



March 9, 2012

Mr. Jeffrey Lippert  
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**Subject:       Site Assessment Report  
                  Lovers Lane Lead Site  
                  Comstock Park, Kent County, Michigan  
                  Technical Direction Document No. TO-01-11-11-0028  
                  OTIE Contract No. EP-S5-10-10**

Dear Mr. Lippert:

OTIE is submitting the enclosed Site Assessment Report for the Lovers Lane Lead Site located in Comstock Park, Michigan. If you have any questions or comments about the report or need additional copies, please contact me at (312) 220-7000, extension 24 or Raghu Nagam at (312) 220-7005.

Sincerely,

Naren Babu  
Project Manager

Enclosure

cc:       Raghu Nagam, START Program Manager

**SITE ASSESSMENT REPORT  
LOVERS LANE LEAD SITE  
COMSTOCK PARK, KENT COUNTY, MICHIGAN**

Prepared for:

U.S. Environmental Protection Agency, Region 5  
Emergency Response Branch  
9311 Groh Road  
Grosse Ile, MI 48138-1697

TDD No.:	TO-01-11-11-0028
Date Prepared:	March 9, 2012
Contract No.:	EP-S5-10-10
Prepared by:	OTIE
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- A PHOTOGRAPHIC LOG
- B VALIDATED ANALYTICAL DATA PACKAGE

## 1. INTRODUCTION

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OTIE was tasked to conduct a Site Assessment (SA) and prepare a SA report in accordance with the requirements of U.S. Environmental Protection Agency (U.S. EPA) Technical Direction Document (TDD) No. TO-01-11-11-0028 under the Superfund Technical Assessment and Response Team (START) Contract No. EP-S5-10-10. The scope of this TDD was to conduct a SA at the Lovers Lane Lead Site (Site) in Comstock Park, Kent County, Michigan. START was tasked to prepare a site-specific Health and Safety Plan (HASP), Field Sampling and Analysis Plan (SAP), subcontract an analytical laboratory and an excavation contractor service, collect liquid and solid samples, evaluate analytical data, document on-site conditions with written logbook notes and still photographs, and prepare this SA report.

N. Babu was the START Project Manager and C. Schulz assisted with the sampling activities conducted on December 20<sup>th</sup>, 2011. Follow-up samplings were conducted on January 5<sup>th</sup>, 2012 and February 9<sup>th</sup>, 2012. This SA Report summarizes the Site background, discusses the assessment activities, provides a summary of the analytical data, and discusses potential site-related threats. The attachments for this report include a photographic log of the Site (Appendix A) and validated sample analytical results (Appendix B).

## 2. SITE BACKGROUND

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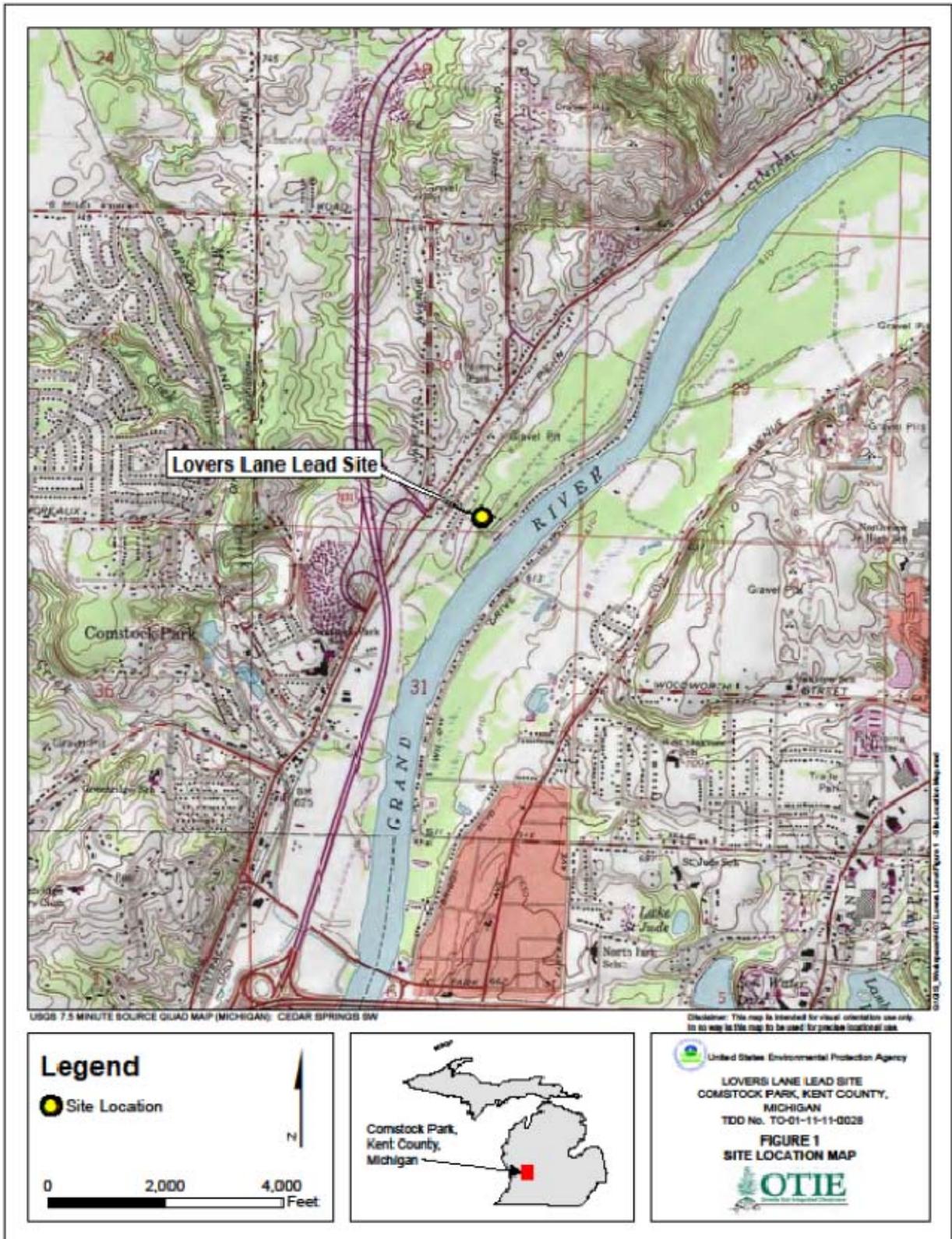
This section provides Site background information and a history of the Site.

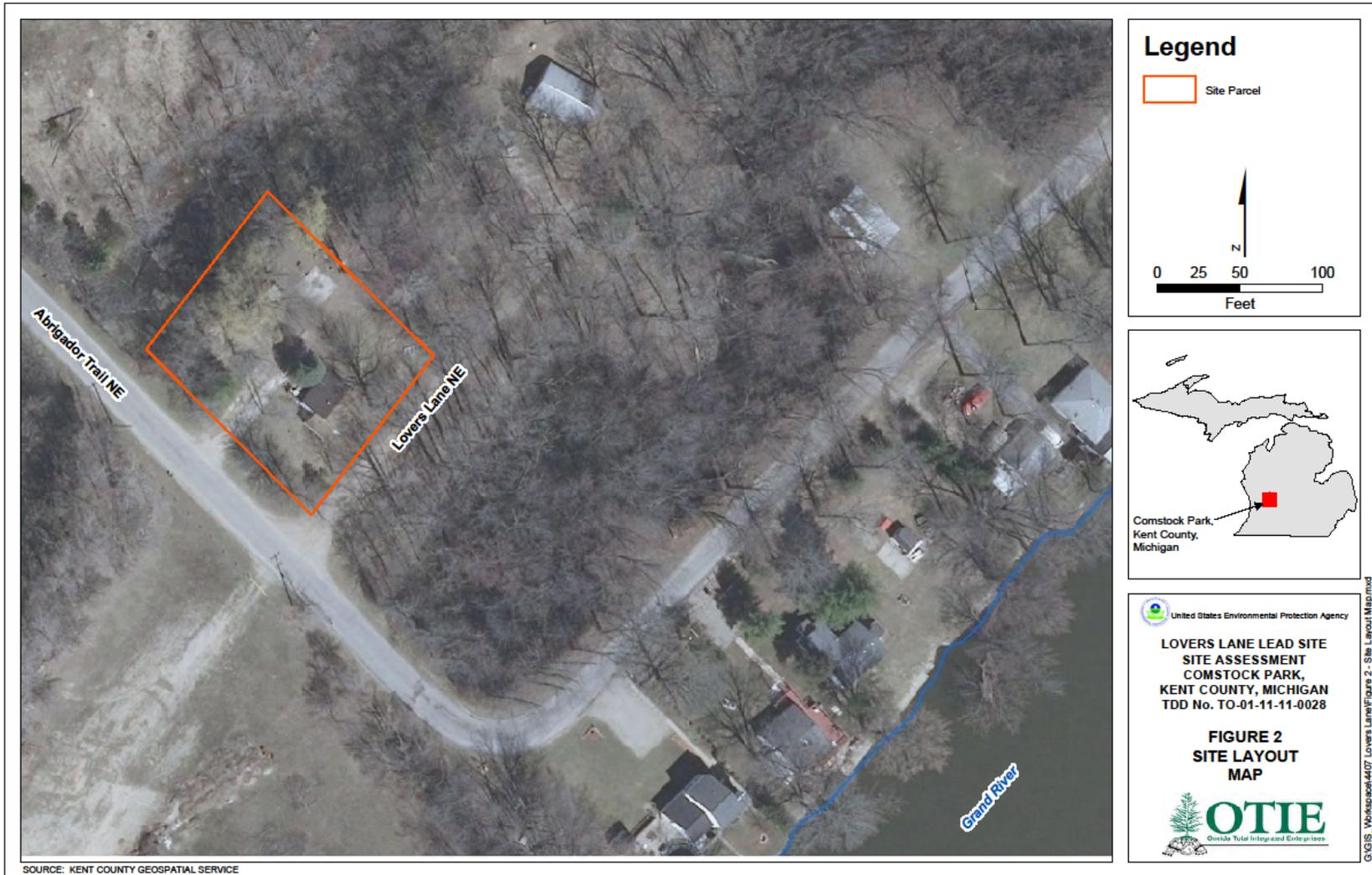
### 2.1 Site Description

The 0.39 acre property is located at 4407 Lovers Lane NE in Comstock Park, Michigan on the northern corner of Abrigador Trail NE and Lovers Lane NE (Figure 1– Site Location Map). The geographical coordinates for the residential house are 43.0438 degrees north latitude and -85.65526 degrees west longitude. The Site consists of a one story house with a possible crawl space and grass-covered lawn and trees surrounding the house (Figure 2 – Site Layout Map) and is located in a residential area surrounded by Abrigador Trail to the southwest, a wooded area to the northwest, a residential property with at least one building to the northeast, and Lovers Ln NE to the southeast. There is a creek that runs on the west side of the house and flows into Grand River, located approximately 0.1 miles from the site. The Site is notorious for flooding every spring from the Grand River. Residents near the Site often park their automobiles blocks away from their homes and use boats for transportation during flooding or wade through knee-deep water to reach their homes located near the Grand River.

### 2.2 Site History

During excavation activities while installing a water main in the right-of-way on Abrigador Trail, a large number of broken lead-acid automobile battery casings were encountered. Analysis of soil associated with these battery casings revealed Toxicity Characteristic Leaching Procedure (TCLP) lead concentrations as high as 53 milligrams per liter (mg/L). The federal TCLP limit for lead characteristic hazardous waste is 5 mg/L. Based on visual observations by the contractor and Michigan Department of Environmental Quality (MDEQ) staff, the battery casings disposal area likely extends further into the right-of-way of Abrigador Trail and perhaps into the Lovers Lane Lead Site property. The resident on the Site does not have access to municipal water and utilizes groundwater. The MDEQ has referred the Site to U.S. EPA Region 5 Superfund Division to conduct a SA and abate any environmental and human health threats resulting from the broken lead-acid battery casings found on the Site.





### 3. SITE ASSESSMENT ACTIVITIES

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Site Assessment and reconnaissance activities at the Lovers Lane Lead site, including sampling events, are discussed below. The SAP was developed for the SA prior to fieldwork. The SAP described the data quality objectives (DQO), sampling strategy, sampling locations, sampling methodology, and analytical procedures to be used during the SA. This section summarizes field investigation activities including Site reconnaissance (subsection 3.1) and sampling activities (subsection 3.2). Table 1 presents a summary of all samples collected and their associated locations. Photographic documentation is provided as Appendix A.

#### 3.1 Site Reconnaissance

On December 20<sup>th</sup>, 2011, U.S. EPA On-Scene Coordinators (OSCs) J. Lippert and B. Nightingale, OTIE START personnel N. Babu and C. Schulz mobilized to the Site and met with Young's Environmental crew. Young's Environmental was subcontracted by START to conduct exploration test pits to unearth battery casings and facilitate in START sampling. A Health and Safety meeting was held, including a discussion about the elements of HASP and SAP at the Site. Prior to conducting the Site reconnaissance, START calibrated MultiRAE® Plus five-gas air monitoring equipment. MultiRAE® includes a photoionization detector that measures/senses volatile organic compounds (VOCs), carbon monoxide (CO), hydrogen sulfide (H<sub>2</sub>S), lower explosive limit (LEL), and oxygen (O<sub>2</sub>). The Site reconnaissance activities were conducted outside the residential building in Level D personal protective equipment (PPE) in accordance with the approved site-specific HASP. Air monitoring was conducted in the breathing zone throughout the Site reconnaissance using the MultiRAE®.

One house and a storage shed are located on the Site property. A tree in front of the house (south corner) appeared to be dying. (Photo 1 in Appendix A). A drinking water well is located in the middle of the front yard. A basketball hoop and a fire-pit were observed northeast of the house. Broken lead-acid battery casings were observed in the yard at several locations. MultiRAE® readings were all below background levels for VOCs, CO, H<sub>2</sub>S, and LEL. O<sub>2</sub> was around 20.9% throughout the Site reconnaissance.

### 3.2 Sampling Activities

Following the Site reconnaissance activities on December 20<sup>th</sup>, 2011, subsurface soil sampling was conducted to determine the presence of contaminated soils and the need for a removal action. Locations where broken battery casings were observed were selected for conducting exploratory test-pits. Prior to mobilizing to the site, all utility locations were flagged on the Site. Young's Environmental excavated the test pits with an Excavator.

Four exploratory test pits were excavated on four sides of the house: Test Pit #1 on the west side, Test Pit #2 on the north side, Test Pit #3 on the south side and Test Pit #4 on the east side of the house. Plastic sheeting was used as a barrier on the ground surrounding each Test Pit and the excavated soil was stored on top of the plastic sheeting. Broken lead-acid battery casings were observed between two and four feet below ground surface (bgs) in Test Pit #1. Hard clay was observed around five feet bgs. START collected a composite soil sample, LL-SB-001, from the excavated soil for analysis of total and TCLP metals. Additionally, START collected an aliquot of soil for waste characterization analysis (LL-WS-01). At 5 to 6 feet bgs, water was observed in Test Pit #1. A sample of this water was collected and labeled as LL-WW-001 and preserved with nitric acid (HNO<sub>3</sub>) for total metals analysis.

Test Pit #2 was excavated on the north side the house. Broken battery casings were observed between one and five feet bgs. No water was encountered during excavation in the second Test Pit. A composite soil sample, LL-SB-002, was collected from the soil excavated from Test Pit #2. START also collected the second aliquot of soil for waste characterization analysis.

Test Pit #3 was excavated on the south side of the house. Battery casings were observed from just below the grass layer surface to three feet bgs. The third soil aliquot was collected to add to the composite waste characterization soil sample along with soil sample LL-SS-003.

Test Pit #4 was excavated on the east side of the house. This Test Pit was excavated to about six feet bgs and no battery casings were observed. Native sand was found throughout the Test Pit. No samples were collected from Test Pit #4.

A composite soil sample, LL-WS-001, was collected from the three aliquots obtained from test pits #1, #2 and #3 for the following waste characterization analyses: TCLP metals, TCLP VOCs, TCLP semi volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs) and pH determination. After the samples

were collected, all the Test Pits were back-filled and re-stored to the original condition. Young's environmental decontaminated the bucket on the excavator prior to their departure from the Site.

