resourceful. naturally.
engineering and environmental consultants

BARR

Technical Memorandum

To: Alex Smith, Enbridge Energy
From: Ryan Erickson and Kaitlin Johnson
Subject: 2019 Tank 22 Mixer Release Response
WI Spill #: 13715 ID 20190318NO16-1
Date: July 9, 2019
Project: 49161374.07 005

This memorandum summarizes the environmental response and assessment activities performed by Enbridge Energy (Enbridge) and Barr Engineering (Barr) following a crude oil release from a mixer on the south side of Tank 22 at Enbridge Superior Terminal (Terminal) in Superior, Wisconsin (Figure 1).

Background

On February 28, 2019, Enbridge discovered that a tank mixer on the south side of Tank 22 had released crude oil onto the ground surface (Figure 2). Enbridge Pipe Line Maintenance (PLM) personnel responded to the site to assess and repair the release source and conduct initial environmental remediation activities. The PLM reported that approximately 6.5 gallons had been released from the mixer. The PLM excavated the hydrocarbon-impacted material beneath the mixer to a depth of approximately 1 foot below ground surface (bgs). However, after the initial remedial actions, free-product was still present in the gravel fill material on the north sidewall of the excavation (Photo 1). Additional excavation of the impacted soil was not completed at the time due to frozen ground conditions and the excavation's proximity to Tank 22 and associated tank infrastructure. Excavated impacted soil was stockpiled in the Terminal Soil Management Area (SMA) until offsite management was approved.

On March 7, 2019 Enbridge PLM installed four product-recovery sumps. The PLM periodically returned to the site to monitor environmental conditions and to recover free-product in the excavation and sumps with a vacuum truck, as needed. Enbridge returned to the site on May 2, 2019 to complete final remedial excavation activities of accessible impacted soil.

Enbridge Environment requested that Barr complete the following activities:

- document PLM remediation activities and environmental conditions at the time of those activities,
- assist with the characterization and offsite management coordination of hydrocarbon-impacted soil,
- field screen and sample soil from the final excavation extents to document residual impacts, and
- prepare a memorandum summarizing the environmental actions taken and the environmental conditions encountered.

The Wisconsin Department of Natural Resources (WDNR) was notified about the mixer release on March 18, 2019 and spill number #13715 ID 20190318NO16-1 was assigned to the site. The associated WDNR communication is provided in Attachment A. Two historical Bureau for Remediation and Redevelopment Tracking System (BRRTS) sites were identified within the Tank 22 containment basin (Figure 2). BRRTS site #0216220009 is associated with an August 28, 1998, 40-barrel (bbl) release. This site has been closed by the WDNR. BRRTS site #0216556786 was established in 2011 to address historical impacts that were

encountered during a 2010 Tank 22 tank floor replacement project. The WDNR has closed this site with continuing obligations.

Field Activities

Barr was on site March 7, 11, and 14, and May 2, 2019 to complete the field activities described below.

On March 7, Barr documented environmental site conditions, PLM remediation activities, and the installation of four PVC product-recovery sumps (*Sump-1* through *Sump-4*; Figure 2; Attachment B). Barr collected free-product sample *TK22-Product-1* from the remedial excavation for a Total and Saturated Hydrocarbon Analysis by modified US EPA method 8015 (8015-MOD) at the ATS Laboratory (ATS) in Ann Arbor, Michigan. The fingerprint sample laboratory report and an initial results-interpretation communication (email dated March 14, 2019) are provided in Attachment C. Barr collected waste characterization sample *TK22-Stockpile-1* from the contaminated soil stockpile for analysis of Benzene, Toluene, Ethyl Benzene, and Xylene (BTEX) and Diesel Range Organics (DRO) at the ALS Laboratory (ALS) in Holland, Michigan. Additional information about soil disposal is included in the *Material Management* section of this memorandum and the waste characterization laboratory report is provided in Attachment D.

On March 11, and 14, Barr documented environmental site conditions and the PLM product recovery activities. The field observations are documented on the Site Investigation Field sampling and Screening Logs in Attachment B.

On May 2, Barr observed PLM remedial excavation activities and documented the environmental conditions in the final excavation extents through field screening and analytical sample collection, as required per the 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 the field log in 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 confirmation sample *TK22-S-1* was collected from the excavation's southern sidewall to delineate residual impacts in the final excavation. The sample was submitted to ALS for analysis of petroleum volatile organic compounds (PVOC) and naphthalene. The laboratory results are summarized in Table 1 and the laboratory report is provided in Attachment C.

Results

On March 7, soil was excavated with a hydrovacuum truck (hydrovac) to install product recovery sumps *Sump-1, Sump-2,* and *Sump-3* around the mixer release location within approximately 3 feet of Tank 22. *Sump-4* was installed approximately 10 feet south of the mixer (Photos 2 and 3; Figure 2; Attachment B). The sump excavations were approximately one foot in diameter. The sumps near the tank were advanced to approximately 4 feet in depth and *Sump-4* was advanced to 5 feet in depth. Gravel roadbed material was observed from approximately 0 to 3 feet in depth in the sumps near the tank. Clay soil was identified below the roadbed material up to 5 feet bgs. A one-inch thick layer of ice was observed in the road fill material at approximately 1.7 feet bgs. The ice layer was the inferred depth of a perched water table in the tank area fill material. Free-product was observed in the roadbed fill material above the inferred perched

water table (1.7 feet bgs) in the *Sump 1*, *2*, and *3* excavations and trace amounts of product were observed from 1.7 to 3 feet bgs. Hydrocarbon impacts were not identified in the clay soil below 3 feet bgs or in *Sump 4*.

