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BARR

Technical Memorandum

To: Alex Smith, Enbridge Energy
From: Ryan Erickson
Subject: Superior Terminal Tank 10 Line Response
WDNR SERTS ID: 20180726NO16-1
Date: January 14, 2019
Project: 49161374.04 019 001

This memorandum summarizes the environmental response activities performed by Barr Engineering (Barr) at the request Enbridge Energy (Enbridge) following the discovery of a Tank 10 line subsurface crude oil release within the tank containment basin at the Enbridge Superior Terminal (Terminal) in Superior, Wisconsin (Figure 1).

Background

On July 26, 2018, Enbridge personnel discovered evidence of a crude oil release on the ground surface in the western corner of the Tank 10 containment basin (Figure 2). Enbridge Pipe Line Maintenance (PLM) personnel immediately responded to the site and excavated the Tank 10 pipeline (Photos 1 and 2). PLM discovered a small hole in the bottom of the line near the bend in the pipe. PLM immediately initiated product capture and recovery efforts (Photos 3 and 4) and began the installation of a sleeve over the section of pipeline with the hole. When the release point was excavated, Enbridge estimated that leak rate was approximately 0.5 gallons of oil per minute and that approximately 2 barrels had been released. Note that the pre-excavation release rate may have been limited by the presence of the fat clay soil in this location. Free-product released from the hole during repair activities was captured in a plastic basin, recovered with a vacuum truck, and injected back into the pipeline system. Contaminated soil was excavated and stockpiled at the Superior Terminal Soil Management Area (SMA) until off-site disposal was coordinated, as discussed in the *Material Management* section of this memo.

Enbridge Environment was notified upon discovery of the release. On July 26, 2018, Enbridge requested Barr's assistance with the following activities:

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

The Wisconsin Department of Natural Resources (WDNR) was notified on July 26, 2018 (Attachment A) and Spills Electronic Reporting and Tracking System (SERTS) number # 20180726NO16-1 was assigned to the site.

Field Activities

Barr was on site July 26, July 30, August 20, and November 14, 2018 to complete the field activities listed above. Barr documented PLM response activities on July 26. Barr returned to the site on July 30, August 20, and November 14 to document conditions in the excavation and the presence of residual impacts.

On July 26, July 30, and November 14, Barr used soil field screening and sampling methods to document the environmental conditions in the stockpiles and the excavation, as described in the WDNR Enbridge Superior Terminal Site Investigation and Response Action Plan (SI/RAP) (2014).

Field screening samples were tested for the presence of organic vapors using a 10.6eV photoionization detector (PID). Samples were also inspected for the presence of other potential indicators of petroleum impacts such as odor, discoloration and sheen. The PID readings and physical observations were documented on a site investigation field sampling and screening log (Attachment B). Soil with headspace readings greater than 10 parts per million (ppm) or presenting other evidence of hydrocarbon contamination (e.g., hydrocarbon odor, sheen, the presence of free product) were considered impacted.

Analytical samples *TK10-B* and *TK10-S* were collected from the final excavation extents on November 14 to document residual analyte concentrations. *TK10-B* was collected beneath the eastern end of the exposed pipeline and *TK10-S* was collected from the direct contact zone. The samples were submitted to the ALS Laboratory (ALS) in Holland, Michigan for analysis of Petroleum Volatile Organic Compounds (PVOCs) plus Naphthalene. The laboratory results are summarized in Table 1 and the lab report is provided in Attachment C. Waste characterization analytical samples *TK10-Stockpile-1 and TK10-Stockpile-2* were collected on July 26 from the impacted soil stockpile for landfill disposal purposes (Attachment D). The analytical samples were submitted to ALS for analysis of benzene, toluene, ethyl benzene, and xylenes (BTEX) and diesel range organics (DRO).

Results

On July 26, July 30, August 20, and November 14, 2018, Barr documented the conditions at the site. Soil with residual crude oil impacts was observed beneath the ends of the exposed pipeline in July and August (Photo 6); therefore additional remedial excavation was requested. The final remedial excavation was completed in November. The final excavation was approximately 50 feet long by 25 feet wide by up to 10 feet deep (Photos 7 and 8; Figure 2; Attachment B). Soil in the excavation bottom and sidewalls was a fat clay. Groundwater was not observed in the excavation.

Barr collected thirteen field screening soil samples (*R-1* through *R-13*) from the excavation stockpiles on July 26. Soil from the contaminated stockpiles had headspace readings up to 555 parts per million (ppm) and product was observed. Soil from the clean stockpile had headspace readings below 10 ppm and no other evidence of contamination was identified.

Barr collected eight field screening soil samples from the excavation sidewalls and bottom on July 30 (*B-1*, *S-1* through *S-7*) and eleven samples on November 14 (*B-1* through *B-3*, *S-1* through *S-8*). Headspace readings from soil from the excavation extents were between 0.5 and 5.7 ppm. Other evidence of hydrocarbon impacts (e.g., odor, product), with the exception of a light petroleum odor in sample *B-1* (11/14/2018).

Analyte concentrations in the confirmation samples *TK10-B* and *TK10-S* were below the WDNR Industrial Direct Contact Residual Contaminant Levels (RCLs) and the WDNR Groundwater RCLs, with the exception of sample *TK10-B* that had a benzene concentration (0.15 mg/kg) that exceeded the Groundwater RCL (0.0051 mg/kg). The laboratory results are summarized in Table 1 and the ALS report is provided in Attachment C.

Based on the field screening results and the field observations, remedial excavation activities were concluded and the excavation was backfilled with clean fill material.

Receptor Survey

No direct contact risks were identified based on field screening and analytical sampling results. No impacts to surface water were identified and there is little risk for future impacts based on the location of the site within the tank containment berm. There are no potential vapor receptors within 300 feet of the site and therefore the risk of hazardous vapor accumulation is low. Employees on site are also required to wear four-gas detectors that would alert them to a potentially hazardous atmosphere.

Groundwater at the Superior Terminal is sampled on a bi-annual basis from the terminal's monitoring well network and Enbridge will conduct its next sampling event in the spring of 2019. The monitoring results are submitted to WDNR. The nearest downgradient monitoring well is MW-20 which is located 430 feet south of the release site.

Material Management

During excavation activities, soil with evidence of hydrocarbon impacts was segregated from soil without identified impacts. Characterization soil samples *TK10-Stockpile-1* and *TK10-Stockpile-2* were collected from the impacted stockpile and submitted to ALS for analysis of BTEX and DRO. Enbridge submitted the waste characterization sample data to the VONCO V landfill in Duluth, Minnesota and requested permission to manage the soil at that facility. The soil disposal request was approved and 155.63 tons of soil were hauled to the facility in August 2018. The waste characterization laboratory report, the profile application, the landfill approval letter, and a landfill activity summary report are provided in Attachment D.

Discussion and Recommendations

The Tank 10 Line crude oil release was reported to the WDNR based on the estimated release volume (2 barrels). Upon discovery, Enbridge immediately initiated repair and remediation activities. Based on analytical sampling, there is no evidence of residual soil contamination exceeding WDNR Direct Contact Zone RCL criteria. Some residual soil contamination exceeding the Groundwater RCL criteria for benzene remains below the direct contact zone at the base of the excavation (8 feet below ground surface). Additional excavation of this material was not feasible due to the presence of Terminal infrastructure. The excavation was backfilled with clean fill.

There is no identified risk to direct contact, surface water, or vapor receptors associated with the residual contamination from this release. The risk to groundwater from the residual contamination will be addressed through the facility-wide hydrogeologic performance standard established for the Superior Terminal.

The WDNR will be notified of any identified change in environmental conditions at the site. As part of this hydrogeologic performance standard Enbridge will continue to monitor groundwater conditions of the facility and, if evidence of contamination is identified, it will be reported to the WDNR and managed in accordance with the approved *Facility-wide SI/RAP* and *Addendum*.

Based on the *Facility-Wide SI/RAP* and *Addendum* site classification and the conditions encountered in the field, the pathway to site closure will be to transfer the site to the Superior Terminal Facility-Wide Site (BRRTS#: 02-16-560657) and no additional response or investigation will be required. Upon WDNR approval, Enbridge will prepare an *Enbridge Superior Terminal Facility-Wide Continuing Obligations GIS Registry Update* and submit it, along with associated fees, to facilitate the modification of the facility-wide continuing obligation registry.

Attachments:

Site Photos	1 through 8
Figure 1	Site Location
Figure 2	Site Layout
Figure 3	Receptor Survey
Attachment A	WDNR SERTS Communication
Attachment B	Site Investigation Field Sampling and Screening Logs
Attachment C	ALS Laboratory Report
Attachment D	Material Management Documents

Site Photos

Photo 1

Photo 2

Photo 1: Release response excavation location in the western corner of the Tank 10 containment basin. Photo taken facing northeast on 7/26/2018.
Photo 2: Release response excavation. Photo taken facing northeast on 7/26/2018.