U.S. EPA OSC B. Nightingale and START N. Babu met with the home owner and gathered additional information regarding the site. Owner claimed that she buys bottled water and does not drink the well water. START purged the tap water in the kitchen for 15 minutes and collected water sample, LL-DW-001 for total metals analysis and preserved with HNO<sub>3</sub>.

START preserved all samples on ice, packaged them in a cooler, and hand-delivered the samples to Trimatrix Laboratories in Grand Rapids, Michigan on December 20<sup>th</sup>, 2011.

START were tasked to conduct additional sampling activities to further delineate the extent of contamination in surface soil and evaluate direct contact exposure criteria.

On January 5<sup>th</sup>, 2012, START N. Babu mobilized to the Site to conduct a second round of sampling activities. START selected nine locations around the house for surface soil sampling. Soil sample LL-SS-001 was collected north of the fire pit. Soil sample LL-SS-002 was collected west of the fire pit, Soil sample LL-SS-003 was collected just north of the basketball hoop, Soil sample LL-SS-004 was collected north of the house, Soil sample LL-SS-005 was collected south of the storage shed, Soil sample LL-SS-006 was collected in front of the dumpsters, Soil sample LL-SS-007 was collected near the well located south of the house, Soil sample LL-SS-008 was collected near the creek located west of the house, Soil sample LL-SS-009 was a duplicate grab sample of sample LL-SS-008, and Soil sample LL-SS-010 was collected on the sloped area in the northernmost corner of the property. The ten soil samples were collected for total Michigan-ten (MI-10) metals analysis and hand-delivered to Trimatrix Laboratories in Grand Rapids, Michigan. Upon receipt of the analytical data, four samples, LL-SS-004, LL-SS-005, LL-SS-007 and LL-SS-010 with the highest total lead results were requested for additional TCLP analysis.

On February 9<sup>th</sup>, 2012, U.S. EPA J. Lippert and START C. Schulz conducted additional sampling activities. A total of five surface soil samples were collected during the third round of sampling. Sample LL-21 was collected from the southern corner of the site, Sample LL-22 was collected from the western corner of the Site on the north side of the driveway, Sample LL-23 was collected on the hill in the northern corner of the Site near the creek, Sample LL-24 was collected in front of the house on the southwest side of the driveway, and Sample LL-25 was collected on the southwest side of the basketball

hoop in front of the shed, northwest of the house. Samples were packaged, preserved on ice, and hand-delivered to Trimatrix Laboratories in Grand Rapids, MI for total MI-10 metals analysis. Upon receipt of the analytical data, three samples, LL-21, LL-22, and LL-24, with the highest total lead results were requested for additional TCLP analysis.

For a complete list of the sample locations and descriptions see Table 1. All sample locations are shown in Figure 3.

**Table 1  
Sampling Location and Description  
Lovers Lane Lead Site Assessment  
Comstock Park, Kent County, Michigan**

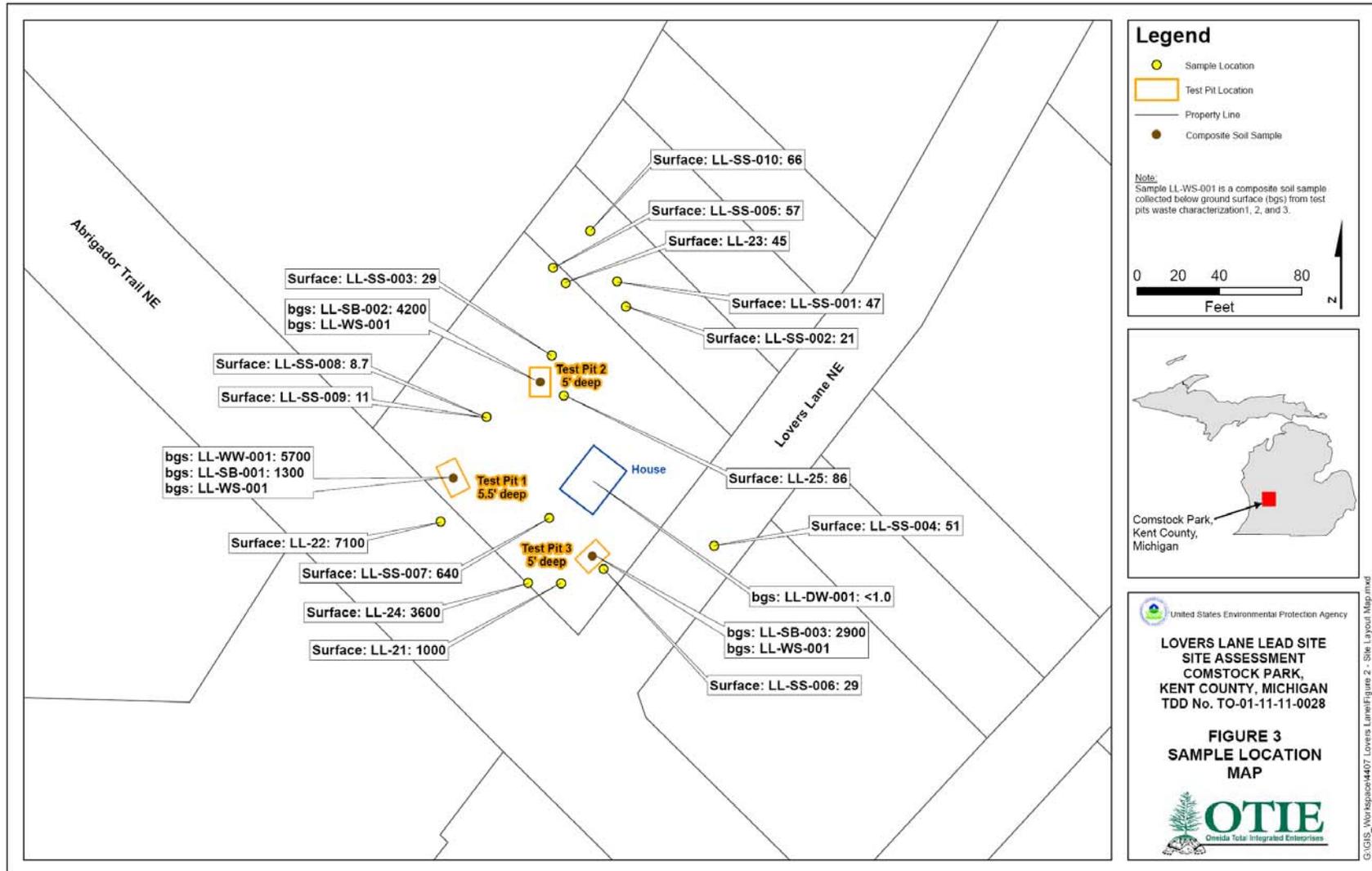
Sample ID	Sample Description	Date Collected	Latitude	Longitude
LL-SB-001	Composite soil sample collected below ground surface from Test Pit 1	12/20/2011	--	--
LL-SB-002	Composite soil sample collected below ground surface from Test Pit 2	12/20/2011	--	--
LL-SB-003	Composite soil sample collected below ground surface from Test Pit 3	12/20/2011	--	--
LL-WS-001	Waste characterization soil sample collected below ground surface from Test Pits 1, 2, &3	12/20/2011	--	--
LL-WW-001	Water sample infiltrated from Test Pit # 1	12/20/2011	--	--
LL-DW-001	Drinking water sample collected from kitchen tap inside the house	12/20/2011	--	--
LL-SS-001	Surface soil sample collected north of the fire pit	1/5/2012	43.0440333	-85.6551167
LL-SS-002	Surface soil sample collected west of the fire pit	1/5/2012	43.0440000	-85.6551000
LL-SS-003	Surface soil sample collected north of the basketball hoop	1/5/2012	43.0439333	-85.6552333
LL-SS-004	Surface soil sample collected north of the house	1/5/2012	43.0436833	-85.6549333
LL-SS-005	Surface soil sample collected south of the shed	1/5/2012	43.0440500	-85.6552333
LL-SS-006	Surface soil sample collected west of the trash bins	1/5/2012	43.0436500	-85.6551333
LL-SS-007	Surface soil sample collected near the well	1/5/2012	43.0437167	-85.6552333
LL-SS-008	Surface soil sample collected near the creak	1/5/2012	43.0438500	-85.6553500
LL-SS-009	Duplicate of sample LL-SS-008	1/5/2012	43.0438500	-85.6553500
LL-SS-010	Surface soil sample collected on the sloped area in the northernmost corner of the Site property	1/5/2012	43.0441000	-85.6551667
LL-21	Surface soil sample collected in the front yard in the south corner of site.	2/9/2012	43.04363	-85.65521
LL-22	Surface soil sample collected in the front yard in the west corner of site, northwest of driveway.	2/9/2012	43.04371	-85.65543
LL-23	Surface soil sample collected on the hill behind the house in the north corner of site, near basketball hoop and creek.	2/9/2012	43.04403	-85.655521
LL-24	Surface soil sample collected in front of house, southwest of the driveway.	2/9/2012	43.04363	-85.65527
LL-25	Southwest of basketball hoop, in front of shed, northwest of house.	2/9/2012	43.04388	-85.65521

**Notes:**

Sample IDs containing "SB", "WS", and "SS" were soil samples collected from subsurface soil, waste soil, and surface soil respectively.

Sample IDs containing "WW" and "DW" were water samples collected from infiltrated water, and drinking water respectively.





#### 4. ANALYTICAL RESULTS

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START reviewed the sample analytical data and supporting quality assurance/quality control (QA/QC) data provided by Trimatrix Laboratories. The validated analytical data package is included in Appendix B. Based on START's data validation, the data are acceptable for use as qualified.

Metal analytical results of soil samples collected during the site assessment are shown in Table 2. Results in Table 2 were compared against values listed by MDEQ in the Remediation and Redevelopment Division (RRD) Operational Memoranda Part 201 Cleanup Criteria Residential Direct Contact Criteria (DCC) and drinking water protection criteria (DWPC). The soil DCC represents a soil concentration that is protective against adverse health effects due to long-term ingestion of and dermal contact with contaminated soil. DWPC is developed to assess the potential for hazardous substances in soil to leach and impact groundwater at concentrations greater than the applicable generic drinking water, groundwater surface water interface, and groundwater contact criteria.

TCLP metals analytical results for soil samples collected during the site assessment are shown in Table 3. Results in Table 3 were compared against values in 40 Code of Federal Regulations (CFR) Section 261.22-261.24 listed for identifying characteristics of hazardous waste.

Results for the water samples and waste characterization sample collected during the site assessment are shown in Table 4 and Table 5, respectively. Results in Table 4 were compared against federal maximum contaminant levels (MCLs) and values listed by MDEQ Part 201 Cleanup Criteria groundwater contact criteria and Residential Drinking Water Criteria. Results in Table 5 were compared against values in 40 Code of Federal Regulations (CFR) Section 261.22 and 261.24.

The total lead analytical results exceeded the MDEQ Part 201 residential DCC for four surface soil samples. Surface soil samples LL-SS-007, LL-21, LL-22, and LL-24 exceeded the MDEQ Part 201 residential DCC of 400 mg/Kg for lead with reported values of 640 mg/Kg, 1,000 mg/Kg, 7,100 mg/Kg, and 3,600 mg/Kg, respectively.

The soil TCLP lead analytical results exceeded the federal TCLP hazardous waste limit for six soil samples. Soil samples LL-SB-001, LL-SB-002, LL-SB-003, LL-21, LL-22, and LL-24 exceeded the federal TCLP limit of 5 mg/L for lead with reported values of 940 mg/L, 13 mg/L, 46 mg/L, 6.3 mg/L, 120 mg/L and 12 mg/L respectively.

Analytical results of the metals in tap water sample, LL-DW-001, were below the federal MCL limits. Lead was detected at 5,700  $\mu\text{g/L}$  in the infiltrated water sample, LL-WW-001, collected from Test Pit #1. There is no lead value listed by MDEQ under Groundwater Contact Criteria (GCC) due to insufficient data to develop the criterion. GCC is determined for exposure to groundwater contaminants by a utility worker during work activities such as utility line repair, maintenance, and installation etc.

TCLP analytical result of the waste characterization soil sample, LL-WS-001, exceeded the federal TCLP limit of 5 mg/L for lead with a reported value of 36 mg/L.

**Table 2**  
**Total Metals Soil Analytical Results**  
**Lovers Lane Lead Site Assessment**  
**Comstock Park, Kent County, Michigan**

ANALYTE	MDEQ Part 201 Residential Direct Contact Criteria (mg/Kg)	MDEQ Part 201 Residential Drinking Water Protection Criteria (mg/Kg)	Total Metals (mg/kg Dry wt.)								
			LL-SB-001	LL-SB-002	LL-SB-003	LL-SS-001	LL-SS-002	LL-SS-003	LL-SS-004	LL-SS-005	LL-SS-006
Arsenic	7.6	4.6	2.5	10	4.1 J	<b>22</b>	<b>5.9</b>	2.2	1.7	<b>5.8</b>	1.6
Barium	37,000	1,300	22	1,200	130	61	29	35	25	31	29
Cadmium	550	6.0	0.079	4.5	0.70 J	0.67	0.36	0.34	0.37	0.26	0.20
Chromium (VI)	2,500	30	6.6	24	9.9 J	18	13	9.5	6.2	11	6.0
Copper	20,000	5,800	NA	NA	NA	46	20	19	8.6	9.3	14
Lead	400	700	<b>1,300</b>	<b>4,200</b>	<b>2,900</b>	47	21	29	51	57	29
Mercury	160	1.7	ND	0.24	ND	ND	ND	ND	ND	ND	ND
Selenium	2,600	4.0	ND	0.93	0.25	ND	ND	ND	ND	ND	ND
Silver	2,500	4.5	ND	0.22	0.063	0.066	0.077	0.053	ND	0.055	ND
Zinc	170,000	2,400	NA	NA	NA	230	71	82	57	65	53

Notes:  
mg/Kg dry wt.- milligram per kilograms dry weight  
ND – analyte was analyzed for but not detected  
NA – analyte not analyzed  
**BOLD-** analytical result exceeded the Residential Drinking Water Protection Criteria  
**BOLD-** analytical result exceeded both the Residential Direct Contact Criteria and the Residential Drinking Water Protection Criteria  
Samples were collected on December 20<sup>th</sup>, 2011, January 5<sup>th</sup>, 2012, and February 9<sup>th</sup>, 2012 under START contract EP-S5-10-10.  
Analyses were conducted by Trimatrix Laboratories, Inc. under TDD No: TO-01-11-11-0028

**Table 2 (continued)**  
**Total Metals Soil Analytical Results**  
**Lovers Lane Lead Site Assessment**  
**Comstock Park, Kent County, Michigan**

ANALYTE	Residential Direct Contact Criteria (mg/Kg)	Residential Drinking Water Protection Criteria (mg/Kg)	Total Metals (mg/kg Dry wt.)								
			LL-SS-007	LL-SS-008	LL-SS-009	LL-SS-010	LL-21	LL-22	LL-23	LL-24	LL-25
Arsenic	7.6	4.6	2.4	3.7	3.5	<b>490</b>	2.8	<b>7.2</b>	<b>160</b>	<b>5.2</b>	3.6
Barium	37,000	1,300	29	46	43	99	27	51	60	71	35
Cadmium	550	6.0	0.10	0.11	0.12	0.59	0.13	0.24	0.53	0.27	0.11
Chromium (VI)	2,500	30	8.8	13	13	<b>300</b>	8.8	12	<b>96</b>	14	14
Copper	20,000	5,800	7.0	9.4	8.8	470	5.1	7.1	150	7.3	7.6
Lead	400	700	<b>640</b>	8.7	11	66	<b>1,000</b>	<b>7,100</b>	45	<b>3,600</b>	86
Mercury	160	1.7	ND	ND	ND	ND	ND	ND	ND	0.053	ND
Selenium	2,600	4.0	ND	ND	ND	0.22	0.23	0.34	0.29	0.40	0.23
Silver	2,500	4.5	0.048	0.080	0.066	0.093	ND	0.079	ND	0.052	ND
Zinc	170,000	2,400	24	33	33	240	24	35	170	39	28

Notes:

mg/Kg dry wt.- milligram per kilograms dry weight

ND – analyte was analyzed for but not detected

NA – analyte not analyzed

**BOLD-** analytical result exceeded the Residential Drinking Water Protection Criteria

**BOLD-** analytical result exceeded both the Residential Direct Contact Criteria and the Residential Drinking Water Protection Criteria

Samples were collected on December 20<sup>th</sup>, 2011, January 5<sup>th</sup>, 2012, and February 9<sup>th</sup>, 2012 under START contract EP-S5-10-10.

