

**FINAL TRIP REPORT
FOR THE
FORMER MOHR ORCHARD
SURFACE SOIL SAMPLING
NORTH WHITEHALL TOWNSHIP, PENNSYLVANIA**

Prepared for

U.S. Environmental Protection Agency Region 3
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Submitted by

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EPA Contract No. EP-S3-05-02

Technical Direction Document No. E33-020-08-07-025
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March 2, 2009

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START Backup POC

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1.0 INTRODUCTION

Under Eastern Area Superfund Technical Assessment and Response Team (START) Contract No. EP-S3-05-02, Technical Direction Document (TDD) No. E33-020-08-07-025, U.S. Environmental Protection Agency (EPA) Region 3 tasked Tetra Tech EM Inc., (Tetra Tech) to assist with surface soil sampling activities at the Former Mohr Orchard site (site) in North Whitehall Township, Pennsylvania. Surface soil sampling began in August 2008. The final soil samples were collected in December 2008. The surface soil sampling activities were to assist in determining if the historical orchard operations, specifically use of lead-arsenate pesticide, resulted in a threat to human health and/or the environment.

This trip report provides site background information in Section 2.0, describes geology in Section 3.0, describes site activities in Section 4.0, summarizes analytical results in Section 5.0, and presents conclusions in Section 6.0. References are listed after the text.

2.0 BACKGROUND

A private citizen living on former orchard property collected a soil sample from a depth of 6 to 12 inches below ground surface (bgs). This sample was analyzed by a private laboratory and resulted in a concentration of 140 parts per million (ppm) of arsenic. This result was reported to the Agency for Toxic Substance and Disease Registry (ATSDR), and the EPA is performing an assessment at the request of the Pennsylvania Department of Environment and ATSDR. This section provides background information on the site, including its location, description, and history of site activities and investigations.

2.1 SITE LOCATION

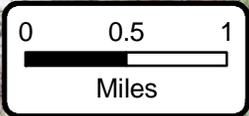
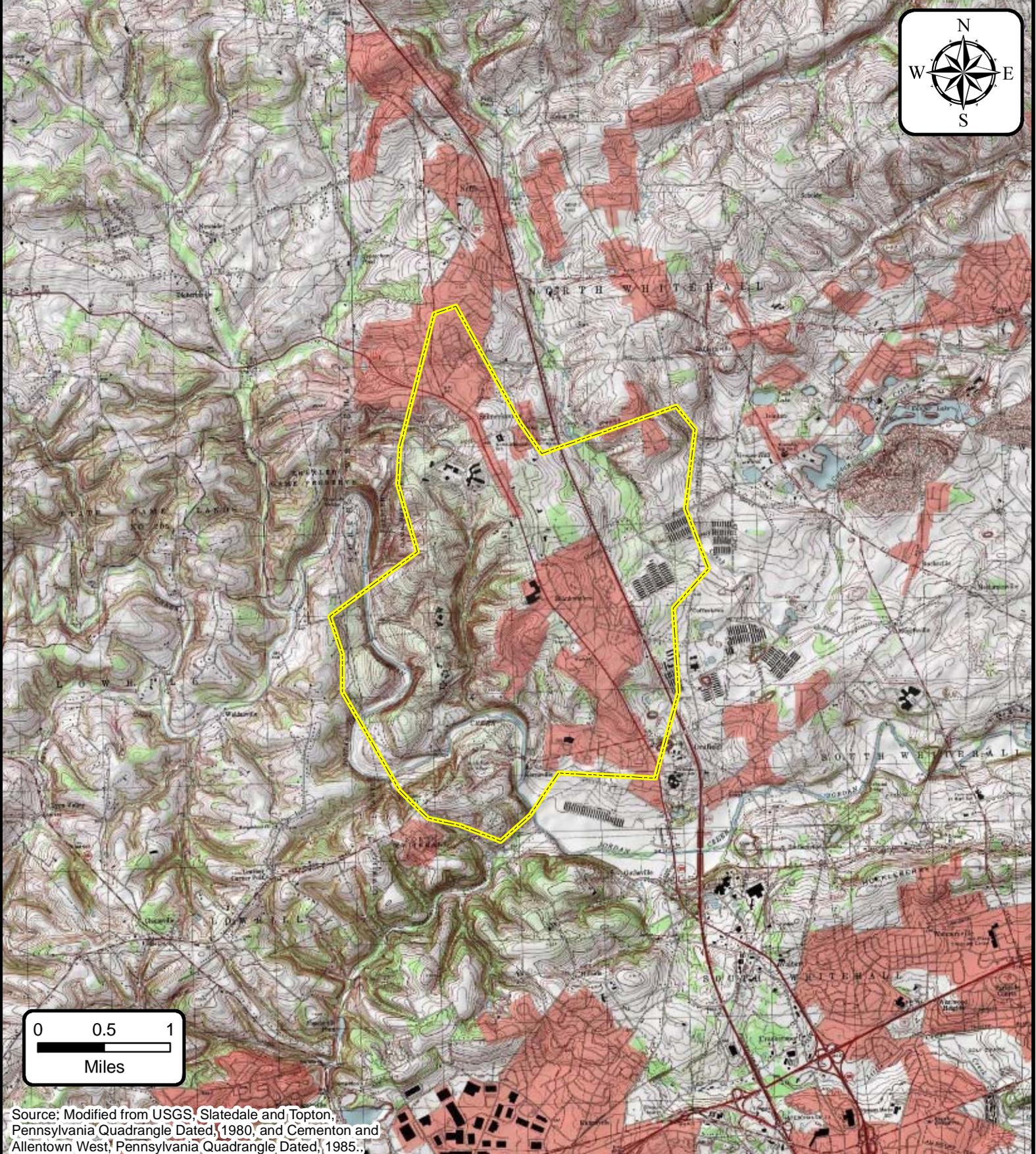
The site is located within North Whitehall Township and a small portion of South Whitehall Township, in Lehigh County, Pennsylvania. The geographic coordinates of the approximate center of the site are 40.6464° north latitude and 75.6014° west longitude (U.S. Geological Survey [USGS] 1980).

Numerous small unnamed ponds are located within the immediate vicinity of the site. Jordan Creek and Mill Creek are located approximately 0.25 mile west and northwest of the site, respectively. The Site Location Map is provided as Figure 1.

2.2 SITE DESCRIPTION

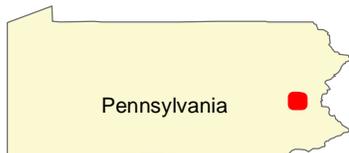
The Former Mohr Orchard site is primarily located within North Whitehall Township, made up of different types of property, including farmland, woodland, residential, commercial, and industrial properties. Pennsylvania State Route 309 runs north-south through the center of the site, through Schnecksville to the north and Orefield to the south. US Interstate 476 also runs north-south through the site. The nearest access to Interstate 476 is located approximately five miles to the south. Figure 2, Aerial Photograph, shows an aerial photo of the Former Mohr Orchard site. The site ranges between approximately 400 feet and 700 feet above mean sea level and consists primarily of rolling hills on the western portion of the site and generally slopes to the east on the eastern portion of the site (USGS 1999).

The sampling assessment was limited to properties currently zoned for residential or other public use (i.e. schools, child day care, parks, etc), and not currently utilized for crop fields. Commercial, industrial, and undeveloped land are not included as part of this sampling assessment. Former orchard areas are defined by information provided by people knowledgeable of the historical use of the area, in addition to historical aerial photographs.



Source: Modified from USGS, Slatedale and Topton, Pennsylvania Quadrangle Dated, 1980, and Cementon and Allentown West, Pennsylvania Quadrangle Dated, 1985.

Approximate Site Location = 



Legend

 Site Boundary

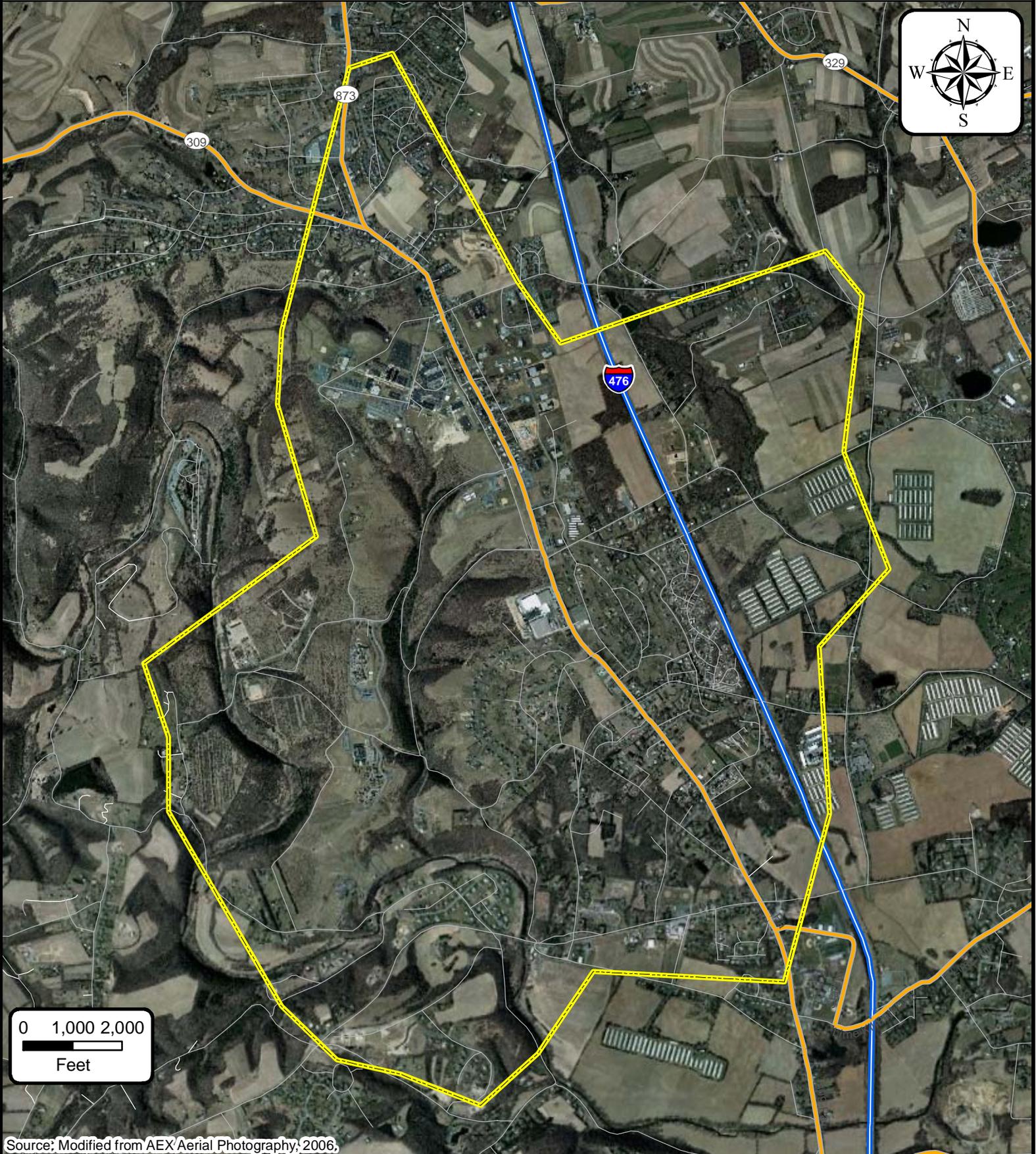
Former Mohr Orchard North Whitehall Township, Pennsylvania

Figure 1
Site Location Map

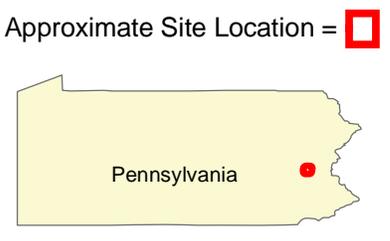
TDD No. 0210200807025
EPA Contract No. EP-S3-05-02

Map created on July 25, 2008
by A. Dye, Tetra Tech EMI





Source: Modified from AEX Aerial Photography, 2006.



Legend
 Site Boundary

**Former Mohr Orchard
 North Whitehall Township, Pennsylvania**

Figure 2
 Aerial Photograph

TDD No. 0210200807025
 EPA Contract No. EP-S3-05-02

Map created on July 25, 2008
 by A. Dye, Tetra Tech EMI



3.0 SITE GEOLOGY

The site is located in the Great Valley Section of the Ridge and Valley physiographic province of Pennsylvania. The Great Valley Section consists of a very broad lowland that lies south of Blue Mountain in southeastern Pennsylvania. The lowland has gently undulating hills eroded into shales and siltstones on the north side of the valley and a lower elevation flatter landscape developed on limestones and dolomites on the south side. Local relief is generally less than 100 feet, particularly in the carbonate area, but may be up to 300 feet in the shale area. Elevation ranges from 140 feet to 1,100 feet. Several large rivers such as the Susquehanna and Schuylkill Rivers cut across the Great Valley. However, most of the well-defined drainage originates on the slopes of Blue Mountain and flows across the shales (Pennsylvania Department of Conservation and Natural Resources [DCNR] 2009).

The site itself is underlain primarily by Lower Martinsburg Shale, a gray to dark-gray shale and slate. A portion of the site on the east is underlain by Jacksonburg Limestone, and to the south by Illinoian terminal moraine, surrounding Jordan Creek (DCNR 2009).

No drilling occurred during site activities.

4.0 SITE ACTIVITIES

From August through December 2008, surface soil samples were collected and analyzed onsite for arsenic and lead concentrations. Tetra Tech documented and photographed site activities in accordance with Tetra Tech Standard Operating Procedure (SOP) No. 024, "Recording of Notes in Field Logbook" (Tetra Tech 1999a). Photographic documentation is provided in Appendix A. This section discusses activities which occurred during surface soil sampling activities.

4.1 BACKGROUND SURFACE SOIL SAMPLING

On August 15, 2008, Tetra Tech and EPA mobilized to the site to collect background soil samples. Twelve soil samples were collected from properties within or close to the site area, but not physically located on former orchard property. Former orchard property was determined by historical aerial photography provided by North Whitehall Township. Grab samples were

collected from each location. All soil sample locations are identified on Figure 3, Background Sample Locations and Results. All samples were collected in accordance with the Tetra Tech SOP No. 005, "Soil Sampling" (Tetra Tech 1999c). Samples were collected with a dedicated plastic scoop. The soil was homogenized in place with the plastic scoop before being placed into a labeled, self-sealing plastic bag. The geographic location where each soil sample was collected was recorded with a Trimble global positioning system (GPS) device. Table 1 summarizes the August 23, 2008 sampling activities.

TABLE 1 - BACKGROUND SURFACE SOIL SAMPLING SUMMARY

Sample Identifier	Laboratory Identifier	Sample Matrix	Sample Date	Collection Time	Analysis
FMO-081508-BKG-SS01	MC0289	Surface Soil	8/15/08	9:30	Lead/Arsenic
FMO-081508-BKG-SS02	MC0290	Surface Soil	8/15/08	10:24	Lead/Arsenic
FMO-081508-BKG-SS03	MC0291	Surface Soil	8/15/08	10:51	Lead/Arsenic
FMO-081508-BKG-SS05	MC0292	Surface Soil	8/15/08	11:30	Lead/Arsenic
FMO-081508-BKG-SS06	MC0293	Surface Soil	8/15/08	12:12	Lead/Arsenic
FMO-081508-BKG-SS07	MC0294	Surface Soil	8/15/08	14:23	Lead/Arsenic
FMO-081508-BKG-SS08	MC0295	Surface Soil	8/15/08	14:50	Lead/Arsenic
FMO-081508-BKG-SS09	MC0296	Surface Soil	8/15/08	15:16	Lead/Arsenic
FMO-081508-BKG-SS10	MC0297	Surface Soil	8/15/08	15:43	Lead/Arsenic
FMO-081508-BKG-SS11	MC0298	Surface Soil	8/15/08	16:03	Lead/Arsenic
FMO-081508-BKG-SS12	MC0299	Surface Soil	8/15/08	16:18	Lead/Arsenic
FMO-081508-BKG-SS13	MC02A0	Surface Soil	8/15/08	16:30	Lead/Arsenic

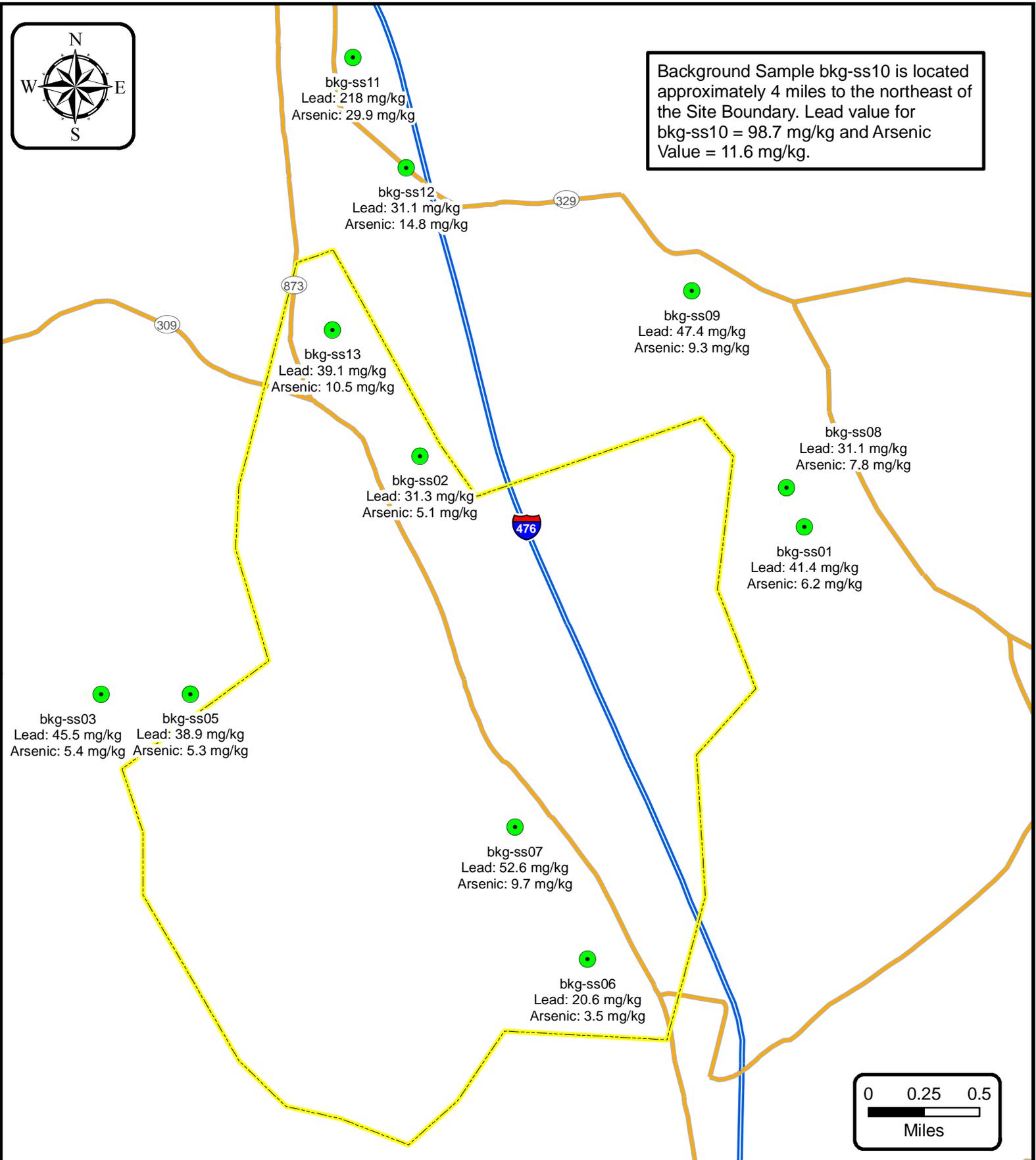
Notes:

BKG - Background
 SS - Surface Soil

FMO - Former Mohr Orchard



Background Sample bkg-ss10 is located approximately 4 miles to the northeast of the Site Boundary. Lead value for bkg-ss10 = 98.7 mg/kg and Arsenic Value = 11.6 mg/kg.



