logbooks in accordance with GL-MA-E-012 for Inorganic CLP Sample Digestions.

12.4 Sample spills should be handled as stated in Section 8.3.

13.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

13.1 Routine Preventative and Special Operational (Failure)

13.2 Routine Preventative Maintenance (PM) Procedures are done as follows:

Frequency	Procedure
When Needed	Clean nebulizer tip after use. Replace peripump sample introduction tubing. Change pump hoses on drain systems. Check drain waste collection containers, and empty as necessary. Check Neslab water level and add water if required. Clean/replace interface cones. Clean/replace nebulizer. Clean/replace torch. Check/replace water filter.
Quarterly	Change oil in interface rotary pump (or as needed). Clean ion lenses 4-6 months (or as needed).
6 Months	Clean air filters.
12 months	Change pump oil in backing rotary pump. Evaluate/replace EM (electron multiplier)

13.3 Non-Routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

13.3.1 If the instrument will not function properly see the trouble shooting section in the appropriate Maintenance Manual.

13.3.2 If the analyst is unable to determine cause/fix instrument, the GEL Service Engineer is called, and if needed, the manufacturer's customer support number may be found in the owner's maintenance manuals.

13.4 Refer to ICP-MS Maintenance Logbook for routine records. Service call records are also available.

14.0 PREPARATION OF STANDARD SOLUTION AND QUALITY CONTROL SAMPLES

14.1 Source standards records are recorded in AlphaLIMS.

14.2 Recommended Suppliers: Refer to source log and use Approved Vendors List maintained in Procurement.

14.3 Standards are receipted, labeled, prepared and stored in accordance with the GL-LB-E-007 for Laboratory Standards Documentation, and GL-LB-E-015 for Control of Laboratory Standards.

15.0 INSTRUMENT CALIBRATION

15.1 Samples may be analyzed manually or automatically.

15.1.1 Tuning for each instrument is performed daily according to the following directions and criteria.

15.1.2 PE 6100/ 9000: Aspirate a tuning solution consisting of 10 µg/L each of ^{9}Be, ^{24}Mg, ^{59}Co, ^{115}In and ^{208}Pb. Perform 5 replicates. Manufacturer's recommended tune criteria are as follows:

Parameter	Starting Point

^{9}Be	± 0.10 amu
^{24}Mg	± 0.10 amu
^{59}Co	± 0.10 amu
^{115}In	± 0.10 amu
^{208}Pb	± 0.10 amu
Ba^{++} net intensity mean	< 0.05 or 5%
CeO net intensity mean	< 0.03 or 3%
Be, Mg, Co, In, Pb net intensity RSD	< 5%
Resolution at 10% peak height	< 0.9 amu

15.1.3 Conduct additional tuning procedures as specified by client contract or other methodology.

15.1.4 If any of the preceding tune criteria does not meet the recommended requirements, investigate the problem, correct the situation, and reanalyze the tune sequence.

15.1.5 Standardization: Standardization is required on a daily basis.

15.2 Internal standards are used as appropriate for the analytes of interest. The internal standards are made in the appropriate acid and contain varying concentrations of such elements as ^{6}Li, ^{45}Sc, ^{74}Ge, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, ^{175}Lu, ^{181}Ta, ^{209}Bi. The internal standard solution is mixed in-line with the sample stream using a dedicated channel of the peristaltic pump. Internal standards may be added at the time of analysis as an alternative to in-line mixing. Alternate internal standards may be used to meet client needs.

15.3 Calculations are described in the instrument manual.

15.4 Calibration standards vary according to method and equipment. A minimum of two, a blank and value standard, is required.

15.5 For required quality control standards refer to Section 21.0.

15.6 For continuing calibration requirements refer to Section 21.0.

15.7 For what to do when initial or continuing calibrations fail to meet requirements, refer to Section 21.0.

16.0 INSTRUMENT PERFORMANCE REQUIREMENTS

16.1 Before samples may be analyzed to generate reportable data, the instrument must have been tuned and calibrated. Also, the Initial Calibration Verification (ICV), which is prepared from an independent source, Initial Calibration Blank (ICB), the Reportable Detection Limit (CRDL), the Interference Check Standards (ICS-A, ICS-AB), and the Linear Range Standards (LRS) must meet the requirements stated in Section 21.2 for each analyte being reported, unless otherwise required by methodology, clients or contracts.

16.2 The instrument calibration and all continuing verification data is maintained in the printed hard copy file. The printouts are kept in chronological order by instrument.

Recent files are in the metals laboratory; older records are archived in short or long-term storage.

17.0 ANALYST AND METHOD VERIFICATION REQUIREMENTS

17.1 Analyst training is conducted and certified in accordance with the GL-HR-E-002 for Employee Training.

17.2 Method performance is verified by the conductance of MDL studies in accordance with GL-LB-E-001 for The Determination of Method Detection Limits, and by the evaluation of LCS and LCS duplicates for each batch of samples.

