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JOSEPH GUY COMMUNITY CENTER
KWETHLUK, ALASKA

BROWNFIELD CLEANUP ACTION PLAN

June 5, 2012

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Alaska Department of Environmental Conservation

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ACRONYMS AND ABBREVIATIONS

AST ..... Aboveground storage tank
Bgs ..... below ground surface
BSI ..... Bethel Services, Inc.
BTEX ..... Benzene, toluene, ethylbenzene, and xylenes
CIS ..... Alaska Community Database Community Information Summaries
CoC ..... Chain of custody
COPC ..... Contaminants of potential concern
CSM ..... Conceptual site model
cy ..... cubic yards
DEC ..... Alaska Department of Environmental Conservation
DQO ..... Data quality objectives
DRO ..... Diesel-range organics
E&E ..... Ecology and Environment, Inc.
EPA ..... U.S. Environmental Protection Agency
GPS ..... Global Positioning System
GRO ..... Gasoline-range organics
IDW ..... Investigation-derived waste
JGCC ..... Joseph Guy Community Center
KTRC ..... Kwethluk Tribal Resident Council
mg/kg ..... milligrams per kilogram
mg/L ..... milligrams per liter
OASIS ..... OASIS Environmental, Inc., an ERM company
OVK ..... Organized Village of Kwethluk
pdf ..... portable document format
PID ..... Photoionization detector
PM ..... Project manager
PPE ..... Personal protective equipment
ppmv ..... parts per million by volume
QAPP ..... Quality Assurance Project Plan
QA/QC ..... Quality assurance/quality control
RCRA ..... Resource Conservation Recovery Act
RRO ..... Residual-range organics
SPLP ..... Synthetic Precipitation Leaching Procedure
SVOC ..... Semi-volatile organic compound
TAL ..... Target analyte list
TBA ..... Targeted Brownfields Assessment
TCLP ..... Toxicity Characteristic Leaching Procedure
XRF ..... X-ray Fluorescence Spectrometer

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1. INTRODUCTION

OASIS Environmental, Inc., an ERM company (OASIS) was contracted by the Alaska Department of Environmental Conservation (DEC) to demolish a burned building, the former Joseph Guy Community Center (JGCC), in Kwethluk, Alaska and dispose of the building materials, debris, and ash. The project also involves confirmation sampling the footprint of the building, removal of known semi-volatile organic compounds contaminated soil, and removal / management of known petroleum contaminated soil associated with an aboveground storage tank (AST) that served the building. The ultimate purpose of the project is to assist the Organized Village of Kwethluk and the City of Kwethluk in revitalizing the site by making the property ready for reuse. The EPA has provided funding for demolition of the JGCC, removal of contaminated soil, and confirmation sampling of building footprint and the excavations associated with the contaminated soil.

1.1. Site Description and Background

The following community and site information was gathered from the Alaska Community Database Community Information Summaries (CIS) and from the 2010 Targeted Brownfields Assessment (TBA) by Ecology and Environment, Inc. (E&E, 2011). Kwethluk is located approximately 12 miles east of Bethel, Alaska on the Kwethluk River, a tributary to the Kuskokwim River (Figure 1). The community lies at 60.81220° North Latitude and -161.435830° West Longitude (Section 05, T008N, R069W, Seward Meridian.) It is a Yup'ik community with a population of 741. The City of Kwethluk provides water treatment, honeybucket, washeteria, and refuse services. Residents haul water for household use.

The community relies on air transportation for year-round freight and passenger service, with a state-owned gravel airstrip and seaplane base. Snowmachines, all-terrain vehicles and skiffs are used for local travel and the river becomes an ice road during the winter.

The 5,000 square-foot JGCC was built between 1998 and 2002. It is owned by the Organized Village of Kwethluk (OVK) and housed the Kwethluk Indian Reorganization Act Council and eight village social services. It was also used for community functions. The center was primarily constructed of metal with steel I-beam supports and joists with corrugated sheet metal walls and roof. The floor was built of combustible materials. The building was built on a raised earthen platform covered by a geotextile liner and polystyrene foam. Interior walls were constructed of particle board and sheet rock.

1.2. Brief Site History

The JGCC building burned in April 2006 and the possible release of contaminants posed a risk to human health through building debris and soil / groundwater contamination. The possible sources of contamination included a former glycol heating system and

burned electronic waste. It is unlikely that any fluorescent light fixtures in the building contain polychlorobiphenyls as the building was built in the late 1990's.

In 2010, E&E performed a TBA funded by the U. S. Environmental Protection Agency (EPA). The TBA involved collecting eight surface soil samples from the building interior for analysis of Target Analyte List (TAL) metals and semi-volatile organic compounds (SVOC). Five of the eight samples were also analyzed for dioxins and furans.

All eight of the samples contained at least one TAL metal result that exceeded DEC cleanup levels. Only six of the twenty TAL metals exceeded cleanup levels including antimony, arsenic, chromium, cobalt, copper, and nickel. None of the samples exceeded DEC or EPA regulatory criteria for SVOC or dioxins/furans.

Eighteen exterior co-located surface/subsurface soil samples were collected and analyzed for TAL metals and SVOC. Six of the samples were also analyzed for dioxins/furans. Two surface soil samples exceeded DEC cleanup levels for SVOC; a sample located on the west side of the building had a bis(2-ethylhexyl)phthalate result of 2.7 milligrams per kilogram (mg/kg), exceeding the DEC cleanup level of 1.3 mg/kg; a sample located on the south side of the building had a n-nitroso-di-n-propylamine result of 0.042 mg/kg, exceeding the cleanup level of 0.0011 mg/kg.

Two surface soil samples were collected from the former location of an AST that contained heating oil and analyzed for diesel-range organics (DRO) and residual-range organics (RRO). One of the samples had a DRO result of 9,000 mg/kg, exceeding the DEC cleanup level for DRO of 250 mg/kg.

Eight wipe samples were collected from the interior and exterior building walls and analyzed for dioxins/furans. All of the wipe samples were positive for dioxins/furans. No regulatory criteria exist for wipe samples.

Twelve bulk samples were collected of suspected asbestos containing building materials. No asbestos was present in any of the samples.

1.3. Conceptual Site Model

A preliminary conceptual site model (CSM) is discussed in the following subsections. CSM and graphic are included in Appendix A.

1.3.1. Contaminants of Potential Concern (COPC)

The TBA determined that surface soil at the site contains several COPC. Table 1 presents the COPC and the corresponding regulatory criteria. All criteria are based on the DEC Method Two Migration-to-Groundwater Soil Cleanup Levels except for the cobalt value. The DEC does not have a cleanup level for cobalt. The cobalt value is based on the EPA Regional Protection of Groundwater Soil Screening Level.

Table 1: COPC and Regulatory Criteria

Contaminant	Regulatory Criteria (mg/kg)
Antimony	3.6
Arsenic	3.9
Chromium	25
Cobalt	0.21
Copper	460
Nickel	86
Bis(2-ethylhexyl)phthalate	13
N-nitroso-di-n-propylamine	0.0011
DRO	250

1.3.2. Exposure Pathways Determination

As detailed in the CSM scoping form and associated graphics (Appendix A), exposure via the following pathways may occur at the site:

- Incidental soil ingestion,
- Dermal absorption of contaminants from soil,
- Inhalation of fugitive dust,
- Ingestion of groundwater,
- Ingestion of surface water, and
- Inhalation of outdoor air.

As contaminants are present in the surface soil, the incidental soil ingestion pathway is complete. Arsenic and SVOCs are able to permeate the skin so dermal absorption of contaminants from soil pathway is complete. Chromium is a COPC and it may be present in the top 2 centimeters of soil, making the inhalation of fugitive dust pathway complete.

Groundwater contamination has not been evaluated at the site, but since contaminants are present in the soil at concentrations that indicate migration to groundwater is possible, and the groundwater in Kwethluk has not been determined by the DEC to be unusable as a drinking water source, the ingestion of groundwater pathway is considered complete.

Ingestion of surface water pathway is considered complete because surface contaminants could reach the Kwethluk River through run-off or groundwater flow. It is not known whether the river is used as a seasonal drinking water source.

The ingestion of wild and farmed foods pathway is considered incomplete because the site is in an open, cleared area that would not likely be used for harvesting foods.

The inhalation of outdoor air pathway is considered complete because DRO is a volatile contaminant and is present in the surface soil. Although this pathway is complete, it is

unlikely that sufficient quantities of this compound are present in the soil to present a risk to human health.

The inhalation of indoor air pathway is not considered complete because DRO is the only volatile contaminant of concern at the site and the DEC will generally not require an vapor intrusion evaluation if the only contaminants of concern are GRO, DRO, and RRO.

1.3.3. Receptors

The site is not currently used as a residence and is not expected to be used for this purpose in the future, so residents are not considered receptors. It is not currently used as a place of work but may be once the building is replaced so commercial workers are considered future receptors only.

Site visitors and trespassers may occupy the site for short periods and can be considered both current and future receptors. Construction workers during demolition and construction of a new building on the property are considered both current and future receptors. As mentioned above, subsistence harvesters are not considered receptors as the building is located on a cleared piece of land.

1.4. Permitting

The DEC has requested and received concurrence of a "No Historic Property Affected" finding for the Brownfield Cleanup Action from the Alaska State Historical Preservation Office. The U. S. Fish and Wildlife Service provided concurrence that no federally listed or proposed species or designated or proposed critical habitat is present within the work area of this project. Both concurrence letters are included as Appendix B.

1.5. Project Objectives

The project objective is to make the JGCC property ready for building a new community center. This project is designed to meet that objective through the following tasks:

- Demolish the JGCC and transport all non-hazardous building materials to Bethel, Alaska for recycling or disposal. Any obvious hazardous materials will be staged at a location designated by the stakeholders.
- Collect confirmation samples from the building footprint for analysis of six TAL metals.
- Prepare the Kwethluk landfill to accept four waste streams; the top 2 to 3 inches of ash/soil/debris from the building footprint, known diesel-contaminated soil associated with the former AST adjacent to the building, and two small volumes of SVOC-contaminated soil adjacent to the building, but not related to the fire.
- Scrape the top few inches of ash/soil/debris from the former JGCC footprint and transport it to the Kwethluk landfill.
- Excavate diesel-contaminated soil associated with a former heating oil AST and store at the Kwethluk landfill in bulk polyethylene sacks.

- Excavate surface soil in two locations of known SVOC contamination and store soil from each location in separate bulk sacks at the Kwethluk landfill.
- Collect confirmation samples of the excavations and diesel and SVOC contaminated soil stockpile.

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2. FIELD WORK

The field work will be completed in two phases. The demolition phase is scheduled from April 2 to April 15, 2012. The soil excavation, sampling, and stockpiling work is scheduled for June 2012. The tasks are listed and described below and methods are explained in the following section. All fieldwork will be conducted and all field and laboratory quality assurance criteria for this project will be performed in accordance with the January 2010 DEC *Draft Field Sampling Guidance*.

2.1. General

OASIS is a prime contractor for this project. OASIS' subcontractor, Bethel Services, Inc. (BSI) will demolish the JGCC and will haul the materials to Bethel where they will be disposed of or staged for recycling. OASIS will rent equipment for the excavation and stockpiling phase from Kwethluk Tribal Resident Council (KTRC). A local Hazardous Waste and Emergency Response trained operator will be hired in Kwethluk to perform the excavation services.

2.2. Building Demolition

BSI will demolish the building in early April 2012. All unburned materials, with the exception of any potentially hazardous materials such as electronic waste, will be placed in trucks and hauled to the Bethel landfill over the ice road on the Kwethluk and Kuskokwim Rivers (Figure 2). Any small, non-metal debris will be transported to the Kwethluk landfill. The OVK requested and received concurrence from the DEC that the building materials and debris may be handled as solid waste rather than hazardous waste. The concurrence letter is included in Appendix B.

2.2.1. Building Footprint Soil Screening and Sampling

During preparation for this project, two three-point composite samples were collected of the ash/soil mixture within the building footprint. The samples were analyzed for metals using a Toxic Characteristic Leaching Procedure (TCLP). The sample results are presented in Table 2.

Table 2: TCLP Metal Results

Sample ID	Analytical Results (mg/L)					
	Antimony	Arsenic	Chromium	Cobalt	Copper	Nickel
3131201	0.064	ND (0.010)	ND (0.0040)	0.017	0.33	ND (0.030)
3131202	0.036	0.3	0.0067	0.027	0.43	ND (0.030)
RCRA Regulatory Criteria	Not Regulated	5.0	5.0	Not Regulated	Not Regulated	Not Regulated

ND - not detected - reporting limit in parentheses
mg/L - milligrams per liter

Only arsenic and chromium are regulated under Subpart D of the Resource Conservation Recovery Act (RCRA) regulations (40 CFR Part 261). The results for arsenic and chromium in the two composite samples were well below the RCRA regulatory criteria. These results suggest that the soil beneath the building footprint may be treated as solid waste, rather than hazardous waste. Any ash/soil mixture removed with the small debris will also be treated as solid waste and placed at the Kwethluk landfill.

OASIS will return to the site on June 6, 2012 to collect confirmation samples of the building footprint. The crew will screen the footprint of the building on a 15- by 15-foot grid pattern (Figure 3) using a portable x-ray fluorescence spectrometer (XRF). The screening results will be used to select samples for laboratory analysis of antimony, arsenic, chromium, cobalt, copper, and nickel using EPA Method 6020. Table 3 describes the number of samples and analytical methods for the building footprint confirmation samples. The screening and sampling methods are described in Section 3.

2.3. Utility Locates

OASIS plans to remove contaminated soil to approximately 1 foot below ground surface (bgs). Although it is unlikely that utilities would be buried at this shallow depth, Alaska state law requires that all buried utilities be located before digging to any depth. OASIS will contact the local utilities through the Alaska Dig Line and arrange for utility locates to be completed before any excavation occurs. If it is necessary to advance the excavations below 1 foot bgs, OASIS will stop work and contact the DEC project manager.

2.4. Soil Excavations

During the June field event, OASIS will oversee excavation of contaminated soil from the AST location (excavation area 1 – Figure 3) and the two locations with SVOC contamination (excavation areas 2 and 3 – Figure 3). OASIS will use Global Positioning System (GPS) coordinates to determine the locations of the samples collected during the 2010 TBA. The coordinates of the TBA samples are included on Figure 3 and have been programmed into the GPS to be used onsite.

The excavations will be advanced until clean soil is reached, or until the geotextile liner is encountered (assumed to be 1 foot bgs), or until 5 cubic yards (cy) have been removed, whichever comes first. If either of the latter two situations occurs, OASIS will stop work and contact the DEC project manager before continuing.

OASIS will rent an excavator from KTRC and use a local operator to excavate the contaminated soil. Prior to excavating the soil, OASIS will use heated headspace screening with a photoionization detector (PID) to screen the soil in the three excavation areas. The screening will be used to roughly determine the extent of contaminated soil. The soil will be excavated to the determined extent of contamination. If the PID results are less than 10 parts per million by volume (ppmv) for the SVOC contaminated areas, OASIS will attempt to determine the original location of the 2010 samples and remove one bucket load at that location.

OASIS will collect at least two samples from each of the excavations; one from the floor and one from the sidewall, at the locations with the highest PID results. The AST excavation samples will be analyzed for DRO using Alaska Method (AK) 102, gasoline-range organics (GRO) using AK 101, benzene, toluene, ethylbenzene, and xylenes (BTEX) using EPA Method 8021B, and SVOC using EPA Method 8270D. The SVOC excavation samples will be analyzed for SVOC only. Table 3 details the number of samples and analytical methods for the excavation confirmation samples.

Once analytical samples have been collected, the area will be re-graded to remove any uneven areas caused by removal of the soil.

2.5. Soil Storage

The DEC estimates that the total soil volume to be excavated from all three locations will be approximately 5 cy. The soil must be stored until sample results are received indicating that the contaminant concentrations are low enough to be safely used as cover at the Kwethluk landfill.

OASIS has been unable to locate a local dump truck to haul the soil to the landfill for storage. OASIS plans to place the soil in 1 cy bulk polyethylene sacks and transport the sacks to the landfill using the excavator. The soil from each soil excavation will be placed in a separate sack(s) and each sack will be staged on a pallet at the landfill until results are received from the soil sampling.

OASIS will collect one heated headspace PID screening samples from each of the bulk sacks. The PID results will be used to select samples for laboratory analysis. The PID samples and the corresponding analytical samples will be collected from the same location within each sack.

One sample with the highest PID result will be selected from the AST bulk sacks for laboratory analysis of DRO, GRO, BTEX, and SVOC using the methods described in Section 2.4. Additional volume from the sampled bulk sack will be collected and submitted for Synthetic Precipitation Leaching Procedure (SPLP) extraction and analysis of DRO and SVOC.

One analytical sample will be collected from each of the SVOC bulk sacks and submitted for laboratory analysis of SVOC. Again additional volume will be collected and submitted for SPLP extraction and SVOC analysis. Table 3 describes the analytical methods and number of samples for each set of bulk storage sacks.

TABLE 3. SOIL SAMPLE ANALYSIS SUMMARY

Location	Laboratory Analyses				
	TAL Metals (EPA 6020)	GRO/BTEX (AK 101 / EPA 8021B)	DRO (AK102)	SVOC (EPA 8270D)	SPLP Extraction (EPA 1312)
Building Footprint	9	-	-	-	-
AST Excavation	-	2	2	2	-
SVOC Excavations (2)	-	-	-	4	-
Bulk Sacks - Soil	-	1	1	3	-
Bulk Sacks - SPLP	-	-	1	3	3
Total Primary Samples	9	3	4	12	3
Duplicates (10%)	1	1	1	2	-
Trip Blanks (one per cooler w/ GRO/BTEX)	NA	1	NA	NA	NA
Total Project Samples	10	5	5	14	3

Key:
TAL Metals = Antimony, Arsenic, Chromium, Cobalt, Copper and Nickel (EPA 6020)
BTEX = Benzene, Toluene, Ethylbenzene and Xylene (EPA 8260)
DRO = Diesel-range organics (AK102)
GRO = Gasoline-range organics (AK101)
NA = Not applicable
SVOC = Semivolatile organic compound
- = No samples

3. SAMPLING METHOD

All fieldwork will be conducted and all field and laboratory quality assurance criteria for this project will be performed in accordance with sample collection procedures defined in the January 2010 DEC *Draft Field Sampling Guidance*.

OASIS will collect soil samples using clean, disposable sampling spoons. If collecting surface soil, the spoon will be used to access undisturbed soil and collect the sample. If deeper soil is collected, i.e. from the excavator bucket or from a bulk soil sack, a trowel (excavator bucket) or hand auger (bulk sack) will be used to access soil from below the surface and a clean spoon will be used to access and collect soil that has not touched the trowel or hand auger.

3.1. Field Screening

3.1.1. XRF

The building footprint will be screened using a portable handheld XRF in Soil Mode. The field team has been trained in the safe use of a handheld XRF. If the soil is dry, it will be screened by taking a reading directly from the ground surface (in-situ analysis).

The footprint will be screened on a 15- by 15-foot grid spacing. At each screening location the crew will remove any large or non-representative debris from the soil surface, including rocks, pebbles, twigs, leaves, etc. The ground surface will also be leveled with a trowel, if necessary.

Once the location is prepared, the analyzer will be placed against the ground surface with the nose of the analyzer in contact with the soil and a reading will be initiated. The analyzer will be set to soil mode and all three element ranges will be used (main, low, and high). OASIS will use a measurement time of 40 to 60 seconds on each range, totaling two to three minutes per location. The crew will use professional judgment to determine whether a 40-second measurement time is sufficient by comparing the results with the 60-second measurement time.

If the ground is saturated, the in-situ screening technique will not provide accurate results. In that case, the crew will collect approximately 8 ounces (volumetric) of soil into a re-sealable bag. Each bag of soil will be dried in a toaster oven, sieved to 60-mesh, and placed into a sample cup provided as part of the soil kit for the XRF. Again, a 40 to 60 second measuring time will be used for each element range. The results will be used to select nine footprint samples for laboratory analysis. The laboratory samples will be selected based on the highest screening results and on spatial distribution. Abbreviated XRF soil analysis instructions are included in Appendix C. The entire XRF manual (on CD) will be included with the XRF analyzer.

3.1.2. PID

Field screening of soil samples will be performed using a PID and a heated headspace technique. The PID will be calibrated daily using 100 ppmv isobutylene calibration gas.

Calibration checks will be performed at the end of each day by the field team. Calibration date, time, and results for all instruments will be recorded in a log book. The instrument model and serial number will be recorded.

The samples will be collected and screened in the following manner:

- Partially fill (one-third to one-half) a re-sealable, polyethylene, quart-size bag with the sample to be screened. Ensure that the sample is collected from freshly uncovered soil.
- Agitate the soil within the bag for 15 seconds and place in a warm location to warm the sample to at least 40°F. If no vehicle or nearby building is available to warm the sample, place the bags in the sun on a dark surface.
- Allow headspace vapors to develop in the bag for at least 10 minutes but no longer than one hour.
- Agitate the soil in bag again for 15 seconds.
- Pierce the bag, using the PID, at a point about one-half of the headspace depth.
- Record the highest PID reading in the field notebook.