bkg-ss11
Lead: 218 mg/kg
Arsenic: 29.9 mg/kg

bkg-ss12
Lead: 31.1 mg/kg
Arsenic: 14.8 mg/kg

bkg-ss13
Lead: 39.1 mg/kg
Arsenic: 10.5 mg/kg

bkg-ss09
Lead: 47.4 mg/kg
Arsenic: 9.3 mg/kg

bkg-ss08
Lead: 31.1 mg/kg
Arsenic: 7.8 mg/kg

bkg-ss02
Lead: 31.3 mg/kg
Arsenic: 5.1 mg/kg

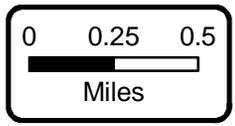
bkg-ss01
Lead: 41.4 mg/kg
Arsenic: 6.2 mg/kg

bkg-ss03
Lead: 45.5 mg/kg
Arsenic: 5.4 mg/kg

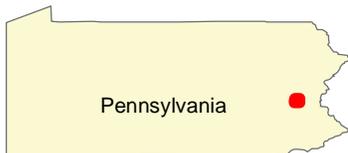
bkg-ss05
Lead: 38.9 mg/kg
Arsenic: 5.3 mg/kg

bkg-ss07
Lead: 52.6 mg/kg
Arsenic: 9.7 mg/kg

bkg-ss06
Lead: 20.6 mg/kg
Arsenic: 3.5 mg/kg



Approximate Site Location = 



Legend

-  Site Boundary
-  Background Samples

**Mohr Orchard
North Whitehall Township, Pennsylvania**

**Figure 3
Background Sample Locations and Results**

TDD No. 0210200807025
EPA Contract No. EP-S3-05-02

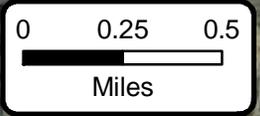
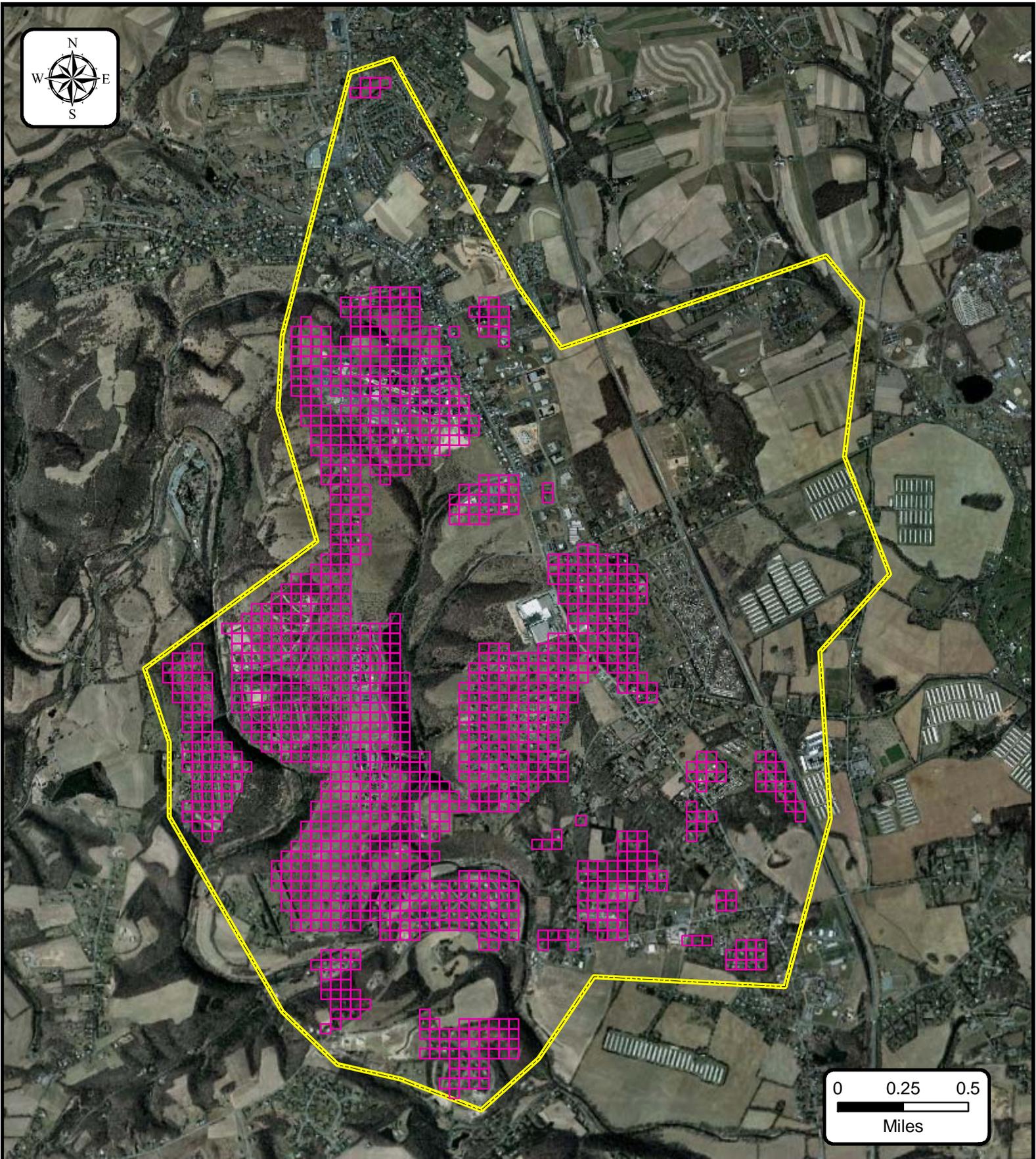
Map created on February 26,
2009 by A. Dye, Tetra Tech EMI



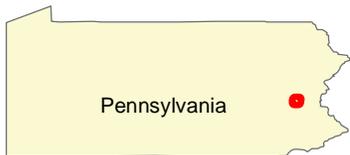
4.2 SURFACE SOIL SAMPLING

With the assistance of North Whitehall Township, areas of the site which were historically utilized for orchard operations, and currently zoned for residential or public use activities, were mapped into 200 foot by 200 foot grids. Sampling grids are identified in Figure 4, Sampling Grid Map. To create these grids, the first grid was selected at a randomly generated point within the site area. EPA utilized an adaptive cluster sampling strategy for this assessment. EPA requested access to collect surface soil composite samples from a random 25% of all sampling grids. Each surface soil sample consisted of 10 randomly selected points within the 200 foot by 200 foot sampling grid. A 10-point composite sample was determined to be a cost-effective balance between collecting enough points to acquire an adequate representation from each grid cell, while not causing too great a dilution within the sample. A 200 foot by 200 foot grid size represents the average residential property size, the selected sampling unit of interest for this assessment. The initial random 25% of sampling grids, identified as proposed Tier I grids, were selected using a graphical information system (GIS) tool which selects random grids while keeping a statistically significant distribution of grids throughout the site. The same GIS tool was utilized for selecting the 10-point composite locations within each grid. Reproducible locations were recorded prior to sample collection in a Trimble GPS. The Trimble GPS was used during sample collection to identify each location. In cases where an obstruction caused sample collection of that point to be impossible, the sample was collected from the nearest accessible surface soil and the change of location was documented.

When field analysis of composite surface soil samples identified a concentration exceeding the screening level for arsenic of 40 ppm, adjacent grids to the north, east, west, and south of the exceeding sample grid were supplemented to the total number of sample grids. These grids were identified as Tier II and Tier III grids. In the situation that the exceeding sample grid is located on the edge of the site boundary, the site boundary was expanded as necessary. The initial proposed sampling grids are identified in Figure 5, Initial 25% of Grids. All sampled grids are identified as Tier I, Tier II, or Tier III grids in Figure 6, Tier I, II, & III Sample Grids.



Approximate Site Location = 



Legend

-  200' x 200' Sampling Grid
-  Site Boundary

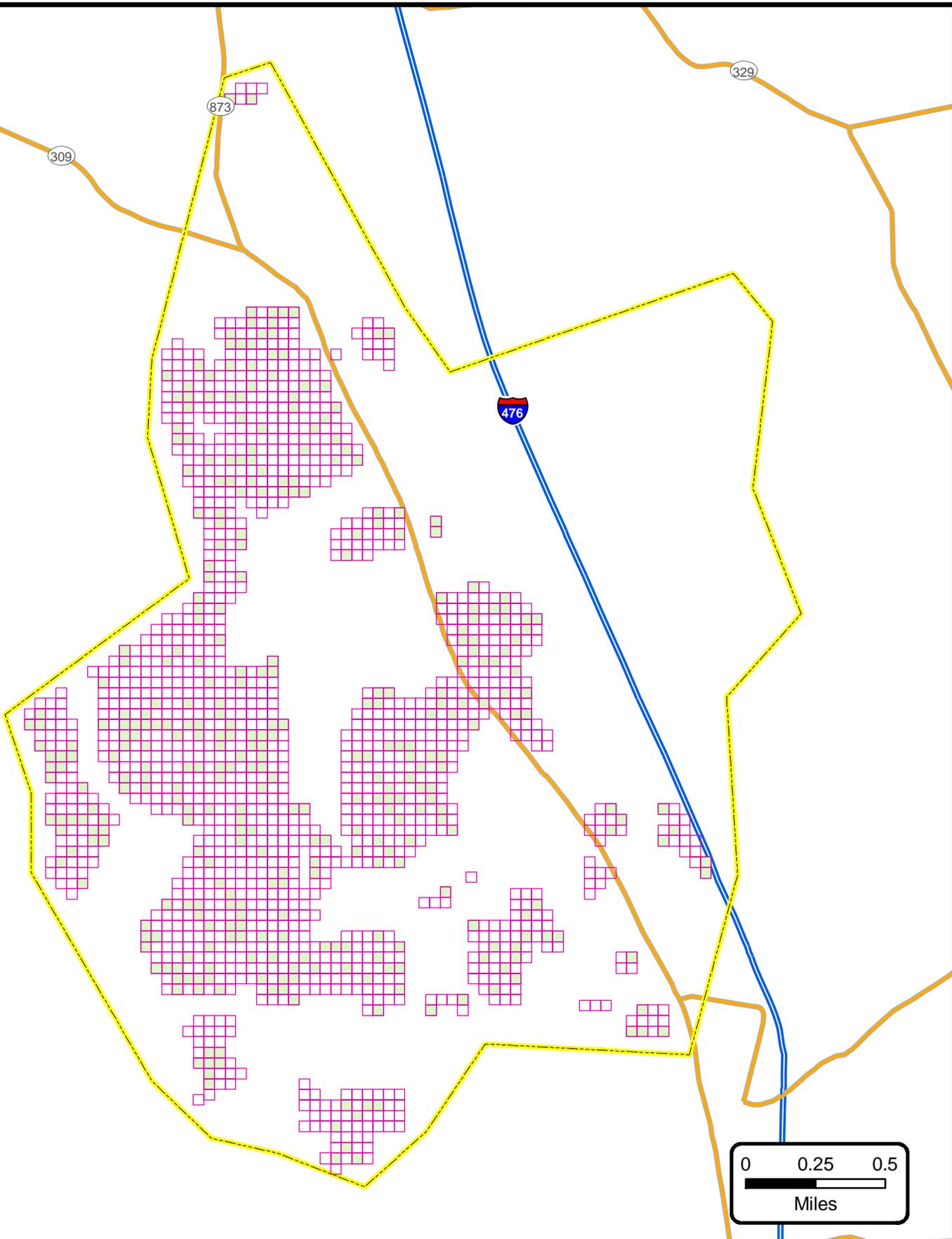
Mohr Orchard North Whitehall Township, Pennsylvania

Figure 4
Sampling Grid Map

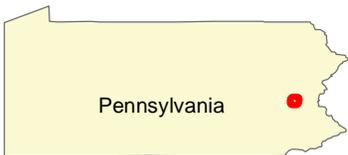
TDD No. 0210200807025
EPA Contract No. EP-S3-05-02

Map created on July 25, 2008
by A. Dye, Tetra Tech EMI





Approximate Site Location = 



Legend

-  200' x 200' Sampling Grid
-  Random_Grids
-  Site Boundary

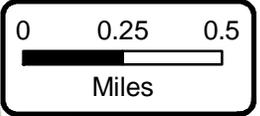
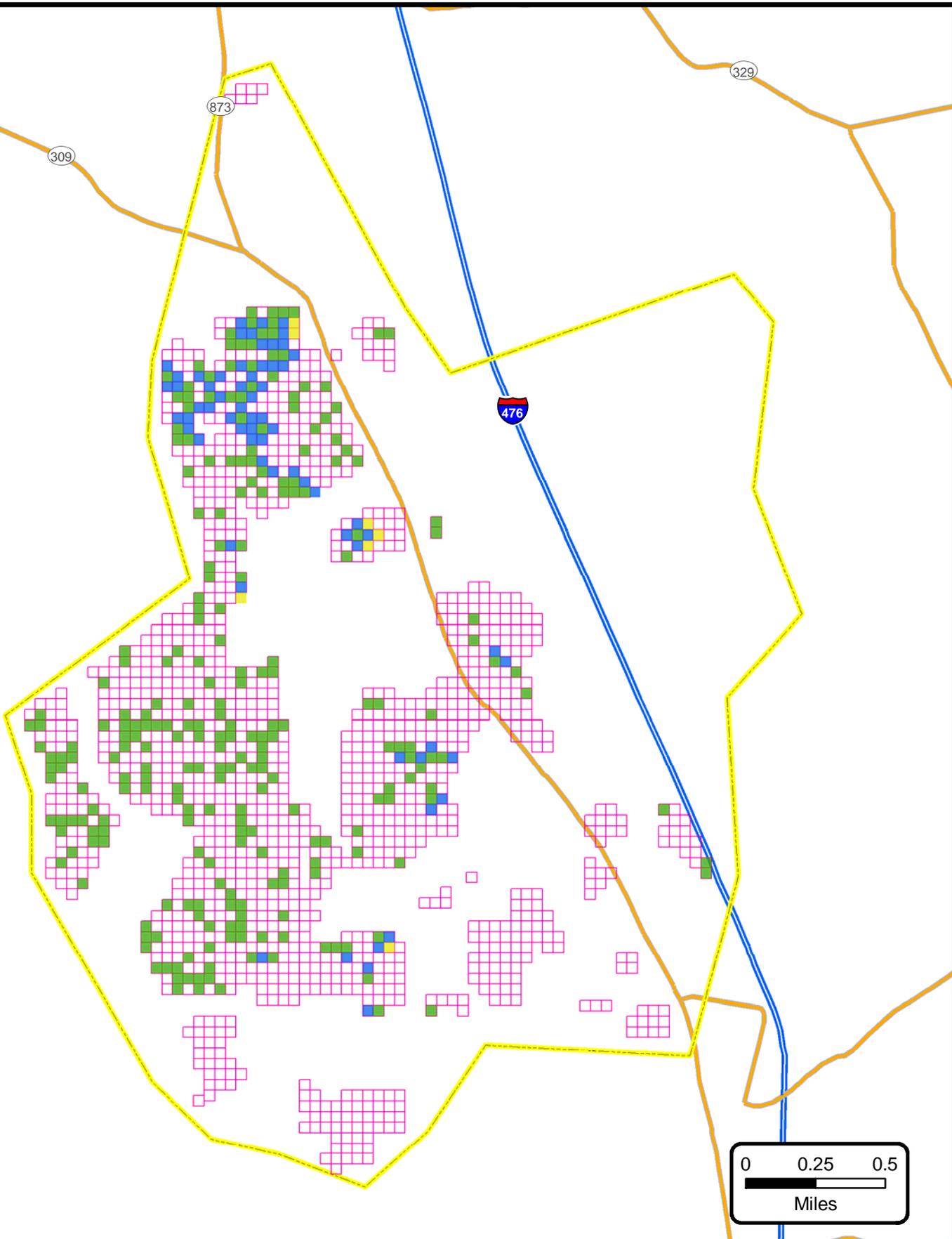
**Mohr Orchard
North Whitehall Township, Pennsylvania**

**Figure 5
Initial 25% of Grids**

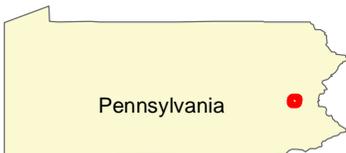
TDD No. 0210200807025
EPA Contract No. EP-S3-05-02

Map created on February 26, 2009
by A. Dye, Tetra Tech EMI





Approximate Site Location = 



Legend

-  Tier I Sample Grid
-  Tier II Sample Grid
-  Tier III Sample Grid
-  200' x 200' Sampling Grid
-  Site Boundary

**Mohr Orchard
North Whitehall Township, Pennsylvania**

**Figure 6
Tier I, II, & III Sample Grids**

TDD No. 0210200807025
EPA Contract No. EP-S3-05-02

Map created on February 26, 2009
by A. Dye, Tetra Tech EMI



Sampling grid locations were identified in the field using a Trimble GPS device. Soil samples were collected in accordance with Tetra Tech Standard Operating Procedure (SOP) No. 005, “Soil Sampling” (Tetra Tech 1999). Non-dedicated sampling equipment, including a pickaxe, was decontaminated between each use in accordance with Tetra Tech SOP No. 002, “General Equipment Decontamination” (Tetra Tech 1999b).

Locations for surface soil samples were prepared for sampling using a steel pickaxe to loosen the topsoil, when necessary. Surface soil samples were collected from the prepared sampling location from 0 to 3 inches bgs using a dedicated plastic scoop. The soil was placed into a labeled, self-sealing plastic bag.

Replicate samples were collected from unique locations within a sampling grid throughout the assessment. This replicate sampling process assists in determining sufficient representation from each grid cell during the assessment. A total of 44 replicate samples were collected during sampling activities. Unique duplicate samples were collected from homogenized samples. This duplicate sample assists in confirmation of adequate homogenization of each composite sample. A total of 28 duplicate samples were collected during sampling activities.

A total of 372 surface soil samples were collected from 327 total grids and 1 biased sampling location within the site area. Any grid that contained any portion of a residential property within the grid was classified residential for reporting purposes. A total of 81 grids that were sampled were classified as residential. The 246 remaining grids were classified as public-use.

Restrictions on soil sampling were generally due to access to grids being denied by property owners. A summary of surface soil samples are included in Appendix B.

4.3 SAMPLE MANAGEMENT

Samples that were shipped to laboratories were handled and packaged in accordance with the Tetra Tech SOP No. 019, “Packaging and Shipping Samples” (Tetra Tech 2000) and with the Tetra Tech “Quality Assurance Project Plan (QAPP) for START” (Tetra Tech 2006). All shipping containers were properly labeled with EPA custody seals and were delivered with signed chain-of-custody forms and appropriate hazard warnings for laboratory personnel.

On August 18, 2008, twelve soil samples from background soil sampling activities were shipped to Bonner Analytical Testing Company of Hattiesburg, MS, via Federal Express under Contract Laboratory Program (CLP) Case Number 37792 for lead and arsenic analysis.

Surface soil samples were prepared for X-ray fluorescence (XRF) analysis in accordance with EPA Method 6200, "Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment" (EPA 1998). Each sample was homogenized in an on-site laboratory within the sealed plastic bag. A 20 to 50 gram aliquot of each sample was dried and sieved through a no. 60-mesh sieve. The dried and sieved aliquots were then placed in dedicated XRF sampling containers for analysis. Each dedicated XRF sampling container was labeled with the corresponding site-specific sampling location identifier. Field blanks prepared from silicon dioxide were collected and prepared with identical equipment and methods as surface soil samples. Non-dedicated sampling equipment, including sieves, pans, and ovens, were decontaminated between each use in accordance with Tetra Tech SOP No. 002, "General Equipment Decontamination" (Tetra Tech 1999).

All surface soil samples were analyzed for total lead by ex situ XRF analysis in accordance with EPA Method 6200. A copy of EPA Method 6200 is provided as Appendix C.

On September 25, 2008 and October 13, 2008, 24 and twelve soil samples from surface soil sampling activities, respectively, were shipped to SVL Analytical Inc. of Kellogg, ID, via Federal Express under CLP Case Number 37845 for lead and arsenic analysis. These samples were analyzed to compare against XRF field data. The EPA On-Scene Coordinator requested that the XRF and laboratory data be reviewed in accordance with the EPA "Quality Assurance/Quality Control Guidance for Removal Activities," EPA/540/4-90/004, April, 1990. The results from SVL Analytical Inc. confirmed the XRF data collected by Tetra Tech. The Data Quality Report is provided in Appendix D.

5.0 ANALYTICAL RESULTS

All analytical results for surface soil samples and background soil samples are summarized in Appendices B and E, respectively. Copies of the validated laboratory analytical data packages are included in the attachment to this report.

5.1 BACKGROUND SURFACE SOIL RESULTS

Twelve surface soil samples were analyzed for lead and arsenic by Bonner Analytical Testing Company. Arsenic concentrations in background samples ranged from 3.5 ppm to 29.9 ppm, with a mean average of approximately 9.9 ppm. Lead concentrations ranged from 20.6 ppm to 218 ppm, with a mean average of approximately 58 ppm. A summary of background surface soil analytical results is provided in Appendix E, and identified on Figure 3, Background Sample Locations and Results. A copy of the validated analytical results is provided in the attachment to this report.

5.2 SURFACE SOIL SAMPLE RESULTS

XRF results for soil samples collected during the surface soil assessment at the Former Mohr Orchard site indicated that arsenic concentrations ranged from non-detect to 149 ppm in surface soil samples collected and analyzed at the site between September and December 2008. The average concentration of arsenic in the surface soil samples was approximately 50 ppm. No applicable action level for arsenic has currently been developed for this site. Lead concentrations ranged from 35 ppm to 1951 ppm in surface soil samples. The average concentration of lead in the surface soil samples was approximately 349 ppm.

There were 400 samples analyzed during this assessment from 327 grids. 81 grids were identified as residential property. 246 grids were identified as public-use property. One sample was analyzed from a biased location.

Arsenic concentrations ranged from non-detect to 148.93 ppm with an average of approximately 49 ppm in residential grid samples. Lead concentrations ranged from 39.39 ppm to 1,833.36 ppm with an average of approximately 308 ppm in residential grid samples.

Arsenic concentrations ranged from non-detect to 140.39 ppm with an average of approximately 50 ppm in public-use grid samples. Lead concentrations ranged from 35.02 ppm to 1,950.84 ppm with an average of approximately 362 ppm in public-use grid samples.

One composite sample was collected from a biased area of no growth within a public use area. Arsenic concentrations were identified at 92.39 ppm, and lead concentrations were identified at 319.88 ppm within this area.

Residential properties were segregated into 14 different areas, based on location. Areas are shown on Figure 7, Residential Grouping Areas. No grids were sampled within Area 1, Area 7, Area 9, Area 11, Area 12, or Area 13. Therefore, no conclusion can be made of surface arsenic and lead concentrations within those areas. Results for areas containing grids that were sampled during this assessment are included in Table 2.

TABLE 2 - RESIDENTIAL SURFACE SOIL SAMPLING SUMMARY

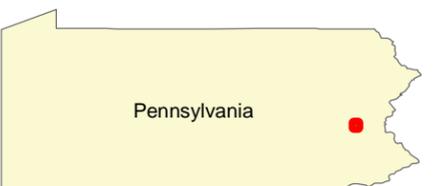
	Number of Grids Sampled	Lead (ppm)			Arsenic (ppm)		
		Maximum	Minimum	Average	Maximum	Minimum	Average
Area 2	8	1,238	74.12	465	138.09	10.2	56
Area 3	10	344.85	156.17	254	73	27.24	50
Area 4	7	174.16	71.4	115	37.77	15.4	26
Area 5	25	814.5	76.54	271	87.99	19.93	48
Area 6	4	1833.36	234.58	851	148.93	36.09	71
Area 8	3	66.36	52.88	61	17.66	10.03	14
Area 10	16	1012.64	39.39	315	120.66	11.38	62
Area 14	3	231.63	182.11	210	45.66	36.22	42

Notes:
ppm = parts per million

A summary of surface soil analytical results from the 2008 sampling event is provided in Appendix B. Copies of the validated laboratory analytical data packages are included in the attachment to this report. A copy of the data quality report is provided in Appendix D.