18.0 ANALYSIS PROCEDURES AND INSTRUMENTAL OPERATION

18.1 All samples are introduced in a set measured quantity, via a peristaltic pump, through a nebulizer into a spray chamber and carried with argon gas through the Radio Frequency (RF) field to generate analyte ions that are selected and measured by the quadruple mass spectrometer.

18.2 Run Sequence

18.2.1 Instrument Calibration executed in accordance with Section 16.

18.2.2 Initial Calibration Verification steps include:

18.2.2.1 ICV
18.2.2.2 ICB
18.2.2.3 CRDL (low level ICV for 6020A)
18.2.2.4 CRI (if required)
18.2.2.5 ICS-A
18.2.2.6 ICS-AB
18.2.2.7 CCV
18.2.2.8 CCB
18.2.2.9 LRS
18.2.2.10 CCV
18.2.2.11 CCB

18.2.3 Sample Run (10 samples or less)

18.2.4 Continuing Calibration Verification:

18.2.4.1 CCV
18.2.4.2 CRDL (for SW-846 Method 6020A only and analyzed at end of batch).
18.2.4.3 CCB

18.2.5 Repeat steps 18.2.3 and 18.2.4 until end of run or verification is out of specification. When the latter occurs, repeat step 18.2.1 and continue with steps 18.2.2 and 18.2.3.

18.2.6 Repeat steps 18.2.3 through 18.2.5 until the end of the run or verification is out of specification. When latter condition occurs, repeat 18.2.1 and continue sequentially through 18.2.7.

18.2.7 Final Verification Steps

18.2.7.1 CRI (if required)

18.2.7.2 ICS-A (if required)

18.2.7.3 ICS-AB (if required)

18.2.7.4 CCV

18.2.7.5 CRDL (for SW-846 Method 6020A only)

18.2.7.6 CCB

NOTE: Method 6020A only requires there to be a closing low level continuing calibration standard (CRDL). For ease of analysis, the CRDL standard can be analyzed every 10 samples.

18.3 For data storage refer to Section 24.1.

18.4 General operation of the instrument.

18.4.1 The Perkin Elmer Model 6100 and Perkin-Elmer Model 9000 are inductively-coupled argon plasma mass spectrometers. The ICP-MS is capable of determining analytes from m/z = 6 (Li) through m/z = 238 (U). The operating software of the system is based on Microsoft Windows.

18.4.2 Set-up

18.4.2.1 Attach the nebulizer argon line to the quick-connect on the nebulizer's argon tube and ensure that it is tightly in place.

18.4.2.2 Attach the nebulizer and endcap to the spray chamber.

18.4.2.3 Attach sample pump tubing to the front peristaltic pump (various sizes may be used depending on need), and attach feed end to endcap.

18.4.2.4 If running manually, place the suction end of the sample tubing in acidified rinse water; otherwise attach it to the autosampler.

18.4.2.5 Ensure that drain hose is connected to the spray chamber and properly plumbed through the drain peristaltic pump.

18.4.3 Run Procedure

18.4.3.1 Refer to the respective owner's manuals for specific instructions for tuning, sequence loading, and method development on each instrument; ELAN 6000/6100 software guide; ELAN 9000 software guide.

18.4.3.2 The owner's manual for each instrument is located in the ICP-MS laboratory.

18.4.4 Typical Analysis Problems

18.4.4.1 Sample Overrange: If a requested element is overrange for a sample, it is necessary to dilute the sample and rerun. Dilution factors must be taken into account when reporting final values.

18.4.4.2 Interferences: Although the ICP-MS can compensate for many interferences with appropriate correction factors, unpredicted interferences may still occur. If the analyst suspects this, then

the sample should be diluted and rerun. Dilution factors must be taken into account when reporting final values.

18.4.4.3 Torch/Sample Introduction Drift: Various changes in torch plasma conditions or sample introduction can have a great effect on the detector counts. They must be corrected or the instrument must be re-standardized.

18.4.4.3.1 If oils or solid samples have been run, and the CCV is not acceptable, allow the instrument to rinse 15 to 20 minutes. If CCV is then acceptable, reanalyze the samples and continue. If the CCV again fails its requirements, re-standardize as necessary.

18.4.4.3.2 If sample introduction drift is suspected, check peristaltic pump tubing for collapse and replace as needed.

18.4.4.3.3 Tubing connections may become loose allowing air to bubble into the sample path. Tighten or replace tubing. If necessary, seal with Parafilm.

18.4.4.4 Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 100 times above the IDL for non-DOE Clients, 100 x the MDL for DOE-Alb Clients, and 50 times the LOQ for DoD QSM in the original sample), an analysis of a five fold (1 + 4) dilution should agree within 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

18.5 Power switches and auxiliaries

18.5.1 Each instrument has one main power switch. Refer to the respective maintenance manuals for the location (ELAN 6100 and ELAN 9000 Hardware Guide). This switch remains on except while servicing the instrument.

