Waste Management Division
PO Box 95, 29 Hazen Drive
Concord, NH 03302

Type of Submittal (Check One-Most Applicable)

☒ Work Scope
☐ Reimbursement Request

☐ UST Facility Report
☐ AST Facility Report

☐ Emergency/Initial Response Action
☐ Groundwater Quality Assessment

☐ Initial Site Characterization
☐ Site Investigation
- Site Investigation Report
- Supplemental Site Investigation Report
- GMZ Delineation
- Source Area Investigation
- Data Submittal
- Annual Summary Report

☐ Unsolicited Phase I Environmental Site Assessment
☐ Closure Documentation

☐ Remedial Action
- Remedial Action Plan
- Bid Plans and Specifications
- Remedial Action Implementation Report

☐ Treatment System and POE O&M
☐ Activity and Use Restriction

☐ Temporary Surface Water Discharge Permit

☐ Groundwater Management Permit
- Permit Application
- Renewal Application
- Deed Recordation Documentation
- Abutter Notification Documentation
- Release of Recordation

☐ Data Submittal
☐ Annual Summary Report

SITE SPECIFIC QUALITY ASSURANCE PROJECT PLAN

Ferrari Realty Trust Property
93-119 Memorial Street
Franklin, New Hampshire
NHDES Site #198606087

Prepared For:

Franklin Falls Downtown Development Authority
361 Central Street
Franklin, New Hampshire 03235
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JUDD R. NEWCOMB
No. 00799

Prepared By:
CREDERE ASSOCIATES, LLC
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Phone: (207) 828-1272 ext. 16
Contact: Judd Newcomb, CG, PG

October 3, 2014

Recommended Risk Category (check one)

☐ 1. Immediate Human Health Risk (Impacted water supply well, etc.)

☐ 2. Potential Human Health Risk (Water supply well within 1,000' or Site within SWPA)

☐ 3. Free Product or Source Hazard

☐ 4. Surface Water Impact

☐ 5. No Alternate Water Available/No Existing Wells in Area

☐ 6. Alternate Water Available/High Level Groundwater Contamination (>1,000 X AGQS)

☐ 7. Alternate Water Available/Low Level Groundwater Contamination (<1,000 X AGQS)

☐ 8. No AGQS Violation/No Source Remaining

☐ Closure Recommended

1. TITLE AND APPROVAL PAGE

SITE-SPECIFIC QUALITY ASSURANCE PROJECT PLAN (SSQAPP) ADDENDUM TO NEW HAMPSHIRE GENERIC QAPP RFA #14123

PROPERTY:
Ferrari Realty Trust Property
NHDES #198606087
93-119 Memorial Street, Franklin, New Hampshire
EPA Brownfields Assessment Grant # BF-96176301

PREPARED BY:
Credere Associates, LLC
776 Main Street, Westbrook, Maine 04092
(207) 828-1272

October 3, 2014

Below is a listing of the names, titles, signatures, and signature dates of officials approving this Site Specific Quality Assurance Project Plan (SSQAPP) Addendum:

Alan Peterson 10/14/2014
Alan Peterson
EPA Brownfields Project Officer
Date

Steph DiMattei 10-14-14
Stephen DiMattei
EPA Quality Assurance Officer
Date

10-14-14
Jeff Hayes
Lakes Region Planning Commission, Brownfields Assessment Grantee
Date

10/14/2014
Rebecca Williams
NHDES Project Manager
Date

Judd R. Newcomb 10/14/2014
Judd Newcomb, PG, CG
Credere Associates, LLC QA/QC Manager
Date

Rip Patten 10/14/2014
Rip Patten, PE, LSP, LEED-AP
Credere Associates, LLC Program Manager
Date

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APPENDICES

2. INTRODUCTION

Credere Associates, LLC (Credere) was retained by Lakes Region Planning Commission (LRPC) to prepare this Site-Specific Quality Assurance Project Plan (SSQAPP). LRPC is using funding from a U.S. Environmental Protection Agency (EPA) Brownfields Assessment Grant (Grant number: BF-96176301) to conduct assessment activities at the Ferrari Realty Trust Property located at 93-119 Memorial Street in the City of Franklin, New Hampshire (the Site).

This SSQAPP presents the following information:

- Problem definition including a Site description and summary of background information for the Site
- Project description and timeline
- Preliminary conceptual site model (CSM)
- Assessment objectives and proposed sampling design and rationale
- Site-specific field sampling and analytical methodology
- Regulatory standards applicable to the Site for each proposed sampling media

This SSQAPP was prepared to be used in concert with Credere's Generic Quality Assurance Project Plan (QAPP) EPA Quality Assurance Tracking: Request for Assistance (RFA) #14123 revision dated September 4, 2014, which was prepared for all of Credere's EPA Brownfields work in New Hampshire. The quality assurance and quality control (QA/QC) procedures outlined in Credere's Generic QAPP will be followed for this investigation program including sample collection, handling, and analysis of samples; chain-of-custody; and data management, documentation, validation and usability assessment. Sampling as outlined in this SSQAPP will not occur until receipt of approval from EPA and the New Hampshire Department of Environmental Services (NHDES).

Figure 1 shows the general location of the Site in Franklin, New Hampshire; **Figure 2** presents pertinent Site features and proposed sampling locations; and **Figure 3** is a Project Organization Flow Chart for the Ferrari Realty Trust Property project team.

3. PROBLEM DEFINITION

3.1 SITE DESCRIPTION

The 1.219-acre Site is part of the Franklin Falls Historic District and consists of two buildings including the Stanley Mill in the eastern portion of the Site and a former Armory in the western portion of the Site. The Site is identified by the City of Franklin as Map 117, Lot 142 and is currently owned by Ferrari Realty Trust/Ferrari Trustees L & C. Below is a description of the two buildings including details of conditions observed during the Phase I ESA Site reconnaissance; however, Credere was limited in their ability to observed portions of the Site due to thick vegetation.

Stanley Mill

The Stanley Mill was observed to be a two-story mill building with a concrete and stone foundation. The original southeast portion of the building was constructed between 1897 and 1911. The mill building was expanded over time and reached its current configuration by 1929.

The building consisted of several large open rooms and hallways. The main rooms were accessed by sliding steel fire doors. A boiler room was located extending off the southwest portion of the building. An electric elevator was located in the southeast portion of the building. The building was vacant and generally empty with the exception of some debris. The building was in poor condition with water damaged floors, walls, and roofs.

Former Armory

The former Armory building was observed to be a single-story masonry structure. The original eastern portion of the Site building was constructed between 1911 and 1923 as a large open single room space. The western portion of the building was added between 1948 and 1956 and contains several rooms and a loading dock area. The former Armory building was vacant and mostly empty with the exception of an electrical hoist and debris. A fill and vent pipe were observed at the former Armory near the northwest corner of the building, and an underground storage tank (UST) is suspected to be located in this area. The building was in fair condition and appeared to need only cosmetic repairs.

3.2 SITE HISTORY

The Site

The Site was first developed by the G. W. Griffin and Company as a hacksaw manufacturing mill (Stanley Mill) between 1897 and 1911. The mill was expanded over the next few decades, and by 1929 the building reached its current configuration. The building was occupied by the G. W. Griffin Company through at least 1967 and likely through 1979 based on historical ownership records. Stanley Tool purchased the Site in 1979 and occupied the Site through at least 1986 when ownership was transferred to Franklin Falls Trust. It is presumed the building was vacated shortly thereafter.

The Armory was constructed between 1911 and 1923 and was occupied by the New Hampshire National Guard. G.W. Griffin and Company purchased the Armory in 1935, and the New Hampshire National Guard moved across Memorial Street to the location of the current Franklin Community Center. By 1948, the former Armory was used for storage by the French Mills, Inc., a tenant of G.W. Griffin. An addition was added to the western side of the former Armory by 1956; and by 1964 the former Armory was used as a mica depot. The former Armory was used most recently by PrETCo, a processing equipment manufacturer as indicated by the sign on the side of the building, and for storage by a local construction company. The former Armory has been vacant since approximately 2010.

Surrounding Area

The surrounding area was developed as early as 1884 with a lumber mill to the southeast. Several textile mills, garages, and distribution facilities were located upstream of the Site to the east. Chemicals, coal, dyes, and oils were used and stored at these facilities.

The adjoining property south of the Site (Riverbend Mill) was developed with the Acme Knitting Machine and Needle Company by 1911. By 1961, the factory was converted to the Shepard Grocery Company and was used as a warehouse/storage facility through at least 1967. By 1990, the building was occupied by various commercial and industrial businesses including a chapter of the Red Cross, a gymnastics center, non-ferrous casting, an attorney, wood worker, and nautilus company. The adjoining mill was vacant at the time of our Site visit.

3.3 PRIOR INVESTIGATIONS

The following prior environmental reports were identified for the Site.

Tank Closure Summary for Memorial Street Property, Lakes Region Environmental Contractors, Inc., January 12, 1999

A 10,000-gallon No. 2 fuel oil UST was removed from the Site on December 7, 1998. Based on the figure included in the Tank Closure Summary and conditions observed at the Site, the UST was located east of the Stanley Mill building.

Upon removal, the UST was observed to have minor pitting; however, no evidence of a release was observed within the excavation. Results from one confirmatory composite sample collected from the excavation base were below applicable NHDES Soil Remediation Standards (SRS). Based on the results of the confirmatory sample and observed subsurface conditions, no further assessment was recommended. The report was reviewed by NHDES on February 3, 1999, and no further action was required.

Phase I Environmental Site Assessment (ESA), August 8, 2014, Credere

Credere prepared a Phase I ESA for the Site dated August 8, 2014. Based on reviews of historical sources, environmental databases, interviews, information provided by the City of Franklin, a Site reconnaissance, and judgment by the Environmental Professional, the following recognized environmental conditions (RECs) were identified in connection with the Site:

- REC #1 – Disposal of industrial waste as fill at the Site
- REC #2 – Evidence of a UST near the northwest corner of the former Armory
- REC #3 – Presumed ACMs in waste form in the basement of the Stanley Mill
- REC #4 – Observed staining on a former transformer pad near the northeast corner of the Stanley Mill
- REC #5 – Threat of release from corroded drum in the former Armory and drums in the Stanley Mill
- REC #6 – Release to surface soil from hopper east of the Stanley Mill
- REC #7 - Release or threat of release from possible subsurface fuel oil supply lines in the boiler house
- REC #8 – Likely presence of surface soil impacts throughout the Site from historical Site use

Additionally, the following environmental findings, which did not meet ASTM E 1527-13's definition of a REC, historical REC (HREC), controlled REC (CREC), or *de minimis* condition (DMC), warranted the opinion of an Environmental Professional, were identified:

- Environmental Finding #1 – Observed possible hazardous building materials throughout the Site
- Environmental Finding #2 – Upstream historical industrial operations
- Environmental Finding #3 – Former UST removed from the east side of the Stanley Mill building

Based on the RECs and environmental findings identified during this Phase I ESA, Credere recommended the following:

- Immediate stabilization and removal of the corroded drum in the former Armory and removal of the drums in the Stanley Mill
- A Phase II investigation to confirm or dismiss the RECs and environmental findings identified in this Phase I ESA
- A Hazardous Building Materials Survey (HBMS) to assess the presence of hazardous building materials throughout the Site
- A supplemental Site reconnaissance in the late fall or spring when thick vegetation and foliage is not present

4. PROJECT DESCRIPTION & TIMELINE

4.1 REDEVELOPMENT SCENARIO

Franklin Falls Downtown Development Authority (FFDDA) intends to redevelop the Site to remove potential environmental concerns, preserve the historic significance, and return the property to a useful condition. Current proposed reuse is a boutique bed and breakfast with function rooms, a restaurant, and for a satellite college campus.

4.2 PROPOSED PROJECT TIMELINE

The following schedule is proposed for the assessment work. This is a dynamic schedule and tasks may be performed later based on document regulatory review time and contractor availability.

TENTATIVE DATE	ACTION
August 2014	Submit Draft SSQAPP
September 2014	EPA and NHDES SSQAPP Review Period
October 2014	Finalize SSQAPP and Begin Phase II ESA and HBMS
November 2014	Submit Draft Phase II ESA and HBMS Summary Report
December 2014	NHDES Review Period
January 2014	Finalize Phase II ESA and HBMS Summary Report

5. CONCEPTUAL SITE MODEL

A CSM was developed using the findings from prior environmental investigations and the Phase I ESA and will be updated in subsequent reports as new information becomes available. This CSM includes a description of the physical setting of the Site, contaminants of concern (COCs), extent of contamination, exposure pathways, and potential human and environmental receptors.

5.1 PHYSICAL SETTING

Topography

Based on Credere's observations and the United States Geological Survey (USGS) Topographic Map of the Franklin Quadrangle, New Hampshire, topography at the Site is generally flat to gently sloping to the north. The northern property boundary is further characterized by a steep northward slope that meets the Winnipesaukee River. Elevation at the Site is approximately 300 feet above mean sea level. An excerpt from the USGS map has been included as **Figure 1**.

Geology

Surficial Geology

According to the physical Setting Source Summary in the Environmental Database Report (EDR) reviewed during the Phase I ESA, which is derived from the US Department of Agriculture's Soil Conservation Service National Cooperative Survey, Site soils are mapped as Paxton soils, which typically consist of well drained very stony fine sandy loam with slow infiltration rates.

Credere also observed fill material through the surface of the northern edge of the Site.

Bedrock Geology

According to the Bedrock Geologic Map of New Hampshire, bedrock beneath the Site consists of Lower Silurian meta-argillite, meta-conglomerate, and calc-silicate rocks of the lower part of the Rangley Formation. Rocks were formed by metamorphosis of sedimentary deposits in the Central Maine Composite Terrane (Central Maine Trough) and igneous intrusive rocks. No bedrock outcrops were observed during Credere's Site reconnaissance.

Hydrology

The nearest surface water body to the Site is the Winnipesaukee River, which is located along the northern edge of the Site. The Winnipesaukee River flows west and joins the Pemigewasset River approximately 0.6-miles south of the Site. Storm drains were observed along Memorial Street, which likely accept runoff from the southern portion of the Site. Stormwater likely infiltrates permeable areas of the northern portion of the Site or flows overland to the Winnipesaukee River.

Based on observed grades and mapped topography, groundwater at the Site is presumed to flow to the north towards the Winnipesaukee River. However, due to the oxbow path of the river, groundwater at the adjoining property to the south flows southwest as documented by prior investigations at that property.

5.2 CURRENT CONTAMINANTS OF CONCERN

Based on the historical use of the Site, current COCs include volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) associated with the use of degreasers at the Stanley Mill and the suspect UST at the former Armory, heavy metals associated with the historical manufacturing of metals tools, and polychlorinated biphenyls (PCBs) typically found in cutting and lubricating oils used in machining during the time period of the mill's operation and associated with the former transformer pad.

Asbestos, lead contained in lead-based paint (LBP), and PCBs in building materials may be present in/on the current Site building and are also considered COCs.

5.3 EXTENT OF CONTAMINATION

The extent of COCs at the Site is currently unknown based on lack of prior environmental assessment at the Site; however, based on Site observations and historical information reviewed during the Phase I ESA, COCs are potentially located in the following areas.

- VOCs and SVOCs are potentially located in areas where cutting and lubricating oils or degreasers were used, handled (e.g. loading docks), stored (e.g. the basement or former garage), or possibly discharged to the surface or subsurface (e.g. the floor drain/discharge in the Stanley Mill and industrial fill), as well as in the area of the suspect UST at the former Armory. In addition, fuel oil related petroleum hydrocarbons may be in the location of the UST or in the vicinity of the fuel oil supply lines.

- Metals may be found in surface soil throughout the Site due to the long history of metals parts manufacturing, in areas of material handling areas (e.g. loading docks and the garage), and in areas of industrial waste disposal (e.g. the fill areas along the north edge of the Site, and the hopper east of the Stanley Mill).

- PCBs are potentially located in oil handling areas (e.g. loading docks), in surface soils due to tracking of oils across the Site by trucks or by foot traffic, surrounding the former transformer pad, in the industrial waste fill, and in areas of concrete staining within the Site buildings.

- If ACM, PCBs, and LBP are identified in/on the Site buildings they are presumed to be confined to the Site buildings or immediately surrounding the Site buildings in soil.