Source: Modified from AEX Aerial Photography, 2006.

<p>Approximate Site Location = </p>  <p>Pennsylvania</p>	<p>Legend</p> <ul style="list-style-type: none"> Limited Access Granted Within Area for Assessment Sampling Access Not Granted Within Area for Assessment Sampling No Additional Sampling will be Conducted Site Boundary <p>0 1,000 2,000 Feet</p>	<h2>Mohr Orchard</h2> <h3>North Whitehall Township, Pennsylvania</h3>	
		<h3>Figure 7</h3> <h4>Residential Grouping Areas</h4>	
		TDD No. 0210200807025 EPA Contract No. EP-S3-05-02	Map created on February 26, 2009 by A. Dye, Tetra Tech EMI
		TETRA TECH	

REFERENCES

- Pennsylvania Department of Conservation and Natural Resources. 2009. Pennsylvania Geologic Survey. On-line address: <http://www.dcnr.state.pa.us/topogeo/index.aspx>.
- Tetra Tech EM Inc. (Tetra Tech). 1999. "Recording of Notes in Field Logbook." Standard Operating Procedure (SOP) No. 024. November.
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- Tetra Tech. 2006. "Quality Assurance Project Plan [QAPP] for START." August.
- U.S. Geological Survey (USGS). 1980. 7.5-Minute Series Topographic Quadrangle Map, Slatedale and Topton, Pennsylvania.
- USGS. 1999. 7.5-Minute Series Topographic Map of Slatedale and Topton, Pennsylvania Quadrangle.

APPENDIX A

Photographic Documentation Log

(2 pages)

Client: EPA Region 3
Site Name: Former Mohr Orchard
Location: North Whitehall Township, PA

Prepared by: Tetra Tech
Photographer: TTEMI
TDD Number: E33-020-08-07-025

Photograph No. 1

Photograph Date: September 2008

Description: Sampling crew collecting surface soil grid sample.



Photograph No. 2

Photograph Date: September 2008

Description: Example of primarily undisturbed orchard area located within the site.



Client: EPA Region 3
Site Name: Former Mohr Orchard
Location: North Whitehall Township, PA

Prepared by: Tetra Tech
Photographer: TTEMI
TDD Number: E33-020-08-07-025

Photograph No. 3

Photograph Date: October 2008

Description: Sample team collecting soil samples from open field area.



Photograph No. 4

Photograph Date: October 2008

Description: Sample team collecting sample point using dedicated plastic scoop and self-sealing plastic bag.



APPENDIX B

Surface Soil Sampling Data Tables

(8 pages)

UNIQUE ID	PPM PB	PB Qualifier	PPM As	As Qualifier	Grid Type	Area No.
001	74.12		17.68	J	R	2
002	165.36		22.76		R	2
003	268.08		53.9		R	2
004	272		22.79		R	2
005	416.17		86.7		R	2
006	820.43		92.32		R	2
007	1238.74		138.09		R	2
008	Not recorded		10.2		R	2
009	156.17		27.24		R	3
010	219.14		53.17		R	3
011	229.55		39.46		R	3
012	235.92		73		R	3
013	240.22		58.33		R	3
014	262.09		55.94		R	3
015	268.08		57.76		R	3
016	280.49		37		R	3
017	300.75		53.25		R	3
018	344.85		47.98		R	3
019	71.4		24.69		R	4
020	89.02		18.59	J	R	4
021	100.41		25.07		R	4
022	101.72		27.43		R	4
023	118.58		31.64		R	4
024	151.64		37.77		R	4
025	174.16		15.4	J	R	4
026	76.54		19.93	J	R	5
027	89.07		29.37		R	5
028	93.88		26.78		R	5
029	143.79		30.75		R	5
030	155.89		37.53		R	5
031	167.15		27.54		R	5
032	178.64		55.44		R	5
033	186.37		41.37		R	5
034	192.74		56.52		R	5
035	193.68		45.44		R	5
036	203.5		43.5		R	5
037	209.83		25.29		R	5
038	212.66		35.71		R	5
039	229.39		51.45		R	5
040	230.74		51.59		R	5
041	259.21		51.26		R	5
042	267.46		44.91		R	5
043	284		54.8		R	5
044	288.54		60.04		R	5
045	302.44		50.3		R	5
046	359.1		64.2		R	5
047	426.01		58.94		R	5
048	581.69		77.45		R	5
049	629.93		77.63		R	5
050	814.5		87.99		R	5
051	234.58		36.09		R	6

UNIQUE ID	PPM PB	PB Qualifier	PPM As	As Qualifier	Grid Type	Area No.
052	526.49		55.21	J	R	6
053	809.66		44.26	J	R	6
054	1833.36		148.93	J	R	6
055	52.88		17.66	J	R	8
056	63.59		13.36	J	R	8
057	66.36		10.03	J	R	8
058	39.39		< LOD	U	R	10
059	48.87		11.38	J	R	10
060	155.4		49.1		R	10
061	177.32		39.54		R	10
062	189.21		44.48		R	10
063	193.45		49.43		R	10
064	201.2		56.6		R	10
065	251.95		66.98		R	10
066	258.62		67.38		R	10
067	261.82		66.06		R	10
068	298.82		58.76		R	10
069	379.17		86.94		R	10
070	425.59		74.52		R	10
071	549.4		< LOD	U	R	10
072	604.4		77.89		R	10
073	1012.64		120.66		R	10
074	182.11		36.22		R	14
075	215.6		45.66		R	14
076	231.63		44.23		R	14
077	35.02		7.77	J	PU	N/A
078	40.64		13.95	J	PU	N/A
079	42.96		18.23	J	PU	N/A
080	44.39		< LOD	U	PU	N/A
081	46.83		7.96	J	PU	N/A
082	49.09		11.69	J	PU	N/A
083	49.2		16.54	J	PU	N/A
084	50.14		9.85	J	PU	N/A
085	50.49		14.27	J	PU	N/A
086	52.29		9.83	J	PU	N/A
087	54.62		11.85	J	PU	N/A
088	56.44		17.14	J	PU	N/A
089	56.76		11.06	J	PU	N/A
090	57.15		23.36		PU	N/A
091	62.88		20.25		PU	N/A
092	63.28		15.69	J	PU	N/A
093	63.73		19.86	J	PU	N/A
094	64.88		15.09	J	PU	N/A
095	65.39		11.28	J	PU	N/A
096	65.79		18.74	J	PU	N/A
097	66.21		18.44	J	PU	N/A
098	66.96		13.68	J	PU	N/A
099	67.17		14.17	J	PU	N/A
100	68.09		22.97		PU	N/A
101	69.02		12.69	J	PU	N/A
102	69.3		21.83		PU	N/A

UNIQUE ID	PPM PB	PB Qualifier	PPM As	As Qualifier	Grid Type	Area No.
103	72.34		11.31	J	PU	N/A
104	77.93		16.72	J	PU	N/A
105	78.82		21.21		PU	N/A
106	78.83		16.58	J	PU	N/A
107	80.01		19.6	J	PU	N/A
108	82.14		22.44		PU	N/A
109	85.52		21.2		PU	N/A
110	88.99		15.36	J	PU	N/A
111	91.6		14.04	J	PU	N/A
112	91.74		28.74		PU	N/A
113	93.87		24.12		PU	N/A
114	101.33		19.94	J	PU	N/A
115	102.48		20.15		PU	N/A
116	104.46		11.96	J	PU	N/A
117	108.35		19.11	J	PU	N/A
118	109.43		26.17		PU	N/A
119	111.27		24.45		PU	N/A
120	113.46		16.27	J	R	N/A
121	113.5		29.32		PU	N/A
122	114.33		24.39		PU	N/A
123	119.11		18.38	J	PU	N/A
124	120.05		29.36		PU	N/A
125	122.61		19.72	J	PU	N/A
126	124.57		21.18		PU	N/A
127	124.69		26.81		PU	N/A
128	125.58		35.78		PU	N/A
129	127.56		15.75	J	PU	N/A
130	131.39		29.8		PU	N/A
131	131.5		17.41	J	PU	N/A
132	131.83		25.62		PU	N/A
133	132.48		21.01		PU	N/A
134	133.48		28.99		PU	N/A
135	135.98		36.64		PU	N/A
136	138.36		27.93		PU	N/A
137	140.98		17.07	J	PU	N/A
138	142.82		23.39		PU	N/A
139	143.5		25.12		PU	N/A
140	146.31		19.33	J	PU	N/A
141	146.39		24.21		PU	N/A
142	148.66		22.4		PU	N/A
143	153.34		28.81		PU	N/A
144	154.66		25.11		PU	N/A
145	157.3		31.67		PU	N/A
146	159.57		33.86		PU	N/A
147	162.86		26.45		PU	N/A
148	167.01		41.12		PU	N/A
149	168.52		19.72	J	PU	N/A
150	169.22		26.26		PU	N/A
151	172.87		22.18		PU	N/A
152	174.02		39.54		PU	N/A
153	174.24		46.76		PU	N/A

UNIQUE ID	PPM PB	PB Qualifier	PPM As	As Qualifier	Grid Type	Area No.
154	175.91		41.26		PU	N/A
155	177.08		30.25		PU	N/A
156	177.3		19.74	J	PU	N/A
157	178		24.88		R	N/A
158	184.29		39.07		PU	N/A
159	184.48		35.7		PU	N/A
160	184.75		24.69		PU	N/A
161	187.61		36.54		PU	N/A
162	188		45.98		PU	N/A
163	190.71		17.95	J	PU	N/A
164	190.73		39.3	J	PU	N/A
165	193.77		60.65		PU	N/A
166	195.26		27.66		PU	N/A
167	195.56		47.14		PU	N/A
168	200.58		40.64		PU	N/A
169	204.3		29.64		PU	N/A
170	206.27		30.36		PU	N/A
171	207.01		26.19		PU	N/A
172	208.5		42.18		PU	N/A
173	210.08		41.66		PU	N/A
174	212.98		28.41		PU	N/A
175	213.7		30.47		PU	N/A
176	215.05		34.45		PU	N/A
177	215.22		45.32		PU	N/A
178	215.5		38.42		PU	N/A
179	216.65		23.05		R	N/A
180	218.58		35.52		PU	N/A
181	220.26		33.5		PU	N/A
182	220.52		48.21		PU	N/A
183	220.87		51.29		PU	N/A
184	221.86		35.37		PU	N/A
185	227.6		46.98		PU	N/A
186	228.46		25.4		PU	N/A
187	229.08		56.41		PU	N/A
188	231.18		36.34		PU	N/A
189	232.34		27.21		PU	N/A
190	234.7		53.23	J	PU	N/A
191	235.17		67.93		PU	N/A
192	236.63		58.8		PU	N/A
193	237.39		49.39		PU	N/A
194	239.2		49.14		PU	N/A
195	241.38		27.87		PU	N/A
196	242.09		59.04		PU	N/A
197	242.45		49.13		PU	N/A
198	244.15		39.34		PU	N/A
199	244.18		47.97		PU	N/A
200	245.34		39.34		PU	N/A
201	246.03		68.74		PU	N/A
202	246.35		74.71		PU	N/A
203	246.82		44.63		PU	N/A
204	248.6		55.9		PU	N/A

UNIQUE ID	PPM PB	PB Qualifier	PPM As	As Qualifier	Grid Type	Area No.
205	248.72		40.55		PU	N/A
206	249.24		35.67		PU	N/A
207	252.44		40.93		PU	N/A
208	253.02		57.66		PU	N/A
209	254.2		40.63		PU	N/A
210	256.67		36.29		PU	N/A
211	257.48		54.01		PU	N/A
212	258.61		53.63	J	PU	N/A
213	259.21		55.98	J	PU	N/A
214	260.95		43.36		PU	N/A
215	260.98		57.5		PU	N/A
216	262.89		60.13		PU	N/A
217	270.43		43.88		PU	N/A
218	272.72		66.68		PU	N/A
219	275.48		39.12		PU	N/A
220	276.33		55.74		PU	N/A
221	276.52		51.93		PU	N/A
222	277.9		46.44		PU	N/A
223	280.74		64.64		PU	N/A
224	286.29		71.89		PU	N/A
225	288.63		47.39		PU	N/A
226	291.88		45.84		PU	N/A
227	294.47		57.81		PU	N/A
228	295.05		47.16		PU	N/A
229	299.33		59.65		PU	N/A
230	304.05		27.3		PU	N/A
231	305.54		41.75		PU	N/A
232	308.29		69.84		PU	N/A
233	309.61		47.9		PU	N/A
234	310.19		76.02		PU	N/A
235	312.73		51.06		PU	N/A
236	314.18		28.18		PU	N/A
237	314.24		35.95		PU	N/A
238	315.62		65.55		PU	N/A
239	315.95		44.79		PU	N/A
240	318.08		41.77		PU	N/A
241	319.77		63.24		PU	N/A
242	319.88		92.39	J	Biased	N/A
243	320.57		71.5		PU	N/A
244	335.83		67.82		PU	N/A
245	336.08		56.52		PU	N/A
246	336.46		57.18		PU	N/A
247	337.13		68.95		PU	N/A
248	341.15		57.01		PU	N/A
249	344.93		66.89		PU	N/A
250	349.99		55.5		PU	N/A
251	352.11		66.35		PU	N/A
252	352.65		57.75		PU	N/A
253	355.58		63.51		PU	N/A
254	360.87		66.92		PU	N/A
255	368.22		70		PU	N/A

UNIQUE ID	PPM PB	PB Qualifier	PPM As	As Qualifier	Grid Type	Area No.
256	371.15		47		R	N/A
257	371.9		46.45		PU	N/A
258	381.79		85.5		PU	N/A
259	382.84		44.68		PU	N/A
260	385.63		58.71		PU	N/A
261	385.95		47.71		PU	N/A
262	394.8		76.85		PU	N/A
263	424.44		64.84		PU	N/A
264	424.97		49.65		PU	N/A
265	437.2		69.94		PU	N/A
266	439.97		72.47		PU	N/A
267	447.16		53.79		PU	N/A
268	465.81		65.12	J	PU	N/A
269	466.79		54.51	J	PU	N/A
270	475.56		70.66		PU	N/A
271	480.76		87.22		PU	N/A
272	495.68		61.53	J	PU	N/A
273	497.88		64.12		PU	N/A
274	513.51	J	53.7	J	PU	N/A
275	514.45		77.08		PU	N/A
276	515.08		65.34		PU	N/A
277	521.81		71.16		PU	N/A
278	526.5		60	J	PU	N/A
279	534.23		55.89		PU	N/A
280	536.37		104.96		PU	N/A
281	539.18		93.91		PU	N/A
282	557.66		82.07		PU	N/A
283	601.31		81.33	J	PU	N/A
284	606.9		96.87	J	PU	N/A
285	617.85		131.52		PU	N/A
286	628.54		96.42	J	PU	N/A
287	634.33		77.66		PU	N/A
288	649.14		76.13		PU	N/A
289	649.68		111.8		PU	N/A
290	651.64		69.46		PU	N/A
291	662.78		128.57		PU	N/A
292	666.21		72.94		PU	N/A
293	667.57		82.92		PU	N/A
294	689.7		78.38	J	PU	N/A
295	700.85		55.31	J	PU	N/A
296	716.61		113.93	J	PU	N/A
297	722.04		54.75	J	PU	N/A
298	724.79		85.59		PU	N/A
299	739.8		81.79		PU	N/A
300	785.28		85.3		PU	N/A
301	795.67		60.69	J	PU	N/A
302	818.52		71.1		PU	N/A
303	824.06		80.89	J	PU	N/A
304	835.27		57.61		PU	N/A
305	866.72		92.24		PU	N/A
306	871.57		128.69		PU	N/A

UNIQUE ID	PPM PB	PB Qualifier	PPM As	As Qualifier	Grid Type	Area No.
307	894.31		73.54		PU	N/A
308	909.46		56.3		PU	N/A
309	917.06		132.55		PU	N/A
310	938.35		94.38		PU	N/A
311	961.02		68.86		PU	N/A
312	972.16		75.72		PU	N/A
313	978.59		54.37		PU	N/A
314	983.31		98.26		PU	N/A
315	1003.42		77.59	J	PU	N/A
316	1088.96		91.57		R	N/A
317	1142.49		140.39	J	PU	N/A
318	1188.02		72.42		PU	N/A
319	1223.85		68	J	PU	N/A
320	1236.73		122.57		PU	N/A
321	1319.74		103.13	J	PU	N/A
322	1353.8		133.14	J	PU	N/A
323	1400.82		106.19		PU	N/A
324	1689.66		129.45	J	PU	N/A
325	1795.68		93.15	J	PU	N/A
326	1834.87		99.28		PU	N/A
327	1863.59		120		PU	N/A
328	1950.84		133.51		PU	N/A

As - Arsenic

Grid Identifiers removed due to privacy regulations.

J - Estimated Value

Notes:

Pb - Lead

ppm - parts per million

U - not detected



LABORATORY COMPARISON DATA

FORMER MOHR ORCHARD

XRF Run No.	Nominal Seconds	Pb (ug/g)	Pb Error	Lab Confirmation Result (ug/g)	% D	As (ug/g)	As Error	Lab Confirmation Result (ug/g)	Qualifier	% D
67	300	155.89	6.48	161	-3.277952402	37.53	5.62	31.0		17.3994138
100	300	212.66	7.03	229	-7.683626446	35.71	5.94	31.5		11.78941473
133	300	286.98	8.84	286	0.341487212	41.84	7.43	40.0		4.397705545
146	300	167.01	7.06	165	1.203520747	41.12	6.14	34.5		16.09922179
189	300	184.48	6.87	190	-2.992194276	35.7	5.86	36.2		-1.400560224
206	300	187.61	6.89	210	-11.93433186	36.54	5.88	34.1		6.677613574
300	300	245.34	7.88	244	0.54618081	38.46	6.64	31.8		17.31669267
306	300	234.58	7.77	251	-6.999744224	36.09	6.54	33.6		6.899418121
310	300	184.29	7.23	179	2.87047588	39.07	6.2	34.9		10.67315076
311	300	256.67	8.52	250	2.59866755	36.29	7.13	30.6		15.67925048
348	150	1353.8	26.92	1400	-3.412616339	129.2	22.26	117		9.442724458
354	300	190.73	6.92	197	-3.28736958	39.3	5.93	37.4		4.834605598
361	150	1833.36	33.57	1820	0.728716673	148.93	27.59	123		17.41086416
420	150	35.02	5.99	29.8	14.90576813	7.77	4.96	7.6		2.187902188
499	300	318.08	8.52	345	-8.463279678	41.77	7.12	36.5		12.61671056
500	300	135.98	6.09	142	-4.427121636	36.64	5.32	31.6		13.75545852
503	300	248.72	7.81	265	-6.545513027	40.01	6.59	36.4		9.022744314
***	150			78.2	N/A	10.2	6.5	12.0		-17.64705882
565	300	229.55	7.72	241	-4.988020039	39.46	6.55	36.0		8.768373036
566	300	125.58	6.06	132	-5.112279025	35.78	5.31	29.7		16.99273337
721	300	215.5	6.9	231	-7.192575406	38.42	5.86	33.3		13.3263925
722	300	270.43	8.69	257	4.966164996	43.88	7.35	38.2		12.9443938
725	300	252.44	8.39	242	4.135636191	40.93	7.09	39.7		3.005130711
730	300	254.2	8.22	266	-4.642014162	40.63	6.94	35.1		13.61063254
946	300	174.02	6.93	177	-1.712446845	39.54	5.99	34.9	J	11.73495195
987	150	41.47	6.29	37.5	9.573185435	10.58	5.3	10.2	J	3.59168242
994	150	1238.74	28.27	1230	0.705555645	138.09	23.54	128	J	7.30682888
995	150	820.43	22.73	835	-1.775898005	92.32	18.92	105	J	-13.73483536
1040	150	617.85	19.6	631	-2.128348305	131.52	17.01	115	J	12.56082725
1102	300	280.92	8.08	286	-1.808344013	34.52	6.73	31.0	J	10.19698725
1134	150	48.87	6.5	44.0	9.965213833	11.38	5.47	7.0	J	38.48857645
1142	300	267.19	7.89	267	0.071110446	35.6	6.58	31.9	J	10.39325843
1143	300	231.18	7.75	229	0.942988148	36.34	6.53	32.0	J	11.9427628
1172	300	305.54	8.79	306	-0.150553119	41.75	7.35	42.0	J	-0.598802395
1178	300	253.02	8.37	244	3.564935578	42.53	7.09	42.1	J	1.011051023
1192	300	208.5	7.61	196	5.995203837	42.18	6.52	34.0	J	19.39307729

%D = Percent difference between XRF result and laboratory result.

*** = Sample not recorded in XRF. Relying on record in field notes.

APPENDIX C

EPA Method 6200

(31 pages)

METHOD 6200

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

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed in Table 1 for soil and sediment samples. Some common elements are not listed in Table 1 because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). They are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed in Table 1 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.

1.2 Detection limits depend on several factors, 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. General instrument detection limits for analytes of interest in environmental applications are shown in Table 1. These detection limits apply to a clean matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (600-second) count times. These detection limits are given for guidance only and will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of field performance-based detection limits is presented in Section 13.4 of this method. The clean matrix and field performance-based detection limits should be used for general planning purposes, and a third detection limit discussed, based on the standard deviation around single measurements, should be used in assessing data quality. This detection limit is discussed in Sections 9.7 and 11.3.

1.3 Use of this method is restricted to personnel either trained and knowledgeable in the operation of an XRF instrument or under the supervision of a trained and knowledgeable individual. This method is a screening method to be used with confirmatory analysis using EPA-approved methods. This method's main strength is as a rapid field screening procedure. The method detection limits (MDL) of FPXRF are above the toxicity characteristic regulatory level for most RCRA analytes. If the precision, accuracy, and detection limits of FPXRF meet the data quality objectives (DQOs) of your project, then XRF is a fast, powerful, cost effective technology for site characterization.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use sealed radioisotope sources to irradiate samples with x-rays. X-ray tubes are used to irradiate samples in the laboratory and are beginning to be incorporated into field portable instruments. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This later 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 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) or beta (β), 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 15.7 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: 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 standard.