18.5.2 The computer system has 3 power cords all connected to a switchable power strip: the computer main, the monitor and the printer power cords. The power to these is left on while the instrument is not in use for short times (e.g. overnight).

18.5.2.1 The main computer switch is on the front of the CPU.

18.5.2.2 The ***monitor switch*** is on the front of the monitor itself.

NOTE: If the computer is left in use without an analyst present for an extended period of time, turn the monitor off to prevent screen burn-in.

18.5.2.3 The ***printer switch*** is on the lower front of the printer. Refer to printer manual for operating instructions.

18.5.3 Argon gas is provided through a manifold from the storage tank. Under normal conditions a constant argon flow is available.

18.5.4 Start-up

18.5.4.1 Before starting, ensure that the power is on and the vacuum system is switched on.

18.5.4.2 The owner's manual for each instrument provides specific instructions for ignition of the plasma (refer to Section 18.5.1 for software manuals).

18.5.4.3 The plasma should be steady and should not flicker.

18.5.4.4 Once ignition has occurred, levels may be adjusted; it is best to set levels appropriate to the method by loading or editing an appropriate tune file and allow 15 to 30 minutes for warm-up.

18.5.5 Shutdown

18.5.5.1 When the plasma is off, the instrument is either in Standby or Shutdown mode. The ICP-MS should be completely shut down only in case of major maintenance, relocation of the instrument, or when the lab is closed for an extended time.

18.5.5.2 Daily shutdown (if required): Refer to the respective owner's manual shutdown procedures (Refer to Section 18.5.1 for software manuals.)

18.6 Sample quantity requirements are approximately 5 mL for each run with the nebulizer. If out of specification or range, further sample may be necessary for reruns.

18.7 The autosampler can be used by attaching introduction tube to sample introduction system, and rinsing tubing to peristaltic pump and following autosampler table set-up and procedures. To define a sequence, refer to the appropriate software manual (refer to Section 18.5.1).

19.0 CALCULATIONS AND DATA REDUCTION METHODS

19.1 Any dilutions, concentrations, or preparation factors must be taken into account prior to reporting data to a client.

$$\text{Relative Percent Difference} = \frac{100 * |\text{Sample 1 Value} - \text{Sample 2 Value}|}{\frac{(\text{Sample 1 Value} + \text{Sample 2 Value})}{2}}$$

$$\text{Matrix Spike Recovery} = \frac{100 * (\text{Spike Value} * \text{DF} * \text{PF} - \text{Sample Value} * \text{DF} * \text{PF})}{\text{Spike Nominal Concentration} * \text{DF} * \text{PF}}$$

$$\text{Post Spike Recovery} = \frac{100 * (\text{Spike Value} - \text{Sample Value})}{\text{Spike Nominal Concentration}}$$

$$\text{LCS Recovery} = \frac{100 * (\text{Sample Value} * \text{DF} * \text{PF})}{\text{Nominal Concentration} * \text{DF} * \text{PF}}$$

Where: Sample Value = instrument reading for the sample
Spike Value = instrument reading for the spiked sample
DF = Dilution Factor
PF = Preparation Factor

Relative Error Ratio (RER) 2 sigma equation:

$$RER = \frac{|\text{Sample Activity} - \text{Duplicate Activity}|}{\sqrt{(\text{Sample 2 sigma TPU}/1.96)^2 + (\text{Duplicate 2 sigma TPU}/1.96)^2)}} = \leq 3$$

NOTE: Activity calculations can be found in Appendix 4 of this SOP. Two sigma TPU calculations can be found in section 6.0 of SOP GL-QS-E-014.

19.2 Care must be taken that the correct units are being employed.

20.0 DATA RECORDING

20.1 ICP-MS data are generally stored to the hard disk drive of the ICP-MS computer system and printed at the instrument as each sample is analyzed.

20.2 ICP-MS samples are generally analyzed in three replicates (minimum of 2) and the reported value is the average of the replicates. The data for individual replicates is stored in the computer.

20.3 Data are processed locally by manual or programmable procedures to eliminate unused data, to enter dilution factors, and to enter relevant conversion factors prior to uploading to AlphaLIMS.

21.0 QUALITY CONTROL REQUIREMENTS

21.1 Frequency of Quality Control Activities (also refer to Appendix 1)

21.1.1 Initial Calibration Verification (ICV) is performed immediately following each calibration and Continuing Calibration Verification (CCV) is performed after at least every 10 samples.

21.1.2 Initial Calibration Blank (ICB) is performed immediately following the ICV and Continuing Calibration Blanks (CCB) must run with each CCV.

21.1.3 An Interference Check Standard (ICS) is analyzed at the beginning of each analytical run and at least once every twelve hours (if required). Additional requirements may be specified by client contract or methodology.