This technique will also be used to assess the boundaries of contaminated soil in each excavation and to help select locations for analytical confirmation samples.

3.2. Analytical Sampling

Analytical samples will be selected based on the highest screening results and on spatial distribution. Spatial distribution will be important for representing the building footprint with analytical samples. The analytical samples will be collected from the same location as the screening sample and will immediately be placed in sample jars and preserved, if appropriate.

The GRO/BTEX samples will be collected first. A 25-gram sample of soil will be placed with minimum disturbance directly into a tared 4-ounce jar with a Teflon®-lined septum fused to the lid. The sample will be immediately preserved with 25 milliliters of methanol. Any visible grit will be removed from the jar threads before sealing the jar to prevent leakage of the methanol. The jars will then be placed on ice to maintain the sample temperature at 4 degrees Celsius (°C) ± 2°C. As unpreserved DRO samples are being collected from each location, no additional moisture content samples will be collected.

The DRO, SVOC, and TAL metals samples will be collected directly into 4-ounce amber, non-preserved, sample jars. The SPLP extraction DRO and SVOC samples will be collected into 8-ounce amber, non-preserved jars. Separate jars will be collected for the DRO, SVOC, and TAL Metals samples. The samples will be placed on ice to maintain the sample temperature at 4°C ± 2°C.

Table 3 details the number of samples and analytical methods for each phase of the investigation. The appropriate containers, holding times, and other quality control measures are included in Section 4.

A new pair of nitrile gloves and new stainless steel sampling spoon will be used for collection of each soil sample. Visual soil observations and sample collection location will be recorded in the field notebook.

Samples will be delivered to TestAmerica in Anchorage, a DEC-approved laboratory, under proper chain-of-custody (CoC) procedures. Sample identifications as well as date and time collected will be entered on the CoC form, and the appropriate analyses should be requested for each sample. The CoC should be included in the cooler (or one of the coolers in a batch) and the cooler(s) sealed with custody seals. The cooler(s) must contain enough ice to maintain a temperature of 4°C± 2°C and must be delivered to the laboratory within enough time to assure analysis within holding times.

3.3. Sample Identification

OASIS will use an alpha-numeric code for sample identification numbers. The sample code for laboratory samples is broken down as follows in Table 4:

TABLE 4. SAMPLE IDENTIFICATION SCHEME

Digits	Description	Code Examples
1-2	Year	12
3-6	Location Code	JGCC
7-9	Sequential Sample Number	101
10-11	Sample Type:	Symbol:
	Soil Sample	SO
	Trip Blank	TB

Example: 12-JGCC-101-SO (2012 Joseph Guy Community Center, Sample No. 01, Soil Sample).

3.4. Investigation-Derived Waste

The Investigation-derived waste (IDW) will include five waste streams. The building and its contents will be treated as one waste stream. This waste stream has been determined to be non-hazardous. The DEC has issued a letter concurring with the OVK that the waste and debris generated by this project can be handled as solid waste for disposal (Appendix B). The metal materials will be delivered to the Bethel landfill via the ice road on the rivers between Kwethluk and Bethel.

Any small, non-metal debris with incidental soil/ash mixture will be transported to the Kwethluk landfill. As mentioned in Section 2.2, this waste stream has been characterized as non-hazardous by TCLP sample results.

The diesel- and SVOC-contaminated soil will be placed in 1 cy bulk polyethylene sacks at the Kwethluk landfill. The soil from each of the three excavations will be placed in separate sacks. A concurrence letter for placement at the landfill is included in Appendix B.

All sampling supplies and personal protective equipment (PPE) will be disposable and will be considered one waste stream. These materials will be placed in garbage bags and disposed of at the Kwethluk landfill.

No decontamination water will be generated during this project. All sampling supplies/PPE will be disposable and the heavy equipment will be dry-decontaminated using a stiff broom to remove the soil. The tracks/wheels of the equipment are not expected to come in contact with the contaminated soil.

4. QUALITY ASSURANCE / QUALITY CONTROL

This section includes the quality assurance / quality control (QA/QC) procedures for the field personnel regarding training, documentation, instrument calibration, sample collection and sample management. Laboratory QA/QC procedures are included in the Quality Assurance Project Plan (QAPP) in Appendix D.

4.1. Personnel

This demolition portion of the project will be overseen by Lisa Nicholson. The soil screening and sampling portion of the project will be performed by Rena Bryan and Leslie Davis. BSI will provide building demolition services and will haul the metal debris to Bethel where it will be disposed of or staged for recycling. Sampling will consist of collecting confirmation samples of the building footprint and of the AST-related contaminated soil and the SVOC contaminated soil. All sampling field personnel meet the definition of "qualified person" per 18 AAC 75.990(100).

4.2. Field Procedures

All fieldwork and laboratory analyses will be conducted in accordance with the May 2010 Draft Field Sampling Guidance (DEC 2010).

4.2.1. Field Documentation

Field documentation will include sample identification labels, photographs, laboratory analysis requests, and permanently bound field logs. A field logbook will be maintained by the OASIS field team leader to record a detailed description of all field activities and samples collected. The logbook will be maintained as part of the permanent record for the site.

Pages will not be removed from any data logbook for any reason. Any possible corrections will be made by drawing a single line through the original entry, so that the original entry still can be read. The corrections will be written alongside the crossed-out entry. The corrections will be initialed and dated. The logbook will be maintained as part of the permanent record for the site.

Activities and observations to be noted in the logbook include the following items:

- Name of author and date and time of entry
- Names and affiliations of personnel on-site
- Location of activity and site conditions
- Field observations and comments
- Documentation of instrument calibration
- Weather conditions
- Locations of site photographs
- Site sketches

- Health and safety comments

4.2.2. Calibration of Instruments

All equipment will be calibrated, maintained and operated according to manufacturer recommendations. The lot number and expiration date of the isobutylene calibration gas used will be recorded in a calibration logbook for the PID. The appropriate pages of the PID calibration log will be included in the project report.

4.2.3. Sample Collection

Sampling supplies will be dedicated to each discrete sample location. Sample matrices will have minimal disturbance prior to collection. Sample containers will be sealed, labeled, and placed in a cooler following collection. Samplers will use dedicated PPE to prevent cross-contamination between samples. Field personnel will collect samples in a manner that preserves the integrity of the sample matrix.

4.2.4. Sample Labeling

Each sample container will be sealed and labeled immediately after collection. Sample labels will be completed using waterproof ink and will be affixed firmly to the sample containers with clear, waterproof tape. A sample code will be assigned to each sample as an identification number to track collected samples. The sample label will provide the following information: Project name; Date and time of collection; Sample identification number; Analysis required; and Preservation method used.

Duplicate samples will be numbered sequentially without any additional identification so that the laboratory cannot identify the quality control purpose of the sample.

Labels will not be placed on pre-tared containers provided by the laboratory for volatile analyses. Information will be written on the pre-labeled containers. If the container does not have a label, the container will be placed in a resealable bag, and the label will be placed on the outside of the bag. The bagged sample will then be placed into a second bag that will serve to protect loss or damage to the sample label. Duplicate samples will be numbered sequentially without any additional identification so that the laboratory cannot identify the quality control purpose of the sample. Trip blanks will be numbered sequentially for the project.

After a sample is collected, pertinent information such as sample identification number, date and time of sample collection, sample collection method, description of sample, and any field screening measurements will be recorded in the field logbook. Additionally, sample identification and the associated location shall be documented in the field logbook. Cooler identification shall be included to link samples with associated coolers used to ship samples to the laboratory.

4.2.5. Chain-of-Custody, Sample Packaging

A CoC record will be completed and shipped with the samples. The CoC will include the OASIS project number. Proper sample custody is maintained through adherence to the procedures listed below.

- Custody seals will be placed in two locations over the lid/cooler edge and secured with clear packing tape.
- A CoC record must accompany the coolers in which the samples are packed. When transferring samples, the individuals relinquishing and receiving the coolers must sign, date, and note the time on the CoC record. This record documents sample custody transfer.

Samples must be packaged carefully to avoid breakage or contamination and must be shipped to the laboratory at proper temperatures. Adherence to the following sample package requirements is essential:

- Sample container lids must never be mixed. All lids must remain with their original container.
- Environmental samples must be cooled to 4°C ± 2°C to preserve many chemical constituents.

Any remaining space in the cooler should be filled with inert packing material such as bubble wrap, newspaper, etc. Under no circumstances should material such as sawdust, sand, or Styrofoam peanuts be used.

4.2.6. Shipping

Environmental samples will be properly packaged and labeled inside an insulated cooler for transport to TestAmerica in Anchorage, Alaska. TestAmerica will provide pre-filled shipping documents for use by the field team when dropping a cooler for shipment. Custody seals will be placed over the lid/cooler edge and secured with clear packing tape. The custody seal must remain intact during transfer to the laboratory.

4.3. Quality Assurance and Quality Control

Quality control procedures are used to ensure that data are useable for their intended purpose. Specific objectives of quality control samples are listed as follows:

- All samples collected at the site are consistent with project objectives.
- Samples are identified, preserved, and transported in a manner such that the data are representative of the actual site conditions.
- Information is not lost in sample transfer.
- The data are legally defensible.

As part of the field QA/QC program, field duplicates and trip blank samples will be collected.

4.3.1. Quality Control Samples

Quality control samples will be collected and prepared to assess potential errors introduced during sample collection, handling, and analyses. As part of the QA/QC program, field duplicates will be collected for all project analytes, and trip blanks will be collected and analyzed for GRO/BTEX analyses only.

Field duplicate samples will be collected to verify the reproducibility of data within the project laboratory. In general, at least one field duplicate sample will be collected for every ten project samples per matrix. The locations of field duplicates will be determined in the field. The duplicate samples will be handled, labeled, and documented in the same manner as regular field samples to prevent potential bias in the laboratory results. Field duplicates will not be identified and will be labeled in the same manner as other field samples on the CoC forms.

Trip blanks will accompany the sample containers sent into the field from the laboratory. Trip blanks are used when samples are collected for GRO/BTEX analysis and are included in all shipments back to the laboratory that contain samples to be analyzed for GRO/BTEX.

4.3.2. Field Duplicates

Field duplicate samples are collected to verify the reproducibility of data. At least one field duplicate sample will be collected, per DEC requirements, for every ten samples of a particular matrix type and will be submitted to the project laboratory. Field duplicate samples will be collected from a location expected to contain soil or groundwater impact exceeding the method reporting limits, so that the field duplicate samples result in meaningful relative percent difference (precision) determinations.

Field duplicate samples for all analyses, except for GRO/BTEX, will consist of a fraction of the homogenized sample material collected for a designated field sample. To reduce the possibility of analyte volatilization, soil for GRO/BTEX field duplicate samples will not be homogenized.

All field duplicate samples will be handled, labeled, and documented in the same manner as associated samples to prevent biased sample results. Field duplicates will not be identified as such on the CoC forms but labeled as a regular field samples.

At least one field duplicate sample location will be chosen where a full set of sample analytes will be collected, such as at a soil boring where SVOCs are sampled.

4.3.3. Trip Blanks

Trip blanks will accompany each sample cooler containing sample bottles sent to the field from the project laboratory when volatile samples are shipped back to the project laboratory. The field team will identify which volatile samples are enclosed with a specified trip blank in a specific cooler.

4.4. Analytical Procedures

A list of the analytical methods, holding times, sample containers, and preservation requirements is provided in Table 5. Quality control requirements are provided in Table 6.

TABLE 5:. SAMPLE STORAGE, CONTAINMENT, AND PRESERVATION SUMMARY

Parameter	Analytical Methods	Holding Times	Containers	Preservation	
GRO/BTEX	AK 101 / SW 8021B	14 days	1 – 4 oz pre-weighed amber glass	Methanol	4±2°C
DRO	AK102	14 days	1 - 4 oz. amber glass; 1 – 8 oz amber glass for SPLP	None	4±2°C
SVOC	SW 8270D	14 days	1 - 4 oz. amber glass; 1 – 8 oz amber glass for SPLP		
TAL Metals	SW 6020	180 days	1 - 4 oz. amber glass		

TABLE 6: QUALITY CONTROL REQUIREMENTS

Analytical Parameter	Analytical Method	Matrix	Number of Primary Samples	Number of Trip Blank Samples	Number of Duplicate Samples	Total Number of Samples
GRO/BTEX	AK 101 / SW 8021B	Soil	4	1 per cooler	1 per 10	6*
DRO	AK102	Soil	4	0	1 per 10	5
SPLP DRO	SW1312 AK102	Soil	2	0	0	2
SVOC	SW8270D	Soil	7	0	1 per 10	8
SPLP SVOC	SW1312 SW8270D	Soil	4	0	0	4
TAL Metals	6020	Soil	9	0	1 per 10	10

** Includes one trip blank*
RPD = Relative percent difference

4.5. Laboratory Data Quality Objectives

Data quality objectives (DQOs) have been established for this project to ensure that the soil and water sampling data is of sufficient quantity and quality to accomplish the following:

- Monitor potential petroleum hydrocarbon concentrations to verify conformance with Method Two soil cleanup levels for the groundwater migration pathway in the under 40-inch precipitation zone, as specified in 18 AAC 75.341.
- Ensure the integrity of the results is legally defensible.

The DQOs are included in the QAPP in Appendix D.

4.6. Data Reduction, Review, and Reporting

Review of all analytical data will be performed by a qualified professional experienced in data validation and review procedures. All data will be validated and reviewed in accordance with appropriate EPA procedural guidance documents and DEC regulatory guidance documents. The reference documents include EPA Functional Guidelines for Organic Data Review (EPA 2008a), Inorganic Data Review (EPA 2010), DEC Environmental Laboratory Data and Quality Assurance Requirements, Technical Memorandum (DEC 2009) and DEC Laboratory Data Review Checklist (DEC 2010). The DQOs are included in the QAPP in Appendix D.

5. REPORTING

The field activities for this project will be presented to the DEC in a complete report. OASIS will advise DEC of any alteration from this work plan due to site conditions or unforeseen changes in the project scope.

Following the receipt of laboratory data and completion of field activities, OASIS will prepare and submit a final comprehensive report that meets the requirements of 18 AAC 75.380 for review and approval by the DEC project manager (PM). The report will consist of a narrative, tables, figures, site photographs, laboratory data, and data quality review, which will include the DEC quality assurance/quality control checklist for data quality.

The draft report will be submitted via email for DEC review and comment. OASIS will deliver the text in Word format and the complete report in portable document format (pdf). The final report will be revised based on DEC comments.

All deliverables will be submitted to the DEC Contract Manager, and e-copy deliverables will be copied to the DEC PM simultaneously. The final report will be submitted as six hard copies and in pdf format on CDs included with each hard copy report.

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6. REFERENCES

Alaska Community Database Community Information Summaries (CIS), 2012. Kwethluk. http://commerce.alaska.gov/dcra/commdb/CF_CIS.htm

Alaska Department of Environmental Conservation (DEC), 2009. Technical Memorandum: Environmental Laboratory Data and Quality Assurance Requirements. March.

DEC, 2010. Laboratory Data Review Checklist. January.

Ecology and Environment (E&E), 2011. Former Joseph Guy Community Center ARRA Funded Targeted Brownfields Assessment, Kwethluk, Alaska. March, 2011.

U.S. Environmental Protection Agency (EPA), 2008. *Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA-540-R-08-01). June.

EPA, 2010. *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA-540-R-10-011). January.

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FIGURES

SOURCE: DCRA COMMUNITY PROFILE MAPS, SEPT. 07 2007, 1 FT. PER PIXEL

V:\PROJECT DRAWINGS\ADEC\KWETHLUK BROWN FIELDS\WORKPLAN\0158196_KWSITE_F2.dwg May 24, 2012.

FIGURE 1

SITE MAP

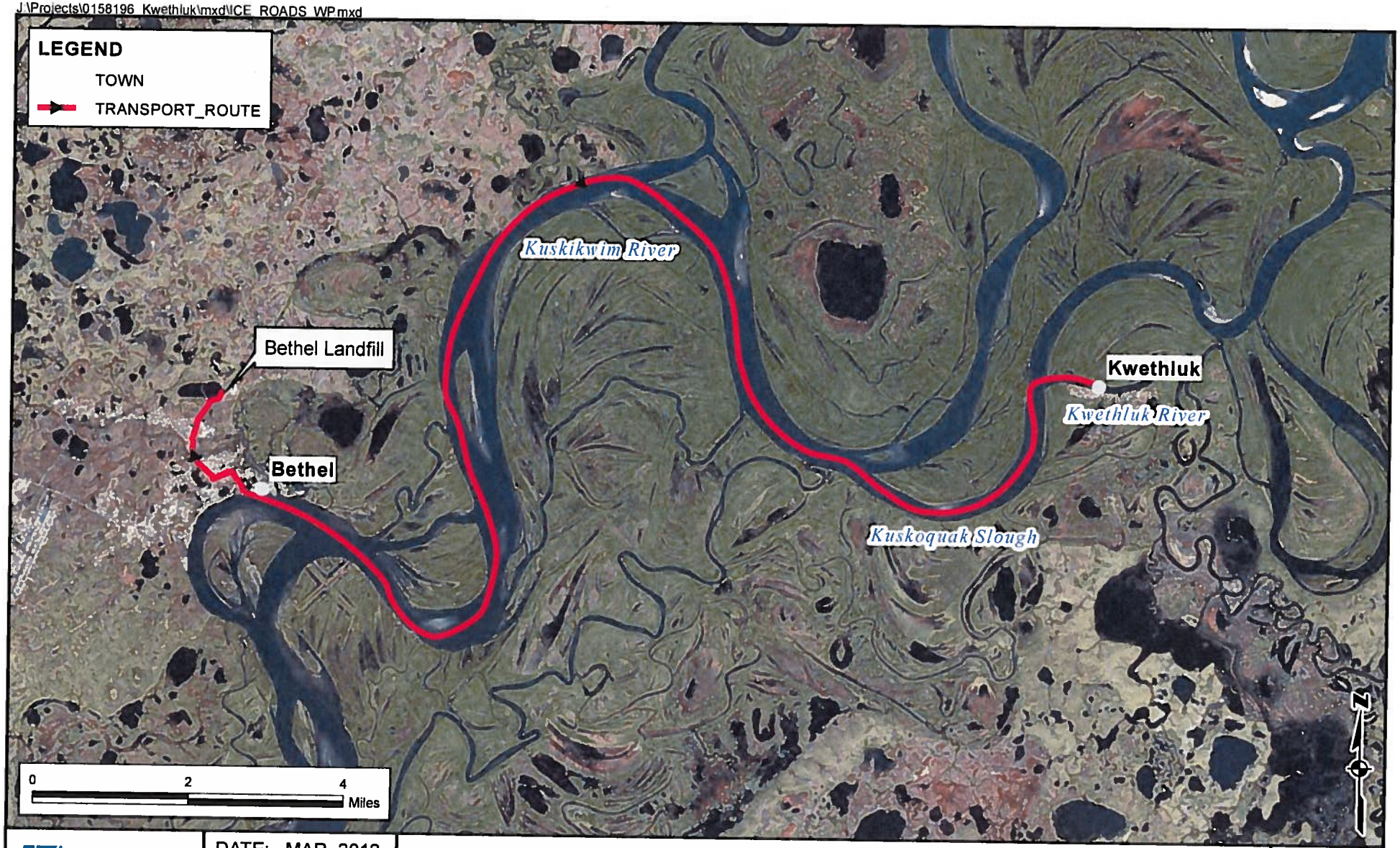
JOSEPH GUY COMMUNITY CENTER CLEAN-UP ACTION PLAN
Kwethluk, Alaska