Analyses were conducted by Trimatrix Laboratories, Inc. under TDD No: TO-01-11-11-0028

**Table 3  
TCLP Metals Soil Analytical Results  
Lovers Lane Lead Site Assessment  
Comstock Park, Kent County, Michigan**

ANALYTE	40 CFR Section 261 TCLP Regulatory Limit (mg/L)	TCLP RCRA 8 Metals (mg/L)			TCLP Lead (mg/L)						
		LL-SB-001	LL-SB-002	LL-SB-003	LL-SS-004	LL-SS-005	LL-SS-007	LL-SS-010	LL-21	LL-22	LL-24
Barium	100.0	0.42 J	0.66	0.53	NA	NA	NA	NA	NA	NA	NA
Cadmium	1.0	ND	0.064	ND	NA	NA	NA	NA	NA	NA	NA
Lead	5.0	<b>940 J</b>	<b>13</b>	<b>46</b>	ND	ND	2.5 J	ND	<b>6.3</b>	<b>120</b>	<b>12</b>

Notes:

TCLP - Toxicity Characteristic Leaching Procedure

mg/L- milligram per liter

ND – analyte was analyzed for but not detected

NA – analyte not analyzed

**BOLD**- analytical result exceeded the 40 CFR Section 261 TCLP Regulatory Limit

Samples were collected on December 20<sup>th</sup>, 2011, January 5<sup>th</sup>, 2012, and February 9<sup>th</sup>, 2012 under START contract EP-S5-10-10.

Analyses were conducted by Trimatrix Laboratories, Inc. under TDD No: TO-01-11-11-0028

**Table 4  
Water Sample Analytical Results  
Lovers Lane Lead Site Assessment  
Comstock Park, Kent County, Michigan**

<b>Analyte</b>	<b>Water from Test pit #1</b>		<b>Tapwater from the residence</b>		
<b>Analyte</b>	<b>MDEQ Part 201 Groundwater Contact Criteria (µg/L)</b>	<b>LL-WW-001</b>	<b>MDEQ Part 201 Residential Drinking Water Criteria (µg/L)</b>	<b>Federal MCL (µg/L)</b>	<b>LL-DW-001</b>
Arsenic	4,300	19 J	10	10	ND
Barium	14,000,000	230	2,000	2,000	42
Cadmium	190,000	0.68	5	5	ND
Chromium	290,000,000	18	100	100	ND
Lead	ID	5,700	4	15	ND
Silver	1,500,000	0.27	34	NL	ND

Notes:

Sample ID containing “WW” was the infiltrated water sample from Test Pit #1.

Sample ID containing “DW” was a tap water sample from the on-site residence.

ID - insufficient data to develop criterion.

NL – Not listed under Federal MCLs

ND – analyte was analyzed for but not detected above the method detection limit

µg/L- microgram per liter

Samples were collected on December 20<sup>th</sup>, 2011, January 5<sup>th</sup>, 2012, and February 9<sup>th</sup>, 2012 under START contract EP-S5-10-10.

Analyses were conducted by Trimatrix Laboratories, Inc. under TDD No: TO-01-11-11-0028

<p align="center"><b>Table 5</b>  <b>Waste Characterization Sample Analytical Results</b>  <b>Lovers Lane Lead Site Assessment</b>  <b>Comstock Park, Kent County, Michigan</b></p>		
<b>ANALYTE</b>	<b>40 CFR Section 261 Regulatory Limit<sup>1</sup></b>	<b>LL-WS-001</b>
<b>pH (SU)</b>	<2 or >12.5	7.7
<b>TCLP Metals (mg/L)</b>		
<b>Barium</b>	100	0.75
<b>Lead</b>	5	<b>36</b>
<b>TCLP VOCs (mg/L)</b>		
TCLP VOCs	varies	ND
<b>TCLP SVOCs (mg/L)</b>		
TCLP SVOCs	varies	ND
<b>PCBs (mg/Kg)</b>		
PCB Aroclors	NL	ND

Notes:

<sup>1</sup> - Hazardous Waste Characterization Criteria according to 40 CFR Sections 261.21-261.24

SU - Standard Unit

VOCs - Volatile Organic Compounds

SVOCs - Semi Volatile Organic Compounds

PCBs - Poly Chlorinated Biphenyls

NL - Not listed under Federal MCLs

ND - analyte was analyzed for but not detected above the method detection limit

mg/L - milligrams per liter

mg/Kg - milligrams per kilogram

**BOLD-** analyte exceeded the 40 CFR Section 261 criteria

Samples were collected on December 20<sup>th</sup>, 2011, January 5<sup>th</sup>, 2012, and February 9<sup>th</sup>, 2012 under START contract EP-S5-10-10.

Analyses were conducted by Trimatrix Laboratories, Inc. under TDD No: TO-01-11-11-0028

## 5. POTENTIAL SITE RELATED THREATS

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Threats posed by the Site were evaluated in accordance with the National Oil and Hazardous Substances Pollution Contingency Plan (NCP) criteria for initiating a removal action listed under Title 40 of the CFR, Section 300.415(b) (2). Paragraph (b) (2) of 40 CFR Section 300.415 lists factors to be considered when determining the appropriateness of a potential removal action at a site. Potential site-related threats to human health and the environment were evaluated based on the criteria listed in 40 CFR, Sections 261.20 through 261.24, and MDEQ residential DCC and DWPC. Factors that are applicable to the Site for the appropriateness of a removal action are discussed below.

### **Actual or potential exposure of nearby human populations, animals, or the food chain to hazardous substances or pollutants or contaminants**

During the Site Assessment, broken pieces of lead battery casings were spotted on the top surface of the yard at several locations all around the property. Layers of lead battery casings were found at 1-foot to 5-foot bgs in three of the four test pit locations. Total metals analytical results of five surface soil samples collected during the site assessment exceeded the MDEQ Part 201 residential DCC of 400 mg/Kg for lead. TCLP metals analytical results of seven surface and subsurface soil samples collected during the SA exceeded the federal TCLP hazardous characteristic limit of 5 mg/L for lead. A sample of the water infiltrated into one of the test pits was reported with 5,700 µg/L lead.

As a probable human carcinogen, lead may cause significant health effects through any route of exposure. Lead exposure even at low levels can permanently affect children resulting in nervous system damage, learning disabilities, attention deficit disorder, and decreased intelligence. Lead exposures at high levels may cause more serious health effects such as seizures, unconsciousness, and possibly death. (ATSDR, 2012)

The Site has no fencing around the property with unrestricted access from all sides of the Site.

The presence of elevated levels of lead in soil and groundwater poses a threat to the residents on-site and off-site through direct exposure since the Site has unrestricted access.

**High levels of hazardous substances or pollutants or contaminants in soils largely at or near the surface, that may migrate.**

TCLP metals analytical results of seven surface and subsurface soil samples collected during the SA exceeded the federal lead TCLP limit of 5 mg/L to be classified as hazardous characteristic. TCLP lead concentrations in surface and subsurface soil indicate that the lead in soil can potentially leach into storm water or water from gardening and migrate into groundwater or run off-site to the nearby Grand River.

**Weather conditions that may cause hazardous substances or pollutants or contaminants to migrate or be released**

Broken lead-acid battery casings were found in surface soils and in many layers of subsurface soils during the excavation on December 20<sup>th</sup>, 2011. The site is located approximately 0.1 miles from the Grand River and in an area that is notorious for flooding every spring from the Grand River. Locals often wade through knee-deep water to reach their homes located along the river. The water runoff from these flooding events on the site may cause the lead to migrate off-site and/or leach into surrounding soils and residences.

**The availability of other appropriate federal or state response mechanisms to respond to the release**

MDEQ requested the assistance of U.S. EPA Region 5 Emergency Response Branch to help evaluate and mitigate a possible threat posed by the Lovers Lane Lead Site. This request was made to U.S. EPA since MDEQ does not have appropriate state response mechanisms or resources to respond.

## 6. SUMMARY

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On December 20th, 2011; January 5th, 2012; and February 9th, 2012, U.S. EPA and START conducted SA activities at the Lovers Lane Lead Site in Comstock Park, Michigan. During sampling activities, soil and water samples were collected and submitted for pH, total and TCLP metals, PCBs, TCLP VOCs, and TCLP SVOCs analysis.

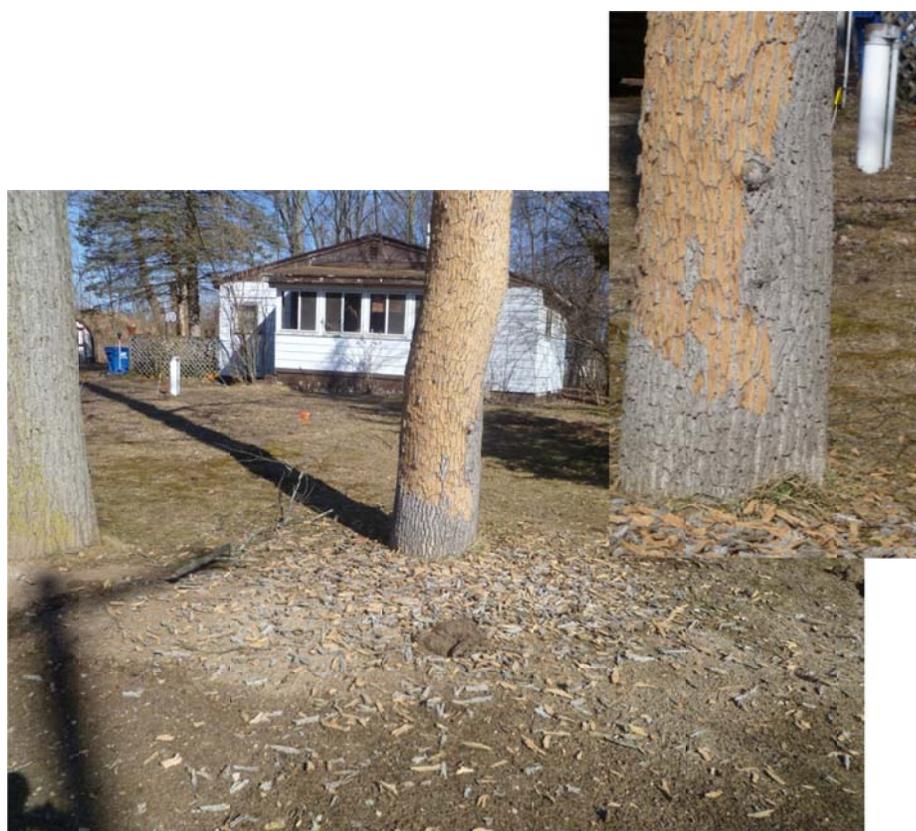
Sample analytical results were evaluated against the characteristics of hazardous waste as listed under 40 CFR, Sections 261.22 through 261.24 and against values listed by MDEQ in the Remediation and Redevelopment Division (RRD) Operational Memoranda Part 201 Cleanup Criteria residential DCC and DWCP. Surface and subsurface soil samples indicated the presence of elevated levels of lead. Surface soil samples contained levels of total lead above MDEQ residential DCC posing a threat of exposure to the residents on-site and off-site. Surface and subsurface soil samples contained TCLP lead levels above federal TCLP limit, posing a threat of release to the groundwater and the nearby Grand River. Thus, a removal action is warranted at this Site to abate threats to human health and the environment.

## 7. REFERENCES

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Agency for Toxic Substances & Disease Registry (2012). Toxic Substances Portal-Lead. Retrieved on March 02, 2012 from <http://www.atsdr.cdc.gov/substances/toxsubstance.asp?toxid=22>.

**APPENDIX A**  
**PHOTOGRAPHIC LOG**  
**(5 Pages)**



**Photograph No.:** 1      **Photographer:** Carly Schulz      **Orientation:** Northeast  
**TDD Number:** TO-01-11-11-0028      **Contract:** EP-S5-10-10, OTIE      **Date:** 02/09/2012  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** Tree that appears to be dying in front yard near well. The residential house is visible in the background.



**Photograph No.:** 2      **Photographer:** Naren Babu      **Orientation:** Northeast  
**TDD Number:** TO-01-11-11-0028      **Contract:** EP-S5-10-10, OTIE      **Date:** 12/20/2011  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** View of Test Pit #1, excavated just northwest of the driveway in the front yard.



**Photograph No.:** 3      **Photographer:** Naren Babu      **Orientation:** Downward  
**TDD Number:** TO-01-11-11-0028      **Contract:** EP-S5-10-10, OTIE      **Date:** 12/20/2011  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** View of layers of soil inside Test Pit #1.



**Photograph No.:** 4      **Photographer:** Naren Babu      **Orientation:** Downward  
**TDD Number:** TO-01-11-11-0028      **Contract:** EP-S5-10-10, OTIE      **Date:** 12/20/2011  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** Infiltrated groundwater at approximately five feet bgs inside Test Pit #1.



**Photograph No.:** 5      **Photographer:** Naren Babu      **Orientation:** Northeast  
**TDD Number:** TO-01-11-11-0028      **Contract:** EP-S5-10-10, OTIE      **Date:** 12/20/2011  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** View of Test Pit #1 after backfills.



**Photograph No.:** 6      **Photographer:** Naren Babu      **Orientation:** Northeast  
**TDD Number:** TO-01-11-11-0028      **Contract:** EP-S5-10-10, OTIE      **Date:** 12/20/2011  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** View of broken lead-acid battery casings and soil in the bucket excavated from Test Pit #2.



**Photograph No.:** 7                      **Photographer:** Naren Babu                      **Orientation:** Downward  
**TDD Number:** TO-01-11-11-0028   **Contract:** EP-S5-10-10, OTIE                      **Date:** 12/20/2011  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** Plastic lead-acid battery casing found on top of soil throughout site.



**Photograph No.:** 8                      **Photographer:** Naren Babu                      **Orientation:** North  
**TDD Number:** TO-01-11-11-0028   **Contract:** EP-S5-10-10, OTIE                      **Date:** 01/05/2012  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** Soil Sample LL-SS-007 collected southeast of the well in front of the house.



**Photograph No.:** 9      **Photographer:** Carly Schulz      **Orientation:** North  
**TDD Number:** TO-01-11-11-0028      **Contract:** EP-S5-10-10, OTIE      **Date:** 02/09/2012  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** Surface soil sample LL-21 collected southwest of the house near Lovers Lane Street NE.



**Photograph No.:** 10      **Photographer:** Carly Schulz      **Orientation:** East  
**TDD Number:** TO-01-11-11-0028      **Contract:** EP-S5-10-10, OTIE      **Date:** 02/09/2012  
**Site Name & Location:** Lovers Lane Site, Kent County, Michigan.  
**Subject:** Surface soil sample LL-22 collected in front of house northwest of the driveway.

**APPENDIX B**

**VALIDATED LABORATORY ANALYTICAL RESULTS**



## MEMORANDUM

**Date:** February 1, 2012

**To:** Naren Babu, Project Manager, OTIE  
Superfund Technical Assessment and Response Team (START) for Region 5

**Prepared by:** Renea Anglin, START chemist for Region 4

**QA/QC** Keely Meadows

**Concurrence by:**

**Subject:** Data Validation for  
Lovers Lane lead Site  
Comstock, MI  
Project TDD No. TO-01-11-11-0028

Laboratory: Trimatrix Laboratories, Grand Rapids, MI.  
Sample Delivery Group (SDG): 1112328

### 1.0 INTRODUCTION

The START chemist for Region 4 validated analytical data for 3 soil samples for Total RCRA 8 Metals, 1 soil sample for polychlorinated biphenyls (PCB) and % solids, and 2 water samples for Total RCRA 8 Metals. Samples were collected at the Lovers Lane Lead site on December 20, 2011. The samples were analyzed under SDG 1112328 by Trimatrix Laboratories of Grand Rapids, MI using U.S. Environmental Protection Agency (U.S. EPA) methods 6020A, 7471A, 7470A, 8082, and 3550B.