21.1.4 The PQL standard is analyzed after each calibration and recommended at least every 10 samples between the CCV and CCB analyses for SW-846 Method 6020A. Method 6020A requires analysis at the end of every batch and only recommends analysis every 10 samples. This standard may also be labeled as a CRDL standard. For CLP work, the CRI is analyzed at the beginning and at the end of each analytical run, and at a minimum of two times per eight hours. For CLP analyses the CRI is analyzed at two times the CRDL or two times the IDL, whichever is greater.

21.1.5 A method blank (MB) is performed for each batch of 20 or fewer samples or per client requirement.

21.1.6 A matrix spike (MS) and a duplicate (DUP), or matrix spike and matrix spike duplicate (MSD) are analyzed for each batch of 20 or fewer samples or per client requirements.

21.1.7 A laboratory control sample (LCS) is analyzed with each batch of 20 or fewer samples. An LCS duplicate (LCS DUP) may be added if required by the client.

21.1.8 Serial dilutions or analytical spikes are analyzed to confirm the presence or absence of interferences when analyzing a new matrix type. The serial dilution is generally performed at a 5x of the test sample.

21.1.9 When performing CLP analyses, the serial dilution must be performed on a sample from each SDG of 20 samples or less.

21.1.10 When performing CLP analyses, a post-spike must be performed on the original sample when the matrix spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added. Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x the CRDL, whichever is greater. Post-spike analysis is not required for silver.

21.2 Acceptance Limits (also refer to Appendix 2)

21.2.1 ICV results must be between 90% and 110% of the true values for work under EPA SW-846 Method 6020A or EPA Method 200.8. CCV results must be between 90% and 110% of the true values for work under EPA Method 200.8 or SW846 Method 6020A.

NOTE: The ICV is the second source standard and may be used as the CCV as it also will show calibration verification.

21.2.2 ICB and CCB results must have an absolute value less than the Reporting Limit (RL). For DoD QSM analysis, the absolute value must be less than the LOD. If this is not the case, the reason for the out-of-control condition must be found and corrected, or any data reported must be 10 times greater than the absolute value for the element or less than the RL.

21.2.3 Interference Check Sample results must be monitored at the beginning of an analytical run or once every 12 hours, whichever is more frequent, for work under SW-846. The ISCA and ICSAB must recover 80-120% the reporting level for the spiked analysis and must have an absolute value less that 2x the reporting level for the non-spiked analyte. For DoD QSM, the ICSA must have an absolute value of less than the absolute value of the LOD analytes.

21.2.4 The Linear Range Standard (LRS) is analyzed within the calibration verification read back and must fall between 90% and 110% of the true values. Meeting these criteria allows target analyte concentration to be reported up to the LRS concentration thus extending the calibration range of the instrument. Any sample concentrations above the LRS concentration will be diluted to fall below the concentration of the linear calibration range standard.

21.2.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 30% and 120% (or 70% and 130% for SW-846 Method 6020A) of the intensity of that internal standard in the initial calibration then the sample must be diluted five fold (1 + 4) and reanalyzed with the addition of appropriate amounts of internal standards. The intensity levels of the internal standards for the calibration blanks (ICB and CCB) and instrument check standards (ICV and CCV) must agree within $\pm$ 20% of the intensity level of the internal standard of the original calibration solution. For work done under EPA Method 200.8, the internal standard responses of any one internal standard must not deviate more than 60% to 125% of the original response in the calibration blank. Five internal standards (^{45}Sc, ^{74}Ge, ^{115}In, ^{175}Lu, and ^{181}Ta) are used to cover the mass ranges reported. Refer to Appendix 3 for list of internal standard/ analyte associations. Other exotic analytes may be used as needed. Refer to Section 15.2.

21.2.6 Method blank results must be lower than the PQL or less than 10% of the determined value of all samples in the batch. When performing work under EPA Method 200.8, if LRB (laboratory reagent blank) values are 10% or more of the analyte level determined for a sample or are 2.2 times the analyte MDL, then fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained. For DoD QSM work, the absolute value must be less than ½ RL or less than 10% of the determined value of all samples in the batch. Al, Fe, Mg, Ca, Na, and K must be less than the RL.

21.2.7 LCS results, and LCS duplicate (if performed), must be within process control limits as established by Statistical Process Control, manufacturer's certification, or method requirements.

21.2.8 Matrix spikes with recoveries between 75% and 125% suggest the absence of interference for work under EPA Method 200.8. Matrix spikes with recoveries between 75% and 125% suggest the absence of interference for work under SW-846 Method 6020 and SW-846 Method 6020A. Matrix spikes with recoveries between 80% and 120% suggest the absence of inteferences for work under DoD QSM.