DATE: MAY 2012
CHKD: L.C.N.
DRAWN: D.R.F.
PROJ. No.: 0158196

825 W. 8th Ave., Anchorage, AK 99501, (907) 258-4880

oasis ENVIRONMENTAL

J:\Projects\0158196_Kwethluk\mxd\ICE_ROADS_WP.mxd



oasis ENVIRONMENTAL

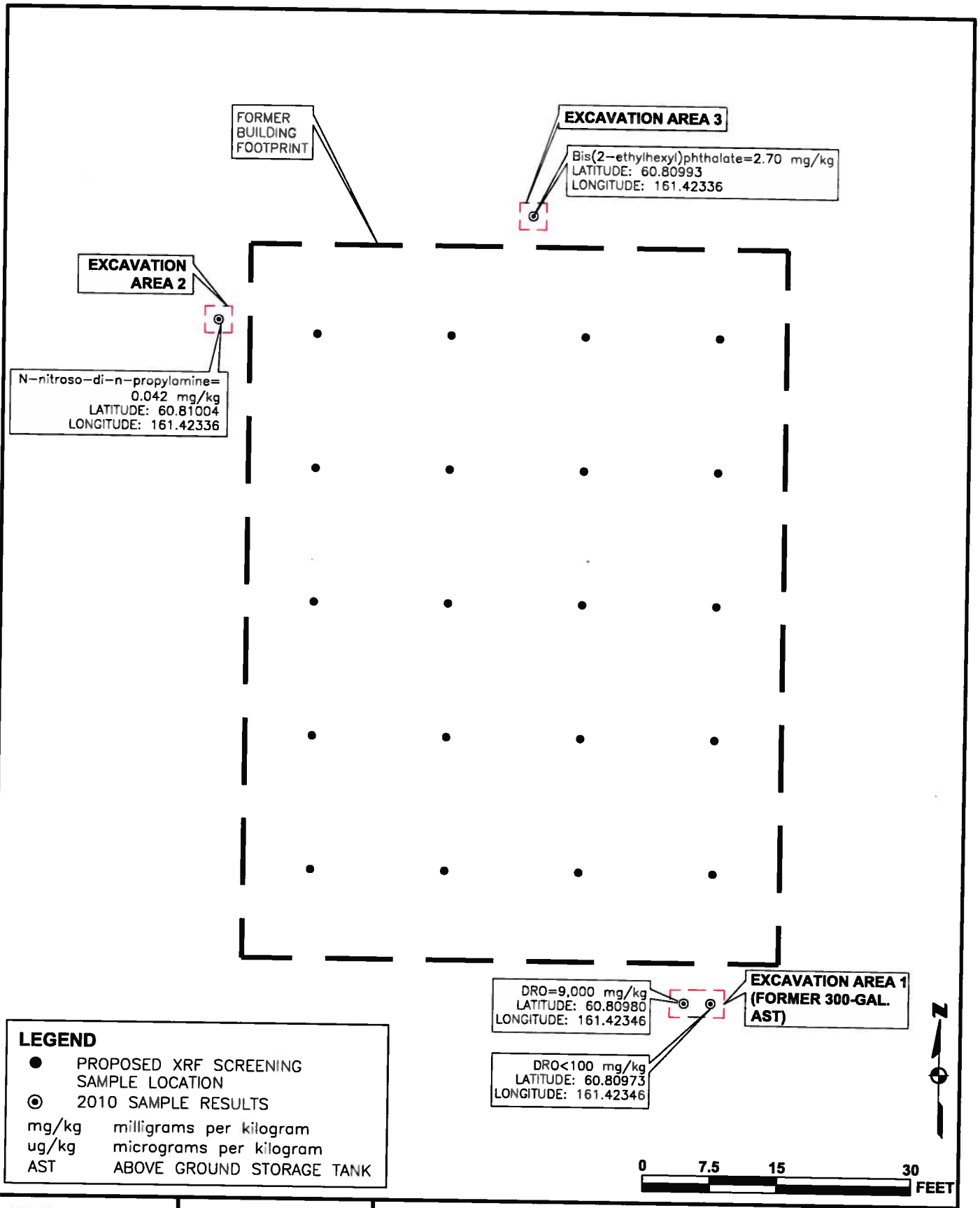
DATE: MAR. 2012
CHKD: DRAFT
DRWN: D.R.F.
PROJ. No.: 0158196

825 W. 8th Ave., Anchorage, AK 99501, (907) 258-4880

ICE ROAD TRANSPORT ROUTE TO BETHEL LANDFILL

Joseph Guy Community Center Clean-up Action Plan
Kwethluk, Alaska

FIGURE
2



LEGEND

- ● PROPOSED XRF SCREENING SAMPLE LOCATION
- ⊙ 2010 SAMPLE RESULTS
- mg/kg milligrams per kilogram
- ug/kg micrograms per kilogram
- AST ABOVE GROUND STORAGE TANK

V:\PROJECT DRAWINGS\ADEC\KWETHLUK BROWN FIELDS\WORKPLAN\0158196_KWSITE_F3.dwg Jun 01, 2012.

oasis ENVIRONMENTAL

DATE: MAY 2012
CHKD: L.C.N.
DRAWN: D.R.F.
PROJ. No.: 0158196

825 W. 8th Ave., Anchorage, AK 99501, (907) 258-4880

SAMPLE LOCATION MAP

JOSEPH COMMUNITY CENTER
CLEAN-UP ACTION PLAN
Kwethluk, Alaska

FIGURE
3

APPENDIX A

Conceptual Site Model

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Print Form

Human Health Conceptual Site Model Scoping Form

Site Name: Joseph Guy Community Center, Kwethluk, Alaska

File Number: 2424.57.001

Completed by: Lisa Nicholson

Introduction

The form should be used to reach agreement with the Alaska Department of Environmental Conservation (DEC) about which exposure pathways should be further investigated during site characterization. From this information, summary text about the CSM and a graphic depicting exposure pathways should be submitted with the site characterization work plan and updated as needed in later reports.

General Instructions: Follow the italicized instructions in each section below.

1. General Information:

Sources *(check potential sources at the site)*

- ☐ USTs
- ☒ ASTs
- ☐ Dispensers/fuel loading racks
- ☐ Drums
- ☐ Vehicles
- ☐ Landfills
- ☐ Transformers
- ☒ Other: Burned building debris

Release Mechanisms *(check potential release mechanisms at the site)*

- ☐ Spills
- ☒ Leaks
- ☐ Direct discharge
- ☒ Burning
- ☐ Other:

Impacted Media *(check potentially-impacted media at the site)*

- ☒ Surface soil (0-2 feet bgs*)
- ☒ Subsurface soil (>2 feet bgs)
- ☐ Air
- ☐ Sediment
- ☐ Groundwater
- ☐ Surface water
- ☐ Biota
- ☐ Other:

Receptors *(check receptors that could be affected by contamination at the site)*

- ☐ Residents (adult or child)
- ☒ Commercial or industrial worker
- ☒ Construction worker
- ☐ Subsistence harvester (i.e. gathers wild foods)
- ☐ Subsistence consumer (i.e. eats wild foods)
- ☒ Site visitor
- ☒ Trespasser
- ☒ Recreational user
- ☐ Farmer
- ☐ Other:

* bgs - below ground surface

2. Exposure Pathways: *(The answers to the following questions will identify complete exposure pathways at the site. Check each box where the answer to the question is "yes".)*

a) Direct Contact -

1. Incidental Soil Ingestion

Are contaminants present or potentially present in surface soil between 0 and 15 feet below the ground surface? (Contamination at deeper depths may require evaluation on a site-specific basis.) ☒

If the box is checked, label this pathway complete: Complete

Comments:

Surface soil contains the following contaminants that exceed regulatory criteria: antimony, arsenic, chromium, cobalt, copper, nickel, bis((2-ethylhexyl)phthalate, n-nitroso-di-n-propylamine, and DRO.

2. Dermal Absorption of Contaminants from Soil

Are contaminants present or potentially present in surface soil between 0 and 15 feet below the ground surface? (Contamination at deeper depths may require evaluation on a site specific basis.) ☒

Can the soil contaminants permeate the skin (see Appendix B in the guidance document)? ☒

If both boxes are checked, label this pathway complete: Complete

Comments:

Soil contains arsenic and SVOCs, contaminants that can permeate the skin.

b) Ingestion -

1. Ingestion of Groundwater

Have contaminants been detected or are they expected to be detected in the groundwater, or are contaminants expected to migrate to groundwater in the future? ☒

Could the potentially affected groundwater be used as a current or future drinking water source? Please note, only leave the box unchecked if DEC has determined the ground-water is not a currently or reasonably expected future source of drinking water according to 18 AAC 75.350. ☒

If both boxes are checked, label this pathway complete: Complete

Comments:

DRO could potentially migrate to the groundwater.

2. Ingestion of Surface Water

Have contaminants been detected or are they expected to be detected in surface water, or are contaminants expected to migrate to surface water in the future? ☒

Could potentially affected surface water bodies be used, currently or in the future, as a drinking water source? Consider both public water systems and private use (i.e., during residential, recreational or subsistence activities). ☒

If both boxes are checked, label this pathway complete: Complete

Comments:

The building is about 500 feet south of the Kwethluk River.

3. Ingestion of Wild and Farmed Foods

Is the site in an area that is used or reasonably could be used for hunting, fishing, or harvesting of wild or farmed foods? ☐

Do the site contaminants have the potential to bioaccumulate (see Appendix C in the guidance document)? ☒

Are site contaminants located where they would have the potential to be taken up into biota? (i.e. soil within the root zone for plants or burrowing depth for animals, in groundwater that could be connected to surface water, etc.) ☐

If all of the boxes are checked, label this pathway complete: Incomplete

Comments:

The site would not likely be used for subsistence hunting or harvesting.

c) Inhalation-

1. Inhalation of Outdoor Air

Are contaminants present or potentially present in surface soil between 0 and 15 feet below the ground surface? (Contamination at deeper depths may require evaluation on a site specific basis.) ☒

Are the contaminants in soil volatile (see Appendix D in the guidance document)? ☐

If both boxes are checked, label this pathway complete: Incomplete

Comments:

The only volatile contaminant of concern at the site is DRO. The DEC will generally not require an evaluation for vapor intrusion of the only COCs at the site are GRO, DRO, and RRO.

2. Inhalation of Indoor Air

Are occupied buildings on the site or reasonably expected to be occupied or placed on the site in an area that could be affected by contaminant vapors? (within 30 horizontal or vertical feet of petroleum contaminated soil or groundwater; within 100 feet of non-petroleum contaminted soil or groundwater; or subject to "preferential pathways," which promote easy airflow like utility conduits or rock fractures) ☒

Are volatile compounds present in soil or groundwater (see Appendix D in the guidance document)? ☒

If both boxes are checked, label this pathway complete: Complete

Comments:

See #1 above.

3. **Additional Exposure Pathways:** *(Although there are no definitive questions provided in this section, these exposure pathways should also be considered at each site. Use the guidelines provided below to determine if further evaluation of each pathway is warranted.)*

Dermal Exposure to Contaminants in Groundwater and Surface Water

Dermal exposure to contaminants in groundwater and surface water may be a complete pathway if:

- Climate permits recreational use of waters for swimming.
- Climate permits exposure to groundwater during activities, such as construction.
- Groundwater or surface water is used for household purposes, such as bathing or cleaning.

Generally, DEC groundwater cleanup levels in 18 AAC 75, Table C, are assumed to be protective of this pathway.

Check the box if further evaluation of this pathway is needed: ☐

Comments:

Inhalation of Volatile Compounds in Tap Water

Inhalation of volatile compounds in tap water may be a complete pathway if:

- The contaminated water is used for indoor household purposes such as showering, laundering, and dish washing.
- The contaminants of concern are volatile (common volatile contaminants are listed in Appendix D in the guidance document.)

Generally, DEC groundwater cleanup levels in 18 AAC 75, Table C, are assumed to be protective of this pathway.

Check the box if further evaluation of this pathway is needed: ☐

Comments:

Inhalation of Fugitive Dust

Inhalation of fugitive dust may be a complete pathway if:

- Nonvolatile compounds are found in the top 2 centimeters of soil. The top 2 centimeters of soil are likely to be dispersed in the wind as dust particles.
- Dust particles are less than 10 micrometers (Particulate Matter - PM_{10}). Particles of this size are called respirable particles and can reach the pulmonary parts of the lungs when inhaled.
- Chromium is present in soil that can be dispersed as dust particles of any size.

Generally, DEC direct contact soil cleanup levels in Table B1 of 18 AAC 75 are protective of this pathway because it is assumed most dust particles are incidentally ingested instead of inhaled to the lower lungs. The inhalation pathway only needs to be evaluated when very small dust particles are present (e.g., along a dirt roadway or where dusts are a nuisance). This is not true in the case of chromium. Site specific cleanup levels will need to be calculated in the event that inhalation of dust containing chromium is a complete pathway at a site.

Check the box if further evaluation of this pathway is needed: ☒

Comments:

Soil in footprint of building known to contain up to 44 mg/kg chromium.

Direct Contact with Sediment

This pathway involves people's hands being exposed to sediment, such as during some recreational, subsistence, or industrial activity. People then incidentally ingest sediment from normal hand-to-mouth activities. In addition, dermal absorption of contaminants may be of concern if the the contaminants are able to permeate the skin (see Appendix B in the guidance document). This type of exposure should be investigated if:

- Climate permits recreational activities around sediment.
- The community has identified subsistence or recreational activities that would result in exposure to the sediment, such as clam digging.

Generally, DEC direct contact soil cleanup levels in 18 AAC 75, Table B1, are assumed to be protective of direct contact with sediment.

Check the box if further evaluation of this pathway is needed: ☐

Comments:

4. Other Comments *(Provide other comments as necessary to support the information provided in this form.)*

HUMAN HEALTH CONCEPTUAL SITE MODEL GRAPHIC FORM

Site: Joseph Guy Community Center, Kwethluk, Alaska

Completed By: Lisa Nicholson

Date Completed: 3/5/2012

***Instructions*: Follow the numbered directions below. Do not consider contaminant concentrations or engineering/land use controls when describing pathways.**

(1) *Check the media that could be directly affected by the release.*

(2) *For each medium identified in (1), follow the top arrow and check possible transport mechanisms. Check additional media under (1) if the media acts as a secondary source.*

(3) *Check all exposure media identified in (2).*

(4) *Check all pathways that could be complete. The pathways identified in this column must agree with Sections 2 and 3 of the Human Health CSM Scoping Form.*

(5) *Identify the receptors potentially affected by each exposure pathway: Enter "C" for current receptors, "F" for future receptors, "C/F" for both current and future receptors, or "I" for insignificant exposure.*

Media	Transport Mechanisms	Exposure Media	Exposure Pathway/Route	Current & Future Receptors: Residents (adults or children)	Commercial or industrial workers	Site visitors, trespassers, or recreational users	Construction workers	Farmers or subsistence harvesters	Subsistence consumers	Other
☑ Surface Soil (0-2 ft bgs)	☑ Direct release to surface soil (check soil)	☑ soil	☑ Incidental Soil Ingestion	F	C/F	C/F				
	☑ Migration to subsurface (check soil)		☑ Dermal Absorption of Contaminants from Soil	F	C/F	C/F				
	☑ Migration to groundwater (check groundwater)		☑ Inhalation of Fugitive Dust	F	C/F	C/F				
	☑ Volatilization (check air)	☑ groundwater	☑ Ingestion of Groundwater	C/F	C/F	C/F				
	☑ Runoff or erosion (check surface water)		☐ Dermal Absorption of Contaminants in Groundwater							
	☐ Uptake by plants or animals (check biota)		☐ Inhalation of Volatile Compounds in Tap Water							
	☐ Other (list):	☐ air	☑ Inhalation of Outdoor Air	F	C/F	C/F				
☐ Subsurface Soil (2-15 ft bgs)	☐ Direct release to subsurface soil (check soil)		☐ Inhalation of Indoor Air							
	☐ Migration to groundwater (check groundwater)		☐ Inhalation of Fugitive Dust							
	☐ Volatilization (check air)	☑ surface water	☑ Ingestion of Surface Water		C/F					
	☐ Uptake by plants or animals (check biota)		☐ Dermal Absorption of Contaminants in Surface Water							
	☐ Other (list):		☐ Inhalation of Volatile Compounds in Tap Water							
☐ Ground-water	☐ Direct release to groundwater (check groundwater)	☐ sediment	☐ Direct Contact with Sediment							
	☐ Volatilization (check air)	☐ biota	☐ Ingestion of Wild or Farmed Foods							
	☐ Flow to surface water body (check surface water)									
	☐ Flow to sediment (check sediment)									
	☐ Uptake by plants or animals (check biota)									
	☐ Other (list):									
☐ Surface Water	☐ Direct release to surface water (check surface water)									
	☐ Volatilization (check air)									
	☐ Sedimentation (check sediment)									
	☐ Uptake by plants or animals (check biota)									
	☐ Other (list):									
☐ Sediment	☐ Direct release to sediment (check sediment)									
	☐ Resuspension, runoff, or erosion (check surface water)									
	☐ Uptake by plants or animals (check biota)									
	☐ Other (list):									

Revised, 10/01/2010

APPENDIX B

Concurrence Letters

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T 8.12.2011 3130-2R ADEC

STATE OF ALASKA

DEPARTMENT OF ENVIRONMENTAL CONSERVATION
DIVISION OF SPILL PREVENTION AND RESPONSE
CONTAMINATED SITES PROGRAM

SEAN PARNELL, GOVERNOR
610 University Avenue
Fairbanks, AK 99709-3643
PHONE: (907) 451-5174
FAX: (907) 451-5105
www.dec.state.ak.us

File: 2424.57.001 **RECEIVED**

AUG 04 2011

August 1, 2011

Judith Bittner
Chief, Office of History and Archaeology
Alaska Department of Natural Resources
550 West 7th Avenue, Suite 1310
Anchorage, AK 99501-3565

OHA

No Historic Properties Affected
Alaska State Historic Preservation Officer
Date: 8.12.2011
File No. 3130-2R ADEC SHPO

Re: Request concurrence of "No Historic Property Affected" finding for environmental site assessment and clean-up activities at the Former Joseph Guy Community Center in Kwethluk, Alaska

Dear Ms. Bittner:

The Alaska Department of Environmental Conservation (DEC) seeks concurrence that the environmental site assessment and cleanup activities to be performed at the above site will comply with policies of the State Historic Preservation Office. The project is described below. See Figure 1 for a vicinity map of Kwethluk, Alaska. See Photo 1 for an aerial photo of the community with the site labeled.

Former Joseph Guy Community Center:
The Former Joseph Guy Community Center (Center) was destroyed by fire in April 2006 (see Photo 2). The Center was built with a combination of federal, state, and private funds between 1998 and 2002 on land owned by the Organized Village of Kwethluk (OVK). The burned-out structure is adjacent to both the post office and Head Start buildings, and is across the street from the Lower Kuskokwim School District school.

The 0.5-acre site is located in Section 5, Township 8 North, Range 69 West, Seward Meridian at 60.810278N, -166.423945W.

The U.S. Environmental Protection Agency (EPA) provided a Targeted Brownfields Assessment (TBA) of the site in 2010, and the report of findings was published in 2011. The limited sampling found three types of soil contamination above cleanup levels at the site: 1) the footprint of the Center is contaminated with Target Analyte List (TAL) metals, 2) the location of the former aboveground storage tank next to the Center is contaminated with diesel range organics, and 3) two locations

adjacent to the structure's footprint are contaminated with semi-volatile organic compounds.

In order to protect the community from the physical and environmental hazards, the building debris and contaminated soils at the site will be removed so that the property can be reused. The removal area would include the entire footprint of the building plus the three areas outside of the structure where concentrations of contaminants above cleanup levels were detected. The depth for the excavations is estimated at 1 foot, or to the depth of the geotextile liner which was observed at a depth of approximately 6 to 12 inches during field sampling. For the three spots outside the structure slated for excavation, a 10 ft^2 area around each of the sampling locations may be removed to a depth not to exceed 3 to 4 feet. An excavator or backhoe would be used for the soil removal. If contamination is found to exist outside the boundary of excavation in any direction, either through visual observation, presence of an odor, or field screening results, the excavation could continue until all contamination is removed or delineated. It is anticipated that this removal action will be limited in extent.

After the building footprint, location of the former aboveground storage tank, and locations of SVOC contamination are excavated as described above, soil samples would be collected from the areas using hand tools to confirm the success of the cleanup. Either hand shovels/trowels, or small-diameter (up to two inches) slide-hammer probes or augers will be used to advance into the subsurface to a maximum depth of five feet. Samples will be collected from the core and the remaining soil material returned to the same hole or location from which it was derived.

If cultural resources are found during digging or other ground altering activities associated with this project, work would be stopped immediately and the Office of History and Archaeology would be consulted regarding the significance of the find and the appropriate actions to be taken.

Project Schedule:
We intend to complete the field work for this project between September 2011 and May 2012. A unique opportunity to coordinate with the Alaska Native Tribal Health Consortium (ANTHC) for use of their heavy equipment mobilized to Kwethluk for a sewer/water project exists during this timeframe. This coordination would reduce the cost of the site cleanup, enhancing the viability of the venture. Your assistance in meeting this schedule is greatly appreciated.

If you have any questions, please contact me by phone at (907) 451-5174 or via email at melinda.brunner@alaska.gov.

Alternatively, you can call John Carnahan at (907) 451-2166 or email him at john.carnahan@alaska.gov.

Sincerely,

Melinda Brunner
Environmental Program Specialist

Attachments: Figure 1, Photos 1 and 2

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United States Department of the Interior

FISH AND WILDLIFE SERVICE
Anchorage Fish & Wildlife Field Office
605 West 4th Avenue, Room G-61
Anchorage, Alaska 99501-2249

In reply refer to:
AFWFO

August 5, 2011

Ms. Melinda Brunner
Alaska Department of Environmental Conservation
Contaminated Sites - Reuse & Redevelopment Program
610 University Avenue
Fairbanks, AK 99709

Re: ESA Comments on Kwethluk site assessment and cleanup (*Consultation number 2011-0182*)

Dear Ms. Brunner:

This responds to your email of August 1, 2011, to Ellen Lance, requesting our concurrence that the above-referenced project is not likely to affect any endangered or threatened species. We are providing the following comments pursuant to section 7 of the Endangered Species Act of 1973 (16 U.S.C. 1531 *et seq.* as amended; ESA).

Our records indicate that there are no federally listed or proposed species, or designated or proposed critical habitat, within the action area of the proposed project. Therefore, requirements of section 7 of the ESA have been satisfied, and no further consultation pursuant to ESA section 7 is required at this time. However, you are required to re-initiate section 7 consultation, on behalf of EPA, if project plans change, if a new species is listed, or critical habitat is determined that may be affected by the identified action.