Laboratory data were validated using guidelines set forth in the U.S. EPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data Review (EPA-540-R-08-01, June 2008), NFG for Inorganic Data Review (EPA-540-R-10-011, January 2010), and applicable methodologies. The purpose of the chemical data quality evaluation process is to assess the usability of data for the project decision-making process.

Organic data validation consisted of a review of the following QC audits:

- Chain of custody and sample receipt forms review
- Sample preservation and holding time
- Blank results
- Surrogate recoveries
- Matrix spike and Matrix Spike Duplicate (MS/MSD) recovery results
- Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) recovery results

Inorganic data validation consisted of a review of the following QC audits:

- Chain of custody and sample receipt forms review
- Sample preservation and holding time
- Blank results
- Duplicate Sample Results
- LCS recovery results
- MS/MSD recovery results

Section 2.0 of this memorandum discusses the results of organic data validation. Section 3.0 of this memorandum discusses the results of inorganic data validation. Section 4.0 presents an overall assessment of the data. The attachment to this memorandum contains the laboratory reporting forms as well as START's handwritten data qualifications where warranted.

## **2.0 ORGANIC DATA VALIDATION RESULTS**

The results of START's organic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted (see attachment):

- J – The analyte was detected. The reported concentration was considered estimated.
- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives established for the project.

### **2.1 SOIL SAMPLES BY METHOD 8082**

#### ***2.1.1 SAMPLE HANDLING***

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Soil samples were collected on December 20, 2011 and were received on ice

No discrepancies were noted.

#### ***2.1.2 SAMPLE PRESERVATION AND HOLDING TIME***

Samples were shipped on ice and were analyzed within holding time criteria.

No discrepancies were noted.

#### ***2.1.3 BLANK RESULTS***

The purpose of laboratory blank analysis is to determine the existence and magnitude of contamination resulting from laboratory activities. A laboratory method blank sample (1114773MB) was run with this SDG.

No laboratory method blank detects were noted.

#### ***2.1.4 SURROGATE RECOVERIES***

Laboratory performance on individual samples is established by means of fortifying each sample with surrogate compounds. The surrogate spike compound included Decachlorobiphenyl and Tetrachloro-m-xylene.

The surrogate was within limits for samples analyzed in this SDG.

### ***2.1.5 MS/MSD RECOVERY RESULTS***

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

No MS/MSD samples were requested for this analysis.

### ***2.1.6 LCS RECOVERY RESULTS***

Data for the LCS is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS was fortified and analyzed with each batch of samples. The LCS accuracy performance is measured by %R.

All recoveries were within QC limits.

## **3.0 INORGANIC DATA VALIDATION RESULTS**

The results of START's inorganic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted:

- J – The analyte was detected. The reported concentration was considered estimated.
- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives established for the project.

## **3.1 SOIL SAMPLES BY METHOD 6010 B/7471**

### ***3.1.1 SAMPLE HANDLING***

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Soil samples were collected on December 20, 2011 and were received on ice.

### ***3.1.2 SAMPLE PRESERVATION AND HOLDING TIME***

Samples were analyzed within the holding time criteria. No discrepancies were noted.

### ***3.1.3 BLANK RESULTS***

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities. A laboratory method blank sample (1114600MB) for method 6020A and a laboratory method blank sample (1114521MB) for method 7471A were run with this SDG.

No laboratory method blank detects were noted.

### ***3.1.4 LCS RECOVERY RESULTS***

The LCS serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. The LCS is fortified with each analyte of interest and analyzed with each batch of samples. The LCS accuracy performance is measured by %R.

The LCS recoveries were within acceptable recovery limits.

### ***3.1.5 MS/MSD RECOVERY RESULTS***

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The MS/MSD accuracy performance is measured by %R.

An MS/MSD was not requested with this SDG; however, a MS/MSD was run on sample LL-SB-003. MS/MSD results were not available for Barium or Lead due to the large dilutions necessary on the native sample. The MS had an Arsenic result biased high at 127%R. The MSD had a Cadmium result biased high at 171%R and a Chromium result biased low at 72%R. In addition, the RPD for Cadmium was outside QC limits at 39%RPD. Mercury was biased high in the MS at 124%R and MSD at 125%R. Therefore, Arsenic, Cadmium, and Chromium were flagged as J in sample LL-SB-003. Mercury will be flagged as UJ in sample LL-SB-003.

### ***3.1.6 GENERAL LABORATORY OBSERVATIONS***

Samples LL-SB-001, LL-SB-002, and LL-SB-003 had Barium and Lead reported from dilutions, which raised the reporting limit for these two analytes. Sample LL-SB-003 also had a dilution on Chromium, which raised the reporting limit.

## **3.2 WATER SAMPLES BY METHOD 6020A/7471A**

### ***3.2.1 SAMPLE HANDLING***

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Water samples were collected on December 20, 2011 and were received on ice. No discrepancies were noted.

### ***3.2.2 SAMPLE PRESERVATION AND HOLDING TIME***

Samples were analyzed within the holding time criteria. No discrepancies were noted.

### ***3.2.3 BLANK RESULTS***

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities. A laboratory method blank sample (1114524MB) for method 6020A and a laboratory method blank sample (1114639MB) for method 7470A were run with this SDG.

No laboratory method blank detects were noted.

### ***3.2.4 LCS RECOVERY RESULTS***

The LCS serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. The LCS is fortified with each analyte of interest and analyzed with each batch of samples. The LCS accuracy performance is measured by %R.

The LCS/LCSD %R were all within acceptable recovery limits.

### ***3.2.5 MS/MSD RECOVERY RESULTS***

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The MS/MSD accuracy performance is measured by %R.

No MS/MSD was requested for these analyses for this SDG; however, a MS/MSD was run on sample LL-WW-001. No results were reported for Lead due to the high level of lead in the native sample. The MS had an Arsenic recovery biased low at 73%. Therefore, Arsenic was flagged as J in sample LL-WW-001.

### ***3.2.6 GENERAL LABORATORY OBSERVATIONS***

Sample LL-WW-001 had Barium and Lead reported from dilutions which raised the reporting limit for these two analytes.

## **4.0 OVERALL ASSESSMENT OF DATA**

The analytical results meet the data quality objectives defined by the applicable method and validation guidance documentation. The analytical data is usable and acceptable as reported by the laboratory.

**ATTACHMENT**  
**SUMMARY OF VALIDATED ANALYTICAL RESULTS**  
**AND**  
**CHAIN-OF-CUSTODY**

**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112328</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-SB-001</b>	Sampled:	12/20/11 09:00
Lab Sample ID:	<b>1112328-01</b>	Sampled By:	N.B., C.S.
Matrix:	Soil	Received:	12/20/11 13:50
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>2.5</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:25	MSM	1114600
<b>Barium</b>	<b>22</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	12/28/11 14:51	MSM	1114600
<b>Cadmium</b>	<b>0.079</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:25	MSM	1114600
<b>Chromium</b>	<b>6.6</b>	0.20	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:25	MSM	1114600
<b>Lead</b>	<b>1300</b>	50	mg/kg dry wt.	500	USEPA-6020A	12/27/11 16:35	MSM	1114600
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	12/22/11 11:59	KLV	1114521
Selenium	<0.20	0.20	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:25	MSM	1114600
Silver	<0.050	0.050	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:25	MSM	1114600

*SA*  
1-30-12

**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112328</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-WW-001</b>	Sampled:	12/20/11 09:15
Lab Sample ID:	<b>1112328-02</b>	Sampled By:	N.B., C.S.
Matrix:	Water	Received:	12/20/11 13:50

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>19</b> J	1.0	ug/L	1	USEPA-6020A	01/04/12 11:57	DSC	1114524
<b>Barium</b>	<b>230</b>	5.0	ug/L	5	USEPA-6020A	01/03/12 12:30	DSC	1114524
<b>*Cadmium</b>	<b>0.68</b>	0.20	ug/L	1	USEPA-6020A	01/03/12 09:39	DSC	1114524
<b>*Chromium</b>	<b>18</b>	5.0	ug/L	1	USEPA-6020A	01/03/12 09:39	DSC	1114524
<b>*Lead</b>	<b>5700</b>	100	ug/L	100	USEPA-6020A	01/03/12 12:20	DSC	1114524
Mercury	<0.20	0.20	ug/L	1	USEPA-7470A	12/29/11 14:25	DSC	1114639
Selenium	<2.0	2.0	ug/L	1	USEPA-6020A	01/03/12 09:39	DSC	1114524
<b>*Silver</b>	<b>0.27</b>	0.20	ug/L	1	USEPA-6020A	01/03/12 09:39	DSC	1114524

\*See Statement of Data Qualifications

*Handwritten signature and date: 1-30-12*

**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112328</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-SB-002</b>	Sampled:	12/20/11 09:50
Lab Sample ID:	<b>1112328-03</b>	Sampled By:	N.B., C.S.
Matrix:	Soil	Received:	12/20/11 13:50
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>10</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	12/27/11 16:38	MSM	1114600
<b>Barium</b>	<b>1200</b>	20	mg/kg dry wt.	200	USEPA-6020A	12/27/11 16:37	MSM	1114600
<b>Cadmium</b>	<b>4.5</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:26	MSM	1114600
<b>Chromium</b>	<b>24</b>	1.0	mg/kg dry wt.	5	USEPA-6020A	12/27/11 16:38	MSM	1114600
<b>Lead</b>	<b>4200</b>	100	mg/kg dry wt.	1000	USEPA-6020A	12/27/11 15:23	MSM	1114600
<b>Mercury</b>	<b>0.24</b>	0.046	mg/kg dry wt.	1	USEPA-7471A	12/22/11 12:04	KLV	1114521
<b>Selenium</b>	<b>0.93</b>	0.20	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:26	MSM	1114600
<b>Silver</b>	<b>0.22</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:26	MSM	1114600

*Handwritten signature and date: 1-30-12*

### ANALYTICAL REPORT

Client:	Oneida Total Integrated Enterprises	Work Order:	1112328
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	LL-SB-003	Sampled:	12/20/11 10:20
Lab Sample ID:	1112328-04	Sampled By:	N.B., C.S.
Matrix:	Soil	Received:	12/20/11 13:50
Percent Solids:	n/a		

#### Total Metals by EPA 6000/7000 Series Methods

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
*Arsenic	4.1 <i>J</i>	0.10	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:28	MSM	1114600
*Barium	130	2.5	mg/kg dry wt.	25	USEPA-6020A	12/27/11 16:54	MSM	1114600
*Cadmium	0.70 <i>J</i>	0.050	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:28	MSM	1114600
*Chromium	9.9 <i>J</i>	1.0	mg/kg dry wt.	5	USEPA-6020A	12/27/11 16:45	MSM	1114600
*Lead	2900	100	mg/kg dry wt.	1000	USEPA-6020A	12/27/11 15:10	MSM	1114600
*Mercury	<0.050 <i>uJ</i>	0.050	mg/kg dry wt.	1	USEPA-7471A	12/22/11 12:09	KLV	1114521
Selenium	0.25	0.20	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:28	MSM	1114600
*Silver	0.063	0.050	mg/kg dry wt.	1	USEPA-6020A	12/27/11 15:28	MSM	1114600

\*See Statement of Data Qualifications

*Signature* 1-30-12

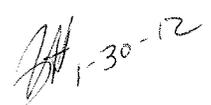
**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112328</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-WS-001</b>	Sampled:	12/20/11 10:45
Lab Sample ID:	<b>1112328-05</b>	Sampled By:	N.B., C.S.
Matrix:	Soil	Received:	12/20/11 13:50
Unit:	mg/kg dry	Prepared:	12/29/11 By: BJH
Dilution Factor:	1	Analyzed:	12/30/11 By: MSZ
QC Batch:	1114773	Analytical Batch:	1L30018
Percent Solids:	87		

**\*Polychlorinated Biphenyls (PCBs) by EPA Method 8082**

CAS Number	Analyte	Analytical Result	RL
12674-11-2	PCB-1016	<0.38	0.38
11104-28-2	PCB-1221	<0.38	0.38
11141-16-5	PCB-1232	<0.38	0.38
53469-21-9	PCB-1242	<0.38	0.38
12672-29-6	PCB-1248	<0.38	0.38
11097-69-1	PCB-1254	<0.38	0.38
11096-82-5	PCB-1260	<0.38	0.38
<b>Surrogates:</b>			
		<b>% Recovery</b>	<b>Control Limits</b>
	<i>Decachlorobiphenyl</i>	93	48-136
	<i>Tetrachloro-m-xylene</i>	93	61-123

\*See Statement of Data Qualifications



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112328</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-WS-001</b>	Sampled:	12/20/11 10:45
Lab Sample ID:	<b>1112328-05</b>	Sampled By:	N.B., C.S.
Matrix:	Soil	Received:	12/20/11 13:50

**Physical/Chemical Parameters by EPA/APHA/ASTM Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Percent Solids	<b>87</b>	0.1	%	1	USEPA-3550B	12/22/11 14:00	CLB	1114568

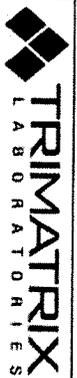
*Handwritten signature and date: 1-30-12*

### ANALYTICAL REPORT

Client:	Oneida Total Integrated Enterprises	Work Order:	1112328
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	LL-DW-001	Sampled:	12/20/11 12:45
Lab Sample ID:	1112328-06	Sampled By:	N.B., C.S.
Matrix:	Water	Received:	12/20/11 13:50

#### Total Metals by EPA 6000/7000 Series Methods

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Arsenic	<1.0	1.0	ug/L	1	USEPA-6020A	01/04/12 12:10	DSC	1114524
<b>Barium</b>	<b>42</b>	1.0	ug/L	1	USEPA-6020A	01/03/12 10:02	DSC	1114524
Cadmium	<0.20	0.20	ug/L	1	USEPA-6020A	01/03/12 10:02	DSC	1114524
Chromium	<5.0	5.0	ug/L	1	USEPA-6020A	01/03/12 10:02	DSC	1114524
Lead	<1.0	1.0	ug/L	1	USEPA-6020A	01/03/12 10:02	DSC	1114524
Mercury	<0.20	0.20	ug/L	1	USEPA-7470A	12/29/11 14:51	DSC	1114639
Selenium	<2.0	2.0	ug/L	1	USEPA-6020A	01/03/12 10:02	DSC	1114524
Silver	<0.20	0.20	ug/L	1	USEPA-6020A	01/03/12 10:02	DSC	1114524



5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512  
Phone (616) 975-4500 Fax (616) 942-7463  
www.trimatrixlabs.com

### Chain of Custody Record

Analyses Requested

Form No. **091603B**  
Pg. **1** of **1**

ESSENTIALS

For Lab Use Only  
Cart **4**  
VOA Rack/Tray **4528**  
Receipt Log No. **1717**  
Project Chemist: **Lisa Harvey**  
Work Order No. **112328**

Client Name: **Onoda Total Integrated Enterprises**  
Address: **100 West Monroe, Suite 300**  
City, State Zip: **Chicago, IL 60603**  
Phone/Fax: **312-220-7000 (cell) 312-656-7695**  
Email: **NPAK@OTIE.COM**

Project Name: **Lovers Lane Lead Site, Cornstock, MI**  
Client Project No. / P.O. No.  
Invoice To:  Client  Other (comments)Contact/Report To: **NAREN BARDU**

Container Type (corresponds to Container Packing List)	16	16	17	17	17	17	17	17	17
Total RCRA 8 (soil)									
TCLP-RCRA 8 (soil)									
TCLP-VOC									
TCLP-SVOC									
PCB, %S (Soil)									
Total RCRA 8 (waste)									
TCLP-RCRA 8 (waste)									
pH (waste)									
<b>TOTAL METALS WATER</b>									

Number of Containers Submitted

ESSENTIALS  
A NONE pH-7  
B HNO<sub>3</sub> pH<2  
C H<sub>2</sub>SO<sub>4</sub> pH<2  
D 1+1 HCl pH<2  
E NaOH pH>12  
F ZnAc<sub>2</sub>/NaOH pH>12  
G MeOH  
H Other (note below)