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 and Chapter Three for additional definitions.

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, 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 no As being reported 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 by an EPA-approved method.

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 SW-846 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 data quality objectives.

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, the confirmatory method used was Method 3050, and the FPXRF data

compared very well with regression correlation coefficients (r^2 often exceeding 0.95, except for barium and chromium. See Table 9 in Section 17.0). The critical factor is that the digestion procedure and analytical reference method used should meet the data quality objectives (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 Section 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 to 20°F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 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 operators 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. Licenses for radioactive materials are of two types; (1) general license which is usually provided by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) specific license which is issued to named persons for the operation of radioactive instruments as required by local state agencies. 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. A copy of the radioactive material licenses 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. Finally, an additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.2 Radiation monitoring equipment should be used with the handling 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 should be worn in the area of most frequent 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.

5.3 Refer to Chapter Three for guidance on some proper safety protocols.

6.0 EQUIPMENT AND SUPPLIES

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: Most 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, cadmium (Cd)-109, americium (Am)-241, and curium (Cm)-244. These sources may be contained in a probe along with a window and the detector; the probe is 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. 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 required 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 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. 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 most FPXRF instruments operated in the intrusive mode, the probe is rotated so that the window faces upward. 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 $\text{Si}(\text{Li})$. 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 $\text{Si}(\text{Li})$ detector must be cooled to at least -90°C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a $\text{Si}(\text{Li})$ detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 liter. 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; $\text{Si}(\text{Li})$ -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 parts per million on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 100 to 500 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, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery chargers.

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

6.4 X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (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 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 required 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.

7.2 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.

7.2.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of ten 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.

7.2.2 Each sample should be oven-dried for 2 to 4 hours at a temperature of less than 150°C. If mercury is to be analyzed, a separate sample portion must remain undried, 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 ground with a mortar and pestle and passed through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.2.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 grams 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 grams 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.

7.3 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 method detection limits. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.4 Standard Reference Materials: Standard reference materials (SRM) 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.

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 Refer to Chapter One for additional guidance on quality assurance protocols. All field data sheets and quality control data should be maintained for reference or inspection.

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 Section 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (> 10 to 20°F).

The energy calibration check should be run at a frequency consistent with manufacturers 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.

9.2.1 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 radioactive 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: instrument blanks and method blanks. An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window.

9.3.1 The instrument blank can be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the method detection limits 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. To be acceptable, a method blank must not contain any analyte at a concentration above its method detection limit. If an analyte's concentration exceeds its method detection limit, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

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.

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 detection limit, but decreases sample throughput.

9.6 Detection Limits: Results for replicate analyses of a low-concentration sample, SSCS, or SRM can be used to generate an average site-specific method detection and quantitation limits. In this case, the method detection limit is defined as 3 times the standard deviation of the results for the low-concentration samples and the method quantitation limit is defined as 10 times the standard deviation of the same results. Another means of determining method detection and quantitation limits involves use of counting statistics. In FPXRF analysis, the standard deviation from counting statistics is defined as $\text{SD} = (\text{N})^{1/2}$, where SD is the standard deviation for a target analyte peak and N is the net counts for the peak of the analyte of interest (i.e., gross counts minus background under the peak). Three times this standard deviation would be the method detection limit and 10 times this standard deviation would be the method quantitation limit. If both of the above mentioned approaches are used to calculate method detection limits, the larger of the standard deviations should be used to provide the more conservative detection limits.

This SD based detection limit criteria must be used by the operator to evaluate each measurement for its useability. A measurement above the average calculated or manufacturer's detection limit, but smaller than three times its associated SD, should not be used as a quantitative measurement. Conversely, if the measurement is below the average calculated or manufacturer's detection limit, but greater than three times its associated SD. It should be coded as an estimated value.

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 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^2) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r^2 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: 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 required, 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 required.

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 Section 7.2. 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 Section 7.2; 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 required. 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 required to perform an adequate empirical calibration. The number of required 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 interference. 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 on in 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, one requirement is that any large or nonrepresentative debris be removed from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Another requirement is that the soil surface 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 data for this method, this modest amount of sample preparation was found to take less than 5 minutes 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 required detection limits.

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 grams or 250 cm³, which is enough soil to fill an 8-ounce jar. 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 Section 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 homogenization procedure using the fluorescein dye required 3 to 5 minutes per sample. As demonstrated in Sections 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, it can be used without the more labor intensive steps of drying, grinding, and sieving given in Sections 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps must 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 grams) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hours in the convection or toaster oven at a temperature not greater than 150°C. 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 minutes 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 MDLs of the procedure or DQOs of the analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in parts per million and can be downloaded to a PC, which can provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation.

13.0 METHOD PERFORMANCE

13.1 This section discusses four performance factors, field-based method detection limits, precision, accuracy, and comparability to EPA-approved methods. The numbers presented in Tables 4 through 9 were generated from data obtained from six FPXRF instruments. 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.

13.2 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 HgI₂ 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.

13.3 All data presented in Tables 4 through 9 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.

13.4 Field-Based Method Detection Limits: The field-based method detection limits are presented in Table 4. The field-based method detection limits were determined by collecting ten replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected method detection limits. Based on these ten replicate measurements, a standard deviation on the replicate analysis was calculated. The method detection limits presented in Table 4 are defined as 3 times the standard deviation for each analyte.

The field-based method detection limits were generated by using the count times discussed earlier in this section. All the field-based method detection limits were calculated for soil samples that had been dried and ground and placed in a sample cup with the exception of the MAP Spectrum Analyzer. This instrument can only be operated in the in situ mode, meaning the samples were moist and not ground.

Some of the analytes such as cadmium, mercury, silver, selenium, and thorium were not detected or only detected at very low concentrations such that a field-based method detection limit could not be determined. These analytes are not presented in Table 4. Other analytes such as calcium, iron, potassium, and titanium were only found at high concentrations (thousands of mg/kg) so that reasonable method detection limits could not be calculated. These analytes also are not presented in Table 4.

13.5 Precision Measurements: The precision data is presented in Table 5. 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 5 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the MDL 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 5. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the MDLs so that an RSD value calculated at 5 to 10 times the MDL 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 6 shows these results. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the detection limit of 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 6 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 6 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 versus 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 7 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 7 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 7. 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 7.

Table 8 provides a more detailed summary of accuracy data for one FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. Table 8 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 9. Similar trends in the data were seen for all instruments.

Table 9 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. 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--in situ, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not ground; and preparation 4--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 9 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 9 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.

Section 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 required to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 minutes. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 minutes per sample. Lastly, when grinding and sieving is conducted, time must 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 Hewitt, A.D. 1994. "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_{α} Peak Normalization Analysis." *American Environmental Laboratory*. Pages 24-32.

13.8.2 Piorek, S., and J.R. Pasmore. 1993. "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. Volume 2, Pages 1135-1151.

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 Street N.W., Washington D.C. 20036, (202) 872-4477.

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. 1994. Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction.
3. TN Spectrace. Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.
4. Unpublished SITE data, recieved from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 through 9 and a method procedure flow diagram.

**TABLE 1
INTERFERENCE FREE DETECTION LIMITS**

Analyte	Chemical Abstract Series Number	Detection Limit 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: References 1, 2, and 3

**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	458	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: Reference 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: Reference 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
FIELD-BASED METHOD DETECTION LIMITS (mg/kg)^a

Analyte	Instrument					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	55	NR	NR	NR	NR	NR
Arsenic	60	50	55	50	110	225
Barium	60	NR	30	400	NR	NR
Chromium	200	460	210	110	900	NR
Cobalt	330	NR	NR	NR	NR	NR
Copper	85	115	75	100	125	525
Lead	45	40	45	100	75	165
Manganese	240	340	NR	NR	NR	NR
Molybdenum	25	NR	NR	NR	30	NR
Nickel	100	NR	NA	NA	NA	NR
Rubidium	30	NR	NR	NR	45	NR
Strontium	35	NR	NR	NR	40	NR
Tin	85	NR	NR	NR	NR	NR
Zinc	80	95	70	NA	110	NA
Zirconium	40	NR	NR	NR	25	NR

Source: Reference 4

^a MDLs are related to the total number of counts taken. See Section 13.3 for count times used to generate this table.

NR Not reported.

NA Not applicable; analyte was reported but was not at high enough concentrations for method detection limit to be determined.

**TABLE 5
PRECISION**

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the MDL					
	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

Source: Reference 4

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

NR Not reported.

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

**TABLE 6
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

Source: Reference 4

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

ND Not detected.

NR Not reported.

**TABLE 7
ACCURACY**

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: Reference 4

- 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 8
ACCURACY FOR TN 9000^a**

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	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: Reference 4

- ^a All concentrations in milligrams per kilogram.
- %Rec. Percent recovery.
- ND Not detected.
- NA Not applicable.
- No data.

**TABLE 9
REGRESSION PARAMETERS FOR COMPARABILITY¹**

	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

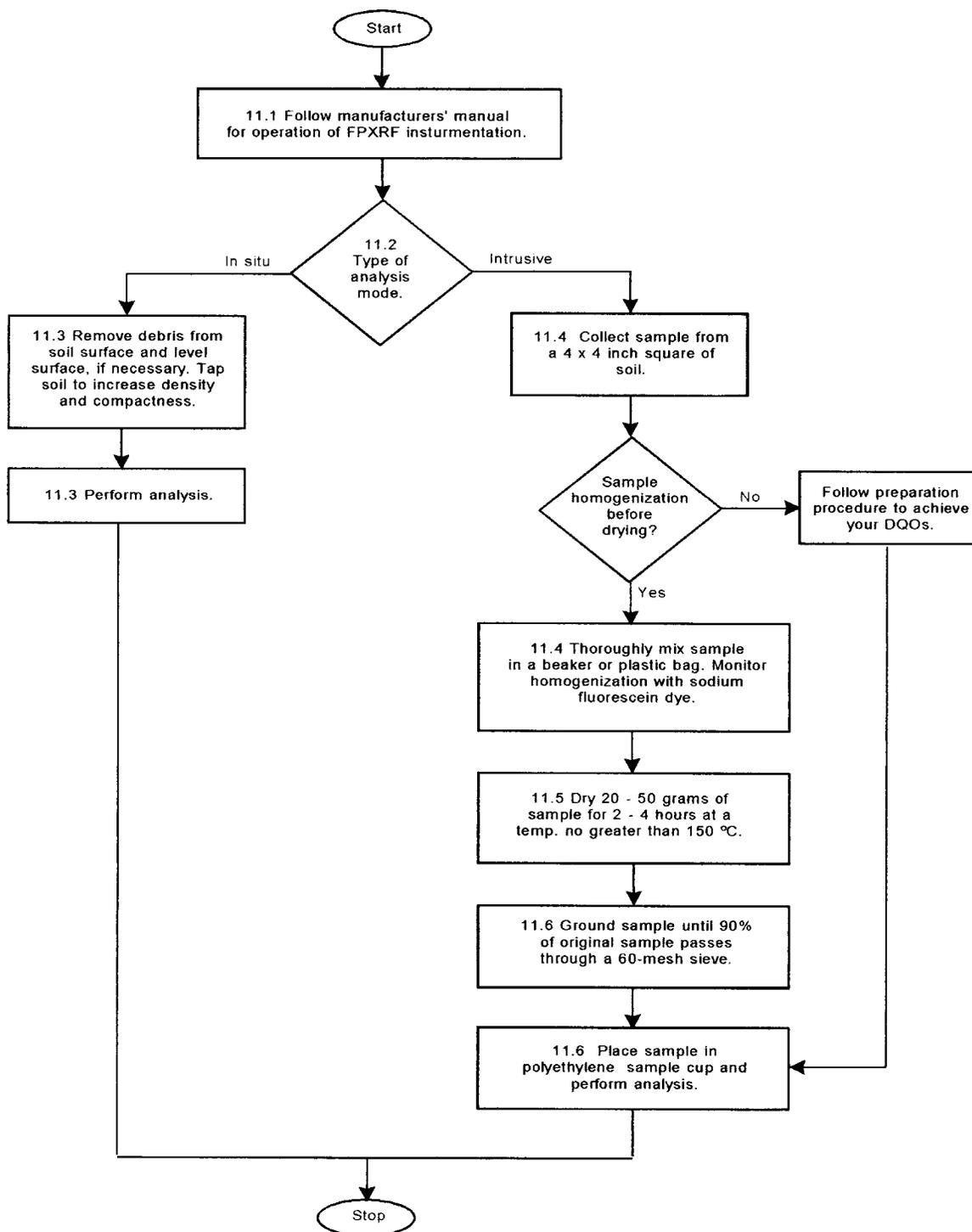
	Lead				Zinc				Chromium			
	n	r ²	Int.	Slope	n	r ²	Int.	Slope	n	r ²	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	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: Reference 4

- ¹ Log-transformed data
- n Number of data points
- r² 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 D

Data Quality Report

(3 pages)

December 31, 2008

Mr. Richard Fetzer (3HS31)
On-Scene Coordinator (OSC)
U.S. Environmental Protection Agency
1650 Arch Street
Philadelphia, PA 19103-2029

Subject: Former Mohr Orchard Site - Data Quality Report
EPA Contract No: EP-S3-05-02
Technical Direction Document No: E33-020-08-07-025
Document Tracking No. 0599

Dear Mr. Fetzer:

This report provides a general review of the Tetra Tech EM Inc. (Tetra Tech) x-ray fluorescence (XRF) analytical data package for the approximately 388 composite soil samples collected at the Former Mohr Orchard site in North Whitehall Township, Lehigh County, Pennsylvania, and analyzed on September 8 through November 9, 2008. The U.S. Environmental Protection Agency (EPA) asked that the samples be analyzed for arsenic using the Niton Model 700 XRF instrument. Later, lead was added as an analyte and the relevant results were extracted from the instrument's files, which include more than 20 metals. As part of the XRF assurance process, 7.5 percent of the samples (28 samples) were sent as Case No. 37485 to SVL Analytical, Inc., of Kellogg, Idaho, for confirmation analysis through the EPA Contract Laboratory Program (CLP). EPA validated the CLP reports so this report focuses on the review of the XRF data only.

The samples were analyzed for lead and arsenic by Tetra Tech using the ex-situ variant of EPA SW-846 Method 6200 from "Test Methods for Evaluation Solid Waste", September 1986. The samples were prepared for analysis by drying and sieving to minimize heterogeneity.

The XRF data package was reviewed in accordance with the EPA, "Region III Modifications to National Functional Guidelines for Inorganic Data Review Multi-Media, Multi-Concentration," April 1993, to level IM1 for inorganic analysis. Those guidelines were modified, as appropriate to conform to Method 6200 and the requirements of Tetra Tech's "Sampling and Analysis Plan for the Former Mohr Orchard Site, North Whitehall Township, Lehigh County, Pennsylvania" (SAP), dated September 4, 2008.

On the whole, the analyses went well, although there were minor problems with the data that caused data qualifiers to be applied to some results. There is a good correlation between the XRF and CLP data, as shown on the spreadsheet "Comparison XRF data.xls". For lead, the percent differences between the analyses averaged 4.7 percent, with a maximum of 14.9 percent for sample N34. For arsenic, the percent differences averaged 11.2 percent, with a maximum of 38.5 percent for sample W62. The higher percent differences are associated with the lower concentration results. The highest percent differences for both metals are associated with absolute differences that are similar to, or less than, the counting error that is inherent in the XRF analyses. Because of this characteristic of the XRF technology, all XRF results that were less than 20 milligrams per kilogram (mg/kg), the approximate level of the low-concentration standards for both metals, were flagged "J" to indicate that they are estimated.

The analyst followed the daily routine as established by Method 6200 and Tetra Tech's SAP. The instrument (method) blanks contained no detectable analytes. Sample replicates and continuing calibration standards (blank, low level, intermediate level, and high level) were analyzed as required at the start of each day, after every 20 samples, and at the end of the day to verify precision and accuracy. As documented in the daily summaries (included as "XRFdaily.zip"), there were occasional irregularities, but generally most results were acceptable and appropriate corrective actions were

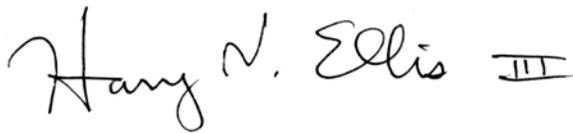
Mr. Richard Fetxer, OSC
Former Mohr Orchard Site
December 31, 2008
Page 2 of 2

performed when necessary. One notable example of this was at the start of the second day of analysis, when it was noted that the counting time of 90 seconds used with most of the samples on the first day was inadequate to provide acceptable sensitivity and precision. Therefore all following analyses were performed with a counting time of 150 seconds or longer and the samples analyzed on the first day were re-analyzed using the new conditions. Therefore the sample results from September 8, 2008, were rejected and the re-analyses used in lieu. The only significant irregularity with this daily routine occurred in the intermediate replicate/calibration run on September 16, 2008. The low-level standard for arsenic had an excessive percent difference (30.5 percent) from its known concentration. Therefore all arsenic results from that day that were less than the intermediate standard were flagged "J" to indicate that they are estimated and may be biased high.

Many field duplicates were analyzed and almost all of them gave very comparable results. For decision making, one should use the average results of these analyses. There were two exceptions. The first was sample AI60. The second analysis (identified as Reading No. 1001) had much higher concentrations than the first analysis. However, the next analysis (Reading No. 1002) was a high-concentration calibration standard that had concentrations very similar to those of Reading No. 1001. There seems to have been some sort of a mix-up with sample identification. Therefore Reading No. 1001 was flagged "R" to indicate that it is rejected and only Reading No. 1000 should be used for sample AI60. The other exception involved sample W52, which was analyzed three times. The arsenic results are generally similar for all, but the third analysis (Reading No. 913) had a lead concentration less than half that of the other two analyses. There is no apparent reason for this discrepancy. Therefore the results for Reading No. 913 were flagged "R" to indicate that they are rejected. Because of the uncertainty as to the cause of this discrepancy, the results from the other two analyses (Readings Nos. 792 and 912) were flagged "J" to indicate that they are considered estimated.

Tetra Tech recommends that the data be accepted as qualified. The qualified XRF results can be used in decision-making as if they were CLP results. The validated results are summarized in the spreadsheet "ValXRFDData.xls". Please contact me at (312) 201-7756 regarding any aspect of this report.

Sincerely,

A handwritten signature in black ink that reads "Harry N. Ellis III". The signature is written in a cursive style with a horizontal line under the name.

Harry Ellis
Toxicologist

cc: START TDD File

Enclosed: Spreadsheets with Validated Data

SPREADSHEETS WITH VALIDATED DATA

SPREADSHEETS REMOVED DUE TO PRIVACY REGULATIONS

APPENDIX E

Background Soil Sampling Data Table

(1 page)

Sample ID	Arsenic	Lead
BKG-SS01	6.2	41.4
BKG-SS02	5.1	31.3
BKG-SS03	5.4	45.5
BKG-SS05	5.3	38.9
BKG-SS06	3.5	20.6
BKG-SS07	9.7	52.6
BKG-SS08	7.8	31.1
BKG-SS09	9.3	47.4
BKG-SS10	11.6	98.7
BKG-SS11	29.9	218
BKG-SS12	14.8	31.1
BKG-SS13	10.5	39.1

Notes:

All results listed in parts per million
BKG - Background Sample
SS - Surface soil

ATTACHMENT

Validated Data Reports



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
ENVIRONMENTAL SCIENCE CENTER
701 MAPES ROAD
FORT MEADE, MARYLAND 20755-5350

DATE : December 2, 2008

SUBJECT: Region III Data QA Review

FROM : Khin-Cho Thaung *KCT*
Region III ESAT RPO (3ES20)

TO : Richard Fetzer
Regional Project Manager (3HS32)

Attached is the inorganic data validation report for the Former Mohr Orchard site (Case #: 37792, SDG#: MC0290) completed by the Region III Environmental Services Assistance Team (ESAT) contractor under the direction of Region III ESD.

If you have any questions regarding this review, please call me at (410) 305-2743.

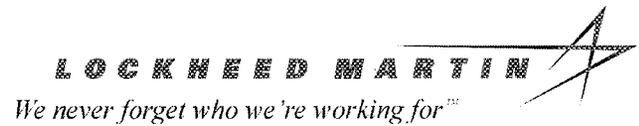
Attachments

cc: Joshua Cope (TTEMI)

TO File #: 0014 TDF#: 11058

OFFICE OF ANALYTICAL SERVICES AND QUALITY ASSURANCE

Lockheed Martin Enterprise Solutions & Services
ESAT Region 3
US EPA Environmental Science Center
701 Mapes Road Ft. Meade, MD 20755-5350
Telephone 410-305-3037 Facsimile 410-305-3597



Date: November 25, 2008

Subject: Inorganic Data Validation (IM2 Level)
Case: 37792
SDG : MC0290
Site : Former Mohr Orchard Arsenic

From: Kurt Roby *KR*
Inorganic Data Reviewer

Mahboobeh Mekanik *am*
Senior Oversight Chemist

To: Khin-Cho Thaug
ESAT Region 3 Project Officer

OVERVIEW

Case 37792, Sample Delivery Group (SDG) MC0290, consisted of twelve (12) soil samples analyzed for arsenic (As) and lead (Pb) by ICP-AES. Samples were analyzed by Bonner Analytical Testing Company (BONNER) according to Contract Laboratory Program (CLP) Statement of Work (SOW) ILM05.4 through the Routine Analytical Services (RAS) program.

SUMMARY

Data were validated according to Region III Modifications to the National Functional Guidelines for Inorganic Data Review, Level IM2. No problems were detected during the validation of this data set.

NOTES

Data for Case 37792, SDG MC0290, were reviewed in accordance with the National Functional Guidelines for Evaluating Inorganic Analyses with Modifications for use within Region III.