This letter relates only to federally listed or proposed species, and designated or proposed critical habitat under the jurisdiction of the U.S. Fish and Wildlife Service; it does not address species under the jurisdiction of the National Marine Fisheries Service, or other responsibilities under the Fish and Wildlife Coordination Act, Clean Water Act, National Environmental Policy Act, Marine Mammal Protection Act, or Bald and Golden Eagle Protection Act, or other legislation.

Thank you for your cooperation in protecting and enhancing endangered, threatened, and other rare species in Alaska. If you have any questions, please contact me at (907) 271-2768 and refer to consultation number 2011-0182.

Sincerely,

Judy Jacobs
Endangered Species Biologist

T:\s7\2011 sec 7\Judy\Completed Consultations\2011-0182 Kwethluk site cleanup concurrence.doc

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ORGANIZED VILLAGE OF KWETHLUK
Kwethluk Indian Reorganization Act Council
P.O. Box 130, 147 Jay Hammond Way, Kwethluk, Alaska 99621
Phone: (907) 757-6714/6715, Fax: (907) 757-6328, Email: kwethlukira @ gmail.com

March 12, 2012

Alaska Department of Environmental Conservation
Attention: John Carnahan, Brownsfield Coordinator
610 University Avenue
Fairbanks, Alaska 99709

Greetings Mr. Carnahan,

The Organized Village of Kwethluk (OVK) is requesting assistance from the Alaska Department of Environmental Conservation (DEC) and the Alaska Native Tribal Health Consortium (ANTHC) in the clean-up of the Former Joseph Guy Memorial Community Center site in Kwethluk, Alaska. The community fully supports this clean-up project, as evidenced by the discussion during the public meeting held in Kwethluk to discuss the project on August 30, 2011. The meeting was attended by representatives from the Organized Village of Kwethluk, City of Kwethluk, Yukon-Kuskokwim Health Corporation, DEC's solid Waste Program, and ANTHC's Division of Environmental Health and Engineering.

Based on the results of EPA's 2010 Targeted Brownsfield Assessment (final report dated March 2011) and documented discussions between DEC's risk assessor and EPA's Resource Conservation and Recovery Act (RCRA) specialist on file with DEC, OVK has determined that the waste and debris to be removed from the site does not constitute a regulated RCRA waste and can therefore be handled as solid waste for disposal.

Please reply with your concurrence to our determination in this matter.

Quyanaq'vaa
(Thank You Very Much!)

Martin Andrew, *President*

Herman N, Evan, *Tribal Administrator*

Cc: City of Kwethluk, Boris Epchook, *Mayor*

STATE OF ALASKA

DEPARTMENT OF ENVIRONMENTAL CONSERVATION
DIVISION OF SPILL PREVENTION AND RESPONSE
CONTAMINATED SITES PROGRAM

SEAN PARNELL, GOVERNOR
610 University Avenue
Fairbanks, AK 99709-3643
PHONE: (907) 451-2166
FAX: (907) 451-5105
www.dec.state.ak.us

File: 2424.57.001

March 12, 2012

Herman N. Evan, Tribal Administrator
Organized Village of Kwethluk
Kwethluk Indian Reorganization Act Council
P.O. Box 130
Kwethluk, Alaska 99621

Re: Concurrence with Solid Waste Determination for Disposal

Dear Mr. Evan:

Thank you for your letter regarding the clean-up of the Former Joseph Guy Community Center, which is being coordinated between the Alaska Department of Environmental Conservation (DEC), the Alaska Native Tribal Health Consortium, the Organized Village of Kwethluk, and the City of Kwethluk.

Having reviewed the Targeted Brownfield Assessment report (March 2011), and our records of correspondence between DEC's risk assessor and the U.S. Environmental Protection Agency's Resource Conservation Recovery Act specialist, we concur with your determination that the waste and debris generated by the clean-up project can be handled as solid waste for disposal.

Please contact me directly if you have any questions.

Sincerely,

John Carnahan
Brownfield Coordinator
Environmental Program Specialist

cc: Boris Epchook, City of Kwethluk
Lisa Nicholson, OASIS Environmental
Michael Roberts, ANTHC
Lori Aldrich, DEC

G:\SPAR\CS\Brownfield Site Files (57)\2424 Kwethluk\2424.57.001 Former Joseph Guy Community Center (Kwethluk)\Concurrence with OVK SW Determination March 12 2012.docx

City of Kwethluk
P.O. Box 50
Kwethluk, Alaska 99621
907-757-6022 Telephone 907-757-6498 Fax
kwtocity@unicom-alaska.com

March 9, 2012

Mr. John B. Carnahan
Brownsfield Coordinator-Contaminated Sites Program
ADEC Spill Prevention and Response
610 University Avenue
Fairbanks, Alaska 99709

Re: Community Center Cleanup Project

Dear Mr. J. Carnahan,

This letter is to follow up our earlier conversation regarding the Community Center Cleanup Project, formerly the Joseph Guy Community Building, and the proposed action plan for debris removal and cleanup of the burned out site.

I appreciate your briefing on the upcoming activities that are to occur during the first week of April 2012 regarding the building and the action plans recommended by the Alaska Native Tribal Health Consortium and the Environmental Remediation Contractor.

The Community of Kwethluk has no objections to the plan for removal of the burned out material and their transportation to a "permitted" landfill, which in this case would be the City of Bethel ADEC Permitted Landfill by means of ground transportation (ice road).

As for the removal and remediation of contaminated soil from the site, the City of Kwethluk, as owner and operator of the landfill, would dedicate a section at the southern end of the community landfill for a cleaning and processing center and site for contaminated soil.

If you have further questions and or concerns, please contact the City Manager, Mr. David Epchook, at the Kwethluk City Office at 907-757-6022 or 907-757-6499.

Respectfully,
City of Kwethluk

Boris L. Epchook, Mayor

Cc: Mr. Martin Andrew, President, Organized Village of Kwethluk
Mr. Chariton A. Epchook, Chairman, Kwethluk, Incorporated
Mr. John Hutchinson, ANTHC, Anchorage
Kwethluk Sanitation Committee
File

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APPENDIX C

XRF Soil Analysis Instructions

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Using Your Analyzer

This section discusses the basics of using your analyzer, no matter the specific type of analysis you wish to perform. First we go over analyzer safety, particularly radiation safety. Using an X-ray based analyzer safely is very important, and not difficult, provided you read, understand, and follow these guidelines. Secondly, we outline the startup procedure we recommend for daily use to ensure that your analyzer is performing properly and at its most efficient level.

Safely and Effectively Using Your Analyzer

CAUTION Niton analyzers are not intrinsically safe analyzers. All pertinent Hot Work procedures should be followed in areas of concern.

Radiation and General Safety

WARNING Always treat radiation with respect. Do not hold your analyzer near the measurement window during testing. Never point your analyzer at yourself or anyone else when the shutter is open.

Radiation and General Safety

This section covers topics related to radiation safety and general safety when using a Thermo Scientific Niton XL3 analyzer. At a minimum all operators of the analyzer should be familiar with the instructions provided in this chapter in order to handle the analyzer in a safe manner. In addition to reading the information presented on the following pages, Thermo Fisher Scientific recommends that instrument users participate in a radiation safety and operational training class.

Radiation Protection Basics

The Niton Model XL3t analyzer contains an x-ray tube which emits radiation only when the user turns the x-ray tube on. When the x-ray tube is on and the shutter is open, as during a measurement, the analyzer emits a directed radiation beam - see Figures 1 and 2. Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure: Time, Distance, and Shielding.

The Niton Model XL3p analyzer contains a radioactive sealed source. Radiation from this source is fully contained within the device when not in use and allowed to escape through the measurement window only while the user is analyzing a sample. Radiation emission is controlled by a shutter. The analyzer emits a directed radiation beam (See Figure 1 and Figure

2) when the shutter is open during a measurement. Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure: Time, Distance, and Shielding.

Time

The longer you are exposed to a source of radiation the longer the radiation is able to interact in your body and the greater the dose you receive. Dose increases in direct proportion to length of exposure.

Distance

The closer you are to a source of radiation, the more radiation strikes you. Based on geometry alone, dose increases and decreases with an inverse-squared relation to your distance from the source of radiation (additional dose rate reduction comes from air attenuation). For example, the radiation dose one foot from a source is nine times greater than the dose three feet from the source. Remember to keep your hands and all body parts away from the front end of the analyzer when the shutter is open to minimize your exposure.

Shielding

Shielding is any material that is placed between you and the radiation source. The more material between you and the source, or the denser the material, the less you will be exposed to that radiation. Supplied or optional test stands are an additional source of shielding for analysis. A backscatter shield accessory is also available and may be appropriate in some applications.

Exposure to Radiation

Human dose to radiation is typically measured in rem, or in one-thousandths of a rem, called millirem (mrem), 1 rem = 1000 mrem. Another unit of dose is the Sievert (Sv), 1 Sv = 100 rem. The allowable limit for occupational exposure in the U.S (and many other countries) is 5,000 mrem/year (50 mSv/year) for deep (penetrating) dose and 50,000 mrem/year (500 mSv/year) for shallow (i.e., skin) dose or dose to extremities. Deep, shallow, and extremity exposure from a properly used Niton XL3t analyzer should be less than 200 mrem per year, (2.0 mSv per year) even if the analyzer is used as much as 2,000 hours per year, with the shutter open continuously. The only anticipated exceptions to the 200 mrem maximum annual dose are: 1) routine and frequent analysis of plastic samples without use of a test stand, backscatter shield, or similar additional protective measures, or 2) improper use where a part of the body is in the primary beam path.

Note NEVER OPERATE THE DEVICE WITH A PART OF YOUR BODY IN THE PRIMARY BEAM PATH OR WITH THE PRIMARY BEAM PATH DIRECTED AT ANYONE ELSE.

Also, consider the use of protective accessories such as a shielded test stand or backscatter shield (or equivalent) when performing routine and/or frequent analysis of any of the following:

- plastic (or similarly low density) samples,
- thin samples (such as foils, circuit boards, and wires)
- samples that are smaller than the analysis window.

Shown in Table 1are the typical background radiation doses received by the average member of the public. The radiation dose limits for radiation workers in the US are also shown in Table 2.

Table 1. Typical Radiation Doses Received (Source: NCRP 1987)

Category	Dose in mrem	Dose in mSv
Average total dose in US (annual)	360	3.6
Average worker exposure (annual)	210	2.1
Average exposure for an underground miner	400	4.0
Exposure for airline crew (1,000 hours at 35,000 ft)	500	5.0
Additional from living in Denver at 5300' (annual)	25	.25
Additional from 4 pCi/l radon in home	1,000	10.0
Typical Chest X-Ray	6	0.06
Typical Head or Neck X-Ray	20	0.2
Typical pelvis/hip x-ray	65	0.65
Typical lumbar spine x-ray	30	0.3
Typical Upper G.I. x-ray	245	2.45
Typical Barium enema x-ray	405	4.05
Typical CAT scan	110	1.10

Table 2. Annual Occupational Dose Limits for Radiation Workers
(Source: Code of Federal Regulations Title 10, Part 20)

Category	Dose in mrem	Dose in mSv
Whole Body	5000	50
Pregnant Worker (during gestation period)	500	5
Eye Dose Equivalent	15,000	150
Shallow dose equivalent to the skin or any extremity or organ	50,000	500
Maximum allowable dose for the general public (annual)	100	1.0
For a Minor	500	5.0

Monitoring your radiation exposure

Individuals can be monitored for the radiation dose they receive by use of radiation dosimetry devices (dosimeters). Monitoring dose using a dosimeter can be a way of identifying improper use and at the same time demonstrating proper use. In some locations, dosimetry is required by regulations and in others it is optional. It is normally required when the user could reasonably be expected to receive in excess of 10% of the annual dose limit. Thermo Fisher Scientific recommends that you determine and obey the local regulatory requirements concerning radiation monitoring of occupational workers.

Two common types of dosimeters are whole-body badges and ring badges. Whole body badges are often attached to the user's torso (e.g., clipped to the collar, shirt pocket, or waist as appropriate). A ring badge is worn on the finger as a measure of maximum extremity dose. When worn, the specific location of the dosimeter should be that part of the body that is expected to receive the highest dose. This location will depend on how the analyzer is used and so it may not be the same for all users. Dosimetry services are offered by many companies. Two companies offering dosimetry services in the USA and much of the world are:

Table 3. Dosimeters

Company	Global Dosimetry Solutions	Landauer, Inc.
Address	2652 McGaw Avenue	2 Science Road
City and State	Irvine, CA 92614	Glenwood, IL 60425-9979
Website	www.dosimetry.com	www.landauerinc.com
Phone Number	(800) 251-3331	(800) 323-8830

Note Wearing a dosimeter badge does not protect you against radiation exposure. A dosimeter badge only measures your exposure (at the dosimeter location).

Pregnancy and Radiation Exposure

International guidance documents (e.g., ICRP Publication 60 and NCRP Publication 116*) recommend that the radiation dose to the embryo/fetus of a pregnant woman should not exceed a total of 500 mrem (10% of normal radiation worker limit) during the gestation period. While this dose limit exceeds the dose limit to a trained operator, pregnant workers may want to take special precautions to reduce their exposure to radiation. For more information see the U.S. NRC Regulatory Guide 8.13 "Instruction Concerning Prenatal Radiation Exposure" which can be found on the resource CD.

* The International Commission on Radiological Protection, ICRP, is an independent Registered Charity, established to advance for the public benefit the science of radiological protection, in particular by providing recommendations and guidance on all aspects of protection against ionizing radiation.

* The National Council on Radiation Protection and Measurements (NCRP) was chartered by the U.S. Congress in 1964 as the National Council on Radiation Protection and Measurements.

How to Use the Niton XL3t Analyzer Safely

The Niton XL3t analyzer is designed to be safe to operate provided that it is used in accordance with manufacturer's instructions. Under conditions of normal use, monitored operators seldom receive a measurable dose and have not been known to receive in excess of 10% of the annual occupational dose limits (a criteria that would require monitoring under regulation in the U.S.). In addition to proper use of the XL3t, it is recommended that you follow these precautions to ensure your safety and the safety of those around you.

Know where the beam is

The primary beam is a directed beam out of the front of the analyzer that can have high dose rates. The secondary beam, or scattered beam, has much lower dose rates.

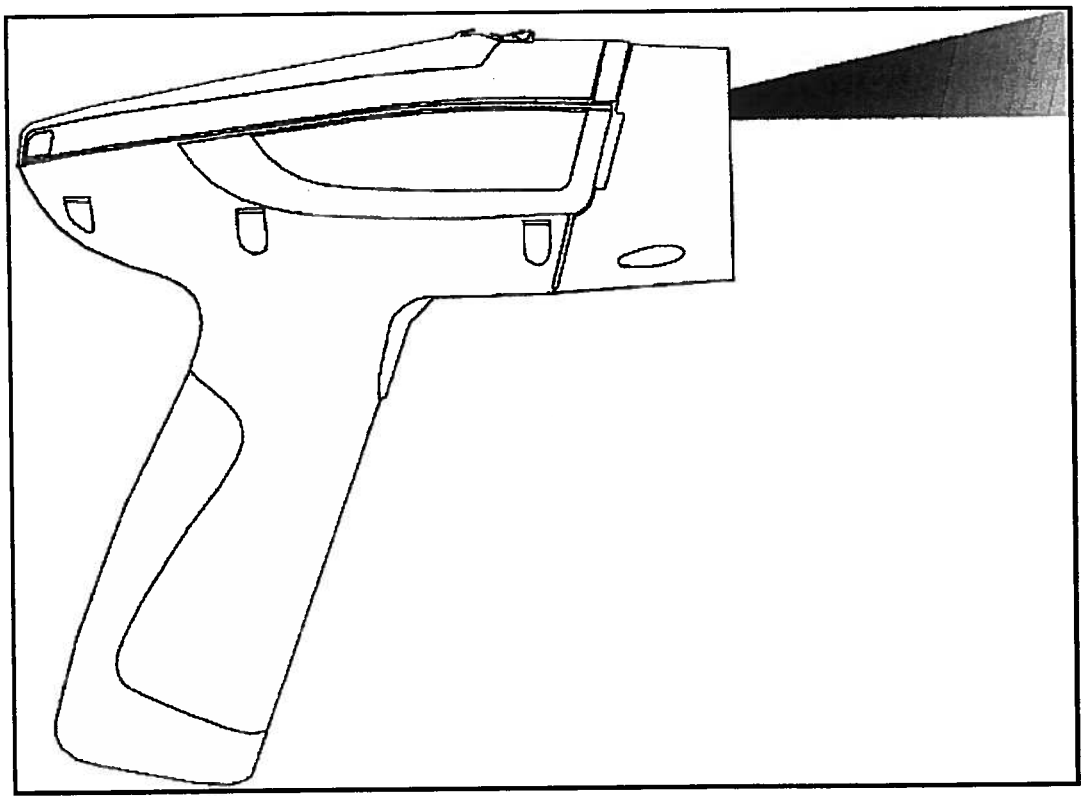


Figure 2. Primary Beam

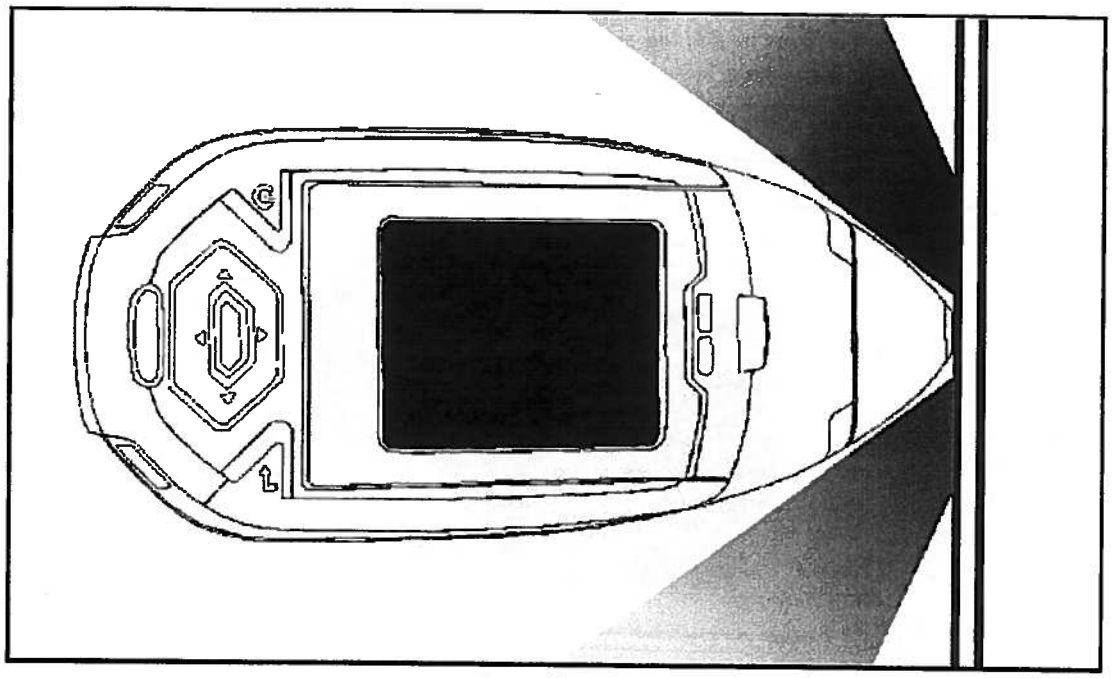


Figure 3. Secondary (Scattered) Beam

The Shutter-Open Indicator Lights

When the lights are flashing, the primary beam is on, and radiation is being emitted from the front of the analyzer.

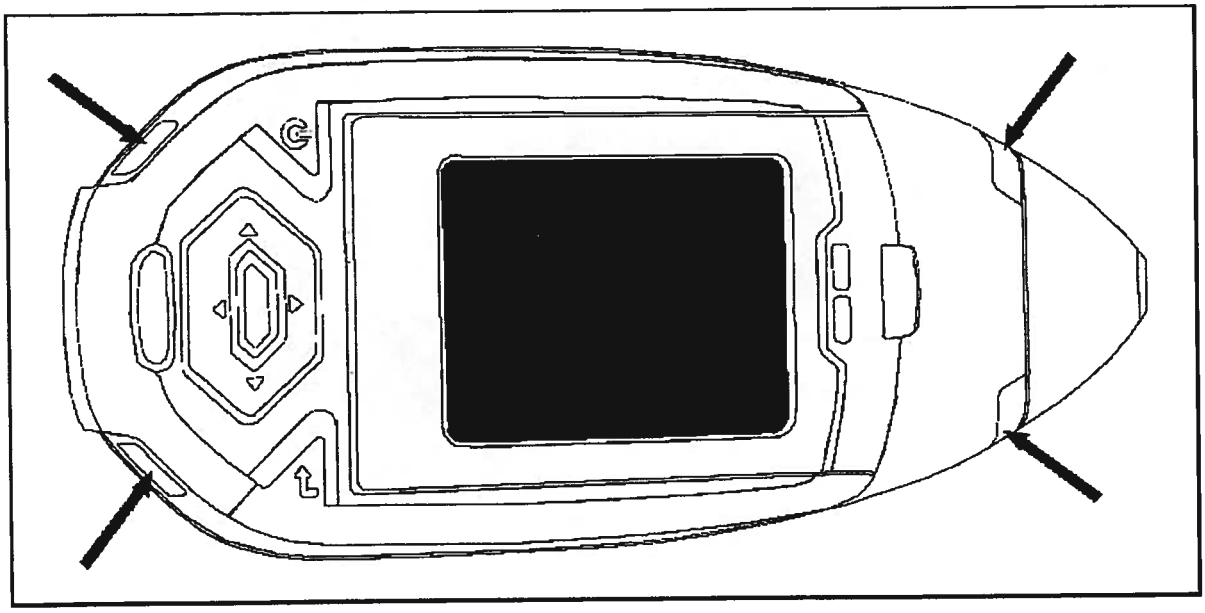


Figure 4. The X-ray Beam Indicator Lights

Handle and Use with Respect

Avoid holding the front of the analyzer when the x-ray tube is energized and the shutter is open. Never point the instrument at yourself or anyone else when the shutter is open and the x-ray tube is energized. Never look into the path of the primary beam.