Schedule	Matrix Code	Sample Number	Field Sample ID	Cooler ID	Sample Date	Sample Time	C	H	M	S	Matrix	16	16	17	17	17	17	17	17	17	Total	Sample Comments	
B		01	LL-5B-001		12/20/11	0900	X			S													
D		02	LL-WM-001		12/20/11	0915				X													
B		03	LL-5B-002		12/20/11	0950	X			S													
B		04	LL-5B-003		12/20/11	1020	X			S													
C		05	LL-W5-001		12/29/11	1045	X			S													
D		06	LLDN-601		12/20/11	1245	X			X													
			TRIP BREAK (3 hrs)		12-20-11	1330																	

Sampled By (print) **NAREN BARDU / SINDLEZ**  
Sampler's Signature *[Signature]*  
How Shipped?  Hand  Carrier  
Tracking No.  
Company **OTIE**

1. Requisitioned By *[Signature]* Date **12/20/11** Time **13:50**  
2. Requisitioned By *[Signature]* Date \_\_\_\_\_ Time \_\_\_\_\_  
3. Requisitioned By *[Signature]* Date **12/20/11** Time **13:50**

1. Received By *[Signature]* Date \_\_\_\_\_ Time \_\_\_\_\_  
2. Received By *[Signature]* Date \_\_\_\_\_ Time \_\_\_\_\_  
3. Received By *[Signature]* Date **12/20/11** Time **13:50**

ORIGINAL - LABORATORY COPY - FIELD/SAMPLER

Onoda (LoversLane 1211.xls) 12/15/2011



## MEMORANDUM

**Date:** February 1, 2012

**To:** Naren Babu, Project Manager, OTIE  
Superfund Technical Assessment and Response Team (START) for Region 5

**Prepared by:** Renea Anglin, START chemist for Region 4

**QA/QC** Keely Meadows

**Concurrence by:**

**Subject:** Data Validation for  
Lovers Lane Lead Site  
Comstock, MI  
Project TDD No. TO-01-11-11-0028

Laboratory: Trimatrix Laboratories, Grand Rapids, MI.  
Sample Delivery Group (SDG): 1112329

### 1.0 INTRODUCTION

The START chemist for Region 4 validated analytical data for 4 soil samples for Toxic Characteristic Leachate Procedure (TCLP) RCRA 8 Metals, 1 soil sample for TCLP Volatile Organic Compounds (VOCs), TCLP Semi-Volatile Organic Compounds (SVOCs), and pH. Samples were collected at the Lovers Lane Lead site on December 20, 2011. The samples were analyzed under SDG 1112329 by Trimatrix Laboratories of Grand Rapids, MI using U.S. Environmental Protection Agency (U.S. EPA) methods 1311/6010C, 1311/7470A, 1311/8260, 1311/8270 and 9045C.

Laboratory data were validated using guidelines set forth in the U.S. EPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data Review (EPA-540-R-08-01, June 2008), NFG for Inorganic Data Review (EPA-540-R-10-011, January 2010), and applicable methodologies. The purpose of the chemical data quality evaluation process is to assess the usability of data for the project decision-making process.

Organic data validation consisted of a review of the following QC audits:

- Chain of custody and sample receipt forms review
- Sample preservation and holding time
- Blank results
- Surrogate recoveries
- Matrix spike and Matrix Spike Duplicate (MS/MSD) recovery results
- Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) recovery results

Inorganic data validation consisted of a review of the following QC audits:

- Chain of custody and sample receipt forms review
- Sample preservation and holding time
- Blank results
- Duplicate Sample Results
- LCS recovery results

- MS/MSD recovery results

Section 2.0 of this memorandum discusses the results of organic data validation. Section 3.0 of this memorandum discusses the results of inorganic data validation. Section 4.0 presents an overall assessment of the data. The attachment to this memorandum contains the laboratory reporting forms as well as START's handwritten data qualifications where warranted.

## **2.0 ORGANIC DATA VALIDATION RESULTS**

The results of START's organic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted (see attachment):

- J – The analyte was detected. The reported concentration was considered estimated.
- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives established for the project.

## **2.1 TCLP SAMPLES BY METHOD 1311/8260**

### ***2.1.1 SAMPLE HANDLING***

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Soil samples were collected on December 20, 2011 and were received on ice

No discrepancies were noted.

### ***2.1.2 SAMPLE PRESERVATION AND HOLDING TIME***

Samples were shipped on ice and were analyzed within holding time criteria.

No discrepancies were noted.

### ***2.1.3 BLANK RESULTS***

The purpose of laboratory blank analysis is to determine the existence and magnitude of contamination resulting from laboratory activities. A laboratory method blank sample (1114675MB) was run with this SDG.

No laboratory method blank detects were noted.

### ***2.1.4 SURROGATE RECOVERIES***

Laboratory performance on individual samples is established by means of fortifying each sample with surrogate compounds. The surrogate spike compound included Dibromofluoromethane, 1,2-Dichloroethane-d4, Toluene-d8, and 4-Bromofluorobenzene.

The surrogates were within limits for samples analyzed in this SDG.

### ***2.1.5 MS/MSD RECOVERY RESULTS***

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

No MS/MSD samples were requested for this analysis.

### ***2.1.6 LCS RECOVERY RESULTS***

Data for the LCS is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS was fortified and analyzed with each batch of samples. The LCS accuracy performance is measured by %R.

All recoveries were within QC limits.

## **2.2 TCLP SAMPLES BY METHOD 1311/8270**

### ***2.2.1 SAMPLE HANDLING***

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Soil samples were collected on December 20, 2011 and were received on ice

No discrepancies were noted.

### ***2.2.2 SAMPLE PRESERVATION AND HOLDING TIME***

Samples were shipped on ice and were analyzed within holding time criteria.

No discrepancies were noted.

### ***2.2.3 BLANK RESULTS***

The purpose of laboratory blank analysis is to determine the existence and magnitude of contamination resulting from laboratory activities. A laboratory method blank sample (1114531MB) was run with this SDG.

No laboratory method blank detects were noted.

### ***2.2.4 SURROGATE RECOVERIES***

Laboratory performance on individual samples is established by means of fortifying each sample with surrogate compounds. The surrogate spike compounds included 2-Fluorophenol, Phenol-d6, Nitrobenzene-d5, 2-Fluorobiphenyl, 2,4,6-Tribromophenol, and o-Terphenyl.

The surrogates were within limits for samples analyzed in this SDG.

### ***2.2.5 MS/MSD RECOVERY RESULTS***

Data for MS/MSD are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis.

No MS/MSD samples were requested for this analysis.

### ***2.2.6 LCS RECOVERY RESULTS***

Data for the LCS is generated to provide information on the accuracy of the analytical method and on the laboratory performance. The LCS was fortified and analyzed with each batch of samples. The LCS accuracy performance is measured by %R.

Pentachlorophenol was biased low at 35%R. Therefore, pentachlorophenol was flagged as UJ in sample LL-WS-001.

## **3.0 INORGANIC DATA VALIDATION RESULTS**

The results of START's inorganic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted:

- J – The analyte was detected. The reported concentration was considered estimated.
- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives established for the project.

## **3.1 TCLP SAMPLES BY METHOD 1311/6010C/7471**

### ***3.1.1 SAMPLE HANDLING***

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Soil samples were collected on December 20, 2011 and were received on ice.

### ***3.1.2 SAMPLE PRESERVATION AND HOLDING TIME***

Samples were analyzed within the holding time criteria. No discrepancies were noted.

### ***3.1.3 BLANK RESULTS***

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities. A laboratory method blank sample (1114528MB) for method 6020A and a laboratory method blank sample (1114623MB) for method 7470A were run with this SDG.

No laboratory method blank detects were noted.

### ***3.1.4 LCS RECOVERY RESULTS***

The LCS serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. The LCS is fortified with each analyte of interest and analyzed with each batch of samples. The LCS accuracy performance is measured by %R.

The LCS recoveries were within acceptable recovery limits.

### ***3.1.5 MS/MSD RECOVERY RESULTS***

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The MS/MSD accuracy performance is measured by %R.

An MS/MSD was not requested with this SDG; however a MS/MSD was run on sample LL-SB-001. MS/MSD results were not available for Lead due to the large dilutions necessary on the native sample. The MSD had a Barium result biased low at 37%R; in addition, the RPD for Barium was outside QC limits at 29%RPD. Mercury was biased low in the MS at 76%R and MSD at 74%R. Barium will be flagged as J and Mercury will be flagged as UJ in sample LL-SB-001.

### ***3.1.6 GENERAL LABORATORY OBSERVATIONS***

Samples LL-SB-001 had Lead reported from a dilution, which raised the reporting limit for Lead. The lab also noted that the Lead in sample LL-SB-001 failed the serial dilution test. Therefore, Lead was flagged as J in sample LL-SB-001.

## **4.0 OVERALL ASSESSMENT OF DATA**

The analytical results meet the data quality objectives defined by the applicable method and validation guidance documentation. The analytical data is usable and acceptable as reported by the laboratory.

**ATTACHMENT**  
**SUMMARY OF VALIDATED ANALYTICAL RESULTS**  
**AND**  
**CHAIN-OF-CUSTODY**

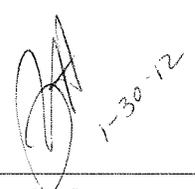
**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112329</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-SB-001</b>	Sampled:	12/20/11 09:00
Lab Sample ID:	<b>1112329-01</b>	Sampled By:	N.B., C.S.
Matrix:	Soil	Received:	12/20/11 13:50
Percent Solids:	n/a		

**TCLP Metals by EPA 1311/6000/7000 Series Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Arsenic	<0.50	0.50	5	mg/L	1	USEPA-6010C	12/23/11 09:32	KLV	1114528
<b>*Barium</b>	<b>0.42</b>	0.35	J 100	mg/L	1	USEPA-6010C	12/23/11 09:32	KLV	1114528
Cadmium	<0.050	0.050	1	mg/L	1	USEPA-6010C	12/23/11 09:32	KLV	1114528
Chromium	<0.25	0.25	5	mg/L	1	USEPA-6010C	12/23/11 09:32	KLV	1114528
<b>*Lead</b>	<b>940</b>	250	J 5	mg/L	1000	USEPA-6010C	12/23/11 10:47	KLV	1114528
*Mercury	<0.00020	0.00020	KJ 0.2	mg/L	1	USEPA-7470A	12/29/11 07:58	DSC	1114623
Selenium	<0.20	0.20	1	mg/L	1	USEPA-6010C	12/23/11 09:32	KLV	1114528
Silver	<0.050	0.050	5	mg/L	1	USEPA-6010C	12/23/11 09:32	KLV	1114528

\*See Statement of Data Qualifications



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112329</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-SB-002</b>	Sampled:	12/20/11 09:50
Lab Sample ID:	<b>1112329-02</b>	Sampled By:	N.B., C.S.
Matrix:	Soil	Received:	12/20/11 13:50
Percent Solids:	n/a		

**TCLP Metals by EPA 1311/6000/7000 Series Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Arsenic	<0.50	0.50	5	mg/L	1	USEPA-6010C	12/23/11 09:53	KLV	1114528
<b>Barium</b>	<b>0.66</b>	0.35	100	mg/L	1	USEPA-6010C	12/23/11 09:53	KLV	1114528
<b>Cadmium</b>	<b>0.064</b>	0.050	1	mg/L	1	USEPA-6010C	12/23/11 09:53	KLV	1114528
Chromium	<0.25	0.25	5	mg/L	1	USEPA-6010C	12/23/11 09:53	KLV	1114528
<b>Lead</b>	<b>13</b>	0.25	5	mg/L	1	USEPA-6010C	12/23/11 09:53	KLV	1114528
Mercury	<0.00020	0.00020	0.2	mg/L	1	USEPA-7470A	12/29/11 08:12	DSC	1114623
Selenium	<0.20	0.20	1	mg/L	1	USEPA-6010C	12/23/11 09:53	KLV	1114528
Silver	<0.050	0.050	5	mg/L	1	USEPA-6010C	12/23/11 09:53	KLV	1114528

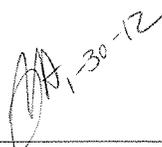


**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112329</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-SB-003</b>	Sampled:	12/20/11 10:20
Lab Sample ID:	<b>1112329-03</b>	Sampled By:	N.B., C.S.
Matrix:	Soil	Received:	12/20/11 13:50
Percent Solids:	n/a		

**TCLP Metals by EPA 1311/6000/7000 Series Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Arsenic	<0.50	0.50	5	mg/L	1	USEPA-6010C	12/23/11 09:58	KLV	1114528
<b>Barium</b>	<b>0.53</b>	0.35	100	mg/L	1	USEPA-6010C	12/23/11 09:58	KLV	1114528
Cadmium	<0.050	0.050	1	mg/L	1	USEPA-6010C	12/23/11 09:58	KLV	1114528
Chromium	<0.25	0.25	5	mg/L	1	USEPA-6010C	12/23/11 09:58	KLV	1114528
<b>Lead</b>	<b>46</b>	0.25	5	mg/L	1	USEPA-6010C	12/23/11 09:58	KLV	1114528
Mercury	<0.00020	0.00020	0.2	mg/L	1	USEPA-7470A	12/29/11 08:17	DSC	1114623
Selenium	<0.20	0.20	1	mg/L	1	USEPA-6010C	12/23/11 09:58	KLV	1114528
Silver	<0.050	0.050	5	mg/L	1	USEPA-6010C	12/23/11 09:58	KLV	1114528



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112329</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-WS-001</b>	Sampled:	12/20/11 10:45
Lab Sample ID:	<b>1112329-04</b>	Sampled By:	N.B., C.S.
Matrix:	Waste	Received:	12/20/11 13:50

**Physical/Chemical Parameters by EPA/APHA/ASTM Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
pH	7.7	0.1		pH Units	1	USEPA-9045C	12/27/11 10:23	KRK	1114679



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1112329</b>
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	<b>LL-WS-001</b>	Sampled:	12/20/11 10:45
Lab Sample ID:	<b>1112329-04</b>	Sampled By:	N.B., C.S.
Matrix:	Waste	Received:	12/20/11 13:50

**TCLP Metals by EPA 1311/6000/7000 Series Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Arsenic	<1.0	1.0	5	mg/L	1	USEPA-6010C	12/23/11 10:02	KLV	1114528
<b>Barium</b>	<b>0.75</b>	0.70	100	mg/L	1	USEPA-6010C	12/23/11 10:02	KLV	1114528
Cadmium	<0.10	0.10	1	mg/L	1	USEPA-6010C	12/23/11 10:02	KLV	1114528
Chromium	<0.50	0.50	5	mg/L	1	USEPA-6010C	12/23/11 10:02	KLV	1114528
<b>Lead</b>	<b>36</b>	0.50	5	mg/L	1	USEPA-6010C	12/23/11 10:02	KLV	1114528
Mercury	<0.00040	0.00040	0.2	mg/L	1	USEPA-7470A	12/29/11 08:32	DSC	1114623
Selenium	<0.40	0.40	1	mg/L	1	USEPA-6010C	12/23/11 10:02	KLV	1114528
Silver	<0.10	0.10	5	mg/L	1	USEPA-6010C	12/23/11 10:02	KLV	1114528

*Handwritten signature and date: 1-30-12*

### ANALYTICAL REPORT

Client:	Oneida Total Integrated Enterprises	Work Order:	1112329
Project:	Lovers Lane Lead Site, Comstock, MI	Description:	Laboratory Services
Client Sample ID:	LL-WS-001	Sampled:	12/20/11 10:45
Lab Sample ID:	1112329-04	Sampled By:	N.B., C.S.
Matrix:	Waste	Received:	12/20/11 13:50
Unit:	mg/L	Prepared:	12/26/11 By: DLV
Dilution Factor:	100	Analyzed:	12/27/11 By: JDM
QC Batch:	1114675	Analytical Batch:	1L27006