ATTACHMENTS

Appendix A Glossary of Data Qualifier Codes
Appendix B Data Summary Form(s)
Appendix C Chain of Custody Records
Appendix D Laboratory Case Narrative

DCN: 37792_ MC0290

Appendix A

Glossary of Data Qualifier Codes

GLOSSARY OF DATA QUALIFIER CODES (INORGANIC)

CODES RELATED TO IDENTIFICATION

(confidence concerning presence or absence of analytes):

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

B = Not detected substantially above the level reported in laboratory or field blanks.

R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

CODES RELATED TO QUANTITATION

(can be used for both positive results and sample quantitation limits):

J = Analyte Present. Reported value may not be accurate or precise.

K = Analyte present. Reported value may be biased high. Actual value is expected to be lower.

L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ = Not detected, quantitation limit may be inaccurate or imprecise.

UL = Not detected, quantitation limit is probably higher.

OTHER CODES

Q = No analytical result.

Appendix B

Data Summary Forms

DATA SUMMARY FORM: INORGANIC

Case #: 37792
 Site :
 Lab. :

SDG : MC0290
 FORMER MOHR ORCHARD ARSENIC
 BONNER

Number of Soil Samples : 12
 Number of Water Samples : 0

Sample Number :	MC02A0	MC0289	MC0290	MC0291	MC0292						
Sampling Location : (Prefix : FMO-81508-)	BKG-SS13	BKG-SS01	BKG-SS02	BKG-SS03	BKG-SS05						
Matrix :	Soil	Soil	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	8/15/2008	8/15/2008	8/15/2008	8/15/2008	8/15/2008						
Time Sampled :	16:30	09:30	10:24	10:51	11:30						
%Solids :	77.7	67.3	73.9	77.2	81.7						
Dilution Factor :	1.0	1.0	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	10.5		6.2		5.1		5.4		5.3	
*LEAD	1	39.1		41.4		31.3		45.5		38.9	

Sample Number :	MC0293	MC0294	MC0295	MC0296	MC0297						
Sampling Location : (Prefix : FMO-81508-)	BKG-SS06	BKG-SS07	BKG-SS08	BKG-SS09	BKG-SS10						
Matrix :	Soil	Soil	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	8/15/2008	8/15/2008	8/15/2008	8/15/2008	8/15/2008						
Time Sampled :	12:12	14:23	14:50	15:16	15:43						
%Solids :	72.5	78.0	65.8	74.4	76.3						
Dilution Factor :	1.0	1.0	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	3.5		9.7		7.8		9.3		11.6	
*LEAD	1	20.6		52.6		31.1		47.4		98.7	

Sample Number :	MC0298	MC0299									
Sampling Location : (Prefix : FMO-81508-)	BKG-SS11	BKG-SS12									
Matrix :	Soil	Soil									
Units :	mg/Kg	mg/Kg									
Date Sampled :	8/15/2008	8/15/2008									
Time Sampled :	16:03	16:18									
%Solids :	74.1	82.8									
Dilution Factor :	1.0	1.0									
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	29.9		14.8							
*LEAD	1	218		31.1							

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample quantitation limits: (CRQL * Dilution Factor) / (%Solids/ 100)

Revised 09/99

Appendix C

Chain of Custody Records



**USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record**

Case No: 37792

DAS No:

R

Region: Project Code: Account Code: CERCLIS ID: Spill ID: Site Name/State: Project Leader: Action: Sampling Co:	3 CT4349 NONE ALG Former Mohr Orchard/PA Erik Armistead Preliminary Assessment Tetra Tech EM Inc.	Date Shipped: Carrier Name: Airbill: Shipped to:	8/18/2008 FedEx 961942977746 Bonner Analytical Testing Company 2703 Oak Grove Road Hattiesburg MS 39402 (601) 264-2854	Relinquished By <i>Erik Armistead</i> 8/17/08 14:00	Sampler Signature: Received By	(Date / Time) (Date / Time)
---	--	---	---	---	-----------------------------------	--------------------------------

INORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	PRESERVATIVE/ Bottles	TAG No./	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	QC Type
MC0295	Soil (0"-12")/ Erik Armistead	L/G	As (14)	771 (Ice Only) (1)	FMO-081508-BKG-SS08 S:	8/15/2008	14:50 ✓		
MC0296	Soil (0"-12")/ Erik Armistead	L/G	As (14)	772 (Ice Only) (1)	FMO-081508-BKG-SS09 S:	8/15/2008	15:16 ✓		
MC0297	Soil (0"-12")/ Erik Armistead	L/G	As (14)	773 (Ice Only) (1)	FMO-081508-BKG-SS10 S:	8/15/2008	15:43 ✓		
MC0298	Soil (0"-12")/ Erik Armistead	L/G	As (14)	774 (Ice Only) (1)	FMO-081508-BKG-SS11 S:	8/15/2008	16:03 ✓		
MC0299	Soil (0"-12")/ Erik Armistead	L/G	As (14)	775 (Ice Only) (1)	FMO-081508-BKG-SS12 S:	8/15/2008	16:18 ✓		
MC02A0	Soil (0"-12")/ Erik Armistead	L/G	As (14)	776 (Ice Only) (1)	FMO-081508-BKG-SS13 S:	8/15/2008	16:30 ✓		

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: As = Arsenic, As-Water = Arsenic - Water	Concentration: L = Low, M = Low/Medium, H = High.	Type/Designate: Composite = C, Grab = G	Shipment lead?

TR Number: 3-375524367-081708-0001



Inorganic Traffic Report & Chain of Custody Record

DAS No:

Region: 3	Date Shipped: 8/18/2008	Carrier Name: FedEx	STATION LOCATION	ORGANIC SAMPLE No.	OC Type
Project Code: CT4349	Airbill: 961942977746	Shipped to: Bonner Analytical Testing 2703 Oak Grove Road Hattiesburg MS 39402 (601) 264-2854			
Account Code:					
CERCLIS ID: NONE					
Spill ID: ALG					
Site Name/State: Former Mohr Orchard/PA					
Project Leader: Erik Armistead					
Action: Preliminary Assessment					
Sampling Co: Tetra Tech EIM Inc.					

INORGANIC SAMPLE No.	MATRIX/SAMPLER	CONC/TYPE	ANALYSIS/TURNAROUND	PRESERVATIVE/ Bottles	TAG No./	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	OC Type
MC0284	Potable Well/ Erik Armistead	L/G	As-Water (14)	760 (HNO3) (1)		FMO--81508-BKG-PW04 S:	8/15/2008 11:20 ✓		
MC0285	Potable Well/ Erik Armistead	L/G	As-Water (14)	761 (HNO3) (1)		FMO--81508-BKG-PW06 S:	8/15/2008 12:28 ✓		
MC0286	Potable Well/ Erik Armistead	L/G	As-Water (14)	762 (HNO3) (1)		FMO--81508-BKG-PW07 S:	8/15/2008 14:31 ✓		
MC0287	Potable Well/ Erik Armistead	L/G	As-Water (14)	763 (HNO3) (1)		FMO--81508-BKG-PW09 S:	8/15/2008 15:20 ✓		
MC0288	Potable Well/ Erik Armistead	L/G	As-Water (14)	764 (HNO3) (1)		FMO--81508-BKG-PW11 S:	8/17/2008 13:38 ✓		
MC0289	Soil (0"-12")/ Erik Armistead	L/G	As (14)	765 (Ice Only) (1)		FMO-081508-BKG-SS01 S:	8/15/2008 9:30 ✓		
MC0290	Soil (0"-12")/ Erik Armistead	L/G	As (14)	766 (Ice Only) (1)		FMO-081508-BKG-SS02 S:	8/15/2008 10:24 ✓		
MC0291	Soil (0"-12")/ Erik Armistead	L/G	As (14)	767 (Ice Only) (1)		FMO-081508-BKG-SS03 S:	8/15/2008 10:51 ✓		
MC0292	Soil (0"-12")/ Erik Armistead	L/G	As (14)	768 (Ice Only) (1)		FMO-081508-BKG-SS05 S:	8/15/2008 11:30 ✓		
MC0293	Soil (0"-12")/ Erik Armistead	L/G	As (14)	769 (Ice Only) (1)		FMO-081508-BKG-SS06 S:	8/15/2008 12:12 ✓		
MC0294	Soil (0"-12")/ Erik Armistead	L/G	As (14)	770 (Ice Only) (1)		FMO-081508-BKG-SS07 S:	8/15/2008 14:23 ✓		

Handwritten notes:
 rec'd 10/15/08
 91038
 100454

Shipment for Case Complete? Y	Sample(s) to be used for laboratory OC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: AS = Arsenic, As-Water = Arsenic - water	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Used?

TR Number: 3-375524367-081708-0001

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
 Send Copy to Sample Management Office, Attn: Heather Bauer, C.S.C. 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

U.S. EPA Region III Analytical Request Form

ASQAB USE ONLY	
RAS#	Analytical TAT
DAS#	14
NSR#	

37792

2008TO3N302DC6C A3LGRS00

Date: 8/13/2008		Site Activity: RS - Removal	
Site Name: Former Mohr Orchard ARSENIC ASSESSMENT			
City: OREFIELD		Street Address: GOLDEN DR. & ROME CT	
State: PA		Longitude: 75.60078° W	
Latitude: 40.64622° N		CERCLIS #: --	
Program: Superfund		Acct. #: 08F3A505302DC6CA3LGRS00-	
Site ID: A3LG		Operable Unit:	
Site Specific QA Plan Submitted: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		Title: Mohr Orchard Abbreviated SAP	
Date Approved: 8/11/2008		E-mail: Fetzer.rich@epa.gov	
EPA Project Leader: RICHARD FETZER		Cell Phone #:	
Phone#: 215-341-6307		Cell Phone #: 215-768-8114	
Request Preparer: JOSHUA COPE		Cell Phone #: 267 446 2837	
Site Leader: ERIK ARMISTEAD		E-mail: Erik.armistead@ttemi.com	
Contractor: Tetra Tech EM Inc			
EPA CO/PO: Lorrrie Murray/Karen Wodarczyk		Method: ILM05.4 ICP-AES	
#Samples 15	Matrix: soil	Method: ILM05.4 ICP-AES	
#Samples 1	Matrix: water blank	Method: ILM05.4 ICP-AES	
#Samples	Matrix:	Method:	
#Samples	Matrix: 29099	Method: 9-25 Dec 9-25 (7TAT) MCO290	
#Samples	Matrix: 29100	Method:	
#Samples	Matrix: 29101	Method:	
#Samples	Matrix:	Method:	
#Samples	Matrix:	Method:	
Ship Date From: 8/18/2008		Ship Date To: 8/22/2008	
Org. Validation Level		Inorg. Validation Level IM2	
Unvalidated Data Requested: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		If Yes, TAT Needed: <input type="checkbox"/> 24hrs <input type="checkbox"/> 48hrs <input type="checkbox"/> 72hrs <input type="checkbox"/> 7days <input checked="" type="checkbox"/> Other (Specify) 14 days	
Validated Data Package Due: <input type="checkbox"/> 14 days <input type="checkbox"/> 21 days <input checked="" type="checkbox"/> 30 days <input type="checkbox"/> 42 days <input type="checkbox"/> Other (Specify)		14/16	
Electronic Data Deliverables Required: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		(EDDs will be provided in Region 3 EDD Format)	
Special Instructions: Detection limits for Arsenic CAS#7440382 are 1 mg/kg for soil and 10 ug/L for water. Hold samples for 90 days for possible additional analysis.			

Appendix D

Laboratory Case Narrative

COVER PAGE

Lab Name: Bonner Analytical Testing Company Contract: EPW06055
 Lab Code: BONNER Case No: 37792 NRAS No.: _____ SDG No: MC0290
 SOW No.: ILM05.4

EPA Sample No.	Lab Sample ID
<u>MC0289</u>	<u>0809236-01</u>
<u>MC0290</u>	<u>0809236-02</u>
<u>MC0290D</u>	<u>8091902-DUP1</u>
<u>MC0290S</u>	<u>8091902-MS1</u>
<u>MC0291</u>	<u>0809236-03</u>
<u>MC0292</u>	<u>0809236-04</u>
<u>MC0293</u>	<u>0809236-05</u>
<u>MC0294</u>	<u>0809236-06</u>
<u>MC0295</u>	<u>0809236-07</u>
<u>MC0296</u>	<u>0809236-08</u>
<u>MC0297</u>	<u>0809236-09</u>
<u>MC0298</u>	<u>0809236-10</u>
<u>MC0299</u>	<u>0809236-11</u>
<u>MC02A0</u>	<u>0809236-12</u>

		ICP-AES	ICP-MS
Were ICP-AES and ICP-MS interelement corrections applied?	(Yes/No)	<u>YES</u>	<u>YES</u>
Were ICP-AES and ICP-MS background corrections applied?	(Yes/No)	<u>YES</u>	<u>YES</u>
If yes, were raw data generated before application of background corrections?	(Yes/No)	<u>NO</u>	<u>NO</u>

Comments: _____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature:  Name: Christopher M. Bonner
 Date: 11/18/08 Title: Inorganic Laboratory Manager

Bonner Analytical Testing Company



2703 Oak Grove Road, Hattiesburg, MS 39402
Phone: (601) 264-2854 Fax: (601) 268-7084

SDG NARRATIVE:

SDG Number: MC0290

Case Number: 37792

Contract Number: EPW06055

Sample Receipt:

On October 9, 2008, we were asked to reanalyze SDG MC0290. On August 19, 2008, we received 12 soil samples under FedEx airbill number 9619 4297 7746. Custody seals were present and intact. Cooler temp was determined to be 1.5°C. Samples were received in good condition except for the following discrepancies:

1. There is no QC listed on the TR/COC for SDG MC0289. We would like to use MC0289 as QC. Please advise if this is acceptable.

Resolution: In accordance with previous direction from Region 3, the laboratory will select a sample for laboratory QC as long as the sample is not a PE, blank, or rinsate sample. The laboratory will note the issue in the Case/SDG Narrative, notify the SMO coordinator of the sample selected for laboratory QC, and proceed with the analysis of the samples.

SMO will note that the laboratory selected samples MC0284 (SDG MC0284) and MC0289 (SDG MC0289) as laboratory QC.

2. The laboratory was instructed on 9/24/08 to perform the reanalysis of the 12 soil samples for ICP-AES (Pb) with a start date of 9/24/08 and a TAT of 7 days; however, due to laboratory oversight the reanalysis of the soil samples was not performed.

Resolution 2: Per John Nebelsick, the laboratory will perform the reanalysis of the 12 soil samples for ICP-AES (As, Pb). The start date will be 10/9/08 and the TAT is 7 days. The reanalysis is billable. The laboratory will submit the data under SDG MC0290. The previously submitted reanalyzed ICP-AES (As) data under SDG MC0290 will be disregarded. The laboratory will note the issue in the Case/SDG Narrative and proceed with the reanalysis of the samples.

Metals

No Discrepancies

CSF:

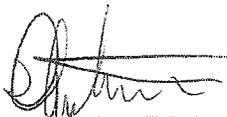
No Discrepancies

Sample Equation:

Lab ID Q809236-01 EPA Sample # MCO289

Date & Time 9/22/2009 @ 1220

Metals: $41.7 \mu\text{g/L}$ (0.100 L) 100% 1000 g 1 mg
(Analyte As) * (1.00 g) 67.3% 1 kg 1000 μg = 6.2 mg
Kg

Authorized by 
Daniel Antrim
Document Control Officer

Bonner Analytical Testing Company

Total Solids

SDG No: MC0289 Case No.: 37792 Batch No.: 082108A

Date Began: 08/21/08 Time Began: 17:00 Temperature Began: 109.0
 Date Finished: 08/22/08 Time Finished: 11:00 Temperature Finished: 108.0

EPA Sample ID	MC0289	MC0290	MC0291	MC0292	MC0293	MC0294	MC0295	MC0296	MC0297	MC0298
Laboratory ID	0808283-01	0808283-02	0808283-03	0808283-04	0808283-05	0808283-06	0808283-07	0808283-08	0808283-09	0808283-10
Pan ID	1	2	3	4	5	6	7	8	9	10
Pan Weight	1.01	0.99	0.97	1.03	1.02	1.00	1.02	1.00	1.01	1.01
Pan + Sample (Initial)	6.43	6.21	6.06	6.71	7.13	6.10	6.29	6.20	6.24	6.30
Sample Weight (Initial)	5.42	5.22	5.09	5.68	6.11	5.10	5.27	5.20	5.23	5.29
Pan + Sample (Final)	4.66	4.85	4.90	5.67	5.45	4.98	4.49	4.87	5.00	4.93
Sample Weight (Final)	3.65	3.86	3.93	4.64	4.43	3.98	3.47	3.87	3.99	3.92
Total Solids	67.3%	73.9%	77.2%	81.7%	72.5%	78.0%	65.8%	74.4%	76.3%	74.1%
% RSD	3.06%									

Sample ID	MC0299	MC02A0								
Laboratory ID	0808283-11	0808283-12	0808283-13	0808283-14	0808283-15	0808283-16	0808283-17	0808283-18	0808283-19	0808283-20
Pan ID	11	12	13	14	15	16	17	18	19	20
Pan Weight	0.99	1.01								
Pan + Sample (Initial)	7.05	6.58								
Sample Weight (Initial)	5.06	5.57	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Pan + Sample (Final)	5.01	5.34								
Sample Weight (Final)	5.02	4.33	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Total Solids	82.8%	77.7%	#DIV/0!							

Weighed By _____ Date: _____
 Analyst: _____ Date: _____
 Supervisor: *[Signature]* Date: *2/29/18*

Bethany Whitehead

From: Walsh, Colin [cwalsh20@fedcsc.com]
Sent: Thursday, October 09, 2008 2:05 PM
To: Bethany Whitehead; Chris Bonner
Cc: slizys.dan@epa.gov; Harris.Carroll@epamail.epa.gov; thaung.khin-cho@epa.gov; kwedar.john@epa.gov; Nebelsick.John@epamail.epa.gov; Bauer, Heather
Subject: Region 03 | Case 37792 | Lab BONNER | Issue Reanalysis | FINAL

Beth,

Summary Start

-Record of Communication Update-

This ROC has been updated to include that the laboratory will reanalyze the 12 soil samples for ICP-AES (As, Pb) and replace SDG MC0290 with the new reanalysis data.

Please note that per John Nebelsick, the laboratory will perform the reanalysis of the 12 soil samples for ICP-AES (As, Pb). The start date will be 10/9/08 and the TAT is 7 days. The reanalysis is billable. The laboratory will submit the data under SDG MC0290. The previously submitted reanalyzed ICP-AES (As) soil data under SDG MC0290 will be disregarded. The laboratory will note the issue in the Case/SDG Narrative and proceed with the reanalysis of the samples.

Summary End

Please let me know if you have any questions or problems.

Thanks,

Colin

Colin G. Walsh
Environmental Coordinator - Region 3
CSC

15000 Conference Center Drive, Chantilly, VA 20151
 Civil Division | (p) 703-818-4544 | (f) 703-818-4602 | cwalsh20@fedcsc.com | www.csc.com

 10/9/08, 12:30 PM, Phone conversation between John Nebelsick (EPA) and Heather Bauer (SMO). John indicated that the laboratory will reanalyze the 12 soil samples for ICP-AES (As, Pb). The laboratory will resubmit SDG MC0290 with the ICP-AES (As, Pb) reanalysis data. The previously submitted reanalyzed ICP-AES (As) soil data under SDG MC0290 will be disregarded. The start date will be 10/9/08 and the TAT is 7 days. The reanalysis is billable.

From: Walsh, Colin
Sent: Monday, September 22, 2008 10:00 AM
To: Chris Bonner; 'Bethany Whitehead'
Cc: 'slizys.dan@epa.gov'; Harris.Carroll@epamail.epa.gov; thaung.khin-cho@epa.gov; kwedar.john@epa.gov
Subject: Region 03 | Case 37792 | Lab BONNER | Issue Reanalysis | FINAL

Chris,

Summary Start

11/18/2008

Bethany Whitehead

From: Walsh, Colin [cwalsh20@fedcsc.com]
Sent: Thursday, October 09, 2008 1:56 PM
To: Bethany Whitehead; Chris Bonner
Cc: slizys.dan@epa.gov; Harris.Carroll@epamail.epa.gov; thaung.khin-cho@epa.gov; kwedar.john@epa.gov; Nebelsick.John@epamail.epa.gov; Bauer, Heather
Subject: Region 03 | Case 37792 | Lab BONNER | Issue Reanalysis | FINAL

Beth,

Summary Start

-Record of Communication Update-

This ROC has been updated to include that the laboratory will reanalyze the 12 soil samples for ICP-AES (As, Pb) and replace SDG MC0290 with the new reanalysis data.

Issue: The laboratory was instructed on 9/24/08 to perform the reanalysis of the 12 soil samples for ICP-AES (Pb) with a start date of 9/24/08 and a TAT of 7 days; however, due to laboratory oversight the reanalysis of the soil samples was not performed.

Resolution: Per John Nebelsick, the laboratory will perform the reanalysis of the 12 soil samples for ICP-AES (As, Pb). The start date will be 10/9/08 and the TAT is 7 days. The reanalysis is billable. The laboratory will submit the data under SDG MC0290. The previously submitted reanalyzed ICP-AES (As) data under SDG MC0290 will be disregarded. The laboratory will note the issue in the Case/SDG Narrative and proceed with the reanalysis of the samples.

Summary End

Please let me know if you have any questions or problems.

Thanks,

Colin

Colin G. Walsh
Environmental Coordinator - Region 3
CSC

15000 Conference Center Drive, Chantilly, VA 20151
Civil Division | (p) 703-818-4544 | (f) 703-818-4602 | cwalsh20@fedcsc.com | www.csc.com

10/9/08, 12:30 PM, Phone conversation between John Nebelsick (EPA) and Heather Bauer (SMO). John indicated that the laboratory will reanalyze the 12 soil samples for ICP-AES (As, Pb). The laboratory will resubmit SDG MC0290 with the ICP-AES (As, Pb) reanalysis data. The previously submitted reanalyzed ICP-AES (As) data under SDG MC0290 will be disregarded. The start date will be 10/9/08 and the TAT is 7 days. The reanalysis is billable.