Follow a Radiation Protection Program

Your organization should establish, document, and follow a Radiation Protection Program. An example of such a program can be found on the resource CD (provided with the instrument).

Take Proper Care of your Niton XL3

Keeping your analyzer maintained in good condition will help minimize the risk of accidental exposure. Mechanical malfunction of the shutter can be avoided by maintaining the measurement window, as described in the User Guide. This prevents foreign objects from entering your analyzer

Avoid Over-Exposures

Direct contact with the window could result in overexposures in the times indicated inTable 4 below.

Table 4. Potential Exposure Limit Times

Location of Dose	Limit	Time to Reach Limit
Deep Dose / Whole Body	5 rem (50 mSv)	2.1 minutes
Shallow Dose / Extremities	50 rem (500 mSv)	0.95 minutes
Member of Public (i.e. untrained operator)	0.1 rem (1 mSv)	2.5 seconds*

Extremity is defined by the NRC as the hand, elbow, arm below the elbow, foot, knee, or leg below the knee. Whole Body is defined by the NRC as the head, trunk (including male gonads), arms above the elbow, or legs above the knee.
*Based on maximum deep dose rate and US exposure limit.

Safe Handling of Samples

As mentioned many times in this chapter, never place any part of your body in the path of the x-ray beam. There is always a safe way to handle samples whether they are small, irregularly shaped, or of low density. Never look into the path of the primary beam.

Small Samples

A small sample would be any sample that is smaller than the measurement window. Small samples present a unique risk because they don't block the entire beam path. The difficulty with placing small samples down on a work surface to analyze them is that you may get readings from the work surface that interfere with analytical results. A test stand is an effective way of analyzing small samples accurately and safely. Never hold samples during analysis or look into the path of the primary beam.

Irregularly Shaped Samples

Irregularly shaped samples may not allow the proximity button to be depressed, or they may not entirely cover the primary beam and cause additional scattering. A back scatter shield is a safe way of reducing your radiation exposure while effectively analyzing an irregularly shaped sample.

Low Density Materials (such as plastics).

X-rays are attenuated more through denser materials and less through low density materials such as plastic. This causes higher dose rates in the scattered radiation. If you are frequently handling low density samples, you should consider the use of test stands, backscatter shields, or the equivalent.

Niton XL3t Radiation Profile

Radiation Meter Information

Model: Bicron MicroRem

SN: 2057

Cal Due: 10/10/2009

Background Radiation Level

<0.01 mr/hr

Table 1-4 - Niton XL3t Radiation Profile - Scatter Measurements - mRem/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	Plastic	40	3.5	2
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	Soil	8	0.4	0.07
20	100	S, P, T, M, E (Low Filter)	Aluminum	0.015	0.01	0.01
20	100	S, P, T, M, E (Low Filter)	Stainless	0.015	0.01	0.01
20	100	S, P, T, M, E (Low Filter)	Plastic	0.13	0.015	0.015
20	100	S, P, T, M, E (Low Filter)	Soil	0.015	0.015	0.015
15	100	IP, EM (Low Filter)	Aluminum	0.015	0.015	0.015
15	100	IP, EM (Low Filter)	Stainless	0.015	0.015	0.015

* GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil, E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Scatter Measurements off various substrates - Dose Rates in mRem/hr

Table 1-5 - Niton XL3t Radiation Profile - Scatter Measurements - µSv/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, GM, S, T, IP, PM, DA, M, E (P (Main Filter) S, T, M, E (High Filter)	Plastic	400	35	20
50	40	EM, GM, S, T, IP, PM, DA, M, E (P (Main Filter) S, T, M, E (High Filter)	Soil	80	4	0.7
20	100	S, P, T, M, E (Low Filter)	Aluminum	0.15	0.1	0.1
20	100	S, P, T, M, E (Low Filter)	Stainless	0.15	0.1	0.1
20	100	S, P, T, M, E (Low Filter)	Plastic	1.3	0.15	0.15
20	100	S, P, T, M, E (Low Filter)	Soil	0.15	0.15	0.15
15	100	IP, EM (Low Filter)	Aluminum	0.15	0.15	0.15
15	100	IP, EM (Low Filter)	Stainless	0.15	0.15	0.15

Notes:
Scatter measurements were taken at a radius of 5 or 30 cm around the nose of the analyzer with the highest scatter dose rate being recorded.
Scatter Measurements off various substrates - Dose Rates in µSv/hr
* GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil, E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Table 1-6 Niton XL3t Radiation Profile - In Beam Measurements - Rem/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30 cm Shallow
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	110	410	8.4	1.3
20	100	S, P, T, M, E (Low Filter)	150	3200	0.52	0.05
15	100	IP, EM (Low Filter)	14.0	1100	0.43	0.042

In Beam Measurements - Dose Rates in Rem/hr
* GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil,
E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic
Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-7 Niton XL3t Radiation Profile - In Beam Measurements - mSv/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Shallow
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	1100	4,100	84.0	13
20	100	S, P, T, M, E (Low Filter)	1500	32000	5.2	0.50
15	100	IP, EM (Low Filter)	140	11000	4.3	0.42

Notes:
In beam dose rates were measured using thermoluminescent dosimeters (TLDs) or Optically Stimulated Luminescent Dosimeters (OSL).
In Beam Measurements - Dose Rates in mSv/hr
* GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil,
E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic
Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Niton XL3t GOLDD Plus Radiation Profile

Table 1-8 - Niton XL3t GOLDD Plus Radiation Profile - In Beam Measurements - mSv/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Deep
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	440	1300	74	5.3
50	40	GM, PM, M, S, IP (Main)	1500	3800	360	22
20	100	EM, P, TA, M, S, T (Low)	690	19000	190	9.2
15	133.3	GM (Low)	240	13000	50	2.3
8	200	GM, P, M (Light)	0.30	17000	0.10	<0.003

Notes:
*SAMPLE TYPES (MODES)
GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin
Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-9 - Niton XL3t GOLDD Plus Radiation Profile - In Beam Measurements - Rem/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Deep
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	44	130	7.4	0.53
50	40	GM, PM, M, S, IP (Main)	150	380	36	2.2
20	100	EM, P, TA, M, S, T (Low)	69	1900	19	0.92
15	133.3	GM (Low)	24	1300	5.0	0.23
8	200	GM, P, M (Light)	0.030	1700	0.010	<0.0003

Notes:
*SAMPLE TYPES (MODES)
GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin
Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-10 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - mRem/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Steel	0.14	<0.01	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Aluminum	2	<0.01	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Soil	2	0.04	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Plastic	10	0.45	6
50	40	GM, PM, M, S, IP (Main)	Steel	0.3	<0.01	<0.01
50	40	GM, PM, M, S, IP (Main)	Aluminum	4	0.01	0.01
50	40	GM, PM, M, S, IP (Main)	Soil	4	0.09	<0.01
20	100	EM, P, TA, M, S, T (Low)	Soil	<0.01	<0.01	<0.01
20	100	EM, P, TA, M, S, T (Low)	Plastic	0.07	<0.01	<0.01
15	133.3	GM (Low)	Steel	<0.01	<0.01	<0.01

Table 1-10 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - mRem/hr

15	133.3	GM (Low)	Aluminum	<0.01	<0.01	<0.01
8	200	GM, P, M (Light)	<0.01 (no detectable scatter radiation) at any location for steel, aluminum, soil, or plastic sample types			

Notes:
*SAMPLE TYPES (MODES)
GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin

Table 1-11 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - µSv/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Steel	1.4	<0.1	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Aluminum	20	<0.1	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Soil	20	0.4	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Plastic	100	4.5	60
50	40	GM, PM, M, S, IP (Main)	Steel	3	<0.1	<0.1
50	40	GM, PM, M, S, IP (Main)	Aluminum	40	0.1	0.1

Table 1-11 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - µSv/hr

50	40	GM, PM, M, S, IP (Main)	Soil	40	0.9	<0.1
20	100	EM, P, TA, M, S, T (Low)	Soil	<0.1	<0.1	<0.1
20	100	EM, P, TA, M, S, T (Low)	Plastic	0.7	<0.1	<0.1
15	133.3	GM (Low)	Steel	<0.1	<0.1	<0.1
15	133.3	GM (Low)	Aluminum	<0.1	<0.1	<0.1
8	200	GM, P, M (Light)	<0.1 (no detectable scatter radiation) at any location for steel, aluminum, soil, or plastic sample types			

Note *SAMPLE TYPES (MODES)

GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin

Niton XL3p Radiation Profile

Table 1-12 - Niton XL3p Radiation Profile - In Beam Measurements

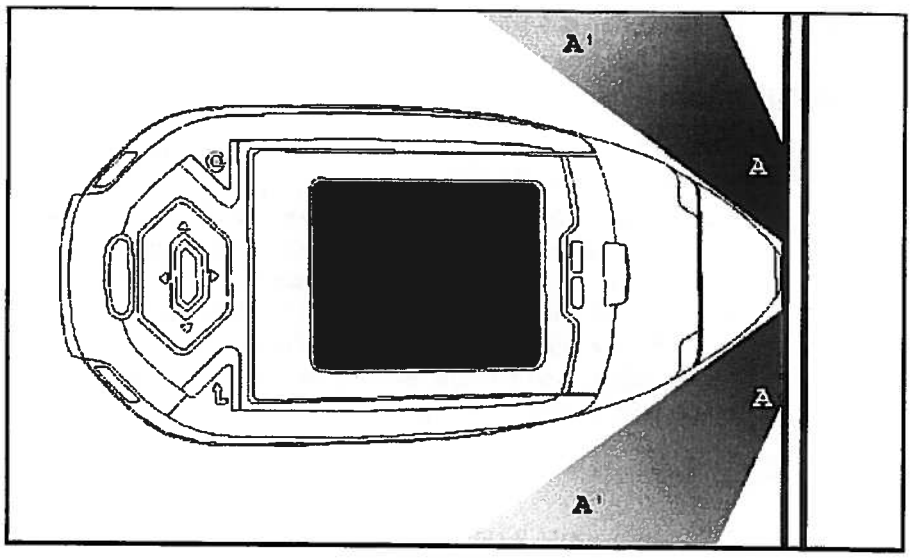
Distance From Window	Dose Rate (mSv/hr)	Dose Rate (mrem/hr)
5 cm	0.45	45
30 cm	0.03	3.0
100 cm	0.003	0.3

Table 1-13 - Niton XL3p Radiation Profile - Scatter Measurements - mSv/hr

Location	Plastic Substrate	Wood Substrate	Soil Substrate	Aluminum Substrate	Steel Substrate
Max Scatter @ 5cm from Snout (A')	0.06	0.03	0.018	0.01	0.0042
Max Scatter @ Trigger (B)	0.0038	0.002	0.0015	0.0048	0.0003

Table 1-14 - Niton XL3p Radiation Profile - Scatter Measurements - mRem/hr

Location	Plastic Substrate	Wood Substrate	Soil Substrate	Aluminum Substrate	Steel Substrate
Max Scatter @ 5cm from Snout (A')	6.0	3.0	1.8	1.0	0.42
Max Scatter @ Trigger (B)	0.38	0.2	0.15	0.48	0.03



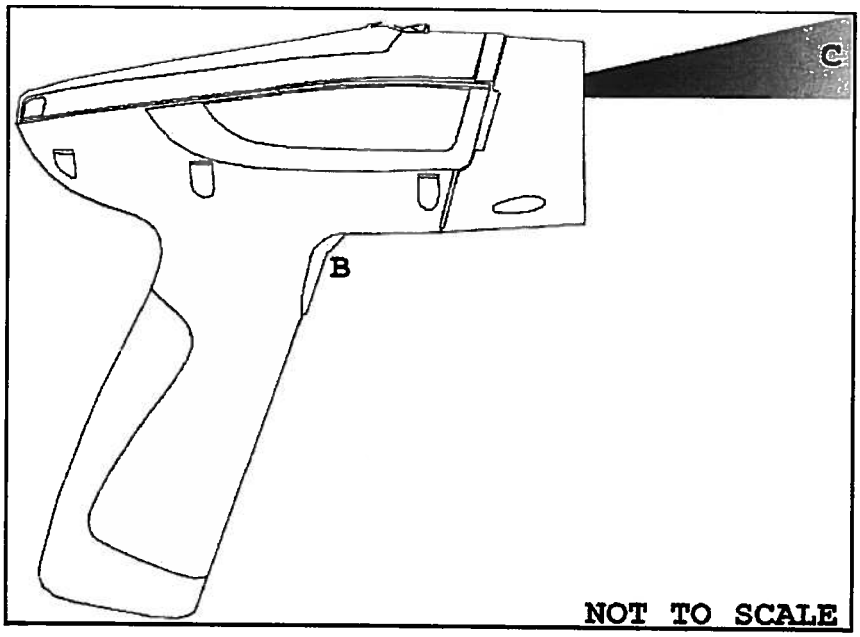


Figure 5. Primary and Secondary Dose Locations (Not to Scale)

Primary Radiation

Niton XL3t and XL3t GOLDD

Primary radiation is radiation that is produced by the analyzer and emitted out through the measurement window. Individuals should never place any part of their body in the primary beam path when the x-ray tube is on. There should always be a sample in contact with the measurement window when the x-ray tube is on. The sample will absorb most of the primary-beam radiation unless it is smaller than the instrument's measurement window or of low density and/or thickness. Caution should be taken when analyzing samples that are small, thin, and/or low in density as they may allow much more of the primary beam to escape. In-beam primary radiation dose rates are listed in Table 1-6, 1-7, 1-8, 1-9, and their location identified relative to the analyzer in Figure 4 as Dose Point C.

Niton XL3p

Primary radiation is radiation that is produced by the analyzer and emitted out through the measurement window. Individuals should never place any part of their body in the primary beam path when the shutter is open. There should always be a sample in contact with the measurement window when the shutter is open. The sample will absorb most of the primary-beam radiation unless it is smaller than the instrument's measurement window or of low density and/or thickness. Caution should be taken when analyzing samples that are small, thin, and/or low in density as they may allow much more of the primary beam to escape. In-beam primary radiation dose rates are listed in Table 1-6, 1-7, 1-8, 1-9, and their location identified relative to the analyzer inFigure 4 as Dose Point C.

Secondary Radiation

Under conditions of normal and proper use, individuals can be exposed to secondary (or "scattered") radiation. Secondary radiation is low-level radiation that emanates from the sample being analyzed as a result of primary beam radiation scattering in the sample or primary beam radiation inducing fluorescent x-rays in the sample. Dose points A, A' and B in Figure 4 are examples of where you can encounter secondary radiation. The magnitude of this secondary radiation is sample dependent. Higher density samples such as steel will emit the lowest levels as they absorb most primary and secondary radiations. Lower density samples such as aluminum, wood, and especially plastic, will produce higher levels of secondary radiation.

Niton XL3t and XL3t GOLDD

Secondary radiation dose rates are listed in Tables 1-4, 1-5, 1-10, and 1-11, for a few common sample types over a wide range of densities.

Niton XL3p

Secondary radiation dose rates are listed in Tables 1-13 and 1-14 for a few common sample types over a wide range of densities.

Holding Samples

The operator is reminded that one should never hold samples during analysis, doing so will result in higher than necessary exposure to secondary radiation and could expose the operator directly to the much higher primary-beam dose rates.

Deep and Shallow Dose

You will find in the tables that shallow dose rates are listed for some dose points. All dose rates listed in the In-Beam Primary Radiation Tables are deep dose unless they are specifically identified as shallow dose. Deep dose is dose from penetrating radiation that is delivered to both skin and underlying tissues and organs and is the type most commonly referred to when describing external radiation hazards. Occupational deep dose is limited to a maximum of 5 rem (50 mSv) per year in the United States and most countries internationally. Deep dose is measured at 1.0 cm below the skin surface.

Shallow dose is often referred to as "skin dose" because it is a result of low penetrating radiation that only interacts with the skin. Shallow dose is limited to a maximum of 50 rem (500 mSv) per year in the United States and most countries internationally. Shallow dose is listed for primary in-beam dose points only because the low penetrating radiation that causes shallow dose is nearly all absorbed by a sample and does not produce any significant secondary radiation. Shallow dose is measured at a point 0.007 cm below the surface.

Proper and Improper Operation

Storage and Transportation

Storage

Regulations in nearly all locations will require that you store your analyzer locked in a secured area to prevent access, use, and/or removal by unauthorized individuals. Storage requirements will vary by location, particularly with regard to storage at temporary job sites or away from your primary storage location such as hotels and motels and in vehicles. You should contact your local Radiation Control Authority to identify the specific storage requirements in your jurisdiction.

Transportation

Niton XL3t and XL3t GOLDD

There are no X-ray tube specific US Department of Transportation (DOT) or International Air Transport Association (IATA) radiation regulations regarding shipping the Niton XL3t analyzer. It is recommended that you ship the analyzert in its carrying case and an over-pack to protect the sensitive measuring equipment inside the analyzer. Do NOT ship the analyzer with the battery pack connected to the analyzer.

Niton XL3p

For Thermo Fisher Scientific, Niton Analyzers (Niton Analyzers), in the United States, the government agency that has primary authority and regulations which apply to transportation is the Department of Transportation (DOT) (Code of Federal Regulations 49 Parts 100 to 185).

In addition, the EPA, OSHA, and the NRC also have regulations that touch on the transportation of hazardous substances.

The International Air Transport Association (IATA) has recommendations that every major air carrier has incorporated into their policies for the air transport of hazardous substances.

The enforcement of DOTregulations is carried out by the Department of Transportation, the Federal Aviation Administration, the Federal Highway Administration, the Federal Railroad Administration, the Coast Guard, and Customs and Border Protection. State agencies may also enforce state DOT regulations.

The major aspects of the regulations include:

• "training to recognize hazards and how to safely deal with hazardous substances,

• "classification and identification of packages to inform of hazards,

• "protective packaging to safely transport hazardous materials,

• "hazard communication to inform personnel of hazards in a package,

and

• "incident reporting to inform regulators of incidents.

As part of shipping hazardous substances, you should be trained in:

• "a general awareness and a familiarity with the general provisions of the DOT and hazardous materials regulations,

• "training that is function specific and be applicable to the daily work performed,

• "the recognition and identification of hazardous substances,

• "the specific requirements for functions performed,

• "security measures to keep a package secure.

• "safety issues as related to safe handling and hazard communication.

• "knowledge of emergency response information, self-protective procedures, and accident prevention procedures.

Employers are responsible for providing the proper training (every three years for DOT) to employees, testing employee knowledge, and record keeping.

The DOT Hazardous Material Regulations set the criteria for determining the hazard class and the proper shipping name for hazardous materials. The Hazard Classes as follows:

Class

1. Explosives

2. Gases

3. Flammable and Combustible Liquids

4. Flammable solids, combustible materials, and dangerous when wet materials

5. Oxidizers and organic peroxides

6. Toxic materials and infectious substances

7. Radioactive Materials

8. Corrosive Materials

9. Miscellaneous dangerous goods

Niton analyzers with radioisotopes are Hazard Class 7. The Hazard Class of the material being transported tells you which Parts of the regulations are required.

A UN number is assigned to each type of hazardous material. It is the letters "UN" followed by a four digit numerical code, which allows emergency responders to identify the chemical being shipped. The UN number for Niton analyzers is "UN 2911".

There is also an official name designation for Niton Analyzers called the Proper Shipping Name. The proper Shipping Name for the Niton Analyzers is "Radioactive material, excepted package-instruments and articles".

A reportable quantity (RQ) is listed in DOT regulations for each hazardous material (e.g., each radioactive isotope). If you are shipping more than the reportable quantity in a package, that package must be marked clear and legibly with the letters "RQ". The Niton XL3p analyzer contains a 30 mCi (1.11 GBq) Am-241 source and is therefore considered a reportable quantity requiring the RQ marking when being transported.

Also, the quantity of Am-241 that is used in a Model XL3p Analyzer requires a special form certification for transport as an excepted package. This Special Form certificate must accompany the instrument during shipment. Thermo Fisher Scientific will provide the Special Form certificate for any analyzer with Am-241."

The type of protective packaging used is dependent on the nature of the material to be packaged. All packaging must be designed to prevent a release of hazardous material during normal transportation or storage of the material. The classification of package used for Niton Analyzers is designated as an "Excepted Package". Always ship the analyzer in its original plastic case to ensure that the packaging used meets the regulatory requirements for an Excepted Package.