5.4 EXPOSURE PATHWAYS AND POTENTIAL RECEPTORS

Exposure pathways describe how a human or environmental receptor comes into contact with contaminants that may be present at the Site. Exposure pathways presented in the CSM include the following:

Active Ingestion:	The active ingestion pathway represents exposure which may occur through the active ingestion of contaminant concentrations via a drinking water supply well, through agricultural products, or through direct consumption of soil (typically by children).
Inhalation:	This pathway is primarily associated with groundwater contamination within 30 feet of an occupied structure when groundwater elevation is less than 15 feet below surface grade, or when depth to groundwater is unknown. This pathway is applicable when receptors may inhale impacted media in the form of contaminated vapor.
Dermal Absorption:	Exposure via dermal absorption occurs when receptors are exposed to chemical concentrations present in soil, groundwater, surface water, or hazardous building materials through direct contact with the skin.
Incidental Uptake:	This pathway is applicable when receptors may incidentally inhale or ingest impacted media in the form of contaminated dust, chips, or airborne asbestos fibers.

Potential Receptors are categorized by duration of exposure and intensity of use at the Site. The receptor categories described in the CSM include the following:

Commercial Workers:	Commercial receptors are those which are present at the Site for long durations but with low intensity exposure such as indoor office workers.
Recreational or Park User:	Park users are characterized by low duration (i.e. less than two hours per day) and low intensity usage such as that which would occur during activities such as walking, shopping, and bird watching.
Excavation or Construction Worker:	Excavation or construction workers are present at the Site for short durations though intensity of use is high, such as during non-routine activities including construction or utility work. Examples include utility and construction contractors and landscapers.
Terrestrial Biota:	These receptors include flora and fauna which may be exposed to contaminants in their respective environments.

5.6 CONCEPTUAL SITE MODEL SUMMARY

Under current conditions, exposure to possible remaining or unassessed contamination at the Site is limited. However, the planned use of the Site as a bed and breakfast, event center, and for a satellite college increases exposure to future occupants of the Site.

Under the proposed reuse, potential receptors include construction workers during construction, future employees of the Site, future students of the college, and future patrons of the bed and breakfast and event center, as well as terrestrial biota. If contaminants are detected near the floor drain discharge pipe, aquatic biota may also be affected. Exposure pathways include ingestion by terrestrial and aquatic biota; inhalation of contaminants in indoor air due to the potential for vapor intrusion if soil or groundwater near the Site buildings have been impacted by VOCs; dermal absorption through terrestrial and aquatic biota, use of the property by future occupants, and construction workers contact with soil or groundwater during construction; and incidental uptake of contaminated dust or asbestos fibers both during construction and by future occupants.

6. SAMPLING DESIGN

6.1 OBJECTIVE

The main objective of this assessment is to assess the RECs and environmental findings and fulfill the recommendations identified during the Phase I ESA. Data collected at the Site during this assessment will be considered in the planning stages for possible cleanup activities and Site reuse. The following tasks are proposed to address this objective:

- Perform a ground penetrating radar (GPR) survey
- Advance soil borings and collect soil samples from each boring location
- Collect surface soil samples
- Install groundwater monitoring wells and collect groundwater samples
- Excavate test pits and collect soil samples from each test pit
- Collect samples from areas of stained concrete
- Perform an asbestos survey of the Site buildings and collect samples of suspect ACM
- Perform PCB-containing building material survey of the Site buildings and collect samples of suspect PCB-containing materials
- Perform a LBP screening of the Site buildings and perimeter soil and collect surface soil samples from the building perimeter
- Perform an inventory of universal and/or hazardous wastes present in the Site buildings

Specific sampling methodologies are described in **Section 7**. **Table 1** includes the number and type of samples that are proposed to be collected with accompanying rationale, selected analytical methods, and sample volume and preservation details. **Table 2** is a Standard Operating Procedure (SOP) reference table detailing the version of each SOP that will be used during the field sampling program.

6.2 GROUND PENETRATING RADAR SURVEY

Credere will contract with Vermont Underground Locators (VUL) to perform a GPR survey at the Site to assess if the suspect UST behind the former Armory is currently present, assess the remainder of the Site (particularly surrounding the Stanley Mill boiler house), and clear test pit and soil boring locations for utilities (in addition to DigSafe).

6.3 SOIL BORING ADVANCEMENT & SOIL SAMPLING

Six (6) soil borings (CA-SB-1 through CA-SB-6) will be advanced at the Site. Soil samples will be collected from either the surface to assess surface soil impacts from historical use of the Site; from the area of greatest observed contamination or from the groundwater interface to assess possible light non aqueous phase liquid (LNAPL) or dense non-aqueous phase liquid (DNAPL) impacts associated with historical use of the Site; from the observe fill interval to assess impacts associated with the industrial waste fill along the river; and from the first encountered native soil below the fill to assess the leaching of contaminants to native material or delineate the extent of fill impacts.

One (1) surface soil sample will be collected from soil boring CA-SB-1 from 0 to 2-feet to assess surface soil conditions at the Site.

Two (2) soil samples will be collected from soil boring CA-SB-2: one surface soil sample will be collected from 0 to 2 feet, and one subsurface sample will be collected from the depth interval of greatest observed contamination. In the absence of evidence of contamination, the subsurface samples will be collected from the groundwater interface.

One (1) subsurface soil sample will be collected from soil boring CA-SB-3 from the depth interval of greatest observed contamination to assess the potential for fuel oil releases to have occurred from the fuel oil supply lines (CA-SB-3). In the absence of evidence of contamination, the subsurface samples will be collected from the groundwater interface.

Three (3) soil samples will be collected from CA-SB-4, CA-SB-5, and CA-SB-6: one from the interval of observed industrial fill material, one from the first encountered native material below the fill, and one from the area of greatest observed contamination. In the absence of evidence of contamination the sample from the area of greatest observed contamination will be collected from the groundwater interface. If the first encountered native material is within the same interval as the water table and no evidence of contamination is identified, or within the same interval as the area of greatest observed contamination, only two samples will collected: one from the fill material and one from first encountered native material. The first encountered native material samples will be placed on hold pending the results of the above fill material analyses. If results of the fill material analyses exceed their applicable NHDES SRSs, then the associated native material sample will be authorized for analysis in an effort to vertically delineate the extent of impacts associated with the fill. If only two soil samples are collected from any of the borings, VOCs and SVOCs will initially be authorized for analysis and only the PCBs and priority pollutant metals will be placed on hold pending the results of the above fill material sample.

Seven (7) surface soil samples will be collected from 0 to 2 feet using hand tools (hand auger or trowel) to assess possible sources of surficial contamination identified during the Phase I ESA including the historical use of the Site (CA-SS-1 through CA-SS-4), the floor drain discharge pipe on the banks of the Winnipesaukee River (CA-SS-5), the former transformer area (CA-SS-6), and the metal dust hopper (CA-SS-7). Surface soil samples locations will be biased towards evidence of contamination (e.g. staining, fill materials, etc.).

Proposed sample locations are depicted on **Figure 2,** and the rationale for each sample is summarized in **Table 1**.

6.4 MONITORING WELL INSTALLATION & GROUNDWATER SAMPLING

Soil borings CA-SB-1 through CA-SB-6 will be completed as groundwater monitoring wells (CA-MW-1 through CA-MW-6). Groundwater samples will be collected from the newly installed wells after development and at least a 14 day stabilization period. Groundwater samples will be collected to assess groundwater at the upgradient most position of the Site (CA-MW-1), impacts associated with the storage of oil and hazardous materials in the former garage (CA-MW-2), groundwater downgradient of the Stanley Mill boiler house (CA-MW-3), groundwater downgradient of the Stanley Mill and within the industrial fill material (CA-MW-4), groundwater downgradient of the basement floor drain and within the fill material (CA-MW-5), and downgradient of the former Armory and within the fill material (CA-MW-6).

Proposed monitoring well locations are depicted on **Figure 2,** and the rationale for each sample is summarized on **Table 1**.

6.5 TEST PIT EXCAVATION & SOIL SAMPLING

Three test pits (CA-TP-1 through CA-TP-3) are proposed to be excavated by ENPRO Services Inc. (ENPRO) to observe the extent of the industrial fill material along the northern side of the Site. Two (2) soil samples will be collected from each test pit from the observed fill interval and from the first encountered native soil beneath the fill. Additional test pits may be excavated to further delineate the visual extent of fill; however, no additional samples will be collected from these additional test pits.

Proposed test pit locations are depicted on **Figure 2,** and the rationale for each sample is summarized on **Table 1**.

6.6 CONCRETE SAMPLING

Three (3) concrete samples (CA-CC-1 through CA-CC-3) will be collected from areas of observed staining within the former Armory, the Stanley Mill basement, and from the former transformer pad. Samples will be analyzed for PCBs to assess if the concrete floors/pads are regulated as PCB remediation waste as a result of a historical release of presumably PCB containing materials.

6.7 ASBESTOS SAMPLING

Credere will perform a survey of the Site buildings to identify suspect ACM, and each suspect ACM will be sampled. Sample results will be used to properly manage ACM during renovation to the Site buildings. Twenty-five (25) samples of suspect ACMs will be collected in triplicate (i.e. 75 total ACM samples). This sampling will be performed in accordance with NHDES Chapter Env-A 1800 – Asbestos Management and Control. The number of samples actually collected will be dependent on the number and volume of suspect ACMs that are encountered, but will not exceed 75 individual samples without project team approval.

6.8 PCB-CONTAINING BUILDING MATERIAL SAMPLING

To assess the potential presence of PCB-containing building materials, the buildings will be inspected and suspect materials will be inventoried and considered for sampling. Materials that typically contain PCBs include caulk/sealants, paint, and mastics/adhesives that were manufactured between approximately 1930 and 1980 and are most commonly in areas that endure high wear, weather, high heat, or moisture. Typical materials and locations where PCBs are encountered include, but are not limited to:

- exterior caulks and sealants around doors and windows or within expansion joints
- wall paints in high heat or moisture areas such as boiler rooms, equipment rooms, or basements
- floor paints in high traffic areas such as hallways or building entrances
- mastics beneath floor tiles

Considering the size of the Site buildings and variety of building materials that are expected to be inventoried, 12 suspect PCB-containing building materials (CA-PCB-1 through CA-PCB-12) that are more likely to contain PCBs will be collected for analysis. One field duplicate from each distinctive type of material sampled (e.g. caulks/sealants/adhesives and paints) to a maximum of two duplicate samples will be collected for analysis. Samples will be collected to assess if any hazards are present associated with PCBs in building materials and if the building materials are regulated as PCB bulk product waste as defined by 40 CFR 761.3. If based on the initial results, additional assessment of PCB-containing building materials is needed, approval for additional samples will be proposed and approved under a separate SSQAPP amendment. Data will be used to properly manage building materials that may contain PCBs during renovations to the Site buildings.

6.9 LBP SCREENING

Painted surfaces throughout the Site buildings will be screened for lead in LBP using an X-ray fluorescence (XRF) meter. LBP is defined as paint with a lead concentration of 1.0 milligrams per square centimeter (mg/cm^2) or greater in accordance with the United States Department of Housing, Chapter 7: Lead-Based Paint Inspections, 1997 Revision (HUD Guidelines) and with the Chapter 130 – Lead Paint Poisoning Prevention and Control of New Hampshire Statues (Chapter 130). The number of screening points will be dependent on the number of different types/colors of painted surfaces encountered in/on the Site buildings.

If LBP is identified on the exterior of the former Armory (the current exterior or sub-layers), soil surrounding the building will be screened for lead using an XRF meter to assess if chipping or flaking of LBP (presently or in the past) from the Site building has impacted Site surface soil. Soil surrounding the former Armory will be presumed to be impacted if XRF screening results exceed 240 parts per million (ppm) (40% error range for the XRF relative to the NHDES Soil Remediation Standard (SRS) for lead of 400 milligrams per kilogram (mg/kg)).

The Stanley Mill building is not painted on the exterior and exterior screening will not be conducted at the Stanley Mill.

6.10 LBP PERIMETER SOIL SAMPLING

If surface soil surrounding the former Armory is found to be impacted by LBP (see **Section 6.9**), surface soil samples will be collected from soil surrounding the building to quantify soil concentrations from chipping or flaking LBP from the exterior of the Site building. Two (2) samples locations will be selected from the south side of the former Armory, one (1) location will be selected from the east side of the Site building (in additional to a separate surface soil sample proposed along this wall, CA-SS-3), two (2) locations will be selected from the north side of the former Armory, and one (1) location will be selected from the west side of the former Armory (in addition to a separate surface soil sample proposed along this wall, CA-SS-4). Samples locations will be biased to locations of the highest XRF screening values.

Two samples will be collected from each location (a total of 12 samples) at 0 to 0.5-feet and 0.5 to 1-foot. The 0.5 to 1-foot samples will be placed on hold pending the results of the 0 to 0.5-feet samples. If results of the related 0 to 0.5-feet sample exceed applicable NHDES SRSs, the associated 0.5 to 1-foot sample analysis will be authorized to delineate the vertical extent of lead impacted soil

No analytical soil samples will be collected if LBP is not found on the exterior of the Site building or if soil XRF field screening results do not exceed 240 ppm.

6.11 UNIVERSAL/HAZARDOUS WASTE INVENTORY

Materials that once removed will meet the definition of universal/hazardous waste include, but are not limited to, fluorescent lighting, smoke detectors, thermostats (that contain mercury), and lead acid batteries. These types of materials at the Site will be inventoried. Inventory results will be used to properly manage universal and/or hazardous wastes during renovation or demolition of the Site building.

7. SAMPLING & ANALYTICAL METHODS REQUIREMENTS

The proposed sampling activities will be conducted according to **Table 1**. Field activities will be conducted in accordance with Credere's Generic QAPP RFA #14123 and the SOPs referenced on **Table 2**.

7.1 GROUND PENETRATING RADAR SURVEY

VUL will perform their GPR survey by transecting the Site in a grid pattern. Anomalies will be reported to Credere in real time, and Credere will document the location of the anomalies, if any. The GPR results will be summarized in the Phase II ESA report.

7.2 SOIL BORING ADVANCEMENT & SOIL SAMPLING

Six (6) soil borings (CA-SB-1 through CA-SB-6) will be advanced at the Site. Soil borings CA-SB-1 through CA-SB-3 will be advanced to approximately 5 feet below the water table, and soil borings CA-SB-4 through CA-SB-6 will be advanced to the first confining layer below the water table or to bedrock. Borings will be advanced using direct push drilling methods with a Geoprobe® macrocore sampling device, or equivalent. Soil cores will be collected continuously using dedicated, disposable polyethylene liners. Macrocores will be individually logged, evidence of contamination will be noted, and soil will be field screened for total volatile organic compounds (VOCs) using a Thermo 580B OVM PID (or similar) calibrated with a 100 part per million by volume (ppm_v) isobutylene gas with a response factor of 1.0 ppm_v. Soil will be screened in accordance with the NHDES HWRB-12 jar headspace technique SOP.

Visible asphalt and base materials, landscaping materials, and other organic detritus will be removed prior to sampling.

Sample target depths are summarized in **Section 6.4** and on **Table 1**, which is to be used as a field guide.

In all soil samples, representative soil from a 2-foot interval will be sampled from the macrocore or from the surface using decontaminated hand tools (hand auger or trowel) and placed in a decontaminated stainless steel bowl. Volatile samples (VOCs) collected from boring locations will be collected directly from the macrocores using a dedicated soil syringe immediately after opening to prevent loss of volatiles and degradation. Soil for other analyses will be homogenized and placed in laboratory provided glassware. Proposed sample analysis for each respective sample as well as the required volume and preservation is provided on **Table 1**. Soil samples will be stored on ice and submitted to ARA for analysis.

Excess soil from each boring or surface sample location will be returned to its place of origin within the borehole or to the surface surrounding the borehole.

7.3 MONITORING WELL INSTALLATION & GROUNDWATER SAMPLING

Each boring will be completed as a groundwater monitoring well. Each groundwater monitoring well will be constructed using 10 feet of 0.010-inch slotted PVC screen with at least 5 feet of screen below the depth of the water table and enough solid PVC riser to reach the ground surface. The well annulus will be filled with No. 2 washed silica sand with a bentonite seal and the wells will be completed with flush mounted road boxes or stand pipes depending on the location.

Following installation, each monitoring well's top of PVC elevation will be surveyed to an onsite benchmark, if available, or an arbitrary datum if necessary to allow for the determination of relative groundwater elevations and the calculation of groundwater flow at the Site.

Each well will be developed by overpumping and agitation methods. The wells will be purged until a total of at least three well volumes have been removed and turbidity has been reduced to less than 20 NTUs.