From: Beth Whitehead [mailto:bwhitehead@batco.com]
Sent: Wednesday, October 08, 2008 4:44 PM
To: Walsh, Colin
Cc: Chris Bonner
Subject: RE: Region 03 | Case 37792 | Lab BONNER | Issue Reanalysis | FINAL

11/18/2008



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
ENVIRONMENTAL SCIENCE CENTER
701 MAPES ROAD
FORT MEADE, MARYLAND 20755-5350

DATE : November 5, 2008

SUBJECT: Region III Data QA Review

FROM : Khin-Cho Thaung *KCT*
Region III ESAT RPO (3ES20)

TO : Richard Fetzner
Regional Project Manager (3HS32)

Attached is the inorganic data validation report for the Former Mohr Orchard site (Case #: 37845, SDG#: MC02Y5) completed by the Region III Environmental Services Assistance Team (ESAT) contractor under the direction of Region III ESD.

If you have any questions regarding this review, please call me at (410) 305-2743.

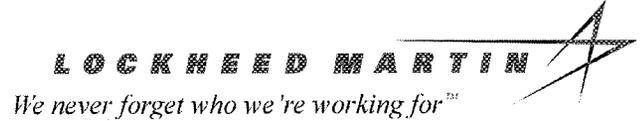
Attachments

cc: Joshua Cope (TTEMI)

TO File #: 0014 TDF#: 11001

OFFICE OF ANALYTICAL SERVICES AND QUALITY ASSURANCE

Lockheed Martin Enterprise Solutions & Services
ESAT Region 3
US EPA Environmental Science Center
701 Mapes Road Ft. Meade, MD 20755-5350
Telephone 410-305-3037 Facsimile 410-305-3597



Date: November 5, 2008

Subject: Inorganic Data Validation (IM2 Level)
Case: 37845
SDG : MC02Y5
Site : Former Mohr Orchard Arsenic

From: Kurt Roby *KR*
Inorganic Data Reviewer

Mahboobeh Mekanik *mm*
Senior Oversight Chemist

To: Khin-Cho Thaug
ESAT Region 3 Project Officer

OVERVIEW

Case 37845, Sample Delivery Group (SDG) MC02Y5, consisted of twelve (12) soil samples analyzed for arsenic (As) and lead (Pb) by ICP-AES. Samples were analyzed by SVL Analytical, Inc. (SVL) according to Contract Laboratory Program (CLP) Statement of Work (SOW) ILM05.4 through the Routine Analytical Services (RAS) program.

SUMMARY

Data were validated according to Region III Modifications to the National Functional Guidelines for Inorganic Data Review, Level IM2. Areas of concern with respect to data usability are listed below.

Data in this case have been impacted by an outlier present in the ICP serial dilution analysis. The detail of this outlier is discussed under "Minor Problem," specific samples affected are outlined in "Table 1A" and qualified analytical results for all samples are summarized on the Data Summary Form (DSF).

MINOR PROBLEM

Percent difference (%D) in the ICP serial dilution analysis was outside the control limit (>10%) for arsenic (As). Positive results for this analyte in all samples are estimated due to possible matrix interferences and have been qualified "J" on the DSF.

NOTES

Per laboratory case narrative, insufficient sample volumes were received by the laboratory to perform percent solids determination. Samples were received in XRF cups, appearing dried and pulverized. Per Region III, the laboratory was directed to report percent solids as 100%.

Data for Case 37845, SDG MC02Y5, were reviewed in accordance with the National Functional Guidelines for Evaluating Inorganic Analyses with Modifications for use within Region III.

ATTACHMENTS

INFORMATION REGARDING REPORT CONTENT

Table 1A is a summary of qualifiers applied to the laboratory-generated results during data validation.

Table 1A	Summary of qualifiers on data summary forms after data validation
Table 1B	Codes used in comments column of Table 1A
Appendix A	Glossary of Data Qualifier Codes
Appendix B	Data Summary Form(s)
Appendix C	Chain of Custody Records
Appendix D	Laboratory Case Narrative

DCN: 37845_MC02Y5

TABLE 1A
SUMMARY OF QUALIFIERS ON DATA SUMMARY
FORM AFTER DATA VALIDATION

Case 37845, SDG MC02Y5

<u>ANALYTE</u>	<u>SAMPLES AFFECTED</u>	<u>POSITIVE</u> <u>VALUES</u>	<u>NON-</u> <u>DETECTED</u> <u>VALUES</u>	<u>BIAS</u>	<u>COMMENTS*</u>
As	All samples	J			ISD (10.4%)

* See explanation of comments in Table 1B

TABLE 1B
CODES USED IN COMMENTS COLUMN

ISD = Percent difference (%D) in the ICP serial dilution analysis was outside the control limit (>10%) [%D is in parenthesis]. Positive results are estimated.

Appendix A

Glossary of Data Qualifier Codes

GLOSSARY OF DATA QUALIFIER CODES (INORGANIC)

CODES RELATED TO IDENTIFICATION

(confidence concerning presence or absence of analytes):

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

B = Not detected substantially above the level reported in laboratory or field blanks.

R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

CODES RELATED TO QUANTITATION

(can be used for both positive results and sample quantitation limits):

J = Analyte Present. Reported value may not be accurate or precise.

K = Analyte present. Reported value may be biased high. Actual value is expected to be lower.

L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ = Not detected, quantitation limit may be inaccurate or imprecise.

UL = Not detected, quantitation limit is probably higher.

OTHER CODES

Q = No analytical result.

Appendix B

Data Summary Forms

DATA SUMMARY FORM: INORGANIC

Case #: 37845
 Site :
 Lab. :

SDG : MC02Y5
 FORMER MOHR ORCHARD ARSENIC
 SVL

Number of Soil Samples : 12
 Number of Water Samples : 0

Sample Number :	MC02Y5	MC02Y6	MC02Y7	MC02Y8	MC02Y9						
Sampling Location : (Prefix : FMO08-)	SS-AG22	SS-011	SS-Q07	SS-Q13	SS-S07						
Matrix :	Soil	Soil	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	10/1/2008	10/8/2008	10/2/2008	10/2/2008	10/2/2008						
Time Sampled :	10:35	17:07	14:27	08:53	14:50						
%Solids :	100.0	100.0	100.0	100.0	100.0						
Dilution Factor :	1.0	1.0	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	34.9	J	42.0	J	10.2	J	31.0	J	31.9	J
*LEAD	1	177		306		37.5		286		267	

Sample Number :	MC02Z0	MC02Z1	MC02Z2	MC02Z3	MC02Z4						
Sampling Location : (Prefix : FMO08-)	SS-S09	SS-U03	SS-V03	SS-V12	SS-W02						
Matrix :	Soil	Soil	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	10/2/2008	10/3/2008	10/3/2008	10/2/2008	10/6/2008						
Time Sampled :	15:00	12:20	12:32	18:20	11:50						
%Solids :	100.0	100.0	100.0	100.0	100.0						
Dilution Factor :	1.0	1.0	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	32.0	J	105	J	128	J	42.1	J	115	J
*LEAD	1	229		835		1230		244		631	

Sample Number :	MC02Z5	MC02Z6									
Sampling Location : (Prefix : FMO08-)	SS-W13	SS-W62									
Matrix :	Soil	Soil									
Units :	mg/Kg	mg/Kg									
Date Sampled :	10/2/2008	10/8/2008									
Time Sampled :	18:07	10:25									
%Solids :	100.0	100.0									
Dilution Factor :	1.0	1.0									
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	34.0	J	7.0	J						
*LEAD	1	196		44.0							

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample quantitation limits: (CRQL * Dilution Factor) / (%Solids/ 100) .

Revised 09/99

Appendix C

Chain of Custody Records



**USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record**

Case No: 37845

DAS No:

R

Region: 3		Date Shipped: 10/13/2008		Chain of Custody Record	
Project Code: CT4362		Carrier Name: FedEx		Sampler Signature:	
Account Code:		Airbill: 857499852809		Received By (Date / Time)	
CERCLIS ID: None		Shipped to: SVL Analytical Inc One Government Gulch Kellogg ID 83837 (208) 784-1258		1.	
Spill ID: ALG				2.	
Site Name/State: Former Mohr Orchard Assessment/PA				3.	
Project Leader: Erik Armistead				4.	
Action: Preliminary Assessment					
Sampling Co: Tetra Tech EM Inc.					

INORGANIC SAMPLE NO.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	PRESERVATIVE/ Bottles	TAG No./	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	QC Type
MC02Y2	Potable Well/ Erik Armistead	L/G	Pb/As - Wa (14)	1022 (HNO3) (1)		FMO08-PW-AE6301	S: 10/10/2008 8:45		
MC02Y3	Potable Well/ Erik Armistead	L/G	Pb/As - Wa (14)	1023 (HNO3) (1)		FMO08-PW-X6201	S: 10/9/2008 17:42		
MC02Y4	Potable Well/ Erik Armistead	L/G	Pb/As - Wa (14)	1024 (HNO3) (1)		FMO08-PW-X6202	S: 10/10/2008 16:18		
MC02Y5	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1025 (Ice Only) (1)		FMO08-SS-AG22	S: 10/1/2008 10:35		
MC02Y6	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1026 (Ice Only) (1)		FMO08-SS-O11	S: 10/8/2008 17:07		
MC02Y7	Soil (0"-12")/ Erik Armistead	L/C	Pb/As - So (14)	1027 (Ice Only) (1)		FMO08-SS-Q07	S: 10/2/2008 14:27		
MC02Y8	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1028 (Ice Only) (1)		FMO08-SS-Q13	S: 10/2/2008 8:53		
MC02Y9	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1029 (Ice Only) (1)		FMO08-SS-S07	S: 10/2/2008 14:50		
MC02Z0	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1030 (Ice Only) (1)		FMO08-SS-S09	S: 10/2/2008 15:00		
MC02Z1	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1031 (Ice Only) (1)		FMO08-SS-U03	S: 10/3/2008 12:20		
MC02Z2	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1032 (Ice Only) (1)		FMO08-SS-V03	S: 10/3/2008 12:32		

Shipment for Case Complete? N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: Pb/As - So = Lead and Arsenic - Soil, Pb/As - Wa = Lead and Arsenic - Water	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____

TR Number: 3-375524367-101008-0001

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

REGIONAL



**USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record**

Case No: 37845

DAS No:

R

Region: Project Code: Account Code: CERCLIS ID: Spill ID: Site Name/State: Project Leader: Action: Sampling Co:	3 CT4362 None ALG Former Mohr Orchard Assessment/PA Erik Armistead Preliminary Assessment Tetra Tech EM Inc.	Date Shipped: Carrier Name: Airbill: Shipped to:	10/13/2008 FedEx 857499852809 SVL Analytical Inc One Government Gulch Kellogg ID 83837 (208) 784-1258
Chain of Custody Record		Sampler Signature:	Received By (Date / Time)
Relinquished By		1	
2.			
3.			
4.			

INORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	QC Type
MC02Z3	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1033 (Ice Only) (1)	FMO08-SS-V12	S: 10/2/2008 18:20 ✓		-
MC02Z4	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1034 (Ice Only) (1)	FMO08-SS-W02	S: 10/6/2008 11:50 ✓		-
MC02Z5	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	1035 (Ice Only) (1)	FMO08-SS-W13	S: 10/2/2008 18:07 ✓		-
MC02Z6	Soil (0"-12")/ Erik Armistead	L/C	Pb/As - So (14)	1036 (Ice Only) (1)	FMO08-SS-W62	S: 10/8/2008 10:25 ✓		-

Shipment for Case Complete? N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: Pb/As - So = Lead and Arsenic - Soil, Pb/As - Wa = Lead and Arsenic - Water	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____

TR Number: 3-375524367-101008-0001

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

REGION COPY

9-8-08 275

ASQAB USE ONLY	
RAS#	CT4362 Analytical TAT
DAS#	14
NSF#	

U.S. EPA Region III Analytical Request Form

37845

Date: 9/4/2008		Site Activity: Removal Site Evaluation	
Site Name: Former Mohr Orchard Arsenic		Street Address: Orefield, Pennsylvania	
City: North Whitehall Township		State: PA	Latitude: 40.64622° N
Program: Superfund		Acct. #: 2008T03 N 302DC6CA3LGRS00	Longitude: 75.60078° W
Site ID: A3LG		CERCLIS #: PAN00306624	Operable Unit:
Site Specific QA Plan Submitted: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		Title: Draft Former Mohr Orchard SAP	Date Approved:
EPA Project Leader: RICHARD FETZER	Phone#: 215-341-6307	Cell Phone #:	E-mail: Fetzer.rich@epa.gov
Request Preparer: JOSHUA COPE	Phone#: 610-364-2130	Cell Phone #: 215-768-8114	E-mail: Joshua.cope@ttemi.com
Site Leader: ERIK ARMISTEAD	Phone#: 610-364-2151	Cell Phone #: 267 446 2837	E-mail: Erik.armistead@ttemi.com
Contractor: Tetra Tech EM Inc			
#Samples 85	Matrix: soil	Parameter: Arsenic and Lead	Method: ILM05.4 ICPAES 290403
#Samples 10-50	Matrix: potable water	Parameter: Total Arsenic	Method: ILM05.4 ICPMS 29044
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
Ship Date From: 9/12/2008		Ship Date To: 10/3/2008	Org. Validation Level
Unvalidated Data Requested: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		If Yes, TAT Needed: <input type="checkbox"/> 24hrs <input type="checkbox"/> 48hrs <input type="checkbox"/> 72hrs <input type="checkbox"/> 7days <input checked="" type="checkbox"/> Other (Specify) 14 days	Inorg. Validation Level IM2
Validated Data Package Due: <input type="checkbox"/> 14 days <input type="checkbox"/> 21 days <input checked="" type="checkbox"/> 30days <input type="checkbox"/> 42 days <input type="checkbox"/> Other (Specify)		14/16	
Electronic Data Deliverables Required: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes (EDDs will be provided in Region 3 EDD Format)			
Special Instructions: Potable waters will ship weekly 9/12, 9/19, and 9/26. Soils will be shipped at end of the sampling event between 9/26 and 10/3. Detection limits are Lead @ 1 mg/kg and Arsenic @ 1 mg/kg for soil and Arsenic @ 1 ug/L for potable water. Reg'd regional CDC 10-29-08			

Appendix D

Laboratory Case Narrative

USEPA-CLP

COVER PAGE

Lab Name: SVL Analytical, Inc. Contract: EP-W-06-056
 Lab Code: SVL Case No: 37845 NRAS No.: _____ SDG No: MC02Y5
 SOW No.: ILM05.4

EPA Sample No.	Lab Sample ID
<u>MC02Y5</u>	<u>MC02Y5</u>
<u>MC02Y5D</u>	<u>MC02Y5D</u>
<u>MC02Y5S</u>	<u>MC02Y5S</u>
<u>MC02Y6</u>	<u>MC02Y6</u>
<u>MC02Y7</u>	<u>MC02Y7</u>
<u>MC02Y8</u>	<u>MC02Y8</u>
<u>MC02Y9</u>	<u>MC02Y9</u>
<u>MC02Z0</u>	<u>MC02Z0</u>
<u>MC02Z1</u>	<u>MC02Z1</u>
<u>MC02Z2</u>	<u>MC02Z2</u>
<u>MC02Z3</u>	<u>MC02Z3</u>
<u>MC02Z4</u>	<u>MC02Z4</u>
<u>MC02Z5</u>	<u>MC02Z5</u>
<u>MC02Z6</u>	<u>MC02Z6</u>

	(Yes/No)	<u>ICP-AES</u>	<u>ICP-MS</u>
Were ICP-AES and ICP-MS interelement corrections applied?		<u>YES</u>	<u>NO</u>
Were ICP-AES and ICP-MS background corrections applied?		<u>YES</u>	<u>NO</u>
If yes, were raw data generated before application of background corrections?		<u>NO</u>	<u>NO</u>

Comments: _____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: *Kirby L. Gray* Name: KIRBY L. GRAY
 Date: 10/29/2008 Title: TECHNICAL DIRECTOR

SDG NARRATIVE

LAB NAME: SVL ANALYTICAL, INC.
LAB CODE: SVL
CONTRACT NO.: EP-W-06-056

CASE: 37845
SDG: MC02Y5

SAMPLES RECEIVED FOR ICP-AES ON OCTOBER 14, 2008.
SAMPLE TAGS WERE PRESENT.
COOLER TEMPERATURE INDICATOR BOTTLE ABSENT.

ISSUE 1: A TEMPERATURE BLANK WAS NOT INCLUDED WITH THE SAMPLES RECEIVED ON 10/10/08 AND 10/14/08. THE TEMPERATURE WAS DETERMINED TO BE 0.2 DEGREES CELSIUS AND 4.0 DEGREES CELSIUS BY IR GUN FOR THE SHIPMENTS RECEIVED ON 10/10/08 AND 10/14/2008, RESPECTIVELY.

RESOLUTION 1: IN ACCORDANCE WITH PREVIOUS DIRECTION FROM REGION 3, THE LABORATORY WILL NOTE THE ISSUE, AND THE METHOD USED TO DETERMINE THE TEMPERATURE, IN THE CASE/SDG NARRATIVE AND PROCEED WITH THE ANALYSIS OF THE SAMPLES.

ISSUE 2: LABORATORY QC IS NOT DESIGNATED ON THE TR/COC; HOWEVER, THE SCHEDULING NOTIFICATION FORM LISTS THAT LABORATORY QC IS REQUIRED. THE LABORATORY WOULD LIKE TO SELECT SAMPLES MC02X9 (SDG MC02X9) AND MC02Y5 (SDG MC02Y5) AS LABORATORY QC.

RESOLUTION 2: IN ACCORDANCE WITH PREVIOUS DIRECTION FROM REGION 3, THE LABORATORY WILL SELECT A SAMPLE FOR LABORATORY QC AS LONG AS THE SAMPLE IS NOT A PE, BLANK, OR RINSATE SAMPLE. THE LABORATORY WILL NOTE THE ISSUE IN THE CASE/SDG NARRATIVE, NOTIFY THE SMO COORDINATOR OF THE SAMPLE SELECTED FOR LABORATORY QC, AND PROCEED WITH THE ANALYSIS OF THE SAMPLES. SMO WILL NOTE THAT THE LABORATORY SELECTED SAMPLES MC02X9 (SDG MC02X9) AND MC02Y5 (SDG MC02Y5) AS LABORATORY QC.

ISSUE 3: THERE IS INSUFFICIENT VOLUME TO PERFORM % SOLIDS FOR SDG MC02Y5. THE SOIL SAMPLES APPEAR TO BE DRIED AND PULVERIZED AND WERE RECEIVED IN XRF CUPS. THE LABORATORY WOULD LIKE TO KNOW IF THEY CAN REPORT PERCENT SOLIDS AS 100%?

RESOLUTION 3: PER REGION 3, THE LABORATORY CAN REPORT THE % SOLIDS AS 100% FOR THE SAMPLES IN SDG MC02Y5. THE LABORATORY WILL NOTE THE ISSUE IN THE CASE/SDG NARRATIVE AND PROCEED WITH THE ANALYSIS OF THE SAMPLES.

ICP-AES EXAMPLE CALCULATION: MC02Y5 LEAD

$$\text{CONCENTRATION (DRY WT.) (MG/KG)} = \frac{C \times V}{W \times S} \times \text{DF}$$

C = CONCENTRATION (mg/L)
V = FINAL SAMPLE VOLUME IN LITERS (L)
W = WET SAMPLE WEIGHT (kg)
S = % SOLIDS/100
DF = DILUTION FACTOR

$$\frac{1.77122 \text{ mg/L} \times 0.1 \text{ L}}{0.001 \text{ kg} \times 100.0/100} \times 1 = 177 \text{ mg/kg}$$



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
ENVIRONMENTAL SCIENCE CENTER
701 MAPES ROAD
FORT MEADE, MARYLAND 20755-5350

DATE : October 22, 2008

SUBJECT: Region III Data QA Review

FROM : Khin-Cho Thaung *KCT*
Region III ESAT RPO (3ES20)

TO : Richard Fetzer
Regional Project Manager (3HS32)

Attached is the inorganic data validation report for the Former Mohr Orchard site (Case #: 37845, SDG#: MC02P4) completed by the Region III Environmental Services Assistance Team (ESAT) contractor under the direction of Region III ESD.

If you have any questions regarding this review, please call me at (410) 305-2743.

Attachments

cc: Joshua Cope (TTEMI)

TO File #: 0014 TDF#: 1055

OFFICE OF ANALYTICAL SERVICES AND QUALITY ASSURANCE



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Lockheed Martin Enterprise Solutions & Services
ESAT Region 3
US EPA Environmental Science Center
701 Mapes Road Ft. Meade, MD 20755-5350
Telephone 410-305-3037 Facsimile 410-305-3597

DATE: October 21, 2008

SUBJECT: Level IM2 Inorganic Data Validation for Case 37485
SDG: MC02P4
Site: Former Mohr Orchard

FROM: Shilpa Udani
Inorganic Data Reviewer

Through: Mahboobeh Mecanic *Plus*
SM Senior Data Review Chemist

TO: Colleen Walling
ESAT Region 3 Project Officer

OVERVIEW

Case 37485, Sample Delivery Group (SDG) MC02P4, consisted of sixteen (16) soil samples submitted to SVL (SVL) for arsenic (As) and lead (Pb) analyses. The sample set included no field Quality Control (QC) samples. Samples were analyzed in accordance with Contract Laboratory Program (CLP) Statement of Work (SOW) ILM05.4 through the Routine Analytical Services (RAS) program.

SUMMARY

Data were validated according to the Region III Modifications to the National Functional Guidelines for Inorganic Data Review level IM2. No problems regarding data usability were noted during the review of this data set. The analytical results for this sample set are summarized on a single Data Summary Form (DSF) in Appendix B.

NOTE

Samples were received in XRF cups with insufficient sample size for percent solids determination. Results are reported on an "as received" basis.

Data for Case 37845, SDG MC02P4, were reviewed in accordance with National Functional Guidelines for Evaluating Inorganic Analyses with Modifications for use within Region III.

ATTACHMENTS

INFORMATION REGARDING REPORT CONTENT

APPENDIX A GLOSSARY OF DATA QUALIFIER CODES

APPENDIX B DATA SUMMARY FORM(S)

APPENDIX C CHAIN OF CUSTODY RECORD(S)

APPENDIX D LABORATORY CASE NARRATIVE(S)

DCN: 37845_ MC02P4. IM2

Appendix A

Glossary of Data Qualifier Codes

GLOSSARY OF DATA QUALIFIER CODES (INORGANIC)

CODES RELATED TO IDENTIFICATION

(confidence concerning presence or absence of analytes):

- U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.
- (NO CODE) = Confirmed identification.
- B = Not detected substantially above the level reported in laboratory or field blanks.
- R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

CODES RELATED TO QUANTITATION

(can be used for both positive results and sample quantitation limits):

- J = Analyte Present. Reported value may not be accurate or precise.
- K = Analyte present. Reported value may be biased high. Actual value is expected to be lower.
- L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.
- UJ = Not detected, quantitation limit may be inaccurate or imprecise.
- UL = Not detected, quantitation limit is probably higher.

OTHER CODE

- Q = No analytical result.

Appendix B

Data Summary Forms (DSFs)

DATA SUMMARY FORM: INORGANIC

Case #: 37845

SDG : MC02P4

Number of Soil Samples : 16

Site :

FORMER MOHR ORCHARD

Number of Water Samples : 0

Lab. :

SVL

Sample Number :	MC02P4	MC02P5	MC02P6	MC02P7	MC02P8						
Sampling Location :	FMO08-SS-A40	FMO08-SS-AF22	FMO08-SS-AJ53	FMO08-SS-AM47	FMO08-SS-C45						
Matrix :	Soil	Soil	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	9/15/2008	9/18/2008	9/8/2008	9/5/2008	9/12/2008						
Time Sampled :	17:40	09:55	12:35	10:50	08:50						
%Solids :	100.0	100.0	100.0	100.0	100.0						
Dilution Factor :	1.0	1.0	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	123		29.7		31.5		31.0		33.6	
*LEAD	1	1820		132		229		161		251	

Sample Number :	MC02P9	MC02Q0	MC02Q1	MC02Q2	MC02Q3						
Sampling Location :	FMO08-SS-H49	FMO08-SS-J46	FMO08-SS-L45	FMO08-SS-M38	FMO08-SS-N34						
Matrix :	Soil	Soil	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	9/12/2008	9/15/2008	9/15/2008	9/11/2008	9/16/2008						
Time Sampled :	10:45	11:20	12:35	16:20	14:37						
%Solids :	100.0	100.0	100.0	100.0	100.0						
Dilution Factor :	1.0	1.0	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	117		31.6		36.5		30.6		7.6	
*LEAD	1	1400		142		345		250		29.8	

Sample Number :	MC02Q4	MC02Q5	MC02Q6	MC02Q7	MC02Q8						
Sampling Location :	FMO08-SS-O13	FMO08-SS-P10	FMO08-SS-P13	FMO08-SS-Q20	FMO08-SS-R07						
Matrix :	Soil	Soil	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	9/16/2008	9/10/2008	9/18/2008	9/12/2008	9/10/2008						
Time Sampled :	15:30	08:55	09:15	17:35	11:25						
%Solids :	100.0	100.0	100.0	100.0	100.0						
Dilution Factor :	1.0	1.0	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	36.4		36.2		36.0		37.4		34.1	
*LEAD	1	265		190		241		197		210	

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample quantitation limits: (CRQL * Dilution Factor) / (%Solids/ 100)

Revised 09/99

DATA SUMMARY FORM: INORGANIC

Case #: 37845

SDG : MC02P4

Site :

FORMER MOHR ORCHARD

Lab. :

SVL

Sample Number :		MC02Q9							
Sampling Location :		FMO08-SS-T03							
Matrix :		Soil							
Units :		mg/Kg							
Date Sampled :		9/18/2008							
Time Sampled :		12:30							
%Solids :		100.0							
Dilution Factor :		1.0							
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	12.0							
*LEAD	1	78.2							

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample quantitation limits: $(CRQL * Dilution Factor) / (\%Solids / 100)$

Revised 09/99

Appendix C

Chain-of-Custody Records



**USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record**

Case No: 37845

R

DAS No:

Region: 3	Date Shipped: 9/25/2008	Chain of Custody Record	
Project Code: CT4362	Carrier Name: FedEX	Relinquished By	Sampler Signature:
Account Code:	Airbill: 857499852978	(Date / Time)	Received By
CERCLIS ID: None	Shipped to: SVL Analytical Inc	1	
Spill ID: ALG	One Government Gulch	2.	
Site Name/State: Former Mohr Orchard Assessment/PA	Kellogg ID 83837	3.	
Project Leader: Erik Armistead	(208) 784-1258	4.	
Action: Preliminary Assessment			
Sampling Co: Tetra Tech EM Inc.			

INORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	QC Type
MC02P1	Potable Well/ Erik Armistead	L/G	Pb/As - Wa (14)	951 (HNO3) (1)	FMO08-PW-BE6901	S: 9/25/2008 12:49		
MC02P2	Potable Well/ Erik Armistead	L/G	Pb/As - Wa (14)	952 (HNO3) (1)	FMO08-PW-BK5401	S: 9/19/2008 8:52		
MC02P3	Potable Well/ Erik Armistead	L/G	Pb/As - Wa (14)	953 (HNO3) (1)	FMO08-PW-AD8101	S: 9/23/2008 10:08		
MC02P4	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	954 (Ice Only) (1)	FMO08-SS-A40	S: 9/15/2008 17:40		
MC02P5	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	955 (Ice Only) (1)	FMO08-SS-AF22	S: 9/18/2008 9:55		
MC02P6	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	956 (Ice Only) (1)	FMO08-SS-AJ53	S: 9/8/2008 12:35		
MC02P7	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	957 (Ice Only) (1)	FMO08-SS-AM47	S: 9/5/2008 10:50		
MC02P8	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	958 (Ice Only) (1)	FMO08-SS-C45	S: 9/12/2008 8:50		
MC02P9	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	959 (Ice Only) (1)	FMO08-SS-H49	S: 9/12/2008 10:45		
MC02Q0	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	960 (Ice Only) (1)	FMO08-SS-J46	S: 9/15/2008 11:20		
MC02Q1	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	961 (Ice Only) (1)	FMO08-SS-L45	S: 9/15/2008 12:35		

Shipment for Case Complete? N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: Pb/As - So = Lead and Arsenic - Soil, Pb/As - Wa = Lead and Arsenic - Water	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____

TR Number: 3-375524367-092508-0001





**USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record**

Case No: 37845

R

Region: 3	Date Shipped: 9/25/2008	Carrier Name: FedEx	Airbill: 857499852978	Shipped to: SVL Analytical Inc One Government Gulch Kellogg ID 83837 (208) 784-1258
Project Code: CT4362	Carrier Name: FedEx	Airbill: 857499852978		
CERCLIS ID: None				
Spill ID: ALG				
Site Name/State: Former Mohr Orchard Assessment/PA				
Project Leader: Erik Armistead				
Action: Preliminary Assessment				
Sampling Co: Terra Tech EM Inc.				

Chain of Custody Record		
Relinquished By	(Date / Time)	(Date / Time)
1.		
2.		
3.		
4.		

Sampler Signature: _____
Received By: _____
(Date / Time)

INORGANIC SAMPLE NO.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE NO.	QC Type
MC02Q2	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	962 (Ice Only) (1)	FMO08-SS-M38	S: 9/11/2008 16:20		--
MC02Q3	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	963 (Ice Only) (1)	FMO08-SS-N34	S: 9/16/2008 14:37		--
MC02Q4	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	964 (Ice Only) (1)	FMO08-SS-O13	S: 9/16/2008 15:30		--
MC02Q5	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	965 (Ice Only) (1)	FMO08-SS-P10	S: 9/10/2008 8:55		--
MC02Q6	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	966 (Ice Only) (1)	FMO08-SS-P13	S: 9/18/2008 9:15		--
MC02Q7	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	967 (Ice Only) (1)	FMO08-SS-Q20	S: 9/12/2008 17:35		--
MC02Q8	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	968 (Ice Only) (1)	FMO08-SS-R07	S: 9/10/2008 11:25		--
MC02Q9	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	969 (Ice Only) (1)	FMO08-SS-T03	S: 9/18/2008 12:30		--
MC02R0	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	970 (Ice Only) (1)	FMO08-SS-T40	S: 9/22/2008 11:22		--
MC02R1	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	971 (Ice Only) (1)	FMO08-SS-U11	S: 9/10/2008 14:30		--
MC02R2	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	972 (Ice Only) (1)	FMO08-SS-U48	S: 9/23/2008 15:56		--

Shipment for Case Complete? N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: Pb/As - So = Lead and Arsenic - Soil, Pb/As - Wa = Lead and Arsenic - Water	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____

TR Number: 3-375524367-092508-0001

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

REGION COPY

9-8-08 JTS

ASQAB USE ONLY	
RAS#	CT4362
DAS#	
NSF#	
Analytical TAT 14	

U.S. EPA Region III Analytical Request Form

37845

Date: 9/4/2008		Site Activity: Removal Site Evaluation	
Site Name: Former Mohr Orchard Arsenic <i>Arsenic Assessment</i>		Street Address: Orefield, Pennsylvania	
City: North Whitehall Township		Longitude: 75.60078° W	
State: PA		Latitude: 40.64622° N	
Program: Superfund		Acct. #: 2008T03 N 302DC6CA3LGRS00	
Site ID:		CERCLIS #: <i>NONE</i>	
Spill ID: A3LG		Operable Unit:	
Site Specific QA Plan Submitted: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		Title: Draft Former Mohr Orchard SAP	
EPA Project Leader: RICHARD FETZER		Phone#: 215-341-6307	
Request Preparer: JOSHUA COPE		Cell Phone #: 215-768-8114	
Site Leader: ERIK ARMISTEAD		Cell Phone #: 267 446 2837	
Contractor: Tetra Tech EM Inc		EPA CO/PO: Lorrie Murray/Karen Wodarczyk	
#Samples 85	Matrix: soil	Parameter: Arsenic and Lead	<i>SVL</i>
#Samples 10-50	Matrix: potable water	Parameter: Total Arsenic	
#Samples	Matrix:	Parameter:	
Ship Date From: 9/12/2008		Ship Date To: 10/3/2008	
Unvalidated Data Requested: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		If Yes, TAT Needed: <input type="checkbox"/> 24hrs <input type="checkbox"/> 48hrs <input type="checkbox"/> 72hrs <input type="checkbox"/> 7days <input checked="" type="checkbox"/> Other (Specify) 14 days	
Validated Data Package Due: <input type="checkbox"/> 14 days <input type="checkbox"/> 21 days <input checked="" type="checkbox"/> 30days <input type="checkbox"/> 42 days <input type="checkbox"/> Other (Specify)		14/16	
Electronic Data Deliverables Required: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes (EDDs will be provided in Region 3 EDD Format)			
Special Instructions: Potable waters will ship weekly 9/12, 9/19, and 9/26. Soils will be shipped at end of the sampling event between 9/26 and 10/3. Detection limits are Lead @ 1 mg/kg and Arsenic @ 1 mg/kg for soil and Arsenic @ 1 ug/L for potable water.			

Appendix D

Laboratory Case Narrative

USEPA-CLP

COVER PAGE

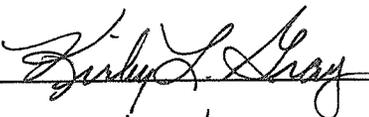
Lab Name: SVL Analytical, Inc. Contract: EP-W-06-056
 Lab Code: SVL Case No: 37845 NRAS No.: _____ SDG No: MC02P4
 SOW No.: ILM05.4

EPA Sample No.	Lab Sample ID
<u>MC02P4</u>	<u>MC02P4</u>
<u>MC02P4D</u>	<u>MC02P4D</u>
<u>MC02P4S</u>	<u>MC02P4S</u>
<u>MC02P5</u>	<u>MC02P5</u>
<u>MC02P6</u>	<u>MC02P6</u>
<u>MC02P7</u>	<u>MC02P7</u>
<u>MC02P8</u>	<u>MC02P8</u>
<u>MC02P9</u>	<u>MC02P9</u>
<u>MC02Q0</u>	<u>MC02Q0</u>
<u>MC02Q1</u>	<u>MC02Q1</u>
<u>MC02Q2</u>	<u>MC02Q2</u>
<u>MC02Q3</u>	<u>MC02Q3</u>
<u>MC02Q4</u>	<u>MC02Q4</u>
<u>MC02Q5</u>	<u>MC02Q5</u>
<u>MC02Q6</u>	<u>MC02Q6</u>
<u>MC02Q7</u>	<u>MC02Q7</u>
<u>MC02Q8</u>	<u>MC02Q8</u>
<u>MC02Q9</u>	<u>MC02Q9</u>

		ICP-AES	ICP-MS
Were ICP-AES and ICP-MS interelement corrections applied?	(Yes/No)	<u>YES</u>	<u>NO</u>
Were ICP-AES and ICP-MS background corrections applied?	(Yes/No)	<u>YES</u>	<u>NO</u>
If yes, were raw data generated before application of background corrections?	(Yes/No)	<u>NO</u>	<u>NO</u>

Comments: _____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature:  Name: KIRBY L. GRAY
 Date: 10/13/2008 Title: TECHNICAL DIRECTOR

SDG NARRATIVE

2

LAB NAME: SVL ANALYTICAL, INC.
LAB CODE: SVL
CONTRACT NO.: EP-W-06-056

CASE: 37845
SDG: MC02P4

SAMPLES RECEIVED FOR ICP-AES AND MERCURY ON SEPTEMBER 26, 2008.
SAMPLE TAGS WERE PRESENT.
COOLER TEMPERATURE INDICATOR BOTTLE ABSENT.

-MISSING TEMPERATURE BLANK-

ISSUE 1: A TEMPERATURE BLANK WAS NOT INCLUDED WITH THE SAMPLES RECEIVED ON 9/26/08. THE TEMPERATURE WAS DETERMINED TO BE 1.6 DEGREES CELSIUS BY IR GUN.

RESOLUTION 1: IN ACCORDANCE WITH PREVIOUS DIRECTION FROM REGION 3, THE LABORATORY WILL NOTE THE ISSUE, AND THE METHOD USED TO DETERMINE THE TEMPERATURE, IN THE CASE/SDG NARRATIVE AND PROCEED WITH THE ANALYSIS OF THE SAMPLES.

-INSUFFICIENT/INAPPROPRIATE DESIGNATION OF LABORATORY QC-

ISSUE 2: LABORATORY QC IS NOT DESIGNATED ON THE TR/COC; HOWEVER, THE SCHEDULING NOTIFICATION FORM LISTS THAT LABORATORY QC IS REQUIRED. THE LABORATORY WOULD LIKE TO SELECT SAMPLES MC02P4 (SDG MC02P4), MC02R0 (SDG MC02R0), AND MC02N0 (SDG MC02N0) AS LABORATORY QC.

RESOLUTION 2: IN ACCORDANCE WITH PREVIOUS DIRECTION FROM REGION 3, THE LABORATORY WILL SELECT A SAMPLE FOR LABORATORY QC AS LONG AS THE SAMPLE IS NOT A PE, BLANK, OR RINSATE SAMPLE. THE LABORATORY WILL NOTE THE ISSUE IN THE CASE/SDG NARRATIVE, NOTIFY THE SMO COORDINATOR OF THE SAMPLE SELECTED FOR LABORATORY QC, AND PROCEED WITH THE ANALYSIS OF THE SAMPLES. SMO WILL NOTE THAT THE LABORATORY SELECTED SAMPLES MC02P4 (SDG MC02P4), MC02R0 (SDG MC02R0), AND MC02N0 (SDG MC02N0) AS LABORATORY QC.

ISSUE 3: THERE IS INSUFFICIENT VOLUME TO PERFORM % SOLIDS FOR SDGS MC02P4 AND -R0. THE SOIL SAMPLES APPEAR TO BE DRIED AND PULVERIZED AND WERE RECEIVED IN XRF CUPS. THE LABORATORY WOULD LIKE TO KNOW IF THEY CAN REPORT PERCENT SOLIDS AS 100%?

RESOLUTION 3: PER REGION 3, THE LABORATORY CAN REPORT THE % SOLIDS AS 100% FOR THE SAMPLES IN SDGS MC02P4 AND -R0. THE LABORATORY WILL NOTE THE ISSUE IN THE CASE/SDG NARRATIVE AND PROCEED WITH THE ANALYSIS OF THE SAMPLES.

ICP-AES EXAMPLE CALCULATION: MC02P4 LEAD

$$\text{CONCENTRATION (DRY WT.) (MG/KG)} = \frac{C \times V}{W \times S} \times \text{DF}$$

- C = CONCENTRATION (mg/L)
- V = FINAL SAMPLE VOLUME IN LITERS (L)
- W = WET SAMPLE WEIGHT (kg)
- S = % SOLIDS/100
- DF = DILUTION FACTOR

$$\frac{18.2013 \text{ mg/L} \times 0.1 \text{ L}}{0.001 \text{ kg} \times 100.0/100} \times 1 = 1820 \text{ mg/kg}$$

Lab Name SVL ANALYTICAL					Page 1 of 3	
Received By (Print Name) C. FLORES					Log-in Date 9/26/2008	
Received By (Signature) <i>C. Flores / CPS</i>						
Case Number 37845		Sample Delivery Group No. MC02N0/MC02P4/MC02R0			NRAS Number N/A	
Remarks:				Corresponding		Remarks: Condition of Sample Shipment, etc.
		EPA Sample #	Aqueous Sample pH	Sample Tag #	Assigned Lab #	
1. Custody Seal(s)	<u>Present</u> /Absent* <u>Intact</u> /Broken	MC02N0	<2	940	MC02N0	INTACT
2. Custody Seal Nos.	N/A	MC02N1	<2	941	MC02N1	INTACT
3. <u>Traffic Reports/Chain of Custody Records</u> or Packing Lists	<u>Present</u> /Absent*	MC02N2	<2	942	MC02N2	INTACT
4. Airbill	<u>Airbill/Sticker Present</u> /Absent*	MC02N3	<2	943	MC02N3	INTACT
5. Airbill No.	857499852978	MC02N4	<2	944	MC02N4	INTACT
6. Sample Tags	<u>Present</u> /Absent*	MC02N5	<2	945	MC02N5	INTACT
Sample Tag Numbers	<u>Listed</u> /Not Listed on Traffic Report/ <u>Chain of Custody Record</u>	MC02N6	<2	946	MC02N6	INTACT
7. Sample Condition	<u>Intact</u> /Broken*/Leaking	MC02N7	<2	947	MC02N7	INTACT
8. Cooler Temperature Indicator Bottle	<u>Present</u> /Absent*	MC02N8	<2	948	MC02N8	INTACT
9. Cooler Temperature	1.6°C	MC02N9	<2	949	MC02N9	INTACT
10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree?	<u>Yes</u> /No*	MC02P0	<2	950	MC02P0	INTACT
11. Date Received at Lab	9/26/2008	MC02P1	<2	951	MC02P1	INTACT
12. Time Received	13:10	MC02P2	<2	952	MC02P2	INTACT
Sample Transfer		MC02P3	<2	953	MC02P3	INTACT
Fraction N/A	Fraction N/A	MC02P4	N/A	954	MC02P4	INTACT
Area # RecCLP Clr 2B/	Area # N/A	MC02P5	N/A	955	MC02P5	INTACT
By N/A	By N/A	MC02P6	N/A	956	MC02P6	INTACT
On N/A	On N/A	MC02P7	N/A	957	MC02P7	INTACT

* Contact SMO and attach record of resolution

Reviewed By <i>[Signature]</i>	N/A	Logbook No.	N/A
Date 09/29/2008	N/A	Logbook Page No.	N/A

Lab Name SVL ANALYTICAL				Page 2 of 3		
Received By (Print Name) C. FLORES				Log-in Date 9/26/2008		
Received By (Signature) <i>C. Flores / CFS</i>						
Case Number 37845		Sample Delivery Group No. MC02N0/MC02P4/MC02R0		NRAS Number N/A		
Remarks:		EPA Sample #	Aqueous Sample pH	Corresponding Sample Tag #	Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	<u>Present</u> /Absent* <u>Intact</u> /Broken	MC02P8	N/A	958	MC02P8	INTACT
2. Custody Seal Nos.	N/A	MC02P9	N/A	959	MC02P9	INTACT
3. <u>Traffic Reports/Chain of Custody Records</u> or Packing Lists	<u>Present</u> /Absent*	MC02Q0	N/A	960	MC02Q0	INTACT
4. Airbill	<u>Airbill</u> /Sticker <u>Present</u> /Absent*	MC02Q1	N/A	961	MC02Q1	INTACT
5. Airbill No.	857499852978	MC02Q2	N/A	962	MC02Q2	INTACT
6. Sample Tags	<u>Present</u> /Absent*	MC02Q3	N/A	963	MC02Q3	INTACT
Sample Tag Numbers	<u>Listed</u> /Not Listed on Traffic Report/ <u>Chain of Custody Record</u>	MC02Q4	N/A	964	MC02Q4	INTACT
7. Sample Condition	<u>Intact</u> /Broken*/Leaking	MC02Q5	N/A	965	MC02Q5	INTACT
8. Cooler Temperature Indicator Bottle	<u>Present</u> /Absent*	MC02Q6	N/A	966	MC02Q6	INTACT
9. Cooler Temperature	1.6°C	MC02Q7	N/A	967	MC02Q7	INTACT
10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree?	<u>Yes</u> /No*	MC02Q8	N/A	968	MC02Q8	INTACT
11. Date Received at Lab	9/26/2008	MC02Q9	N/A	969	MC02Q9	INTACT
12. Time Received	13:10	MC02R0	N/A	970	MC02R0	INTACT
Sample Transfer		MC02R1	N/A	971	MC02R1	INTACT
Fraction N/A	Fraction N/A	MC02R2	N/A	972	MC02R2	INTACT
Area # RecCLP Clr 2B/	Area # N/A	MC02R3	N/A	973	MC02R3	INTACT
By N/A	By N/A	MC02R4	N/A	974	MC02R4	INTACT
On N/A	On N/A	MC02R5	N/A	975	MC02R5	INTACT

Contact SMO and attach record of resolution

Reviewed By <i>[Signature]</i>	N/A	Logbook No.	N/A
Date <i>09/29/2008</i>	N/A	Logbook Page No.	N/A

Lab Name SVL ANALYTICAL				Page 3 of 3	
Received By (Print Name) C. FLORES				Log-in Date 9/26/2008	
Received By (Signature) <i>C. Flores</i>					
Case Number 37845		Sample Delivery Group No. MC02N0/MC02P4/MC02R0			NRAS Number N/A
Remarks:	EPA Sample #	Aqueous Sample pH	Corresponding		Remarks: Condition of Sample Shipment, etc.
			Sample Tag #	Assigned Lab #	
1. Custody Seal(s) Present/Absent* Intact/Broken	MC02R6	N/A	976	MC02R6	INTACT
2. Custody Seal Nos. N/A	MC02R7	N/A	977	MC02R7	INTACT
3. Traffic Reports/Chain of Custody Records or Packing Lists Present/Absent*	N/A	N/A	N/A	N/A	N/A
4. Airbill Airbill/Sticker Present/Absent*	N/A	N/A	N/A	N/A	N/A
5. Airbill No. 857499852978	N/A	N/A	N/A	N/A	N/A
6. Sample Tags Present/Absent*	N/A	N/A	N/A	N/A	N/A
Sample Tag Numbers Listed/Not Listed on Traffic Report/Chain of Custody Record	N/A	N/A	N/A	N/A	N/A
7. Sample Condition Intact/Broken*/Leaking	N/A	N/A	N/A	N/A	N/A
8. Cooler Temperature Indicator Bottle Present/Absent*	N/A	N/A	N/A	N/A	N/A
9. Cooler Temperature 1.6°C	N/A	N/A	N/A	N/A	N/A
10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree? Yes/No*	N/A	N/A	N/A	N/A	N/A
11. Date Received at Lab 9/26/2008	N/A	N/A	N/A	N/A	N/A
12. Time Received 13:10	N/A	N/A	N/A	N/A	N/A
Sample Transfer		N/A	N/A	N/A	N/A
Fraction N/A	Fraction N/A	N/A	N/A	N/A	N/A
Area # RecCLP Clr 2B/	Area # N/A	N/A	N/A	N/A	N/A
By N/A	By N/A	N/A	N/A	N/A	N/A
On N/A	On N/A	N/A	N/A	N/A	N/A

* Contact SMO and attach record of resolution.