Shipments of radioactive materials must have proper Labeling and Marking.

Niton analyzers have a Marking requirement (i.e., UN number and RQ if applicable), but not a Labeling requirement (i.e., diamond shaped hazmat labels), and vehicles transporting these analyzers are not required to have "Placards".

When reading the DOT regulations, you will find the following information useful.

Thermo Fisher Scientific, Niton Analyzers are shipped:

• "Under the proper shipping name "Radioactive material, excepted package-instruments or articles" in accordance with 49 CFR 173.424,

• "with the radiation level at 10 cm from the unpacked instrument surface less than 10 mrem/hr (0.1 mSv/hr) "
Note A Niton Analyzer in proper condition will be less than 0.5 mrem/hr (0.005 mSv/hr) at 10 cm,

• "with the radiation level at the package surface less than 0.5 mrem/hr (0.005 mSv/hr)"
Note A Niton Analyzer in proper condition will be less than 0.05 mrem/hr (0.0005 mSv/hr) at the surface of the case,

• "with all radioactive sources as "solid", "sealed sources"

• "Am-241 listed in A1 column of 173.435 (270 Ci) (Special Form capsule)

• "with the package design meeting the requirements of 173.410

• "package marked with "UN2911"

• "with the Am-241 source, the package is marked with "RQ"

• "meeting the 173.424 criteria for labeling and marking requirements

For any shipment: Include in the package a current copy of the instrument Leak Test.

Include a list of emergency numbers in the package.

For Am-241, include the Special Form Certificate in the package.

Always ship in supplied plastic case, with the case secured against accidental opening. Always ship with the battery disconnected.

When shipping by air: Ship with the proper IATA marking (See IATA Dangerous Goods regulations Figure 10.7.8.A), UN 2911, and proper shipping name.

"RQ" marking and "dangerous goods declaration" are required.

When shipping by Ground: Ship under proper marking "UN2911"

"RQ" marking and shipping papers are required (Note: a dangerous goods declaration form can be used to meet the shipping paper requirement).

A "Dangerous Goods Declaration" can be obtained by the air carrier that you will be using. Instructions can also be obtained from the same source.

Carefully follow the directions given by the air carrier. Several typed copies will be required.

Shipping papers contain all of the same information as a Dangerous Goods Declaration, but do not have a specified format for that information.

- At a minimum, a properly prepared shipping paper clearly identifies the hazardous substance by its proper shipping name, hazard class and division, UN identification number, packing group (if applicable), and total quantity. It also has consigner information, consignee information, and a shipper's declaration that the package is in compliance with the DOT regulations.

The elements of hazard warning information are communicated through shipping documents, packaging markings, and written emergency response information.

The DOT & FAA Hazardous Materials Regulations require the carrier to report all incidents involving hazardous materials.

An "incident" involves the unintended release of hazardous materials (Am-241), suspected radioactive contamination, if the general public is evacuated for an hour or more, or the flight pattern or routine of an aircraft is altered.

For any "incident", contact the Company Radiation Safety Officer or Responsible Party and the state radiation control program.

Any "incident" needs to reported to the:

Hazardous Materials Information Center

1-800-467-4922

Mon-Fri 9AM-5PM Eastern

Leak Tests (Niton XL3p Only)

The Niton XL3p Series analyzer contains a radioactive source that must be periodically leak tested. The purpose of leak testing is to verify the integrity of the source encapsulation. A leak test sample is obtained by wiping exterior surfaces of the device with moderate pressure using a cotton swab, filter paper, or whichever wiping media is supplied by the analysis laboratory. Leak test samples are then typically analyzed at a laboratory, although some device users have the equipment and licensed authority to perform this analysis

Unless specified otherwise by your local authority or radioactive material license, the gauge must be leak tested at intervals not to exceed 6 months. In the US, leak test samples may be acquired by any end-user, however the analysis of the sample must be performed by an organization licensed to do so. If you are using a vendor to perform the laboratory analysis of the leak test sample, they will send you a leak test kit which comes with complete instructions for performing the test. These vendors will also typically send you a reminder when it is time to perform the next leak test on your instrument. Please follow the test kit instructions carefully, and promptly mail the test samples to the laboratory. They will send you a leak test certificate soon after. Keep one copy of the leak test certificate with the device at all times (i.e., in the case) and another copy safely on file.

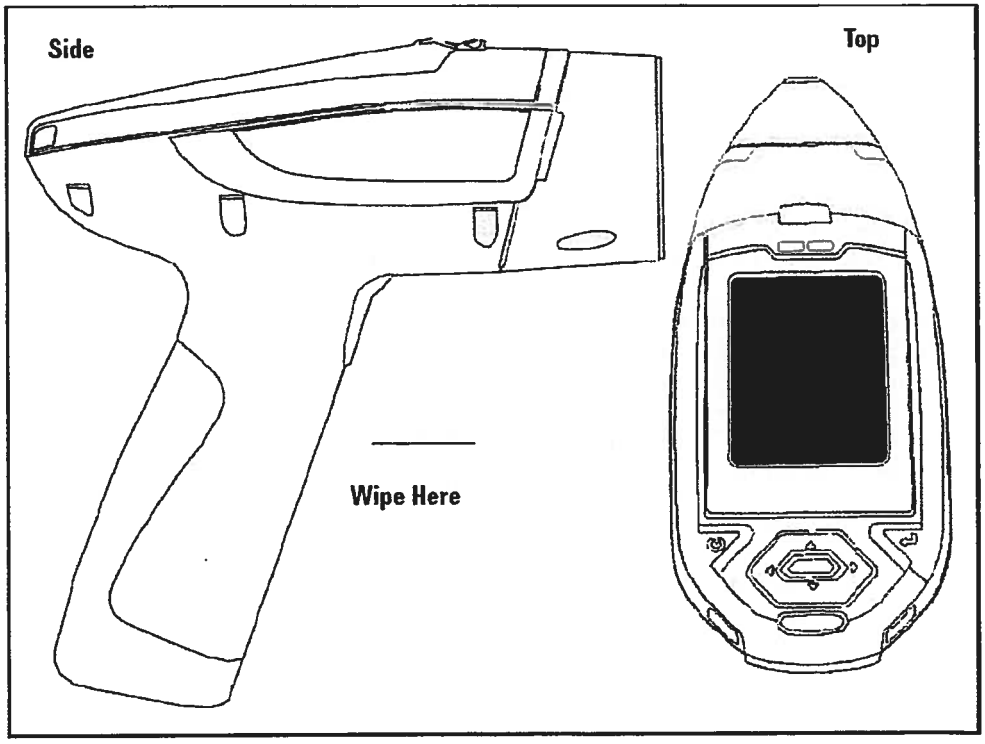


Figure 6. Wipe Test Locations

CAUTION REMOVE THE BATTERY WHILE PERFORMING A WIPE TEST TO BE SURE THAT THE SHUTTERS ARE NOT OPEN DURING THIS PROCEDURE!

Lost or Stolen Instrument

THIS PAGE CONTAINS EMERGENCY CONTACT INFORMATION THAT SHOULD BE AVAILABLE TO THE OPERATOR AT ALL TIMES.

If the Niton XL3t analyzer is lost or stolen, notify your Radiation Safety Officer (RSO) or the equivalent responsible individual at your company or institution immediately. Your company's RSO, as well as other important emergency contacts, are listed below. Your company RSO may need to notify the x-ray tube regulatory authority and the local police. It is also recommended that a notification is made to Thermo Fisher Scientific.

Damaged Instrument

Minor Damage

If the instrument is intact but there is indication of an unsafe condition such as a cracked case, a shutter mechanism failure, or the lights remain flashing after a measurement is terminated, follow these steps:

1. Stop using the instrument

2. Remove the battery. The x-ray tube can not produce radiation when the battery is disconnected. The instrument is now safe to handle.

3. Place the instrument securely in the holster.

4. Place the instrument in the carrying case that came with the instrument.

5. Notify your Radiation Safety Officer (RSO) or the equivalent responsible individual at your company or institution immediately.

6. You or your RSO should call Thermo Fisher Scientific at one of their contact numbers listed below for additional instructions and guidance.

Major Damage

If the instrument is severely damaged:

1. Perform the same steps as described above for minor damage. There will be no radiation hazard as long as the battery is removed from the instrument.

2. Place all components in a plastic bag and contact Thermo Fisher Scientific.

Emergency Response Information

Please Complete the Following Emergency Response Information and Keep with the Analyzer at All Times

NITON ANALYZER EMERGENCY CONTACT INFORMATION

The Company RSO is:_____

RSO Telephone Number:_____

Regulatory Agency Emergency Number:_____

Local Fire Department:_____

Local or State Police Department:_____

Thermo Fisher Scientific's Niton Analyzer Contact Numbers

Main Number (USA): (800) 875-1578

Additional Radiation Emergency #'s: (978) 790-8269 or (617) 901-3125

Outside the USA - Local Niton Service Center:_____

Europe

Niton Analyzers Europe

Munich, Germany

Phone: +49 89 3681 380

Fax: +49 89 3681 3830

Email: niton.eur@thermofisher.com

Asia

Niton Analyzers Asia

Hong Kong

Phone: +852 2869-6669

Fax: +852 2869-6665

Email: niton.asia@thermofisher.com

Registration and Licensing

As a user of a Niton XL3 analyzer, you may be required to register or obtain a license with your local radiation control authority. In the US, if you intend to do work with your analyzer in states other than your own, you may be required to register there as well. See the Safety and Compliance Web Hub for much more information.

Regarding Safety Devices for the Open Beam Configuration:

In the US, you may be required to file for an exemption, "variance letter", with your state if there is a requirement for a safety device that would prevent entry of an extremity into the primary beam. If you need assistance with the exemption letter, you may contact the radiation safety group.

Registration and Licensing FAQ

Soil Analysis

Niton XL3 X-Ray Fluorescence (XRF) Analyzer – Version 7.0 and Higher

Analyzing Soil Samples Using the Thermo Scientific Niton® XL3 Series X-Ray Fluorescence (XRF) Analyzer with Software Version 7.0 and Higher

Standard Operating Procedure

Note - Each user should read the Thermo Scientific Niton XL3 Series User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly (US only), or contact their local representative for product and safety training. For more information, visit www.niton.com.

PREPARATORY TASKS

1. Insert a charged battery into the analyzer and turn it on. See Battery Installation and Charging for detailed instructions. Follow the screen instructions and "Log On" as the operator using either the default password or one designated by the user in an NDU file.

2. Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.

3. Verify that the date is set properly for data tracking purposes. From the Main Menu, select the System icon then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by "Closing" out of the Specs screen and selecting the Date & Time icon from the System Menu. See Setting the Date and Time for detailed instructions. Select the Return Button to go back to the Main Menu.

4. (Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. See Connecting the XRF Analyzer to Your PC for more information.

5. During analysis and System Check, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.

6. From the Main Menu, select the System Check icon, and select the Yes Button. (Figure 1.)

6.1. System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.

6.2. If the unit shows a failure error, then perform a second System Check by selecting the Recheck Button. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.

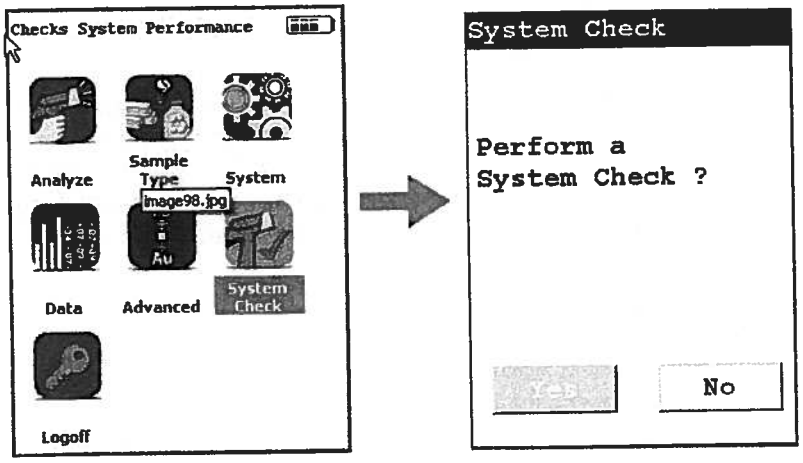


Figure 99. System Check Menu Path

7. Verify and/or change filter position time.

Thermo Scientific Niton XL3 analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements in different matrices. In Soil Mode, up to three filters may be selected, depending on the elements required. The amount of time that the analyzer spends in each filter position is user definable. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement. To verify or change the length of time each filter will be active, do the following:

7.1 From the Main Menu select the Advanced icon, then the Element Range icon (Figure 1-2)

In the drop-down menu, select Soils Mode.

7.2 Select the box next to High Range, Main Range, and Low Range so that a check mark appears in each box.

7.3 Enter the desired length of time for each filter. For example, set values of 60 seconds for the Main Range, 60 seconds for the Low Range, and 60 seconds for the High Range as shown in Figure 3.

7.4 Select the Save Button and return to the Main Menu by selecting the Return icon.

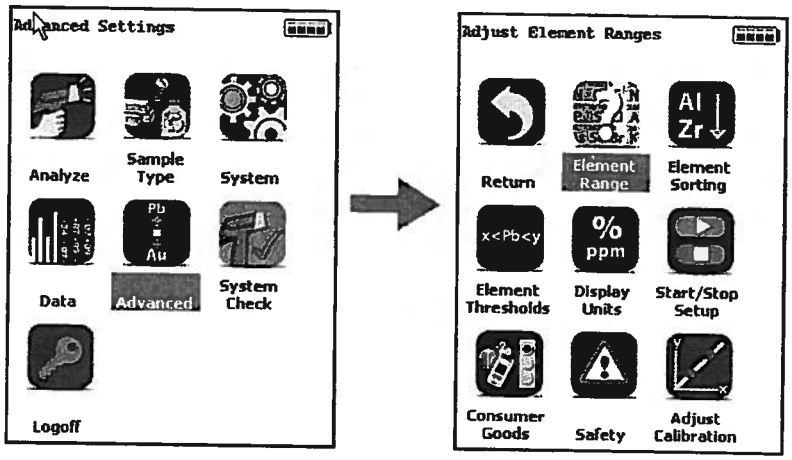


Figure 100. Element Range Menu Path

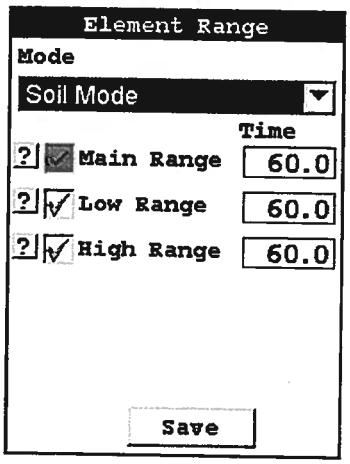


Figure 101. Setting Element Ranges

8.1 Verify instrument measurement accuracy (QC check). From the Main Menu, select the Sample Type icon, then the Soils & Minerals icon, then the Soils icon. You can also use this procedure with TestAll Geo Mode. See GENERAL TESTING PROTOCOL below for more information.

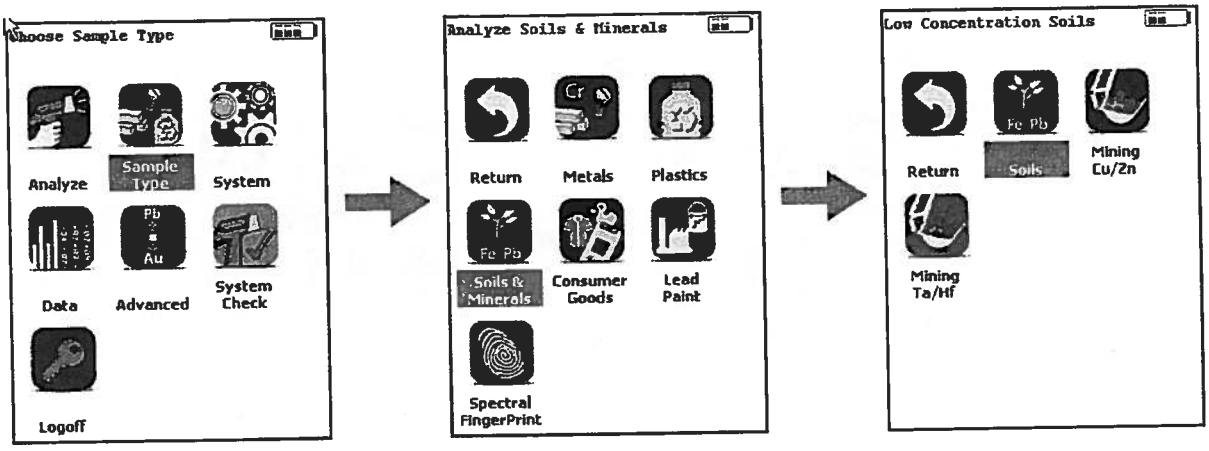


Figure 102. Soil Analysis Menu Path

8.1.1. Place TILL4 Reference Sample (P/N 180-601) (or any other reference sample supplied with your instrument) in the test stand for analysis, or set the reference sample on a clean surface. The unlabeled side should be presented to the spectrometer. Place the front end of the instrument against the sample with the Analysis Window centered on the sample.

8.1.2. Take one 180-second measurement on this reference sample (60 sec. on each Range). Ensure that the analyzer has the maximum test time set to 180 seconds or greater. From the Main Menu select the Advanced icon, then the Start/Stop Setup icon. Edit the Max. Time dialog box to display the proper setting. See Start/Stop Setup for more information on the Start/Stop Menu.

8.1.3. Compare the results with the acceptable ranges as listed in the original accompanying Certificate of Calibration sheet, the results should fall within these specified values. Depending on the element calibrations provided with your Thermo Scientific Niton XRF analyzer, some of the reference sample elements may not be displayed. If your instrument is not displaying all elements, check the Element Display option to determine which elements are being displayed. To force elements not displayed to display, from the Main Menu, select the Advanced icon. Select the Element Sorting icon, then select Soil Mode from the menu. Select the element you wish to modify, select Always Display from the drop down list, then select the Save Button. Repeat this process for each element you want to change, then select the Close Button, select the Close Button again, then select the Return icon.

8.1.4 If the analyzer reports values within the acceptable ranges of the reference values, it is ready for measurement of unknown samples. Proceed to GENERAL TESTING PROTOCOL section.

8.1.5 If the analyzer reports values outside the acceptance tolerance ranges specified in the accompanying Certificate of Calibration sheet, repeat the System Check described in step 5, then repeat the reference sample analysis in step 7.1

8.1.6 If the analyzer again fails to meet the acceptable tolerance ranges specified in the accompanying Certificate of Calibration sheet, please contact Thermo Fisher Scientific or your local Thermo Fisher representative for assistance.

GENERAL TESTING PROTOCOL

Soil analysis may be performed as either In-Situ Analysis (i.e., in place) or Ex-Situ Analysis and (typically) prepared. The results obtained from the XRF instrument may be different on the same sample when comparing an unprepared with a fully prepared sample. This is generally caused by sample inhomogeneity, particle size effects and moisture content. The more time and effort you spend on preparing the sample properly will allow you to obtain close-to laboratory grade data, whereas in-situ analysis may only provide "screening" type data. The decision to prepare or leave a sample unprepared may be determined by your Data Quality Objectives (DQOs).

Soil mode is a Compton normalized calibration which works over a fairly limited element concentration range. It is highly recommended that samples containing high concentrations of elements (such as ore samples) be analyzed using the Mining mode (see Mining Analysis).

In-Situ Analysis

This is the most basic type of measurement as there is little to no sample preparation performed. It is typically used for providing screening data to help rapidly define an area of contamination for example.

1. It is highly recommended that you read and follow the USEPA Method 6200 for soil analysis.

2. After performing a QC check (see above), place the In-Situ Test Guard over the front nose of the instrument. This test guard has 2 main purposes, firstly it helps to keep the front of the instrument clean by preventing soil particles from collecting on the window. Secondly it provides a solid base for the Proximity Button to engage properly, which can be difficult on very loose or powdery samples.

3. Set the appropriate range and analysis times. The analysis times should have been previously determined See Adjusting the Element Range to set and save the filter settings.

4. If desired, set beeps to activate at the end of the predetermined measurement time. See Setting up Audio Cues for instructions to set beep times.

5. Remove any large or non-representative debris from the soil surface before analysis. This debris includes rocks, pebbles, twigs, leaves, concrete, etc. If the surface is unlevel, it may be smoothed with a stainless steel trowel or other similar implement. If the soil is saturated with water, allow it to dry before starting the analysis.

6. Grasp the analyzer by the handle, ensuring the wrist strap is properly placed on the wrist.

7. Enter the sample name, GPS co-ordinates or any other pertinent information regarding the sample to maintain appropriate chain-of-custody. See Entering Soil Sample ID for details.

8. Position the analyzer on the desired analysis spot, ensuring that the nose of the analyzer is making contact with the soil, and then initiate a reading.

Continue the reading for the predetermined measurement time, and stop the reading when measurement is complete.