21.2.9 The relative percent difference (RPD) between a sample and a sample duplicate should be within ± 20% if the analyte concentration in the sample or duplicate is greater than 5 times the RL. If either the sample or duplicate concentration is less than 5 times the RL, the results should agree within the absolute value of the RL. Results less than the MDL or IDL are not evaluated. The relative error ratio (RER) between a sample and a sample duplicate should be $\leq$ 3%.

21.2.10 Serial dilution results that agree within 10% of the original analytical results, if the original results are greater than 100 times the instrument detection limit or greater than 50 times the LOQ, suggest the absence of interference.

21.2.11 Post spikes with recoveries between 75% and 125% under EPA Method 200.8 and SW-846 Method 6020, and between 80% and 120% under SW-846 6020A suggest the absence of interference.

21.3 Out-of-Control Situations

21.3.1 ICV and/or CCV failure requires recalibration of the instrument and/or preparation of new standard solutions. Samples analyzed prior to or after calibration verifications that are not acceptable for required analytes must be reanalyzed. An ICV or CCV that has failed may be rerun once only if there is an attributable cause known to have affected the CCV only and not the previous samples. Examples of an acceptable cause may be a sample tip out of solution during analysis, an incorrectly prepared CCV, or obvious carryover in the CCV from a very high sample immediately prior to the CCV. If a CCV is reanalyzed, the data must be lined through, initialed and dated, and the reason for the rerun must be documented on the raw data. In addition, corrective action should be taken to eliminate the cause of the initial CCV failure to prevent future occurrence.

21.3.2 ICB and CCB failure requires recalibration of the instrument and/or calibration blank solution to be remade. The CCB is acceptable if the level of analyte in the corresponding sample(s) is 10 times greater or less than the PQL for the failing element. For DoD QSM work, the absolute values must be less than the LOD or less than 10% of the determined value of all samples in the batch.

21.3.3 ICS failure requires that the instrument be re-calibrated or the interferences be corrected, via recalculation, of Interelement Correction Factors so that the ICS can be read within the required limits before samples are analyzed. ICS failure at the end of an analysis period will require that the samples' ICSA run for the affected analyte(s) during that period to be reanalyzed. The ICSA and ICSAB must recover 80-120% for the spiked analytes and must have an absolute value less than 2x the reporting level for the non- spiked analytes. For DoD QSM, the ICSA must have an absolute value of less than the LOD for the non-spiked analytes.

21.3.4 LRS failures limit the reportable calibration range to the high standard in the calibration curve. Any sample concentration that falls above the high calibration standard will be diluted to fall within the calibration range.

21.3.5 Internal Standard failure requires one or more of the following: five-fold dilution of the sample, correction of the problem, termination of analysis, recalibration of the instrument, and/or reanalysis of the affected samples depending on whether the failure is due to the samples or the instrumental drift.

21.3.6 Method blank results higher than the PQL and greater than 10% of any sample value in that batch that has concentrations above the PQL require that batch be redigested and reanalyzed. If the method blank results are less than -2x PQL there may be significant interference, calibration, or contamination problems with the sample, instrument, or calibration

standards that must be resolved before the batch can be analyzed. For DoD QSM work, the absolute value must be less than ½ RL or less than 10% of the determined value of all samples in the batch. Al, Fe, Ca, Mg, Na, and K must be less than RL.

21.3.7 Matrix spikes, duplicates and spike duplicates are used only as indicators of method effectiveness on that sample and will not be used as acceptability criteria for the process, unless a special requirement of the client.

21.3.8 LCS and/or LCS duplicate results outside of established acceptance limits require the batch to be redigested and reanalyzed.

21.3.9 When analytical results suggest the presence of interference, one of the methods listed in Section 18.4.4.2 should be employed.

21.3.10 The CRDL standard should be evaluated, but no action is required if the results fall outside of the 70-130% advisory window. For DoD QSM, the CRDL standard must be 80-120% of the true value or recalibration is required. For SW-846 Method 6020A, the CRDL standard must be 70-130% of the true value or recalibration is required. This also includes the low level continuing calibration verification standard analyzed every 10 samples. Sample results can be evaluated for reporting if they are at least 2x the CRDL for a given analyte.

21.4 Corrective actions taken for data not conforming to the requirements in Section 21.2 are stated in Section 21.3. If these corrective actions can be taken by the analyst prior to the acceptance of the data, then no nonconformance documentation is required. However, if these corrective actions include redigestion of the batch or sample, if the data have already been accepted, or if the corrective action requires an instrument service call, then a nonconformance and/or corrective action report should be completed. This report includes the date, person requesting the action, sample(s) or batch(s) affected, and action requested, all provided by the requester. The person taking the action will provide any pertinent comments, their signature, and the date the action is completed. The disposition of the nonconformance will then be verified by the Quality Systems specialist. These reports will be kept on file. Refer to GL-QS-E-002 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items.

21.5 Analytical data are evaluated for conformance with the requirements stated in Section 21.2 by the analyst during and/or after the analysis, but before the data are entered into AlphaLIMS. Data may be accepted or rejected by the analyst at this point or by the data reviewer(s) as stated in Section 22.0.

22.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURES

22.1 After samples are analyzed, the data must go through the review process before it can be reported out of the lab. The analyst who performed the analysis will review the raw data prior to uploading it into AlphaLIMS. The upload process may be handled by the analyst or by a data entry clerk.

22.2 After the upload is complete, an AlphaLIMS data report is generated that will be passed (along with the batch sheet, data review checklist, and the raw data) to

another analyst for a peer review. Discrepancies found in this review will be resolved before the batch data are passed and the status updated.

22.3 When this peer review is completed and all of the data are found to be acceptable, the reviewer signs and dates the batch data report and updates the status of the batch to done. If the sample is for a Federal client, the batch data report and all relevant information (dependent upon client contract) should be copied and delivered to the packaging group. If the reviewer determines that the reviewed data are not acceptable and requires additional work or correction, data are returned to the analyst or representative with an appropriate explanation.

22.4 Listed below are the data review responsibilities of the Analyst and Peer Reviewer:

22.4.1 Analyst Review is performed by the analyst who generated the data before the data are submitted for entry into AlphaLIMS. Analyst completes and attaches the run data cover sheet to the printout.

22.4.2 Peer Analyst Review is performed by an individual who did not perform the analysis but is familiar with the analytical method used and the reporting requirements. This person will review the complete data report after all data are entered, will ensure that any data entry corrections are made before data are approved, and will complete the reviewer portion of the data review check list. If the data do not meet the necessary quality requirements and need further analysis, they are returned to the analyst or representative.

22.5 A Third Review is performed for Data Packages (CLP/CLP-Like) and/or by client requirement. This review is by the Metals Team Validator or other qualified person.

22.6 Specific items that are reviewed at each level include the following:

22.6.1 Analyst Review: Before data is submitted for entry into AlphaLIMS.

22.6.1.1 All analyses in the batch are completed.

22.6.1.2 Any corrections and comments on the data are properly initialed and dated.

22.6.1.3 Proper standard identification numbers appear on the runlog to ensure traceability from the data to original source standards.

22.6.1.4 All data are complete and accurate in the AlphaLIMS data report.

22.6.1.5 Data acceptance limit criteria identified in Section 21.2 are met or an explanation given.

22.6.2 Peer Analyst Review: Before data are submitted for further review or update:

22.6.2.1 All data are complete and accurate in the AlphaLIMS Data Report.

22.6.2.2 Any exceptions or shortcomings have been sufficiently explained or corrected.

22.6.2.3 Data are reported in the proper units or an explanation is given.

22.6.2.4 Prep factor and dilution calculations by AlphaLIMS are present in the data report and AlphaLIMS calculations are correct.

22.6.2.5 LCS and Spike Recoveries and RPD calculations by AlphaLIMS are correct, and the values are within control limits.

22.7 When the data review is completed and the data have been reported out of the lab, they are bound with the batching sheet and kept on file in the lab.

22.8 The complete data review process requires the use of the Prep Log Book, Prep data report, batch sheet, AlphaLIMS data report, raw instrument data, and runlog.

23.0 DATA REPORTING

23.1 To report data after the review process has been completed:

23.1.1 Enter the AlphaLIMS program through an available terminal and select DATA MENU, BATCH ITEMS, CHANGE BATCH STATUS.

23.1.2 Enter the Batch Number and "Submit."

23.1.3 Use the down arrow key to move the cursor down the new status column to the end. Change status from REVW to DONE and "Save."

24.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

24.1 Records of the instrumental analysis, operation, and maintenance are maintained as follows:

24.1.1 Run logs are an accurate chronology of what the instrument did during a specified period of time. The logs detail the standard name or sample number for each standardization or analysis, the analyst identification, and the date and time of each analysis or standardization. This information is periodically retrieved from the instrument data files, printed in chronological sequence, and maintained as documentation of the sequence of events and as a reference for the raw data.

24.1.2 Extraction/Digestion Logs are maintained in accordance with the established procedures in the areas where these processes are performed.

24.1.3 Instrument Maintenance Logs are chronological representations of all maintenance activities involving the instrument operation. This record is kept in bound composition books and consists of the details of the action, who performed it, when it was performed, and when instrument was returned to operation.

24.1.4 Batch Sheets accompany the batch of samples through digestion and analysis and then accompany the raw analytical data through data entry and data review until the batch status is changed to "DONE" in the laboratory. This record is maintained in the Metals group files.

24.1.5 Batch Data Reports are generated to be used in the peer data review. After the batch is reviewed, corrected if necessary, and updated to "DONE," the batch data report is stored with the Batch sheet.

24.1.6 Raw Instrument Data are generated as the analyses are performed and from AlphaLIMS after the data are entered and attached to the batch sheet to be used in the data review process and kept in the Metals group files.