Reviewed By <i>Debra Gray</i>	N/A	Logbook No. N/A
Date 09/29/2008	N/A	Logbook Page No. N/A



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
ENVIRONMENTAL SCIENCE CENTER
701 MAPES ROAD
FORT MEADE, MARYLAND 20755-5350

DATE : October 22, 2008

SUBJECT: Region III Data QA Review

FROM : Khin-Cho Thaung *KCT*
Region III ESAT RPO (3ES20)

TO : Richard Fetzer
Regional Project Manager (3HS32)

Attached is the inorganic (Arsenic Assessment) data validation report for the Former Mohr Orchard site (Case #: 37845, SDG#: MC02R0) completed by the Region III Environmental Services Assistance Team (ESAT) contractor under the direction of Region III ESD.

If you have any questions regarding this review, please call me at (410) 305-2743.

Attachments

cc: Joshua Cope (TTEMI)

TO File #: 0014

TDF#: 1046

OFFICE OF ANALYTICAL SERVICES AND QUALITY ASSURANCE

Lockheed Martin Enterprise Solutions & Services
ESAT Region 3
US EPA Environmental Science Center
701 Mapes Road Ft. Meade, MD 20755-5350
Telephone 410-305-3037 Facsimile 410-305-3597

LOCKHEED MARTIN
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Date: October 21, 2008

Subject: Inorganic Data Validation (IM2 Level)
Case: 37845
SDG : MC02R0
Site : Former Mohr Orchard Arsenic Assessment

From: Kurt Roby *KR*
Inorganic Data Reviewer

M Mahboobeh Mecanic *M*
Senior Oversight Chemist

To: Colleen Walling
ESAT Region 3 Project Officer

OVERVIEW

Case 37845, Sample Delivery Groups (SDG) MC02R0, consisted of eight (8) soil samples analyzed for arsenic (As) and lead (Pb) by ICP-MS. Samples were analyzed by SVL Analytical, Inc. (SVL) according to the Contract Laboratory Program (CLP) Statement of Work (SOW) ILM05.4 through the Routine Analytical Services (RAS) program.

SUMMARY

Data were validated according to Region III Modifications to the National Functional Guidelines for Inorganic Data Review, Level IM2. No problems were detected during the validation of this data set.

NOTES

The samples shipped under Case 37845 were received pulverized, dried in XRF cups and with insufficient quantity to perform percent solids determination. Per Region III, the laboratory was directed to report all samples as 100% solid.

Data for Case 37845, SDG MC02R0, were reviewed in accordance with the National Functional Guidelines for Evaluating Inorganic Analyses with Modifications for use within Region III.

ATTACHMENTS

Appendix A Glossary of Data Qualifier Codes
Appendix B Data Summary Form(s)
Appendix C Chain of Custody Records
Appendix D Laboratory Case Narrative

DCN: 37845_MC02R0

Appendix A

Glossary of Data Qualifier Codes

GLOSSARY OF DATA QUALIFIER CODES (INORGANIC)

CODES RELATED TO IDENTIFICATION

(confidence concerning presence or absence of analytes):

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

B = Not detected substantially above the level reported in laboratory or field blanks.

R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

CODES RELATED TO QUANTITATION

(can be used for both positive results and sample quantitation limits):

J = Analyte Present. Reported value may not be accurate or precise.

K = Analyte present. Reported value may be biased high. Actual value is expected to be lower.

L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ = Not detected, quantitation limit may be inaccurate or imprecise.

UL = Not detected, quantitation limit is probably higher.

OTHER CODES

Q = No analytical result.

Appendix B

Data Summary Forms

DATA SUMMARY FORM: INORGANIC

Case #: 37845
 Site :
 Lab. :

SDG : MC02R0
 FORMER MOHR ORCHARD ARSENIC ASSESSMENT
 SVL

Number of Soil Samples : 8
 Number of Water Samples : 0

Sample Number :	MC02R0	MC02R1	MC02R2	MC02R3	MC02R4						
Sampling Location :	FMO08-SS-T40	FMO08-SS-U11	FMO08-SS-U48	FMO08-SS-V08	FMO08-SS-V14						
Matrix :	Soil	Soil	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	9/22/2008	9/10/2008	9/23/2008	9/9/2008	9/9/2008						
Time Sampled :	11:22	14:30	15:56	11:32	18:27						
%Solids :	100.0	100.0	100.0	100.0	100.0						
Dilution Factor :	1.0	1.0	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag		
ARSENIC	1	35.1		34.9		39.7		40.0		34.5	
*LEAD	1	266		179		242		286		165	

Sample Number :	MC02R5	MC02R6	MC02R7						
Sampling Location :	FMO08-SS-V41	FMO08-SS-W12	FMO08-SS-W35						
Matrix :	Soil	Soil	Soil						
Units :	mg/Kg	mg/Kg	mg/Kg						
Date Sampled :	9/22/2008	9/10/2008	9/22/2008						
Time Sampled :	17:55	14:45	10:00						
%Solids :	100.0	100.0	100.0						
Dilution Factor :	1.0	1.0	1.0						
ANALYTE	CRQL	Result	Flag	Result	Flag	Result	Flag	Result	Flag
ARSENIC	1	38.2		31.8		33.3			
*LEAD	1	257		244		231			

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample quantitation limits: $(CRQL * Dilution Factor) / (\%Solids / 100)$

Revised 09/99

Appendix C

Chain of Custody Records



**USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record**

Case No: 37845

DAS No:

R

Region: 3	Date Shipped: 9/25/2008	Chain of Custody Record		Sampler Signature:
Project Code: CT4362	Carrier Name: FedEx	Relinquished By	(Date / Time)	Received By
Account Code:	Airbill: 857499852978	1		(Date / Time)
CERCLIS ID: None	Shipped to: SVL Analytical Inc	2.		
Spill ID: ALG	Former Mohr Orchard Assessment/PA	3.		
Site Name/State: Erik Armistead	Preliminary Assessment	4.		
Project Leader: Erik Armistead	Tetra Tech EM Inc.			
Action: Preliminary Assessment				
Sampling Co: Tetra Tech EM Inc.				

INORGANIC SAMPLE NO.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	QC Type
MC02Q2	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	962 (Ice Only) (1)	FMC08-SS-M38	S: 9/11/2008 16:20		--
MC02Q3	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	963 (Ice Only) (1)	FMC08-SS-N34	S: 9/16/2008 14:37		--
MC02Q4	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	964 (Ice Only) (1)	FMC08-SS-O13	S: 9/16/2008 15:30		--
MC02Q5	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	965 (Ice Only) (1)	FMC08-SS-P10	S: 9/10/2008 8:55		--
MC02Q6	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	966 (Ice Only) (1)	FMC08-SS-P13	S: 9/18/2008 9:15		--
MC02Q7	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	967 (Ice Only) (1)	FMC08-SS-Q20	S: 9/12/2008 17:35		--
MC02Q8	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	968 (Ice Only) (1)	FMC08-SS-R07	S: 9/10/2008 11:25		--
MC02Q9	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	969 (Ice Only) (1)	FMC08-SS-T03	S: 9/18/2008 12:30		--
MC02R0	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	970 (Ice Only) (1)	FMC08-SS-T40	S: 9/22/2008 11:22		--
MC02R1	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	971 (Ice Only) (1)	FMC08-SS-U11	S: 9/10/2008 14:30		--
MC02R2	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	972 (Ice Only) (1)	FMC08-SS-U48	S: 9/23/2008 15:56		--

Shipment for Case Complete? N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: Pb/As - So = Lead and Arsenic - Soil, Pb/As - Wa = Lead and Arsenic - Water	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____

TR Number: 3-375524367-092508-0001

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

REGION 9000



**USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record**

Case No: 37845

DAS No:

R

Region: 3	Date Shipped: 9/25/2008	Carrier Name: FedEx	STATION LOCATION	ORGANIC SAMPLE No.	QC Type
Project Code: CT4362	Carrier Name: FedEx	Airbill: 857499862978			
Account Code:	Airbill: 857499862978	Shipped to: SVL Analytical Inc One Government Gulch Kellogg ID 83837 (208) 784-1258			
CERCLIS ID: None					
Spill ID: ALG					
Site Name/State: Former Mohr Orchard Assessment/PA					
Project Leader: Erik Armistead					
Action: Preliminary Assessment					
Sampling Co: Tetra Tech EM Inc.					

INORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	PRESERVATIVE/ Bottles	TAG No./	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	QC Type
MC02R3	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	973 (Ice Only) (1)		FMO08-SS-V08	S: 9/9/2008 11:32		--
MC02R4	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	974 (Ice Only) (1)		FMO08-SS-V14	S: 9/9/2008 18:27		--
MC02R5	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	975 (Ice Only) (1)		FMO08-SS-V41	S: 9/22/2008 17:55		--
MC02R6	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	976 (Ice Only) (1)		FMO08-SS-W12	S: 9/10/2008 14:45		--
MC02R7	Soil (0"-12")/ Erik Armistead	M/C	Pb/As - So (14)	977 (Ice Only) (1)		FMO08-SS-W85	S: 9/22/2008 10:00		--

Shipment for Case Complete? N	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: Pb/As - So = Lead and Arsenic - Soil, Pb/As - Vva = Lead and Arsenic - Water	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____

TR Number: 3-375524367-092508-0001

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, Attn: Heather Bauer, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4602

REGION COPY

9-8-08 975
 ASQAB USE ONLY
 RASH# CT4362 Analytical TAT
 DASH# 14
 NSF#

U.S. EPA Region III Analytical Request Form

37845

Date: 9/4/2008		Site Activity: Removal Site Evaluation	
Site Name: Former Mohr Orchard-Arsenic <i>Arsenic Assessment</i>		Street Address: Orefield, Pennsylvania	
City: North Whitehall Township	State: PA	Latitude: 40.64622° N	Longitude: 75.60078° W
Program: Superfund	Acct. #: 2008T03 N 302DC6CA3LGRS00	CERCLIS #: <i>NONE</i>	
Site ID:	Spill ID: A3LG	Operable Unit:	
Site Specific QA Plan Submitted: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		Title: Draft Former Mohr Orchard SAP	Date Approved:
EPA Project Leader: RICHARD FETZER	Phone#: 215-341-6307	Cell Phone #:	E-mail: Fetzter.rich@epa.gov
Request Preparer: JOSHUA COPE	Phone#: 610-364-2130	Cell Phone #: 215-768-8114	E-mail: Joshua.cope@ttemi.com
Site Leader: ERIK ARMISTEAD	Phone#: 610-364-2151	Cell Phone #: 267 446 2837	E-mail: Erik.armistead@ttemi.com
Contractor: Tetra Tech EM Inc			
#Samples 85	Matrix: soil	Parameter: Arsenic and Lead	Method: <i>ILM05.4 ICPAES</i> <i>290483</i>
#Samples 10-50	Matrix: potable water	Parameter: Total Arsenic	Method: <i>ILM05.4 ICPMS</i> <i>29044</i>
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
Ship Date From: 9/12/2008	Ship Date To: 10/3/2008	Org. Validation Level	Inorg. Validation Level IM2
Unvalidated Data Requested: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		If Yes, TAT Needed: <input type="checkbox"/> 24hrs <input type="checkbox"/> 48hrs <input type="checkbox"/> 72hrs <input type="checkbox"/> 7days <input checked="" type="checkbox"/> Other (Specify) 14 days	<i>14 Day TAT</i>
Validated Data Package Due: <input type="checkbox"/> 14 days <input type="checkbox"/> 21 days <input checked="" type="checkbox"/> 30days <input type="checkbox"/> 42 days <input type="checkbox"/> Other (Specify)		<i>14/16</i>	
Electronic Data Deliverables Required: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes (EDDs will be provided in Region 3 EDD Format)			
Special Instructions: Potable waters will ship weekly 9/12, 9/19, and 9/26. Soils will be shipped at end of the sampling event between 9/26 and 10/3. Detection limits are Lead @ 1 mg/kg and Arsenic @ 1 mg/kg for soil and Arsenic @ 1 ug/L for potable water.			

Appendix D

Laboratory Case Narrative

USEPA-CLP

COVER PAGE

Lab Name: SVL Analytical, Inc. Contract: EP-W-06-056
 Lab Code: SVL Case No: 37845 NRAS No.: _____ SDG No: MC02R0
 SOW No.: ILM05.4

EPA Sample No.	Lab Sample ID
<u>MC02R0</u>	<u>MC02R0</u>
<u>MC02R0D</u>	<u>MC02R0D</u>
<u>MC02R0S</u>	<u>MC02R0S</u>
<u>MC02R1</u>	<u>MC02R1</u>
<u>MC02R2</u>	<u>MC02R2</u>
<u>MC02R3</u>	<u>MC02R3</u>
<u>MC02R4</u>	<u>MC02R4</u>
<u>MC02R5</u>	<u>MC02R5</u>
<u>MC02R6</u>	<u>MC02R6</u>
<u>MC02R7</u>	<u>MC02R7</u>

		ICP-AES	ICP-MS
Were ICP-AES and ICP-MS interelement corrections applied?	(Yes/No)	<u>YES</u>	<u>NO</u>
Were ICP-AES and ICP-MS background corrections applied?	(Yes/No)	<u>YES</u>	<u>NO</u>
If yes, were raw data generated before application of background corrections?	(Yes/No)	<u>NO</u>	<u>NO</u>

Comments: _____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: *Kirby L. Gray* Name: KIRBY L. GRAY
 Date: 10/07/2008 Title: TECHNICAL DIRECTOR

SDG NARRATIVE

LAB NAME: SVL ANALYTICAL, INC.
LAB CODE: SVL
CONTRACT NO.: EP-W-06-056

CASE: 37845
SDG: MC02R0

SAMPLES RECEIVED FOR ICP-AES AND MERCURY ON SEPTEMBER 26, 2008.
SAMPLE TAGS WERE PRESENT.
COOLER TEMPERATURE INDICATOR BOTTLE ABSENT.

-MISSING TEMPERATURE BLANK-

ISSUE 1: A TEMPERATURE BLANK WAS NOT INCLUDED WITH THE SAMPLES RECEIVED ON 9/26/08. THE TEMPERATURE WAS DETERMINED TO BE 1.6 DEGREES CELSIUS BY IR GUN.

RESOLUTION 1: IN ACCORDANCE WITH PREVIOUS DIRECTION FROM REGION 3, THE LABORATORY WILL NOTE THE ISSUE, AND THE METHOD USED TO DETERMINE THE TEMPERATURE, IN THE CASE/SDG NARRATIVE AND PROCEED WITH THE ANALYSIS OF THE SAMPLES.

-INSUFFICIENT/INAPPROPRIATE DESIGNATION OF LABORATORY QC-

ISSUE 2: LABORATORY QC IS NOT DESIGNATED ON THE TR/COC; HOWEVER, THE SCHEDULING NOTIFICATION FORM LISTS THAT LABORATORY QC IS REQUIRED. THE LABORATORY WOULD LIKE TO SELECT SAMPLES MC02P4 (SDG MC02P4), MC02R0 (SDG MC02R0), AND MC02N0 (SDG MC02N0) AS LABORATORY QC.

RESOLUTION 2: IN ACCORDANCE WITH PREVIOUS DIRECTION FROM REGION 3, THE LABORATORY WILL SELECT A SAMPLE FOR LABORATORY QC AS LONG AS THE SAMPLE IS NOT A PE, BLANK, OR RINSATE SAMPLE. THE LABORATORY WILL NOTE THE ISSUE IN THE CASE/SDG NARRATIVE, NOTIFY THE SMO COORDINATOR OF THE SAMPLE SELECTED FOR LABORATORY QC, AND PROCEED WITH THE ANALYSIS OF THE SAMPLES. SMO WILL NOTE THAT THE LABORATORY SELECTED SAMPLES MC02P4 (SDG MC02P4), MC02R0 (SDG MC02R0), AND MC02N0 (SDG MC02N0) AS LABORATORY QC.

ISSUE 3: THERE IS INSUFFICIENT VOLUME TO PERFORM % SOLIDS FOR SDGS MC02P4 AND -R0. THE SOIL SAMPLES APPEAR TO BE DRIED AND PULVERIZED AND WERE RECEIVED IN XRF CUPS. THE LABORATORY WOULD LIKE TO KNOW IF THEY CAN REPORT PERCENT SOLIDS AS 100%?

RESOLUTION 3: PER REGION 3, THE LABORATORY CAN REPORT THE % SOLIDS AS 100% FOR THE SAMPLES IN SDGS MC02P4 AND -R0. THE LABORATORY WILL NOTE THE ISSUE IN THE CASE/SDG NARRATIVE AND PROCEED WITH THE ANALYSIS OF THE SAMPLES.

ICP-AES EXAMPLE CALCULATION: MC02R0 LEAD

$$\text{CONCENTRATION (DRY WT.) (MG/KG)} = \frac{C \times V}{W \times S} \times \text{DF}$$

C = CONCENTRATION (mg/L)

V = FINAL SAMPLE VOLUME IN LITERS (L)

W = WET SAMPLE WEIGHT (kg)

S = % SOLIDS/100

DF = DILUTION FACTOR

$$\frac{2.66016 \text{ mg/L} \times 0.1 \text{ L}}{0.001 \text{ kg} \times 100.0/100} \times 1 = 266 \text{ mg/kg}$$

Kirby Gray

From: Walsh, Colin [cwalsh20@fedcsc.com]
Sent: Monday, September 29, 2008 10:18
To: Kirby Gray
Cc: slizys.dan@epa.gov; Harris.Carroll@epamail.epa.gov; thaung.khin-cho@epa.gov; kwedar.john@epa.gov
Subject: Region 03 | Case 37845 | Lab SVL | Issue Multiple | FINAL

Kirby,

Summary Start

-Missing temperature blank-

Issue 1: A temperature blank was not included with the samples received on 9/26/08. The temperature was determined to be 1.6 degrees Celsius by IR Gun.

Resolution 1: In accordance with previous direction from Region 3, the laboratory will note the issue, and the method used to determine the temperature, in the Case/SDG Narrative and proceed with the analysis of the samples.

-Insufficient/inappropriate designation of laboratory QC-

Issue 2: Laboratory QC is not designated on the TR/COC; however, the Scheduling Notification Form lists that laboratory QC is required. The laboratory would like to select samples MC02P4 (SDG MC02P4), MC02R0 (SDG MC02R0), and MC02N0 (SDG MC02N0) as laboratory QC.

Resolution 2: In accordance with previous direction from Region 3, the laboratory will select a sample for laboratory QC as long as the sample is not a PE, blank, or rinsate sample. The laboratory will note the issue in the Case/SDG Narrative, notify the SMO coordinator of the sample selected for laboratory QC, and proceed with the analysis of the samples.

SMO will note that the laboratory selected samples MC02P4 (SDG MC02P4), MC02R0 (SDG MC02R0), and MC02N0 (SDG MC02N0) as laboratory QC.

Summary End

Please note that the analysis of the water samples should be ICP-MS (As, Pb) per the most recent Scheduling Notification Form received.

Please let me know if you have any further questions or problems.

Thanks,

Colin

Colin G. Walsh
Environmental Coordinator - Region 3
CSC

15000 Conference Center Drive, Chantilly, VA 20151
 Civil Division | (p) 703-818-4544 | (f) 703-818-4602 | cwalsh20@fedcsc.com | www.csc.com

From: Kirby Gray [mailto:kirbyg@svl.net]
Sent: Monday, September 29, 2008 12:57 PM
To: Walsh, Colin
Subject: RE: Case 37845 | TR/COCs

9/29/2008