9. Confirm that the readings are appropriate and then proceed to next sampling area. Repeat steps 6 and 7.

10. Per USEPA Method 6200, a minimum of 1 sample for every 20 analyses (i.e. 5%) should be taken and submitted for laboratory analysis. Make sure that as best you can the sample taken is the sample that you read, samples can vary immensely over very short distances (a few cm).

11. After completing all the required analysis, rerun the QC standard to confirm stability of the instrument.

12. Download all results (unknown sample readings, QC samples, and system check) and save as an appropriately named file. The ndt file created during the download is encrypted and provides data integrity.

Ex-Situ Analysis

This is a more labor-intensive type of analysis as the sample undergoes full preparation. It is typically used to quickly provide on-site personnel with laboratory grade data without the wait for laboratory analysis, allowing more immediate and accurate decision making to occur.

1. It is highly recommended that you read and follow the USEPA Method 6200 for soil analysis.

2. Samples are procured in the field and generally taken to a fixed location such as an office or even a vehicle for preparation and analysis. Sample collection is very important as samples collected incorrectly may bias the results. As for the in-situ analysis, non-representative debris should be removed (such as twigs, leaves, rocks etc.) before sample collection begins. The sample is collected from a 4" * 4" square that is 1" deep (10cm * 10cm * 2.5cm deep). This provides enough material to fill an 8oz or 250cm3 specimen jar. Label the jar immediately to maintain chain-of-custody requirements.

3. Collect all samples in a similar fashion, and return samples to sample preparation area.

4. Sample preparation must then be performed, samples are dried and homogenized, and then ground and sieved.

5. Samples may be dried rapidly in either a convection or a toaster oven (if the work site has electricity), or spread out on a tray and air dried (this takes longer) if power is not available. It is generally not recommended to dry samples in a microwave oven.

6. The dried sample should then be well mixed and then split using a technique such as coning and quartering that will ensure an even particle size distribution (so you do not bias the results). This procedure should be repeated until you have approximately 1oz (25g) of representative sample. This sample is then ground with a pestle and mortar until it passes a 60-mesh (250?m) sieve.

Note - The remaining unprepared sample should be returned to the container to allow duplicate XRF or laboratory analysis if required. It is possible to fully prepare the entire sample, but this can be a lengthy process and in-field work does not always allow the analyst the time to do this.

7. The ground sample should then be cupped. The 32mm Sample Cups should be assembled on a clean surface (to prevent cross-contamination). Place the outer ring on a clean surface. Lay a piece of Mylar Film 2.5" dia. 6m thick or Polypropylene Film 2.5" dia. 4m thick over the top of the outer ring making sure it is evenly situated over the ring. Take the inner ring and press gently but firmly into the inner ring until it is completely together. Check the film to ensure there are no rips or tears. Fill the cup with the prepared sample and tamp down to ensure there are no air gaps and that the sample is densely packed. When the cup is filled to approximately ¼" (6mm) from the top, add a piece of Filter Paper 2.4cm dia. and then a piece of Polystuffing over the filter (this will prevent the sample moving in the cup if it is inverted). Snap on the cap and label to maintain identification of sample.

8. Perform instrument QC check (see above).

9. The sample cups may then be analyzed. It is best to analyze these in Test Stands so that the cup is held firmly against the instrument window and the operator is shielded from any back-scattered radiation.

10. Enter the sample name, GPS co-ordinates or any other pertinent information regarding the sample to maintain appropriate chain-of-custody. See Entering Soil Sample ID for details.

11. Confirm that the readings are appropriate and then proceed to analyzing next cup.

12. Per USEPA Method 6200, a minimum of 1 sample for every 20 analyses (i.e. 5%) should be taken and submitted for laboratory analysis. Some of the remaining split can be used for this purpose.

13. After completing all the required analysis, rerun the QC standard to confirm stability of the instrument.

14. Download all results (unknown sample readings, QC samples, and system check) and save as an appropriately named file. The ndt file created during the download is encrypted and provides data integrity.

Entering Soil Sample ID

1. From the Main Menu, select the Sample Type icon, then the Soils & Minerals icon, then the Soils icon which brings you into the Ready to Test screen.

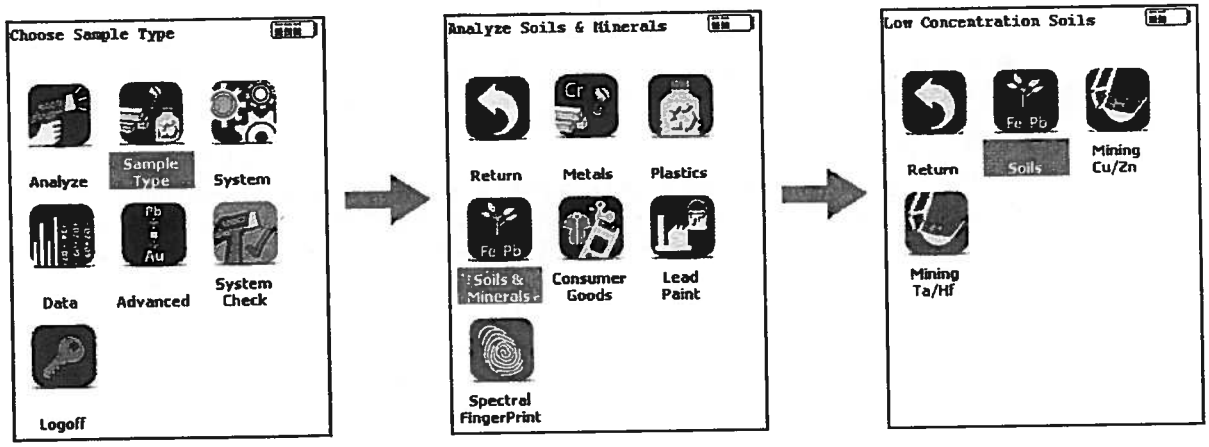


Figure 103. Soil Analysis Menu Path

2. From the Ready to Test screen, select the Data Entry button, then select the Keyboard icon next to the word Sample. This takes you to the virtual keyboard with which you can enter the sample identification. When complete, select the Return Button to take you back to the Ready to Test screen.

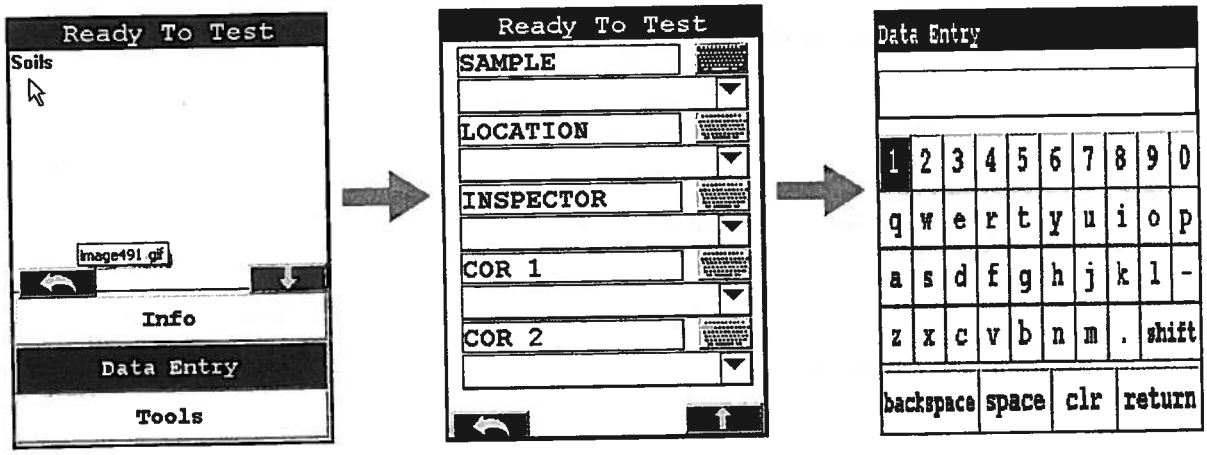


Figure 104. Soil ID Data Entry

3. Additional sample identification information may be entered into other fields if required.

APPENDIX D

Quality Assurance Project Plan

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QUALITY ASSURANCE PROJECT PLAN

JOSEPH GUY COMMUNITY CENTER
KWETHLUK, ALASKA

June 5, 2012

Prepared for:

Alaska Department of Environmental Conservation

Prepared by:

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ENVIRONMENTAL

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Quality Assurance Project Plan

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ATTACHMENT

A: EPA Method 6200

1. INTRODUCTION

The purpose of this Quality Assurance Project Plan (QAPP) for the Joseph Guy Community Center (JGCC) located in Kwethluk, Alaska is to describe data quality objectives; work to be performed toward meeting the project objectives; and methods used to obtain valid data of documented quality. The QAPP presents requirements for performing analytical procedures including specific measurement objectives for chemical analyses, sampling and calibration procedures, sample custody, data review and reporting, and internal quality control (QC) checks. The specified quality assurance (QA) procedures adhere to applicable regulatory requirements, laboratory standard operating procedures, and/or other specifications necessary for compliance with industry-accepted standards and contract requirements.

This document should be used in concert with the JGCC Brownfield Cleanup Action work plan. The QAPP and work plan together will comprise the sample and analysis plan for the site.

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2. PROJECT MANAGEMENT

This section presents the roles, responsibilities, and objectives of the participants involved in the assessment of the site in Kwethluk.

2.1. Project Organization

The following list describes the roles of the participants in this project.

- Alaska Department of Environmental Conservation (DEC) Project Manager: Serves as the overall project coordinator and primary point of contact. Responsible for facilitating communication between all parties involved with the project. Has final review and approval authority for all elements of the project.
- EPA Project Manager: Point of contact with EPA. Provides coordination between DEC and EPA.
- EPA QA Officer: Authority to review and approve the QAPP. Has authority to perform QC on any aspect of project performance to determine compliance with QA requirements.
- OASIS Environmental, Inc., an ERM Company (OASIS) Contract Manager: Serves as principal reviewer for this project and responsible for overall execution of the term contract that OASIS has with DEC's Division of Spill Prevention and Response. Responsible for appropriating necessary resources to complete the project.
- OASIS QA Officer: Reviews and approves in-house project QA objectives and QC elements. Responsible for checking or auditing all aspects of project execution and proposing corrective actions where needed.
- OASIS Project Manager: Provides coordination for all aspects of the project. Implements project plans and consults with DEC project manager on deviations. Acts as primary point of contact between DEC and OASIS. Responsible for coordinating resources necessary to complete the project.

2.2. Problem Definition/Background

OASIS was contracted by the DEC to demolish a burned building, the former JGCC, in Kwethluk, Alaska and dispose of the building materials, debris, and ash. The project also involves confirmation sampling the footprint of the building, removal of known semi-volatile organic compounds contaminated soil, and removal / management of known petroleum contaminated soil associated with an aboveground storage tank (AST) that served the building. The ultimate purpose of the project is to assist the Organized Village of Kwethluk and the City of Kwethluk in revitalizing the site by making the property ready for reuse. Figure 1 of the main work plan shows the location of the site. . The EPA has provided funding for demolition of the JGCC, removal of contaminated soil, and confirmation sampling of building footprint and the excavations associated with the contaminated soil.

The JGCC building burned in April 2006 and the possible release of contaminants posed a risk to human health through building debris and soil / groundwater contamination. The possible sources of contamination included a former glycol heating system and burned electronic waste. It is unlikely that any fluorescent light fixtures in the building contain polychlorinated biphenyls as the building was built in the late 1990's.

In 2010, Ecology and Environment (E&E) performed a TBA funded by the U. S. Environmental Protection Agency (EPA). The TBA (E&E 2011) involved collecting eight surface soil samples from the building interior for analysis of Target Analyte List (TAL) metals and SVOC. Five of the eight samples were also analyzed for dioxins and furans.

All eight of the samples contained at least one TAL metal result that exceeded DEC cleanup levels. Only six of the twenty TAL metals exceeded cleanup levels, including antimony, arsenic, chromium, cobalt, copper, and nickel. None of the samples exceeded DEC or EPA regulatory criteria for SVOC or dioxins/furans.

Eighteen exterior co-located surface/subsurface soil samples were collected and analyzed for TAL metals and SVOC. Six of the samples were also analyzed for dioxins/furans. Two surface soil samples exceeded DEC cleanup levels for SVOC; a sample located on the west side of the building had a bis(2-ethylhexyl)phthalate result of 2.7 milligrams per kilogram (mg/kg), exceeding the DEC cleanup level of 1.3 mg/kg; a sample located on the south side of the building had a n-nitroso-di-n-propylamine result of 0.042 mg/kg, exceeding the cleanup level of 0.0011 mg/kg.

Two surface soil samples were collected from the former location of an AST that contained heating oil and analyzed for diesel-range organics (DRO) and residual-range organics (RRO). One of the samples had a DRO result of 9,000 mg/kg, exceeding the DEC cleanup level for DRO of 250 mg/kg.

Eight wipe samples were collected from the interior and exterior building walls and analyzed for dioxins/furans. All of the wipe samples were positive for dioxins/furans. No regulatory criteria exist for wipe samples.

Twelve bulk samples were collected of suspected asbestos containing building materials. No asbestos was present in any of the samples.

2.3. Project Description

The description of this project presents a scope of work necessary to meet the project definition outlined in the previous subsection. This QAPP sets guidelines for assessing project performance in meeting the following project objectives:

- Develop a Brownfield Cleanup Action work plan (hereafter, "project work plan) according to the DEC guidance on preparation of work plans and reporting (DEC 2009).
- Demolish the JGCC and transport all non-hazardous metal building materials to Bethel, Alaska for recycling or disposal.

- Collect confirmation samples from the building footprint for analysis of six TAL metals.
- Prepare the Kwethluk landfill to accept four waste streams; the top 2 to 3 inches of ash/soil/debris from the building footprint, known diesel contaminated soil associated with the former AST and two separate volumes of soil from known SVOC-contaminated soil adjacent to the building.
- Scrape the top few inches of ash/soil/debris from the former JGCC footprint and transport it to the Kwethluk landfill.
- Excavate diesel-contaminated soil associated with a former heating oil AST and store at the Kwethluk landfill in bulk polyethylene sacks.
- Excavate surface soil in two locations of known SVOC contamination and store soil from each location in separate bulk sacks at the Kwethluk landfill.
- Collect confirmation samples of the excavations and diesel and SVOC contaminated soil bulk sacks.

The schedule for this project is based on completion dates for the following tasks:

Task	Start Date	Completion Date
Develop and submit draft project work plan for demolition phase.	March 5, 2012	March 30, 2012
Complete and submit final demolition plan	March 30, 2012	March 30, 2012
Complete demolition of JGCC	April 2, 2012	April 10, 2012
Conduct project work plan for entire project (including sampling and excavation activities)	May 1, 2012	May 24, 2012
Develop and submit QAPP to DEC/EPA	May 31, 2012	June 4, 2012
Receive comments from DEC/EPA on QAPP	June 1, 2012	June 6, 2012
Complete and submit final project work plan	May 24, 2012	June 6, 2012
Complete and submit final QAPP	June 6, 2012	June 6, 2012

2.4. Quality Objectives and Criteria

The data quality objectives (DQOs) of this project are to provide data of documented quality to characterize the nature and extent of recognized environmental conditions at the site based on DEC soil and groundwater cleanup levels as presented in Alaska Administrative Code Title 18, Chapter 75 (18 AAC 75). The DEC does not regulate cobalt and the DQOs for cobalt are based on the EPA Regional Protection of Groundwater Soil Screening Level. All samples collected during this project will be analyzed by either published EPA or DEC methods. Analytical data will be compared to defined laboratory DQOs. The laboratory, or analytical, DQOs are the performance criteria of precision, accuracy, representativeness, comparability, and completeness of the tests. The following subsections detail these performance criteria.

2.4.1. Precision

Precision measures the reproducibility of repetitive measurements and gives information about the consistency of methods. Analytical precision is the measurement of the variability associated with duplicate analyses. The laboratory control sample (LCS) determines the precision of laboratory operations. If the recoveries of the analytes in the LCS are within established control limits, then precision is within limits. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch. Rather, the comparison is between a QC sample and its duplicate QC sample or a QC sample analyzed in a previous batch.

Total precision is the measurement of the variability associated with the entire sampling and analysis process. The analysis of duplicate field samples measures variability introduced by field operations, and the analysis of matrix spike duplicate samples measures variability introduced by the sample matrix and by laboratory operations. This project will use both types of duplicate samples to assess field and analytical precision. The precision measurement is determined using the relative percent difference (RPD) between the original and duplicate sample results.

The following formula is used to calculate precision:

$$RPD = \frac{(100) \times (S1 \quad S2)}{(S1 + S2)/2}$$

where:

S1 = primary sample value

S2 = duplicate sample value

Table A1 presents the precision goals that have been established for this project. Precision goals will be met if duplicate analyses of LCS agree within RPDs specified in Table A1. RPDs for LCS that are outside specified criteria indicate the analytical system is out of control and will require corrective action, which likely means re-analysis.

2.4.2. Accuracy

Accuracy is a measurement of correctness and includes components of random error and systemic error. A measurement is accurate when the value reported does not differ from the true value. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into a field sample (matrix spike [MS] sample) or a blank matrix (LCS) prior to analysis. Surrogate compound recovery is another spiking technique used to assess method accuracy for each sample analyzed for volatile and semi-volatile compounds.

The following formula is used to calculate accuracy:

$$\% \text{ Recovery} = \frac{\text{Reported Analyte Concentration} \times 100}{\text{Actual Analyte Concentration}}$$

Table 1 presents accuracy goals. These goals are considered be met if individual LCS recoveries are within listed criteria. LCS recoveries outside criteria indicate the analytical system is out of control and requires corrective action, which likely means re-analysis.

2.4.3. Representativeness

Representativeness is a measure of the degree to which data accurately and precisely represent a population (i.e., sample matrix or media). Sampling procedures are designed to ensure that the data will be representative of the media sampled. This includes the selection of sample location, the manner of sample collection and handling techniques to avoid contamination or compromise of sample integrity, and proper chain of custody of samples. Additionally, the sampling design should include a sufficient number of samples and level of confidence that analysis of these samples will detect the contaminants of concern, if present.

Objectives for representativeness will be defined for each sampling and analysis task and will be a function of the investigative objectives. Representativeness will be achieved in part through use of the standard sampling and analytical procedures described in this QAPP and in the project work plan.

2.4.4. Comparability

Comparability is the measure of confidence that two data sets contribute to a common evaluation. Comparability with respect to laboratory analyses pertains to method type comparison, holding times, and quantitation limits. The following items are evaluated when assessing data comparability:

- Determining if two data sets or batches contain the same set of parameters;
- Determining if the units used for each data set are convertible to a common scale;
- Determining if similar analytical procedures and quality assurance were used to collect data for both data sets;
- Determining if the analytical instruments used for both data sets have approximately similar detection levels; and
- Determining if samples within data sets were selected and collected in a similar manner.

The comparability objective for this project is to produce data with the greatest degree of comparability possible.

2.4.5. Completeness

Completeness is calculated for the aggregation of each analyte measured. The number of valid results divided by the number of possible individual analyte results, expressed as

a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not rejected through data validation. The requirement for completeness is 90%.

The following formula is used to calculate completeness:

$$\% \text{ completeness} = \frac{\text{number of valid results} \times 100}{\text{number of possible results}}$$

For any instances of samples that could not be analyzed, the numerator of this calculation becomes the number of valid results minus the number of possible results not reported.

2.5. Special Training

Any field personnel will be required to have 40-hour hazardous waste and emergency response (HAZWOPER) training. In addition, sampling personnel are required to meet the requirements of a "qualified person" per the Alaska Administrative Code Title 18, Chapter 75, Article 9, Section 990 (18 AAC 75.990(100)).

2.6. Documentation and Records

Pertinent documentation for this project will be included with the project report, when it is produced. The documentation will include analytical data packages, chain-of-custody documentation, copies of logbooks, documentation of corrective actions, and photographs. In addition, additional documentation may be provided to DEC, if requested. These items include electronic data deliverables, correspondence, original chain-of-custody documentation, original logbooks, original corrective action forms, and referenced materials.

3. DATA GENERATION AND AQUISITION

This section describes the process for data generation and acquisition to ensure that appropriate methods for sampling, measurement and analysis, data collection and generation, data handling, and QC activities are employed and documented.

3.1. Sampling Process Design

Samples will be collected from areas at the site that may have been impacted by contaminants mentioned below. Selection of analytical sample locations will be biased so as to increase the probability that contamination, if present, is detected. Sample media will include only soil. Any significant deviation from the planned sampling locations or number of samples to be collected will be discussed with the DEC project manager as needed.

Samples will be analyzed by field screening techniques and/or a fixed-base laboratory. The use of field screening techniques will be maximized in order to provide more real-time data and generate a larger data set at reduced expenditure. Fixed-base laboratory analysis will be used for confirmation analysis.

The contaminants of concern at the site include DRO, n-nitroso-di-n-propylamine, bis(2-ethylhexyl)phthalate, antimony, arsenic, chromium, cobalt, copper, and nickel. Table A1 presents QA/QC summaries for the methods.

3.2. Sampling Methods

Soil samples will be collected based on the process described in Section III of the *Draft Field Sampling Guidance* (DEC 2010). Additionally, samples will be collected and preserved based on the requirements of the analytical method used to analyze the samples. Table A2 provides sample method standards for each proposed analysis.