### TCLP Volatile Organics by EPA Method 1311/8260B

CAS Number	Analyte	Analytical Result	RL	Action Limit
71-43-2	Benzene	<0.10	0.10	0.5
56-23-5	Carbon Tetrachloride	<0.10	0.10	0.5
108-90-7	Chlorobenzene	<0.10	0.10	100
67-66-3	Chloroform	<0.10	0.10	6
107-06-2	1,2-Dichloroethane	<0.10	0.10	0.5
75-35-4	1,1-Dichloroethene	<0.10	0.10	0.7
78-93-3	2-Butanone (MEK)	<5.0	5.0	200
127-18-4	Tetrachloroethene	<0.10	0.10	0.7
79-01-6	Trichloroethene	<0.10	0.10	0.5
75-01-4	Vinyl Chloride	<0.10	0.10	0.2

**Surrogates:**

	% Recovery	Control Limits
<i>Dibromofluoromethane</i>	99	79-124
<i>1,2-Dichloroethane-d4</i>	91	75-128
<i>Toluene-d8</i>	99	87-113
<i>4-Bromofluorobenzene</i>	92	70-121

*BAH* 1-30-12

### ANALYTICAL REPORT

Client: <b>Oneida Total Integrated Enterprises</b>	Work Order: <b>1112329</b>
Project: Lovers Lane Lead Site, Comstock, MI	Description: Laboratory Services
Client Sample ID: <b>LL-WS-001</b>	Sampled: 12/20/11 10:45
Lab Sample ID: <b>1112329-04</b>	Sampled By: N.B., C.S.
Matrix: Waste	Received: 12/20/11 13:50
Unit: mg/L	Prepared: 12/28/11 By: JTS
Dilution Factor: 1	Analyzed: 12/29/11 By: DWJ
QC Batch: 1114531	Analytical Batch: 1L30024

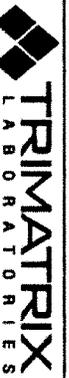
#### \*TCLP Semivolatile Organic Compounds by EPA Method 1311/8270C

CAS Number	Analyte	Analytical Result	RL	Action Limit
106-46-7	1,4-Dichlorobenzene	<0.0050	0.0050	7.5
121-14-2	2,4-Dinitrotoluene	<0.0050	0.0050	0.13
118-74-1	Hexachlorobenzene	<0.0050	0.0050	0.13
87-68-3	Hexachlorobutadiene	<0.0050	0.0050	0.5
67-72-1	Hexachloroethane	<0.0050	0.0050	3
98-95-3	Nitrobenzene	<0.0050	0.0050	2
110-86-1	Pyridine	<0.050	0.050	5
*87-86-5	Pentachlorophenol	<0.0050 <i>u.s</i>	0.0050	100
88-06-2	2,4,6-Trichlorophenol	<0.0050	0.0050	2
95-95-4	2,4,5-Trichlorophenol	<0.0050	0.0050	400
95-48-7	2-Methylphenol	<0.0050	0.0050	200
108-39-4	3+4-Methylphenol	<0.0050	0.0050	200

<i>Surrogates:</i>	<i>% Recovery</i>	<i>Control Limits</i>
<i>2-Fluorophenol</i>	<i>35</i>	<i>20-121</i>
<i>Phenol-d6</i>	<i>23</i>	<i>10-105</i>
<i>Nitrobenzene-d5</i>	<i>69</i>	<i>38-141</i>
<i>2-Fluorobiphenyl</i>	<i>65</i>	<i>41-132</i>
<i>2,4,6-Tribromophenol</i>	<i>52</i>	<i>20-142</i>
<i>o-Terphenyl</i>	<i>73</i>	<i>39-148</i>

\*See Statement of Data Qualifications

*DWJ 1-30-12*



5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512  
Phone (616) 975-4500 Fax (616) 942-7463  
www.trimatrixlabs.com

### Chain of Custody Record

Lab No. **096031A**  
Pg. **1** of **1**

*WASTE SAMPLES*

Analyses Requested

For Lab Use Only	Client Name	Project Name	Container Type (corresponds to Container Packing List)
Cart # <b>4</b>	Onelda Total Integrated Enterprises	Lovers Lane Land Site, Constock, MI	A
VOA Recd/Tray # <b>444528</b>	Address	Client Project No. / P.O. No.	A
Receipt Log No. <b>17.17</b>	100 West Monroe, Suite 300		A
Project Chemist	City, State Zip	Invoice To	A
<b>Lisa Harvey</b>	Chicago, IL 60603	<input type="radio"/> Client	A
Work Order No. <b>112379</b>	Phone/Fax: 312-220-7000 (cell 312-656-7685)	<input type="radio"/> Other (comments)	A
	Email: <b>NAEEN@OTTE.COM</b>	Contact/Report To	A
		<b>NAEEN BREX</b>	A

Schedule	Matrix Code	Sample Number	Field Sample ID	Cooler ID	Sample Date	Sample Time	Number of Containers Submitted										Total	Sample Comments
							18	16	17	17	17	17	17	17	17	17		
3		01	LL-SB-001		12/20/11	0900	X	X									1	Pregone in H <sub>2</sub> O <sub>2</sub>
3		02	LL-SB-002		12/20/11	0915	X	X									1	
3		03	LL-SB-003		12/20/11	1020	X	X									1	
A		04	LL-WS-001		12/20/11	1045	X	X	X	X							3	Revised in H <sub>2</sub> O <sub>2</sub>
			LDN-601		12/20/11	1245	X	X									1	
			Trip break (3 hrs)		12-20-11	1350											3	Do not analyze (MIL)

Sampled By (Print)	How Shipped?	Hand	Carrier	Comments
<b>NAEEN BREX</b>		X		
Sampler's Signature	Tracking No.			
<i>[Signature]</i>				
Company	1 Requisitioned By	Date	Time	2 Requisitioned By
<b>OTTE</b>	<i>[Signature]</i>	12/20/11	1350	
	3 Received by Lab By	Date	Time	
	<i>[Signature]</i>	12/20/11	1350	

Onelda Lovorslane 1211.xls

ORIGINAL - LABORATORY

COPY - FIELD/SAMPLER

12/15/2011



## MEMORANDUM

**Date:** February 1, 2012

**To:** Naren Babu, Project Manager, OTIE  
Superfund Technical Assessment and Response Team (START) for Region 5

**Prepared by:** Renea Anglin, START chemist for Region 4

**QA/QC** Keely Meadows

**Concurrence by:**

**Subject:** Data Validation for  
Lovers Lane Lead Site  
Comstock, MI  
Project TDD No. TO-01-11-11-0028

Laboratory: Trimatrix Laboratories, Grand Rapids, MI.  
Sample Delivery Group (SDG): 1201043

### 1.0 INTRODUCTION

The START chemist for Region 4 validated analytical data for 10 soil samples for Total Michigan (MI)-10 Metals. Samples were collected at the Lovers Lane Lead Site in Comstock, MI on January 5, 2012. The samples were analyzed under SDG 1201043 by Trimatrix Laboratories of Grand Rapids, MI using U.S. Environmental Protection Agency (U.S. EPA) methods 6020A and 7471A.

Laboratory data were validated using guidelines set forth in the U.S. EPA Contract Laboratory Program National Functional Guidelines (NFG) for Inorganic Data Review (EPA-540-R-10-011, January 2010), and applicable methodologies. The purpose of the chemical data quality evaluation process is to assess the usability of data for the project decision-making process.

Inorganic data validation consisted of a review of the following QC audits:

- Chain of custody and sample receipt forms review
- Sample preservation and holding time
- Blank results
- Duplicate Sample Results
- LCS recovery results
- MS/MSD recovery results

Section 2.0 of this memorandum discusses the results of inorganic data validation. Section 3.0 presents an overall assessment of the data. The attachment to this memorandum contains the laboratory reporting forms as well as START's handwritten data qualifications where warranted.

### 2.0 INORGANIC DATA VALIDATION RESULTS

The results of START's inorganic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted:

- J – The analyte was detected. The reported concentration was considered estimated.

- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives established for the project.

## **2.1 SOIL SAMPLES BY METHOD 6020A/7471A**

### **2.1.1 SAMPLE HANDLING**

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Soil samples were collected on January 5, 2012 and were received on ice.

Samples were not received at  $<6^{\circ}\text{C}$ . Sample cooler temperature was  $9.4^{\circ}\text{C}$  upon laboratory receipt. However, the cooler was hand delivered to the laboratory and the time of last sample collection was 15:05 and time for receipt of the cooler at the laboratory was 16:00. This is insufficient time for cooling of the soil samples; therefore, no further action was taken.

### **2.1.2 SAMPLE PRESERVATION AND HOLDING TIME**

Samples were analyzed within the holding time criteria. No discrepancies were noted.

### **2.1.3 BLANK RESULTS**

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities. A laboratory method blank sample (1201018MB) for method 6020A, and a laboratory method blank sample (1201097MB) for method 7470A were run with this SDG.

Zinc was detected in the method blank at 2.2 mg/kg. All samples had Zinc recoveries greater than 5x the blank contamination level ( $>11$  mg/kg). Therefore, no further action was taken.

### **2.1.4 LCS RECOVERY RESULTS**

The LCS serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. The LCS is fortified with each analyte of interest and analyzed with each batch of samples. The LCS accuracy performance is measured by %R.

The LCS recoveries were within acceptable recovery limits.

### **2.1.5 MS/MSD RECOVERY RESULTS**

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The MS/MSD accuracy performance is measured by %R.

An MS/MSD was not requested with this SDG; however a MS/MSD was run on sample LL-SB-001. MS/MSD results were not available for Zinc due to the large dilutions necessary on the native sample. The MS had a Barium result biased low at 37%R, and the MSD had a Barium recovery biased low at 55.2%. The MSD had a Copper result biased low at 2%R. The MS had a recovery for Lead biased low at 34%R, and the MSD had a recovery for Lead biased low at 0%R. The detected levels for Barium, Copper, and Lead in the

native sample were greater than 10x the spike levels and would mask the spike, so these cannot be evaluated for MS/MSD and no further action was taken.

### ***2.1.1 FIELD DUPLICATES***

Data for field duplicates were collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples.

Sample LL-SS-008 had a duplicate collected (LL-SS-009) for metals. No deficiencies were noted.

### ***2.1.2 GENERAL LABORATORY OBSERVATIONS***

Sample LL-SS-001 had Arsenic, Barium, Chromium, Copper, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 6 analytes. Sample LL-SS-002 had Barium, Chromium, Copper, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 5 analytes. Sample LL-SS-003 had Barium, Chromium, Copper, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 5 analytes. Sample LL-SS-004 had Barium, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 3 analytes. Sample LL-SS-005 had Barium, Chromium, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 4 analytes. Sample LL-SS-006 had Barium, Copper, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 4 analytes. Sample LL-SS-007 had Barium, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 3 analytes. Sample LL-SS-008 had Barium, Chromium, and Zinc reported from dilutions, which raised the reporting limit for these 3 analytes. Sample LL-SS-009 had Arsenic, Barium, Chromium, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 4 analytes. Sample LL-SS-010 had Arsenic, Barium, Chromium, Copper, Lead, and Zinc reported from dilutions, which raised the reporting limit for these 6 analytes.

## **3.0 OVERALL ASSESSMENT OF DATA**

The analytical results meet the data quality objectives defined by the applicable method and validation guidance documentation. The analytical data is usable and acceptable as reported by the laboratory.

**ATTACHMENT**  
**SUMMARY OF VALIDATED ANALYTICAL RESULTS**  
**AND**  
**CHAIN-OF-CUSTODY**

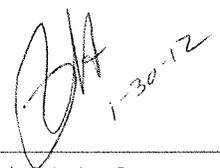
**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-001</b>	Sampled:	01/05/12 13:45
Lab Sample ID:	<b>1201043-01</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>22</b>	1.0	mg/kg dry wt.	10	USEPA-6020A	01/10/12 12:29	DSC	1201018
<b>*Barium</b>	<b>61</b>	1.0	mg/kg dry wt.	10	USEPA-6020A	01/10/12 12:29	DSC	1201018
<b>Cadmium</b>	<b>0.67</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 10:25	DSC	1201018
<b>*Chromium</b>	<b>18</b>	2.0	mg/kg dry wt.	10	USEPA-6020A	01/10/12 12:29	DSC	1201018
<b>*Copper</b>	<b>46</b>	1.0	mg/kg dry wt.	10	USEPA-6020A	01/10/12 12:29	DSC	1201018
<b>*Lead</b>	<b>47</b>	1.0	mg/kg dry wt.	10	USEPA-6020A	01/10/12 12:29	DSC	1201018
Mercury	<0.046	0.046	mg/kg dry wt.	1	USEPA-7471A	01/11/12 11:28	CKD	1201097
Selenium	<0.20	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 10:25	DSC	1201018
<b>Silver</b>	<b>0.066</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 10:25	DSC	1201018
<b>*Zinc</b>	<b>230</b>	50	mg/kg dry wt.	50	USEPA-6020A	01/10/12 12:09	DSC	1201018

\*See Statement of Data Qualifications



**ANALYTICAL REPORT**

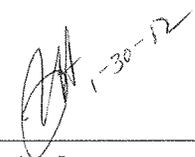
Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-002</b>	Sampled:	01/05/12 13:55
Lab Sample ID:	<b>1201043-02</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>5.9</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 10:55	DSC	1201018
<b>Barium</b>	<b>29</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 12:50	DSC	1201018
<b>Cadmium</b>	<b>0.36</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 10:55	DSC	1201018
<b>Chromium</b>	<b>13</b>	1.0	mg/kg dry wt.	5	USEPA-6020A	01/10/12 12:50	DSC	1201018
<b>Copper</b>	<b>20</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 12:50	DSC	1201018
<b>Lead</b>	<b>21</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 12:50	DSC	1201018
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	01/11/12 11:42	CKD	1201097
Selenium	<0.20	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 10:55	DSC	1201018
<b>Silver</b>	<b>0.077</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 10:55	DSC	1201018
<b>*Zinc</b>	<b>71</b>	10	mg/kg dry wt.	10	USEPA-6020A	01/10/12 12:47	DSC	1201018

\*See Statement of Data Qualifications

Page 3 of 17



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-003</b>	Sampled:	01/05/12 14:05
Lab Sample ID:	<b>1201043-03</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>2.2</b>	0.094	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:02	DSC	1201018
<b>Barium</b>	<b>35</b>	0.47	mg/kg dry wt.	5	USEPA-6020A	01/10/12 12:57	DSC	1201018
<b>Cadmium</b>	<b>0.34</b>	0.047	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:02	DSC	1201018
<b>Chromium</b>	<b>9.5</b>	0.94	mg/kg dry wt.	5	USEPA-6020A	01/10/12 12:57	DSC	1201018
<b>Copper</b>	<b>19</b>	0.47	mg/kg dry wt.	5	USEPA-6020A	01/10/12 12:57	DSC	1201018
<b>Lead</b>	<b>29</b>	0.47	mg/kg dry wt.	5	USEPA-6020A	01/10/12 12:57	DSC	1201018
Mercury	<0.047	0.047	mg/kg dry wt.	1	USEPA-7471A	01/11/12 11:47	CKD	1201097
Selenium	<0.19	0.19	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:02	DSC	1201018
<b>Silver</b>	<b>0.053</b>	0.047	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:02	DSC	1201018
<b>*Zinc</b>	<b>82</b>	24	mg/kg dry wt.	25	USEPA-6020A	01/10/12 12:54	DSC	1201018

\*See Statement of Data Qualifications



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-004</b>	Sampled:	01/05/12 14:15
Lab Sample ID:	<b>1201043-04</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>1.7</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:06	DSC	1201018
<b>Barium</b>	<b>25</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 13:52	DSC	1201018
<b>Cadmium</b>	<b>0.37</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:06	DSC	1201018
<b>Chromium</b>	<b>6.2</b>	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:06	DSC	1201018
<b>Copper</b>	<b>8.6</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:06	DSC	1201018
<b>Lead</b>	<b>51</b>	1.0	mg/kg dry wt.	10	USEPA-6020A	01/10/12 13:48	DSC	1201018
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	01/11/12 11:52	CKD	1201097
Selenium	<0.20	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:06	DSC	1201018
Silver	<0.050	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:06	DSC	1201018
<b>*Zinc</b>	<b>57</b>	10	mg/kg dry wt.	10	USEPA-6020A	01/10/12 13:48	DSC	1201018