25.0 LABORATORY WASTE HANDLING AND DISPOSAL: SAMPLES, EXTRACTS, DIGESTATES, AND REAGENTS

25.1 Standard solutions that must be disposed are taken to the Waste Disposal coordinator for disposal in accordance with Laboratory Waste Management Plan, GL-LB-G-001.

25.2 Sample digestates are stored in the lab for a specified period of time following analysis. At this time, they are composited into a waste container that is picked up by the Waste Management Technician for proper disposal.

25.3 Radioactive Waste:

25.3.1 Samples returned to sample storage

25.3.2 Drain waste collected in the radioactive waste carboy is dumped when full into the appropriate 55 gallon drum sitting outside the ICPMS laboratory. Ultimate disposal of liquid radioactive waste done by waste management department.

25.3.3 Implements, vials, gloves, etc., are wrapped and labeled with radioactive tape and placed in the radioactive waste container in high bay area.

25.3.4 Expired Standard Solutions: Refer to Section 25.1.

26.0 REFERENCES

26.1 Perkin Elmer ELAN 9000 Hardware Guide

26.2 Perkin Elmer ELAN 6100 Hardware Guide

26.3 Perkin Elmer ELAN 9000 Software Guide

26.4 Perkin Elmer ELAN 6000/6100 Software Guide

26.5 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods. Volume 1A, USEPA SW-846, Third Edition, Revision 2, September 1994:

26.5.1 Method 6020A, "Inductively Coupled Plasma – Mass Spectrometry," Revision 1, February 2007

26.5.2 Method 6020, "Inductively Coupled Plasma – Mass Spectrometry," Revision 0, September 1994

26.5.3 Method 3005A, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy", Revision 1, July 1992

26.5.4 Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy," Revision 1, July 1992

26.5.5 Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils", Revision 2, December 1996.

26.6 USEPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry," Revision 5.4, May 1994.

26.7 2001 Annual Book of ASTM Standard: D4698-92 "Standard Practice for Total Digestion of Sediment Samples for Chemical Analysis of Various Metals."

26.8 Conversion Constants for Uranium Isotopes, Dr. Robert Litman, April 2009.

26.9 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010.

26.10 U.S. Department of Energy, Quality Systems for Analytical Services (DOE QSAS). Rev 2.6, November 2010.

27.0 HISTORY

Revision 22: Editorial corrections only.

APPENDIX 1: FREQUENCY OF QUALITY CONTROL ACTIVITIES

(For illustrative purposes only)

Frequency of Quality Control Activities

Method/ Frequency	SW-846 6020	EPA 200.8	SW-846 6020A
Calibration Std readbacks	Not required	Not required	Not required
Linear Range Std	Quarterly (may be more frequent per contract)	Quarterly (may be more frequent per contract)	Quarterly (may be more frequent per contract)
ICV	Per calibration	Per calibration	Per calibration
ICB	Per calibration	Per calibration	Per calibration
PQL/CRI (CLP)	Per calibration	Per calibration	Per calibration and at end of each analytical batch.
ICSA	Per calibration	Per calibration	Per calibration
ICSAB	Per calibration; every 12 hours after	Per calibration	Per calibration; every 12 hours after
CCV	Every 10 instrument runs	Every 10 instrument runs	Every 10 instrument runs
CCB	Every 10 instrument runs	Every 10 instrument runs	Every 10 instrument runs
Method Blank	5% or per batch	5% or per batch	5% or per batch
LCS – liquid LCS – soil	5% or per batch	5% or per batch	5% or per batch
Matrix Spikes	5% or per request	10% or per request	5% or per request
Sample Duplicates	5% or per request	5% or per request	5% or per request
Serial Dilutions	5% or per request	5% or per request	5% or per request
Matrix Spike Duplicates	5% or per request	10% or per request	5% or per request
Post-Digestion Spikes	5% or per request	10% or per request	5% or per request
Linear Range Standard (LRS)	Per calibration	Per calibration	Per calibration