Following development, Credere will allow at least two weeks for the monitoring wells to equilibrate with the surrounding aquifer prior to sampling. Depth to groundwater and non-aqueous phase liquid (NAPL) thickness, if present, will then be measured. Groundwater elevations will be calculated relative to the top of the well casing and elevations will be mapped to assess the groundwater flow direction.

Credere will sample each well in accordance with **Table 1** using low-flow sampling methodologies. During sampling, groundwater will be periodically monitored for temperature, pH, oxidation-reduction potential, specific conductivity, dissolved oxygen, and turbidity using a multi-parameter meter and an in-line flow-through cell until parameters have stabilized over a period of three readings, spaced at least 5 minutes apart or at a spacing to allow for a complete exchange of flow through the flow-through cell based on the flow-through cell volume and flow rate. Upon stabilization of field parameters, groundwater samples will be collected immediately after the pump and directly into the appropriate bottle ware in order of decreasing volatility. Proposed sample analysis for each respective sample as well as the required volume and preservation is provided on **Table 1**. Groundwater samples will be stored on ice and submitted to ARA for analysis.

7.4 TEST PIT EXCAVATION & SOIL SAMPLING

Soil will be removed from the test pits by an excavator and stockpiled adjacent to the test pit on polyethylene sheeting to prevent surface contamination of the adjoining area. Test pits will be excavated until native soil is encountered unless the maximum extent of the excavator is encountered first. The sidewalls of the test pit will be logged in the field by a Credere geologist and the thickness of the fill will be measured.

Two soil samples will be collected from each of three proposed test pits. Soil will be collected from the entire observed fill interval (i.e. a variable thickness) and from the two foot interval of first encountered native soil below the fill. Soil from each interval to be sampled will be collected into a decontaminated stainless steel bowl and homogenized. Soil will be transferred to laboratory provided glassware and submitted to ARA for analysis in accordance with **Table 1**. The first encountered native material samples will be placed on hold pending the results of the above fill material samples. If results of the fill material analyses exceed their applicable NHDES SRSs, then the associated native material sample will be authorized for analysis in an effort to vertically delineate the extent of impacts associated with the fill.

After sampling, soil will be returned to the excavation in the approximate order it was removed. Soil will be compacted with the excavator bucket in 1-foot lifts. The surface will be finished such that no hazards are protruding from the ground (e.g. sheets of metals, large metal scraps).

7.5 CONCRETE SAMPLING

Concrete samples will be collected from three areas of observed staining (CA-CC-1 through CA-CC-3). A hammer drill with a 1-inch carbide drill bit will be used to pulverize the concrete for sampling. A half inch depth should be measured and marked on the drill bit. An aluminum foil mat with a 1-inch diameter hole will be placed over the location to be sampled. A 0.5-inch depth hole will be advanced through the aluminum foil hole using the hammer drill. Concrete dust will be collected in a glass container to be analyzed for PCBs. Multiple 0.5-inch holes in adjacent locations may be advanced to obtain adequate volume for sample analysis. Additionally, a stainless steel scoopula or bulb syringe may be used to extract concrete dust from the 0.5-inch hole. Dedicated sampling tools will be used at each location to prevent cross contamination.

7.6 ASBESTOS SAMPLING

Any sampling of suspect ACM at the Site will be conducted by a New Hampshire Certified Asbestos Inspector and in accordance with Chapter 130. Three discrete bulk samples will be collected from each type of homogenous suspect ACM (25 suspected ACMs sampled in triplicate for a total of 75 samples). Minor destructive sampling may be required. Samples will be analyzed by EMSL Analytical, Inc. (EMSL) of South Portland, Maine, using Polarized Light Microscopy (PLM) according to EPA Method 600/R-93/116.

7.7 PCB-CONTAINING BUILDING MATERIAL SAMPLING

The buildings will be surveyed to locate the materials that in Credere's experience are more likely to contain concentrations of PCBs exceeding the PCB bulk waste criteria. Twelve (12) samples (CA-PCB-1 through CA-PCB-12) will be collected using dedicated disposable tools and placed in laboratory provided glassware. Samples will be submitted to ARA for analysis of PCBs by EPA Method 8082 using soxhlet extraction method 3540C.

7.8 LBP SCREENING

Painted surfaces will be screened for the presence of lead in the form of LBP using an XRF meter. Each accessible color and type of paint throughout the Site building will be screened. Paints with screening concentrations of lead exceeding 1.0 mg/cm^2 will be considered LBP. If the white paint (or sub-layers) on the exterior of the former Armory is identified as LBP, soil around the perimeter of the building will be screened to assess if flaking or chipping paint has impacted Site soils. Soil will be screened in 10-foot intervals around the perimeter of the Site building with a focus on areas of the building with the most significant chipping paint.

XRF precision will be assessed by performing precision measurements at one soil screening location. A 7 time replicate will be performed and the relative standard deviation (RSD) will be calculated (RSD = (SD/mean concentrations) x 100). RSD should not exceed 20 percent. If LBP screening results are within the instrument error range (0.6 to 1.1 mg/cm^2), the presumed LBP coated surface will be screened in triplicate at three adjoining locations on the same surface to assure similar results. If the RSD exceeds the relative percent difference, the XRF will be recalibrated and locations selected for analytical samples will be rescreened to confirm the elevated concentration. Additionally, if the precision test fails, the soil analytical results will be relied upon for future risk assessment and/or remediation planning. An SOP for use of an XRF is included in **Appendix B**.

7.9 LBP PERIMETER SOIL SAMPLING

If soil is found to be impacted by LBP chips (See **Section 6.10**), 12 soil samples will be collected from 6 locations (CA-SS-LBP-1 through CA-SS-LBP-6). Samples will be collected using a hand auger. Soil from 0 to 0.5-feet will be collected by hand auger, placed in a decontaminated stainless steel bowl, and homogenized. Soil will then be transferred to laboratory provided glassware and submitted to ARA to be analyzed for lead by EPA Method 6010C. Soil from 0.5-1-foot will be collected by the same method and placed on hold pending the results of the 0 to 0.5-foot sample. If results of the 0 to 0.5-foot sample exceed the NHDES lead SRS of 400 mg/kg, the associated 0.5 to 1-foot sample will be authorized for analysis.

7.10 UNIVERSAL/HAZARDOUS WASTE INVENTORY

Materials as described in **Section 6.12** will be manually counted to inventory what will require disposal as universal or hazardous wastes prior to building demolition and preparation of the Site for residential use.

8. REGULATORY STANDARDS

Sample results will be compared to the applicable state and/or federal standards/guidelines described below. **Appendix A** includes Analytical Sensitivity and Project Criteria Tables for the Site, which compares regulatory standards for each contaminant to the analytical limits of the laboratory method used.

8.1 SOIL ANALYTICAL RESULTS

Soil analytical results will be compared to the New Hampshire Code of Administrative Rules Chapter Env-Or 600 – Contaminated Site Management Table 600-2 SRSs.

8.2 GROUNDWATER ANALYTICAL RESULTS

Groundwater analytical results will be compared to the New Hampshire Code of Administrative Rules Chapter Env-Or 600 – Contaminated Site Management Table 600-1 Ambient Groundwater Quality Standards (AGQS) and Table 2, Method 1 Groundwater Standards as revised in February 2013.

8.3 CONCRETE ANALYTICAL RESULTS

PCB results from the three concrete samples will be compared to the remediation waste cleanup guidelines of 1 or 25 mg/kg for low or high occupancy areas, respectively, based on the future reuse of each area sampled in accordance with 40 CFR 761.61.

8.4 ASBESTOS RESULTS

Laboratory analytical results for asbestos bulk samples will be compared to the 1% limit specified in Chapter Env-A 1800 – Asbestos Management and Control.

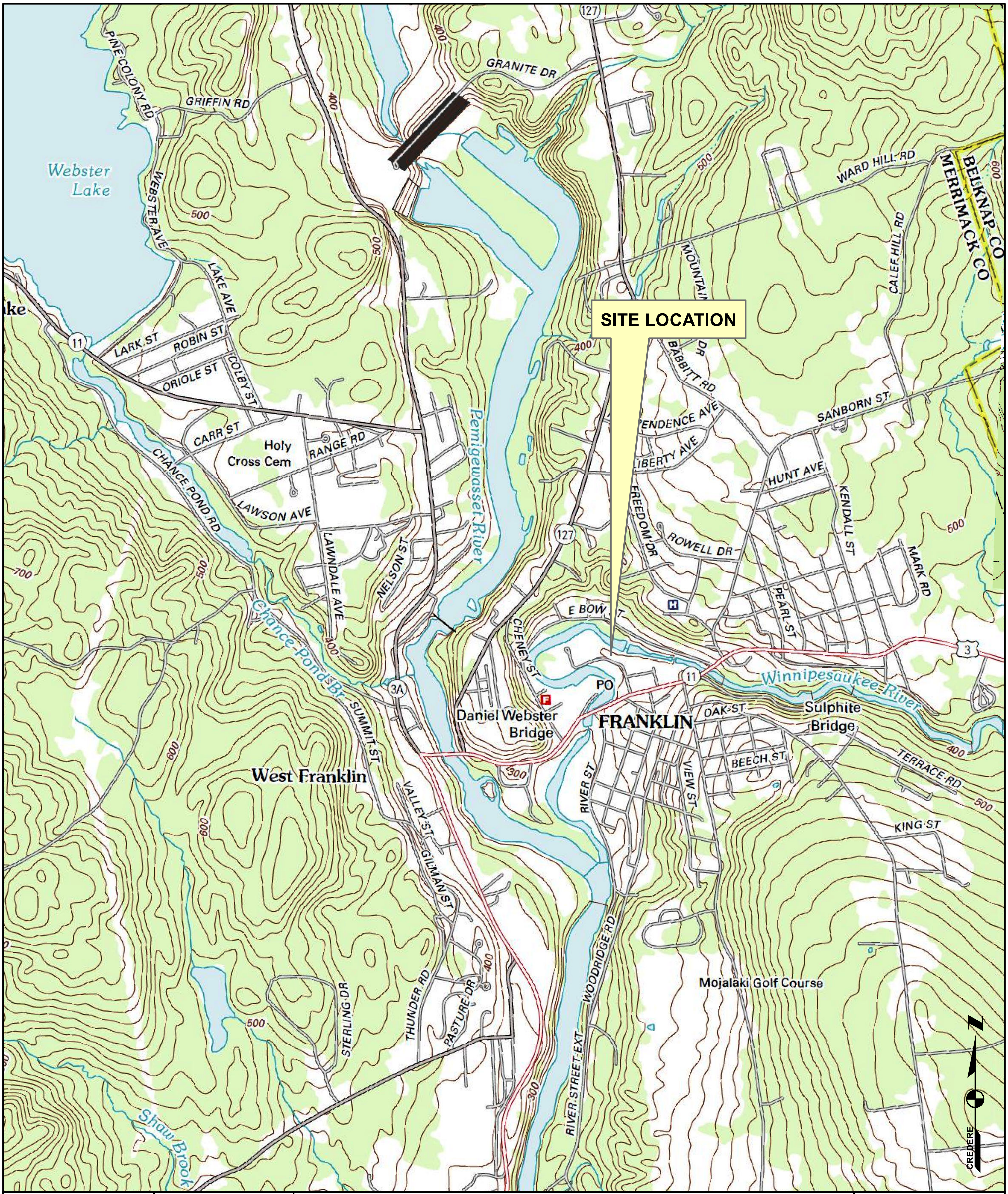
8.5 PCBS IN BUILDING MATERIALS RESULTS

PCB containing building materials will be compared to the 40 CFR 761.3 definition of PCB bulk product waste. Results will be compared to the 50 mg/kg threshold criteria.

8.6 LBP SCREENING RESULTS

Screening results will be compared to the 1.0 mg/cm^2 HUD Guideline and Chapter 130.

FIGURES



DRAWN BY: **SWM**
DATE: **7/15/2014**
CHECKED BY: **ASD**
PROJECT: **14001247**

FIGURE 1
SITE LOCATION PLAN

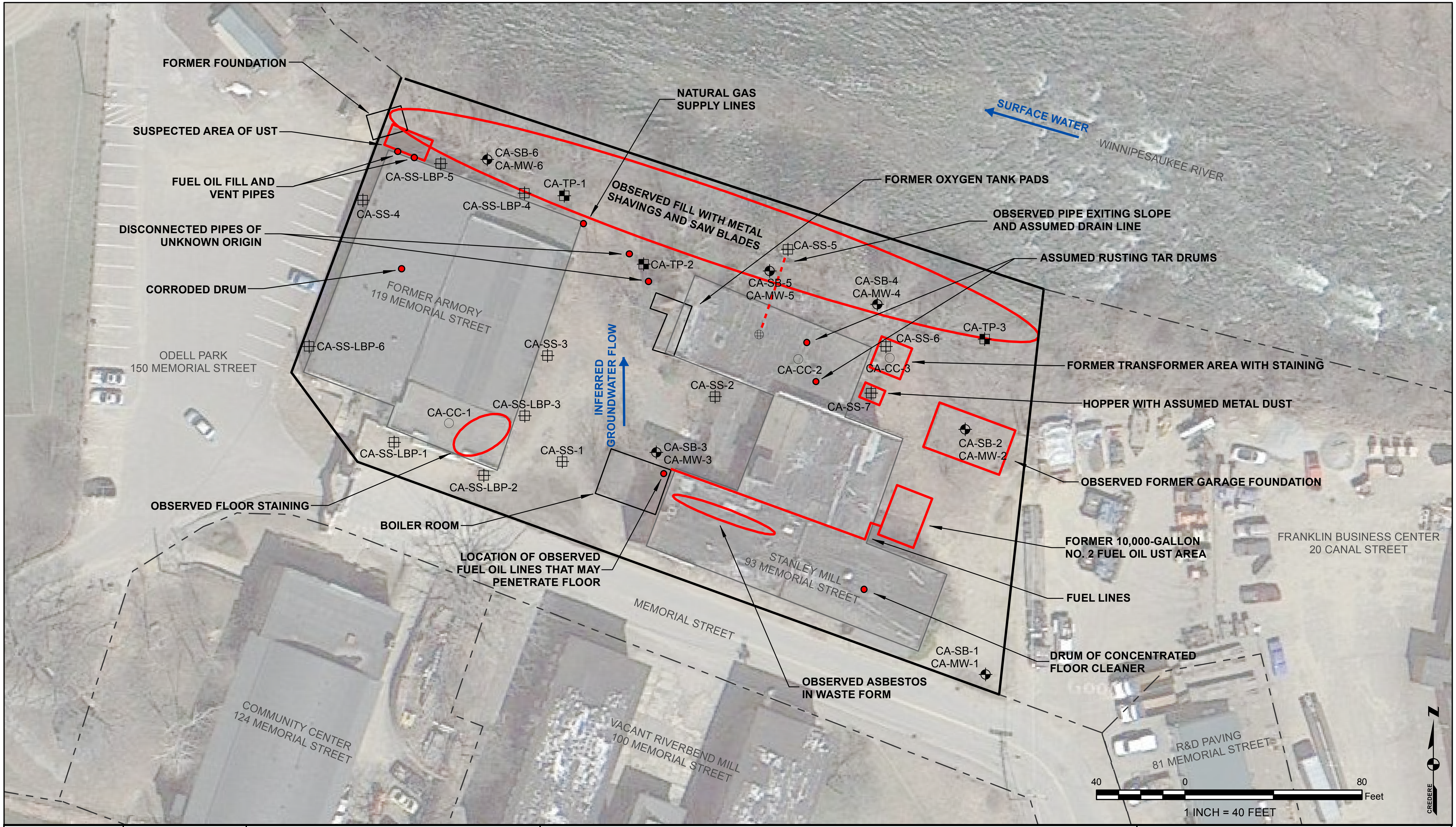
Credere Associates, LLC
776 MAIN STREET
WESTBROOK, MAINE
Tel. 207.828.1272
Fax 207.887.1051
WWW.CREDERELLC.COM

FERRARI REALTY TRUST PROPERTY
93-119 MEMORIAL STREET
FRANKLIN, NEW HAMPSHIRE 03235

1,000 0 2,000
Feet
1 inch = 2,000 feet.

Data Source: USGS Quadrangles: Franklin, New Hampshire 7.5 minute series 2012

T:\Data\NH\Town\Franklin\14001247-Ferrari Mill



DRAWN BY: **SWM**
CHECKED BY: **ASD**
DATE: **8/21/2014**
PROJECT: **14001247**

Credere Associates, LLC
776 MAIN STREET
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Community Economy Environment

FIGURE 2
SAMPLE LOCATION PLAN

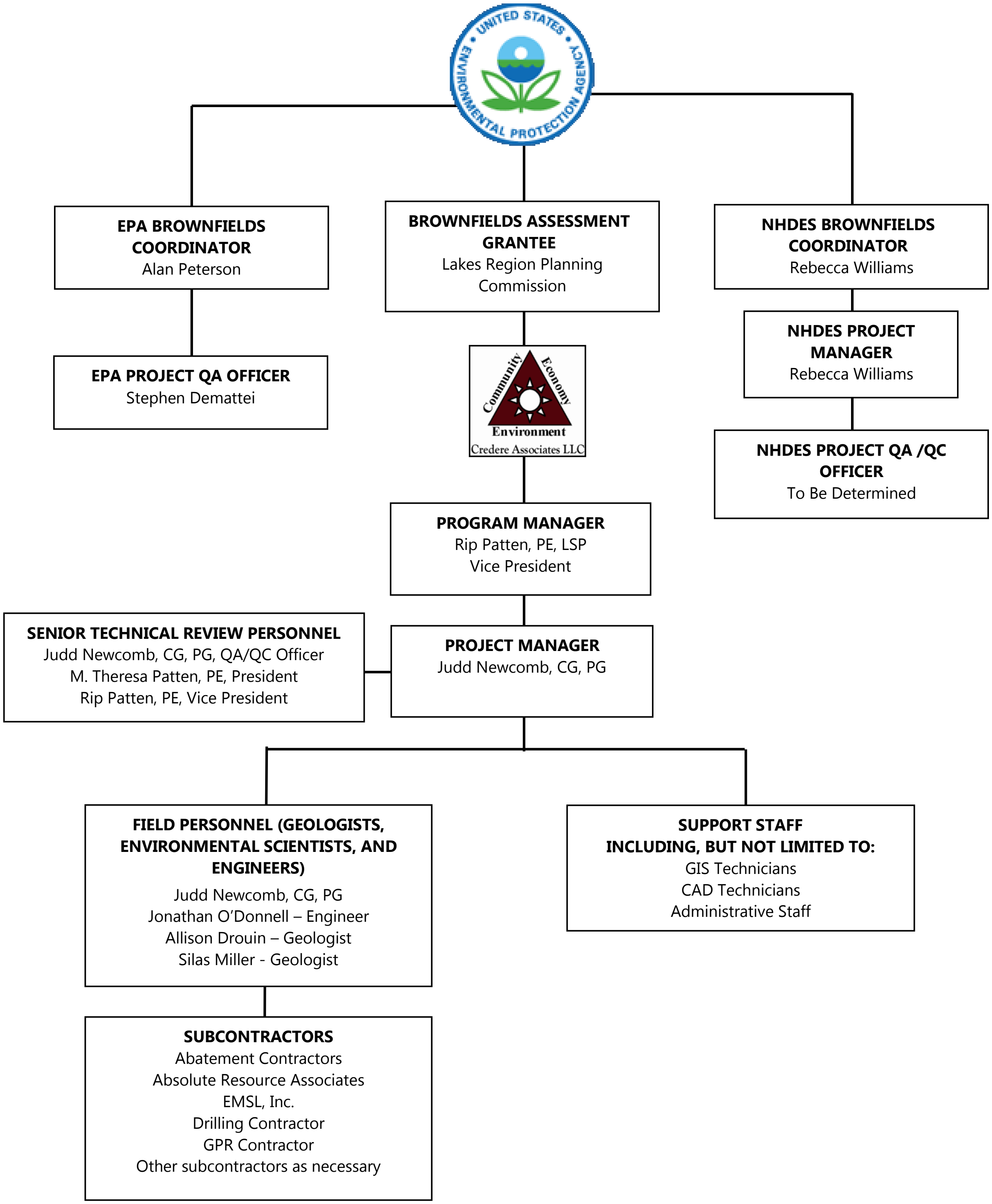
FERRARI REALTY TRUST PROPERTY
93-119 MEMORIAL STREET
FRANKLIN, NEW HAMPSHIRE 03235

□	AREA OF POTENTIAL CONCERN
●	POINT OF POTENTIAL CONCERN
⊕	FLOOR DRAIN
▣	TEST PIT
⊕	SOIL BORING AND MONITORING WELL
⊞	SOIL SAMPLE
○	CONCRETE SAMPLE
- - -	INFERRED LINES
—	SITE BOUNDARY
— -	PARCEL BOUNDARY
—	BUILDING BOUNDARY

NOTES:
1. EXISTING CONDITIONS FEATURES SHOWN ON THIS PLAN ARE APPROXIMATE AND ARE BASED ON INFORMATION OBTAINED FROM THE CITY OF FRANKLIN TAX MAP 117, 2013 ORTHO IMAGES AND THE SITE RECONNAISSANCE PERFORMED ON JULY 9, 2014.

T:\Data\NH\Town\Franklin\14001247\Ferrari Mill

Figure 3 – Project Organization Flow Chart



EPA BROWNFIELDS COORDINATOR
Alan Peterson

EPA PROJECT QA OFFICER
Stephen Demattei

BROWNFIELDS ASSESSMENT GRANTEE
Lakes Region Planning Commission

PROGRAM MANAGER
Rip Patten, PE, LSP
Vice President

PROJECT MANAGER
Judd Newcomb, CG, PG

SENIOR TECHNICAL REVIEW PERSONNEL
Judd Newcomb, CG, PG, QA/QC Officer
M. Theresa Patten, PE, President
Rip Patten, PE, Vice President

NHDES BROWNFIELDS COORDINATOR
Rebecca Williams

NHDES PROJECT MANAGER
Rebecca Williams

NHDES PROJECT QA /QC OFFICER
To Be Determined

FIELD PERSONNEL (GEOLOGISTS, ENVIRONMENTAL SCIENTISTS, AND ENGINEERS)
Judd Newcomb, CG, PG
Jonathan O'Donnell – Engineer
Allison Drouin – Geologist
Silas Miller - Geologist

SUBCONTRACTORS
Abatement Contractors
Absolute Resource Associates
EMSL, Inc.
Drilling Contractor
GPR Contractor
Other subcontractors as necessary

SUPPORT STAFF
INCLUDING, BUT NOT LIMITED TO:
GIS Technicians
CAD Technicians
Administrative Staff

TABLES

Table 1: Sample Reference Table
Ferrari Realty Trust Property
93-119 Memorial Street, Franklin, New Hampshire

Media to be Collected	Proposed Sample IDs[2]	Sample Type	Sample Rational	Sample Depth (feet bgs)	Field Analysis/ Observations	No. of Samples for Analysis	QA/QC Samples	Analytical Method	Sample Container Information & Preservative (per location)*,[1]	Laboratory To be Used
Soil	CA-SB-1	Surface soil	-To assess surface soil impacts for historical use of the Site (REC #8) and document surface soil condition in support of Site reuse	0-2	PID Screening Visual Olfactory	1	1 Field Duplicates 1 MS/MSD (metals only)	SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2- 8 oz amber glass	Absolute Resource Associates, Portsmouth, NH
	CA-SB-2	Surface soil	-To assess soil in the former garage where oil and hazardous materials were reportedly stored (REC #8)	0-2		1		SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2- 8 oz amber glass	
		Subsurface soil	-To assess soil beneath the former garage where oil and hazardous materials were reportedly stored (REC #8)	Area of greatest observed contamination or groundwater interface		1		VOC (EPA Method 8260) SVOCs (EPA Method 8270D) PCBs (EPA Method 8082) Priority pollutant metals (EPA Method 6010C & 7471B)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
	CA-SB-3	Subsurface soil	-To assess a possible release from fuel oil supply lines downgradient of the boiler room (REC #7) and Site impacts for historical use (REC #8)	Area of greatest observed contamination or groundwater interface		1		VOC (EPA Method 8260) SVOCs (EPA Method 8270D) TPH (EPA Method 8015C) Priority pollutant metals (EPA Method 6010C & 7471B)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
	CA-SB-4[4]	Subsurface soil	-To assess the industrial fill material (REC #1)	Observed fill interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
		Subsurface soil	-To assess the leaching of contaminants to native material beneath the fill or delineate the extent of contamination associated with the industrial waste fill (REC #1)	First encountered native material beneath the industrial waste fill[3]		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
		Subsurface soil	-To assess possible contamination migrating downgradient of the Stanley Mill (REC #4, REC #6, REC #8)	Area of greatest observed contamination or groundwater interface		1		VOC (EPA Method 8260) SVOCs (EPA Method 8270D)	1 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
	CA-SB-5[4]	Subsurface soil	-To assess the industrial fill material (REC #1)	Observed fill interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
		Subsurface soil	-To assess the leaching of contaminants to native material beneath the fill or delineate the extent of contamination associated with the industrial waste fill (REC #1)	First encountered native material beneath the industrial waste fill[3]		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
		Subsurface soil	-To assess possible contamination migrating downgradient of the Stanley Mill and the floor drain (REC #8)	Area of greatest observed contamination or groundwater interface		1		VOC (EPA Method 8260) SVOCs (EPA Method 8270D)	1 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
	CA-SB-6[4]	Subsurface soil	-To assess the industrial fill material (REC #1)	Observed fill interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
		Subsurface soil	-To assess the leaching of contaminants to native material beneath the fill or delineate the extent of contamination associated with the industrial waste fill (REC #1)	First encountered native material beneath the industrial waste fill[3]		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
		Subsurface soil	-To assess possible contamination migrating downgradient of the former Armory (REC #8)	Area of greatest observed contamination or groundwater interface		1		VOC (EPA Method 8260) SVOCs (EPA Method 8270D)	1 - 8 oz amber glass 1 - 40 mL VOA (methanol)	

Table 1: Sample Reference Table
Ferrari Realty Trust Property
93-119 Memorial Street, Franklin, New Hampshire

Media to be Collected	Proposed Sample IDs[2]	Sample Type	Sample Rational	Sample Depth (feet bgs)	Field Analysis/ Observations	No. of Samples for Analysis	QA/QC Samples	Analytical Method	Sample Container Information & Preservative (per location)*,[1]	Laboratory To be Used
Soil	CA-TP-1	Subsurface soil	-To assess the industrial fill material (REC #1)	Observed fill interval	PID Screening Visual Olfactory	1	1 Field Duplicates 1 MS/MSD (metals only)	VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	Absolute Resource Associates, Portsmouth, NH
		Subsurface soil	-To assess the leaching of contaminants to native material beneath the fill or delineate the extent of contamination associated with the industrial waste fill (REC #1)	First encountered native material beneath the industrial waste fill[3]		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
	CA-TP-2	Subsurface soil	-To assess the industrial fill material (REC #1)	Observed fill interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
		Subsurface soil	-To assess the leaching of contaminants to native material beneath the fill or delineate the extent of contamination associated with the industrial waste fill (REC #1)	First encountered native material beneath the industrial waste fill[3]		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
	CA-TP-3	Subsurface soil	-To assess the industrial fill material (REC #1)	Observed fill interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
		Subsurface soil	-To assess the leaching of contaminants to native material beneath the fill or delineate the extent of contamination associated with the industrial waste fill (REC #1)	First encountered native material beneath the industrial waste fill[3]		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass 1 - 40 mL VOA (methanol)	
	CA-SS-1	Surface soil	-To assess surface soil impacts from historical use of the Site (REC #8) and document surface soil condition in support of Site reuse	0-2		1		SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass	
	CA-SS-2	Surface soil	-To assess surface soil impacts near former loading dock (REC #8) and document surface soil condition in support of Site reuse	0-2		1				
	CA-SS-3	Surface Soil	-To assess surface soil impacts in areas of material handling (REC #8), assess possible impacts from LBP on the former Armory (Environmental Finding #1), and document surface soil condition in support of Site reuse	0-2		1				
	CA-SS-4	Surface Soil		0-2		1				

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Ferrari Realty Trust Property
93-119 Memorial Street, Franklin, New Hampshire

Media to be Collected	Proposed Sample IDs[2]	Sample Type	Sample Rational	Sample Depth (feet bgs)	Field Analysis/ Observations	No. of Samples for Analysis	QA/QC Samples	Analytical Method	Sample Container Information & Preservative (per location)*,[1]	Laboratory To be Used
Soil	CA-SS-5	Surface Soil	-To assess the discharge location of the floor drain from the Stanley Mill basement where materials were stored (REC #8)	0-2	PID Screening Visual Olfactory	1	1 Field duplicate 1 MS/MSD	SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass	Absolute Resource Associates, Portsmouth, NH
	CA-SS-6	Surface Soil	-To assess surface soil surrounding the stained former transformer pad (REC #4)	0-2		1		PCBs (EPA Method 8082)	1 - 8 oz clear glass	
	CA-SS-7	Surface soil	-To assess the metal dust discharged to surface soil in the area of the hopper (REC #6)	0-2		1		SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7471B) PCBs (EPA Method 8082)	2 - 8 oz amber glass	
	CA-SS-LBP-1 through CA-SS-LBP-6	Surface soil	Samples will be collected if the exterior of the Site building is found to contain LBP and if soil surrounding the Site building is screened to be impacted by lead. Samples will be biased towards the locations of highest XRF screening values to assess impacts to soil from possible chipping of LBP from the Site building (Environmental Finding #1)	0-0.5	XRF Screening	6		Lead (EPA Method 6010C)	1 - 8 oz clear glass	
				0.5-1		6				
Groundwater	CA-MW-1		-To document upgradient groundwater conditions at the Site	Screened interval	pH, ORP, DO, specific conductivity, turbidity, temperature	1	1 Field duplicate 1 MS/MSD	VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7470A)	2 - 1L amber jars 1 - 40 mL VOAs (HCl) 1 - 500 mL poly (HNO3)	
	CA-MW-2		-To assess groundwater beneath the former garage where oil and hazardous materials were reportedly stored (REC #8)	Screened interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7470A)	2 - 1L amber jars 1 - 40 mL VOAs (HCl) 1 - 500 mL poly (HNO3)	
	CA-MW-3		-To assess a possible release from fuel oil supply lines downgradient of the boiler room (REC #7) and groundwater impacts from historical Site use (REC #8)	Screened interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7470A)	2 - 1L amber jars 1 - 40 mL VOAs (HCl) 1 - 500 mL poly (HNO3)	
	CA-MW-4		-To assess groundwater downgradient of the Stanley Mill and within the industrial waste fill that may be entering the Winnipesaukee River (REC #1, REC #4, REC #6, REC #8)	Screened interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7470A)	2 - 1L amber jars 1 - 40 mL VOAs (HCl) 1 - 500 mL poly (HNO3)	
	CA-MW-5		-To assess groundwater downgradient of the Stanley Mill that may have been impacted by discharges to the Stanley Mill floor drain (REC #8)	Screened interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7470A)	2 - 1L amber jars 1 - 40 mL VOAs (HCl) 1 - 500 mL poly (HNO3)	
	CA-MW-6		-To assess groundwater downgradient of the former Armory and within the industrial waste fill that may be entering the Winnipesaukee River (REC #1 and REC #8)	Screened interval		1		VOCs (EPA Method 8260B) SVOCs (EPA Method 8270D) Priority pollutant metals (EPA Method 6010C & 7470A)	2 - 1L amber jars 1 - 40 mL VOAs (HCl) 1 - 500 mL poly (HNO3)	

Table 1: Sample Reference Table
Ferrari Realty Trust Property
93-119 Memorial Street, Franklin, New Hampshire

Media to be Collected	Proposed Sample IDs[2]	Sample Type	Sample Rational	Sample Depth (feet bgs)	Field Analysis/ Observations	No. of Samples for Analysis	QA/QC Samples	Analytical Method	Sample Container Information & Preservative (per location)*,[1]	Laboratory To be Used
Building Materials	CA-CC-1	Concrete	-To assess the area of staining at the southern end of the former Armory building (REC #8)	0 to 0.5-inches	Visual	1	2 Field Duplicates (1 caulk/sealant/ adhesive type material and 1 paint)	PCBs (EPA Method 8082 with soxhlet extraction)	1 - 4 oz glass	Absolute Resource Associates, Portsmouth, NH
	CA-CC-2	Concrete	-To assess the area of staining surround the turbine in the basement of the Site building (REC #8)			1				
	CA-CC-3	Concrete	-To assess the area of staining observed on the former transformer pad near the northeast corner of the Stanley Mill (REC #4)			1				
	CA-PCB-1 through CA-PCB-12	Building materials	-To assess for the presence of PCB containing building materials in the Site buildings (Environmental Finding #1). Data will be used to properly manage building materials during Site renovations.	NA		12				
Asbestos	CA-PACM-1(A-C) through CA-PACM-25(A-C)	Bulk Materials	-Three samples will be collected from each suspected asbestos-containing material (REC #3, Environmental Finding #1)	NA	Visual	25	Triplicate Sampling	Polarized Light Microscopy EPA 600/R-93/116	Plastic zipper bags	EMSL Analytical, Inc., South Portland, ME

Notes:

1 - All samples will be chilled to 4°C (+/- 2°C) and submitted to the laboratory on ice.

2 - If contamination is identified in the UST excavation, CA-UST-1 and CA-UST-2 will be collected from the areas of greatest PID response. If no contamination is encountered one composite sample (CA-CompUST-1) consisting of aliquots from the four sidewalls and base of the excavation will be collected. If no UST is encountered, one grab sample will be collected from the end point of the piping (CA-Pipe-1).

3 - Samples will be placed on hold pending the results of the above fill material samples. If results of the fill material samples exceed applicable regulatory standards, the associated native sample will be authorized for analysis in an effort to delineate the extent of fill.

4 - If the first encountered native material is within the same interval as the water table and no evidence of contamination is identified, or within the same interval as the area of greatest observed contamination, only two samples will collected: duplicate samples will not be collected from the same interval.

* - Additional details regarding analytical method, sample preservation, sample volume, and hold times can be found in Appendix D of Credere's Generic Maine QAPP.

"greatest observed contamination" shall be defined as the interval of highest PID response, visual staining, or sheens.

MS/MSD - Matrix Spike/Matrix Spike Duplicate
NA - not applicable
bgs - below ground surface
DO - Dissolved Oxygen
ORP - Oxidation-reduction potential
SVOCs - semi-volatile organic compounds
VOC - volatile organic compounds
Priority pollutant metals: Sb, As, Be, Cd, Cr, Cu, Pb, Hg, Ni, Se, Ag, Ti, Zn
PCB - polychlorinated biphenyl
XRF-X-ray fluorescence meter

Table 2: Standard Operating Procedure (SOP) Reference Table
Ferrari Realty Trust Property
93-119 Memorial Street, Franklin, New Hampshire

Field SOPs

SOP	SOP Description	Date
Credere-001	SOP for Field Measurement of Groundwater Level	March 2008
Credere-002	SOP for Geoprobe Sampling	38991
Credere-003	SOP for Test Pit Sampling	October 2006
Credere-004	SOP for Log Book Entries	October 2006
Credere-007	SOP for EM and GPR Surveys (SOP by: Northeast Geophysical Services)	October 2006
Credere-009	SOPs for Typical Asbestos Bulk and Air Sampling (SOP by: Environmental Safety & Hygiene Associates, Inc.)	NA
HWRB-1	Water Level Measurements, Revision 2	December 2011
HWRB-2	Calculation of Purge Volume, Revision 1	January 2012
HWRB-9	Low Flow Groundwater Purging and Sampling, Revision 5	January 2012
HWRB-11	Soil Sampling, Revision 1	January 2012
HWRB-12	Jar headspace Technique for Field Screening Soil Samples, Revision 2	January 2012
HWRB-15	Decontamination, Revision 3	January 2012
HWRB-17	Calibration of Field Instruments, Revision 4	January 2012
HWRB-18	Chain of Custody, Sample Handling & Shipping, Revision 2	January 2012
RWM-DR-025	Protocol for Collecting Data Using an Innov-X Field Portable X-Ray Fluorescence Spectrometer for Certain metals in Solid Media (Included in Appendix B)	Febraury 29, 2009
EPASOP#2048	Monitoring Well Installation	March 18, 1996
EIASOP_POROUSSAMPLING1	Standard Operating Procedure for Sampling Porous Surfaces for PCBs	May 5, 2011
EIASOP_SOILSAMPLING2	Standard Operating Procedure for Soil, Sediment and Solid Waste Sampling	Rev #2, February 13, 2004
EPA 600/R-93/116	Method for the Determination of Asbestos in Bulk Building Materials	July 1993
EQAGUI-DO	Quality Assurance Bulletin for Calibration of Dissolved Oxygen Meters	February 2006
EQASOP_FieldCalibrat	Standard Operating Procedure Calibration of Field Instruments	Rev #2, January 19, 2010
EQASOP-GW 001	Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells	Rev #3, January 19, 2010

Laboratory SOPs

SOP	SOP Description	Date
EMSL: PLM SOP	Polarized Light Microscopy	November 12, 2010
RL-4	Analysis of Polychlorinated Biphenyls in Soil and Water Extracts by EPA 8082	January 2013
RL-5	Trace Metals by ICP EPA 200.7/6010C	January 2013
RL-6	Mercury Analysis by Cold Vapor Methods 245.1, 7470A/7471B	January 2013
RL-7	Analysis of Diesel Range Organics in Extracts of Soil and Water by Method 8015 and Method 8100	August 2011
RL-9	Analysis of VOCs in Water and Solid Samples by EPA Method 8260B	June 2012
RL-12	Preparation and analysis of PAHs, Base/Neutrals, and Acids by EPA Method 8270D	August 2011
RL-28	Soxhlet Extraction by EPA method 3540C	August 2011

APPENDIX A

Analytical Sensitivity and Project Criteria Tables

As of the date of this SSQAPP Addendum, the current state and/or federal standards have been reviewed for accuracy.

VOCs in Soil by EPA Method 8260C

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
1,1,1,2-tetrachloroethane	0.1	0.8
1,1,1-trichloroethane	0.1	78
1,1,2,2-tetrachloroethane	0.1	4
1,1,2-trichloroethane	0.1	0.1
1,1-dichloroethane	0.1	3
1,1-dichloroethe	0.1	2
1,1-dichloropropene	0.1	NE
1,2,3-trichlorobenzene	0.1	4.9
1,2,3-trichloropropane	0.1	0.2
1,2,4-trichlorobenzene	0.1	19
1,2,4-trimethylbenzene	0.1	130
1,2-dibromo-3-chloropropane (DBCP)	0.1	0.1
1,2-dibromoethane (EDB)	0.1	0.1
1,2-dichlorobenzene	0.1	88
1,2-dichloroethane	0.1	0.1
1,2-dichloropropane	0.1	0.1
1,3,5-trichlorobenzene	0.1	340
1,3,5-trimethylbenzene	0.1	96
1,3-dichlorobenzene	0.1	150
1,3-dichloropropane	0.1	160*
1,4-dichlorobenzene	0.1	7
1,4-dioxane	2	5
2,2-dichloropropane	0.1	NE
2-butanone (MEK)	0.3	51
2-chlorotoluene	0.1	15
2-hexanone	0.5	20
4-chlorotoluene	0.1	2,400
4-isopropyltoluene	0.1	3,400
4-methyl-2-pentanone (MIBK)	0.4	29
acetone	2	75
benzene	0.1	0.3
bromobenzene	0.1	6.2
bromochloromethane	0.1	8.3
bromodichloromethane	0.1	0.1
bromoform	0.1	0.1
bromomethane	0.2	0.3
carbon disulfide	0.1	460
carbon tetrachloride	0.1	12
chlorobenzene	0.1	28
chloroethane	0.1	NE
chloroform	0.1	0.73
chloromethane	0.1	3
cis-1,2-dichloroethene	0.1	NE
cis-1,3-dichloropropene	0.1	NE
dibromochloromethane	0.1	1
dibromomethane	0.1	25*
dichlorodifluoromethane	0.1	1,000
diethyl ether	0.1	3,900
diisopropyl ether (DIPE)	0.1	10
ethyl t-butyl ether (ETBE)	0.1	0.7

VOCs in Soil by EPA Method 8260C

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
ethylbenzene	0.1	140
hexachlorobutadiene	0.1	7
isopropylbenzene	0.1	330
m&p-xylenes	0.1	500**
methyl t-butyl ether (MTBE)	0.1	0.2
methylene chloride	0.1	0.1
naphthalene	0.1	5
n-butylbenzene	0.1	110
n-propylbenzene	0.1	85
o-xylene	0.1	500**
sec-butylbenzene	0.1	130
styrene	0.1	17
t-amyl-methyl ether (TAME)	0.1	3
t-butanol (TBA)	2	2
tert-butylbenzene	0.1	100
tetrachloroethene (ethylene, PCE)	0.1	2
tetrahydrofuran (THF)	0.5	200
toluene	0.1	100
trans-1,2-dichloroethene (ethylene)	0.1	9
trans-1,3-dichloropropene	0.1	NE
trichloroethene (TCE)	0.1	0.8
trichlorofluoromethane	0.1	1,000
vinyl chloride	0.1	1

Notes:

All values are in mg/kg.

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards and Appendix E, Method 1 Soil Standards from NHDES Risk Characterization and Management Policy, unless marked with an *.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed May 2014). Regional Screening Levels for Chemical Contaminants at Superfund Sites (Residential Soil). http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

** NDHES mixed isomer standard.

NE = Regulatory guideline not established

VOCs in Groundwater by EPA Method 8260C

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standards[1]	
		AGQS and GW-1	GW-2
1,1,1,2-tetrachloroethane	2	70	NE
1,1,1-trichloroethane	2	200	27,000
1,1,2,2-tetrachloroethane	2	2	120
1,1,2-trichloroethane	2	5	20
1,1-dichloroethane	2	81	130
1,1-dichloroethene	1	7	630
1,1-dichloropropene	2	NE	NE
1,2,3-trichlorobenzene	2	0.7*	NE
1,2,3-trichloropropane	2	40	NE
1,2,4-trichlorobenzene	2	70	150
1,2,4-trimethylbenzene	2	330	1,300
1,2-dibromo-3-chloropropane (DBCP)	2	0.2	NE
1,2-dibromoethane (EDB)	2	0.05	NE
1,2-dichlorobenzene	2	600	14,000
1,2-dichloroethane	2	5	50
1,2-dichloropropane	2	5	50
1,3,5-trichlorobenzene	2	40	NE
1,3,5-trimethylbenzene	2	330	NE
1,3-dichlorobenzene	2	600	NE
1,3-dichloropropane	2	37*	NE
1,4-dichlorobenzene	2	75	80
1,4-dioxane	50	3	NE
2,2-dichloropropane	2	NE	NE
2-butanone (MEK)	10	4,000	50,000
2-chlorotoluene	2	100	NE
2-hexanone	10	3.8*	NE
4-chlorotoluene	2	25*	NE
4-isopropyltoluene	2	260	NE
4-methyl-2-pentanone (MIBK)	10	2,000	NE
acetone	50	6,000	NE
benzene	2	5	2,900
bromobenzene	2	6,2	NE
bromochloromethane	2	8.3	NE
bromodichloromethane	0.6	0.6	NE
bromoform	2	4	2,800
bromomethane	2	10	10
carbon disulfide	2	70	NE
carbon tetrachloride	2	0.2	10
chlorobenzene	2	7.8	1,500
chloroethane	2	NE	NE
chloroform	2	70	70
chloromethane	2	30	NE
cis-1,2-dichloroethene	2	70	NE
cis-1,3-dichloropropene	2	NE	NE
dibromochloromethane	2	60	NE

VOCs in Groundwater by EPA Method 8260C

Analyte	Laboratory Practical	Regulatory Standards[1]	
dibromomethane	2	0.8	NE
dichlorodifluoromethane	2	1,000	NE
diethyl ether	5	1,400	NE
ethyl t-butyl ether (ETBE)	2	40	NE
ethylbenzene	2	700	1,500
hexachlorobutadiene	0.5	0.5	NE
diisopropyl ether (DIPE)	2	120	NE
isopropylbenzene	2	800	NE
m&p-xylenes	2	10,000**	17,000
methyl t-butyl ether (MTBE)	2	13	2,600
methylene chloride	5	5	24,000
naphthalene	5	20	1,700
n-butylbenzene	2	260	NE
n-propylbenzene	2	260	NE
o-xylene	2	10,000**	17,000
sec-butylbenzene	2	260	NE
styrene	2	100	43,00
t-amyl-methyl ether (TAME)	2	140	NE
t-butanol (TBA)	30	40	NE
tert-butylbenzene	2	260	NE
tetrachloroethene	2	5	240
tetrahydrofuran (THF)	10	154	NE
toluene	2	1,000	50,000
trans-1,2-dichloroethene	2	100	560
trans-1,3-dichloropropene	2	NE	NE
trichloroethene	2	5	20
trichlorofluoromethane	2	2,000	NE
vinyl chloride	2	2	4

Notes:

All values are in ug/L.

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards and Table 2, Method 1 Groundwater Standards for NHDES Risk Characterization and Management Policy (Section. 7.4(4)), unless marked with an *.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed May 2014). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

** - NHDES mixed isomer NOTE

NHDES mixed isomer standard.

NE = Regulatory guideline not established.

TPH in Solids by EPA Method 8100		
Analyte	**Laboratory Practical Quantitation Limit**	**Regulatory Standard[1]**
Total Petroleum Hydrocarbons	200	10,000

Notes:
All values are in mg/kg.
PQLs from Absolute Resource Associates of Portsmouth, New Hampshire
1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards and Appendix E, Method 1 Soil Standards from NHDES Risk Characterization and Management Policy, unless marked with an *.

SVOC in Soil by EPA Method 8270D

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
1,2,4-trichlorobenzene	0.5	19
1,2-dichlorobenzene	0.2	88
1,3-dichlorobenzene	0.2	150
1,4-dichlorobenzene	0.2	7
2,4,5-trichlorophenol	0.2	24
2,4,6-trichlorophenol	0.2	0.7
2,4-dichlorophenol	0.5	0.7
2,4-dimethylphenol	0.2	4
2,4-dinitrophenol	5	0.7
2,4-dinitrotoluene	0.2	0.7
2,6-dinitrotoluene	0.2	0.36
2-chloronaphthalene	0.5	NE
2-chlorophenol	0.5	2
2-methylnaphthalene	0.05	96
2-methylphenol	0.2	0.9
2-nitroaniline	0.2	61
2-nitrophenol	0.2	NE
3,3'-dichlorobenzidine	3	0.7
3-nitroaniline	0.2	NE
4,6-dinitro-2-methylphenol	2	4.9*
4-bromophenyl phenyl ether	0.2	NE
4-chloro-3-methylphenol	0.2	6,100*
4-chloroaniline	0.2	1.3
4-chlorophenyl phenyl ether	0.5	NE
4-methylphenol	0.2	0.7
4-nitroaniline	0.5	25
4-nitrophenol	2	NE
acenaphthene	0.05	340
acenaphthylene	0.05	490
aniline	0.2	43
anthracene	0.05	1000
azobenzene	0.2	5,6
benzidine	3	0.004
benzo(a)anthracene	0.05	1
benzo(a)pyrene	0.05	0.7
benzo(b)fluoranthene	0.05	1
benzo(g,h,i)perylene	0.05	960
benzo(k)fluoranthene	0.05	12
benzoic acid	5	350
benzyl alcohol	0.2	620
bis(2-chloroethoxy)methane	0.2	18
bis(2-chloroethyl)ether	0.2	0.7
bis(2-chloroisopropyl) ether	0.2	5
bis(2-ethylhexyl)phthalate	0.5	72
butyl benzyl phthalate	0.5	280
carbazole	0.2	NE
chrysene	0.05	120
dibenzo(a,h)anthracene	0.05	0.7
dibenzofuran	0.05	0.72

SVOC in Soil by EPA Method 8270D

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
diethyl phthalate	0.5	1000
dimethylphthalate	0.5	700
di-n-butylphthalate	0.5	2,600
di-n-octyl phthalate	0.5	NE
fluoranthene	0.05	960
fluorene	0.05	77
hexachlorobenzene	0.2	0.8
hexachlorobutadiene	0.2	7
hexachlorocyclopentadiene	1	200
hexachloroethane	0.2	0.7
indeno(1,2,3-cd)pyrene	0.05	1
isophorone	0.5	1
naphthalene	0.05	5
nitrobenzene	0.2	5.1
N-nitrosodimethylamine	0.2	0.024
N-nitroso-di-N-propylamine	0.2	0.076
N-nitrosodiphenylamine	0.2	0.19
pentachlorophenol	1	3
phenanthrene	0.05	960
phenol	0.2	56
pyrene	0.05	720

Notes:

All values are in mg/kg.

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards and Appendix E, Method 1 Soil Standards from NHDES Risk Characterization and Management Policy, unless marked with an *.

NE = Regulatory guideline not established

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed May 2014). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

SVOC in Groundwater by EPA Method 8270D

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]	
		AGQS and GW-1	GW-2
1,2,4-trichlorobenzene	5	70	150
1,2-dichlorobenzene	2	600	14,000
1,3-dichlorobenzene	2	600	NE
1,4-dichlorobenzene	2	75	80
2,4,5-trichlorophenol	2	700	NE
2,4,6-trichlorophenol	2	5	NE
2,4-dichlorophenol	5	21	NE
2,4-dimethylphenol	2	140	NE
2,4-dinitrophenol	50	14	NE
2,4-dinitrotoluene	2	10	NE
2,6-dinitrotoluene	2	0.048	NE
2-chloronaphthalene	5	550*	NE
2-chlorophenol	5	35	NE
2-methylnaphthalene	0.5	280	NE
2-methylphenol	2	40	NE
2-nitroaniline	2	19	NE
2-nitrophenol	2	NE	NE
3,3'-dichlorobenzidine	30	1.3	NE
3-nitroaniline	2	NE	NE
4,6-dinitro-2-methylphenol	20	1.2*	NE
4-bromophenyl phenyl ether	2	NE	NE
4-chloro-3-methylphenol	2	1,100*	NE
4-chloroaniline (p-)	2	28	NE
4-chlorophenyl phenyl ether	5	NE	NE
4-methylphenol	2	40	NE
4-nitroaniline	5	3.8	NE
4-nitrophenol	10	NE	NE
acenaphthene	0.5	420	NE
acenaphthylene	0.5	420	NE
aniline	2	12*	NE
anthracene	0.5	2100	NE
azobenzene	2	0.12	NE
benzidine	30	0.8	NE
benzo(a)anthracene	0.5	0.1	NE
benzo(a)pyrene	0.2	0.2	NE
benzo(b)fluoranthene	0.5	0.1	NE
benzo(g,h,i)perylene	0.5	210	NE
benzo(k)fluoranthene	0.5	0.5	NE
benzoic acid	50	28,000	NE
benzyl alcohol	2	200	NE
bis(2-chloroethoxy)methane	5	5.9	NE
bis(2-chloroethyl)ether	2	10	NE
bis(2-chloroisopropyl) ether	2	300	NE
bis(2-ethylhexyl)phthalate	5	6	NE
butyl benzyl phthalate	5	14*	NE
carbazole	2	NE	NE
chrysene	0.5	5	NE
dibenzo(a,h)anthracene	0.5	0.1	NE
dibenzofuran	0.5	0.79	NE
diethyl phthalate	5	1,500	NE

SVOC in Groundwater by EPA Method 8270D

Analyte	Laboratory Practical	Regulatory Standard[1]	
dimethylphthalate	5	50,000	NE
di-n-butylphthalate	5	2,600	NE
di-n-octyl phthalate	2	20	NE
fluoranthene	0.5	280	NE
fluorene	0.5	280	NE
hexachlorobenzene	2	1	NE
hexachlorobutadiene	2	0.5	NE
hexachlorocyclopentadiene	10	50	NE
hexachloroethane	2	1	NE
indeno(1,2,3-cd)pyrene	0.5	0.1	NE
isophorone	5	100	NE
naphthalene	0.5	20	1,700
nitrobenzene	2	0.14	NE
N-nitrosodimethylamine	2	0.00049*	NE
N-nitroso-di-N-propylamine	2	0.011	NE
N-nitrosodiphenylamine	2	12	NE
pentachlorophenol	10	1	NE
phenanthrene	0.5	210	NE
phenol	2	4000	NE
pyrene	0.5	210	NE

Notes:

All values are in ug/L.

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards and Table 2, Method 1 Groundwater Standards for NHDES Risk Characterization and Management Policy (Section. 7.4(4)), unless marked with an *.

* United States Environmental Protection Agency Regions 3, 6, and 9. (accessed May 2014). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

PCBs in Soil by EPA Method 8082A

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
PCB-1016	0.2	1 (Total)
PCB-1221	0.2	
PCB-1232	0.2	
PCB-1242	0.2	
PCB-1248	0.2	
PCB-1260	0.2	

Notes:

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards and Appendix E, Method 1 Soil Standards from NHDES Risk Characterization and Management Policy.

All concentrations in mg/kg

NE = Regulatory guideline not established

PCBs in Building Materials by EPA Method 8082

Analyte	**Laboratory Practical Quantitation Limit**	**Regulatory Standard (40 CFR 761.3)**
PCB-1016	0.2	50 (Total)
PCB-1221	0.2	
PCB-1232	0.2	
PCB-1242	0.2	
PCB-1248	0.2	
PCB-1254	0.2	
PCB-1260	0.2	

Notes:
All values are in mg/kg.

Hg in Soil by EPA Methods 7471B

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
Mercury	0.02	6

Notes:

All values are in mg/kg.

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards and Appendix E, Method 1 Soil Standards from NHDES Risk Characterization and Management Policy.

Hg in Groundwater by EPA Methods 7470A

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
Mercury	0.2	2

Notes:

All values are in ug/L.

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire

[1] - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards and Table 2, Method 1 Groundwater Standards for NHDES Risk Characterization and Management Policy (Section. 7.4(4)).

Asbestos in Solids by PLM by EPA Method 600/R		
Analyte	**Laboratory Practical Quantitation Limit**	**Regulatory Standard[1]**
Asbestos	0.20%	1%

Notes:

1 - New Hampshire Department of Environmental Services Chapter 1800: Asbestos Management Control, October 21, 2008.
PQL from EMSL of Cinnamonsin, New Jersey

Metals in Soil by EPA Method 6010C

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
Antimony	0.3	9
Arsenic	0.5	11
Beryllium	0.2	1
Cadmium	0.2	33
Chromium (III)	2	1,000
Chromium (VI)	2	130
Copper	2	310
Lead	0.5	400
Nickel	2	400
Selenium	2	180
Silver	0.4	89
Tin	2.5	4,700
Zinc	2	1,000

Notes:

All values are in mg/kg.

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards and Appendix E, Method 1 Soil Standards from NHDES Risk Characterization and Management Policy, unless marked with an *.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed May 2014). Regional Screening Levels for Chemical Contaminants at Superfund Sites (Residential Soil). http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

Metals in Groundwater by EPA Method 6010C

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard[1]
Antimony	6	6
Arsenic	8	10
Beryllium	4	4
Cadmium	4	5
Chromium (TOTAL)	50	100
Copper	50	1,300
Lead	8	15
Nickel	50	100
Selenium	50	50
Silver	7	100
Tin	50	1,200
Zinc	50	600

Notes:

All values are in ug/L.

PQLs from Absolute Resource Associates of Portsmouth, New Hampshire
1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards for groundwater, unless marked with an *.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed May 2014). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

** - The chromium VI standard was used because it is the lowest and most conservative chromium standard.

APPENDIX B

Maine DEP Protocol for Collecting Data Using an Innov-X Field Portable X-Ray Fluorescence Spectrometer for Certain Metals in Solid Medium

COVERSHEET
STANDARD OPERATING PROCEDURE

Operation Title: **PROTOCOL FOR COLLECTING DATA USING AN INNOV-X FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETER FOR CERTAIN METALS IN SOLID MEDIA**

Originator:
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Quality Assurance Coordinator
Division of Remediation
Bureau of Remediation and Waste Management

Standard Operating Procedure: **DRWM-DR-025**
Revision: **01**
Date: **February 20, 2009**
Written by: **Jean Firth**
Reviewed by: **Brian Beneski**

Five Year Review No Changes Needed:

Print Name: ________ Signature: ________ Date: ________

Print Name: ________ Signature: ________ Date: ________

Print Name: ________ Signature: ________ Date: ________

Print Name: ________ Signature: ________ Date: ________

1.0 PURPOSE

The purpose of this document is to describe the Maine Department of Environmental Protection, Bureau of Remediation and Waste Management, Division of Remediation's (MEDEP/DR) procedure for collecting data using a Innov-X portable x-ray fluorescence spectrometer (XRF) for certain metals in solid media, paint and dust wipe samples.

2.0 APPLICABILITY

MEDEP/DR is responsible for the investigation and subsequent remediation of hazardous substance, petroleum, and landfill Sites throughout Maine. Part of the investigation of these sites is analysis of samples for metals; the XRF is a useful tool in providing this analysis. This SOP applies to all staff that may use the XRF.

3.0 RESPONSIBILITIES

All MEDEP/DR Staff must follow this procedure when using the Innov-X XRF. All managers and supervisors within MEDEP/DR are responsible for ensuring that their staff are familiar with and adhere to this procedure. *Additionally, before any person is allowed to use the Innov-Xx XRF they* ***MUST:*** *have completed a radiation training course (proof of completion must be submitted to the DR Radiation Safety Officer (RSO)), wear a radiation dosimeter badge and have 8 hours of supervised field use with the instrument by approved Division of Remediation staff.* Safety procedures are described in detail in DR SOP #24 Safety Protocol for Use of the Innov-X X-Ray Fluorescence Metals Analyzer.

A current list of qualified supervisors and operators will be maintained by the DR RSO.

4.0 INTRODUCTION

This standard operating procedure (SOP) is designed to be a guideline for data collection with Innov-X XRF for solid media (e.g. soil, sediment and sludge), lead in painted surfaces and dust wipe samples. This is a field screening method used for: profiling an area, locating sources of contamination, determining the horizontal or vertical extent of contamination or collecting preliminary data that will be used to design a sampling plan. Samples can be analyzed either in-situ methods or by intrusive sample preparation methods. This SOP will outline collecting data using both methods.

5.0 PREPARATION AND GENERAL INFORMATION

5.1 PREPARATION

Prior to conducting any sampling event, a sampling plan should be developed (see SOP DR#014 - Development of a Sampling and Analysis Plan). Clean containers must be used for each sampling event unless in-situ sampling is to be performed.

An evaluation of the site and the elements of concern should be made prior to using the XRF on a site. Then determine if the XRF can analyze for the elements of concern and if the detection limits are acceptable to meet the Data Quality Objectives for the project.

Before sampling, a decision must be made whether to test the material:

- in-situ (in-place),
- as bagged samples (or for sludge, in cups) with a minimum of preparation, or
- in an XRF cup after preparation as described in Section 5.4.

If the primary objective of the sampling event is to determine whether an element is present (rather than accurately measuring how much is present), in-situ or bagged samples are the quickest, simplest way to proceed. (Note: Preparing a sample by drying, milling and sieving will yield greater accuracy.) Even if the objective is to collect samples and prepare them prior to analysis, preliminary direct measurements can help to survey the site.

5.2 EQUIPMENT

Equipment required for this SOP may include:

-- **XRF** – Innov-X X-Ray Fluorescence Spectrum Analyzer
  a) XRF
  b) Batteries and charger
  c) standardization clip
  d) Sample test stand
  e) in-situ sample test stand
  f) standards
  g) Grinder
  h) Mortar and pestle
  i) various size sieves

-- **Sampling implements** - This includes shovels, Geoprobe® soil boring system, dredges, etc, as outlined in the site specific sampling plan. Please refer to the following MEDEP/DR SOPs for using this equipment:
  -- DR#004 - Sampling Surface Water and Sediment
  -- DR#006 - Soil Sampling
  -- DR#007 - Soil Sampling with a Geoprobe Large Bore Sampler

-- **Sample containers** – Whirl pack bags, zipper locking bags or sample cups.
-- **Radiation dosimeter** – must be worn by any department staff using the XRF.

5.3 GENERAL INFORMATION

5.3.1 Radiation Sources

The Innov-X XRF does not contain a radioactive source, which would constantly emit ionizing radiation. The Innov-X has an x-ray tube which can only emit ionizing radiation when the instrument is powered. The instrument will not power the x-ray tube without the battery or handheld computer installed.

5.3.2 Radiation License and Training Requirements

The Innov-X XRF is registered with the Department of Human Services. Only staff who have completed radiation training and are issued a radiation dosimeter badge may use the XRF. Additionally, staff using the XRF must have 8 hours of supervised field use by approved DR Oil and Hazardous Materials Specialists (OHMS).

5.3.3 Detection Limits

An element will only be shown as detected by the XRF if the measured concentration of the sample is at least three times the standard deviation of the measurement. This detection limit will depend on the composition of the sample.

Detection limits depend on several factors: the analyte of interest, times the sample is irradiated, physical matrix effects, chemical matrix effects, and interelement spectral interferences. For more of an explanation of detection limits see Attachment A “EPA Method 6200”. Detected elements are displayed as in the Measurement screen. Non-detected elements are shown as **<** **xx**, where **xx** is the detection limit for that sample. The detection limit for each element is calculated from each sample.

5.3.4 Interferences

Physical matrix interferences result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition.

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.

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.

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.

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.

5.3.5 Precision

The measurement precision for each element displayed appears to the right of the measured concentration, under the heading "**+-**". The **precision** of each measurement is three times the standard deviation.

5.3.6 Maintenance

If there are any problems with how the XRF is working, stop using the instrument and report the problem to the DR's SASS. **Do not attempt to fix the XRF yourself.** Opening the instrument may expose the user to the radiation and will void the warrantee.

5.4 GENERAL PROCEDURE FOR OPERATING THE INNOV-X XRF

Refer to the attached Innov-X User Manual for additional information and figures showing the features of the instrument (Attachment B).

5.4.1 Place a battery in the unit and install the iPAQ. Turn on both the iPAQ (top left hand side) and the XRF (back of the unit).

5.4.2 Make sure the date and time are set correctly on the iPAQ. Data is stored on the instrument by date.

5.4.3 On the iPAQ drop down menu, located at the top left hand side of the screen, choose Innov-X. Note the red light on the end of the instrument will be on when the instrument is on and ready for use. It will flash once the trigger is pulled which indicates the instrument is emitting radiation.

5.4.4 Choose the test mode (soil, paint or dust wipe) from the menu.

5.4.5 The instrument will require you to perform the standardization test at this point. The instrument will not operate without passing this test. Place the standardization clip securely over the sample window, and tap the instruction box on the screen. A small red light on the end of the XRF will begin to flash. This indicates the instrument is operating and emitting radiation. This test will take approximately 1 minute. ***KEEP ALL YOUR BODY PARTS AWAY FROM THE END OF THE INSTRUMENT. MAKE SURE THE INSTRUMENT IS NOT POINTED AT ANYONE AT ANYTIME.*** **All reasonable measures, including labeling, and the concepts of time, distance and shielding should be implemented to limit radiation exposure to as low as reasonably achievable (ALARA).**

Once the standardization is complete, the results will be shown on the screen. If the resolution result is within tolerance limits proceed to the next step. Otherwise run the standardization test again. If the test fails again, turn off the instrument and try again. If the instrument fails a third time you will be prompted to perform a soft restart on the iPAQ. If this fails replace the battery and try again. If you still do not pass call Innov-X customer support (781-938-5005).

5.4.6 Once the instrument has passed the standardization you are ready to begin testing samples.

5.4.7 A padlock icon is also shown on the bottom of the screen. This indicates if the software has been locked or is ready to test. The software will automatically lock when the instrument has not been used for several minutes. This will prevent anyone from inadvertently activating the instrument. To unlock the software, tap on the icon.

5.4.8 If you will be sampling in the soil mode see section 5.0 Soil Sampling and Analysis Procedure below.

6.0 SOIL SAMPLING AND ANALYSIS PROCEDURE

6.1 SOIL ANALYSIS MODEL

6.1.1 After completing the procedure described in section 5.4 there are two buttons shown on the bottom of the touch screen "Start" and "Info". Tap on the Info button to enter information specific to the samples you are analyzing. In soil mode there are preset options such as Operator, Sample method, Sample Number, Sample Depth and Comment. These can be customized to projects when necessary. Fill in the information for the sample before analysis. The analysis will be stored with this information. You need to change the information prior to each sample that is run.

6.1.2 The bottom menu on the screen shows 4 options: File, Edit, View, Options and Help. From these menus the operator can change the settings for the method of analysis (Standard or LEAP) and the time interval for testing. For a complete description of these menus and how to change the settings, see Attachment B.

6.1.3 To begin testing a sample the operator either taps the start button at the bottom of the screen or pulls the trigger. *Note: the software lock may have to be disabled if the instrument has not be used for more than 5 minutes.*

6.1.4 Check the XRF's calibration with testing standards before using the XRF to analyze samples, use standards that are closest to the levels of elements that are expected onsite. Recheck the standards at least once per hour during testing and after analysis has been completed for the day.

EPA Method 6200 <u>Field Portable X-Ray Fluorescence spectrometry for the determination of elemental concentrations in soil and sediment</u> (Attachment C) provides additional information regarding acceptable testing procedures and may be used in place of the procedure described below.

6.2 IN-SITU ANALYSIS

6.2.1 Clear the area selected for analysis of any surface debris or vegetation. Level the area so the XRF sample window will contact the area evenly. Keep in mind that a finer and more homogeneous material will yield more accurate the results. Increased accuracy can be obtained by loosening the soil and letting it dry in the sun before testing.

6.2.2 Hold the XRF on the ground and pull the trigger or place the XRF in the in-situ test stand and pull the trigger. The stand will allow the instrument to stand on its own. If the deadman trigger lock is engaged the trigger must be held for the duration of the analysis.

If the deadman trigger has been disengaged then the analysis will run for the preset time period. The test can be stopped by pulling the trigger again.

Warning: Always treat radiation with respect. Do not put your hand or any other body part on or near the sample window of the XRF while samples are being analyzed. Never point the XRF at yourself or anyone else. ALARA objectives must be considered whenever staff are using an XRF.

The operator is responsible for controlling access in the area in which the XRF is being used. When possible use signs, barricades or caution tape to restrict access. Never allow anyone to enter within 5 feet of the x-ray path.

6.2.3 Watch the results on the display screen to decide when the test has reached the desired level of accuracy or let the analysis run for the allotted time. NOTE: if the instrument is set to run both standard and LEAP analysis consecutively and the test is ended during the standard analysis mode and before the LEAP analysis has begun your data will not be stored.

6.3 IN-SITU DEPTH PROFILING

An in-situ XRF soil test examines only the top few millimeters of soil. To profile the depth of contamination, remove a vertical slice of soil and test several samples from different depths.

6.4 ANALYSIS OF BAGGED SOLID SAMPLES

Depending on the data quality objectives for your site it may be convenient to screen samples collected in plastic bags and analyze them without preparation. Because samples are tested through a bag, test results will tend to be 5-10% lower than test results obtained from direct analysis.

6.4.1 Place 50-100 grams of sample in a clean whirl pack or zipper locking bag. Remove any large stones or debris. Keep in mind that finer and more homogeneous material will yield more accurate results. Increased accuracy can be obtained by letting the sample dry in the sun before testing. Mix the sample thoroughly by kneading the bag.

6.4.2 The accuracy of measurements will be limited by the thickness of the plastic in the bag used. 1 mil-thick polyethylene bags offer a reasonable compromise between accurate readings and bag durability.

6.4.3 Flatten the bag of soil to form a continuous uniform layer of at least 1 cm. (0.4 inch) thickness. Place the sample window flat against the bag and pull the trigger. **Do not hold bagged samples in your hand during testing.**

Warning: Always treat radiation with respect. Do not put your hand or any other body part on or near the sample window of the XRF while samples are being analyzed. Never point the XRF at yourself or anyone else. ALARA objectives must be considered whenever staff is using an XRF.

The operator is responsible for controlling access in the area in which the XRF is being used. When possible use signs, barricades or caution tape to restrict access. Never allow anyone to enter within 5 feet of the x-ray path.

6.4.3 If you are analyzing many samples at one time the easiest way to analyze samples to set up the test stand. See Attachment B for directions to set up the test stand.

6.4.4 When the XRF is in the test stand all operations are conducted from the iPAQ. The red light on top of the test stand will operate in the same way as the red light on top of the XRF. When the instrument is on and capable of emitting radiation the red light will be on constantly. When the light is flashing the instrument is emitting radiation. The instrument cannot emit radiation while the cover is open. The stand is constructed so that all radiation is absorbed by the stand, however, no one should stand behind the test stand while the XRF is being used. The deadman trigger lock cannot be used while the instrument is in the test stand.

6.4.5 Place the sample over the XRF sample window so that the sample is indirect contact with the window. Start the test from the iPAQ.

Signs must be posted restricting access to the area where the XRF is being used while in the test stand. The operator is responsible for restricting access near the XRF while it is being used. No one should be allowed within 5 feet of the x-ray path.

6.4.6 Watch the display screen results to decide when the test has reached the desired level of accuracy and stop the test through the iPAQ or the test will automatically stop when the preset time has expired. NOTE: if the instrument is set to run both standard and LEAP analysis consecutively and the test is ended during the standard analysis mode and before the LEAP analysis has begun your data will not be stored.

6.5 ANALYSIS OF PREPARED SAMPLES

Prepared sample analysis is the most accurate method for determining the concentration of elements in a solid media. Sample preparation minimizes the effects of moisture, large particle size and variations in particle size.

Following this protocol for preparing and testing samples is vital for achieving a level of accuracy comparable with laboratory results. See Attachment B for EPA's approved method for analyzing samples using and XRF (EPA 6200). MEDEP has developed the following preparation method for samples to be analyzed by an XRF.

6.5.1 Collect 50-100 grams of sample to insure that there is enough sample to be representative and unbiased after mixing, grinding, and sieving it. You must have enough sample to half fill the XRF sample cup.

6.5.2 Place the sample in a clean bowl and mix the sample thoroughly by stirring and by rotating the bowl. Gently break up any dirt clods. Don't shake the sample because the sample may become stratified by weight.

6.5.3 If the sample is moist it should be dried. To best prepare a sample for analysis the material should be dry and well homogenized. Ideally, the entire sample should be dried to constant weight, sieved to remove gravel and debris, and ground or milled to a fine powder.

The sample can be dried in several ways:

- Oven dry the sample for approximately 2 hours at 150° C., until the sample reaches a constant weight;
- air dry the sample overnight at room temperature in a shallow pan;
- gently stir and warm the sample in a pan over a hot plate or burner.

Oven, hot plate or burner drying is inappropriate when volatile compounds may be present in the sample. For example, lead present as tetraethyl lead would be driven off by the heat of drying. Some forms of mercury and arsenic are volatile. If mercury is to be analyzed the sample must be air dried.

6.5.4 Sieve the dried sample with the #10 (2mm) mesh and separate out the larger pieces (stones, organic matter, metallic objects).

6.5.5 Grind the sample with a mortar and pestle or electric grinder until the soil particles are fine and homogenous.

6.5.6 Sieve at least 10 grams of the sample through #60 (250 um) and #120 (125 um) mesh. Re-grind the unpassed material until the required fraction is able to pass. Mix the resulting sample.

6.5.7 Place the sample in a sample cup. To assemble a sample cup: *1)* place a circle of mylar film on top of an XRF sample cup. The window goes on the end of the cup with the indented ring. *2)* Secure the film with the collar. The flange inside the collar faces down and snaps into the indented ring of the cup. Inspect the installed film window for continuity and smooth, taut appearance. *3)* Set the cup, window-side down, on a flat surface. Fill it with at least three grams of the prepared sample (no more than half-full). Take care that there are no voids or layering. *4)* Placing the cup film-side down on a flat surface, tamp the sample into the cup. *5)* Fill the cup with polyester fiber stuffing to prevent sample movement. Use aquarium filter or pillow filling as stuffing. A small supply of stuffing comes with the bulk sample kit. *6)* Fasten the cap on the cup.

6.5.8 Analyze the sample with the XRF. The easiest way to analyze samples in cups is to set up the test stand. See Attachment B for directions to set up the test stand.

6.5.9 When the XRF is in the test stand all operations are conducted from the iPAQ. The red light on top of the test stand will operate in the same way as the red light on top of the XRF. When the instrument is on and capable of emitting radiation the red light will be on constantly. When the light is flashing the instrument is emitting radiation. The instrument cannot emit radiation while the cover is open. The stand is constructed so that all radiation is absorbed by the stand, however, no one should stand behind the test stand while the XRF is being used.

6.5.10 Place the sample cup over the XRF sample window so that the cup is indirect contact with the window. Start the test from the iPAQ.

Warning: Always treat radiation with respect. Do not put your hand or any other body part on or near the sample window of the XRF while samples are being analyzed. Never point the XRF at yourself or anyone else. ALARA objectives must be considered whenever staff is using an XRF.

Signs must be posted restricting access to the area where the XRF is being used. The operator is responsible for restricting access near the XRF while it is being used. No one should be allowed within 5 feet of the x-ray path.

6.5.11 Watch the display screen results to decide when the test has reached the desired level of accuracy and stop the test through the iPAQ or the test will automatically stop when the preset time has expired. NOTE: if the instrument is set to run both standard and LEAP analysis consecutively and the test is ended during the standard analysis mode and before the LEAP analysis has begun your data will not be stored.

7.0 LEAD PAINT ANALYSIS AND PROCEDURE

7.1 LEAD PAINT ANALYSIS MODE

7.1.1 After completing the procedure described in section 4.4 there are two buttons shown on the bottom of the touch screen "Start" and "Info". Tap on the Info button to enter information specific to the samples you are analyzing. In there are preset options such as Operator, Location and Comment. These can be customized to projects when necessary. Fill in the information for the sample before analysis. The analysis will be stored with this information. You need to change the information prior to each sample that is run.

7.1.2 The bottom menu on the screen shows 4 options: File, Edit, View, Options and Help. From these menus the operator can change the settings for the method of analysis (Inspection or Fixed time). For a complete description of these menus and how to change the settings, see Attachment B.

7.1.3 Inspection mode automatically ends the test when the analyzer reaches a "Positive" or "Negative" determination with 95% confidence. This is based on a preset action level (the default is 1.0 mg/cm^2).

7.1.4 Fixed time mode always test up to the preset time (default 15 seconds). This returns actual results as opposed to the positive or negative results in the inspection mode.

7.1.5 To begin testing a sample the operator either taps the start button at the bottom of the screen or pulls the trigger. Note the software lock may have to be disabled if the instrument has not been used for more than 5 minutes.

7.1.6 Check the XRF's calibration with testing standard before using the XRF to analyze samples. Recheck the standards at least once every 4 hours during testing and after analysis has been completed for the day.

7.1.7 Hold the analyzer up to the sample to be analyzed. Make sure the sample window is as flat as possible against the sample. Start the analysis either from the iPAQ window or with the trigger. The red light on top of the instrument will flash while the analysis is performed and the instrument is emitting radiation. When at all possible use the instrument with the deadman trigger engaged. This means the operator must hold the trigger during the entire analysis. If the deadman trigger is not engaged the test can be stopped by pulling the trigger again or depending on the test mode the instrument will end the test when a positive or negative result is reached or the preset time period has elapsed.

Warning: Always treat radiation with respect. Do not put your hand or any other body part on or near the sample window of the XRF while samples are being analyzed. Never point the XRF at yourself or anyone else. ALARA objectives must be considered whenever staff are using an XRF.

The operator is responsible for controlling access in the area in which the XRF is being used. When possible use signs, barricades or caution tape to restrict access. Never allow anyone to enter within 5 feet of the x-ray path.

8.0 DUST WIPE ANALYSIS AND PROCEDURE

8.1 DUST WIPE TEST MODE

8.1.1 After completing the procedure described in section 4.4 there are two buttons shown on the bottom of the touch screen "Start" and "Info". Tap on the Info button to enter information specific to the samples you are analyzing. There are preset options such as Operator, Location and Comment to choose from. These can be customized to projects when necessary. Fill in the information for the sample before analysis. The analysis will be stored with this information. You need to change the information prior to each sample that is run.

8.1.2 The bottom menu on the screen shows 4 options: File, Edit, View, Options and Help. From these menus the operator can change the settings for the analysis (e.g. 4 or 8 tests per wipe, area of wipe (default 1ft^2.)) For a complete description of these menus and how to change the settings, see Attachment B.

8.2 SAMPLE PREPARATION

8.2.1 Conduct wipe sample according to Attachment C "Settled Dust Sampling for Lead". However, instead of packaging the wipe for analysis at a laboratory continue as follows.

8.2.2 For best results dry the wipe before analysis.

8.2.3 Fold the wipe so that it will fit into the dust wipe holder as shown in Attachment B. Center the filter in the holder and secure the holder with tape.

8.3 ANALYZING THE DUST WIPE

8.3.1 The XRF can be set to analyze the dust wipe in either 4 or 8 positions on the wipe. If 4 positions are set then they are analyzed in four quadrants of the wipe on the same side. For 8 positions, four quadrants on each side are analyzed.

8.3.2 Place the dust wipe on a flat surface and position the sample window in 1 quadrant of the filter. Pull the trigger. The red light on top of the instrument will flash during analysis indicating the instrument is emitting radiation. When the first position is complete the iPAQ will prompt for the additional readings. Reposition the XRF and tap ok on the screen. Note: If you cancel instead of saying ok the wipe measurement will be aborted and no results will be saved. If you stop the test before any position reading has been completed, no results will be saved.

Warning: <u>Always</u> treat radiation with respect. Do not put your hand or any other body part on or near the sample window of the XRF while samples are being analyzed. Never point the XRF at yourself or anyone else. ALARA objectives must be considered whenever staff are using an XRF.

The operator is responsible for controlling access in the area in which the XRF is being used. When possible use signs, barricades or caution tape to restrict access. Never allow anyone to enter within 5 feet of the x-ray path.

8.3.3 After the last reading has been completed the analyzer will open the results screen and display an average of the readings taken on the dust wipe.

<u>9.0 DOWNLOADING DATA FROM THE XRF</u>

9.1 DOWNLOADING DATA

The Innov-X XRF stores thousands of measurements plus their spectra. This can be downloaded to a computer for reporting in a spreadsheet format. From the Innov-X menu screen choose view on the bottom and then choose results. This will open the last result entered into the iPAQ. Choose “File” on the bottom of the screen them choose “export results”. From this screen you can choose the date and analysis mode for the results (analytical results are saved on the iPAQ by date). After these options have been chosen, click “OK” at the bottom of the screen. The next screen allows you to enter a file name and location to save the file to. The file can then be downloaded to your desk top computer by synchronizing the iPAQ with your computer and saving the data file in an excel format. You must have the iPAQ software installed on your computer. See Attachment B for complete directions on downloading data.

Note: Downloading data does not erase readings. To make room for the next set of data, erase readings after verifying that the data was downloaded successfully.

9.2 ERASING READINGS

Once your data has been downloaded from the i-PAQ the file should be erased. From the Innov-X menu screen choose "view" then "results". This will open the last analysis saved to the iPAQ. Choose "file" at the bottom of the screen then "erase readings". You must enter the administrator password (lower case z). Choose which readings you would like to delete then click "OK". Make sure your data has successfully transferred to your desk top prior to deleting data. See Attachment B for complete directions on erasing data.

10.0 DECONTAMINATION

Decontamination of equipment will follow the MEDEP DR SOP DR#017 - "Decontamination Procedures Protocol". Additionally the following methods may be used in the field:

The mortar, pestle, and grinding mill may be cleaned with dry paper towels. Water will also clean the mortar, pestle, and the mill's container, but be sure each is absolutely dry before they are used for another sample. The mortar and pestle may be cleansed by grinding clean dry sand in the mortar. Use the short bristle brushes (included in the Bulk Testing Kit) to clean the sieves.

11.0 CHAIN OF CUSTODY

For confirmatory samples that are submitted to a fixed laboratory, procedures for chain of custody outlined in MEDEP/DR SOP DR#012 - "Chain of Custody" must be followed.

12.0 DOCUMENTATION

All sampling activities must be documented as outlined in MEDEP/DR SOP DR#013 – "Documentation of Field Activities and Development of a Trip Report". Each sample location will be given a unique sample number. This number will be entered into the XRF with the optical pen and or recorded in the field notes. If no number is entered into the XRF, the default number shown on the XRF screen for that sample will be recorded in the field notes.

13.0 QUALITY ASSURANCE/QUALITY CONTROL

13.1 QUALITY ASSURANCE SAMPLES

Depending on the DQO's for a project the following QA samples may be collected. Any QA sample analyzed will be documented in field notes or in a written report. Calculations for QA samples will also be documented and if QA samples are re analyzed the results of will be documented.

13.1.1 Energy Calibration Check

To determine whether the XRF is operating within resolution and stability tolerances, an energy calibration check should be run. Generally, this is run 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.

13.1.2 Blank Samples

Two types of blank samples should be analyzed for XRF analysis: instrument blanks and method blanks. An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window.

13.1.2.1 Instrument Blank

The instrument blank can be silicon dioxide, a Teflon 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.