\*See Statement of Data Qualifications



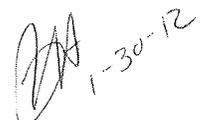
**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-005</b>	Sampled:	01/05/12 14:25
Lab Sample ID:	<b>1201043-05</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>5.8</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:10	DSC	1201018
<b>Barium</b>	<b>31</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 13:59	DSC	1201018
<b>Cadmium</b>	<b>0.26</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:10	DSC	1201018
<b>Chromium</b>	<b>11</b>	1.0	mg/kg dry wt.	5	USEPA-6020A	01/10/12 13:59	DSC	1201018
<b>Copper</b>	<b>9.3</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:10	DSC	1201018
<b>Lead</b>	<b>57</b>	1.0	mg/kg dry wt.	10	USEPA-6020A	01/10/12 13:55	DSC	1201018
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	01/11/12 11:57	CKD	1201097
Selenium	<0.20	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:10	DSC	1201018
<b>Silver</b>	<b>0.055</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:10	DSC	1201018
<b>*Zinc</b>	<b>65</b>	10	mg/kg dry wt.	10	USEPA-6020A	01/10/12 13:55	DSC	1201018

\*See Statement of Data Qualifications



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-006</b>	Sampled:	01/05/12 14:35
Lab Sample ID:	<b>1201043-06</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>1.6</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:13	DSC	1201018
<b>Barium</b>	<b>29</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:06	DSC	1201018
<b>Cadmium</b>	<b>0.20</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:13	DSC	1201018
<b>Chromium</b>	<b>6.0</b>	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:13	DSC	1201018
<b>Copper</b>	<b>14</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:06	DSC	1201018
<b>Lead</b>	<b>29</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:06	DSC	1201018
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	01/11/12 12:02	CKD	1201097
Selenium	<0.20	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:13	DSC	1201018
Silver	<0.050	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:13	DSC	1201018
<b>*Zinc</b>	<b>53</b>	10	mg/kg dry wt.	10	USEPA-6020A	01/10/12 14:02	DSC	1201018

\*See Statement of Data Qualifications



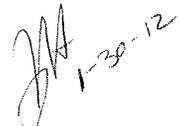
**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-007</b>	Sampled:	01/05/12 14:45
Lab Sample ID:	<b>1201043-07</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>2.4</b>	0.092	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:17	DSC	1201018
<b>Barium</b>	<b>29</b>	0.46	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:12	DSC	1201018
<b>Cadmium</b>	<b>0.10</b>	0.046	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:17	DSC	1201018
<b>Chromium</b>	<b>8.8</b>	0.18	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:17	DSC	1201018
<b>Copper</b>	<b>7.0</b>	0.092	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:17	DSC	1201018
<b>Lead</b>	<b>640</b>	9.2	mg/kg dry wt.	100	USEPA-6020A	01/10/12 14:09	DSC	1201018
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	01/11/12 12:19	CKD	1201097
Selenium	<0.18	0.18	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:17	DSC	1201018
<b>Silver</b>	<b>0.048</b>	0.046	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:17	DSC	1201018
<b>*Zinc</b>	<b>24</b>	4.6	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:12	DSC	1201018

\*See Statement of Data Qualifications



### ANALYTICAL REPORT

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-008</b>	Sampled:	01/05/12 14:55
Lab Sample ID:	<b>1201043-08</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

#### Total Metals by EPA 6000/7000 Series Methods

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>3.7</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:21	DSC	1201018
<b>Barium</b>	<b>46</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:16	DSC	1201018
<b>Cadmium</b>	<b>0.11</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:21	DSC	1201018
<b>Chromium</b>	<b>13</b>	1.0	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:16	DSC	1201018
<b>Copper</b>	<b>9.4</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:21	DSC	1201018
<b>Lead</b>	<b>8.7</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:21	DSC	1201018
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	01/11/12 12:24	CKD	1201097
Selenium	<0.20	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:21	DSC	1201018
<b>Silver</b>	<b>0.080</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:21	DSC	1201018
<b>*Zinc</b>	<b>33</b>	5.0	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:16	DSC	1201018

\*See Statement of Data Qualifications

*BA*  
1-30-12

**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-009</b>	Sampled:	01/05/12 14:55
Lab Sample ID:	<b>1201043-09</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>3.5</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:25	DSC	1201018
<b>Barium</b>	<b>43</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:19	DSC	1201018
<b>Cadmium</b>	<b>0.12</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:25	DSC	1201018
<b>Chromium</b>	<b>13</b>	1.0	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:19	DSC	1201018
<b>Copper</b>	<b>8.8</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:25	DSC	1201018
<b>Lead</b>	<b>11</b>	0.50	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:19	DSC	1201018
Mercury	<0.047	0.047	mg/kg dry wt.	1	USEPA-7471A	01/11/12 12:29	CKD	1201097
Selenium	<0.20	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:25	DSC	1201018
<b>Silver</b>	<b>0.066</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:25	DSC	1201018
<b>*Zinc</b>	<b>33</b>	5.0	mg/kg dry wt.	5	USEPA-6020A	01/10/12 14:19	DSC	1201018

\*See Statement of Data Qualifications



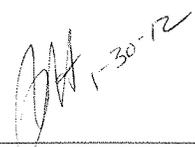
### ANALYTICAL REPORT

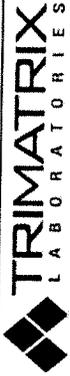
Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201043</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-010</b>	Sampled:	01/05/12 15:05
Lab Sample ID:	<b>1201043-10</b>	Sampled By:	Mr. Naren Babu
Matrix:	Soil	Received:	01/05/12 16:00
Percent Solids:	n/a		

#### Total Metals by EPA 6000/7000 Series Methods

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>490</b>	10	mg/kg dry wt.	100	USEPA-6020A	01/10/12 14:30	DSC	1201018
<b>Barium</b>	<b>99</b>	2.5	mg/kg dry wt.	25	USEPA-6020A	01/10/12 14:34	DSC	1201018
<b>Cadmium</b>	<b>0.59</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:29	DSC	1201018
<b>Chromium</b>	<b>300</b>	20	mg/kg dry wt.	100	USEPA-6020A	01/10/12 14:30	DSC	1201018
<b>Copper</b>	<b>470</b>	10	mg/kg dry wt.	100	USEPA-6020A	01/10/12 14:30	DSC	1201018
<b>Lead</b>	<b>66</b>	2.5	mg/kg dry wt.	25	USEPA-6020A	01/10/12 14:34	DSC	1201018
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	01/11/12 12:34	CKD	1201097
<b>Selenium</b>	<b>0.22</b>	0.20	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:29	DSC	1201018
<b>Silver</b>	<b>0.093</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	01/10/12 11:29	DSC	1201018
<b>*Zinc</b>	<b>240</b>	100	mg/kg dry wt.	100	USEPA-6020A	01/10/12 14:30	DSC	1201018

\*See Statement of Data Qualifications





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Grand Rapids, MI 49512  
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www.trimatrixlabs.com

### Chain of Custody Record

COC No. 09702

Pg. \_\_\_ of \_\_\_

**For Lab Use Only**  
Cart

VOA Rack/Tray

Client Name: Onhelda Total Integrated Enterprises  
Address: 100 West Monroe, Suite 300  
City, State Zip: Chicago, IL 60603  
Phone/Fax: 312-220-7000 (cell 312-656-7685)  
Email:

Project Name: Lovers Lane Lead Site, Comstock, MI  
Client Project No. / P.O. No.:  
Invoice To:  Client  Other (comments)  
Contact/Report To: R NAGY @ OTE  
NEABU @ OTE.COM / 1611

Receipt Log No. 37-11  
Project Chemist: Lisa Harvey  
Work Order No. 1201043

#### Analyses Requested

A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A
Total RCRA 8 (soil)	TCLP-RCRA 8 (soil)	TCLP-VOC	TCLP-SVOC	PCB %S (Soil)	Total RCRA 8 (waste)	TCLP-RCRA 8 (waste)	PH (waste)	Total RCRA 8 (waste)	TCLP-RCRA 8 (waste)	PH (waste)	Total RCRA 8 (waste)	TCLP-RCRA 8 (waste)	PCB %S (Soil)	Total RCRA 8 (waste)	TCLP-RCRA 8 (waste)	PH (waste)	Total RCRA 8 (waste)	TCLP-RCRA 8 (waste)	PCB %S (Soil)

Container Type (corresponds to Container Packing List):  
Number of Containers Submitted: Total Sample Comments

Field Sample ID	Sample Date	Sample Time	Matrix	Comments
1 LL-SS-001	01/05/12	1345	X S	
2 LL-SS-002		1355		
3 LL-SS-003		1405		
4 LL-SS-004		1415		
5 LL-SS-005		1425		
6 LL-SS-006		1435		
7 LL-SS-007		1445		
8 LL-SS-008		1455		
9 LL-SS-009		1455		
10 LL-SS-010		1505		

How Shipped?  Hand  Carrier

Tracking No. \_\_\_\_\_

1. Requisitioned By: [Signature] Date: 11/5/12 Time: 16:00

2. Received By: [Signature] Date: 11/5/12 Time: 16:00

3. Requisitioned By: [Signature] Date: 11/5/12 Time: 16:00

4. Received By: [Signature] Date: 11/5/12 Time: 16:00

Comments: 1-week TAT

Company: OTE

Sampler's Signature: [Signature]

Original - Laboratory

Copy - Field/Sampler





## MEMORANDUM

**Date:** February 1, 2012

**To:** Naren Babu, Project Manager, OTIE  
Superfund Technical Assessment and Response Team (START) for Region 5

**Prepared by:** Renea Anglin, START chemist for Region 4

**QA/QC** Keely Meadows

**Concurrence by:**

**Subject:** Data Validation for  
Lovers Lane Lead Site  
Comstock, MI  
Project TDD No. TO-01-11-11-0028

Laboratory: Trimatrix Laboratories, Grand Rapids, MI.  
Sample Delivery Group (SDG): 1201124

### 1.0 INTRODUCTION

The START chemist for Region 4 validated analytical data for 4 soil samples for Total Characteristic Leachate Procedure (TCLP) Lead. Samples were collected at the Lovers Lane Lead Site in Comstock MI on January 5, 2012. The samples were analyzed under SDG 1201124 by Trimatrix Laboratories of Grand Rapids, MI using U.S. Environmental Protection Agency (U.S. EPA) method 1311/6020A.

Laboratory data were validated using guidelines set forth in the U.S. EPA Contract Laboratory Program National Functional Guidelines (NFG) for Inorganic Data Review (EPA-540-R-10-011, January 2010) and applicable methodologies. The purpose of the chemical data quality evaluation process is to assess the usability of data for the project decision-making process.

Inorganic data validation consisted of a review of the following QC audits:

- Chain of custody and sample receipt forms review
- Sample preservation and holding time
- Blank results
- Duplicate Sample Results
- LCS recovery results
- MS/MSD recovery results

Section 2.0 of this memorandum discusses the results of inorganic data validation. Section 3.0 presents an overall assessment of the data. The attachment to this memorandum contains the laboratory reporting forms as well as START's handwritten data qualifications where warranted.

### 2.0 INORGANIC DATA VALIDATION RESULTS

The results of START's inorganic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted:

- J – The analyte was detected. The reported concentration was considered estimated.

- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives established for the project.

## **2.1 TCLP SAMPLES BY METHOD 1311/6020A**

### ***2.1.1 SAMPLE HANDLING***

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Soil samples were collected on January 5, 2012 and were received on ice.

Samples were not received at  $<6^{\circ}\text{C}$ . Sample cooler temperature was  $9.4^{\circ}\text{C}$  upon laboratory receipt. However, the cooler was hand delivered to the laboratory and the time of last sample collection is 15:05 and time for receipt of the cooler at the laboratory is 16:00. This is insufficient time for cooling of the soil samples; therefore no further action was taken.

### ***2.1.2 SAMPLE PRESERVATION AND HOLDING TIME***

Samples were analyzed within the holding time criteria. No discrepancies were noted.

### ***2.1.3 BLANK RESULTS***

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities. A laboratory method blank sample (12011181MB) for the method was run with this SDG.

No discrepancies were noted.

### ***2.1.4 LCS RECOVERY RESULTS***

The LCS serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. The LCS is fortified with each analyte of interest and analyzed with each batch of samples. The LCS accuracy performance is measured by %R.

The LCS recoveries were within acceptable recovery limits.

### ***2.1.5 MS/MSD RECOVERY RESULTS***

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The MS/MSD accuracy performance is measured by %R.

An MS/MSD was not requested with this SDG; however, a MS/MSD was run on sample LL-SS-007. The MS for Lead was biased low with a recovery of 61%R while the MSD for Lead was biased high at 216%R. The level in the native sample was  $>4x$  the spike level for Lead; therefore, no further action was taken. The RPD for Lead was biased high at 21%RPD; therefore, the Lead level was flagged as J in sample LL-SS-007.

### **3.0 OVERALL ASSESSMENT OF DATA**

The analytical results meet the data quality objectives defined by the applicable method and validation guidance documentation. The analytical data is usable and acceptable as reported by the laboratory.

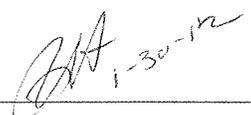
**ATTACHMENT**  
**SUMMARY OF VALIDATED ANALYTICAL RESULTS**  
**AND**  
**CHAIN-OF-CUSTODY**

**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201124</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-004</b>	Sampled:	01/05/12 14:15
Lab Sample ID:	<b>1201124-01</b>	Sampled By:	Mr. Naren Babu
Matrix:	Waste	Received:	01/05/12 16:00

**TCLP Metals by EPA 1311/6000/7000 Series Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Lead	<0.25	0.25	5	mg/L	1	USEPA-6010C	01/16/12 10:28	KLV	1201181

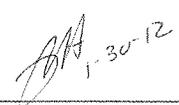


**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201124</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-005</b>	Sampled:	01/05/12 14:25
Lab Sample ID:	<b>1201124-02</b>	Sampled By:	Mr. Naren Babu
Matrix:	Waste	Received:	01/05/12 16:00

**TCLP Metals by EPA 1311/6000/7000 Series Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Lead	<0.25	0.25	5	mg/L	1	USEPA-6010C	01/16/12 10:32	KLV	1201181



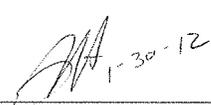
**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201124</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-007</b>	Sampled:	01/05/12 14:45
Lab Sample ID:	<b>1201124-03</b>	Sampled By:	Mr. Naren Babu
Matrix:	Waste	Received:	01/05/12 16:00

**TCLP Metals by EPA 1311/6000/7000 Series Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
*Lead	2.5 J	0.25	5	mg/L	1	USEPA-6010C	01/16/12 10:35	KLV	1201181

\*See Statement of Data Qualifications

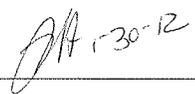


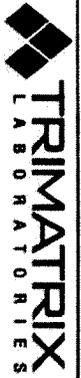
**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1201124</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-SS-010</b>	Sampled:	01/05/12 15:05
Lab Sample ID:	<b>1201124-04</b>	Sampled By:	Mr. Naren Babu
Matrix:	Waste	Received:	01/05/12 16:00

**TCLP Metals by EPA 1311/6000/7000 Series Methods**

Analyte	Analytical Result	RL	Action Limit	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Lead	<0.25	0.25	5	mg/L	1	USEPA-6010C	01/16/12 10:52	KLV	1201181





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### Chain of Custody Record

COC No. 09702

Analyses Requested Pg.      of     

For Lab Use Only	Client Name Oreida Total Integrated Enterprises	Project Name Lovers Lane Lead Site, Constock, MI
Client	Address 100 West Monroe, Suite 300	Client Project No. / P.O. No.
VOA Rack/Trey	City, State Zip Chicago, IL 60603	Invoice To <input checked="" type="radio"/> Client <input type="radio"/> Other (comments)
Receipt Log No. 37-11	Project Chemist Lisa Harvey 1201124	Contact/Report To NSARGU@ONE.com
Work Order No. 1201043	Phone/Fax 312-220-7000 (cell 312-656-7885)	Container Type (corresponds to Container Packing List)

Schedule	Matrix Code	Sample Number	Field Sample ID	Cooler ID	Sample Date	Sample Time	Matrix	Number of Containers Submitted	Total	Sample Comments
		01	LL-SS-001		01/05/12	1345	S	17	17	
		02	LL-SS-002			1355	S	17	17	
		03	LL-SS-003			1405	S	17	17	
		04	LL-SS-004			1415	S	17	17	
		05	LL-SS-005			1425	S	17	17	
		06	LL-SS-006			1435	S	17	17	
		07	LL-SS-007			1445	S	17	17	
		08	LL-SS-008			1455	S	17	17	
		09	LL-SS-009			1455	S	17	17	
		10	LL-SS-010			1505	S	17	17	

Sampled By (print) NAEM BARU How Shipped? Hand Carrier ✓

Sampler's Signature [Signature] Tracking No. \_\_\_\_\_

Company ONE

1. Requisitioned By [Signature] Date 1/5/12 Time 16:00

2. Received By [Signature] Date 1/5/12 Time 16:00

3. Requisitioned By [Signature] Date 1/5/12 Time 16:00

4. Received For Lab By [Signature] Date 1/5/12 Time 16:00

Onaida Lower Lane 1211.xls

ORIGINAL - LABORATORY

COPY - FIELD/SAMPLER

12/15/2011



## MEMORANDUM

**Date:** February 29, 2012

**To:** Naren Babu, Project Manager, OTIE  
Superfund Technical Assessment and Response Team (START) for region 5

**Prepared by:** Naren Babu, Project Manager, OTIE  
Superfund Technical Assessment and Response Team (START) for region 5

**QA/QC  
Concurrence by:**

**Subject:** Data Validation for  
Lovers Lane Lead Site  
Comstock Park, Michigan  
Project TDD No. TO-01-11-11-0028

Laboratory: TRIMATRIX Laboratories, Grand Rapids, MI.  
Work Order No. 1202164  
Analyses of Five Soil Samples for total Michigan-ten metals.