APPENDIX 2: ACCEPTANCE LIMITS

Method/ Acceptance Criteria	SW-846 6020	EPA 200.8	DoD QSM	SW-846 6020A
Calibration Std readbacks	Not required	Not required	Not required	Not required
Linear Range Std	± 10% of true value	± 10% of true value	± 10% of true value	± 10% of true value
ICV	90% - 110%	90% - 110%	90%-110%	90% - 110%
ICB	< absolute value of RL	< absolute value of RL	< LOD	< absolute value of RL
PQL/CRI (CLP)	70% - 130% advisory limits only	70% - 130% advisory limits only	80%-120% or investigate and recalibrate	70% - 130% or investigate and recalibrate
ICSA	80-120% for major components; ± 2x RL for non-spiked	80-120% for major components; ± 2x RL for non-spiked	80%-120% for major compounds; ± LOD for non-spiked	80-120% for major components; ± 2x RL for non-spiked
ICSAB	80%-120%	80%-120% (may be requested)	80%-120%	80%-120%
CCV	90% - 110%	90% - 110%	90%-110%	90% - 110%
CCB	± RDL	± RDL	< LOD	± RDL
Method Blank	± RDL	± RDL	± ½ RL except for Al, Fe, Mg, Ca, Na, and K	± RDL
LCS - liquid LCS - soil	80% - 120% current SPC limits	85% - 115% current SPC limits	80%-120%	80% - 120% current SPC limits
Matrix Spikes	75% - 125%, when applicable	75% - 125%, when applicable	80%-120%	75% - 125%, when applicable
Sample Duplicates	0% - 20% when greater than 5X RL, ± RL when less than 5X RL	0% - 20% when greater than 5X RL, ± RL when less than 5X RL	0% - 20% when greater than 5X RL, ± RL when less than 5X RL	0% - 20% when greater than 5X RL, ± RL when less than 5X RL
Serial Dilutions	0% - 10% of initial raw value, when applicable	0% - 10% of initial raw value, when applicable	0% - 10% of initial raw value, when applicable (> 50x LOQ)	0% - 10% of initial raw value, when applicable
Post-digestion spikes	75%-125%, when applicable	75%-125%, when applicable	75%-125%	80%-120%, when applicable
Internal Standards	30%-120%, samples 80%-120% for ICB, ICV, CCV, CCB	60%-125% for all	30%-120%	70%-130%, for all
Matrix Spike Duplicate	0% - 20% when greater than 5X RL, ± RL when less than 5X RL	0% - 20% when greater than 5X RL, ± RL when less than 5X RL	0% - 20% when greater than 5X RL, ± RL when less than 5X RL	0% - 20% when greater than 5X RL, ± RL when less than 5X RL
Linear Range Standard (LRS)	90%-110%, or up to the high calibration standard	90%-110%, or up to the high calibration standard	90%-110%, or up to the high calibration standard	90%-110%, or up to the high calibration standard

APPENDIX 2-Cont'd

Method/ Acceptance Criteria	SW-846 6020	EPA 200.8	DoD QSM	SW-846 6020A
Sample Duplicates RER activity	≤ 3%	≤ 3%	≤ 3%	≤ 3%

APPENDIX 3: INTERNAL STANDARDS WITH ASSOCIATED ANALYTES/ISOTOPES

IS ^{45}Sc	IS ^{74}Ge	IS ^{115}In	IS ^{175}Lu	IS ^{181}Ta
^{7}Li	^{66}Zn	^{88}Sr	^{133}Cs	^{133}Cs
^{9}Be	^{67}Zn	^{90}Zr	^{135}Ba	^{135}Ba
^{11}B	^{68}Zn	^{93}Nb	^{137}Ba	^{137}Ba
^{23}Na	^{75}As	^{98}Mo	^{139}La	^{139}La
^{24}Mg	^{77}Se	^{102}Ru	^{140}Ce	^{140}Ce
^{27}Al	^{82}Se	^{105}Pd	^{141}Pr	^{141}Pr
^{31}P	^{83}Kr	^{107}Ag	^{142}Nd	^{142}Nd
^{39}K		^{111}Cd	^{152}Sm	^{152}Sm
^{43}Ca		^{114}Cd	^{153}Eu	^{153}Eu
^{47}Ti		^{120}Sn	^{158}Gd	^{158}Gd
^{51}V		^{121}Sb	^{159}Tb	^{159}Tb
^{52}Cr		^{123}Sb	^{195}Pt	^{195}Pt
^{53}Cr			^{197}Au	^{197}Au
^{55}Mn			^{205}Tl	^{205}Tl
^{57}Fe			^{208}Pb	^{208}Pb
^{59}Co			^{232}Th	^{232}Th
^{60}Ni			^{233}U	^{233}U
^{63}Cu			^{234}U	^{234}U
^{65}Cu			^{235}U	^{235}U
			^{236}U	^{236}U
			^{238}U	^{238}U

APPENDIX 4: RADIOCHEMISTRY CONVERSION CALCULATIONS FOR URANIUM ISOTOPES

Conversion for liquids (µg/L x CF = pCi/L)

^{233}U (µg/L to pCi/L) = 9640.6
^{234}U (µg/L to pCi/L) = 6224.9
^{235}U (µg/L to pCi/L) = 2.1615
^{236}U (µg/L to pCi/L) = 64.698
^{238}U (µg/L to pCi/L) = 0.33627

Conversion for solids (mg/kg x CF = pCi/g)

^{233}U (mg/kg to pCi/g) = 9640.6
^{234}U (mg/kg to pCi/g) = 6224.9
^{235}U (mg/kg to pCi/g) = 2.1615
^{236}U (mg/kg to pCi/g) = 64.698
^{238}U (mg/kg to pCi/g) = 0.33627