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BARR

Technical Memorandum

To: Alex Smith, Enbridge Energy
From: Ryan Erickson and Sam Schoenmann
Subject: Manifold 224 Contaminated Soil Response
Date: 8/18/2016
Project: 49161092.04

This memorandum summarizes the field screening, analytical sampling, and waste management activities conducted by Barr Engineering (Barr) at the request of Enbridge Energy (Enbridge) in response to the discovery of crude oil contaminated soil near a Manifold 224 valve at the Enbridge Superior Terminal in Superior, Wiscosnin on June 22, 2016 (Figure 1).

Background

On June 22, 2016, Enbridge Pipe Line Maintenance (PLM) personnel observed darkly-stained soil with a hydrocarbon odor beneath the Manifold 224 Gate Valve 224-BSV-3531 (Photos 1 and 2; Figure 2). PLM personnel responded to the site to determine whether there was an active release at this location and to initiate remedial actions. No active release was identified based on field observations; however, the surficial contamination was indicative of a small, unreported valve release that had occurred at this location. PLM conducted local infrastructure maintenance to prevent future product releases and initiated additional remedial actions to clean-up the contaminated soil. Enbridge Environment was notified about the identified contamination and requested that Barr assist with the following activities:

- assess and document the environmental site conditions during the response actions and after the completion of remedial activities,
- assist with the coordination of the off-site management of contaminated soil, and
- prepare a memorandum summarizing the release response activities and the site environmental conditions upon the completion of cleanup activities.

Field Activities

Barr was onsite on June 22-24, 2016, to assess and document the environmental site conditions and the remediation activities, and to assist with the waste management coordination.

Excavation of soil with observable contamination was accomplished with hand tools and a hydro-vacuum (hydrovac) truck (Photos 3, 4, and 5). The remedial excavation was completed on June 23, 2016 and Barr documented the final site conditions by field-screening soil samples from the excavation sidewalls and base. Field-screening samples were tested for the presence of organic vapors using a 10.6eV photoionization detector (PID) and were inspected for the presence of other potential indicators of hydrocarbon impacts such as odor, discoloration and sheen. The PID readings and physical observations were documented on site investigation field sampling and screening logs (Attachment A). Soil was classified as contaminated if PID headspace readings were greater than 10 parts per million (ppm), or if other physical observations of oil impacts were observed, as outlined in the pending WDNR *Enbridge Superior Terminal Site Investigation and Response Action Plan* (SI/RAP) (2014).

Barr collected analytical soil samples *Manifold-224-S-1* and *Manifold-224-B-1* from the sidewall and base, respectively, of the final excavation extents to document the condition of the soil following remedial excavation activities (Figure 2). The samples were submitted to ALS Environmental in Holland, Michigan and analyzed for petroleum volatile organic compounds (PVOC) and naphthalene. Analyte concentrations were compared to the WDNR Industrial Direct Contact Residual Concentration Limits (RCLs), WDNR Groundwater RCLs, and Cumulative Hazard Index Criteria. Analytical results are summarized below in Table 1 and the laboratory report is provided in Attachment B.

Excavated contaminated soil was transported to the Superior Terminal Soil Management Area (SMA) contaminated-soil staging area where it was stockpiled until off-site disposal could be arranged. Waste characterization sample *Manifold 224 Stockpile-1* was collected from the contaminated stockpile and submitted to ALS Environmental laboratory for analysis as described in the *Waste Disposal Coordination* section below.

Results

The final remedial excavation was approximately 12 feet long by 12 feet wide by 5 feet deep (Photos 4 and 5; Figure 2; Attachment A). Soil within the excavation sidewalls consisted primarily of sand fill material. The ground surface below Manifold 224 and surrounding utilities consisted of gravel fill. Groundwater was present in the excavation at approximately 5 feet below ground surface (bgs).

Barr collected eight field screening soil samples from the final excavation extents (Attachment A). The PID headspace readings ranged from 2.9 ppm to 8.0 ppm and no other evidence of residual hydrocarbon contamination was observed. No hydrocarbon sheen was observed on the surface of the water within the excavation. Barr collected analytical sidewall sample *Manifold-224-S-1* (2 feet bgs) and bottom sample *Manifold-224-B-1* (5 feet bgs) from the final excavation extents.

Analyte concentrations in the direct contact zone sample *Manifold-224-S-1* were below laboratory method detection limits (MDL), below the WDNR Industrial Direct Contact RCL's, below WDNR Groundwater RCL's, and passed the Cumulative Hazard Index criteria (Table 1). Concentrations of benzene, toluene, ethylbenzene, and xylenes (BTEX) were detected in *Manifold-224-B-1* but were below the WDNR Industrial Direct Contact RCL's, below WDNR Groundwater RCL's, and passed the Cumulative Hazard Index criteria, with the exception of benzene (0.1 mg/kg), which exceeded the WDNR Groundwater RCL criteria (0.0051 mg/kg).

Remedial excavation activities were concluded based on field observations and field screening results and the excavation was backfilled with clean fill upon completion of the pipeline maintenance activities.

Table 1 Confirmation Soil Sample Analytical Results *(all analyte concentrations in mg/kg)*

Sample ID	Sample Date	Sample Depth (feet)	1,2,4-Trimethyl benzene	1,3,5-Trimethyl benzene	Benzene	Ethyl benzene	Toluene	Xylenes	Naphthalene
WDNR Groundwater RCLs			*1.3793*	*1.3793*	***0.0051***	*0.785*	*0.5536*	*1.97*	*0.3294*
WDNR Industrial Direct Contact RCLs			*219*	*182*	*7.41*	*37*	*818*	*258*	*26*
Manifold-224-S-1	6/23/2016	2	<0.0066	<0.014	<0.007	<0.008	<0.011	<0.025	<0.0056
Manifold-224-B-1	6/23/2016	5	<0.0085	<0.019	**0.100**	0.020	0.160	0.083	<0.0072

Notes:
BOLD = WDNR Groundwater RCL exceedance
The laboratory report is included in Attachment B.

Waste Disposal Coordination

Barr collected analytical waste characterization soil sample *Manifold 224 Stockpile-1* from the contaminated soil stockpile at the SMA (Photo 6) for laboratory analysis at ALS Environmental. The sample was analyzed for diesel range organics (DRO) and BTEX. The laboratory report was submitted to Vonco V landfill in Duluth, Minnesota with a profile amendment request and was accepted under waste profile #16-065-I. A total of 44.33 tons of contaminated soil were hauled to the landfill on July 19, 2016. The waste profile documents, the waste characterization laboratory report, and the landfill summary report are included in Attachment C.

Conclusions

Crude oil contaminated soil was encountered beneath a Manifold 224 valve at the Superior Terminal. All contaminated soil that was excavated from the site has been disposed of at an off-site landfill facility. Residual contamination was not identified in the final excavation extents through field screening. Confirmation soil sample *Manifold-224-S-1* was collected from the direct contact zone and all analyte concentrations were below laboratory MDL's. Confirmation sample *Manifold-224-B-1* was collected from below the direct contact zone and had a benzene concentration that exceeded the WDNR Groundwater RCL; however, no site-specific groundwater monitoring is required because of the facility-wide groundwater monitoring program that is conducted at the Superior Terminal as part of the hydrogeologic performance standard established in the approved *SI/RAP* (2014). Additional excavation at this location was limited by the presence of pipeline infrastructure. Enbridge will monitor the condition of the site and, if new evidence of contamination is identified, it will be reported and managed appropriately.

Because residual analyte concentrations are below WDNR direct contact RCL's, Barr believes that further remedial action at the site will not be required by the WDNR. Barr recommends that the site be added to the Terminal-wide GIS registry.

Attachments:

Site Photos	1 through 6
Figure 1	Site Location
Figure 2	Site Layout
Attachment A	Site Investigation Field Sampling and Screening Logs
Attachment B	ALS Laboratory Report for Excavation Soil Samples
Attachment C	Waste Disposal Documentation

Site Photos

Photo 1

Photo 2

Photo 1: The stained gravel beneath Manifold 224 Gate Valve 224-BSV-3531. Photo taken facing northeast on June 22, 2016 prior to the remedial excavation.
Photo 2: Western corner of Manifold 224 where the contaminated soil was identified. Gate Valve 224-BSV-3531 is on the left side of the photo. Photo taken facing northeast on June 24, 2016 after the remedial excavation was backfilled.

Photo 3

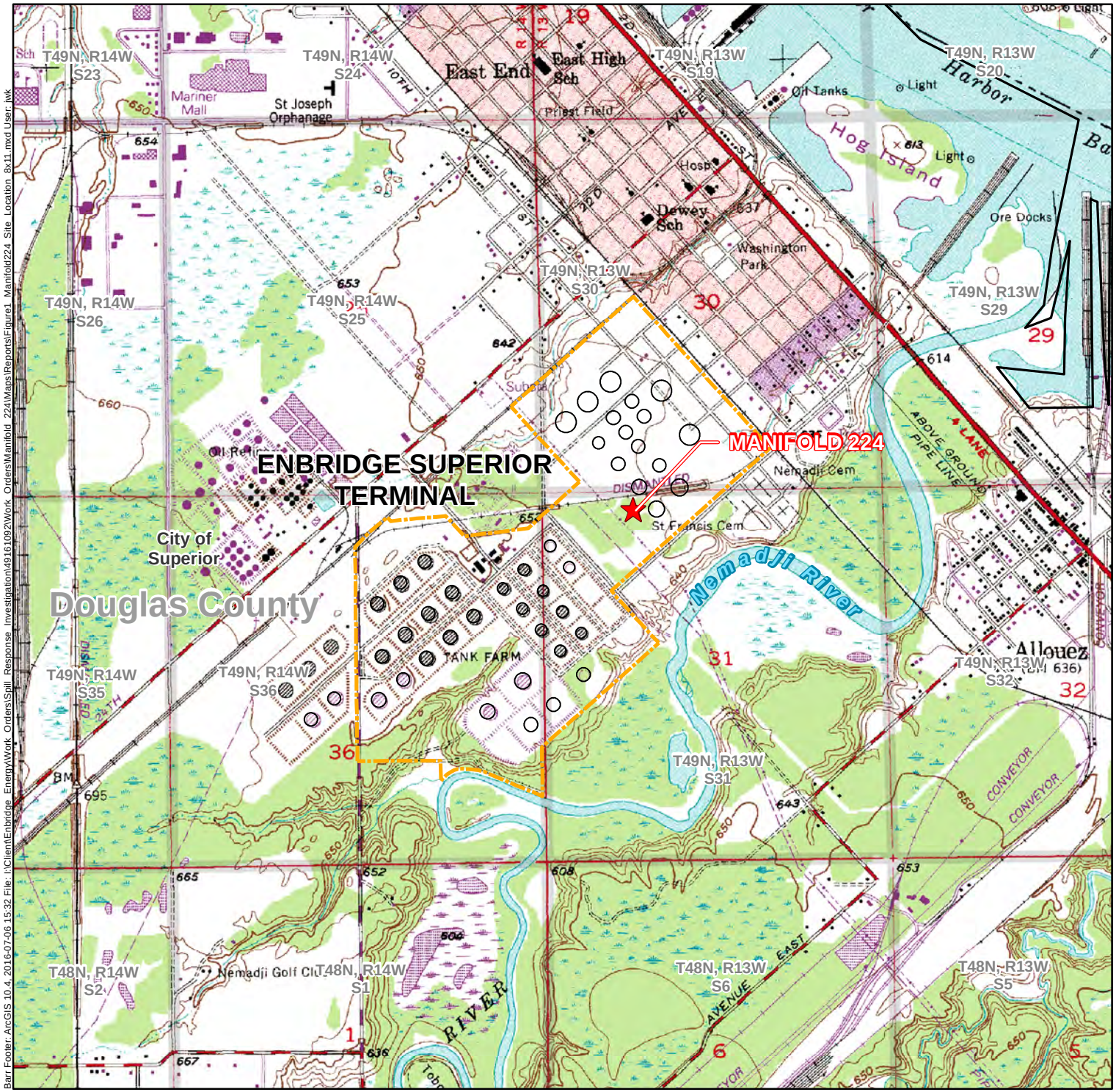
Photo 4

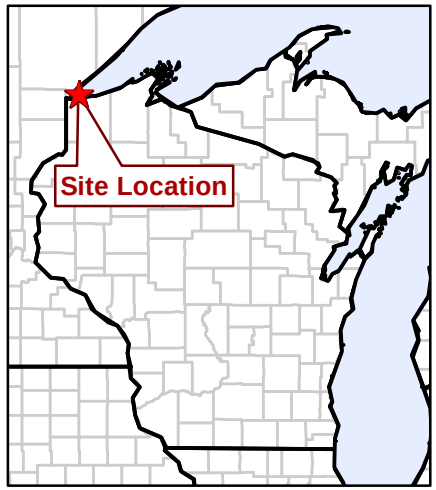
Photo 3: Initial remedial excavation. Contaminated soil was initially stockpiled in a purple tub (bottom right corner of photo). Photo taken facing northeast on June 22, 2016.
Photo 4: Expanded remedial excavation. Photo taken facing north on June 23, 2016.

Photo 5

Photo 6

Photo 5: Manifold 224 remedial excavation. Photo taken on June 23, 2016, facing south.
Photo 6: Manifold 224 contaminated soil in the Superior Terminal Soil Management Area. Photo taken on June 23, 2016.





Site Location

Terminal Property Boundary

N

0 2,000 4,000

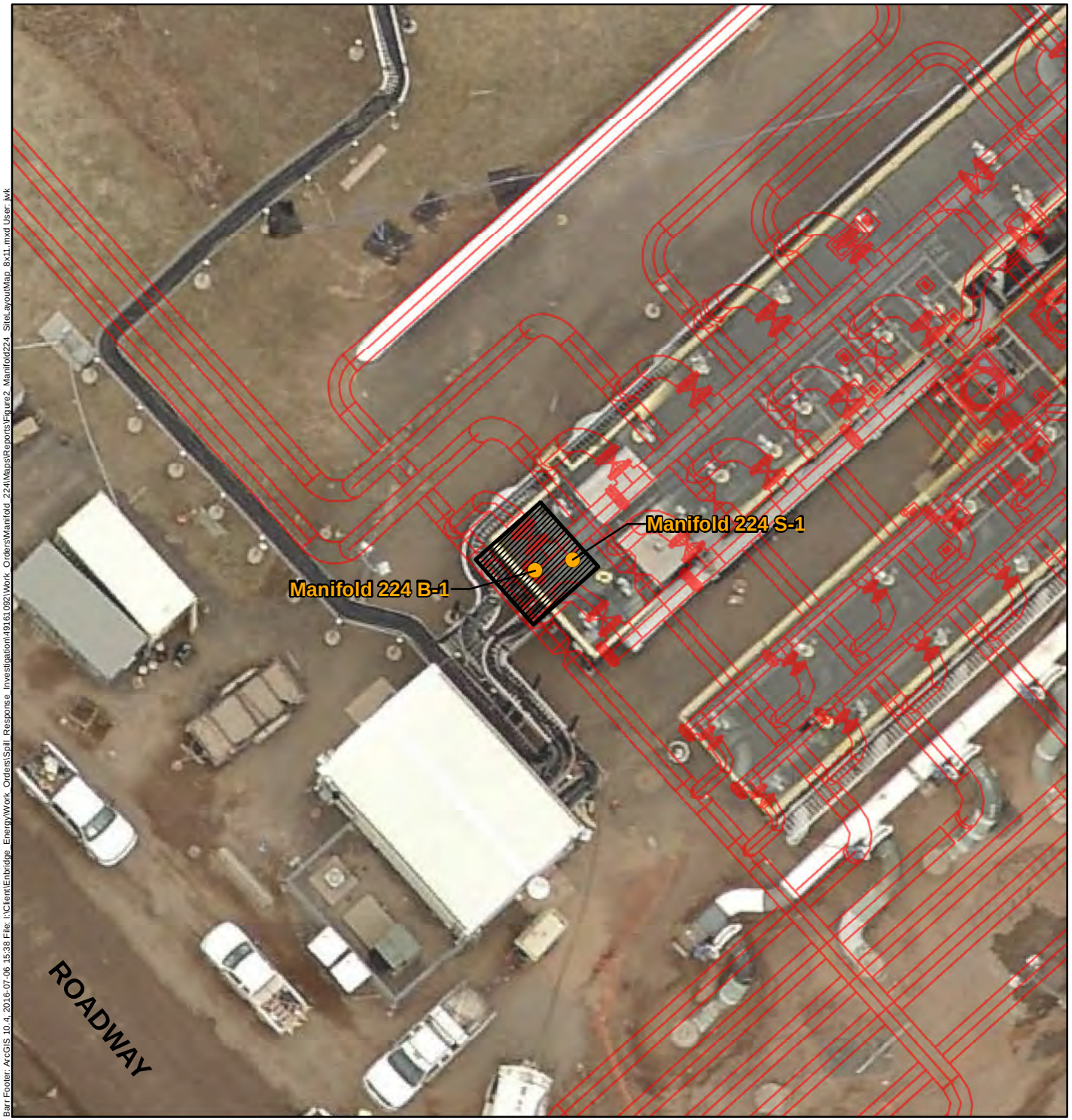
Feet

1 Inch = 2,000 Feet

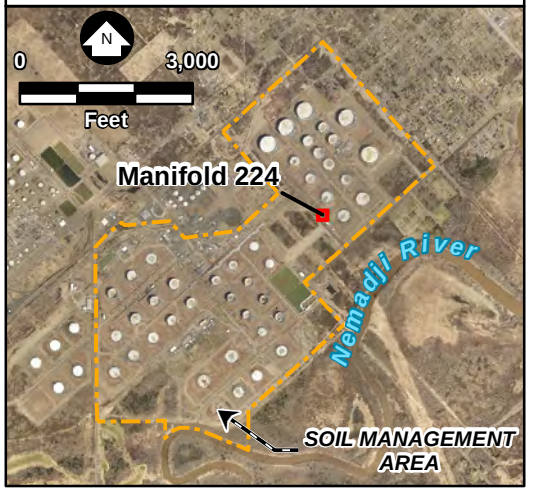
Figure 1

SITE LOCATION
MANIFOLD 224 RESPONSE
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE



ENBRIDGE SUPERIOR TERMINAL



- Analytical Sample Locations
- Excavation Extents
- Pipeline Infrastructure
- Terminal Property Boundary

N

0 20 40

Feet
1 Inch = 20 Feet

Douglas County Imagery Circa 2016

Figure 2

SITE LAYOUT
MANIFOLD 224 RESPONSE
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE

Attachment A:

Site Investigation Field Sampling and Screening Logs

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Location: *Milepost or Facility* Manifold 224 Release Excavation

Equipment used: PID *-ionization detector with* 10.6 *eV lamp*

Background Headspace: 0.1 ppm

0 gas = 0.0 span = 100.0

Post cal. span = 95.6

Date: 6/23/16

Sampler: Jet / MJP

Calibration Time: 13:30

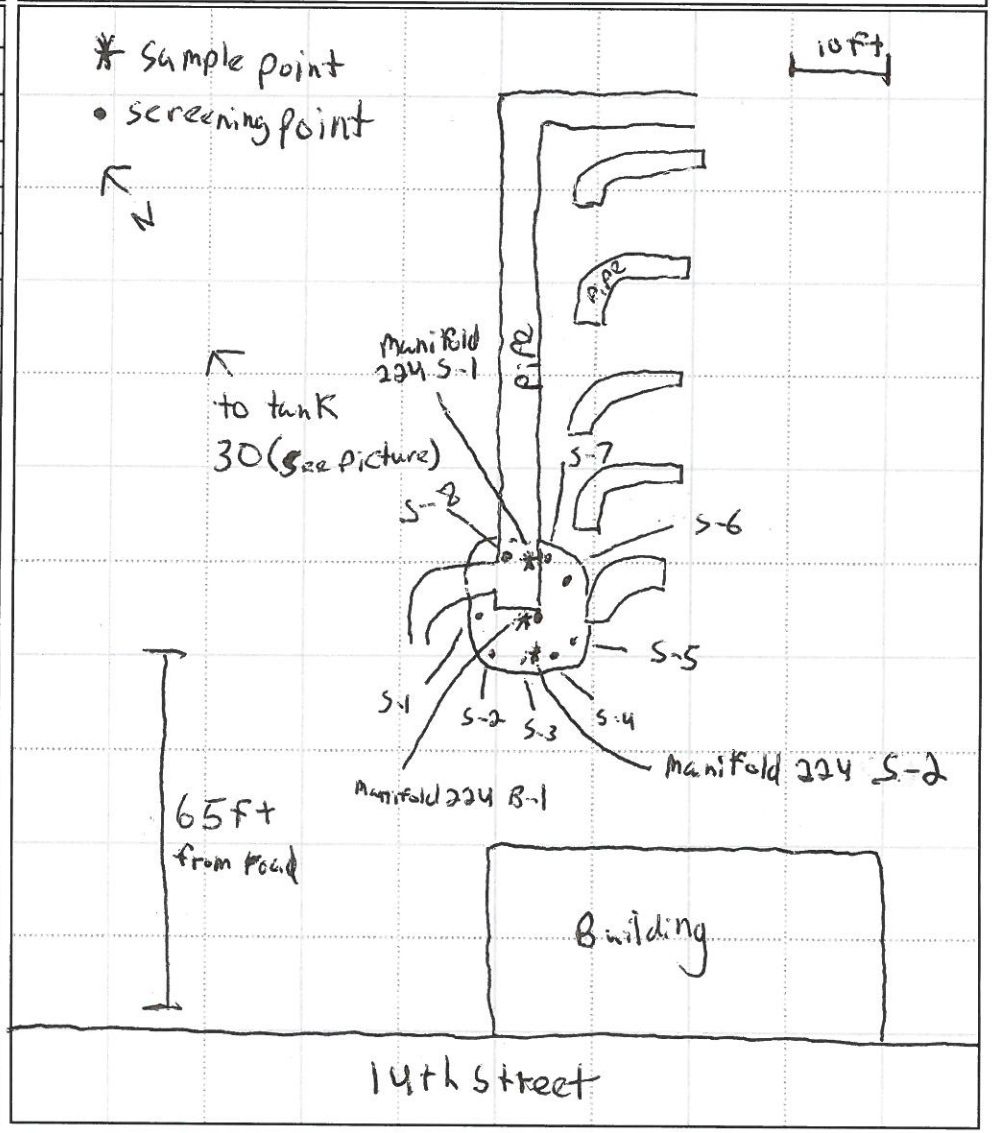
Sample Nomenclature *(Location - sample type - #):* _____

Soil Sample Types: **R** = *Removed Sample* ; **S** = *Sidewall Sample* ; **B** = *Bottom Sample* ; **Stockpile** = *Stockpile Sample*

Sample ID	Depth (FT)	Time (military)	Soil Type (USCS)	Color/ Discolor	Odor/ Sheen	Headspace Reading (ppm)
Example: TK99-S-1	4	16:30	CL	Reddish brown	Petroleum/ Rainbow	275
S-1	2	13:38	Sand	Reddish Brown	N/N	5.4
S-2						2.9
S-3						8.0
S-4		↓				5.2
S-5		13:42				6.1
S-6						6.2
S-7						6.1
S-8	↓	↓	↓	↓	↓	5.5
Manifold 224 B-1	5		Sand	Reddish Brown	N/N	14.3
Manifold 224 S-1	2	13:50				
Manifold 224 S-2	2		↓	↓	↓	Not analyzed

analyzed

SITE SKETCH: *north is up; excavation extents & depths, impacted areas, sample locations, borings, wells, structures, utilities, natural features...* ***1 inch/grid =*** 20 ***FEET***



*See page 2 for Stockpile screening + sample information

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Location: *Milepost or Facility* Manifold 224 Release Stockpile

Equipment used: PID *-ionization detector with* 10.6 *eV lamp* Background Headspace: 1.4 ppm

Sample Nomenclature *(Location - sample type - #):* _____

Soil Sample Types: **R** = *Removed Sample* ; **S** = *Sidewall Sample* ; **B** = *Bottom Sample* ; **Stockpile** = *Stockpile Sample*

Date: 6/23/16

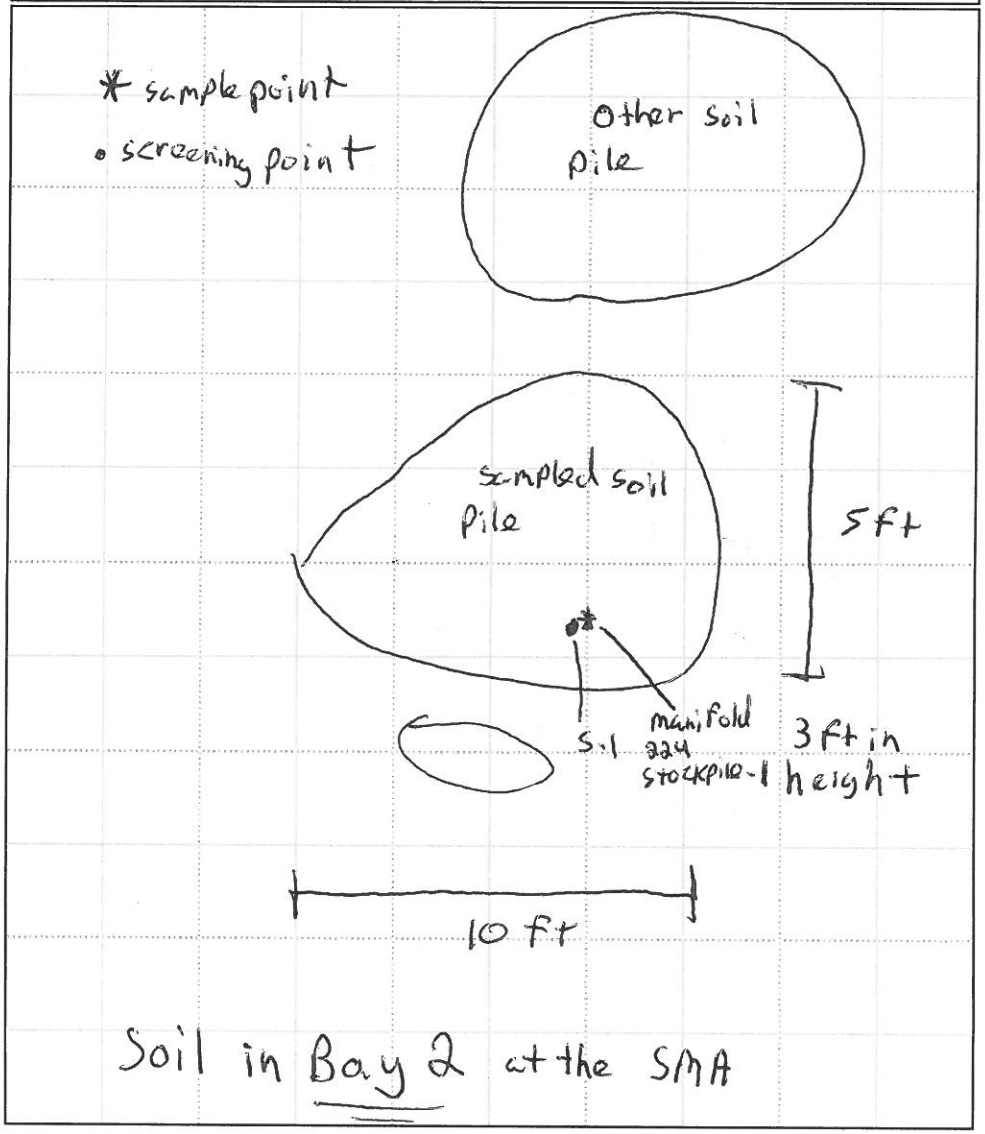
Sampler: JET/AJP

Calibration Time: 13:30

BARR

Sample ID	Depth (FT)	Time (military)	Soil Type (USCS)	Color/ Discolor	Odor/ Sheen	Headspace Reading (ppm)
Example: TK99-S-1	4	16:30	CL	Reddish brown	Petroleum/ Rainbow	275
S-1	-	14:15	sand	Reddish Brown	N/N	242.7
Manifold 224 Stockpile-1		14:15	Sand	Reddish Brown	N	

SITE SKETCH: *north is up; excavation extents & depths, impacted areas, sample locations, borings, wells, structures, utilities, natural features...* ***1 inch/grid =*** 5 ***FEET***



Soil in Bay 2 at the SMA

Attachment B:

ALS Laboratory Report for Excavation Soil Samples

01-Jul-2016

Ryan Erickson
Barr Engineering Company
4300 MarketPointe Drive
Suite 200
Minneapolis, MN 55435

Re: **Manifold 224 Excavation (49161092.04)** Work Order: **16061487**

Dear Ryan,

ALS Environmental received 3 samples on 24-Jun-2016 for the analyses presented in the following report.

The analytical data provided relates directly to the samples received by ALS Environmental and for only the analyses requested.

Sample results are compliant with NELAP standard requirements and QC results achieved laboratory specifications. Any exceptions are noted in the Case Narrative, or noted with qualifiers in the report or QC batch information. Should this laboratory report need to be reproduced, it should be reproduced in full unless written approval has been obtained from ALS Environmental. Samples will be disposed in 30 days unless storage arrangements are made.

The total number of pages in this report is 13.

If you have any questions regarding this report, please feel free to contact me.

Sincerely,

Electronically approved by: Tom Beamish

Tom Beamish
Client Services Coordinator

Certificate No: WI: 399084510

Report of Laboratory Analysis

ALS Group USA, Corp

Date: 01-Jul-16

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Work Order: **16061487**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
16061487-01	Manifold 224 S-1_2	Soil		06/23/16 13:50	06/24/16 10:00	☐
16061487-02	Manifold 224 B-1_5	Soil		06/23/16 13:45	06/24/16 10:00	☐
16061487-03	Trip Blank	Soil		06/23/16 13:45	06/24/16 10:00	☐

ALS Group USA, Corp

Date: 01-Jul-16

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
WorkOrder: **16061487**

QUALIFIERS, ACRONYMS, UNITS

Qualifier	Description
*	Value exceeds Regulatory Limit
a	Not accredited
B	Analyte detected in the associated Method Blank above the Reporting Limit
E	Value above quantitation range
H	Analyzed outside of Holding Time
J	Analyte is present at an estimated concentration between the MDL and Report Limit
n	Not offered for accreditation
ND	Not Detected at the Reporting Limit
O	Sample amount is > 4 times amount spiked
P	Dual Column results percent difference > 40%
R	RPD above laboratory control limit
S	Spike Recovery outside laboratory control limits
U	Analyzed but not detected above the MDL
X	Analyte was detected in the Method Blank between the MDL and Reporting Limit, sample results may exhibit background or reagent contamination at the observed level.

Acronym	Description
DUP	Method Duplicate
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection (see MDL)
LOQ	Limit of Quantitation (see PQL)
MBLK	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
PQL	Practical Quantitation Limit
RPD	Relative Percent Difference
TDL	Target Detection Limit
TNTC	Too Numerous To Count
A	APHA Standard Methods
D	ASTM
E	EPA
SW	SW-846 Update III

Units Reported	Description
% of sample	Percent of Sample
µg/Kg	Micrograms per Kilogram
µg/Kg-dry	Micrograms per Kilogram Dry Weight

ALS Group USA, Corp

Date: 01-Jul-16

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Work Order: 16061487

Case Narrative

Samples for the above noted Work Order were received on 06/24/16. The attached "Sample Receipt Checklist" documents the status of custody seals, container integrity, preservation, and temperature compliance.

Samples were analyzed according to the analytical methodology previously transmitted in the "Work Order Acknowledgement". Methodologies are also documented in the "Analytical Result" section for each sample. Quality control results are listed in the "QC Report" section. Sample association for the reported quality control is located at the end of each batch summary. If applicable, results are appropriately qualified in the Analytical Result and QC Report sections. The "Qualifiers" section documents the various qualifiers, units, and acronyms utilized in reporting.

With the following exceptions, all sample analyses achieved analytical criteria.

Volatile Organics:
No deviations or anomalies were noted.

Wet Chemistry:
No deviations or anomalies were noted.

ALS Group USA, Corp

Date: *01-Jul-16*

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Sample ID: Manifold 224 S-1_2
Collection Date: 06/23/16 01:50 PM

Work Order: 16061487
Lab ID: 16061487-01
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260B**			Prep: SW5035 / 6/24/16		Analyst: **AK**
1,2,4-Trimethylbenzene	U		6.6	33	µg/Kg-dry	1	07/01/16 02:42
1,3,5-Trimethylbenzene	U		14	33	µg/Kg-dry	1	07/01/16 02:42
Benzene	U		7.4	33	µg/Kg-dry	1	07/01/16 02:42
Ethylbenzene	U		7.6	33	µg/Kg-dry	1	07/01/16 02:42
m,p-Xylene	U		15	65	µg/Kg-dry	1	07/01/16 02:42
Naphthalene	U		5.6	110	µg/Kg-dry	1	07/01/16 02:42
o-Xylene	U		11	33	µg/Kg-dry	1	07/01/16 02:42
Toluene	U		11	33	µg/Kg-dry	1	07/01/16 02:42
Xylenes, Total	U		25	98	µg/Kg-dry	1	07/01/16 02:42
Surr: 1,2-Dichloroethane-d4	*102*			*70-130*	*%REC*	1	07/01/16 02:42
Surr: 4-Bromofluorobenzene	*98.4*			*70-130*	*%REC*	1	07/01/16 02:42
Surr: Dibromofluoromethane	*91.8*			*70-130*	*%REC*	1	07/01/16 02:42
Surr: Toluene-d8	*97.0*			*70-130*	*%REC*	1	07/01/16 02:42
MOISTURE		Method: **SW3550C**					Analyst: **EDL**
Moisture	**4.3**		**0.025**	**0.050**	**% of sample**	1	06/28/16 15:23

Note: See Qualifiers page for a list of qualifiers and their definitions.

ALS Group USA, Corp

Date: *01-Jul-16*

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Sample ID: Manifold 224 B-1_5
Collection Date: 06/23/16 01:45 PM

Work Order: 16061487
Lab ID: 16061487-02
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260B**			Prep: SW5035 / 6/24/16		Analyst: **AK**
1,2,4-Trimethylbenzene	U		8.5	42	µg/Kg-dry	1	07/01/16 03:07
1,3,5-Trimethylbenzene	U		19	42	µg/Kg-dry	1	07/01/16 03:07
Benzene	**100**		**9.6**	**42**	**µg/Kg-dry**	1	07/01/16 03:07
Ethylbenzene	20	J	9.9	42	µg/Kg-dry	1	07/01/16 03:07
m,p-Xylene	33	J	19	85	µg/Kg-dry	1	07/01/16 03:07
Naphthalene	U		7.2	140	µg/Kg-dry	1	07/01/16 03:07
o-Xylene	**50**		**14**	**42**	**µg/Kg-dry**	1	07/01/16 03:07
Toluene	**160**		**14**	**42**	**µg/Kg-dry**	1	07/01/16 03:07
Xylenes, Total	83	J	33	130	µg/Kg-dry	1	07/01/16 03:07
Surr: 1,2-Dichloroethane-d4	*102*			*70-130*	*%REC*	1	07/01/16 03:07
Surr: 4-Bromofluorobenzene	*94.0*			*70-130*	*%REC*	1	07/01/16 03:07
Surr: Dibromofluoromethane	*93.0*			*70-130*	*%REC*	1	07/01/16 03:07
Surr: Toluene-d8	*96.5*			*70-130*	*%REC*	1	07/01/16 03:07
MOISTURE		Method: **SW3550C**					Analyst: **EDL**
Moisture	**17**		**0.025**	**0.050**	**% of sample**	1	06/28/16 15:23

Note: See Qualifiers page for a list of qualifiers and their definitions.

ALS Group USA, Corp

Date: *01-Jul-16*

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Sample ID: Trip Blank
Collection Date: 06/23/16 01:45 PM

Work Order: 16061487
Lab ID: 16061487-03
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260B**			Prep: SW5035 / 6/24/16		Analyst: **AK**
1,2,4-Trimethylbenzene	U		6.0	30	µg/Kg	1	07/01/16 03:32
1,3,5-Trimethylbenzene	U		13	30	µg/Kg	1	07/01/16 03:32
Benzene	U		6.8	30	µg/Kg	1	07/01/16 03:32
Ethylbenzene	U		7.0	30	µg/Kg	1	07/01/16 03:32
m,p-Xylene	U		13	60	µg/Kg	1	07/01/16 03:32
Naphthalene	U		5.1	100	µg/Kg	1	07/01/16 03:32
o-Xylene	U		9.7	30	µg/Kg	1	07/01/16 03:32
Toluene	U		9.9	30	µg/Kg	1	07/01/16 03:32
Xylenes, Total	U		23	90	µg/Kg	1	07/01/16 03:32
Surr: 1,2-Dichloroethane-d4	*100*			*70-130*	*%REC*	1	07/01/16 03:32
Surr: 4-Bromofluorobenzene	*97.3*			*70-130*	*%REC*	1	07/01/16 03:32
Surr: Dibromofluoromethane	*92.7*			*70-130*	*%REC*	1	07/01/16 03:32
Surr: Toluene-d8	*97.9*			*70-130*	*%REC*	1	07/01/16 03:32

Note: See Qualifiers page for a list of qualifiers and their definitions.

QC BATCH REPORT

Batch ID: **87810** Instrument ID **VMS6** Method: **SW8260B**

MBLK Sample ID: **MBLK-87810-87810** Units: **µg/Kg-dry** Analysis Date: **06/27/16 11:52 AM**

Client ID: Run ID: **VMS6_160627A** SeqNo: **3895447** Prep Date: **06/24/16** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	U	6	30								
1,3,5-Trimethylbenzene	U	13	30								
Benzene	U	6.8	30								
Ethylbenzene	U	7	30								
m,p-Xylene	U	13	60								
Naphthalene	U	5.1	100								
o-Xylene	U	9.7	30								
Toluene	U	9.9	30								
Xylenes, Total	U	23	90								
Surr: 1,2-Dichloroethane-d4	*995.5*	0	0	*1000*	0	*99.6*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*948*	0	0	*1000*	0	*94.8*	*70-130*	0			
Surr: Dibromofluoromethane	*953*	0	0	*1000*	0	*95.3*	*70-130*	0			
Surr: Toluene-d8	*992.5*	0	0	*1000*	0	*99.2*	*70-130*	0			

LCS Sample ID: **LCS-87810-87810** Units: **µg/Kg-dry** Analysis Date: **06/27/16 10:34 AM**

Client ID: Run ID: **VMS6_160627A** SeqNo: **3895446** Prep Date: **06/24/16** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	1004	6	30	1000	0	100	65-135	0			
1,3,5-Trimethylbenzene	1024	13	30	1000	0	102	65-135	0			
Benzene	1089	6.8	30	1000	0	109	75-125	0			
Ethylbenzene	1042	7	30	1000	0	104	75-125	0			
m,p-Xylene	2090	13	60	2000	0	104	80-125	0			
Naphthalene	993	5.1	100	1000	0	99.3	40-140	0			
o-Xylene	1024	9.7	30	1000	0	102	75-125	0			
Toluene	1096	9.9	30	1000	0	110	70-125	0			
Xylenes, Total	3113	23	90	3000	0	104	75-125	0			
Surr: 1,2-Dichloroethane-d4	*996.5*	0	0	*1000*	0	*99.6*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*972*	0	0	*1000*	0	*97.2*	*70-130*	0			
Surr: Dibromofluoromethane	*994.5*	0	0	*1000*	0	*99.4*	*70-130*	0			
Surr: Toluene-d8	*996*	0	0	*1000*	0	*99.6*	*70-130*	0			

Note: See Qualifiers Page for a list of Qualifiers and their explanation.

Client: Barr Engineering Company
Work Order: 16061487
Project: Manifold 224 Excavation (49161092.04)

QC BATCH REPORT

Batch ID: **87810** Instrument ID **VMS6** Method: **SW8260B**

MS Sample ID: **16061450-03A MS** Units: **µg/Kg-dry** Analysis Date: **07/01/16 01:59 PM**
Client ID: Run ID: **VMS5_160630B** SeqNo: **3903928** Prep Date: **06/24/16** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	1343	7.8	39	1299	0	103	65-135	0			
1,3,5-Trimethylbenzene	1380	17	39	1299	0	106	65-135	0			
Benzene	1345	8.8	39	1299	0	104	75-125	0			
Ethylbenzene	1351	9.1	39	1299	0	104	75-125	0			
m,p-Xylene	2725	18	78	2598	0	105	80-125	0			
Naphthalene	1152	6.7	130	1299	0	88.7	40-140	0			
o-Xylene	1324	13	39	1299	0	102	75-125	0			
Toluene	1333	13	39	1299	0	103	70-125	0			
Xylenes, Total	4049	30	120	3897	0	104	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1321*	0	0	*1299*	0	*102*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1298*	0	0	*1299*	0	*99.9*	*70-130*	0			
Surr: Dibromofluoromethane	*1299*	0	0	*1299*	0	*100*	*70-130*	0			
Surr: Toluene-d8	*1285*	0	0	*1299*	0	*98.9*	*70-130*	0			

MSD Sample ID: **16061450-03A MSD** Units: **µg/Kg-dry** Analysis Date: **07/01/16 02:25 PM**
Client ID: Run ID: **VMS5_160630B** SeqNo: **3903929** Prep Date: **06/24/16** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	1386	7.8	39	1299	0	107	65-135	1343	3.14	30	
1,3,5-Trimethylbenzene	1431	17	39	1299	0	110	65-135	1380	3.6	30	
Benzene	1343	8.8	39	1299	0	103	75-125	1345	0.145	30	
Ethylbenzene	1370	9.1	39	1299	0	106	75-125	1351	1.38	30	
m,p-Xylene	2763	18	78	2598	0	106	80-125	2725	1.4	30	
Naphthalene	1309	6.7	130	1299	0	101	40-140	1152	12.8	30	
o-Xylene	1344	13	39	1299	0	103	75-125	1324	1.46	30	
Toluene	1342	13	39	1299	0	103	70-125	1333	0.68	30	
Xylenes, Total	4107	30	120	3897	0	105	75-125	4049	1.42	30	
Surr: 1,2-Dichloroethane-d4	*1303*	0	0	*1299*	0	*100*	*70-130*	1321	*1.34*	*30*	
Surr: 4-Bromofluorobenzene	*1305*	0	0	*1299*	0	*100*	*70-130*	1298	*0.599*	*30*	
Surr: Dibromofluoromethane	*1297*	0	0	*1299*	0	*99.8*	*70-130*	1299	*0.15*	*30*	
Surr: Toluene-d8	*1285*	0	0	*1299*	0	*99*	*70-130*	1285	*0.0505*	*30*	

The following samples were analyzed in this batch:

16061487-01A	16061487-02A	16061487-03A

Note: See Qualifiers Page for a list of Qualifiers and their explanation.

Client: Barr Engineering Company
Work Order: 16061487
Project: Manifold 224 Excavation (49161092.04)

QC BATCH REPORT

Batch ID: **R190499** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R190499** Units: **% of sample** Analysis Date: **06/28/16 03:23 PM**
Client ID: Run ID: **MOIST_160628B** SeqNo: **3898661** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	U	0.025	0.050								

LCS Sample ID: **LCS-R190499** Units: **% of sample** Analysis Date: **06/28/16 03:23 PM**
Client ID: Run ID: **MOIST_160628B** SeqNo: **3898660** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	100	0.025	0.050	100	0	100	99.5-100.5	0			

DUP Sample ID: **16061007-46A DUP** Units: **% of sample** Analysis Date: **06/28/16 03:23 PM**
Client ID: Run ID: **MOIST_160628B** SeqNo: **3898640** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	19.04	0.025	0.050	0	0	0		20.46	7.19	20	

DUP Sample ID: **16061487-02B DUP** Units: **% of sample** Analysis Date: **06/28/16 03:23 PM**
Client ID: **Manifold 224 B-1_5** Run ID: **MOIST_160628B** SeqNo: **3898647** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	14.58	0.025	0.050	0	0	0		16.68	13.4	20	

The following samples were analyzed in this batch: 16061487-01B 16061487-02B

Note: See Qualifiers Page for a list of Qualifiers and their explanation.

160616487

Barr Engineering Co. Chain of Custody

BARR ☐ Ann Arbor ☐ Bismarck ☒ Duluth ☐ Hibbing ☐ Jefferson City ☐ Minneapolis

Sample Origination State: ☐ KS ☐ MI ☐ MN ☐ MO ☐ ND ☐ SD ☒ WI Other:

COC Number: № 47528

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company: Barr Engineering
Address: 325 S. Lake Ave Duluth	Address:
Name: Ryan Erickson	Name:
email: rerickson@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: Manifold 224 Excavation	Barr Project No: 49161092.04 003 003

Matrix Code: GW = Groundwater, SW = Surface Water, WW = Waste Water, DW = Drinking Water, S = Soil/Solid, SD = Sediment, O = Other

Preservative Code: A = None, B = HCl, C = HNO_3, D = H_2SO_4, E = NaOH, F = MeOH, G = $NaHSO_4$, H = $Na_2S_2O_3$, I = Ascorbic Acid, J = NH_4Cl, K = Zn Acetate, O = Other

Location	Sample Depth Start	Stop	Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform MS/MSD Y / N	Total Number Of Containers	Analysis Requested: Water	Soil: F PVOC + Naphthalene	Soil: % Solids A	Preservative Code	Field Filtered Y/N
1. Manifold 224 S-1	2	-	ft	6/23/16	13:50	S	N	3		2	1	PVOC + Naphthalene	
~~Manifold 224 S-2~~	~~2~~		~~ft~~	~~6/23/16~~	~~13:52~~	~~S~~	~~N~~	~~3~~		~~2~~	~~1~~	~~PVOC + Naphthalene~~	
2 Manifold 224 B-1	5	-	ft	6/23/16	13:45	S	N	3		2	1	PVOC + Naphthalene	
3 Trip Blank	—			6/23/16	13:45	Q/C	N	1		1	-	PVOC + Naphthalene	
Temp Blank	—	—		—	—	Q/C	-	1			-		
6.													
7.													
8.												Standard TAT	
9.													
10.													

BARR USE ONLY		Relinquished by	On Ice? (Y) N	Date	Time	Received by:	Date	Time
Sampled by:	J Tavaldsen / Mike Powers	Michael Powers		6-23-16	16:30			
Barr Proj. Manager:	Ryan Erickson	Relinquished by:	On Ice? Y N	Date	Time	Received by:	Date 6/24/16	Time 1000
Barr DQ Manager:	J Tavaldsen	Samples Shipped VIA: ☐ Courier ☒ Federal Express ☐ Sampler ☐ Other:		Air Bill Number:		Requested Due Date: ☒ Standard Turn Around Time ☐ Rush (mm/dd/yyyy)		
Lab Name:	ALS Environmental	Lab WO:	Temperature on Receipt (°C):			Custody Seal Intact? ☐ Y ☐ N ☐ None		
Lab Location:	Holland, MI							

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Pink Copy: Send to Data Management Administrators.

H:\RLG\STDFORMS\Chain Of Custody Form 2015 RLG Rev. 06/16/15

TAS 5.2C

ALS Group USA, Corp

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **24-Jun-16 10:00**

Work Order: **16061487** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 24-Jun-16 (Date) Reviewed by: Tom Bramish (eSignature) 24-Jun-16 (Date)

Matrices: Soil

Carrier name: FedEx

Shipping container/cooler in good condition?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on shipping container/cooler?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on sample bottles?	Yes ☐	No ☐	Not Present ☑
Chain of custody present?	Yes ☑	No ☐	
Chain of custody signed when relinquished and received?	Yes ☑	No ☐	
Chain of custody agrees with sample labels?	Yes ☑	No ☐	
Samples in proper container/bottle?	Yes ☑	No ☐	
Sample containers intact?	Yes ☑	No ☐	
Sufficient sample volume for indicated test?	Yes ☑	No ☐	
All samples received within holding time?	Yes ☑	No ☐	
Container/Temp Blank temperature in compliance?	Yes ☑	No ☐	
Sample(s) received on ice?	Yes ☑	No ☐	
Temperature(s)/Thermometer(s):	5.2/5.2 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	6/24/2016 12:19:12 PM		
Water - VOA vials have zero headspace?	Yes ☐	No ☐	No VOA vials submitted ☑
Water - pH acceptable upon receipt?	Yes ☐	No ☐	N/A ☑
pH adjusted?	Yes ☐	No ☐	N/A ☑
pH adjusted by:	-		

Login Notes:

Client Contacted: Date Contacted: Person Contacted:

Contacted By: Regarding:

Comments:

CorrectiveAction:

Attachment C:

Waste Disposal Documentation

Vonco V Profile Amendment Request Form

Date: 6/28/2016

Alex Smith

(Contact and Customer Name)

hereby requests an amendment to Vonco V profile # # 16-065-I

to include the following: Profile Name: SUP MLE Manifold Excavations

Reason for Amendment:

Amendment Type: [ ] One time on Request [ ] Permanent addition to Profile

[X] Additional Analytical/MSDS to be added to profile (see attached)

[ ] Volume Increase (specify volume) _____

[ ] Constituent(s) to be added and/or modify current range in chemical composition

[ ] Chemicals or constituents to be added/modified Low High Units

_____ _____

[ ] Other (Specify)

_____

Generator Certification

By signing this form, the Generator hereby certifies:
The information provided in this document, the referenced Vonco V Waste Profile Sheet, and all other referenced documents contain true and accurate descriptions of the waste material. All information regarding known or suspected hazards in the possession of the Generator has been disclosed.

Generator/Customer Signature: _____ Date: 6/29/2016

Company Name: Enbridge Energy

Name (print): Alex Smith Title: Analyst

For Vonco II Use Only

Submitted By: _____ Date: _____

VV Approval: _____ Date: _____

Profile Extension		Analytical Extension	
Original Expiration Date		Analytical Due Date	
Requested Extension Date		Requested Extension Date	
New Expiration Date		New Analytical Due Date	

Conditions: _____

_____

VONCO
Waste Management Campus

VONCO V, LLC
1100 West Gary Street
Duluth, MN 55808

VONCOUSA.com
Office: 218.626.3830
Fax: 218.626.4874

June 30, 2016

Enbridge Energy
Attention: Alex Smith
1100 Louisiana Ave Suite 3300
Houston, TX 77002

RE: Profile # 16-065-I (SUP MLE Manifold Excavations amendment 6-30-16)
Generator: Enbridge Superior Wi Terminal
Waste Stream: contaminated soil

Alex,

Please be advised that the above described waste material is acceptable for disposal at the Vonco V Waste Management Campus Facility in Duluth, MN. The waste material is acceptable per Vonco V (SW-560) Minnesota Pollution Control Agency Industrial Solid Waste Management Plan. The profile is approved for **1000** CY for disposal.

The referenced waste must maintain consistency with what was originally submitted on the waste profile. Vonco V Waste Management Campus must be contacted immediately for any changes in material composition or process generation as further testing and analysis may apply.

Additionally, acceptance is subject to the following conditions:

- The material will be absent of free liquids and must meet the paint filter test.
- A signed waste manifest with the correct profile number shall accompany each load delivered to Vonco V Waste Management Campus.
- All hauling will be in compliance with the Federal and State D.O.T regulations.

Thank you for choosing Vonco V Waste Management Campus. We appreciate your business. If you have any questions or concerns please feel free to contact me @ (218) 730-6361.

Have a great day,

Joe Pesante
Vonco V, LLC

28-Jun-2016

Ryan Erickson
Barr Engineering Company
4300 MarketPointe Drive
Suite 200
Minneapolis, MN 55435

Re: **Manifold 224 Excavation (49161092.04)** Work Order: **16061473**

Dear Ryan,

ALS Environmental received 2 samples on 24-Jun-2016 for the analyses presented in the following report.

The analytical data provided relates directly to the samples received by ALS Environmental and for only the analyses requested.

Sample results are compliant with NELAP standard requirements and QC results achieved laboratory specifications. Any exceptions are noted in the Case Narrative, or noted with qualifiers in the report or QC batch information. Should this laboratory report need to be reproduced, it should be reproduced in full unless written approval has been obtained from ALS Environmental. Samples will be disposed in 30 days unless storage arrangements are made.

The total number of pages in this report is 13.

If you have any questions regarding this report, please feel free to contact me.

Sincerely,

Electronically approved by: Tom Beamish

Tom Beamish
Client Services Coordinator

Certificate No: WI: 399084510

ADDRESS 3352 128th Avenue Holland, Michigan 49424-9263 | PHONE (616) 399-6070 | FAX (616) 399-6185
ALS GROUP USA, CORP Part of the ALS Laboratory Group A Campbell Brothers Limited Company

ALS Group USA, Corp

Date: 28-Jun-16

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Work Order: **16061473**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
16061473-01	Manifold 224 Stockpile - 1	Soil		06/23/16 14:15	06/24/16 10:00	☐
16061473-02	Trip Blank	Soil		06/23/16 14:15	06/24/16 10:00	☐

ALS Group USA, Corp

Date: 28-Jun-16

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
WorkOrder: **16061473**

QUALIFIERS, ACRONYMS, UNITS

Qualifier	Description
*	Value exceeds Regulatory Limit
a	Not accredited
B	Analyte detected in the associated Method Blank above the Reporting Limit
E	Value above quantitation range
H	Analyzed outside of Holding Time
J	Analyte is present at an estimated concentration between the MDL and Report Limit
n	Not offered for accreditation
ND	Not Detected at the Reporting Limit
O	Sample amount is > 4 times amount spiked
P	Dual Column results percent difference > 40%
R	RPD above laboratory control limit
S	Spike Recovery outside laboratory control limits
U	Analyzed but not detected above the MDL
X	Analyte was detected in the Method Blank between the MDL and Reporting Limit, sample results may exhibit background or reagent contamination at the observed level.

Acronym	Description
DUP	Method Duplicate
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection (see MDL)
LOQ	Limit of Quantitation (see PQL)
MBLK	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
PQL	Practical Quantitation Limit
RPD	Relative Percent Difference
TDL	Target Detection Limit
TNTC	Too Numerous To Count
A	APHA Standard Methods
D	ASTM
E	EPA
SW	SW-846 Update III

Units Reported	Description
% of sample	Percent of Sample
µg/Kg	Micrograms per Kilogram
µg/Kg-dry	Micrograms per Kilogram Dry Weight
mg/Kg-dry	Milligrams per Kilogram Dry Weight

ALS Group USA, Corp

Date: 28-Jun-16

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Work Order: 16061473

Case Narrative

Samples for the above noted Work Order were received on 06/24/16. The attached "Sample Receipt Checklist" documents the status of custody seals, container integrity, preservation, and temperature compliance.

Samples were analyzed according to the analytical methodology previously transmitted in the "Work Order Acknowledgement". Methodologies are also documented in the "Analytical Result" section for each sample. Quality control results are listed in the "QC Report" section. Sample association for the reported quality control is located at the end of each batch summary. If applicable, results are appropriately qualified in the Analytical Result and QC Report sections. The "Qualifiers" section documents the various qualifiers, units, and acronyms utilized in reporting.

With the following exceptions, all sample analyses achieved analytical criteria.

Volatile Organics:
No deviations or anomalies were noted.

Extractable Organics:
Batch 87848, Method DRO_Wisconsin_S, Sample DLCSS1-87848: The LCS recovery was below the lower control limit. The sample results may be biased low for DRO (C10-C28).

Batch 87848, Method DRO_Wisconsin_S, Sample DLCSDS1-87848: The LCS recovery was below the lower control limit. The sample results may be biased low for DRO (C10-C28).

No other deviations or anomalies were noted.

Wet Chemistry:
No deviations or anomalies were noted.

ALS Group USA, Corp

Date: *28-Jun-16*

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Sample ID: Manifold 224 Stockpile - 1
Collection Date: 06/23/16 02:15 PM

Work Order: 16061473
Lab ID: 16061473-01
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
DIESEL RANGE ORGANICS BY GC-FID		Method: **PUBL-SW-141**			Prep: PUBL-SW-141 / 6/27/16		Analyst: **IT**
DRO (C10-C28)	**2,200**		**20**	**50**	**mg/Kg-dry**	10	06/27/16 16:09
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260B**			Prep: SW5035 / 6/24/16		Analyst: **AK**
Benzene	**91**		**6.8**	**30**	**µg/Kg-dry**	1	06/24/16 18:34
Ethylbenzene	**2,000**		**7.0**	**30**	**µg/Kg-dry**	1	06/24/16 18:34
m,p-Xylene	**5,500**		**13**	**60**	**µg/Kg-dry**	1	06/24/16 18:34
o-Xylene	**2,900**		**9.7**	**30**	**µg/Kg-dry**	1	06/24/16 18:34
Toluene	**1,700**		**9.9**	**30**	**µg/Kg-dry**	1	06/24/16 18:34
Xylenes, Total	**8,400**		**23**	**90**	**µg/Kg-dry**	1	06/24/16 18:34
Surr: 1,2-Dichloroethane-d4	*99.0*			*70-130*	*%REC*	1	06/24/16 18:34
Surr: 4-Bromofluorobenzene	*120*			*70-130*	*%REC*	1	06/24/16 18:34
Surr: Dibromofluoromethane	*88.1*			*70-130*	*%REC*	1	06/24/16 18:34
Surr: Toluene-d8	*109*			*70-130*	*%REC*	1	06/24/16 18:34
MOISTURE		Method: **SW3550C**					Analyst: **EVB**
Moisture	**6.3**		**0.025**	**0.050**	**% of sample**	1	06/24/16 17:50

Note: See Qualifiers page for a list of qualifiers and their definitions.

ALS Group USA, Corp

Date: *28-Jun-16*

Client: Barr Engineering Company
Project: Manifold 224 Excavation (49161092.04)
Sample ID: Trip Blank
Collection Date: 06/23/16 02:15 PM

Work Order: 16061473
Lab ID: 16061473-02
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260B**			Prep: SW5035 / 6/24/16		Analyst: **AK**
Benzene	U		6.8	30	µg/Kg	1	06/24/16 17:45
Ethylbenzene	U		7.0	30	µg/Kg	1	06/24/16 17:45
m,p-Xylene	U		13	60	µg/Kg	1	06/24/16 17:45
o-Xylene	U		9.7	30	µg/Kg	1	06/24/16 17:45
Toluene	U		9.9	30	µg/Kg	1	06/24/16 17:45
Xylenes, Total	U		23	90	µg/Kg	1	06/24/16 17:45
Surr: 1,2-Dichloroethane-d4	*105*			*70-130*	*%REC*	1	06/24/16 17:45
Surr: 4-Bromofluorobenzene	*96.2*			*70-130*	*%REC*	1	06/24/16 17:45
Surr: Dibromofluoromethane	*94.5*			*70-130*	*%REC*	1	06/24/16 17:45
Surr: Toluene-d8	*95.4*			*70-130*	*%REC*	1	06/24/16 17:45

Note: See Qualifiers page for a list of qualifiers and their definitions.

ALS Group USA, Corp

Date: 28-Jun-16

Client: Barr Engineering Company

QC BATCH REPORT

Work Order: 16061473

Project: Manifold 224 Excavation (49161092.04)

Batch ID: **87848** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK	Sample ID: **DBLKS1-87848-87848**					Units: **mg/Kg**			Analysis Date: **06/27/16 02:10 PM**			
Client ID:		Run ID: **GC8_160627A**				SeqNo: **3896331**		Prep Date: **06/27/16**		DF: **1**		
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	U	2	5.0								

LCS	Sample ID: **DLCSS1-87848-87848**					Units: **mg/Kg**			Analysis Date: **06/27/16 01:40 PM**			
Client ID:		Run ID: **GC8_160627A**				SeqNo: **3896330**		Prep Date: **06/27/16**		DF: **1**		
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	64.86	2	5.0	200	0	32.4	70-120	0			S

LCSD	Sample ID: **DLCSDS1-87848-87848**					Units: **mg/Kg**			Analysis Date: **06/27/16 04:40 PM**			
Client ID:		Run ID: **GC8_160627A**				SeqNo: **3896336**		Prep Date: **06/27/16**		DF: **1**		
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	70.6	2	5.0	200	0	35.3	70-120	64.86	8.48	20	S

The following samples were analyzed in this batch: 16061473-01B

Note: See Qualifiers Page for a list of Qualifiers and their explanation.

Client: Barr Engineering Company
Work Order: 16061473
Project: Manifold 224 Excavation (49161092.04)

QC BATCH REPORT

Batch ID: **87820** Instrument ID **VMS7** Method: **SW8260B**

MBLK Sample ID: **MBLK-87820-87820** Units: **µg/Kg-dry** Analysis Date: **06/24/16 01:58 PM**
Client ID: Run ID: **VMS7_160624A** SeqNo: **3892371** Prep Date: **06/24/16** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	U	6.8	30								
Ethylbenzene	U	7	30								
m,p-Xylene	U	13	60								
o-Xylene	U	9.7	30								
Toluene	U	9.9	30								
Xylenes, Total	U	23	90								
Surr: 1,2-Dichloroethane-d4	*1014*	0	0	*1000*	0	*101*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*962.5*	0	0	*1000*	0	*96.2*	*70-130*	0			
Surr: Dibromofluoromethane	*970.5*	0	0	*1000*	0	*97*	*70-130*	0			
Surr: Toluene-d8	*989*	0	0	*1000*	0	*98.9*	*70-130*	0			

LCS Sample ID: **LCS-87820-87820** Units: **µg/Kg-dry** Analysis Date: **06/24/16 12:17 PM**
Client ID: Run ID: **VMS7_160624A** SeqNo: **3892370** Prep Date: **06/24/16** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1249	6.8	30	1000	0	125	75-125	0			
Ethylbenzene	1004	7	30	1000	0	100	75-125	0			
m,p-Xylene	2461	13	60	2000	0	123	80-125	0			
o-Xylene	1040	9.7	30	1000	0	104	75-125	0			
Toluene	1214	9.9	30	1000	0	121	70-125	0			
Xylenes, Total	3500	23	90	3000	0	117	75-125	0			
Surr: 1,2-Dichloroethane-d4	*942.5*	0	0	*1000*	0	*94.2*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*999*	0	0	*1000*	0	*99.9*	*70-130*	0			
Surr: Dibromofluoromethane	*1006*	0	0	*1000*	0	*101*	*70-130*	0			
Surr: Toluene-d8	*954*	0	0	*1000*	0	*95.4*	*70-130*	0			

MS Sample ID: **16061327-05A MS** Units: **µg/Kg-dry** Analysis Date: **06/27/16 05:55 PM**
Client ID: Run ID: **VMS7_160627A** SeqNo: **3896278** Prep Date: **06/24/16** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1558	8.2	36	1210	0	129	75-125	0			S
Ethylbenzene	1205	8.5	36	1210	10.85	98.7	75-125	0			
m,p-Xylene	2998	16	73	2420	0	124	80-125	0			
o-Xylene	1213	12	36	1210	21.02	98.5	75-125	0			
Toluene	1527	12	36	1210	0	126	70-125	0			S
Xylenes, Total	4211	28	110	3630	21	115	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1185*	0	0	*1210*	0	*98*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1176*	0	0	*1210*	0	*97.2*	*70-130*	0			
Surr: Dibromofluoromethane	*1186*	0	0	*1210*	0	*98*	*70-130*	0			
Surr: Toluene-d8	*1161*	0	0	*1210*	0	*96*	*70-130*	0			

Note: See Qualifiers Page for a list of Qualifiers and their explanation.

Client: Barr Engineering Company
Work Order: 16061473
Project: Manifold 224 Excavation (49161092.04)

QC BATCH REPORT

Batch ID: **87820** Instrument ID **VMS7** Method: **SW8260B**

MSD Sample ID: **16061327-05A MSD** Units: **µg/Kg-dry** Analysis Date: **06/27/16 06:20 PM**
Client ID: Run ID: **VMS7_160627A** SeqNo: **3896279** Prep Date: **06/24/16** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1670	8.2	36	1210	0	138	75-125	1558	6.94	30	S
Ethylbenzene	1390	8.5	36	1210	10.85	114	75-125	1205	14.3	30	
m,p-Xylene	3491	16	73	2420	0	144	80-125	2998	15.2	30	S
o-Xylene	1397	12	36	1210	21.02	114	75-125	1213	14.1	30	
Toluene	1722	12	36	1210	0	142	70-125	1527	12	30	S
Xylenes, Total	4889	28	110	3630	21	134	75-125	4211	14.9	30	S
Surr: 1,2-Dichloroethane-d4	*1109*	0	0	*1210*	0	*91.6*	*70-130*	1185	*6.65*	*30*	
Surr: 4-Bromofluorobenzene	*1157*	0	0	*1210*	0	*95.6*	*70-130*	1176	*1.61*	*30*	
Surr: Dibromofluoromethane	*1172*	0	0	*1210*	0	*96.9*	*70-130*	1186	*1.13*	*30*	
Surr: Toluene-d8	*1182*	0	0	*1210*	0	*97.6*	*70-130*	1161	*1.76*	*30*	

The following samples were analyzed in this batch: 16061473-01A 16061473-02A

Note: See Qualifiers Page for a list of Qualifiers and their explanation.

Client: Barr Engineering Company
Work Order: 16061473
Project: Manifold 224 Excavation (49161092.04)

QC BATCH REPORT

Batch ID: **R190269** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R190269** Units: **% of sample** Analysis Date: **06/24/16 05:50 PM**
Client ID: Run ID: **MOIST_160624C** SeqNo: **3892919** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	0.03	0.025	0.050								J

LCS Sample ID: **LCS-R190269** Units: **% of sample** Analysis Date: **06/24/16 05:50 PM**
Client ID: Run ID: **MOIST_160624C** SeqNo: **3892918** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	100	0.025	0.050	100	0	100	99.5-100.5	0			

DUP Sample ID: **16061472-01B DUP** Units: **% of sample** Analysis Date: **06/24/16 05:50 PM**
Client ID: Run ID: **MOIST_160624C** SeqNo: **3892897** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	33.01	0.025	0.050	0	0	0		37.34	12.3	20	

DUP Sample ID: **16061546-03B DUP** Units: **% of sample** Analysis Date: **06/24/16 05:50 PM**
Client ID: Run ID: **MOIST_160624C** SeqNo: **3892908** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	20.85	0.025	0.050	0	0	0		19.17	8.4	20	

The following samples were analyzed in this batch: 16061473-01C

Note: See Qualifiers Page for a list of Qualifiers and their explanation.

160061473

Barr Engineering Co. Chain of Custody

BARR ☐ Ann Arbor ☐ Bismarck ☐ Duluth ☐ Hibbing ☐ Jefferson City ☐ Minneapolis

Sample Origination State: ☐ KS ☐ MO ☒ WI ☐ MI ☐ ND Other: ☐ MN ☐ SD

COC Number: № 48068

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company: Barr Engineering
Address: 3255 Lake Ave Duluth	Address:
Name: Ryan Erickson	Name:
email: rerickson@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: Manifold 224 Excavation	Barr Project No: 49161092.04 0030

Matrix Code: GW = Groundwater, SW = Surface Water, WW = Waste Water, DW = Drinking Water, S = Soil/Solid, SD = Sediment, O = Other

Preservative Code: A = None, B = HCl, C = HNO_3, D = H_2SO_4, E = NaOH, F = MeOH, G = $NaHSO_4$, H = $Na_2S_2O_3$, I = Ascorbic Acid, J = NH_4Cl, K = Zn Acetate, O = Other

Location	Sample Depth Start	Stop	Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform MS/MSD Y / N	Total Number Of Containers	Analysis Requested: Water	Soil: BTEX	Soil: DRO	Soil: Hold Sample	% Solids	Preservative Code / Field Filtered Y/N
1. Manifold 224 Stockpile-1	—			6/23/16	14:15	S	N	5		2	1	1	1	BTEX, DRO (+) Hold Sample
2. Trip Blank	—			6/23/16	14:15	S	N			1			—	BTEX
3. Temp Blank														
4.														
5.														
6.														
7.														
8.														
9.														
10.														

ASAP TAT

BARR USE ONLY

Sampled by: J. Donaldson / Mike Powell

Barr Proj. Manager: Ryan Erickson

Barr DQ Manager: J. Donaldson

Lab Name: ALS Environmental

Lab Location: Holland

Relinquished by: Michael Powell — On Ice? (Y) N — Date 6-23-16 — Time 16:30

Relinquished by: — On Ice? Y (N) — Date — Time

Received by: — Date — Time

Received by: — Date 6/24/16 — Time 1000

Samples Shipped VIA: ☐ Courier ☒ Federal Express ☐ Sampler ☐ Other:

Air Bill Number:

Lab WO: Temperature on Receipt (°C): Custody Seal Intact? ☐ Y ☐ N ☐ None

Requested Due Date: ☐ Standard Turn Around Time ☒ Rush ASAP (mm/dd/yyyy)

H:\RLG\STDFORMS\Chain Of Custody Form 2015 RLG Rev. 06/16/15

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Pink Copy: Send to Data Management Administrators.

5.2°C

FedEx Express

Package US Airbill

FedEx Tracking Number 8100 9104 2346

Form ID No. 0200

Recipient's Copy

1 From

Date 6/23/16 [redacted]

Sender's Name Jim Taraldsen Phone 218 481-3020

Company Barr Engineering

Address 325 S. Lake Ave. STE 700

Dept./Floor/Suite/Room

City Duluth State MN ZIP 55802-2323

2 Your Internal Billing Reference 49161092.04 003 003

3 To

Recipient's Name Tom Beamish Phone 616 399-6070

Company ALS Environmental

Address 3352 128th Ave.

We cannot deliver to P.O. boxes or P.O. ZIP codes. Dept./Floor/Suite/Room

Hold Weekday — FedEx location address REQUIRED. NOT available for FedEx First Overnight.

Address

Use this line for the HOLD location address or for continuation of your shipping address.

Hold Saturday — FedEx location address REQUIRED. Available ONLY for FedEx Priority Overnight and FedEx 2Day to select locations.

City Holland State MI ZIP 49424

4 Express Package Service *To most locations.

Packages up to 150 lbs.

Next Business Day

- [ ] FedEx First Overnight — Earliest next business morning delivery to select locations. Friday shipments will be delivered on Monday unless Saturday Delivery is selected.
- [x] FedEx Priority Overnight — Next business morning.* Friday shipments will be delivered on Monday unless Saturday Delivery is selected.
- [ ] FedEx Standard Overnight — Next business afternoon.* Saturday Delivery NOT available.

2 or 3 Business Days

- [ ] FedEx 2Day A.M. — Second business morning.* Saturday Delivery NOT available.
- [ ] FedEx 2Day — Second business afternoon.* Thursday shipments will be delivered on Monday unless Saturday Delivery is selected.
- [ ] FedEx Express Saver — Third business day.* Saturday Delivery NOT available.

5 Packaging *Declared value limit $500.

- [ ] FedEx Envelope*
- [ ] FedEx Pak*
- [ ] FedEx Box
- [ ] FedEx Tube
- [x] Other

6 Special Handling and Delivery Signature Options Fees may apply. See the FedEx Service Guide.

- [ ] Saturday Delivery — NOT available for FedEx Standard Overnight, FedEx 2Day A.M., or FedEx Express Saver.
- [x] No Signature Required — Package may be left without obtaining a signature for delivery.
- [ ] Direct Signature — Someone at recipient's address may sign for delivery.
- [ ] Indirect Signature — If no one is available at recipient's address, someone at a neighboring address may sign for delivery. For residential deliveries only.

Does this shipment contain dangerous goods? One box must be checked.

- [x] No
- [ ] Yes As per attached Shipper's Declaration.
- [ ] Yes Shipper's Declaration not required.
- [ ] Dry Ice Dry Ice, 9, UN 1845 ____ x ____ kg
- [ ] Cargo Aircraft Only

Dangerous goods (including dry ice) cannot be shipped in FedEx packaging or placed in a FedEx Express Drop Box. Restrictions apply for dangerous goods — see the current FedEx Service Guide.

7 Payment Bill to:

Enter FedEx Acct. No. or Credit Card No. below.

- [x] Sender Acct. No. in Section 1 will be billed.
- [ ] Recipient
- [ ] Third Party
- [ ] Credit Card
- [ ] Cash/Check
- [ ] Obtain recip. Acct. No.

[redacted]

Total Packages | Total Weight

Credit Card Auth.

CUSTODY SEAL

Project Name: Manifold 224 Excavation Project Number: 49/16-1092.04

Date: 6-22-16 Initials: MJR Signature: Michael Rowe Container # 1 of 1

BARR

JUN 10 10:30A OVERNIGHT 49424 MI-US GRR

fedex.com 1.800.GoFedEx 1.800.463.3339

ALS Group USA, Corp

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **24-Jun-16 10:00**

Work Order: **16061473** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 24-Jun-16 (Date) Reviewed by: Tom Bramish (eSignature) 24-Jun-16 (Date)

Matrices: Soil

Carrier name: FedEx

Shipping container/cooler in good condition?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on shipping container/cooler?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on sample bottles?	Yes ☐	No ☐	Not Present ☑
Chain of custody present?	Yes ☑	No ☐	
Chain of custody signed when relinquished and received?	Yes ☑	No ☐	
Chain of custody agrees with sample labels?	Yes ☑	No ☐	
Samples in proper container/bottle?	Yes ☑	No ☐	
Sample containers intact?	Yes ☑	No ☐	
Sufficient sample volume for indicated test?	Yes ☑	No ☐	
All samples received within holding time?	Yes ☑	No ☐	
Container/Temp Blank temperature in compliance?	Yes ☑	No ☐	
Sample(s) received on ice?	Yes ☑	No ☐	
Temperature(s)/Thermometer(s):	5.2/5.2 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	6/24/2016 11:38:44 AM		
Water - VOA vials have zero headspace?	Yes ☐	No ☐	No VOA vials submitted ☑
Water - pH acceptable upon receipt?	Yes ☐	No ☐	N/A ☑
pH adjusted?	Yes ☐	No ☐	N/A ☑
pH adjusted by:	_		

Login Notes:

Client Contacted: Date Contacted: Person Contacted:

Contacted By: Regarding:

Comments:

CorrectiveAction:

VONCO

Vonco V Waste Management Campus
100 West Gary Street
Duluth, MN 55808
Permit: SW 536

16-065-I ENB SUP Terminal MLE Manifold

Date	Ticket	Customer	Truck	Material	Tons
07/19/2016	276886	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	18.09
07/19/2016	276894	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	12.95
07/19/2016	276906	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	13.29
				Total Tons	***44.33***
				Total Loads	***3***