Photo 3

Photo 4

Photo 3: Tank 10 pipeline with a product recovery basin (beneath pipe bend) and vacuum hose (bottom left to center of photo) and hydrocarbon impacted soil (black staining as shown in Photo 4). The release point is near the bend in the pipe. Photo taken facing southwest on 7/26/2018.
Photo 4: Hydrocarbon impacted soil (black staining) and a product recovery basin and vacuum hose beneath pipeline release location. Photo taken facing southwest on 7/26/2018.

Photo 5

Photo 6

Photo 5: Pipeline repairs (white section near bend) and the northern excavation sidewall. Photo taken facing north on 7/30/2018.
Photo 6: Residual hydrocarbon impacted soil near white oil absorbent pads beneath the Tank 10 pipeline. Note that the black media located away from the pads is blasting sand and not hydrocarbon impacts. Photo taken facing east on 8/20/2018.

Photo 7

Photo 8

Photo 7: Final excavation extents. Photo taken facing east on 11/14/2018.
Photo 8: Bottom of the final project and remedial excavation. No residual hydrocarbon impacts were observed. Photo taken facing northeast on 11/14/2018.

TABLE 1

Soil Sample Analytical Results

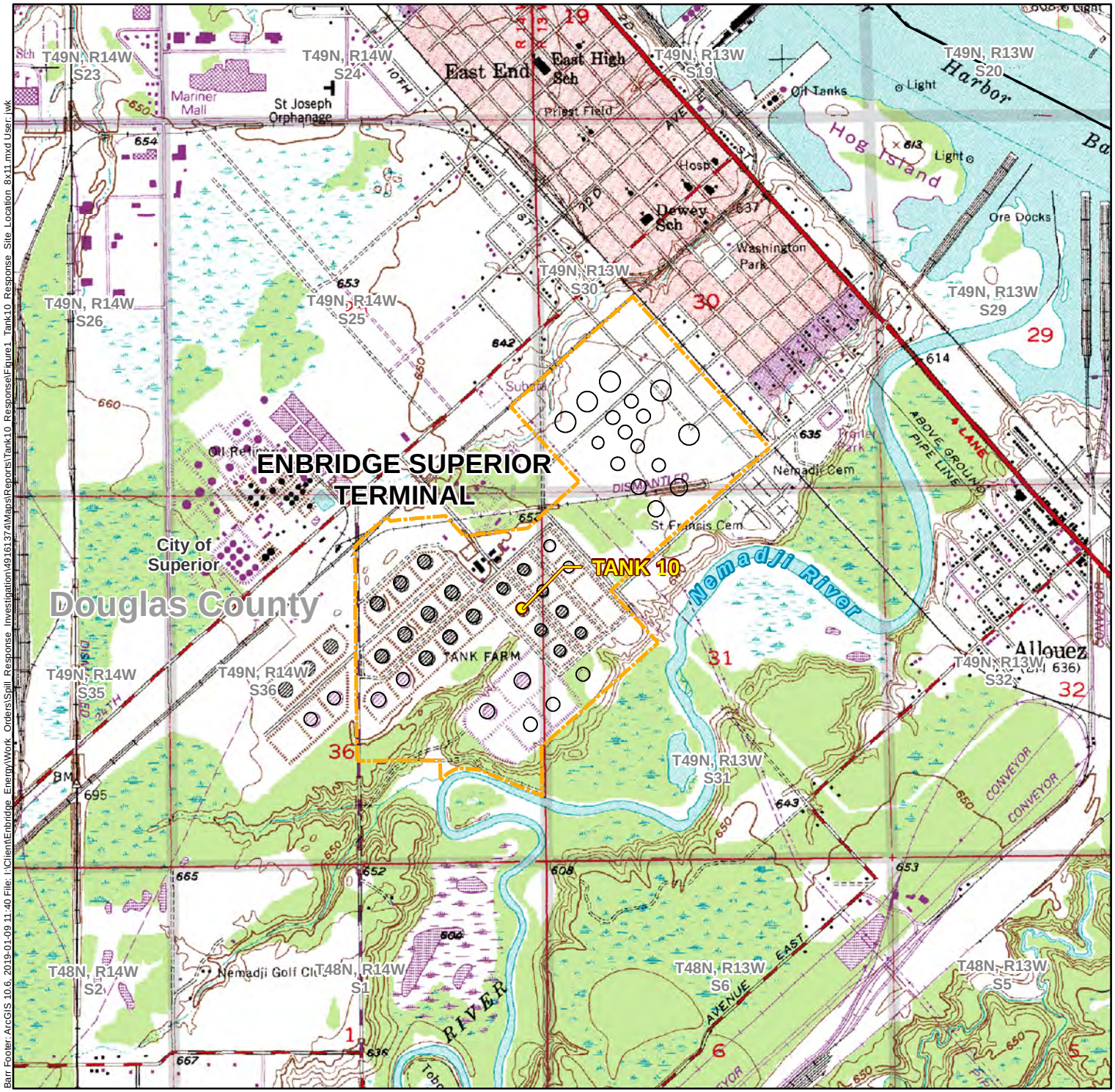
Tank 10 Line Response

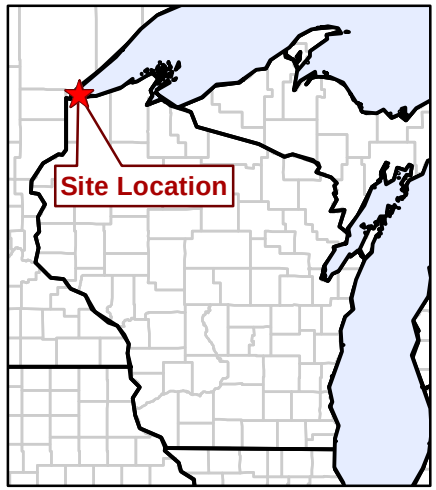
Enbridge Superior Terminal, Superior, WI

(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
Groundwater RCLs	Exceedance = **BOLD**		*1.3793*	*1.3793*	***0.0051***	*0.785*	*0.5536*	*1.97*	*0.3294*
Industrial DC RCLs	No Exceedance		*219*	*182*	*7.41*	*37*	*818*	*258*	*26*
Excavation Samples									
TK10-B	11/14/18	8	0.018	<0.0093	**0.15**	0.0091	0.034	<0.026	0.029
TK10-S	11/14/18	2	<0.0056	<0.0092	<0.0051	<0.0063	<0.0082	<0.026	<0.0083

BOLD = Analyte detections exceeding WDNR Groundwater RCLs.





Site Location

Tank 10

Terminal Property Boundary

N

0 2,000 4,000

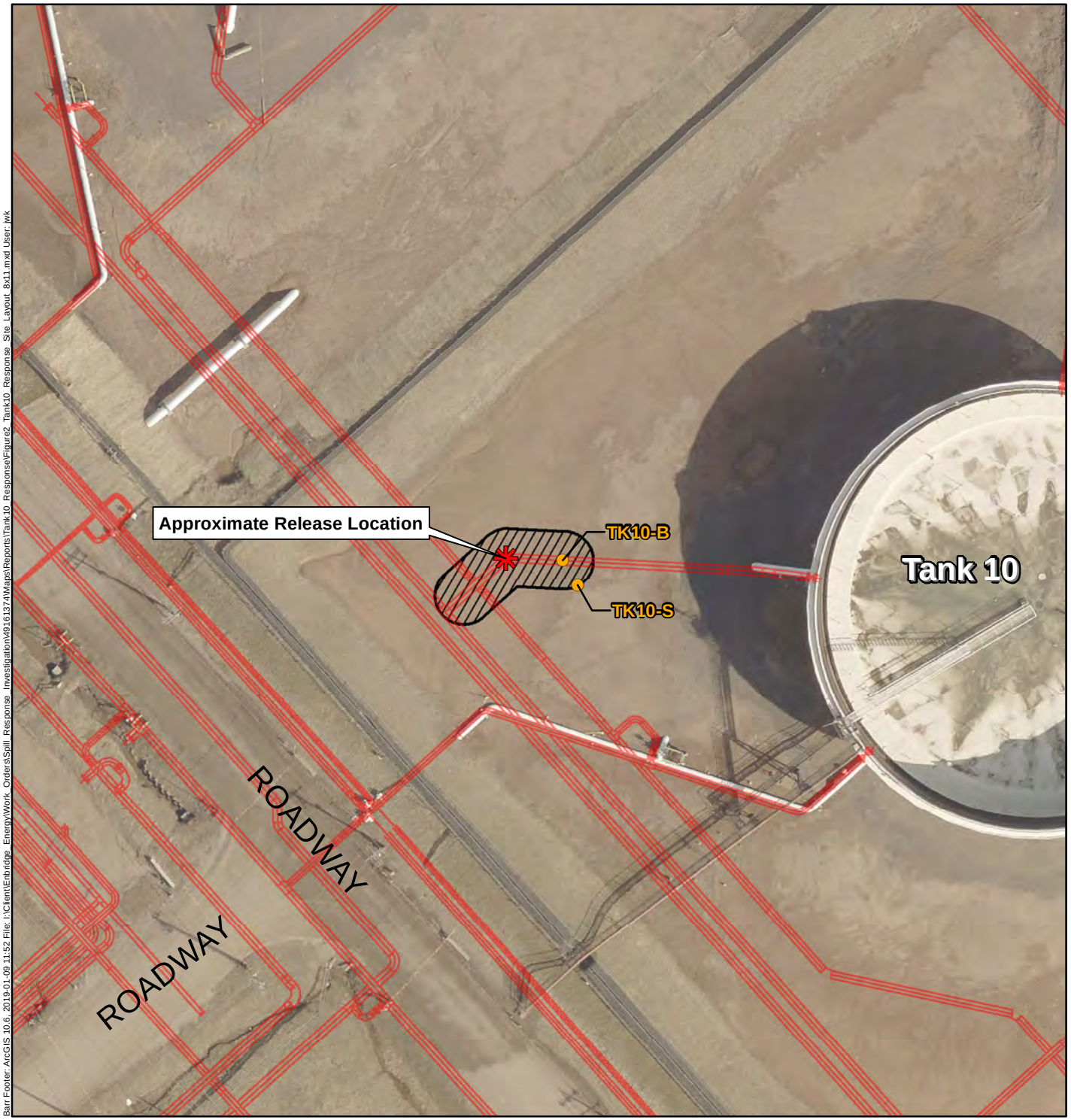
Feet

1 Inch = 2,000 Feet

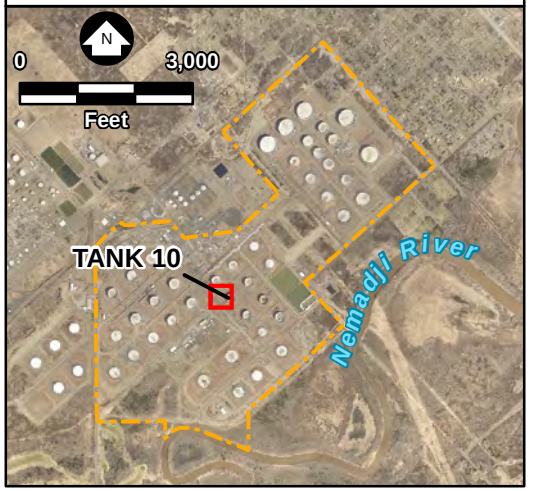
Figure 1

SITE LOCATION
TANK 10 RESPONSE
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE



ENBRIDGE SUPERIOR TERMINAL



- Approximate Release Location
- Analytical Sample Locations
- Excavation Extent; 11/14/2018
- Pipeline Infrastructure
- Terminal Property Boundary

N

0 50 100

Feet

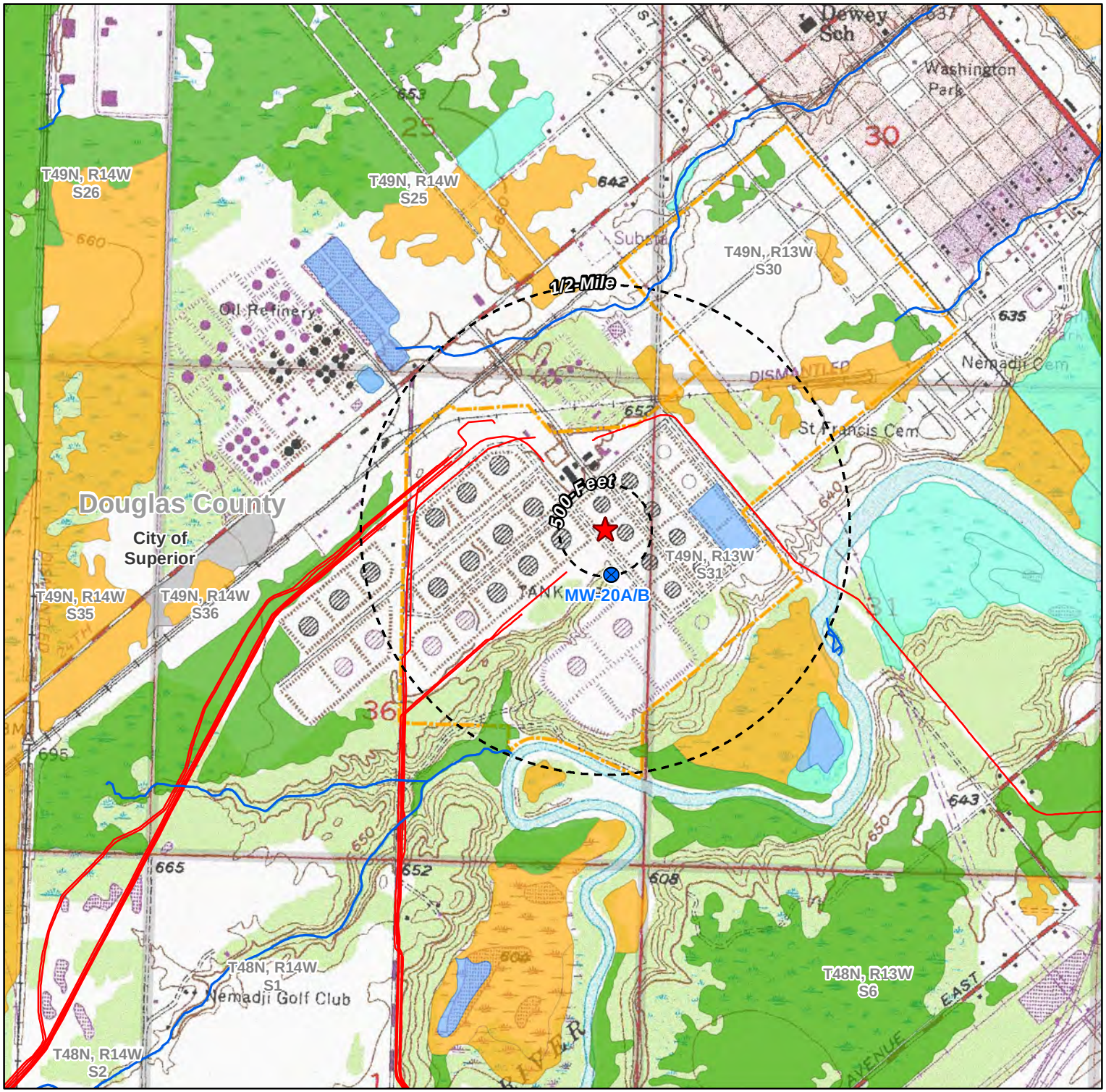
1 Inch = 50 Feet

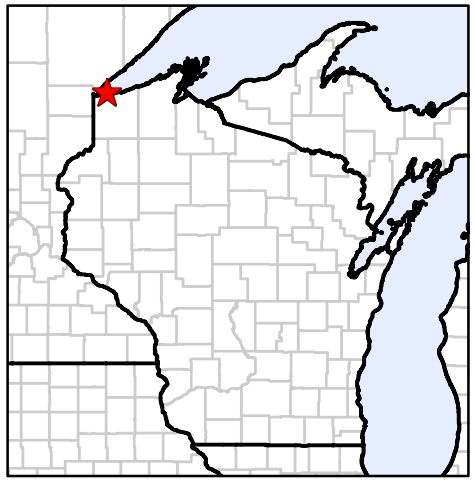
Douglas County Imagery Circa May, 2016

Figure 2

SITE LAYOUT
TANK 10 RESPONSE
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE





Site Location

Enbridge Monitoring Well

Receptor Buffers

Enbridge Pipelines

Terminal Property Boundary

Watercourses

Wisconsin Wetland Inventory

Emergent/wet meadow

Filled/drained wetland

Forested

Open Water

Scrub/Shrub

N

0 1,500 3,000

Feet

1 Inch = 1,500 Feet

Figure 3

RECEPTOR SURVEY
TANK 10 RESPONSE
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE

Barr Footer: ArcGIS 10.6, 2019-01-10 13:26 File: I:\Client\Enbridge_Energy\Work_Orders\Spill_Response_Investigation\49161374\Maps\Reports\Tank10_Response\Figure3_Tank10_Response_Receptor.mxd User: jwk

Attachment A

WDNR SERTS Communication

Ryan E. Erickson

From: Alex Smith <alex.smith@enbridge.com>
Sent: Monday, January 07, 2019 10:12 AM
To: Ryan E. Erickson
Subject: FW: Response Report Updates

Hi Ryan,

Below is the info from WDNR for Tank 10.

Thanks,

Alex Smith
Environmental Advisor, LP US Environment Operations
—
ENBRIDGE
TEL: **715-395-3836**| FAX: 832-325-5511 | CELL: 715-817-8322
119 North 25th Street East, Superior, WI 54880

enbridge.com
Integrity. Safety. Respect.

From: Rahn, Matthew W - DNR [mailto:Matthew.Rahn@wisconsin.gov]
Sent: Monday, January 07, 2019 10:04 AM
To: Alex Smith
Subject: [External] RE: Response Report Updates

Alex,

Below is the reporting information from our SERTS database. Other than that, everything I have in our file is stuff that you sent.

SERTS ID:
20180726NO16-1

Reported:
07/26/2018 12:28

Occurred:
07/26/2018 11:30

Reported by:
TERRI PICTON
COMPLIANCE ADVISOR
ENBRIDGE PIPELINE
theresa.picton@enbridge.com
(715) 718-1208
Also RP Contact

Location:

NO REGION
DOUGLAS COUNTY
SUPERIOR, CITY OF
TERMINAL
2800 E 21ST
SPILL LOCATION

Responsible Party:
ENBRIDGE PIPELINE
119 N 25TH ST E
SUPERIOR, WI 54880
(715) 718-1208

Substance:
CRUDE OIL [CRUDE OIL]
Released Amt: 2 bbl
Recovered Amt: 2 bbl
(Amounts are often estimated)

Cause:
EQUIPMENT FAILURE

Cause Description:
A FITTING ON ONE OF THE ELBOW PIPES FAILED, AND CAUSE THE OIL TO LEAK OUT OF THE PIPE.

Environmental Impact:
CONTAMINATED SOIL.

Weather:
CLOUDY

Contractor:
N/A

Cleanup:
EXCAVATED THE CONTAMINATED THE SOIL, CONTINUE CLEAN UP EFFORTS, TESTING SOIL AND DISPOSING OF SOIL.

Notified MATT RAHN at 12:36 by

Submitted by:
COREY POSTHUMA
(608) 267-7691
coreyl.posthuma@wisconsin.gov

Matthew W. Rahn
Phone: (715) 623-4190 Ext. 3110
Cell Phone: (715) 350-1121
Matthew.Rahn@wisconsin.gov

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Attachment B

Site Investigation Field Sampling and Screening Logs

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Page 1 of 1

Client: Enbridge Energy Date: 7.26.2018

Location: TK 10, Supt Sampled by: TTS

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

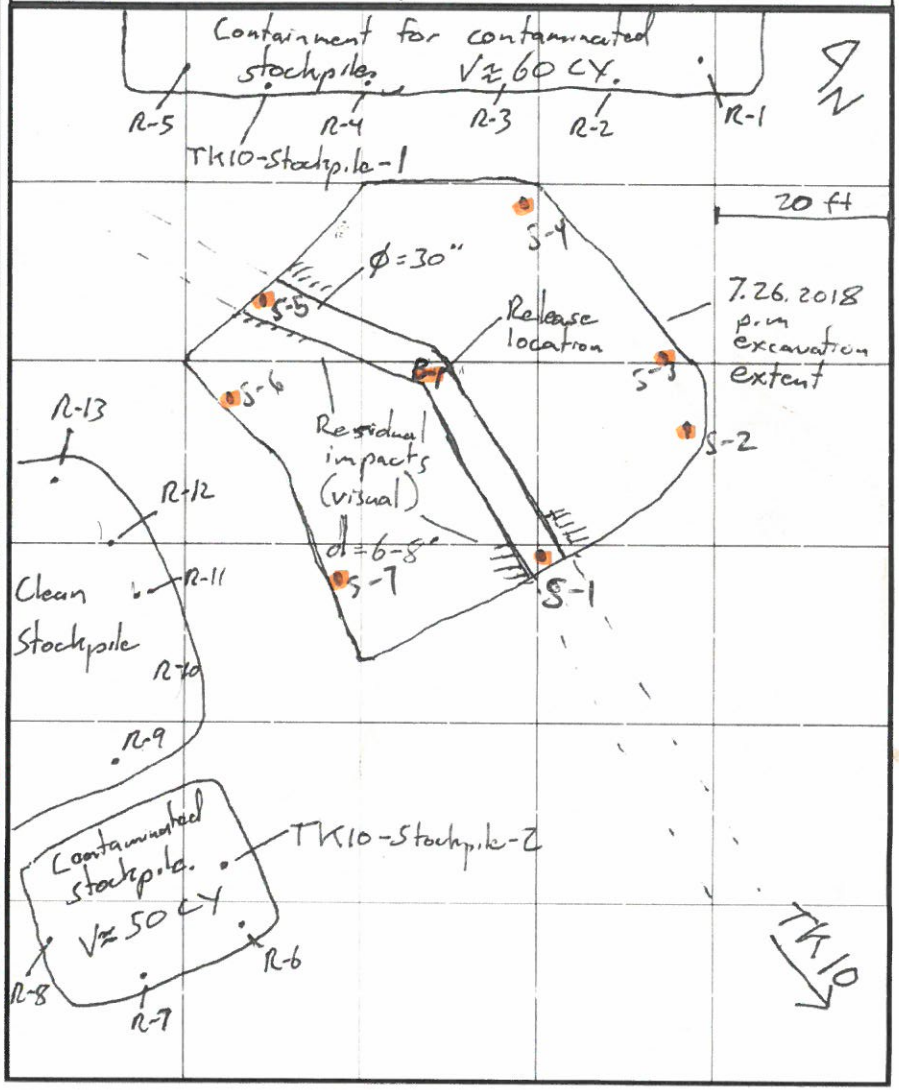
R = Removed **S** = Sidewall **B** = Bottom **Stockpile** = Stockpile

Sample ID	Depth (FT)	Time (military)	Soil Type (USCS)	Color/ Discolor	Odor/ Sheen	Headspace Reading (ppm)
Example: Stockpile-1	*4*	*16:30*	*CL*	*Reddish brown*	*Petroleum/ Rainbow*	*275*
R-1	-	1250	CH	Reddish brown/ blk	Petro/ Rainbow	238.7
R-2	-			"		54.3
R-3	-			brn		230.8
R-4	-			"		555.8
R-5	-			-	Faint/N	10.8
R-6	-	1255			N/N	4.5
R-7	-			brn	Petro/ Rainb	180.6
R-8	-			blk		317.8
R-9	-	1300		N	N/N	3.0
R-10	-			N	N/N	3.4
R-11	-			N	N/N	2.7
R-12	-			N	N/N	3.5
R-13	-			N	N/N	2.9
7/30/18	MAB					
B-1	10	1435	CH	red/N	N/N	2.6
S-1	1-2	1500				1.3
S-2						2.7
S-3						3.7
S-4						3.1
S-5						5.7
S-6						5.3
S-7						2.3

INSTRUMENT: Photoionization detector with 10.6 eV bulb

	Calibration	Bump Test 1	Bump Test 2
Time	1245	1430	1500 7/30
Zero reading (ppm)	0.0	0.0	0.0
Span reading (ppm)	100.0	106.8	97.7
Background (ppm)	0.1	0.1	0.1

SITE SKETCH : *north arrow, scale, excavation extents & depths, impacted areas, sample locations ,borings, wells, structures, utilities, natural features...*



SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Location: *Milepost or Facility* Superior Terminal TK10 Response

Equipment used: Photo -*ionization detector with* 10.6 *eV lamp* Background Headspace: 0.2 ppm

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

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

Date: 11/14/18

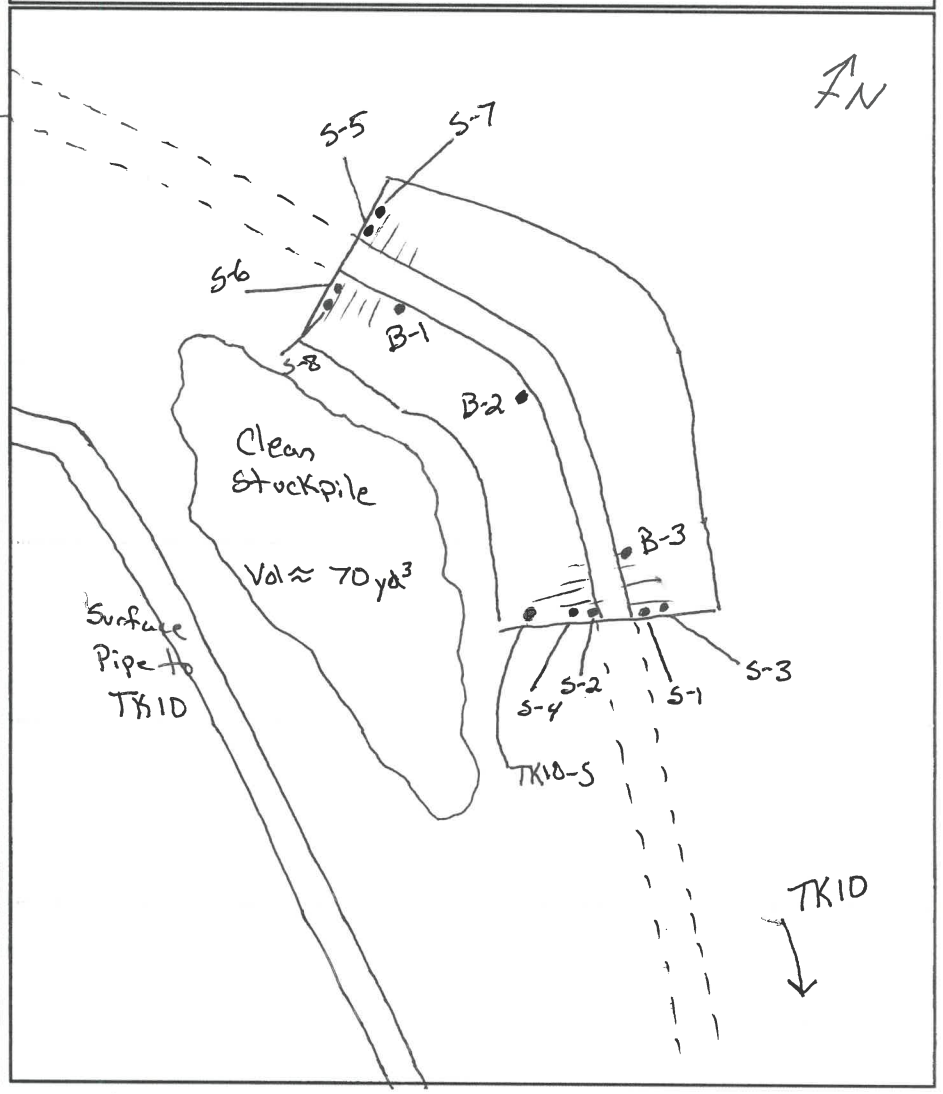
Sampler: PLL

Calibration Time: 0620

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	1-3	0840	CH	Reddish Brown/none	None/none	0.7
S-2	1-3	"	"	"	"	0.5
S-3	6-8	"	"	"	"	0.5
S-4	6-8	"	"	"	"	0.6
S-5	1-3	0905	CH	"	"	2.3
S-6	1-3	"	"	"	"	2.4
S-7	6-8	"	"	"	"	1.7
S-8	6-8	"	"	"	"	0.9
B-1	8-10	0935	CH	Reddish brown/none	Slight Petro/none	2.9
B-2	"	"	"	"	none/none	1.6
B-3	"	"	"	"	N/N	1.7
TK10-B		10:15	Sample location			
TK10-S		10:10	Sample location			

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



Attachment C

ALS Laboratory Report

27-Nov-2018

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

Re: **Tank 10 Response (494161374.04)** Work Order: **18111275**

Dear Ryan,

ALS Environmental received 2 samples on 17-Nov-2018 for the analyses presented in the following report.

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

Sample results are compliant with industry accepted practices and Quality Control 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 11.

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

ADDRESS: 3352 128th Avenue, Holland, MI, USA
PHONE: +1 (616) 399-6070 FAX: +1 (616) 399-6185

Sincerely,

Ehrland Bosworth

Electronically approved by: Ehrland Bosworth

Ehrland Bosworth
Project Manager

Report of Laboratory Analysis

Certificate No: WI: 399084510

ALS Group, USA

Date: 27-Nov-18

Client: Barr Engineering Company
Project: Tank 10 Response (494161374.04)
Work Order: **18111275**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
18111275-01	TK10-B	Soil		11/14/2018 10:15	11/17/2018 09:30	☐
18111275-02	TK10-S	Soil		11/14/2018 10:10	11/17/2018 09:30	☐

ALS Group, USA

Date: 27-Nov-18

Client: Barr Engineering Company
Project: Tank 10 Response (494161374.04)
WorkOrder: **18111275**

QUALIFIERS, ACRONYMS, UNITS

Qualifier	Description
*	Value exceeds Regulatory Limit
**	Estimated Value
a	Analyte is non-accredited
B	Analyte detected in the associated Method Blank above the Reporting Limit
E	Value above quantitation range
H	Analyzed outside of Holding Time
Hr	BOD/CBOD - Sample was reset outside Hold Time, value should be considered estimated.
J	Analyte is present at an estimated concentration between the MDL and Report Limit
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-dry	Micrograms per Kilogram Dry Weight

Client: Barr Engineering Company
Project: Tank 10 Response (494161374.04)
Work Order: 18111275

Case Narrative

Samples for the above noted Work Order were received on 11/17/18. 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. A copy of the laboratory's scope of accreditation is available upon request.

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

Date: *27-Nov-18*

Client: Barr Engineering Company
Project: Tank 10 Response (494161374.04)
Sample ID: TK10-B
Collection Date: 11/14/2018 10:15 AM

Work Order: 18111275
Lab ID: 18111275-01
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 11/19/18		Analyst: **WH**
1,2,4-Trimethylbenzene	18	J	5.6	30	µg/Kg-dry	1	11/20/2018 12:29
1,3,5-Trimethylbenzene	U		9.3	30	µg/Kg-dry	1	11/20/2018 12:29
Benzene	**150**		**5.2**	**30**	**µg/Kg-dry**	1	11/20/2018 12:29
Ethylbenzene	9.1	J	6.4	30	µg/Kg-dry	1	11/20/2018 12:29
m,p-Xylene	21	J	14	61	µg/Kg-dry	1	11/20/2018 12:29
Naphthalene	29	J	8.4	100	µg/Kg-dry	1	11/20/2018 12:29
o-Xylene	U		12	30	µg/Kg-dry	1	11/20/2018 12:29
Toluene	34		8.3	30	µg/Kg-dry	1	11/20/2018 12:29
Xylenes, Total	U		26	91	µg/Kg-dry	1	11/20/2018 12:29
Surr: 1,2-Dichloroethane-d4	*97.8*			*70-130*	*%REC*	1	11/20/2018 12:29
Surr: 4-Bromofluorobenzene	*104*			*70-130*	*%REC*	1	11/20/2018 12:29
Surr: Dibromofluoromethane	*94.8*			*70-130*	*%REC*	1	11/20/2018 12:29
Surr: Toluene-d8	*90.2*			*70-130*	*%REC*	1	11/20/2018 12:29
MOISTURE		Method: **SW3550C**					Analyst: **TRP**
Moisture	**28**		**0.025**	**0.050**	**% of sample**	1	11/26/2018 11:26

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

ALS Group, USA

Date: *27-Nov-18*

Client: Barr Engineering Company
Project: Tank 10 Response (494161374.04)
Sample ID: TK10-S
Collection Date: 11/14/2018 10:10 AM

Work Order: 18111275
Lab ID: 18111275-02
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 11/19/18		Analyst: **AK**
1,2,4-Trimethylbenzene	U		5.6	30	µg/Kg-dry	1	11/21/2018 20:45
1,3,5-Trimethylbenzene	U		9.2	30	µg/Kg-dry	1	11/21/2018 20:45
Benzene	U		5.1	30	µg/Kg-dry	1	11/21/2018 20:45
Ethylbenzene	U		6.3	30	µg/Kg-dry	1	11/21/2018 20:45
m,p-Xylene	U		14	60	µg/Kg-dry	1	11/21/2018 20:45
Naphthalene	U		8.3	100	µg/Kg-dry	1	11/21/2018 20:45
o-Xylene	U		12	30	µg/Kg-dry	1	11/21/2018 20:45
Toluene	U		8.2	30	µg/Kg-dry	1	11/21/2018 20:45
Xylenes, Total	U		26	90	µg/Kg-dry	1	11/21/2018 20:45
Surr: 1,2-Dichloroethane-d4	*110*			*70-130*	*%REC*	1	11/21/2018 20:45
Surr: 4-Bromofluorobenzene	*94.6*			*70-130*	*%REC*	1	11/21/2018 20:45
Surr: Dibromofluoromethane	*87.0*			*70-130*	*%REC*	1	11/21/2018 20:45
Surr: Toluene-d8	*95.7*			*70-130*	*%REC*	1	11/21/2018 20:45
MOISTURE		Method: **SW3550C**					Analyst: **TRP**
Moisture	**21**		**0.025**	**0.050**	**% of sample**	1	11/26/2018 11:26

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

ALS Group, USA

Date: 27-Nov-18

Client: Barr Engineering Company

QC BATCH REPORT

Work Order: 18111275

Project: Tank 10 Response (494161374.04)

Batch ID: **128250** Instrument ID **VMS7** Method: **SW8260C**

MBLK Sample ID: **MBLK-128250-128250** Units: **µg/Kg-dry** Analysis Date: **11/19/2018 11:42 P**

Client ID: Run ID: **VMS7_181119B** SeqNo: **5396028** Prep Date: **11/19/2018** 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	5.6	30								
1,3,5-Trimethylbenzene	U	9.2	30								
Benzene	U	5.1	30								
Ethylbenzene	U	6.3	30								
m,p-Xylene	U	14	60								
Naphthalene	U	8.3	100								
o-Xylene	U	12	30								
Toluene	U	8.2	30								
Xylenes, Total	U	26	90								
Surr: 1,2-Dichloroethane-d4	*972*	0	0	*1000*	0	*97.2*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1009*	0	0	*1000*	0	*101*	*70-130*	0			
Surr: Dibromofluoromethane	*952*	0	0	*1000*	0	*95.2*	*70-130*	0			
Surr: Toluene-d8	*898*	0	0	*1000*	0	*89.8*	*70-130*	0			

LCS Sample ID: **LCS-128250-128250** Units: **µg/Kg-dry** Analysis Date: **11/19/2018 10:40 P**

Client ID: Run ID: **VMS7_181119B** SeqNo: **5396027** Prep Date: **11/19/2018** 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	935.5	5.6	30	1000	0	93.6	65-135	0			
1,3,5-Trimethylbenzene	970.5	9.2	30	1000	0	97	65-135	0			
Benzene	1008	5.1	30	1000	0	101	75-125	0			
Ethylbenzene	943	6.3	30	1000	0	94.3	75-125	0			
m,p-Xylene	1852	14	60	2000	0	92.6	80-125	0			
Naphthalene	926.5	8.3	100	1000	0	92.6	40-140	0			
o-Xylene	957	12	30	1000	0	95.7	75-125	0			
Toluene	896.5	8.2	30	1000	0	89.6	70-125	0			
Xylenes, Total	2809	26	90	3000	0	93.6	75-125	0			
Surr: 1,2-Dichloroethane-d4	*957*	0	0	*1000*	0	*95.7*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1032*	0	0	*1000*	0	*103*	*70-130*	0			
Surr: Dibromofluoromethane	*1022*	0	0	*1000*	0	*102*	*70-130*	0			
Surr: Toluene-d8	*908*	0	0	*1000*	0	*90.8*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 18111275
Project: Tank 10 Response (494161374.04)

QC BATCH REPORT

Batch ID: **128250** Instrument ID **VMS7** Method: **SW8260C**

MS Sample ID: **18111275-01A MS** Units: **µg/Kg-dry** Analysis Date: **11/20/2018 05:08 A**
Client ID: **TK10-B** Run ID: **VMS7_181119B** SeqNo: **5396030** Prep Date: **11/19/2018** 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	961	5.6	30	1000	18.26	94.3	65-135	0			
1,3,5-Trimethylbenzene	994.5	9.2	30	1000	0	99.4	65-135	0			
Benzene	1055	5.1	30	1000	148.1	90.7	75-125	0			
Ethylbenzene	959.5	6.3	30	1000	9.128	95	75-125	0			
m,p-Xylene	1910	14	60	2000	21.3	94.4	80-125	0			
Naphthalene	910	8.3	100	1000	29.41	88.1	40-140	0			
o-Xylene	980	12	30	1000	9.635	97	75-125	0			
Toluene	908	8.2	30	1000	34.48	87.4	70-125	0			
Xylenes, Total	2890	26	90	3000	21	95.6	75-125	0			
Surr: 1,2-Dichloroethane-d4	*985.5*	0	0	*1000*	0	*98.6*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1042*	0	0	*1000*	0	*104*	*70-130*	0			
Surr: Dibromofluoromethane	*1023*	0	0	*1000*	0	*102*	*70-130*	0			
Surr: Toluene-d8	*921*	0	0	*1000*	0	*92.1*	*70-130*	0			

MSD Sample ID: **18111275-01A MSD** Units: **µg/Kg-dry** Analysis Date: **11/20/2018 05:23 A**
Client ID: **TK10-B** Run ID: **VMS7_181119B** SeqNo: **5396031** Prep Date: **11/19/2018** 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	848.5	5.6	30	1000	18.26	83	65-135	961	12.4	30	
1,3,5-Trimethylbenzene	844.5	9.2	30	1000	0	84.4	65-135	994.5	16.3	30	
Benzene	920	5.1	30	1000	148.1	77.2	75-125	1055	13.7	30	
Ethylbenzene	830.5	6.3	30	1000	9.128	82.1	75-125	959.5	14.4	30	
m,p-Xylene	1646	14	60	2000	21.3	81.2	80-125	1910	14.8	30	
Naphthalene	831	8.3	100	1000	29.41	80.2	40-140	910	9.08	30	
o-Xylene	862.5	12	30	1000	9.635	85.3	75-125	980	12.8	30	
Toluene	794	8.2	30	1000	34.48	76	70-125	908	13.4	30	
Xylenes, Total	2508	26	90	3000	21	82.9	75-125	2890	14.1	30	
Surr: 1,2-Dichloroethane-d4	*957*	0	0	*1000*	0	*95.7*	*70-130*	985.5	*2.93*	*30*	
Surr: 4-Bromofluorobenzene	*1044*	0	0	*1000*	0	*104*	*70-130*	1042	*0.192*	*30*	
Surr: Dibromofluoromethane	*988.5*	0	0	*1000*	0	*98.8*	*70-130*	1023	*3.43*	*30*	
Surr: Toluene-d8	*907*	0	0	*1000*	0	*90.7*	*70-130*	921	*1.53*	*30*	

The following samples were analyzed in this batch:

18111275-01A	18111275-02A

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

Client: Barr Engineering Company
Work Order: 18111275
Project: Tank 10 Response (494161374.04)

QC BATCH REPORT

Batch ID: **R250025** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R250025** Units: **% of sample** Analysis Date: **11/26/2018 11:26 A**
Client ID: Run ID: **MOIST_181126A** SeqNo: **5404343** 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-R250025** Units: **% of sample** Analysis Date: **11/26/2018 11:26 A**
Client ID: Run ID: **MOIST_181126A** SeqNo: **5404342** 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: **18111169-15B DUP** Units: **% of sample** Analysis Date: **11/26/2018 11:26 A**
Client ID: Run ID: **MOIST_181126A** SeqNo: **5404302** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	19.39	0.025	0.050	0	0	0	0-0	21.72	11.3	10	R

DUP Sample ID: **18111169-19B DUP** Units: **% of sample** Analysis Date: **11/26/2018 11:26 A**
Client ID: Run ID: **MOIST_181126A** SeqNo: **5404316** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	17.62	0.025	0.050	0	0	0	0-0	17.71	0.509	10	

The following samples were analyzed in this batch: 18111275-01B 18111275-02B

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

18111275

Barr Engineering Co. Chain of Custody

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

BARR ☐ Ann Arbor ☒ Duluth ☐ Hibbing ☐ Minneapolis ☐ Bismarck ☐ Grand Rapids ☐ Jefferson City ☐ Salt Lake City

COC Number: **57726**

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company: SAME
Address: 325 S. Lake Ave.	Address:
Name: Ryan Erickson	Name:
email: REE@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: TANK 10 Response	Barr Project No: 49161374.04 019 ~~001~~

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: Moisture	Soil: PVOC-MTBE + Naphthalene	Soil: % Solids	Preservative Code	Field Filtered Y/N	Comments
										A	A				
										N	N				
1. TK10-B	–	–	–	11/14/2018	10:15	S	N	3		1	2				PVOC-MTBE +
2. TK10-S	–	–	–	11/14/2018	10:10	S	N	3		1	2				Naphthalene
3.															and Moisture
4.															
5.															
6.															
7.															
8.															
9.															
10.															

BARR USE ONLY

Sampled by: PLL

Barr Proj. Manager: REE, LMG

Barr DQ Manager: JET

Lab Name: ALS

Lab Location: Holland

Relinquished by: _____ On Ice? (Y) N Date 11/14/18 Time 12:28 Received by: FED EX

Relinquished by: FED EX On Ice? Y N Date 11/17/18 Time 0930 Received by: _____

Date / Time: _____ / _____

Date / Time: _____ / _____

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

Air Bill Number:

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

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

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

SR2 2.4; EB

H:\RLG\STDFORMS\Chain Of Custody Form 2015 RLG Rev. 01/02/18

ALS Group, USA

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **17-Nov-18 09:30**

Work Order: **18111275** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 19-Nov-18 (Date) Reviewed by: Ehrland Bosworth (eSignature) 19-Nov-18 (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):	2.4/2.4 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	11/19/2018 8:53:22 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:

Attachment D

Material Management Documents

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

VONCOUSA.com
Office: 218.626.3830
Fax: 218.626.4874

July 31, 2018

Enbridge Energy
Attn: Alex Smith
1100 Louisiana Ave. Ste 3300
Houston, TX

RE: Industrial Waste Profile: 18-075-I – Enbridge Energy Superior Terminal

Alex,

Please be advised that the above described waste material is acceptable for disposal of up to **1,000 cubic yards** at the Vonco V Waste Management Campus in Duluth, MN. The waste material is acceptable per the Vonco V Duluth, LLC (SW-536) Minnesota Pollution Control Agency approved Industrial Solid Waste Management Plan.

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. The term of the approval is 3 years and will expire on 7/31/2021.

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 The 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 at: 651-260-6107.

We look forward to working with you,

Aric Olsen
Environmental Manager

VONCO V, LLC.

Industrial Waste Profile Sheet

PROFILE# _____

Designated Facility: Vonco V, LLC.

Permit #536

A. Generator, Waste Site Location

Name	Enbridge Energy Superior Terminal
Site Address	2800 E 21st St
City, State, Zip	Superior, WI, 54880
Contact	Alex Smith
Phone	715-395-3836
Fax	832-325-5511
County	Douglas

B. Billing

Name	Enbridge Energy
Site Address	1100 Louisiana Ave, Ste 3300
City, State, Zip	Houston, TX, 77002
Contact	Alex Smith
Phone	715-398-4795
Fax	832-325-5511

C. Description of Waste

Name of Waste: Hydrocarbon Contaminated Soil - Superior Tank 10 Line

Process Generating Waste: Hydrocarbon contaminated soil from release site

Estimated Volume: 200 cubic yards

Frequency: One time

Physical State: Solid Color: Reddish brown Free Liquids: None

Flash Point (°F): _____ pH: _____ Total Solids: _____

D. Other Comments

E. Sample Information

Check all that apply:

☒ Laboratory Analysis submitted ☐ Material Safety Data Sheet submitted

Laboratory Name: ALS Environmental Sample Date: 7/26/18 Sample I.D.: TK 10 Stockpile -1, TK 10 Stockpile -2

F. Generator Certifications

1. This waste is not a hazardous waste as defined in Minnesota Rules Chapter 7045 or 40 CFR 261.
2. This waste does not contain regulated quantities of PCBs.
3. This waste does not contain regulated quantities of herbicides or pesticides.
4. This waste does not contain infectious wastes as defined in Minnesota Rules Chapter.
5. All information submitted in this and all attached documents contains true and accurate descriptions of this waste. Any sample submitted is representative as defined in 40 CFR 261 Appendix 1 and was obtained by using this or an equivalent sampling method. All relevant information regarding known or suspected hazards in the possession of the generator has been disclosed.

Generator's Signature _____ Title: Environmental Analyst

Print Name: Alex Smith Date: July 31, 18

G. Landfill Approval

My approval is based upon the laboratory analysis of a representative sample and/or material safety data sheets submitted by the generator.

Landfill Signature _____ Date _____

Recertification Date _____

31-Jul-2018

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

Re: **Tank 10 Response (49161374.04)** Work Order: **18071769**

Dear Ryan,

ALS Environmental received 2 samples on 27-Jul-2018 for the analyses presented in the following report.

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

Sample results are compliant with industry accepted practices and Quality Control 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:

ADDRESS: 3352 128th Avenue, Holland, MI, USA
PHONE: +1 (616) 399-6070 FAX: +1 (616) 399-6185

Sincerely,

Electronically approved by: Tom Beamish

Tom Beamish
Senior Project Manager

Report of Laboratory Analysis

ALS Group, USA

Date: 31-Jul-18

Client: Barr Engineering Company
Project: Tank 10 Response (49161374.04)
Work Order: **18071769**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
18071769-01	TK 10 Stockpile -1	Soil		07/26/18 13:15	07/27/18 09:30	☐
18071769-02	TK 10 Stockpile -2	Soil		07/26/18 13:20	07/27/18 09:30	☐

Client: Barr Engineering Company
Project: Tank 10 Response (49161374.04)
WorkOrder: **18071769**

QUALIFIERS, ACRONYMS, UNITS

Qualifier	Description
*	Value exceeds Regulatory Limit
**	Estimated Value
a	Analyte is non-accredited
B	Analyte detected in the associated Method Blank above the Reporting Limit
E	Value above quantitation range
H	Analyzed outside of Holding Time
Hr	BOD/CBOD - Sample was reset outside Hold Time, value should be considered estimated.
J	Analyte is present at an estimated concentration between the MDL and Report Limit
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-dry	Micrograms per Kilogram Dry Weight
mg/Kg-dry	Milligrams per Kilogram Dry Weight

Client: Barr Engineering Company
Project: Tank 10 Response (49161374.04)
Work Order: 18071769

Case Narrative

Samples for the above noted Work Order were received on 07/27/18. 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. A copy of the laboratory's scope of accreditation is available upon request.

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

Volatile Organics:
No deviations or anomalies were noted.

Extractable Organics:
No deviations or anomalies were noted.

Wet Chemistry:
No deviations or anomalies were noted.

ALS Group, USA

Date: *31-Jul-18*

Client: Barr Engineering Company
Project: Tank 10 Response (49161374.04)
Sample ID: TK 10 Stockpile -1
Collection Date: 07/26/18 01:15 PM

Work Order: 18071769
Lab ID: 18071769-01
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 / 7/30/18		Analyst: **RP**
DRO (C10-C28)	**290**		**5.6**	**56**	**mg/Kg-dry**	10	07/30/18 17:33
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 7/27/18		Analyst: **LSY**
Benzene	**190**		**5.1**	**30**	**µg/Kg-dry**	1	07/27/18 22:22
Ethylbenzene	**260**		**6.3**	**30**	**µg/Kg-dry**	1	07/27/18 22:22
m,p-Xylene	**1,400**		**14**	**60**	**µg/Kg-dry**	1	07/27/18 22:22
o-Xylene	**440**		**12**	**30**	**µg/Kg-dry**	1	07/27/18 22:22
Toluene	**880**		**8.2**	**30**	**µg/Kg-dry**	1	07/27/18 22:22
Xylenes, Total	**1,800**		**26**	**90**	**µg/Kg-dry**	1	07/27/18 22:22
Surr: 1,2-Dichloroethane-d4	*101*			*70-130*	*%REC*	1	07/27/18 22:22
Surr: 4-Bromofluorobenzene	*105*			*70-130*	*%REC*	1	07/27/18 22:22
Surr: Dibromofluoromethane	*86.2*			*70-130*	*%REC*	1	07/27/18 22:22
Surr: Toluene-d8	*101*			*70-130*	*%REC*	1	07/27/18 22:22
MOISTURE		Method: **SW3550C**					Analyst: **SBR**
Moisture	**25**		**0.025**	**0.050**	**% of sample**	1	07/30/18 12:08

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

ALS Group, USA

Date: *31-Jul-18*

Client: Barr Engineering Company
Project: Tank 10 Response (49161374.04)
Sample ID: TK 10 Stockpile -2
Collection Date: 07/26/18 01:20 PM

Work Order: 18071769
Lab ID: 18071769-02
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 / 7/30/18		Analyst: **RP**
DRO (C10-C28)	**970**		**5.4**	**54**	**mg/Kg-dry**	10	07/30/18 18:02
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 7/27/18		Analyst: **LSY**
Benzene	**3,600**		**5.8**	**34**	**µg/Kg-dry**	1	07/27/18 22:38
Ethylbenzene	**2,600**		**7.2**	**34**	**µg/Kg-dry**	1	07/27/18 22:38
m,p-Xylene	**16,000**		**81**	**340**	**µg/Kg-dry**	5	07/30/18 15:54
o-Xylene	**4,500**		**13**	**34**	**µg/Kg-dry**	1	07/27/18 22:38
Toluene	**13,000**		**47**	**170**	**µg/Kg-dry**	5	07/30/18 15:54
Xylenes, Total	**21,000**		**150**	**510**	**µg/Kg-dry**	5	07/30/18 15:54
Surr: 1,2-Dichloroethane-d4	*101*			*70-130*	*%REC*	1	07/27/18 22:38
Surr: 1,2-Dichloroethane-d4	*100*			*70-130*	*%REC*	5	07/30/18 15:54
Surr: 4-Bromofluorobenzene	*93.0*			*70-130*	*%REC*	1	07/27/18 22:38
Surr: 4-Bromofluorobenzene	*104*			*70-130*	*%REC*	5	07/30/18 15:54
Surr: Dibromofluoromethane	*83.0*			*70-130*	*%REC*	1	07/27/18 22:38
Surr: Dibromofluoromethane	*95.0*			*70-130*	*%REC*	5	07/30/18 15:54
Surr: Toluene-d8	*113*			*70-130*	*%REC*	1	07/27/18 22:38
Surr: Toluene-d8	*102*			*70-130*	*%REC*	5	07/30/18 15:54
MOISTURE		Method: **SW3550C**					Analyst: **SBR**
Moisture	**25**		**0.025**	**0.050**	**% of sample**	1	07/30/18 12:08

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

ALS Group, USA

Date: 31-Jul-18

Client: Barr Engineering Company

Work Order: 18071769

Project: Tank 10 Response (49161374.04)

QC BATCH REPORT

Batch ID: **122029** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK	Sample ID: **DBLKS1-122029-122029**				Units: **mg/Kg**			Analysis Date: **07/30/18 03:36 PM**			
Client ID:	Run ID: **GC8_180730A**				SeqNo: **5177155**			Prep Date: **07/30/18**		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	0.5	5.0								

LCS	Sample ID: **DLCSS1-122029-122029**				Units: **mg/Kg**			Analysis Date: **07/30/18 04:05 PM**			
Client ID:	Run ID: **GC8_180730A**				SeqNo: **5177156**			Prep Date: **07/30/18**		DF: **1**	
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	7.786	0.5	5.0	10	0	77.9	70-120	0			

LCSD	Sample ID: **DLCSDS1-122029-122029**				Units: **mg/Kg**			Analysis Date: **07/30/18 06:31 PM**			
Client ID:	Run ID: **GC8_180730A**				SeqNo: **5177161**			Prep Date: **07/30/18**		DF: **1**	
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	8.586	0.5	5.0	10	0	85.9	70-120	7.786	9.78	20	

The following samples were analyzed in this batch:

18071769-01C	18071769-02C

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

Client: Barr Engineering Company
Work Order: 18071769
Project: Tank 10 Response (49161374.04)

QC BATCH REPORT

Batch ID: **121973** Instrument ID **VMS9** Method: **SW8260C**

MBLK Sample ID: **MBLK-121973-121973** Units: **µg/Kg-dry** Analysis Date: **07/27/18 01:07 PM**
Client ID: Run ID: **VMS9_180727A** SeqNo: **5172341** Prep Date: **07/27/18** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	U	5.1	30	0	0	0	0-0	0			
Ethylbenzene	U	6.3	30	0	0	0	0-0	0			
m,p-Xylene	U	14	60	0	0	0	0-0	0			
o-Xylene	U	12	30	0	0	0	0-0	0			
Toluene	U	8.2	30	0	0	0	0-0	0			
Xylenes, Total	U	26	90	0	0	0	0-0	0			
Surr: 1,2-Dichloroethane-d4	*1036*	0	0	*1000*	0	*104*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*974*	0	0	*1000*	0	*97.4*	*70-130*	0			
Surr: Dibromofluoromethane	*965*	0	0	*1000*	0	*96.5*	*70-130*	0			
Surr: Toluene-d8	*991*	0	0	*1000*	0	*99.1*	*70-130*	0			

LCS Sample ID: **LCS-121973-121973** Units: **µg/Kg-dry** Analysis Date: **07/27/18 12:22 PM**
Client ID: Run ID: **VMS9_180727A** SeqNo: **5172340** Prep Date: **07/27/18** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1110	5.1	30	1000	0	111	75-125	0			
Ethylbenzene	1165	6.3	30	1000	0	116	75-125	0			
m,p-Xylene	2298	14	60	2000	0	115	80-125	0			
o-Xylene	1146	12	30	1000	0	115	75-125	0			
Toluene	1050	8.2	30	1000	0	105	70-125	0			
Xylenes, Total	3444	26	90	3000	0	115	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1018*	0	0	*1000*	0	*102*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1026*	0	0	*1000*	0	*103*	*70-130*	0			
Surr: Dibromofluoromethane	*1016*	0	0	*1000*	0	*102*	*70-130*	0			
Surr: Toluene-d8	*1014*	0	0	*1000*	0	*101*	*70-130*	0			

MS Sample ID: **18071710-04A MS** Units: **µg/Kg-dry** Analysis Date: **07/27/18 06:36 PM**
Client ID: Run ID: **VMS9_180727A** SeqNo: **5174430** Prep Date: **07/27/18** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1345	6.5	38	1273	0	106	75-125	0			
Ethylbenzene	1375	8.1	38	1273	0	108	75-125	0			
m,p-Xylene	2690	18	76	2545	0	106	80-125	0			
o-Xylene	1333	15	38	1273	0	105	75-125	0			
Toluene	1228	10	38	1273	0	96.4	70-125	0			
Xylenes, Total	4023	33	110	3818	0	105	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1331*	0	0	*1273*	0	*105*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1331*	0	0	*1273*	0	*105*	*70-130*	0			
Surr: Dibromofluoromethane	*1348*	0	0	*1273*	0	*106*	*70-130*	0			
Surr: Toluene-d8	*1307*	0	0	*1273*	0	*103*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 18071769
Project: Tank 10 Response (49161374.04)

QC BATCH REPORT

Batch ID: **121973** Instrument ID **VMS9** Method: **SW8260C**

MSD Sample ID: **18071710-04A MSD** Units: **µg/Kg-dry** Analysis Date: **07/27/18 06:51 PM**
Client ID: Run ID: **VMS9_180727A** SeqNo: **5174431** Prep Date: **07/27/18** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1439	6.5	38	1273	0	113	75-125	1345	6.77	30	
Ethylbenzene	1439	8.1	38	1273	0	113	75-125	1375	4.52	30	
m,p-Xylene	2864	18	76	2545	0	113	80-125	2690	6.28	30	
o-Xylene	1397	15	38	1273	0	110	75-125	1333	4.71	30	
Toluene	1299	10	38	1273	0	102	70-125	1228	5.64	30	
Xylenes, Total	4262	33	110	3818	0	112	75-125	4023	5.76	30	
Surr: 1,2-Dichloroethane-d4	*1365*	0	0	*1273*	0	*107*	*70-130*	1331	*2.55*	*30*	
Surr: 4-Bromofluorobenzene	*1300*	0	0	*1273*	0	*102*	*70-130*	1331	*2.32*	*30*	
Surr: Dibromofluoromethane	*1366*	0	0	*1273*	0	*107*	*70-130*	1348	*1.31*	*30*	
Surr: Toluene-d8	*1259*	0	0	*1273*	0	*98.9*	*70-130*	1307	*3.77*	*30*	

The following samples were analyzed in this batch: 18071769-01A 18071769-02A

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

Client: Barr Engineering Company
Work Order: 18071769
Project: Tank 10 Response (49161374.04)

QC BATCH REPORT

Batch ID: **R241270** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R241270** Units: **% of sample** Analysis Date: **07/30/18 12:08 PM**
Client ID: Run ID: **MOIST_180730B** SeqNo: **5176348** 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-R241270** Units: **% of sample** Analysis Date: **07/30/18 12:08 PM**
Client ID: Run ID: **MOIST_180730B** SeqNo: **5176347** 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: **18071251-01A DUP** Units: **% of sample** Analysis Date: **07/30/18 12:08 PM**
Client ID: Run ID: **MOIST_180730B** SeqNo: **5176326** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	2.17	0.025	0.050	0	0	0	0-0	2.09	3.76	10	

DUP Sample ID: **18071255-07A DUP** Units: **% of sample** Analysis Date: **07/30/18 12:08 PM**
Client ID: Run ID: **MOIST_180730B** SeqNo: **5176336** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	4.61	0.025	0.050	0	0	0	0-0	4.66	1.08	10	

The following samples were analyzed in this batch:

18071769-01B	18071769-02B

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

18071169

Barr Engineering Co. Chain of Custody

BARR ☐ Ann Arbor ☑ Duluth ☐ Hibbing ☐ Minneapolis
☐ Bismarck ☐ Grand Rapids ☐ Jefferson City ☐ Salt Lake City

Sample Origination State: ☐ KS ☐ MO ☐ UT ☐ MI ☐ ND ☑ WI ☐ MN ☐ SD Other: ____

COC Number: 57881
COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company: same
Address: 325 S Lake Ave	Address:
Name: Ryan Erickson	Name:
email: ree@barr.com	email:
Copy to: datamgt@barr.com; jet@barr.com	P.O.
Project Name: Tank 10 Response	Barr Project No: 49161374040 09 100

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 analysis	Soil: % Solids	Preservative Code	Field Filtered Y/N
1. TK 10 Stockpile-1	-	-	-	7/26/2018	1315	S				2	1	2	1		
2. TK 10 Stockpile-2	-	-	-	7/26/2018	1320	S				2	1	2	1		
3.															
4.															
5.															
6.															
7.															
8.															
9.															
10.															

ASAP TAT

BARR USE ONLY		Relinquished by	On Ice?	Date	Time	Received by	Date	Time
Sampled by:	TTS		Y N					
Barr Proj. Manager:	REE LMG	Relinquished by:	On Ice? Y N	Date	Time	Received by:	Date 7/27/18	Time 0936
Barr DQ Manager:	JET							
Lab Name:	ALS	Samples Shipped VIA: ☐ Courier ☑ Federal Express ☐ Sampler ☐ Other:				Air Bill Number:		
Lab Location:	Holland	Lab WO:	Temperature on Receipt (°C):			Custody Seal Intact? ☐ Y ☐ N ☐ None		

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

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. 01/02/18

ALS Group, USA

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **27-Jul-18 09:30**

Work Order: **18071769** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 27-Jul-18 (Date) Reviewed by: Bill Carey (eSignature) 27-Jul-18 (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):	3.4/3.4 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	7/27/2018 10:49:00 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 V Waste Management Campus
1100 West Gary Street
Duluth, MN 55808
Permit: SW 536

18-075-I Superior Terminal Tank 10 Line					
Date	**Ticket**	**Customer**	**Truck**	**Material**	**Tons**
08/02/2018	301649	001342 - Enbridge Pipelines LLC	T53691W	Contaminated Soil Tons	16.05
08/01/2018	301650	001342 - Enbridge Pipelines LLC	S98692W	Contaminated Soil Tons	13.08
08/01/2018	301651	001342 - Enbridge Pipelines LLC	S98692W	Contaminated Soil Tons	13.38
08/01/2018	301652	001342 - Enbridge Pipelines LLC	S98692W	Contaminated Soil Tons	10.82
08/01/2018	301653	001342 - Enbridge Pipelines LLC	S98692W	Contaminated Soil Tons	13.70
08/01/2018	301654	001342 - Enbridge Pipelines LLC	S98692W	Contaminated Soil Tons	12.02
08/02/2018	301664	001342 - Enbridge Pipelines LLC	T53691W	Contaminated Soil Tons	19.43
08/02/2018	301675	001342 - Enbridge Pipelines LLC	T53691W	Contaminated Soil Tons	23.45
08/02/2018	301683	001342 - Enbridge Pipelines LLC	T53691W	Contaminated Soil Tons	20.66
08/02/2018	301696	001342 - Enbridge Pipelines LLC	T53691W	Contaminated Soil Tons	13.04
				Total Tons	**155.63**
				Total Loads	**10**