Every effort will be made to use dedicated sampling equipment to minimize the need for equipment decontamination. If decontamination is necessary, the process will follow the recommended practices in Section VIII (E) of the *Draft Field Sampling Guidance* (DEC 2010). In general, the process will entail the following steps:

- Scrub to remove all visible material;
- Scrub with brushes using Alconox solution;
- Rinse with potable water;
- Double-rinse with organic-free deionized water; and
- Air dry.

The sampling process may generate investigation-derived waste (IDW) from used sampling equipment, used personal protective equipment, and used field screening analytical equipment. Visual contamination will be removed from solid IDW, which then will be bagged and disposed of at as solid waste at the Kwethluk landfill. If generated, aqueous IDW will be poured over the bulk sack associated with the previous sample.

3.3. Sample Handling and Custody

Samples will be packaged carefully to avoid breakage or cross-contamination and will be shipped to the analytical laboratory at required temperatures for specific analytical methods. The following sample packaging requirements will be followed:

- Enclose each sample container individually either in a sealed, clear, plastic bag for shipping;
- Surround sample containers with bubble wrap to prevent breakage from impact;
- Place the samples into the coolers and surround the samples with gel ice;
- Fill any remaining space in the coolers with inert packing material; and
- Tape chain-of-custody documents in a sealed plastic bag under the cooler lid, seal with custody seals, affix a label containing the laboratory name and address, and ship.

Shipping containers will be labeled clearly and custody-sealed for shipment. When custody is relinquished to a shipper, personnel will telephone the analytical laboratory to inform them of the expected arrival of the sample shipment and to advise them of any time constraints on sample analysis.

Samples will be tracked by the use of chain of custody laboratory forms. Each sample will be individually identified on a chain of custody form. These forms will include sample identification number, sample date, sample time, requested analysis, type and number of sample containers, sample preservatives, quality control information, and requested analytical turnaround time. Each form will be signed and dated upon relinquishment to another party, whether shipper, courier, or laboratory, to maintain the custody the samples.

Samples will be identified using a 10 digit alpha-numeric sample number. The sample number will be located on a label that is affixed to the sample container using clear tape. In addition to the sample number, the labels will contain the following information: sample date, sample time, requested analysis, and sample preservation. Table A3 summarizes the 10 digit alpha-numeric sample number scheme.

3.4. Analytical Methods

A combination of field screening and fixed-base laboratory analyses will be used to generate data for this project. Field screening techniques that will be employed are headspace analysis for VOCs by photo-ionization detector (PID) and x-ray fluorescence (XRF) for metals. PID headspace analysis will follow the method outlined in Section III(B) of the *Draft Field Sampling Guidance* (DEC 2010). XRF analysis will follow EPA Method 6200 for field spectrometry, which is attached as Appendix A1 of this QAPP.

Fixed-base laboratory analysis will be performed at a DEC-certified laboratory and will be used to confirm field screening results. The following analytical methods will be used:

- Alaska Method AK-101 for analysis of GRO;
- Alaska Method AK-102 for analysis of DRO;

- EPA Method 8021B for analysis of BTEX;
- EPA Method 8270D for analysis of SVOCs; and
- EPA Method 6020 for analysis of metals;

Table A1 presents QA/QC summaries for proposed analytical methods. Brief narrative descriptions of the EPA methods are provided in the list below:

- AK-101 - This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as gasoline. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. A flame ionization detector (FID), or PID/FID in series, provides detection. Quantitation must be performed by comparing the total chromatographic area between and including C6 (n-hexane) and C9 (n-nonane), to the peak start time of C10 (n-decane), including resolved and unresolved components, based on FID response compared to a blended commercial gasoline standard using forced baseline-baseline integration.
- AK-102 - This method provides gas chromatographic conditions for the detection of semi-volatile petroleum products such as diesels. Samples must be spiked with a surrogate compound and extracted with methylene chloride. The extract is dried and concentrated. An aliquot of the extract must be injected into a capillary column gas chromatograph equipped with a FID, which has been temperature programmed to facilitate separation of organic compounds. Quantitation must be performed by comparing the total chromatographic area between and including the peak start of C10 to the peak start of C25, including both resolved and unresolved components, based on FID response compared to a diesel calibration standard. Integration must be performed using forced baseline-baseline integration.
- EPA 8021B – This method uses a purge-and-trap gas chromatograph and photoionization detector technique. An inert gas is bubbled through a methanol extract/water dilution from soil samples, to transfer the purgeable organic compounds from the liquid to vapor phase. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a gas chromatographic column where they are separated and then detected with a PID.
- EPA 8270D – SVOC extractables in soil samples are analyzed using GC/MS. Organic compounds are extracted from the sample with methylene chloride. The extract is then concentrated by removal of the methylene chloride through evaporation. Compounds of interest are separated and quantified using a GC/MS.
- EPA 6020 – Samples are digested using EPA method SW3005A, SW3020A or SW3050B. Following digestion, trace elements are determined using an inductively couple thermal plasma with a GC/MS.

3.5. Quality Control

QC checks for sample collection will be accomplished by chain-of-custody protocols and field screening and laboratory QA procedures as prescribed in the sampling or analytical methods. QC samples will include blanks, calibration verifications, spikes, duplicates, interference check samples, and serial dilutions. Precision and accuracy requirements are outlined in Table A1.

One temperature blank will be included in each cooler shipped to fixed-base laboratories. Temperature blanks allow a laboratory to obtain a representative measurement of the temperature of samples enclosed in a cooler without disturbing the actual samples.

3.6. Instrument Calibration, Inspection, and Maintenance

The field equipment used during this project may include a GPS unit, an XRF analyzer, and a PID. Testing, inspection, and maintenance of these instruments will be performed in accordance with the manufacturers' recommendations. All field instruments and equipment used for analysis will be serviced and maintained only by qualified personnel.

Equipment calibrations will be documented in an appropriate logbook that will be kept on file. When in use, equipment will be inspected at least twice daily, once before start-up in the morning and again at the end of the work shift before overnight storage. Field sampling task leaders will be responsible for maintaining an adequate inventory of spare parts. In addition to spare parts and supply inventories, a backup supply of much of the equipment and instrumentation for the field sampling will be maintained.

The calibration, inspection, and maintenance of laboratory equipment are the responsibility of subcontract laboratory. The subcontract laboratory's QA procedures, along with manufacturer's recommended guidelines, will dictate these practices.

3.7. Inspection/Acceptance of Supplies and Consumables

Manufacturer QA/QC certification statements will be saved on file for any consumable used during this project. Examples of these include pre-cleaning of sample containers and purity of reagents and calibration standards.

3.8. Non-Direct Measurements

Pre-existing data from the Former Joseph Guy Community Center ARRA Funded Targeted Brownfield Assessment (E&E 2011) conducted at the site has been used to develop site-specific sampling strategy for the project work plan. These data have been called out in the work plan to demonstrate the rationale for selecting the particular sample strategy. All outside materials used to develop a sample strategy have been referenced.

3.9. Data Management

Data generated during this project will include both field screening and fixed-base laboratory analyses. Data will be received from fixed-base analytical laboratories in both

electronic and hard copy formats. Field screening data will be contained in the project logbook. All data will go through validation as described in Section 4. Hard copies of data will be stored on file in the OASIS office. Electronic data will be manipulated for tabular presentation in a report

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4. ASSESSMENT AND OVERSIGHT

This section presents the process for assessing the effectiveness of project implementation and the actions that may be taken when deficiencies are discovered.

4.1. Assessment and Response Actions

The DEC project manager or QA officer may conduct an audit of the field activities for this project. The auditor will have the authority to issue a stop work order upon finding a significant condition that adversely would affect the quality and usability of the data. The DEC project manager will have the responsibility for initiating and implementing response actions to correct the situation. Once the response actions have been implemented, the DEC project manager or designee may perform a follow-up audit to verify and document that the response actions were implemented effectively. OASIS also may perform in-house audits, although no audit is planned for this project.

4.2. Reports to Management

Debriefing of the DEC project manager by the OASIS project manager will occur on a regular schedule decided by these two individuals. The project work plan will provide strategy and methods, and upon approval by DEC will be distributed to involved parties in this project. Similarly, following the receipt of all analytical data, a project report will be prepared, which will include a QA/QC analysis of the project by an OASIS chemist, and upon approval by DEC will be distributed to involved parties in this project.

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5. DATA VALIDATION AND USABILITY

This section presents the process for validation of data to determine whether data conform to the criteria presented in this plan.

5.1. Data Review, Verification, and Validation

The evaluation/assessment of measurement data is required to ensure that the QA objectives for the program are met and that quantitative measures of data quality are provided. The laboratory data review and validation process will conform to EPA Level II data validation protocols. Information provided by the analytical laboratory and field sampling crew will be reviewed and evaluated. An OASIS chemist will perform the data validation to verify conformance to this QAPP and to the analytical methods specified herein. This will include the following:

- Proper sample collection and handling procedures
- Holding times
- Instrument calibration verification
- Internal standards performance criteria
- Laboratory blank analysis
- Reporting limits
- Laboratory replicates
- Matrix spike percent recovery results
- Surrogate percent recovery
- Data completeness and format
- Data qualification

All of the data validations will be performed in accordance with the QA/QC requirements specified in this QAPP, the technical specifications of the analytical methods, and the following documents:

- *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (EPA 2008); and
- *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (EPA 2010).

Field screening data will be examined for adherence to the applicable SOPs and will not be validated.

5.2. Verification and Validation Reporting

Validation deliverables will include a DEC Laboratory Data Review Checklist that will be completed to aid in data validation. In addition, a QA memo will be written, discussing QA conformance and deviations issues that may affect the quality of the data. Data

usability, bases of application of qualifiers, and percentage of qualified data will also be discussed in the QA memo. Report table will include validation qualifiers.

Following data validation and reporting, all project-generated and -compiled data and information will be reconciled with the objectives specified in Subsection 2.3 to assess the overall success of the project. This data assessment, including points of achievement and departure from project-specific objectives, will be discussed in the QA section of the project report.

5.3. Reconciliation with User Requirements

Once the data results are compiled, the OASIS QA officer, OASIS project manager, and DEC project manager or designee will review the sample results to determine if they fall within the acceptance limits as defined in this QAPP. If performance criteria do not meet the project's requirements, the data may be discarded and corrective action will need to be determined by the DEC project manager. If the failure is tied to the analysis, calibration and maintenance techniques will be reassessed as identified by the appropriate laboratory personnel. If the failure is associated with the sample collection, the collection techniques will be re-evaluated as identified by OASIS.

The project's QA officer or project manager will initiate a corrective action in the event that QC results exceed acceptability limits, or upon identification of some other problem or potential problem. These individuals also will initiate corrective action based on QC data or audit results. Typical corrective action procedures depend on the type and severity of the problem and range from use of data qualifier flags to recommending changes to procedures.

6. LEGAL REQUIREMENTS

This section presents the process for assuring that the project complies with the Endangered Species Act and the National Historic Preservation Act.

6.1. Endangered Species Act (ESA)

The DEC has provided OASIS with a letter of concurrence from the U.S. Fish and Wildlife Service stating that the project is not likely to affect any endangered or threatened species.

6.2. National Historic Preservation Act (NHPA)

The DEC has provided a concurrence letter from the Alaska State Historic Preservation Office stating that "No Historic Properties Affected" by the proposed activities for the project area.

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7. REFERENCES

Alaska Department of Conservation (DEC), 2008. 18 AAC 75, Oil and Other Hazardous Substances Pollution Control. October 9.

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TABLES

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Table A1
Quality Assurance/Quality Control Analytical Summary
Joseph Guy Community Center
Kwethluk, Alaska

Sample Matrix	Parameter / Analytical Method	Lab Required Limit of Quantitation (mg/kg)			Precision	Accuracy (% Recovery)
		Compound / Metal	Quantitation Limit	Regulatory Criteria		
Soil	GRO / AK 101	GRO	3.3	300	≤ 20 RPD	60 – 120
		4-Bromofluorobenzene	Surrogates			50 - 150
		a,a,a-Trifluorotoluene				50-150
	DRO / AK 102	N/A	25	250	≤ 50 RPD	75 – 125
		1-chlorooctadecane	Surrogates			50 - 150
	BTEX / EPA 8021B	Benzene	0.0200	0.025	≤ 25 RPD	60 - 140
		Toluene	0.0400	6.5		
		Ethylbenzene	0.0400	6.9		
		Xylenes	0.120	63		
	SVOCs / EPA 8270D	Bis(2-ethylhexyl) phthalate	0.600	13	≤ 60 RPD	64 - 144
		N-Nitrosodi-n-propylamine	0.100	0.0011	≤ 28 RPD	52 - 127
		1-Methylnaphthalene	0.030	6.2	≤ 30 RPD	48 - 148
		2-Methylnaphthalene	0.020	6.1	≤ 27 RPD	65 - 125
		Acenaphthene	0.020	180	≤ 27 RPD	65 - 130
		Acenaphthylene	0.020	180	≤ 28 RPD	69 - 129
		Anthracene	0.020	3,000	≤ 27 RPD	73 - 123
		Benzo[a]anthracene	0.020	3.6	≤ 27 RPD	64 - 124
		Benzo[a]pyrene	0.030	2.1	≤ 30 RPD	68 - 128
		Benzo[b]fluoranthene	0.020	12	≤ 31 RPD	66 - 136
		Benzo[g,h,i]perylene	0.025	38,700	≤ 28 RPD	57 - 142
		Benzo[k]fluoranthene	0.025	120	≤ 31 RPD	63 - 143
		Chrysene	0.025	360	≤ 26 RPD	71 - 126
		Dibenzo(a,h)anthracene	0.040	4.0	≤ 30 RPD	57 - 142
		Fluoranthene	0.020	1,400	≤ 36 RPD	61 - 121
		Fluorene	0.020	220	≤ 31 RPD	68 - 128
		Indeno[1,2,3-cd]pyrene	0.040	41	≤ 29 RPD	59 - 139
		Naphthalene	0.020	20	≤ 26 RPD	64 - 129
		Phenanthrene	0.020	3,000	≤ 28 RPD	65 - 125
		Pyrene	0.020	1,000	≤ 31 RPD	54 - 134

Table A1
Quality Assurance/Quality Control Analytical Summary
Joseph Guy Community Center
Kwethluk, Alaska

Sample Matrix	Parameter / Analytical Method	Lab Required Limit of Quantitation (mg/kg)			Precision	Accuracy (% Recovery)
		Compound / Metal	Quantitation Limit	Regulatory Criteria		
Soil	SVOCs / EPA 8270D	2,4,6-Tribromophenol	Surrogates			28 - 143
		2-Fluorobiphenyl				42 - 140
		2-Fluorophenol				36 - 145
		Nitrobenzene-d5				38 - 141
		Phenol-d5				38 - 149
		Terphenyl-d14				42 - 151
	Metals / EPA 6020	Arsenic	0.5	3.9	≤ 20 RPD	80 - 120
		Arsenic	0.2	3.6		
		Antimony	0.2	25		
		Cobalt	0.2	0.21		
		Copper	0.4	460		
		Nickel	0.5	86		
	Metals / EPA 6200	Antimony	XRF detection limits based on operational run time and source material	3.6	XRF precision based on operational run time and source material	XRF accuracy based on operational run time and source material
		Arsenic		3.9		
		Chromium (total)		25		
		Cobalt		0.21		
		Copper		460		
		Nickel		86		

Notes:
AK = Alaska Method
BTEX = benzene, toluene, ethylbenzene, and xylenes
DRO = diesel-range organics
EPA = U. S. Environmental Protection Agency
GRO = gasoline-range organics
MeOH = methanol
RPD = Relative percent difference
SVOCs = semivolatile organic compounds
XRF = X-ray fluorescence

Table A2
Sample Analysis Summary
Joseph Guy Community Center
Kwethluk, Alaska

Sample Matrix	Parameter / Analytical Method	Sample Preservation	Holding Time	Sample Container(s)
Soil	GRO / AK-101	MeOH; < 4° C	28 days	1 x 4-oz pre-weighted amber
	DRO / AK-102	< 4° C	14 days	1 x 4-oz amber glass
	BTEX / EPA 8021B	MeOH; < 4° C	14 days	1 x 4-oz pre-weighted amber
	SVOCs / EPA 8270D	< 4° C	14 days	1 x 4-oz amber glass
	Metals / EPA 6020	< 4° C	180 days	1 x 4-oz glass

Notes:
AK = Alaska method
BTEX = benzene, toluene, ethylbenzene, and xylenes
C = Celsius
DRO = diesel-range organics
EPA = U. S. Environmental Protection Agency
GRO = gasoline-range organics
MeOH = methanol
oz = ounce
SVOCs = semivolatile organic compounds

Table 3
Sample Identification
Joseph Guy Community Center
Kwethluk, Alaska

Digits	Description	Code Examples
1-2	Year	12
3-6	Location Code	JGCC
7-9	Sequential Sample Number	101
10-11	Sample Type:	Symbol:
	Soil Sample	SO
	Trip Blank	TB

Example: 12-JGCC-101-SO (2012 Joseph Guy Community Center, Sample No. 01, Soil Sample).

ATTACHMENT A

EPA Method 6200

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FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples. The three electron shells include the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α), beta (β), or gamma (γ) etc., which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than

the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments, specifically, in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

3.6 SRM -- Standard reference material; a standard containing certified amounts of metals in soil or sediment.

3.7 eV -- Electron volt; a unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup (i.e., against the cup window), the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95 and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As K_β line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as Method 3050, or a total digestion procedure, such as Method 3052, is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project-specific data quality objectives (DQOs).

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the DQOs of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Sec. 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10° F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) and leak tests should be present with the instrument at all times and available to local and national authorities upon request.

X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs or badges should be worn in the area of maximum exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 FPXRF spectrometer -- An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation sources -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron Fe-55 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of

accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample presentation device -- FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors -- The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 L. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the

units or from PCs. Once the data–storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film -- Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 µm thick.

6.5 Mortar and pestle -- Glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers -- Glass or plastic to store samples.

6.7 Sieves -- 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels -- For smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags -- Used for collection and homogenization of soil samples.

6.10 Drying oven -- Standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Pure element standards -- Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if designated for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.3 Site-specific calibration standards -- Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 °C. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 g of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.4 Blank samples -- The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Follow the manufacturer's instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting,

which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.2 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank samples -- Two types of blank samples should be analyzed for FPXRF analysis, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetraflurorethylene (PTFE) block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the established lower limit of detection should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration verification checks -- A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision measurements -- The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

The equation for calculating RSD is as follows:

$$RSD = (SD/Mean\ Concentration) \times 100$$

where:

RSD	=	Relative standard deviation for the precision measurement for the analyte
SD	=	Standard deviation of the concentration for the analyte
Mean concentration	=	Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

9.7 Confirmatory samples -- The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on project-specific data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument calibration -- Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments, namely: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental parameters calibration -- FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are necessary, although site-specific samples with confirmed and validated analytical results for all elements present could be used.
- Cost is reduced because fewer confirmatory laboratory results or calibration standards are necessary.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Sec. 7.3. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective energy FP calibration -- The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference
C_k = Certified concentration of standard sample
C_s = Measured concentration of standard sample

10.2.2 BFP calibration -- BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended

count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical calibration -- An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Sec. 7.3; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is necessary. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are necessary to perform an adequate empirical calibration. The exact number of standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton normalization method -- The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline reading. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later during analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide example performance data for this method, this modest amount of sample preparation was found to take less than 5 min per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for in situ analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only "screening" type data.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 g or 250 cm^3, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Sec. 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 °C. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 min per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from "nondetect" to tens of thousands of mg/kg. These data are provided for guidance purposes only.

13.3 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode

detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.4 All example data presented in Tables 4 through 8 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from "nondetects" to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the in situ homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square,

measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy measurements -- Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 6. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability -- Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4–intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 8 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 8 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Sec. 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer," Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, February 24-26, 1993, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex, X-MET 920 User's Manual.

2. Spectrace Instruments, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.

3. TN Spectrace, Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, received from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: Refs. 1, 2, and 3
These data are provided for guidance purposes only.

TABLE 2

SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Refs. 1, 2, and 3

TABLE 3

SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Ref. 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4

EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84[a]	NR	24.80[a]	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85[a]	NR	24.92[a]	20.92[a]	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69[a]	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32[a]	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These data are provided for guidance purposes only.
Source: Ref. 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

These data are provided for guidance purposes only.
Source: Ref. 4
[a] These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.
ND Not detected.
NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Ref. 4. These data are provided for guidance purposes only.
n: Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD: Standard deviation; NA: Not applicable; only two data points, therefore, a SD was not calculated.
%Rec.: Percent recovery.
-- No data.

TABLE 7

EXAMPLE ACCURACY FOR TN 9000[a]

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Ref. 4. These data are provided for guidance purposes only.

[a] All concentrations in milligrams per kilogram.

%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.

-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY[1]

	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

Source: Ref. 4. These data are provided for guidance purposes only.

[1] Log-transformed data

n: Number of data points; r^2: Coefficient of determination; Int.: Y-intercept

— No applicable data

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT