New Mexico Environment Department Voluntary Remediation Program Preliminary Work Plan

City of Albuquerque Rail Yards

Albuquerque, Bernalillo County, New Mexico

Prepared for:

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ACRONYMS AND ABBREVIATIONS

ACBM	asbestos-containing building materials
amsl	above mean sea level
AST	aboveground storage tank
ATSF	Atchison, Topeka and Santa Fe
bgs	below ground surface
BNSF	Burlington Northern Santa Fe Railway
CNS	Covenant Not to Sue
COA	City of Albuquerque
CCOC	Conditional Certificate of Completion
COC	Certificate of Completion
COPC	contaminant of potential concern
CSM	conceptual site model
CWE	Central Works Equipment
DBS&A	Daniel B. Stephens and Associates
DRO	diesel range organic
EDB	1,2-dibromoethane
EPA	U.S. Environmental Protection Agency
ERM	Environmental Resources Management, Inc.
ft	feet *or* foot
ft/ft	foot per foot
GRO	gasoline range organic
HAI	Huang & Associates, Inc.
INTERA	INTERA Incorporated
LBP	lead-based paint
LNAPL	light non-aqueous phase liquid
MRO	motor oil range organic
NMAC	New Mexico Administrative Code
NMED	New Mexico Environment Department
NM-GS	New Mexico Ground Water Standard
NMWQCC	New Mexico Water Quality Control Commission

ACRONYMS AND ABBREVIATIONS (CONCLUDED)

PAH	polynuclear aromatic hydrocarbon
PCS	petroleum-contaminated soil
PID	photoionization detector
ppm	part per million
RSL	regional screening level
S&A	sampling and analysis
Site	Albuquerque Rail Yards, Albuquerque, New Mexico
SSL	soil screening level
SWB	Solid Waste Bureau
TPH	total petroleum hydrocarbon
UST	underground storage tank
VISL	vapor intrusion screening level
VOC	volatile organic compound
VRP	Voluntary Remediation Program

1.0 INTRODUCTION

This Preliminary Voluntary Remediation Work Plan developed by INTERA Incorporated (INTERA) presents the results of environmental investigation activities completed to date and proposes additional voluntary investigation/remediation actions for the City of Albuquerque (COA) Rail Yards (Site) in support of Site redevelopment. The COA purchased the Site in 2007 from the Old Locomotive Shops, LLC through Renaissance Development Company, Inc.

Situated between 2nd Street and Commercial Street in downtown Albuquerque, New Mexico, the Site consists of approximately 27 acres (Areas A, B, and C and Tract A) located within the former Atchison, Topeka and Santa Fe (ATSF)/Burlington Northern Santa Fe (BNSF) Central Works Equipment (CWE) Facility Railyard that operated from the 1880s to the early 1990s. As a result of previous operations, the Site sustained environmental impacts from both petroleum hydrocarbon and metals contamination. Contamination is present in both the Site vadose zone (Site soils and soil vapor) and in the saturated zone (Site groundwater) and includes metals adsorbed to soil particles, organic vapors, and organic and inorganic solutes dissolved in groundwater. In addition, both asbestos-containing building materials (ACBM) and lead-based paint (LBP) were used in many of the remaining Site buildings; contamination related to these building materials will also need to be mitigated during any building demolition or building renovation activity.

To best address contamination concerns and help facilitate redevelopment, the COA is seeking to complete Site redevelopment by inclusion in the New Mexico Environment Department (NMED) Voluntary Remediation Program (VRP). By actively participating in the NMED VRP (and upon successful completion of all additional Site investigation/remediation actions), the COA will be able to obtain a Conditional Certificate of Completion (CCOC) and/or Certificate of Completion (COC) for either the entire Site or specific areas of the Site. The COC/CCOC will document that current conditions in a designated area(s) and/or throughout the Site meet applicable environmental quality standards and will provide liability protection for lenders. In addition, once a CCOC or COC is issued, a Covenant Not to Sue (CNS) may be transferred to a selected prospective purchaser and/or future developer or lessor(s) of the Site.

The Site was previously enrolled in the NMED VRP (VRP Site No. 53061001); however, at the time, the Site was not under COA ownership and not all remediation work was completed. As such, NMED has requested a new VRP Application for the Site to be initiated under COA ownership. This work plan represents one required component of the VRP Application and intends to (1) present information to demonstrate the Site continues to meet criteria necessary to participate in the VRP, (2) propose activities to address all remaining environmental concerns identified for the Site such that interested parties may receive all appropriate documentation necessary to complete redevelopment with limited liability, and (3) request NMED VRP support in issuance of a CCOC and/or COC to address contamination concerns in a designated area(s) and/or throughout the Site prior to the initiation of proposed redevelopment activities.

2.0 SITE BACKGROUND

This section briefly summarizes the Site location and general characteristics, the Site physical setting, the Site geology and stratigraphy, the Site hydrology, previous Site environmental investigations, and suspected/known Site contaminants of potential concern (COPCs).

2.1 Location and General Description

The Site, also referred to as the Albuquerque Locomotive Shops and the former ATSF/BNSF CWE facility, is located approximately 1 mile south of the center of downtown Albuquerque in Bernalillo County, New Mexico (**Figure 1**).

The legal description is as follows:

> *The “Albuquerque Old Locomotive Shop” (former A.T. & S.F. Railway Company Machine Shop) is a 27.32 acre tract, more or less, out of the Tract A of the Plat of Tract “A”, A.T. & S.F. Railway Company Machine Shop, 27.321 acre tract located at 2nd and Bridge in Albuquerque in Bernalillo County, New Mexico….*
>
> *Tract “A” of the Plat of Tract A, A.T. & S.F. RAILWAY COMPANY MACHINE SHOP, Albuquerque, Bernalillo County, New Mexico, as the same is shown and designated on the Plat filed in the office of the County Clerk of Bernalillo County, New Mexico on January 25, 1996 as Document Number 1996008744, recorded in Vol. 96C, folio 44, records of Bernalillo County, New Mexico.*

A copy of the Site legal description is provided in **Appendix A.**

The Site operated as a railroad CWE facility from the 1880s to the early 1990s. Activities conducted at the facility included servicing locomotives (blacksmithing, welding, and painting) within the Machine Shop, Boiler Shop, Roundhouse, and other areas; and general servicing and maintenance activities of the facility. The Site also was a central location for the ATSF/BNSF to perform required servicing activities in support of other smaller railyards located nearby. The various types of chemicals used and stored at the Site included: solvents and lye used for parts cleaning; paint; heavy oils, diesel fuel, and other lubricants; and packaged herbicides (INTERA, 2015). In the 1960s, the roundhouse was closed and subsequently demolished. In 1991, all underground storage tanks [USTs] were removed, and the Site was vacated of further industrial/commercial use. Since that time, the Site has largely been unused, except by the film industry. The COA purchased the Site in 2007 and renovated the Blacksmith Shop and Storehouse buildings as interim use/multi-purpose structures in 2013.

2.2 Site Physical Setting

The Site is located at an elevation of approximately 5,000 feet (ft) above mean sea level (amsl). The Site is primarily topographically level, with a slight drop in elevation toward the south.

Today, numerous small and large structures formerly used to support various railroad maintenance activities are still scattered across the Site. The Site is bounded to the west by residential property and to the north and south by commercial/industrial property (INTERA, 2015). Residential properties are located to the east of the Site beyond the adjoining railroad tracks. Between 1996 and 2000, a total of eleven (11) permanent groundwater monitoring wells were installed at Site.

No surface water exists at the Site. The nearest surface water body is the Rio Grande, located approximately 1,500 ft to the west.

2.3 Site Geology and Stratigraphy

The Site is located in the south-central portion of the Albuquerque Basin. This basin is one of the largest of the south-trending series of grabens that form the Rio Grande Drainage Basin, which was formed in response to the Rio Grande Rift (Thorn et al., 1993). The Rio Grande Rift is a north- to south-trending, downdropped area extending for more than 600 miles. The rift is an area of crustal extension originating in central Colorado and extending south through New Mexico to south of the Mexico-Texas border.

The Albuquerque Basin is filled with up to 10,000 ft of clastic sediments. The Santa Fe Formation sediments fill the majority of the basin. The Tertiary and Quaternary Santa Fe Formation is composed of unconsolidated to loosely consolidated gravels, sands, silts, and clays. The thickness of this unit ranges from 2,400 ft on the basin margins to 14,000 ft along the axis of the basin. In the vicinity of the Site, the thickness of this formation is on the order of 4,700 ft. The Santa Fe Group is overlain by Quaternary sediments, which have a similar facies distribution. These post-Santa Fe deposits are alluvial fan and floodplain deposits that are up to 200 ft thick (Thorn et al., 1993).

At the Site, surface and near-surface soils consist of sandy fill and debris and contain classic fining upward sequences typical of a fluvial depositional environment (INTERA, 2012; Innovar, 2011). Fine-grained sediments (sediments containing silts and clay) predominate in the shallow subsurface up to 10 to 15 ft below ground surface (bgs). Below these sediments, coarser-grained units consisting of fine- to coarse-grained sand extend to depths of at least 47 ft bgs. The contact between the fine and coarse units is gradational.

2.4 Site Hydrology

The Santa Fe Group and post-Santa Fe deposits are the principal water-bearing units in the vicinity of the Site and are hydraulically connected (U.S. Army Corps of Engineers, 1979; Thorn et al., 1993). However, the Albuquerque Basin aquifer is anisotropic laterally and vertically due to spatial variations in the lithology of these two water-bearing units (Chamberlin et al., 1992). Clay layers 12 to 15 ft thick are commonly observed in the alluvium of the Albuquerque Basin; these clay layers restrict vertical movement of water and may locally limit hydraulic interconnection between the shallow Quaternary aquifer and the Santa Fe Group aquifer (Thorn et al., 1993). As a result of spatial variations in lithology, the hydraulic transmissivity of the Albuquerque aquifer varies tremendously from less than 10 to 80,000 square ft per day (Thorn et al., 1993). The hydraulic conductivity of the upper part of the Santa Fe Group varies also, but is estimated to average approximately 20 ft per day in the vicinity of the Site (Thorn et al., 1993).

The water table configuration in the Albuquerque area has changed considerably over time due to population growth and the resulting increases in groundwater pumping and use. Regional groundwater flow in the vicinity of the Site before large-scale groundwater development is thought to have been toward the southwest, and this condition existed at least into the mid- to late-1930s (Thorn et al., 1993). More recent groundwater elevation contours constructed from groundwater elevation data obtained from Site wells (1996, 1999, 2010, and 2017), however, indicate that local groundwater flow is now predominately to the east. Hydraulic gradients calculated for horizontal groundwater flow beneath the Site ranged from 0.0042 foot per foot (ft/ft) (November 2016) to 0.006 ft/ft (April 1996 and December 1999) (INTERA, 2015).

Data collected during the mid-1990s from on-Site groundwater monitoring wells indicate the depth to groundwater beneath the Site was between 30 and 40 ft bgs (DBS&A, 1996a). More recent well data indicate that groundwater levels at the Site have increased approximately 5 to 10 ft above this range (INTERA, 2017). This rise in the local water table in the downtown COA area is likely a direct result of recently reduced groundwater pumping from the regional aquifer as the sole source for the area’s water supply. Additionally, as the water levels rise, contaminated vadose zone soils previously not saturated may become saturated. The newly saturated contaminated soils could contribute contamination to area groundwater.

2.5 Previous Investigations

Investigations into the nature and extent of petroleum hydrocarbon and metal contamination at the Site have been ongoing since 1988 and have primarily focused on the extent of the soil contamination and the dissolved-phase groundwater plume (INTERA, 2015). NMED conducted a limited site investigation at the Site in 1988. Characterization activities completed during this investigation included the sampling and analysis (S&A) of surface soils and the installation of two offsite monitoring wells. Results of this investigation indicated the presence of polynuclear aromatic hydrocarbons (PAHs) and metal in soils and trace toluene in groundwater (DBS&A, 1996b). These results initiated a series of additional characterization efforts and some remedial action for one or more portions of the Site; however, remedial actions were limited to small excavation areas. A summary of investigation activities completed for the Site since 1988 is provided in **Table 1.** A summary of remedial actions completed for the Site since 1988 is provided in **Table 2.** A database of all Site environmental data is included in **Appendix D.**

Table 1. Characterization and Investigation Activities

Date	Consultant	Location	Investigation Activity
December 1995	DBS&A	CWE Shops	Phase II Environmental Site Assessment – collected soil and groundwater samples
June 1996	DBS&A	ATSF Railway Company CWE Facility	Water well inventory, soil sampling, groundwater sampling, and aquifer test
July 1996	DBS&A	ATSF Railway Company CWE Facility	Quarterly groundwater monitoring – Stage I Abatement Plan
September 1996	DBS&A	ATSF Railway Company CWE Facility	Plugging and abandonment of on-site water supply wells
December 1996	DBS&A	ATSF Railway Company CWE Facility	Quarterly groundwater monitoring – Stage I Abatement Plan
March 1997	DBS&A	Former ATSF Railway Company CWE Facility	Quarterly groundwater monitoring – Stage I Abatement Plan
January 2000	DBS&A	Former ATSF Railway Company CWE Facility	Groundwater monitoring event
May 2000	Dames & Moore Inc.	Former ATSF Railway Company CWE Facility	Limited Site Investigation – collected soil and groundwater samples, installed wells
September 2000	ERM	CWE Shop Area, Transformer Vandalism Site	Investigated transformer oil leak from 13 vandalized electrical transformers
October 2005	Terracon	Albuquerque Locomotive Shops Area B, Area C, and Tract A	Site characterization and remediation excavation activities
June 2010	HAI	Albuquerque Locomotive Shops Area A	Phase II Environmental Site Assessment – collected soil and groundwater samples
January 2011	Innovar	Albuquerque Locomotive Shops Area B, Area C, and Tract A	Phase II Environmental Site Assessment – focused on nine areas of concern
July 2012	INTERA	Albuquerque Locomotive Shops Area B, Area C, and Tract A	Soil and groundwater sample collection to aid in the delineation of metal and petroleum hydrocarbon contamination
September 2015	INTERA	Conceptual Site Model, City of Albuquerque Rail Yards	Consolidate all previous Site data
February 2017	INTERA	Parcel 1 Additional Characterization Report	Soil, Soil Vapor, ACBM, LBP, and groundwater sampling
February 2017	INTERA	Parcel 2 Additional Characterization Report	Soil, Soil Vapor, ACBM, LBP, and groundwater sampling
February 2017	INTERA	Parcel 3 Additional Characterization Report	Soil Vapor, ACBM, and LBP sampling
February 2017	INTERA	Parcel 4 Additional Characterization Report	Soil, Soil Vapor, ACBM, LBP, and groundwater sampling
February 2017	INTERA	Parcel 5 Additional Characterization Report	Soil Vapor, ACBM and LBP sampling

Table 1. Characterization and Investigation Activities (concluded)

Date	Consultant	Location	Investigation Activity
February 2017	INTERA	Parcel 7 Additional Characterization Report	Soil Vapor, ACBM, and LBP sampling
February 2017	INTERA	Parcel 8 Additional Characterization Report	Soil Vapor, ACBM, and LBP sampling
February 2017	INTERA	Parcel 9 Additional Characterization Report	Soil, Soil Vapor, ACBM, and LBP sampling
February 2017	INTERA	Parcel 10 Additional Characterization Report	Soil, Soil Vapor, ACBM, LBP, and groundwater sampling
February 2017	INTERA	Additional Groundwater Characterization Report	Groundwater sampling
2018	City of Albuquerque Environmental Health Dept.	---	Groundwater and soil vapor sampling

Notes:

DBS&A = Daniel B. Stephens and Associates
ERM = Environmental Resources Management, Inc.
HAI = Huang & Associates, Inc.

Table 2. Excavation Activities

Date	Consultant	Location	Soil Removal Quantity (cubic yards)	Action Driver
2005	Terracon	Roundhouse excavation area	40	Lead
2005	Terracon	Former oil cellar/aboveground storage tank (AST) excavation area (reported to be backfilled with same soil, HAI, 2010)	330	Petroleum
2005	Terracon	Former battery storage excavation area	280	Lead
2005	Terracon	Former sand blasting excavation area	140	Lead

2.6 Contaminants of Potential Concern (COPCs)

The following constituents are identified as Site soil COPCs (INTERA, 2015):

- Metals: antimony, arsenic, chromium, iron, lead, manganese, and thallium
- PAHs: benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, and indeno(1,2,3-cd)pyrene
- TPH DRO and TPH MRO (the sum of TPH-DRO and TPH-MRO)

The following constituents are identified as Site groundwater COPCs (INTERA, 2015):

- PAHs including benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, and indeno(1,2,3-cd)pyrene

- Volatile organic compounds (VOCs) including benzene, toluene, ethyl benzene, and total xylenes (BTEX), total naphthalenes, and ethylene dibromide (EDB)
- TPH (DRO and MRO)
- Metals: barium, benzene, chromium, copper, iron, lead, manganese, and zinc

The following constituents are identified as Site soil vapor COPCs (COA, 2018):

- VOCs including trichloroethene (TCE)

These COPCs were derived from evaluating historical Site analytical data with the applicable New Mexico state regulatory standards including NMED SSLs (NMED, 2017) and the New Mexico Water Quality Control Commission (NMWQCC) Human Health Standards defined in New Mexico Administrative Code (NMAC) 20.6.2.3.3103 (New Mexico Ground Water Standards [NM-GS]) (NMED, 2004). Petroleum contamination was present in both the finer-grained and coarser-grained soil units.

Other Site COPCs established for Site redevelopment activities include:

- ACBM and LBP in Site buildings and structures

3.0 PROPOSED PERFORMANCE STANDARD

Site compliance with the VRP Performance Standard, as defined by NMAC 20.6.3.10 (NMED, 2001), will be established through the completion of a Method 2 assessment for both soil and soil vapor (**Section 3.1** and **Section 3.2,** respectively). For soil, the primary concern is the potential for exposing workers to soil impacted with metals and/or petroleum hydrocarbons during the excavation activities required as part of redevelopment. Current excavations are anticipated in distinct areas up to a depth of approximately 8 ft. Soil contamination removals will be in areas that are accessible; no removals will be conducted under existing buildings. The goal is to achieve soil concentrations for the contaminants of concern that are below applicable NMED residential soil screening levels (SSLs).

Exposure to VOCs as a result of soil vapor intrusion remains an environmental concern for the Site. During Site redevelopment, the primary concern with the presence of VOCs in soil vapor is its potential to adversely impact indoor air quality in new or renovated structures and buildings.

Groundwater is not included as a medium of concern to be addressed through active remediation during redevelopment as it is not anticipated to be directly encountered during Site redevelopment; however, because groundwater contamination is known to be present beneath the Site, groundwater is required to be included as part of the Site long-term monitoring program. Groundwater impacts at the northern half of the COA Yards is limited to elevated manganese, groundwater impacts at the southern half include elevated levels of petroleum hydrocarbons. Groundwater contamination in the southern part of the Site will be addressed in a subsequent VRP Work Plan.

Site groundwater monitoring should occur regularly to establish a record of groundwater quality over time. Site compliance with the VRP Performance Standard (NMED, 2001) will also be established for Site groundwater through the completion of a Method 2 assessment (**Section 3.3**).

3.1 Method 2 Assessment for Soils

Previous Site investigation work has established that shallow subsurface soils in a few distinct areas of the Site contain residual contamination. Of particular concern are (1) construction worker exposure, and (2) the potential generation of regulated waste. Construction activities at the Site may result in excavation of and exposure to soil impacted with elevated metals and VOCs/TPH which requires disposal at an approved disposal facility.

The COA will perform oversight of utility excavations to identify any contaminated soil as the work occurs. This will be performed in utility excavation areas for construction worker health and safety purposes.

The COA will remove and segregate any potential petroleum contaminated soil (PCS) encountered for disposal. Potential PCS shall be defined as any soil observed to contain a total VOC concentration in excess of 100 parts per million (ppm), the NMED TPH soil screening action level (see **Section 5.1** and **Section 5.5**).

Analytical results obtained from soil samples collected during redevelopment activities will be evaluated against applicable (1) New Mexico SSLs as defined by the most current version of NMED Risk Assessment Guidance for Site Investigations and Remediation (NMED, 2017), and (2) the most current version of U.S. Environmental Protection Agency (EPA) Regional Screening Levels (RSLs) (EPA, 2014) if an applicable NMED SSL is not available.

Section 7.0 provides further discussion regarding how the proposed Site characterization activities for Site soil will meet this performance standard.

3.2 Method 2 Assessment for Soil Vapor

Previous Site investigation work has established that shallow subsurface soils in a few distinct areas of the Site as well as shallow groundwater contain residual contamination that may cause the generation and accumulation of vapor-phase constituents in the Site vadose zone. Most of the contamination, both in soil and groundwater, is present in the southern half of the COA Rail Yards and this contamination will be addressed in future VRP Work Plans.

Of particular concern is the potential presence of these constituents within the first 5 ft of the subsurface in Site areas proposed for development of permanent, enclosed structures intended for residential use.

Active soil vapor characterization S&A activities will be performed to help delineate the lateral and vertical extent of vapor-phase contamination in the vadose zone. In particular, sub-slab soil vapor samples shall be collected in the vapor points from historic buildings located within the northern area of the Rail Yards. Previous sampling results have not indicated soil vapor concerns but additional sampling will be conducted to confirm that the soil vapor levels remain below NMED vapor intrusion screening levels (VISLs).

Analytical results obtained from active soil vapor samples collected will be evaluated against the most current applicable New Mexico vapor intrusion screening levels (VISLs) as defined by the most current version of NMED Risk Assessment Guidance for Site Investigations and Remediation (NMED, 2017).

Section 7.0 provides further discussion regarding how the proposed Site characterization activities for Site soil vapor will meet this performance standard.

3.3 Method 2 Assessment for Groundwater

Previous Site investigation work has established that groundwater contamination is present at the Site (**Section 2.6**). Based on the former and existing monitoring well network, the nature and extent of the petroleum hydrocarbon plume is not fully defined to the south, west, or east in the southern portion of the Site.

The nature and extent of metals in groundwater is also not fully defined in the southern portion of the Site. Groundwater currently represents an incomplete exposure pathway: It is not anticipated that excavations will extend into the water table and there is minimal risk of groundwater exposure to future environmental receptors. Groundwater contamination present in the southern half of the COA Rail Yards will be addressed in future VRP Work Plans.

Although groundwater does not currently represent an environmental exposure concern to current or reasonably foreseeable future Site users, groundwater S&A activities will be performed in existing Site monitoring wells to monitor local groundwater levels and plume behavior over time. Both iron and manganese were found to occur at concentrations in excess of applicable NM-GS since monitoring activities began. These occurrences appear to preferentially collocate with the estimated petroleum hydrocarbon dissolved-phase plume suggesting that biodegradation of benzene, total naphthalene, and other petroleum hydrocarbon compounds is occurring. This observation coupled with the absence of petroleum hydrocarbons and the presence of biodegradation parameters in the northern portion of the Site may indicate (1) iron and manganese are present due to the historical use of the northern portion of the property and potentially as natural degradation of petroleum hydrocarbons in and near the ground water table; and/or (2) these concentrations are indicative of background conditions. Due to a regional rising water table, Site groundwater elevations should be monitored to assess the potential of the rising local water table to further contribute to Site soil vapor contamination and to determine if the currently existing Site groundwater monitoring wells remain fully functional.

Analytical results obtained from any groundwater sample collected as part of proposed VRP S&A activities will be evaluated against applicable NM-GS. **Section 7.0** provides further discussion regarding how the proposed Site characterization activities for Site groundwater will meet this performance standard.

4.0 INTERIM REMEDIATION/PROTECTION MEASURES

The COA currently leases the Storehouse and the Blacksmith Shop for Interim Uses and intends to continue to lease these buildings during implementation of this VRP Work Plan. As such, the lease holders of these two buildings will be required to adhere to criteria detailed in this section (**Section 4.0**) while using these spaces. Noncompliance with the protection measures listed herein will result in termination of the lease.

4.1 Storehouse

The historic Storehouse building, also referred to as the Warehouse and/or the WHEELS Warehouse, is a long and narrow structure located in the southwest central portion of the Site and defines part of the Site's west boundary (**Figure 2**). Built in 1914, the Storehouse was used to house an enormous inventory of all manner of parts and supplies for the larger maintenance shops and the entire ATSF railroad system in general.

The COA currently leases the Storehouse to WHEELS for use as storage space for their collections. A 501(c)3 organization dedicated to collecting, preserving and creating educational exhibits about the history of transportation in Albuquerque and New Mexico, the WHEELS collections embrace the history of the railroads, as well as automobiles, horse and wagon, and other modes of transportation, and explore impact(s) the presence of the Rail Yards had on Albuquerque growth and development.

Under the current Site lease agreement, the interim use of the Storehouse is designated as "for storage or warehouse use and only such limited office use appropriate to a storage or warehouse area." Access to the Storehouse by the general public is currently not permitted: only WHEELS employees and volunteers are permitted to access the building and only for such interim uses as designated in the Site lease agreement (City of Albuquerque, 2008).

4.1.1 Required Protection Measures

In order for WHEELS to continue interim utilization of the Storehouse as identified in **Section 4.1** above or for any other use(s) as approved by the COA, environmental concerns for the building must be evaluated and appropriate protection measures identified and implemented. A review of current environmental data available for the Storehouse and immediate vicinity completed by INTERA for the purposes of evaluating required protection measures for the proposed interim uses identified the following as potential environmental concerns: ACBM, LBP, surface and near surface contaminated soils in the surrounding area, and soil vapor intrusion.

ACBM and LBP are present within the Storehouse. No renovation activities will be completed by the current tenant. Any renovation work will be completed by the COA after ACBM and LBP abatement activities are completed, and the renovation area is properly determined to be free of ACBM and LBP by a Certified Industrial Hygienist (CIH).

Controlling access to the Storehouse and the Storehouse's designated parking area and restricting access to other areas of the Site not approved for interim use will be maintained by the COA at all times. For the Storehouse, access to and from the building shall be delineated by fencing and signage. Fencing shall be required to be a minimum of 6 ft tall so as to deter trespassing into surrounding Site areas where access is not permitted. Fencing coupled with appropriate signage shall also clearly identify restricted versus non-restricted Site areas. Signs shall be used to clearly communicate the approved use of the designated area (e.g. "parking area for Storehouse only;" "restricted to tenant employees;" or "access not permitted"). All parking areas designated for Storehouse Interim Use shall be covered by a minimum of 6 inches of gravel. The application of gravel is intended to minimize exposure to residual Site soil contamination by eliminating the potential for direct contact with soil and minimizing the suspension of potentially contaminated soil particulates into the surrounding atmosphere. The addition of well-maintained graveled areas also helps to provide a visual key to Site users, emphasizing areas of approved access and use. If improvements are made to immediate Storehouse building areas (e.g. expansion of the parking lot to the east), upon written approval from the COA, improvements must be completed while adhering to all Interim Use requirements listed above. Any changes to the Interim Use requirements will also require notification and approval by NMED.

4.2 Blacksmith Shop

The Blacksmith Shop is located in the northeast central portion of the Site and defines part of the Site's east boundary (**Figure 2**). Currently, the COA leases the Blacksmith Shop between April and December of each year to a variety of tenants, including the Rail Yards Market and other private users, who use the facility for parties or other social gathering events. When rented, use of the Blacksmith Shop and immediate surrounding area is strictly limited to the following: the building, City-owned portable restrooms, designated walkways present between the buildings located west and south of the Blacksmith Shop, and associated parking lots located north and west of the Blacksmith Shop.

Current rules for any event to be held at the Blacksmith Shop are as follows:

1. There is no potable water available at the Blacksmith Shop.
2. Restrooms are included with the Blacksmith Shop with male, female, and ADA accessible facilities.
3. There are twelve (12) twenty (20) amp separate circuits and five (5) fifty (50) amp connections in the Blacksmith Shop.

4. The potential occupancy of the Blacksmith Shop is nine hundred ninety-nine (999) occupants depending upon the floor plan setup and approvals by the Fire Marshal. The total capacity includes all staff, vendors, and performers inside the space. The renter will be required to monitor the capacity.
5. No heating or cooling systems are provided at the Blacksmith Shop.
6. The COA is not responsible for loud train whistles or passing of trains during events.
7. Parking outside the Blacksmith Shop at un-designated sites or off-site is done at own risk. The COA is not responsible for damages.
8. The COA does not provide any sound or lighting equipment.
9. All doors of the Blacksmith Shop must be locked open during events.
10. It is the renter responsibility to supervise the conduct of all performers, staff, volunteers, vendors, and event guests when they are at the Blacksmith Shop.
11. No riding bicycles or skateboards; no hot air balloons; no onsite body piercing or tattoos.
12. No alcoholic beverages unless alcohol service permits are obtained.
13. No weapons, drug paraphernalia or illegal items.
14. A renter representative must be on-site at the Blacksmith Shop at all times during the rental period.
15. Renter assumes all responsibility for any personal property it brings to the Blacksmith Shop. Renter agrees to hold the COA harmless in the event of any damage or theft of any personal property.

Additional user requirements of the Blacksmith Shop as stipulated by the COA include:

1. Any member/or attendee of an event held at the Blacksmith Shop that is discovered in a restricted area may result in the immediate cancellation of that event, and forfeiture of the damage deposit given to the COA prior to that event. For the Rail Yards Market, which holds multiple events, any representative or member of the renter found to be within the Blacksmith Shop outside of renter rental hours may result in the cancellation of the next scheduled Rail Yards Market event.
2. No modifications of the Blacksmith Shop are allowed without prior written approval by the COA. If approved, any permanent or semi-permanent installment or change to the Blacksmith Shop will require a separate license agreement with the COA prior to being implemented. It is the responsibility of the renter to obtain this Agreement. If any permanent or semi-permanent changes to the Blacksmith Shop are made without prior written approval, then the COA reserves the right to terminate the Agreement or cancel future dates.

4.2.1 Required Protection Measures

A review of current environmental data available for the Blacksmith Shop identified the following potential environmental concerns for interim use: ACBM, LBP, and contaminated soils in the surrounding area. Extensive ACBM and LBP surveys have been conducted previously for the facility and the results of these surveys indicate that ACBM remains in the window glazing and LBP is present on ceiling structures.

To date, neither the ACBM nor the LBP concerns have been completely abated; however, temporary measures to control access and inadvertent exposure to such hazardous materials — sealing off windows, removal of weathered window putty, and prescriptive house cleaning and maintenance measures, such as floor cleaning prior to each planned facility event — continue to be applied to the building as needed. Until ACBM and LBP concerns are fully mitigated, every effort shall be made to prevent the accumulation of dust within the Blacksmith Shop. Repainting of the ceiling of the Blacksmith Shop is also being considered. Such temporary measures shall be incorporated into a master hazardous materials management plan to be drafted for the Blacksmith Shop Interim Use.

Like the Storehouse, the Blacksmith Shop management plan shall outline both short-term and long-term monitoring or maintenance requirements for ACBM and LBP remaining in the building and shall include a schedule for routine building inspections by an appropriately certified inspector. If, during an inspection, material confirmed to contain LBP or ACBM is determined to be of such condition as to represent an immediate and unacceptable exposure, the material shall be properly abated as deemed appropriate by a certified ACBM/LBP inspector during a time period that interim uses are not occurring.

Also like the Storehouse, controlled access to the Blacksmith Shop and restricting access to other areas of the Site not approved for interim use is paramount and must be maintained at all times. Access to and from designated Interim Use areas shall be delineated by 6 ft tall fencing and appropriate signage. All publically-accessible areas in the immediate vicinity of the building utilized for parking shall be covered by gravel a minimum of 6 inches thick. If the COA receives and subsequently approves, in writing, any request for building or area improvements during the interim use period, these improvements must be completed while adhering to the requirements listed above. Any changes to the Interim Use requirements will also require notification and approval by NMED.

Sub-slab soil vapor has been assessed in the Blacksmith Shop and Storehouse in 2017 and 2018. Sub-slab soil gas concentrations identified indicate impact from VOCs, but all concentrations of VOCs identified are below corresponding VISLs (as applicable). The COA will continue to monitoring sub-slab soil gas concentrations by sampling and if VISLs are exceeded, engineering controls will be constructed to mitigate any potential unsafe indoor air conditions.

4.3 Flu Shop

The Flu Shop is located in the northeast central portion of the COA Rail Yards (**Figure 2**). The COA is envisioning similar uses for the Flu Shop as are currently in place at the Blacksmith Shop. The ACMB/LBP in the Flu Shop will be remediated and the building will be remodeled for reuse. Current plans for the remodel are evolving and will be provided to NMED as they become final. NMED can be assured that no long-term occupancy of this building is being planned, only temporary uses as the Blacksmith Shop is the current goal for this building. Further, no use of the Flu Shop will occur until all ACBM and LBP is abated properly. A copy of the ACMB/LBP survey report for the Flu Shop is included in Appendix E along with the ACMB/LBP abatement plan.

4.4 Other Interim Uses

If other Interim Uses of the Site are identified by the COA prior to completion of Site redevelopment, revision of this section (**Section 4.0**) of the VRP Work Plan is required to incorporate such additional uses and the revised section will be resubmitted to NMED for approval prior to proceeding with the Interim Uses. All proposed Interim Uses shall be evaluated to clearly identify what the Interim Uses are, the anticipated duration of operation(s), the associated Site area of impact, and the required protection measures that will be put in place in order for the Interim Uses to be conducted safely.

On resubmission of a revised **Section 4.0** to this VRP Preliminary Work Plan (if necessary), NMED VRP will review and respond with comments and will be modified to address NMED VRP comments. Any additional Interim Uses sought by the COA may commence only upon NMED VRP approval and will be followed by proper implementation and documentation of the recommended actions with the appropriate protection measures.

5.0 PROPOSED SAMPLING AND ANALYSIS ACTIVITIES

To execute Site redevelopment, the COA is seeking to complete all necessary requirements to receive from NMED: (1) a COC or CCOC for the Site or designated areas of the Site that addresses the soil, soil vapor, and groundwater concerns identified for the Site; and (2) a CNS for a potential developer and/or lessor that addresses all environmental media potentially impacted by former Site operations.

To achieve Site redevelopment under the NMED VRP, the following S&A activities are proposed for the Site that will be completed by COA and/or its contractors:

- **S&A Activity 1 –** Soil Characterization during Construction
- **S&A Activity 2 –** Groundwater Investigation and Annual Groundwater Monitoring
- **S&A Activity 3 –** Subsurface Soil Gas Characterization
- **S&A Activity 4 –** ACBM and LBP Survey of Site Buildings and Structures Prior to Construction
- **S&A Activity 5 –** Soil Characterization and Disposal During Construction

Upon completion, results of the VRP S&A activities will be provided to NMED as part of the final Voluntary Remediation Completion Report. Details regarding these S&A activities are provided in the following subsections.

5.1 Soil Characterization During Construction

Soil characterization during construction will be limited to soil sampling in the areas of subsurface utility lines and major excavation areas. Soil samples will be screened in the field using a photoionization detector (PID) and a hand-held X-Ray Fluorescence (XRF) unit. Areas of soil contamination identified to exceed NMED SSLs will be remediated as outlined in **Section 5.5.** Current proposed soil removal areas are shown on **Figure 3.**

5.2 Groundwater Investigation and Annual Groundwater Monitoring

Fluid level data will be collected from all Site wells, and all Site wells will be sampled and analyzed on an annual basis for the parameters listed below. In addition, an assessment of screened intervals should be conducted once groundwater levels are measured. Following this initial assessment, groundwater samples will be collected from all Site monitoring wells on an annual basis.

The COA proposes to install two upgradient groundwater monitoring wells. The proposed locations are shown on **Figure 4.** Two soil borings will be advanced. The soil borings will be a minimum of 6-inches in diameter and continuous soil samples will be collected from each soil boring. The locations of the borings were selected to define the upgradient conditions of the dissolved-phase plume.

The drilling of each soil boring shall be completed using a truck-mounted, low-clearance HSA drilling rig (CME 75 [high torque], 85, 95, or equivalent) equipped with a minimum 6-inch diameter auger. Additionally, to confirm each soil boring location is clear of subsurface utilities, the proposed locations will be cleared by hand digging to approximately 5 ft bgs. Due to drilling in an active roadway and reducing traffic to one-lane, traffic control is necessary, and flaggers will safely direct traffic around work areas.

The targeted depth of the monitoring wells is 35 ft bgs but monitoring well completion depths will be determined based on the measured depth to groundwater at the time of soil boring installation.

The soil borings will be continuously sampled using either a 5-ft-long continuous sampler or an 18-inch-long (or 24-inch-long) split-spoon sampler. Soil samples will be screened for the presence of VOCs using a PID equipped with a 10.6-electron volt lamp. PID readings and a geologic description of the sample will be recorded on the soil boring log maintained by the INTERA scientist or engineer. Soil cores will be lithologically logged in accordance with ASTM Standard D 2488–17e1, *Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)* (ASTM, 2017). A maximum of one soil sample from each soil boring will be submitted for laboratory analyses. A copy of the soil field screening SOP is provided in **Appendix B.**

One soil sample will be collected per boring and submitted for laboratory analyses and analyzed for the Site contaminants of concern. The selection of the samples to be analyzed will be based on field screening results, i.e., the samples having the highest PID readings (or staining/olfactory evidence) will be selected for laboratory analyses. If the PID does not detect any volatile organic vapors and no other evidence of impact is observed, only one sample collected directly above the water table will be submitted. Samples selected for VOC analyses will be extracted in the field with methanol.

Soil cuttings generated during drilling activities will be containerized in US Department of Transportation (DOT)-approved 55-gallon steel drums and labeled accordingly. Two waste profile soil samples from the drummed soil cuttings will be submitted for laboratory analysis of VOCs, TPH, and RCRA metals. Drummed soil cuttings will be transported by a licensed contractor and disposed of at a licensed facility. Drums will be temporarily stored on-Site in a COA-approved staging area pending laboratory analyses prior to removal and disposal.

The two soil borings will be converted to permanent groundwater monitoring wells using methods detailed in ASTM Standard D5092-04, *Standard Practice for Design and Installation of Ground Water Monitoring Wells* (ASTM, 2004).

Each soil boring will be completed as a 2-inch diameter monitoring well, constructed of 2-inch diameter, flush-threaded, schedule-40 PVC, with 15 feet of 0.020-inch slot screen (10 ft below the water table and 5 ft above the water table) and blank casing to the ground surface. Each monitoring well annulus will be backfilled with 10/20 silica sand (filter pack) to approximately 2 ft above the top of the monitoring well screen. Approximately 3 ft of hydrated bentonite clay chips will be placed above the sand pack. Neat cement grout (95% cement and 5% bentonite powder) will be placed above the bentonite seal to approximately 3 ft below grade. The remaining monitoring well annulus and surface pad will be completed with cement. Each monitoring well will be installed with flush-grade, traffic-rated wellhead completions. The wellhead completion will consist of an 8-inch, flush-grade, traffic-rated well vault installed within a 2-ft by 2-ft by 4-inch-thick cement pad.

The new monitoring wells will be developed immediately after well installation and at least 48 hours prior to sampling. Prior to development, monitoring wells will be gauged with an oil/water interface probe to determine if light nonaqueous phase liquid (LNAPL) is present. If LNAPL is not present, monitoring wells will be developed by bailing and pumping for a maximum of one hour. Water produced during development will be discharged to an impermeable surface near the originating well. If LNAPL is present in the monitoring well(s), development will not occur. The temperature, pH, specific conductivity, and relative turbidity (visual method) of the water will be measured and recorded during development.

A survey of the two new monitoring wells (proposed MW-12 and MW-13) and existing wells MW-01 through MW-11 (thirteen [13] monitoring wells total) will be completed after monitoring well installation is completed. The monitoring wells will be located vertically to the nearest 0.01 foot relative to the North American Vertical Datum of 1988 (NAVD 88). The horizontal location of the monitoring wells will be surveyed to the nearest 0.1 foot using the North American Datum of 1983 (NAD 83) and either the State Plane or Latitude-Longitude coordinate system.

The groundwater monitoring well network (13 monitoring wells) will be sampled on an annual basis for a period of at least two (2) years. The groundwater sampling will be scheduled to follow the well installation allowing the newly installed monitoring wells to stabilize for 48-hours following well development. The following activities will be performed:

- Contact the COA Project Manager and the NMED VRP Project Manager at least four days prior to monitoring and sampling.
- Secure transportation and equipment (a vehicle, gauging and sampling equipment, a field logbook, a camera, a tool kit, the Site-specific HASP, etc.).
- Review the HASP and conduct daily safety briefings.

- Remove caps from all monitoring wells to relieve pressure caused by a fluctuating water table.
- Gauge depth to water and depth to LNAPL (as applicable) at groundwater monitoring wells MW-01 through MW-13.
- Collect groundwater samples from 13 monitoring wells with sufficient water and not containing LNAPL and analyze for the constituents outlined below.

Due to groundwater sampling in an active roadway, traffic control is necessary, and flaggers will safely direct traffic around work areas.

The groundwater monitoring wells will be purged a minimum of three saturated well-casing volumes using single-use disposable bailers. In the event that a well is purged dry, the well will be sampled immediately after a sufficient volume of water has recharged into the well to fill sample containers. During purging activities, groundwater quality parameters (specific conductivity, temperature, and pH) will be monitored for stabilization using a YSI 556 MPS water quality meter or similar water quality meter. Groundwater samples will be collected directly from the disposable bailer using an attachable volatile organic analysis collection tip.

Groundwater samples collected for analysis will be placed in 40-milliliter volatile organic analysis vials with zero headspace and preserved with mercuric chloride ($HgCl_2$). All purged water will be discharged on an impermeable surface so that it will evaporate and will not infiltrate into subsurface soil.

Groundwater samples will be submitted for the following laboratory analysis:

- VOCs via EPA Method EPA Method 8260;
- EDB (1,2-dibromoethene) via EPA Method 504.1;
- TPH DRO and MRO via EPA Method 8015B;
- Dissolved Metals: barium, chromium, copper, iron, lead, manganese, and zinc via EPA Method 6010C/200.7/6020/200.8; and,
- PAHs via EPA Method 8310.

After collection, all groundwater samples will be labeled and immediately packed in an ice-chilled cooler for transport to the contract laboratory for analyses. Proper chain-of-custody procedures will be adhered to during sample collection, transport, and delivery.

A comprehensive investigation and groundwater monitoring report will be submitted upon completion of the field activities, receipt of analytical data, and data evaluation. The report will summarize field activities, measurements, and laboratory analytical results (historical and present) associated with groundwater monitoring and sampling. The report will contain the following information:

- Introduction, Scope of Work, and Work Plan Deviations
- Project Description
- Description of Field Activities
- Summary of Data
- Conclusions
- Recommendations
- Figures:
    - Site Plan
    - Soil Concentration Map
    - Potentiometric Surface Map
    - Distribution of VOCs in Groundwater
    - Hydrographs and Temporal Concentration Trends
- Tables:
    - Fluid Level Measurements
    - Laboratory Results (soil and groundwater), including historical groundwater data for existing wells
- Appendices:
    - Soil Boring Logs
    - Monitoring Well Construction Diagrams
    - Field Notes and Forms
    - Laboratory Analytical Reports

The annual groundwater monitoring reports will follow this same outline but will not contain the initial investigation/monitoring well installation activity information.

Annual groundwater monitoring activities will be conducted for a period of at least two years. If any groundwater concentrations of contaminants of concern are identified above their respective New Mexico Water Quality Control Commission (NMWQCC) Standard(s) after this two year period, groundwater monitoring activities as described above will continue on an annual basis until there are no NMWQCC exceedances.

5.3 Subsurface Soil Gas Characterization

Soil Vapor Pins™ have been installed in all the buildings that will remain at the Site. The Vapor Pin™ sampling devices were installed in the concrete floor of each building according to the Vapor

Pin™ standard operating procedure (SOP). A completed copy of the Vapor Pin™ SOP is included in **Appendix C.**

The objective of any additional sub-slab soil vapor sampling is to determine if VOCs are present in soil vapor below the concrete slab at any of the Site buildings. The soil vapor samples will be compared to NMED-established industrial/occupational use vapor intrusion screening levels (VISLs) (NMED, 2017). Soil vapor samples will be collected in the northern area using summa cannisters with detection limits low enough to compare to the NMED VISLs for residential and industrial/occupational use. Soil vapor sample analysis and sampling schedule for the southern buildings will be proposed in subsequent VRP Work Plans.

A map of the previous soil gas sampling locations is shown on **Figure 6.** Future soil gas samples will be collected at either these locations or nearby these previous sampling locations.

Sub-slab soil vapor samples will be collected on an annual basis in the northern area. Each soil vapor sample will be collected using clean, dedicated Teflon-lined polyethylene tubing, and laboratory-provided, dedicated 6-liter (6L) stainless-steel summa cannisters equipped with 8.0 hour orifices (regulators). The soil vapor samples will be analyzed for VOCs via EPA Method TO-15.

Soil vapor will be purged at each sampling location using a $CO_2/O_2/CH_4$ meter until readings stabilize, and then field analyzed using a PID prior to sample collection. The soil vapor sample is then collected by attaching the tubing from the Vapor Pin™ to the summa cannister. In some cases, soil vapor samples may be collected from traditional soil vapor sampling points where Vapor Pins™ are not present or have been damaged/plugged/removed since previous sampling events.

Soil vapor samples are collected through Teflon lined polyethylene tubing attached directly to the Vapor Pin™. The tubing is then connected to a three-way valve which is then connected to the hand-held sampling units and/or the collection vessel (summa cannister).

PID field screening of the soil vapor samples will be conducted using a PID equipped with a 10.6 eV lamp. The PID will also utilize an in-line water trap to eliminate water vapor from entering the PID. Soil vapor will be evacuated from the tubing using the PID internal pump and analyzed for the presence of volatile organics. The PID will provide a reading of volatile organics in ppmv. PIDs screen ionizable organic compound concentrations in air in ppmv for total ionizable organic compounds detected. PIDs detect VOC concentrations at levels between 0 to 1,000 ppmv, with a minimum detection of 0.1 ppmv.

A sample label will be affixed to each summa canister. The label will be completed with the following information written in indelible ink:

- Project name and location,
- Sample identification number,
- Date and time of sample collection,
- Sample collector's initials, and
- Analysis required.

After labeling, each summa cannister will stored in dark plastic bags placed in coolers to protect the sorbent tubes from any damage that may occur in the field or in transit. In addition, coolers insure the integrity of the samples by keeping them at a cool temperature and out of direct sunlight. The soil vapor samples will be delivered to the contract laboratory each day for immediate preparation and analysis.

The soil vapor samples will be collected using stainless steel Vapor Pins™ installed in the concrete floor. Soil vapor will be collected at each Vapor Pin™ from immediately below each building's concrete slab. The Vapor Pins™ will be completed with stainless-steel covers so subsequent sub-slab soil vapor samples can be collected if desired and/or necessary. In some cases, soil vapor samples may be collected from traditional soil vapor sampling points where Vapor Pins™ are not present or have been damaged/removed since previous sampling events.

At the conclusion of field activities, the soil gas sampling results will be documented in the annual groundwater monitoring report and will include a summary table of the applicable soil vapor results, comparison to NMED VISLs for residential and industrial/occupational use (NMED, 2017), and any recommendations for either further Site assessment, indoor air monitoring, or implementation of engineering controls within and/or below a specific building.

5.4 ACBM and LBP Survey of Site Buildings and Structures Prior to Construction

No further ACBM/LBP sampling activities are anticipated. The COA has conducted significant ACBM/LBP sampling efforts at the Site and have a good understanding of all building materials that either contain ACBM and/or are coated with LBP. Specific ACBM/LBP abatement plans have been prepared for certain Site buildings. As redevelopment and use of the other buildings are planned, ACBM/LBP abatement plans will be developed that can be used by the COA and future occupants to guide maintenance, renovation, and demolition activities.

5.5 Soil Characterization and Disposal During Construction

The intent of this S&A activity is to ensure proper handling and disposition of Site soils during construction and to illustrate that remaining Site soils meet the NMED VRP performance standard as described in **Section 3.1** above.

Based on current Site knowledge, the following NMED VRP activities are proposed for the Site:

- Evaluation of Site soils excavated and removed as part of building construction via field screening and laboratory analysis, as required.
- Segregation, characterization, and transportation of PCS and Site debris potentially encountered and exhumed as part of building construction.
- Proper management and disposal of PCS and exhumed Site debris encountered as part of building construction, as required.

These activities are planned to be executed in tandem with redevelopment construction activities.

The potential for residual pockets of contamination in Site soils represents a particular concern for the COA with respect to both construction worker exposure and the potential generation of hazardous waste. To ensure proper handling and disposition of PCS is executed during Site redevelopment, the COA will develop an Impacted Soil Contingency Plan as an Appendix to the Final VRP Work Plan. The Impacted Soil Contingency Plan will outline the approach the COA will use to initially characterize and dispose of impacted soil if impacted soil is encountered during Site redevelopment. A brief overview of the proposed Impacted Soil Contingency Plan is provided below.

During construction, all potential impacted soil encountered shall be removed and segregated for further assessment. Potential impacted soil shall be identified in the field using both visual and olfactory cues and PID field screening methods. If encountered, the areal extent of potential impacted soil will be established via use of the heated headspace method for VOC field screening using a hand-held PID. Excavated soil resulting in PID readings in excess of 100 ppm shall be segregated from other excavated soil until further analysis can be performed to characterize the soil for regulatory-appropriate disposal.

Analysis of segregated soil shall be conducted in compliance with NMED Solid Waste Bureau (SWB) regulations as follows: One representative soil sample shall be collected per 100 cubic yards of potentially impacted soil material exhumed. Upon collection, all representative samples shall be submitted to an approved laboratory for analysis of the following:

- VOCs via EPA Method 8260;
- TPH-GRO, -DRO, and -MRO via EPA Method 8015 modified;
- PAHs via EPA Method 8310;
- EDB via EPA Method 504.1; and,
- Metals: barium, benzene, chromium, copper, iron, lead, manganese, and zinc via EPA Method 6010C/200.7/6020/200.8.

Analytical results obtained from these samples will be further evaluated against applicable NMED SSLs and EPA RSLs to establish the presence or absence of contamination or specialty waste.

If the presence of impacted soil is affirmed, the Site Developer will remove the impacted soil from the Site for disposal at an approved facility.

COA has identified both the COA Cerro Colorado Landfill in Albuquerque and the Waste Management Landfill Facility in Valencia County as local facilities currently approved to accept impacted soil. Once a disposal facility is selected based on volume and concentration, the COA or Site Developer will submit a report to the NMED SWB to document the excavation, transportation, and disposal of impacted soil and/or buried debris. At a minimum, the report shall include information about the following:

- The company performing the excavation and transportation work
- The disposal facility
- The quantity of impacted soil and/or buried debris removed
- Applicable screening levels
- Laboratory analytical results
- Shipping manifests (or truck weight tickets)
- Date(s) of work

The report will also include a photograph log.

This VRP Work Plan proposes removing soil from the battery storage area, the sand blasting area, and a few hot spot areas as shown on **Figure 3.** The soil will be removed and characterized as outlined above.

6.0 PROPOSED REMEDIATION ACTIVITIES

A comprehensive review of previous Site investigations and associated analytical data was completed to determine the current status of environmental conditions for the Site.

1. Soil Removal: Soil contamination is present at the Site in several areas from the ground surface to a depth of 10 ft bgs. The COA will removal contaminated soil in those areas of the northern COA Rail Yards indicated on **Figure 3** (battery storage area and sand blasting area). The volume of soil proposed to be removed is approximately 7,000 cubic yards. The soil will be removed, transported, and disposed of as outlined in **Section 5.5** of the VRP Preliminary Work Plan.

The actual volume of contaminated soil to be excavated at the Site will be determined in the field and will be based on the criteria discussed in this section. The primary criteria to be used in excavating contaminated soil are health and safety of the workers, occupants of nearby buildings, and pedestrians.

Other criteria that will be used to decide what soil is excavated include the following:

- Depth of the excavation
- Concentration of VOCs

The excavation will not extend below a depth of 10 feet. The soil data collected from previous contaminant investigations indicates that an excavation below this depth is not warranted.

The main criteria that will be used to decide what soil is to be excavated are the concentrations of metals (XRF) and VOCs (PID) in soil.

The excavation areas illustrated in **Figure 3** are intended to be general in nature. The final footprint of the excavation is anticipated to coincide with the areal extent of soil contamination. The actual area to be excavated will be determined in the field by an INTERA scientist.

Stockpiling of contaminated soil overnight is not anticipated. If any PCS is stockpiled, the COA will cover the contaminated soil pile with 6-mil polyethylene sheeting that is anchored to prevent wind damage or erosion. Berms will be constructed to prevent liquid leaching from the contaminated soil stockpile.

The COA will load all excavated contaminated soil into trucks and transport the contaminated soil to the Cerro Colorado landfill in Albuquerque, New Mexico for disposal. The contaminated soil from the Site will need a profile prior to waste hauling.

Soil samples will be collected by COA personnel from every 100-cy removed from the Site (prior to the contaminated soil being removed from the Site) and the samples will be analyzed for the COCs.

The contractor will provide a COA-signed copy of the waste manifest for each load to the COA daily. The contractor will be responsible for keeping all weight tickets and providing copies of these to the COA on a daily basis as well. The contractor will transmit copies of the waste manifests and weight tickets to the COA. The COA will provide copies of the waste manifests to NMED as part of the Final Voluntary Remediation Completion Report developed for the Site.

The contractor will load the trucks only to their rated capacity or regulatory limits. The contractor is also responsible for providing all labor and materials necessary to remedy situations involving spills or accidents involving vehicles in transit.

2. Installation of Engineering Controls: Soil contamination is present at the Site in distinct areas at depth (greater than 10 ft). Although these deeper soils do not typically pose an exposure risk for construction workers, if redevelopment plans include excavations to depths greater than 10 ft bgs, potential exposure to PCS will need to be considered. Even if no excavation exceeding 10 ft bgs is planned, deeper PCS at the Site poses an additional human health concern of vapor intrusion (southern half of the Site). For these areas, construction plans for enclosed structures may need to consider the additional installation of engineering controls (vapor intrusion membranes or passive soil vapor venting systems) if additional characterization efforts for soil vapor indicate concern. The installation of vapor intrusion membranes is more likely to occur within the southern half of the COA Rail Yards.
3. Immobilization/Containment of ACBM and LBP Materials: A determination will need to be made once the redevelopment is decided upon as to whether ACBM/LBP abatement or encapsulation needs to occur for those building materials testing positive for the presence of ACBM/LBP. The final building renovation design will have to take these materials into account and a decision will have to be made as to their final deposition. Any remaining ACBM and/or LBP will need to be documented, and a management plan will need to be developed governing how these materials should be handled following renovation activities.

4. Groundwater Remediation: Active groundwater remediation is not proposed at this time. The COA will continue to monitor groundwater by collecting Site groundwater samples on an annual basis. After two years of annual groundwater monitoring, the COA will re-evaluate groundwater concentrations and determine if monitored natural attenuation (MNA) parameters show if sufficient degradation is occurring or if more aggressive

remediation methods are necessary. As stated above, the southern half of the COA Rail Yards and this contamination will be addressed in future VRP Work Plans.

Implementation of one or more of these remedial actions to address residual soil contamination at the Site will be through a phased approach and will depend on the results of the associated proposed additional characterization and sampling efforts (**Section 5.0**).

Analytical results obtained for soil collected at the Site confirm that residual soil contamination is present and, if encountered, this contamination source will be addressed upon excavation and removal of soils as part of the construction planning and oversight as detailed in **Section 5.5** above.

Analytical results obtained for groundwater collected at the Site indicate that groundwater contamination is present as a result of past Site use(s); however, depth to groundwater is documented at approximately 22 ft bgs to 25 ft bgs. Maximum excavation will be 15 ft or less. Groundwater is not anticipated to be encountered by construction during Site redevelopment, and no further action regarding groundwater assessment or remediation is required.

Current Site buildings will be demolished. The location of the buildings to be razed are shown on **Figure 5.** Prior to demolition, all ACBM will be removed and disposed of according to local and state regulations. The following buildings are listed to be demolished in the Site Master Plan within the northern half of the COA Rail Yards:

1. North End of Flu Shop
2. North Wash Room
3. Sheet Metal House
4. Cab Paint Shop
5. Pattern House

The City plans to demolish the Sheep Metal House and the North Wash Room and has yet to make a final determination of which remaining buildings will be demolished. The other buildings within the northern half of the COA Rail Yards will be remediated and remodeled per the COA redevelopment plan.

Several of the buildings within the Site contain wood block floors. The wood blocks are coated with creosote. Previous sampling of the wood blocks has indicated that the wood blocks can be disposed of as municipal solid waste. As demolition and renovation of buildings at the Site occurs, the wood blocks will be disposed of accordingly.

7.0 DISCUSSION OF HOW PROPOSED ACTIVITIES WILL MEET THE VRP PERFORMANCE STANDARD

Performance requirements for projects participating in the NMED VRP program are described in NMAC 20.6.3.10 (NMED, 2001). These performance standards involve four specific activities to meet NMED VRP requirements: (1) identify the problem, (2) quantify the risk, (3) verify the need for remedial action, and (4) identify the remedy. Details regarding how already-completed or currently proposed Site assessments provide sufficient information to support conclusions regarding these activities are discussed further in the subsections below.

7.1 VRP Performance Standard Objective 1

Performance Standard Objective 1 can be defined as identification of "*the source, nature and extent, migration pathways, and environmental fate and transport of contaminants in all environmental media present at the site (i.e., soil, groundwater, surface water, sediment, and/or air).*"

Characterization sampling and analysis efforts were performed at the Site for soil, groundwater, and/or soil vapor. Results of these efforts identified the following:

- Nature of contamination – TPH, VOCs, PAHs, and metals
- Potential contaminant source – Historical Rail Yard operations
- Extent of contamination – Documented areas of soil and groundwater contamination;
- Migration pathways – Leaching; particulate suspension in air; groundwater movement; and soil vapor migration
- Fate and transport of contaminated media – Dermal contact, ingestion, and inhalation

7.2 VRP Performance Standard Objective 2

Performance Standard Objective 2 can be defined as quantification of "*the risk of harm posed by the site to human health, safety, and the environment.*"

Three environmental Site media have been identified as being potentially impacted by one or more COPCs: soil, soil vapor, and groundwater. The relative degree of risk posed by exposure to these COPCs at the Site has been established by directly comparing Site COPC concentrations to applicable standards currently available at the time of comparison. Applicable standards used for this comparison include:

- Soils
    - NMED SSLs (NMED, 2017)

- Groundwater
    - NM-GS (NMED, 2004)
    - EPA maximum contaminant levels [MCLs] (EPA, 2009)
- Soil Vapor
    - NMED VISLs (NMED, 2017)

During Site redevelopment activities, impacted soil, if encountered, will be determined by directly comparing Site COPC concentrations to the most currently available applicable standards for soil.

7.3 VRP Performance Standard Objective 3

Performance Standard Objective 3 can be defined as verification for "*the need to conduct remedial actions at the site to safeguard against such risks.*"

Any potential exposure to impacted soil during construction activities shall be addressed as part of the Impacted Soil Contingency Plan for the Site.

Analysis of Site groundwater data collected indicate that groundwater has been impacted by former Site operations (INTERA, 2017).

Sub-slab soil vapor data indicates that contaminants were identified above NMED VISLs.

The presence of petroleum hydrocarbon contamination in the vadose zone soil and in the dissolved phase can contribute to soil vapor. If the magnitude of these contaminants is great enough, vapor intrusion could result.

Site soil vapor issues will be handled by the use of vapor intrusion liner(s) below any new buildings if warranted. Vapor venting systems may need to be installed around existing structures depending on sample results and future redevelopment scenarios. Any potential subsurface parking garages will be further vented by air exchange rates typically used by below-grade parking structures.

7.4 VRP Performance Standard Objective 4

Performance Standard Objective 4 can be defined as identification of *"the remedial action selection and design, if appropriate."*

The COA will oversee the excavation of Site soils during redevelopment to meet the performance objectives of the VRP. Per the proposed performance standard for soil as described in **Section 3.0,** if any constituent is found to be present in Site soils at a concentration that exceeds an applicable standard (as detailed in this document), the remediation contractor will handle impacted soils as outlined in **Section 5.0** of this Preliminary VRP Work Plan.

Soil vapor issues will be handled as described in **Section 7.3.**

No groundwater remediation is proposed for the Site.

8.0 REFERENCES

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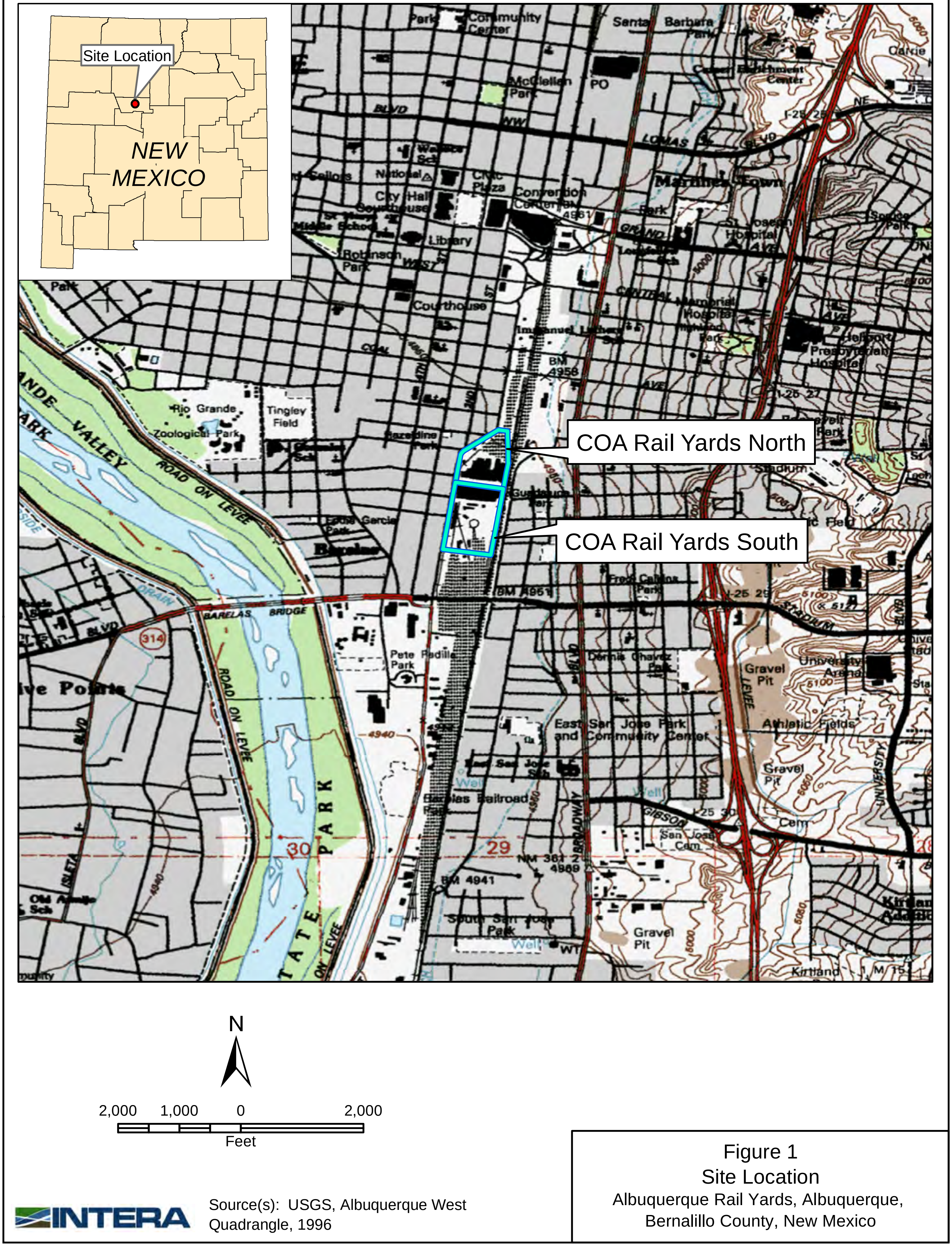
FIGURES



Source(s): USGS, Albuquerque West Quadrangle, 1996

Figure 1
Site Location

Albuquerque Rail Yards, Albuquerque, Bernalillo County, New Mexico

S:\ABQ\COA On-Call 2014\Task_17_Railyard_VRP\Graphics\GIS\MapDocs\WorkPlan_2018\01_SiteLocation.mxd 12/17/2018

Figure 2
Site Plan
Albuquerque Rail Yards, Albuquerque, Bernalillo County, New Mexico

INTERA

Source(s): Aerial – BERNCO GIS website, dated 2016.

Document Path: S:\ABQ\COA On-Call 2014\Task_17_Railyard_VRP\Graphics\GIS\MapDocs\WorkPlan_2018\02_sitePlan.mxd 12/18/2018

⌖ Monitoring Well

▦ Property Boundary

☐ Site Feature

☐ Demolished or Currently Scheduled to be Demolished

🟦 Proposed Soil Removal Area

🟪 Potential Hot Spot Removal Area

🟧 Cover and Manage "In Place"

☐ Removal Area

N

160 80 0 160

Feet

Source(s): Aerial – BERNCO GIS website, dated 2016.

Figure 3
Proposed Soil Removal Areas
Albuquerque Rail Yards, Albuquerque, Bernalillo County, New Mexico

INTERA

Document Path: S:\ABQ\COA On-Call 2014\Task_17_Railyard_VRP\Graphics\GIS\MapDocs\WorkPlan_2018\03_SoilRemoval.mxd 1/11/2019

Figure 4
Proposed Monitoring Wells
Albuquerque Rail Yards, Albuquerque, Bernalillo County, New Mexico

INTERA

Source(s): Aerial – BERNCO GIS website, dated 2016.

Document Path: S:\ABQ\COA On-Call 2014\Task_17_Railyard_VRP\Graphics\GIS\MapDocs\WorkPlan_2018\04_ProposedMWs.mxd 1/14/2019

N

160 80 0 160

Feet

☐ Site Feature

▦ Property Boundary

🟥 Building Demolition

Figure 5
Asbestos/LBP

Albuquerque Rail Yards, Albuquerque,
Bernalillo County, New Mexico

INTERA

Source(s): Aerial – BERNCO GIS website, dated 2016.

Document Path: S:\ABQ\COA On-Call 2014\Task_17_Railyard_VRP\Graphics\GIS\MapDocs\WorkPlan_2018\05_AsbestosLBP.mxd 1/11/2019

Figure 6
Soil Vapor Sample Locations,
Rail Yards North
Albuquerque Rail Yards, Albuquerque,
Bernalillo County, New Mexico

Source(s): Aerial – BERNCO GIS website, dated 2016.

Document Path: S:\ABQ\COA On-Call 2014\Task_17_Railyard_VRP\Graphics\GIS\MapDocs\WorkPlan_2018\06_SVnorth.mxd 12/19/2018

APPENDIX A
LEGAL DESCRIPTION

APPLICATION EXHIBIT "A"
STATE OF NEW MEXICO ENVIRONMENT DEPARTMENT
VOLUNTARY REMEDIATION PROGRAM
LEGAL DESCRIPTION OF THE TRACT OF LAND FOR WHICH VOLUNTARY REMEDIATION IS PLANNED

I. Tract of Land Comprising Site.

The "Albuquerque Old Locomotive Shop" (former A.T. & S.F Railway Company Machine Shop) is a 27.32 acre tract, more or less, out of the Tract A of the Plat of Tract "A", A.T.&S.F. Railway Company Machine Shop, 27.321acre tract located at 2nd and Bridge in Albuquerque in Bernalillo County, New Mexico. Said 27.32 acre voluntary remediation site is more particularly described as follows:

Tract "A" of the Plat of Tract A, A.T. & S.F. RAILWAY COMPANY MACHINE SHOP, Albuquerque, Bernalillo County, New Mexico, as the same is shown and designated on the Plat filed in the office of the County Clerk of Bernalillo County, New Mexico on January 25, 1996 as Document Number 1996008744, recorded in Vol. 96C, folio 44, records of Bernalillo County, New Mexico.

APPENDIX B

FIELD SCREENING STANDARD OPERATION PROCEDURE

23.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) covers commonly-applied field screening techniques that may be utilized by INTERA field personnel to assess particular constituents of concern (COCs) in soil during characterization, remediation, or monitoring of environmental sites as follows:

- Organic vapors via a photoionization detector (PID) or flame ionization detector (FID); and
- Metals via a hand-held x-ray fluorescence (XRF) analyzer.

Utilizing field screening techniques to evaluate the relative degree to which COCs are present during active soil recovery and collection allows a field sampling team to assess:

1. The presence and relative degree to which certain COCs (e.g. volatile organic compounds [VOCs]) are in soil to assess environmental quality;
2. Ambient air quality to assess potential risk (contaminant exposure or explosive hazard from organic vapors or metal particulates) to the field team; and
3. Effluent air stream of treatment systems for regulatory compliance and assess remedial progress.

The procedures included in this SOP focus on field screening and monitoring techniques utilized by field personnel to complete **Objective 1** and **Objective 3** above; field screening, monitoring systems, and associated procedures for the purposes of health and safety (**Objective 2**) are addressed in INTERA SOP 1, *Air Monitoring*, or in the Site-Specific Health and Safety Plan, as applicable.

This SOP should be used in conjunction with procedures presented in the INTERA Site-Specific Health and Safety Plan (SSHASP) and in site-specific investigation planning documents such as a Sampling and Analysis Plan (SAP), Field Sampling Plan (FSP), or Work Plan (WP). This SOP will also be commonly used with and is referenced in the following INTERA SOPs:

- 1, *Air Monitoring*;
- 2, *Decontamination*; and
- 13, *Soil Sampling*.

23.1 Field Screening for Organic Vapors

Field screening of volatile-impacted soil samples is typically performed using either a portable PID or an FID. Professional judgment and site-specific considerations is often the primary method by which an appropriate field instrument (PID vs. FID) is chosen. Factors to consider include, but are not limited to, age of the contaminant source (i.e., underground storage tank [UST] system), soil characteristics, and extent of contaminant degradation.

The applicable manufacturer's instructions shall be available and referenced for equipment calibration, use, and maintenance requirements. **NOTE:** Never use a PID, FID or any other accepted field test instrument unless the instrument is calibrated in the field and in good working order. Only calibration gases having a standard of 100 ppm or greater should be used to calibrate vapor-detecting field instruments. A brief discussion of the PID and FID is presented in **Section 23.1.1** and **Section 23.1.2**, respectively. General procedures applied in the field by INTERA personnel for detecting the presence and overall concentration of organic vapors are presented in **Section 23.1.3** and **Section 23.1.4**.

23.1.1 Application of a Photoionization Detector

PIDs employ ultraviolet radiation to ionize certain chemical molecules. Positive ions and free electrons are formed which migrate to the detector electrode(s), resulting in an electric current that is proportional to chemical concentration at the detector. PIDs are extremely sensitive to aromatic hydrocarbons due to the great efficiency of ionization of pi bonds under ultraviolet radiation. Efficiency of ionization of sigma bonds is lower, resulting in a higher PID detection limit for aliphatic hydrocarbons. The selectivity of the method can be adjusted by selecting lamps of different energies (e.g., 9.6 electron volts [eV], 10.2 eV, 11.7 eV), causing a change in response of chemical with fixed ionization potentials to changing lamp energies.

The PID is usually calibrated using **isobutylene** gas; other VOCs present in soil may produce a relatively greater or lesser response on a concentration basis. This is known as the response factor (RF). Applying a RF allows the user to measure concentration(s) of various gases without actually calibrating the sensor with the targeted gas/COC.

Some considerations when applying a PID for the field screening of organic vapors:

- Cannot be used to detect the presence of methane. Methane has an ionization potential higher than the energies of commercially available lamps.
- If the objective is to monitor for petroleum hydrocarbon vapors, a PID equipped with a 10.2 eV lamp is suitable. If the objective is to monitor for chlorinated compounds, a PID equipped with an 11.7 eV lamp is required.
- Sensitive to analytic interference: For soils containing both low- and high-sensitivity (aromatic) compound(s), the PID tends to conceal the presence of any low-sensitivity compound(s).
- Sensitive to the presence of water vapor: Condensation of water vapor in the lamp can cause fogging and reduced sensitivity of the PID that may result in readings not representative of true soil conditions.
- Readings are relative to the ambient air temperature; the same sample will yield a higher concentration reading at a higher ambient air temperature than at a lower air temperature.

- Can be sensitive to changes in air currents so reading should be performed in a relatively controlled environment or protected area.

23.1.2 Application of a Flame Ionization Detector (FID)

FIDs generate electrical current when gases containing carbon atoms are oxidized to carbon dioxide in a hydrogen flame and potential is applied across the flame. The magnitude of the electric current generated is termed the detector response. FIDs are responsive to hydrocarbon contaminants in a vapor stream and are commonly employed for this purpose. FIDs are durable for field use, and have a wide linear range and nearly uniform response to organic gas species. FIDs are generally unresponsive to inorganic gases and water vapor.

Some considerations when applying a FID for the field screening of organic vapors:

- Although versatile, these detectors are not selective for halogenated compounds (i.e., chlorinated solvents).
- Needs a supply of fuel gas (typically hydrogen). Hydrogen gas is extremely flammable and potentially explosive, therefore, careful safety practices in handling and flame ignition is required.

23.1.3 Procedure – Measurement of VOCs upon Soil Recovery

This section describes the general method of “pass-over” field screening applied by INTERA personnel at sites where VOCs are anticipated in soils to be recovered for further sample characterization, collection, and analysis. Pass-over field screening techniques are often implemented at sites where monitoring of specific VOCs for health and safety purposes may also be required. Depending on the scope of the sampling effort, recovered soil may receive pass-over field screening as well as heated headspace analysis (**Section 23.1.4**); the results of the pass-over field screening may provide direction as to which interval(s) of the recovered soil are of primary interest to collect for additional field screening (**Section 23.1.4**) or offsite analysis.

Pass–over field screening is performed fairly quickly and should be performed immediately upon recovery and exposure of the soil core or cuttings to ambient surface conditions. If discrete soil cores are recovered, an incision with a clean knife or equivalent is made down the length of the sample. The PID or FID sensor is then passed along the length of the incision while partially covering the incision with a hand, and the readings noted in the field notebook and/or on an appropriate field form.

23.1.4 Procedure – Measurement of VOCs via Heated Headspace Analysis

The primary method by which selected intervals of soil are measured for the presence of organic vapors is by application of the heated headspace method. This method is used to estimate the total concentration of VOCs in soil and solid samples between 0 and 1000 parts per million (ppm). For this screening procedure to be effective, the range of VOCs of interest at the site must be known in advance (so the appropriate eV lamp may be selected) and must be detectable by the instrument chosen (anticipated concentration levels).

Results of the heated headspace technique are semi-quantitative: As such, it is strongly recommended that a site sampling design applying this technique shall utilize the field screening in conjunction with a fully quantitative analytical method for final decision-making. In many cases, field screening results are applied to guide further sample collection activities, particularly the collection and selection of “most appropriate” soil for VOC samples for offsite laboratory analysis. Results may also be further used to help identify laboratory samples for which low- or high- concentration preparation procedures should be applied. The utility of applying field screening results to a particular investigation shall be detailed in the SAP/FSP/WP.

Prior to conducting any field screening of soils at a site, verify the following activities have been completed in accordance with established in the SAP/FSP/WP.

1. Verify that equipment required for performing the heated headspace screening method is available, cleaned, and in good working order. Equipment needs may include:
    - Personal Protection Equipment (PPE) as specified in the SSHASP;
    - Appropriate soil collection device, such as a spoon, modified plastic syringe or commercially-available hand coring device;
    - Appropriate sample container such as a clean 0.5- to 1-liter or 16-ounce jar;
    - Aluminum foil and rubber bands;
    - Indelible ink or marker for labeling;
    - Water bath and thermometer or other heating source;
    - Portable gas chromatograph;
    - FID, PID, detector tubes, or other acceptable field instruments;
    - Instrument calibration standards and user’s manual; and
    - Appropriate personnel and equipment decontamination supplies (INTERA SOP 2, *Decontamination*).

NOTE: If the ambient temperature at the site is expected to be below 60° Fahrenheit or 15° Celsius, the sample will require a supplemental heating source prior to analysis as the latent heat from the sun will not be sufficient. A thermometer, water heater, and water bath large enough to hold a sample jar, heat source, and deionized water can be applied; however, heated air from the interior of a vehicle is often easier to implement and is also considered an acceptable method. In all cases, the sample should be kept out of direct sunlight to minimize sample oxidation.

2. As applicable, identify established work zone(s) for field screening, decontamination procedures, and sample management. Appropriate field screening and sample management work zones should be established upwind of any vapor contaminant source (e.g., car exhaust) and out of direct sunlight as hydrocarbons can be oxidized by ultraviolet radiation.
3. Verify that the required field screening equipment has been calibrated per the manufacturer's instructions and the results noted in the field logbook and/or on an appropriate field form.

NOTE: For heated headspace analysis, instrument calibration shall be checked/adjusted no less than once every 10 analyses, or daily, whichever is more frequent.

4. Note any action levels for organic vapors as established in the SAP/FSP/WP.

The heated headspace field screening method of choice applied by INTERA field personnel is the recommended method published by the New Mexico Environment Department (NMED) Petroleum Storage Tank Bureau (NMED, 2000). This method is a variation of EPA's SW846 Field Screening Analytical Method 3815 (EPA, 2007a). General field implementation of the heated headspace field screening method for soils is presented as follows.

1. Fill a 0.5 liter/16 ounce or larger clean glass jar half full of soil sample. Plastic bags or other non-glass containers are not acceptable. A smaller jar may be used when sample volume is limited. Take care to consistently obtain a similar sample volume for each field screening sample collected.
2. Cover top of jar with clean aluminum foil. Place a lid ring, rubber band, or equivalent around the jar to effectively seal the sample, taking care not to compromise or damage the aluminum foil during application.
3. Label sample accordingly and note time and depth of collection.
4. Place sample in a protected location where it can reach a temperature between 60°F to 80°F. Avoid placement in direct sunlight as much as possible to prevent sample oxidation (**see notes above**).
5. Allow aromatic hydrocarbon vapor concentrations to develop in the headspace of the sample jar for 5 to 10 minutes. During the initial stages of headspace development, the sample is to be shaken vigorously for one minute.
6. After the allotted time has elapsed, carefully pierce foil seal with the tip of the sensor from either a PID or FID taking care to create as small of a hole in the seal as possible to avoid excessive loss of any accumulated vapors during measurement.
7. Observe the instrument readout and record the highest (peak) measurement (in ppm) in the field logbook and/or on the appropriate field form (if applicable).

23.2 Field Screening for Metals

Field screening for the presence of certain metals in soil and sediment samples is typically achieved by utilizing XRF spectrometry via an XRF analyzer. XRF is the property of a material to emit X-rays (gamma rays) at a unique frequency (energy) upon electron shell reconfiguration as a result of irradiation by external X-rays of sufficient energy derived from a known radioactive source. Certain types of metals, glasses, and ceramics provide strong XRF responses depending on the radiation source utilized.

An XRF analyzer operates by using one or more internal radioactive source(s) (e.g., cadmium-109, americium-241) to generate X-rays and irradiate a soil sample, or other bulk material such as paint, of interest. The analyzer then detects the X-rays emitted from the soil (as a result of the irradiation) and converts these X-rays into an electric current. The strength of the current is proportional to the energy of the X-ray. Since each metal produces an X-ray at a known unique frequency, electronics (microprocessors) in the analyzer use these differing responses to determine the frequency of every X-ray entering the detector (to determine what metals are present), and how many X-rays at each frequency strike the detector over a certain period of time (to determine the concentration of that metal in the sample).

In support of environmental investigation, XRF analyzers are typically used for two main purposes. First, they may be used to rapidly assess site conditions to support a site reconnaissance. Secondly, they may be used to screen large numbers of soil or sediment samples to minimize the number of samples that are either sent to a laboratory to provide detailed site characterization data or require removal. In either case, XRF field screening data is considered semi-quantitative (unless sufficient site-specific laboratory data is available for correlation – See **Section 23.2.4** for further discussion); therefore, field screening results should be used in conjunction with a fully quantitative analytical method for final decision-making purposes.

23.2.1 Application of a Portable XRF Spectrometer

XRF analyzers are available in a variety of sizes and specifications. Hand-held XRF analyzers, such as Geotech's Innov-x XRF analyzer, are considered most appropriate for environmental characterization investigations as these instruments are easily portable and can be used to measure metal concentrations in both in-situ soils as well as soil grab samples with little sample preparation effort. However, because the analyzer uses an internal radioactive source certain procedural precautions must be in place:

- The operator should always be aware of the instrument's radioactive source and the direction of its beam of X-rays.
- The operator should never point the open source at anyone or allow the x-ray to directly contact a body part (such as a hand).
- When handling the analyzer, proper PPE should be applied at all times.

- As appropriate, special training regarding the use of instruments with radioactive sources may be required.
- State registration may be required to rent or be in possession of the instrument.

If an investigation is considering the use of XRF field screening techniques, these considerations as well as any others identified for the site shall be addressed in the SSHASP.

Another primary consideration when performing XRF field screening is the potential for interferences to impact the resulting data quality including sample preparation error, spectral interferences, and chemical matrix interferences.

- **Preparation Error** – The accuracy of the XRF analysis is strongly impacted by sample homogenization. The more homogeneous the sample, the more accurate the results. There is no control of this limitation when conducting in-situ analysis.
- **Spectral Interference** – Each element has a signature spectrum of energies and relative intensities. Many elements, however, produce X-rays of similar energy and discerning which element produced a detected X-ray is a factor of the detector's resolution capability and the software's ability to fit all of the data to the relative intensities produced by the various wavelengths.
- **Chemical Matrix Interference** – Refers to the effect that one element has on another in producing X-rays that reach the detector. Dominant elemental components of a sample, such as silicon in soils, vary in concentration from sample to sample and therefore so does that element's influence on the other elements in the sample.

Other considerations of the XRF field screening method include:

- **Presence of soil moisture** - Excessive soil moisture biases the results low, i.e., the higher the soil moisture in a particular matrix, the lower the reported concentration relative to the actual concentration. This limitation may be overcome by drying the sample. Without sample drying, XRF measurement results for samples with typical soil moistures within the range of 15% - 25% are routinely reported at values less than laboratory confirmation analysis for the same samples (EPA, 2011). The actual difference may vary significantly for all samples from a site but the XRF results reported by the instrument are typically on the order of 70% - 80% of the laboratory reported value for samples in this moisture range. This factor should be taken into consideration when making decisions based on XRF results (see **Section 23.2.4** for further discussion).
- **Lack of sensitivity with respect to certain constituents** – Due to peak overlaps, some constituents may have problematically high detection limits, i.e., detection limits may be higher than project action levels for certain constituents,

limiting its use for rapid field screening for certain elements. One of the most common examples of this phenomenon is the lead/arsenic constituent pair. When lead and arsenic are being analyzed, the peak overlap problem results in detection limits for arsenic that are several times higher than the typical action levels published for this constituent. It is often necessary to perform confirmatory analysis in the laboratory to obtain analytical results for arsenic, or other constituents with high detection limits, to obtain data in the range necessary for making regulatory decisions.

23.2.2 Procedure –Measurement of Metals via Portable XRF Spectrometry

The general method to be implemented by INTERA personnel by which selected intervals of soil (either in-situ or bagged) are field screened for the presence of metals via a hand-held XRF analyzer method is based on EPA's SW846 Field Screening Analytical Method 6200, *Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment* (EPA, 2007b). This method establishes basic quality assurance (QA) requirements, including calibration; verification; and determination of instrument precision, accuracy, and limit of detection and recommends that a minimum of 5% of all samples tested by field XRF techniques be confirmed by an outside laboratory. A copy of EPA Method 6200 is provided as **Attachment 1**; recommended field XRF analyzer operational checks and calibrations requirements based on this method are summarized in **Section 23.2.3**; and additional QA requirements recommended by EPA for an appropriate XRF sampling design are listed in **Section 23.2.4** (EPA, 2011). Which particular XRF analyzer is chosen to use for an investigation and any details regarding appropriate QA requirements when applying XRF field screening techniques at a particular site (in addition to or in place of those outlined in **Section 23.2.3** and **Section 23.2.4** of this SOP) shall be established in the SAP/FSP/WP.

Prior to conducting any field screening of soils at a site, verify the following activities have been completed in accordance with those established in the SAP or equivalent.

1. Verify equipment required for performing XRF spectrometry is available, cleaned, and in good working order. Equipment needs may include:
    - PPE as specified in the SSHASP;
    - Appropriate soil collection device, such as a spoon, trowel, or hand auger;
    - Soil/rock sample preparation tools such as rock hammer; 2-mm, 250-µm, or 125-µm sieve(s), grinder;
    - Stainless steel bowls or equivalent for soil homogenization;
    - Clean, unused zip-closure plastic bags (or equivalent) for bagged samples;
    - Indelible ink or marker for labeling;
    - XRF field instrument;

  - Instrument calibration standards, method blanks, and user's manual; and
  - Appropriate personnel and equipment decontamination supplies (INTERA SOP 2, Decontamination).
2. As applicable, identify established work zone(s) for field screening, decontamination procedures, and sample management.
3. Verify that the XRF analyzer has been calibrated per the manufacturer's instructions and the results noted in the field logbook.
4. Note any action levels for metals as established in the SAP or equivalent.

23.2.2.1 Collection of an In-situ Measurement

This method is best used to obtain a large number of measurements in a relatively short amount of time. However, results obtained using this field screening technique are generally considered less accurate (biased low) than results obtained using the bagged technique (**Section 23.2.2.2**) (EPA, 2007b). Because of the shallow penetration of the X-rays in typical soils, the measured concentrations are representative of the concentrations present at the surface of the material being measured. If conditions representing concentrations over a greater depth are required by project data quality objectives (DQOs) (i.e., on the order of three to six inches), the bagged technique may be more appropriate.

1. Clear measurement location of any significant vegetation, such as large clumps of grass, and level the location to provide a flat surface on which to place the instrument window.
2. Place a thin piece of Mylar® film or equivalent on the measurement location to protect the window and help prevent instrument damage or contamination.
3. Press analyzer window to the ground surface verifying that the entire area of the window is contacting the soil.
4. Collect a reading over a period of 60 seconds or as otherwise identified in the user's manual or the SAP/FSP/WP.
5. Record measurements in field logbook and/or on an appropriate field form (if applicable) in addition to having the instrument store measurements electronically.

23.2.2.2 Collection of an Bagged Sample Measurement

This method is used to measure concentrations of metals in soil and sediment samples collected from a vertical interval, either as a grab or a composite sample.

1. Collect a representative soil or sediment sample in accordance with the appropriate procedures identified in INTERA SOP 13, *Soil Sampling*.

2. Homogenize and mix sample to achieve a relatively uniform particle matrix. Per the SAP/FSP/WP, additional drying, sieving or grinding of the sample may be required prior to analysis (**Attachment 1**).
3. Place homogenized sample in appropriate container. Typically this is a clean, unused quart-sized zip-closure plastic bag (or equivalent).
4. Place sample on a non-metal table or other flat surface. Spread sample out as evenly as possible to create an even and level surface.
5. Place analyzer directly over plastic bag. Verify that the entire area of the window is contacting the soil sample.
6. Collect a reading over a period of 60 seconds or as otherwise identified in the user's manual or the SAP/FSP/WP. The concentrations reported are representative of the interval sampled, i.e., if the sampler collected the sample from the interval of 0 to 3 inches below ground surface, the reported concentration, assuming thorough homogenization, will be an average of the concentrations over that interval.

NOTE: **NEVER** collect a sample field screening measurement while holding the sample as this will cause undue personal exposure to radiation (emitted X-rays).

7. Record measurements in field logbook and/or on an appropriate field form (if applicable) in addition to having the instrument store measurements electronically.

23.2.3 General Operational Checks and Calibrations Requirements

The selected XRF analyzer shall be maintained and operated in accordance with the manufacturer's instructions and associated user's guide (provided as part of the SAP/FSP/WP) and in accordance with the requirements set forth in EPA Method 6200 (**Attachment 1**). Typical procedure includes initial and periodic performance checks, and the analysis of method blanks and field duplicates. Generally recommended operational checks and calibrations requirements are summarized below.

Prior to each operational period:

1. Turn on the XRF analyzer and allow the analyzer to perform an internal auto-calibration.
2. Conduct an initial performance check using the appropriate National Institute of Standards and Technology (NIST)-traceable standard reference material for the COCs. The value should be within +/- 20% of the stated value of the standard.
3. Perform an initial analysis of a method blank to verify the instrument is not registering false positive results for the COCs.

During operation:

4. Record ambient air temperature for each measurement; if a change by more than 10°F is noted, the instrument will require recalibration.
5. Analyze method blank and reference standards once each hour or every twenty (20) samples, whichever occurs first, and at the end of the period of operation.
6. At least once per day and for every twenty samples (or at an alternate frequency specified in the SAP/FSP/WP), analyze a duplicate using the main sampling technique.
7. Once per day, check instrument precision by analyzing one of the site samples at least seven (7) times in replicate.

23.2.4 Basic Quality Assurance Strategies

Based on limiting factors listed in **Section 23.2.1**, EPA recommends development and application of a confirmatory analytical scheme in conjunction with utilizing XRF field screening data (EPA, 2011). Utilizing a combined XRF field screening/metals confirmation sampling analytical approach has been shown to minimize the number of samples required for offsite laboratory analysis, yet still provide definitive level data, with a high degree of confidence, to the decision maker(s).

Unless otherwise identified in the SAP/FSP/WP, the following combined XRF field screening/metals confirmation sampling analytical approach is recommended (EPA, 2011).

1. Submit 10% of all samples with XRF field screening results for one or more chosen COCs less than approximately 70% - 80% (or other correlation factor developed on actual data, if available) of the applicable site action level for offsite laboratory confirmation analysis via the appropriate method as defined in the SAP/FSP/WP.
2. Submit 100% of all samples with XRF field screening results for one or more chosen COCs equal to approximately 70% - 80% (or other correlation factor developed on actual data, if available) of the applicable site action level for offsite laboratory confirmation analysis via the appropriate method as defined in the SAP/FSP/WP.
3. Submit 10% of all samples with XRF field screening results for one or more chosen COCs greater than approximately 70% - 80% (or other correlation factor developed on actual data, if available) of the applicable site action level for offsite laboratory confirmation analysis via the appropriate method as defined in the SAP/FSP/WP.

23.3 References

Environmental Protection Agency (EPA), 2007a. Method 3815: Screening Solid Samples for Volatile Organics. Revision 0. February.

EPA, 2007b. Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment. Revision 0, February.

EPA, 2011. Operating Procedure Field X-Ray Fluorescence Measurement. SESDPROC-107-R2. USEPA Science and Ecosystem Support Division Region 4. Revision 2. December 20.

New Mexico Environment Department (NMED), 2000. Guidelines for Corrective Action. March 13.

23.4 Forms or Attachments

Attachment 1: USEPA Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment

23.5 Document History

Revision	Effective Date	Lead Author	Summary of Changes
00	2/2015	Kate Herrell	Original version

Attachment 1

USEPA 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^{TM}, Kapton^{TM}, Spectrolene^{TM}, 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:

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

where:

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

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

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

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

10.0 CALIBRATION AND STANDARDIZATION

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

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

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

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

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

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

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

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

The equation to calibrate %D is as follows:

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

where:

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

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

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

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

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

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

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

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

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

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

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

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

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

11.0 PROCEDURE

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

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

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

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

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

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

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

12.0 DATA ANALYSIS AND CALCULATIONS

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

13.0 METHOD PERFORMANCE

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

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

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

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

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

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

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

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square, measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

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

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

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

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

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

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not ground; and preparation 4–intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

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

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

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

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

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

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

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

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

14.0 POLLUTION PREVENTION

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

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

15.0 WASTE MANAGEMENT

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

16.0 REFERENCES

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

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

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

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

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

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

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

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

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

TABLE 2

SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

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

Source: Refs. 1, 2, and 3

TABLE 3

SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

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

Source: Ref. 4

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

TABLE 4

EXAMPLE PRECISION VALUES

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

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

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

NR Not reported.

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

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

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

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

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

ND Not detected.
NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

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

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

TABLE 7

EXAMPLE ACCURACY FOR TN 9000[a]

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

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

[a] All concentrations in milligrams per kilogram.

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

-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY[1]

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

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

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

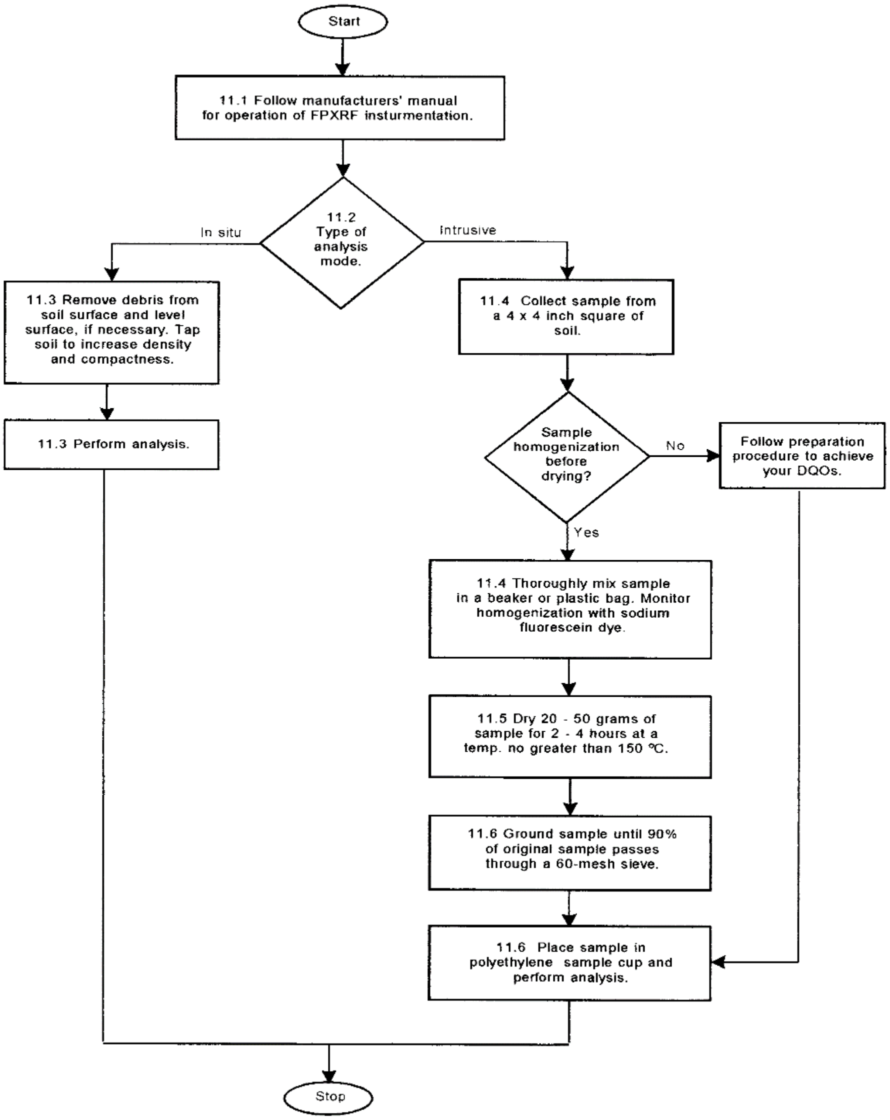
[1] Log-transformed data

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

— No applicable data

METHOD 6200

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



APPENDIX C

VAPOR PIN™ INSTALLATION STANDARD OPERATING PROCEDURE

Cox-Colvin
& ASSOCIATES, INC.
ENVIRONMENTAL SERVICES

Standard Operating Procedure Installation and Extraction of the Vapor Pin™

December 3, 2013

Scope:

This standard operating procedure describes the installation and extraction of the Vapor Pin™ for use in sub-slab soil-gas sampling.

Purpose:

The purpose of this procedure is to assure good quality control in field operations and uniformity between field personnel in the use of the Vapor Pin™ for the collection of sub-slab soil-gas samples.

Equipment Needed:

- Assembled Vapor Pin™ [Vapor Pin™ and silicone sleeve (Figure 1)];
- Hammer drill;
- 5/8-inch diameter hammer bit (Hilti™ TE-YX 5/8" x 22" #00206514 or equivalent);
- 1½-inch diameter hammer bit (Hilti™ TE-YX 1½" x 23" #00293032 or equivalent) for flush mount applications;
- ¾-inch diameter bottle brush;
- Wet/dry vacuum with HEPA filter (optional);
- Vapor Pin™ installation/extraction tool;
- Dead blow hammer;
- Vapor Pin™ flush mount cover, if desired;
- Vapor Pin™ protective cap; and
- VOC-free hole patching material (hydraulic cement) and putty knife or trowel.

Figure 1. Assembled Vapor Pin™.

Installation Procedure:

1) Check for buried obstacles (pipes, electrical lines, etc.) prior to proceeding.

2) Set up wet/dry vacuum to collect drill cuttings.

3) If a flush mount installation is required, drill a 1½-inch diameter hole at least 1¾-inches into the slab.

4) Drill a 5/8-inch diameter hole through the slab and approximately 1-inch into the underlying soil to form a void.

5) Remove the drill bit, brush the hole with the bottle brush, and remove the loose cuttings with the vacuum.

6) Place the lower end of Vapor Pin™ assembly into the drilled hole. Place the small hole located in the handle of the extraction/installation tool over the Vapor Pin™ to protect the barb fitting and cap, and tap the Vapor Pin™ into place using a dead blow hammer (Figure 2). Make sure

the extraction/installation tool is aligned parallel to the Vapor Pin™ to avoid damaging the barb fitting.

Figure 2. Installing the Vapor Pin™.

For flush mount installations, unscrew the threaded coupling from the installation/extraction handle and use the hole in the end of the tool to assist with the installation (Figure 3).

Figure 3. Flush-mount installation.

During installation, the silicone sleeve will form a slight bulge between the slab and the Vapor Pin™ shoulder. Place the protective cap on Vapor Pin™ to prevent vapor loss prior to sampling (Figure 4).

Figure 4. Installed Vapor Pin™.

7) For flush mount installations, cover the Vapor Pin™ with a flush mount cover, using either the plastic cover or the optional stainless-steel Secure Cover.

8) Allow 20 minutes or more (consult applicable guidance for your situation) for the sub-slab soil-gas conditions to equilibrate prior to sampling.

9) Remove protective cap and connect sample tubing to the barb fitting of the Vapor Pin™ (Figure 5).

Figure 5. Vapor Pin™ sample connection.

10) Conduct leak tests in accordance with applicable guidance. If the method of leak testing is not specified, an attractive alternative can be the use of a water dam and vacuum pump, as described in SOP Leak Testing the Vapor Pin™ via Mechanical Means (Figure 6).

Figure 6. Water dam used for leak detection.

11) Collect sub-slab soil gas sample. When finished sampling, replace the protective cap and flush mount cover until the next sampling event. If the sampling is complete, extract the Vapor Pin™.

Extraction Procedure:

1) Remove the protective cap, and thread the installation/extraction tool onto the barrel of the Vapor Pin™ (Figure 7). Continue turning the tool to assist in extraction, then pull the Vapor Pin™ from the hole.

Figure 7. Removing the Vapor Pin™.

2) Fill the void with hydraulic cement and smooth with the trowel or putty knife. Urethane caulk is widely recommended for installing radon systems and can provide a tight seal, but it could also be a source of VOCs during subsequent sampling.
3) Prior to reuse, remove the silicone sleeve and discard. Decontaminate the Vapor Pin™ in a hot water and Alconox® wash, then heat in an oven to a temperature of 130° C.

The Vapor Pin™ to designed be used repeatedly; however, replacement parts and supplies will be required periodically. These parts are available on-line at www.CoxColvin.com.

Replacement Parts:

Vapor Pin™ Kit Case - VPC001
Vapor Pins™ - VPIN0522
Silicone Sleeves - VPTS077
Installation/Extraction Tool - VPIE023
Protective Caps - VPPC010
Flush Mount Covers - VPFM050
Water Dam - VPWD004
Brush - VPB026
Secure Cover - VPSCSS001
Spanner Wrench - VPSPAN001

APPENDIX D

COA RAIL YARDS ENVIRONMENTAL DATABASE (CD)

APPENDIX E

COA RAIL YARDS ASBESTOS AND LBP SURVEY REPORTS (CD)