13.1.2.2 Method Blank

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. To be acceptable, a method blank must not contain any analyte at a concentration above its method detection limit. If an analyte's concentration exceeds its method detection limit, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

13.1.3 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 samples used by the DR will be NIST or other SRM that contains the analytes of interest. These will 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 re-calibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

13.1.4 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. 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.

The equation for calculating RSD is as follows: RSD = (SD/Mean Concentration) x 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.

14.1.5 Confirmatory Samples

The comparability of the XRF analysis is determined by submitting XRF-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 XRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on data quality objectives. The confirmatory analyses can also be used to verify the quality of the XRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the XRF. They should also include samples with analyte concentrations at or near the site action levels. Acceptance criteria for comparison of field and lab samples will be 20% difference of sample results or stated in the site specific QAPP or sampling plan. If the acceptance criteria is exceeded the project manager will evaluate the results to determine if they meet the data quality objectives for the project. If the data quality objectives are not met samples will be re-run or collected again for analysis.

14.2 DEVIATIONS FROM SOPS

All deviations from the procedures outlined in this or in any other SOPs followed for XRF sampling must be documented in field notes.

15.0 REFERENCES

- Innov-X User Manual for Alpha Series XRF.
- EPA Method 6200 Field Portable X-Ray Fluorescence Spectrometry For the Determination of Elemental Concentrations in Soil and Sediment.

ATTACHMENT A

EPA Method 6200 Field Portable X-Ray Fluorescence Spectrometry For the Determination of Elemental Concentrations in Soil and Sediment.

METHOD 6200

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

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

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

1.0 SCOPE AND APPLICATION

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

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

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

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

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

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

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

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

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

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

2.0 SUMMARY OF METHOD

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

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

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

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

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

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

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

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

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

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

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

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

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

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

4.0 INTERFERENCES

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

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

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

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

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

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

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

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

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

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

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

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

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

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

5.0 SAFETY

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

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

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

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

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

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

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

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

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

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

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

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

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

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

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the units or from PCs. Once the data–storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

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

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

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

6.6 Containers -- Glass or plastic to store samples.

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

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

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

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

7.0 REAGENTS AND STANDARDS

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

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

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

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

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

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

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

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

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

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

9.0 QUALITY CONTROL

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

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting, which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

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

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

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

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

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

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

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

The equation for calculating RSD is as follows:

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

where:

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

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

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

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

10.0 CALIBRATION AND STANDARDIZATION

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

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

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

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

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

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

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

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

The equation to calibrate %D is as follows:

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

where:

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

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

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

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

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

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

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

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

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

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

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

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

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

11.0 PROCEDURE

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

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

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

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

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

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

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

12.0 DATA ANALYSIS AND CALCULATIONS

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

13.0 METHOD PERFORMANCE

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

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	K Lines
				Molybdenum to Barium	L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium	K Lines
				Tantalum to Lead	K Lines
				Barium to Uranium	L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium	K Lines
				Tungsten to Uranium	L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium	K Lines
				Lanthanum to Lead	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	K Lines
			Silver to Gadolinium	L Lines
Mo	40-50	17.4	Cobalt to Yttrium	K Lines
			Europium to Radon	L Lines
Ag	50-65	22.1	Zinc to Technicium	K Lines
			Ytterbium to Neptunium	L Lines

Source: Ref. 4

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

TABLE 4

EXAMPLE PRECISION VALUES

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

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

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

NR Not reported.

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

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

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

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

[a] These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.

ND Not detected.
NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

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

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

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

TABLE 7

EXAMPLE ACCURACY FOR TN 9000[a]

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

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

[a] All concentrations in milligrams per kilogram.

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

-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY[1]

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

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

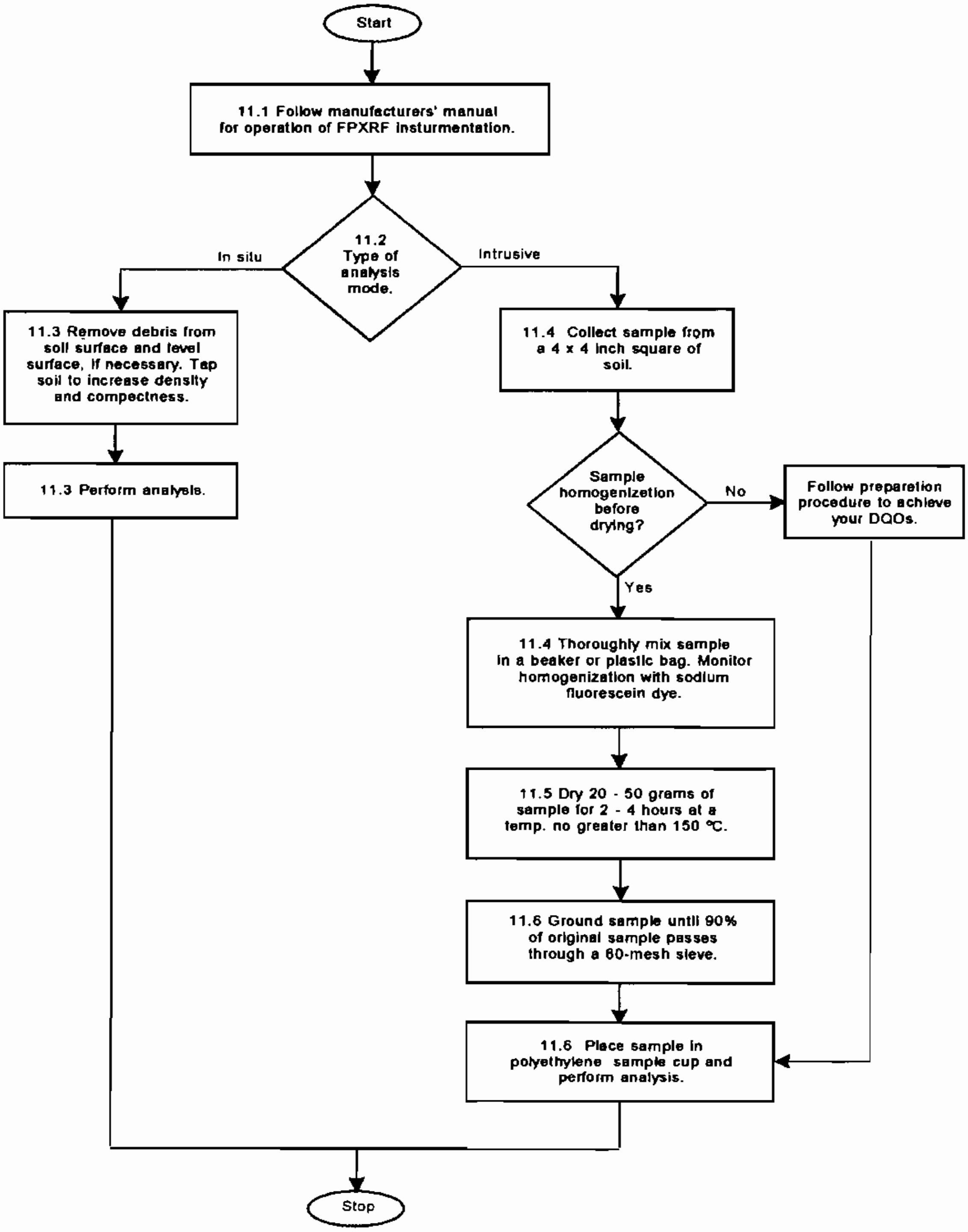
Source: Ref. 4. These data are provided for guidance purposes only.

[1] Log-transformed data

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

— No applicable data

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



ATTACHMENT B

INNOV-X USER Manual for Alpha Series XRF

THE USER MANUAL CAN BE FOUND WITH THE INSTRUMENT IN ITS CASE, OR ON LINE AT INNOV-X.COM

ATTACHMENT C

MEDEP SOP FOR COLLECTION OF DUST WIPE SAMPLES

Part #7	**Section #3**	**Data Quality Assurance**
Description:	Settled Dust Sampling for Lead	
Type:	Lead Sampling	
Date Issued:	April 20, 2004	

1.0 Scope

1.1 This section establishes sampling procedures and protocols for collecting settled dust samples.

2.0 Policy

2.1 Unit staff shall use these procedures for collecting paint chip samples of building components suspected of containing lead-based paint. Unit staff may deviate from these procedures with warranted justification and shall note deviations on the inspection report.

3.0 General Information

Wipe Sampling for Settled Lead-Contaminated Dust. Wipe samples for settled leaded dust can be collected from floors (both carpeted and uncarpeted), interior and sash/sill contact areas, and other reasonably smooth surfaces. Wherever possible, hard surfaces should be sampled. Wipe media should be sufficiently durable so that it is not easily torn, but can be easily digested in the laboratory. Recovery rates of between 80-120% of the true value should be obtained for all media used for wipe sampling. Blank media should contain no more than 25 ug/wipe (the detection limit using Flame Atomic Absorption)

4.0 Sampling Tools and Materials

4.1 **Type of disposable wipe:** Any wipe material that meets the following criteria may be used:

Contains low background lead levels,
Is a single thickness,
Is durable and does not tear easily (do not use Whatman™ filters),
Does not contain aloe,
Can be digested in the laboratory,
Has been shown to yield 80-120% recovery rates from samples spiked with leaded dust (not lead in solution), and
Must remain moist during the wipe sampling process (wipes containing alcohol may be used as long as they do not dry out).

4.2 **Non-sterilized non-powdered disposable gloves.** Disposable gloves are required to prevent cross-sample contamination from hands.

4.3 Non-sterilized polyethylene centrifuge tubes (50 ml size) or equivalent hard-shell container that can be rinsed quantitatively in the laboratory.

4.4 Dust sample collection forms.

4.5 Camera & film to document exact locations (Optional).

4.6 **Template Options.** Masking tape or hard, smooth, reusable templates may be used to define the area to be wiped. Periodic wipe samples should be taken from the templates to determine if the template is contaminated. Disposal templates are also permitted so long as they are not used for more than a single surface. Templates must be larger than $0.1ft^2$, but smaller than $2ft^2$. Templates for floors are typically $1ft^2$. Templates are usually not used for windows due to the variability in size and shape (use masking tape instead).

Note: Masking tape may damage the painted surface.

4.7 Container labels or permanent marker.

4.8 Trash bag or other receptacle (do not use pockets or trash containers at the residence).

4.9 Rack, bag, or box to carry tubes (optional).

4.10 Measuring tape.

4.11 Disposable shoe coverings (optional).

5.0 Single Surface Wipe Sampling Procedure

5.1 Outline Wipe Area:

5.1.1 Floors: Identify the area to be wiped. Do not walk on or touch the surface to be sampled (the wipe area). Apply masking tape to perimeter of the wipe area to form a square or rectangle of about one square foot. No measurement is required at this time. The tape should be positioned in a straight line and corners should be nominally perpendicular. When putting down any template, do not touch the interior wipe area.

5.1.2 Window sills and other rectangular surfaces: Identify the area to be wiped. Do not touch the wipe area. Apply two strips of masking tape across the sill to define a wipe area at least 0.1 square foot in size (approx. 4 inches x 4 inches).

5.2 **Preliminary inspection of the disposable wipes.** Inspect the wipes to determine if they are moist. If they have dried out, do not use them. When using a container that dispenses wipes through a "pop-up" lid, the first wipe in the dispenser at the beginning of the day should be thrown away. The first wipe may be contaminated by the lid and is likely to have dried to some extent. Rotate the container prior to each use to ensure liquid inside the container contacts the wipes.

5.3 **Gloves.** Don a disposable glove on one hand; use a new glove for each sample collected. If two hands are necessary to handle the sample, use new gloves, one for each hand. It is not necessary to wipe the gloved hand before sampling.

5.4 **Collection of sample.** Place the wipe at one corner of the surface to be wiped with wipe fully opened and flat on the surface. For square sample areas, complete a first wipe pass side-to-side as follows. With the fingers together, grasp the wipe between the thumb and the palm. Press down firmly, but not excessively with both the palm and fingers (Do not use only the fingertips or the heel of the hand to hold down the wipe, because there will not be complete contact with the surface and some dust may be missed.) Do not touch the surface with the thumb. Proceed to wipe side-to-side with as many "S"-like motions as are necessary to completely cover the entire wipe area. Exerting excessive pressure on the wipe will cause it to curl. Exerting too little pressure will result in poor collection of dust. Attempt to remove all visible dust from the wipe area.

5.4.1 Next, fold the wipe in half with the contaminated side facing inward. (The wipe can be straightened out by laying it on the wipe area, contaminated side up, and folding it over.) Once folded, place in the top corner of the wipe area and press down firmly with the palm and fingers. Complete a second wipe pass moving from top-to-bottom and wiping the area with "S"-like motions. Attempt to remove all visible dust. Do not touch the contaminated side of the wipe with the hand or fingers. Do not shake the wipe in an attempt to straighten it out, since dust may be lost during shaking.

5.5 For rectangular sample areas: two side-to-side passes must be made over half of this surface, the second pass with the wipe folded so that the contaminated side faces inward. For a window sill, do not attempt to wipe the irregular edges presented by the contour of the window channel. Avoid touching other portions of the window with the wipe. If there are paint chips or gross debris in the window sill, attempt to include as much of it as possible on the wipe. If all of the material cannot be picked up with one wipe, field personnel may use a second wipe at their discretion and insert it in the same container. Consult with the analytical laboratory to determine if they can perform analysis of two wipes as a single sample. When performing single-surface sampling, do not use more than two single surface wipes for each container. If heavily dust-laden, a smaller area should be wiped. It is not necessary to wipe the entire window well but do not wipe less than 0.10 ft^2 (approx. 4" x 4").

6.0 **Packaging the Wipe.** After wiping, fold the wipe with the contaminated side facing inward again, and insert aseptically (without touching anything else) into the centrifuge tube or other hard-shelled container. If gross debris is present, such as paint chips in a window well, make every attempt to include as much of the debris as possible in the wipe.

6.1 **Seal the tube and label with the appropriate identifier.** Record the laboratory submittal sample number on the field sampling form.

7.0 **Area Measurement.** After sampling, measure the surface area wiped to the nearest eighth of an inch using a tape measure or a ruler. The size of the area wiped must be at least 0.10 ft^2 in order to obtain an adequate limit of quantitation. No more than 2 square feet should be wiped with the same wipe or else the wipe may fall apart. Record specific measurements for each area wiped on the field sampling form.

8.0 **Form completion.** Fill out the appropriate field sampling forms completely. Collect and maintain any field notes regarding type of wipe used, lot number, collection protocol, etc.

9.0 **Trash Disposal.** After sampling, remove the masking tape and throw it away in a trash bag. Remove the glove; put all contaminated gloves and sampling debris used for the sampling period into a trash bag. Remove the trash bag when leaving the dwelling. Do not throw away gloves or wipes inside the dwelling unit where they could be accessible to young children, resulting in a suffocation hazard.

10.0 **Blank Preparation.** After sampling the final dwelling unit of the day, but before decontamination, field blank samples should be obtained. Analysis of the field blank samples determines if the sample media is contaminated. Each field blank should be labeled with a unique identifier similar to the others but that identifies the sample as a field blank.

10.1 Blank wipes are collected by removing a wipe from the container with a new glove, shaking the wipe open, refolding as it occurs during the actual sampling procedure, and then inserting it into the centrifuge tube without touching any surface or other object. One blank wipe is collected for each dwelling unit sampled or, if more than one dwelling unit is sampled per day, one blank for every 50 field samples, whichever is less. Also, collect one blank for every lot used. Record the lot number.

11.0 **Lead Inspector Decontamination.** After sampling, wash hands thoroughly with plenty of soap and water. A bathroom in the dwelling unit may be used for this purpose, with the owner's or resident's permission. If there is not running water in the dwelling unit, use wet wipes to clean the hands. During sampling, inspector must not eat, drink, smoke, or otherwise cause hand to mouth contact.

12.0. **Laboratory Submittal.** Submittal Form Preparation. The sample numbers on the sample container must be the same as those on the field sampling form and must also be used on the laboratory submittal form. Confirm that all samples recorded are in fact present on the laboratory submittal form. Chain of custody requirements should be followed.

13.0. **Laboratory Analytical Procedure.** Laboratories analyzing dust samples must participate in the Environmental Lead Laboratory Proficiency Testing Program or equivalent and be an EPA-NLLAP Accredited Laboratory and certified for environmental lead analysis in accordance with the Maine Department of Human Services Chapter 263, "Maine Comprehensive and Limited Environmental Laboratory Certification Rules". The Maine Health and Environmental Testing Laboratory (HETL) currently meets these standards.