### 1.0 INTRODUCTION

The START for region 5 validated Metals for five soil samples. Samples were collected at the Lovers Lane Lead Site, Comstock Park, MI on February 9<sup>th</sup>, 2012. The samples were analyzed under Work Order number 1202164 by TRIMATRIX Laboratories – Grand Rapids, MI using U.S. Environmental Protection Agency (U.S. EPA) method 6000/7000.

Laboratory data were validated using guidelines set forth in the U.S. EPA Contract Laboratory Program National Functional Guidelines Inorganic Data Review (EPA 540/R-94/013, February, 1994) and applicable methodologies. The purpose of the chemical data quality evaluation process is to assess the usability of data for the project decision-making process.

Inorganic data validation consisted of a review of the following QC audits:

- Chain of custody and sample receipt forms review
- Sample preservation and holding time
- Blank results
- Laboratory Control Sample (LCS) recovery results
- Matrix spike and Matrix Spike Duplicate (MS/MSD) recovery results

Section 2.0 of this memorandum discusses the results of inorganic data validation. Section 3.0 presents an overall assessment of the data. The attachment to this memorandum contains the laboratory reporting forms as well as START's handwritten data qualifications where warranted.

## **2.0 INORGANIC DATA VALIDATION RESULTS**

The Results of START's inorganic data validation are summarized below by QC audit reviewed. The data qualifiers listed below were applied to sample analytical results where warranted (see attachment):

- J – The analyte was detected. The reported concentration was considered estimated.
- U – The analyte was not detected.
- UJ – The analyte was not detected. The reporting limit was considered estimated.

After the START project staff received the data packages, they were inventoried for completeness and then reviewed according to matrix-specific protocols and data quality objectives established for the project.

### **2.1 SOIL SAMPLES BY METHOD 6000/7000**

#### ***2.1.1 SAMPLE HANDLING***

Chain of custody documentation and sample receipt forms were reviewed to ensure requested analyses were performed and that samples arrived at the laboratory intact. Soil samples were received intact by the laboratory. No discrepancies were noted.

#### ***2.1.2 SAMPLE PRESERVATION AND HOLDING TIME***

Samples were analyzed in less than one week, well within the holding time limit of six months. No discrepancies were noted.

#### ***2.1.3 BLANK RESULTS***

The assessment of blank analysis results is to determine the existence and magnitude of contamination resulting from laboratory and/or field activities. A laboratory method blank sample for method 6020A and method 7471A were run with this SDG.

No blank detects were noted above the Practical Quantitation Limit (PQL).

#### ***2.1.4 LCS RECOVERY RESULTS***

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Laboratory Control Samples (LCS) were fortified with each analyte of interest and analyzed with each batch of samples. The LCS accuracy performance is measured by Percent Recovery (%R).

LCS recoveries ranged from 80% to 120%. No discrepancies were noted.

#### ***2.1.5 MS/MSD RECOVERY RESULTS***

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The MS/MSD accuracy performance is measured by Percent Recovery (%R).

Data Validation for  
Lovers Lane Lead Site  
Analytical TDD No. TO-01-11-11-0028  
Project TDD No. TO-01-11-11-0028  
Page 3

The MS/MSD results were within the limits.

### **3.0 OVERALL ASSESSMENT OF DATA**

The analytical results meet the data quality objectives defined by the applicable method and validation guidance documentation. The analytical data is usable and acceptable as reported by the laboratory.

**ATTACHEMENT**  
**SUMMARY OF ANALYTICAL RESULTS**  
**AND**  
**CHAIN-OF-CUSTODY**



**ANALYTICAL REPORT**

Client: **Oneida Total Integrated Enterprises**      Work Order: **1202164**  
Project: **Lovers Lane Lead Site, Michigan**      Description: **Laboratory Services**  
Client Sample ID: **LL-21**      Sampled: **02/09/12 10:00**  
Lab Sample ID: **1202164-01**      Sampled By: **C. Schulz**  
Matrix: **Soil**      Received: **02/09/12 13:30**  
Percent Solids: **n/a**

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
Arsenic	2.8	0.10	mg/kg dry wt.	1	USEPA-6020A	02/13/12 12:50	MSM	1202208
Barium	27	0.50	mg/kg dry wt.	5	USEPA-6020A	02/13/12 13:41	MSM	1202208
*Cadmium	0.13	0.050	mg/kg dry wt.	1	USEPA-6020A	02/13/12 12:50	MSM	1202208
Chromium	8.8	1.0	mg/kg dry wt.	5	USEPA-6020A	02/13/12 13:41	MSM	1202208
Copper	5.1	0.10	mg/kg dry wt.	1	USEPA-6020A	02/13/12 12:50	MSM	1202208
*Lead	1000	20	mg/kg dry wt.	200	USEPA-6020A	02/13/12 13:37	MSM	1202208
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	02/13/12 11:42	DSC	1202211
*Selenium	0.23	0.20	mg/kg dry wt.	1	USEPA-6020A	02/13/12 12:50	MSM	1202208
*Silver	<0.050	0.050	mg/kg dry wt.	1	USEPA-6020A	02/13/12 12:50	MSM	1202208
Zinc	24	5.0	mg/kg dry wt.	5	USEPA-6020A	02/13/12 13:41	MSM	1202208

*MB/  
2/29/12*

\*See Statement of Data Qualifications



### ANALYTICAL REPORT

Client: **Oneida Total Integrated Enterprises**      Work Order: **1202164**  
Project: **Lovers Lane Lead Site, Michigan**      Description: **Laboratory Services**  
Client Sample ID: **LL-22**      Sampled: **02/09/12 10:20**  
Lab Sample ID: **1202164-02**      Sampled By: **C. Schulz**  
Matrix: **Soil**      Received: **02/09/12 13:30**  
Percent Solids: **n/a**

#### Total Metals by EPA 6000/7000 Series Methods

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>7.2</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:00	MSM	1202208
<b>Barium</b>	<b>51</b>	1.0	mg/kg dry wt.	10	USEPA-6020A	02/13/12 13:49	MSM	1202208
<b>Cadmium</b>	<b>0.24</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:00	MSM	1202208
<b>Chromium</b>	<b>12</b>	1.0	mg/kg dry wt.	5	USEPA-6020A	02/13/12 13:55	MSM	1202208
<b>Copper</b>	<b>7.1</b>	0.10	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:00	MSM	1202208
<b>Lead</b>	<b>7100</b>	100	mg/kg dry wt.	1000	USEPA-6020A	02/13/12 13:48	MSM	1202208
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	02/13/12 11:56	DSC	1202211
<b>Selenium</b>	<b>0.34</b>	0.20	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:00	MSM	1202208
<b>Silver</b>	<b>0.079</b>	0.050	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:00	MSM	1202208
<b>Zinc</b>	<b>35</b>	5.0	mg/kg dry wt.	5	USEPA-6020A	02/13/12 13:55	MSM	1202208

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2/29/12



**ANALYTICAL REPORT**

Client: **Oneida Total Integrated Enterprises** Work Order: **1202164**  
Project: Lovers Lane Lead Site, Michigan Description: Laboratory Services  
Client Sample ID: **LL-23** Sampled: 02/09/12 10:30  
Lab Sample ID: **1202164-03** Sampled By: C. Schulz  
Matrix: Soil Received: 02/09/12 13:30  
Percent Solids: n/a

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>160</b>	2.3	mg/kg dry wt.	25	USEPA-6020A	02/13/12 13:56	MSM	1202208
<b>Barium</b>	<b>60</b>	0.93	mg/kg dry wt.	10	USEPA-6020A	02/13/12 13:58	MSM	1202208
<b>Cadmium</b>	<b>0.53</b>	0.047	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:01	MSM	1202208
<b>Chromium</b>	<b>96</b>	4.7	mg/kg dry wt.	25	USEPA-6020A	02/13/12 13:56	MSM	1202208
<b>Copper</b>	<b>150</b>	2.3	mg/kg dry wt.	25	USEPA-6020A	02/13/12 13:56	MSM	1202208
<b>Lead</b>	<b>45</b>	0.93	mg/kg dry wt.	10	USEPA-6020A	02/13/12 13:58	MSM	1202208
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	02/13/12 12:01	DSC	1202211
<b>Selenium</b>	<b>0.29</b>	0.19	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:01	MSM	1202208
Silver	<0.047	0.047	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:01	MSM	1202208
<b>Zinc</b>	<b>170</b>	23	mg/kg dry wt.	25	USEPA-6020A	02/13/12 13:56	MSM	1202208

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*2/29/12*



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1202164</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-24</b>	Sampled:	02/09/12 10:40
Lab Sample ID:	<b>1202164-04</b>	Sampled By:	C. Schulz
Matrix:	Soil	Received:	02/09/12 13:30
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>5.2</b>	0.093	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:03	MSM	1202208
<b>Barium</b>	<b>71</b>	0.93	mg/kg dry wt.	10	USEPA-6020A	02/13/12 14:00	MSM	1202208
<b>Cadmium</b>	<b>0.27</b>	0.046	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:03	MSM	1202208
<b>Chromium</b>	<b>14</b>	0.93	mg/kg dry wt.	5	USEPA-6020A	02/13/12 14:01	MSM	1202208
<b>Copper</b>	<b>7.3</b>	0.093	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:03	MSM	1202208
<b>Lead</b>	<b>3600</b>	46	mg/kg dry wt.	500	USEPA-6020A	02/13/12 13:59	MSM	1202208
<b>Mercury</b>	<b>0.053</b>	0.050	mg/kg dry wt.	1	USEPA-7471A	02/13/12 12:06	DSC	1202211
<b>Selenium</b>	<b>0.40</b>	0.19	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:03	MSM	1202208
<b>Silver</b>	<b>0.052</b>	0.046	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:03	MSM	1202208
<b>Zinc</b>	<b>39</b>	4.6	mg/kg dry wt.	5	USEPA-6020A	02/13/12 14:01	MSM	1202208

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*2/29/12*



**ANALYTICAL REPORT**

Client:	<b>Oneida Total Integrated Enterprises</b>	Work Order:	<b>1202164</b>
Project:	Lovers Lane Lead Site, Michigan	Description:	Laboratory Services
Client Sample ID:	<b>LL-25</b>	Sampled:	02/09/12 11:00
Lab Sample ID:	<b>1202164-05</b>	Sampled By:	C. Schulz
Matrix:	Soil	Received:	02/09/12 13:30
Percent Solids:	n/a		

**Total Metals by EPA 6000/7000 Series Methods**

Analyte	Analytical Result	RL	Unit	Dilution Factor	Method	Date Time Analyzed	By	QC Batch
<b>Arsenic</b>	<b>3.6</b>	0.094	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:14	MSM	1202208
<b>Barium</b>	<b>35</b>	0.47	mg/kg dry wt.	5	USEPA-6020A	02/13/12 14:04	MSM	1202208
<b>Cadmium</b>	<b>0.11</b>	0.047	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:14	MSM	1202208
<b>Chromium</b>	<b>14</b>	0.94	mg/kg dry wt.	5	USEPA-6020A	02/13/12 14:04	MSM	1202208
<b>Copper</b>	<b>7.6</b>	0.094	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:14	MSM	1202208
<b>Lead</b>	<b>86</b>	2.4	mg/kg dry wt.	25	USEPA-6020A	02/13/12 14:03	MSM	1202208
Mercury	<0.050	0.050	mg/kg dry wt.	1	USEPA-7471A	02/13/12 12:11	DSC	1202211
<b>Selenium</b>	<b>0.23</b>	0.19	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:14	MSM	1202208
Silver	<0.047	0.047	mg/kg dry wt.	1	USEPA-6020A	02/13/12 13:14	MSM	1202208
<b>Zinc</b>	<b>28</b>	4.7	mg/kg dry wt.	5	USEPA-6020A	02/13/12 14:04	MSM	1202208

*MS*  
*2/29/12*

**TRIMATRIX LABORATORIES**  
5560 Corporate Exchange Court SE  
Grand Rapids, MI 49512  
Phone (616) 975-4500 Fax (616) 942-7463  
www.trimatrixlabs.com

**Chain of Custody Record** COC No. **09893**  
Pg. **1** of **1**

**For Lab Use Only**

Client Name: **ONCIDA TOTAL INTERESTED ENTERPRISES (SITE)**  
 Address: **100 W MONROE, SUITE 300**  
 City, State Zip: **CHICAGO, IL 60603**  
 Phone/Fax: **(312) 465-1050 / (312) 220-7050**  
 Email: **ENR@ENR.COM**

Project Name: **LOWES LANE LEAD SITE, COMSDA PERK MI**  
 Client Project No. / P.O. No.:  
 Invoice To:  Client  Other (comments)

Work Order No.: **1303164**  
 Project Chemist:  
 Phone/Fax: **(312) 465-1050 / (312) 220-7050**  
 Email: **ENR@ENR.COM**

Schedule	Matrix Code	Sample Number	Field Sample ID	Cooler ID	Sample Date	Sample Time	C O M P O S I T I O N	Matrix	Number of Containers Submitted	Total	Sample Comments
		01	LL-21		2/9/12	1000	X	S	1	1	40ZGIRSS-PR
		02	LL-22		2/9/12	1020	X	S	1	1	
		03	LL-23		2/9/12	1030	X	S	1	1	
		04	LL-24		2/9/12	1040	X	S	1	1	
		05	LL-25		2/9/12	1100	X	S	1	1	
										0	
										0	
										0	
										0	
										0	
										0	
										0	

Metals (TOTAL):  
 Container Type (corresponds to Container Packing List):  
 Number of Containers Submitted:  
 Total: 5

Preservatives:  
 A NONE pH-7  
 B HNO<sub>3</sub> pH-2  
 C H<sub>2</sub>SO<sub>4</sub> pH-2  
 D 1+1 HCl pH-2  
 E NaOH pH-12  
 F ZnAcOH pH-9  
 G MeOH  
 H Other (note below)

How Shipped?  Tracking No.  Hand  Carrier   
 1. Received By: *[Signature]* Date: 2/9/12 Time: 1330  
 2. Received By: *[Signature]* Date: 2/9/12 Time: 1330  
 3. Received By: *[Signature]* Date: 2/9/12 Time: 1330

Comments:  
 STANDARD TEST SHOULD BE MICHIGAN - TEN METALS FOR ALL SAMPLES. THINDEST \*  
 \*NOTE THE ANALYSES

Sampled By (print): **CHARV SCHUIZ**  
 Sampler's Signature: *[Signature]*  
 Company: **OTIE**