On the same day, Barr collected free-product sample *TK22-Product-1* for fingerprint analysis by ATS. The ATS laboratory analysts review indicated that the crude oil sample had little to no weathering (Attachment C- email dated March 14, 2019).

On March 11 and 14, 2019, free-product was observed on the surface of water within the remedial excavations. On March 11, product in the *Sump 1*, *2*, and *3* excavations was approximately 0.25 inches thick, and no impacts were observed in the *Sump 4* excavation. On March 14, product was present in the *Sump 1*, *2*, and *3* excavations but it did not cover the entire water surface (Photos 4 and 5). No impacts were observed in the *Sump 4* excavation.

On May 2, 2019, PLM personnel excavated accessible impacted soil beneath the mixer and excavated a small test pit approximately 20 feet east of the mixer (Figure 2; Attachment B-Sheet 4). Excavation activities were limited by the presence of above and below ground tank infrastructure. *Sump-1*, *Sump-2,* and *Sump-3* were permanently removed during excavation activities. The final excavation was approximately 17 feet long (west to east) by 4 feet wide (south to north) and up to 3 feet in depth (Photos 6, 7; Figure 2; Attachment B-Sheet 4). The test pit excavation east of the mixer was approximately 4 feet in diameter and up to 3 feet deep (Photos 8, 9). Soil/fill in the excavations consisted of approximately 3 feet of road bed fill material overlying red clay soil. The perched water table was approximately 1.5 feet below the gravel road surface prior to excavation activities. Hydrocarbon-impacted water containing free-product and a rainbow sheen was recovered with a vacuum truck and oil absorbent pads.

Barr observed soil conditions and collected field screening soil samples from the final remedial excavations to identify residual impacts. Fill material with a hydrocarbon odor, dark staining, and trace amounts of product were identified along the north sidewall of the excavation near the tank. Additional excavation of the identified residual impacts along the north side of the excavation, along the tank wall, was not feasible based on the presence of tank infrastructure. In the southern excavation sidewall, headspace readings were between 0.0 and 1.6 ppm, and no evidence of residual hydrocarbon impacts was observed. Soil in the eastern test pit excavation had headspace readings of 0.1 and 0.2 and no evidence of impacts were observed in the soil.

Analytical soil confirmation sample *TK22-S-1* was collected from the southern sidewall near the *S3* field screening point at 2 feet bgs to document conditions away from the observed residual impacts beneath the tank (Figure 2; Attachment B-Sheet 4). Analyte concentrations were below WDNR Industrial Direct Contact Residual Contaminant Levels (RCLs) and WDNR Groundwater RCLs. The laboratory results are summarized in Table 1 and the ALS laboratory report is provided in Attachment C.

Based on the field observations and not being able to safely excavate additional impacted soil closer to the tank, remedial excavation activities were concluded and the excavation and test pit were backfilled with clean road fill. A slotted PVC pipe (*Sump-5*) was installed in the excavation 10 feet east of the mixer before backfill material was placed to facilitate future product monitoring and recovery, as needed, with a vacuum truck (Photo 6; Figure 2; Attachment B).

Material Management

On March 7, Barr collected analytical waste characterization soil sample *Tank22-Stockpile-1* from the contaminated soil stockpile at the SMA for laboratory analysis at ALS Laboratory. The sample was analyzed for DRO and BTEX. The laboratory report and a waste profile application were submitted to the VONCO V landfill in Duluth, Minnesota and the soil was accepted and assigned waste profile #19-017-I. A total of 46.48 tons of contaminated soil were hauled to the landfill on March 29, 2019. The waste profile documents, the waste characterization laboratory report, and the landfill summary report are included in Attachment D.

Receptor Survey

Fill material containing limited residual free-product is likely still present beneath the tank. However, the excavations were backfilled with clean fill material, the site will be monitored by facility personnel, and personnel working at the Terminal are aware of the possibility of encountering impacted soil. Therefore, the direct contact risk is deemed to be low. No impacts to surface water were identified and there is little risk of future surface water impacts based on the remedial actions completed and the site's location within the tank containment basin. Residual free-product was identified in shallow fill material beneath Tank 22; however, soil approximately 10 feet to the south of the mixer had residual analyte concentrations below the WDNR Groundwater RCLs. The groundwater pathway at the Superior Terminal is also addressed on a facility-wide basis through the established hydrogeologic performance standard approved by the WDNR. Enbridge samples its monitoring well network on a biannual basis. The nearest downgradient monitoring well is MW-6 located 400 feet to the south of the release site (Figure 3). The nearest structures are slab-on-grade terminal buildings approximately 300 feet southeast of the site. The risk of hazardous vapor accumulation in those structures is low because the buildings are above ground buildings with minimal human occupancy. Terminal employees are also required to wear four-gas detectors that would alert them to a potentially hazardous atmosphere.

Discussion and Conclusions

The 2019 Tank 22 Mixer release (WI SPILL #13715 ID 20190318NO16-1) was reported to the WDNR based on the estimated release volume (6.5 gallons). Enbridge recovered accessible free-product and impacted soil; however, tank infrastructure limited those actions. Based on field observations, fill material containing residual hydrocarbon impacts remain near the tank within the direct contact zone. Soil and water approximately 10 feet to the south of the mixer and tank did not show signs of residual hydrocarbon impacts and no receptors were identified as being at risk. Enbridge intends to continue to monitor environmental conditions below the mixer and in the sump and recover free-product if observed.

Based on the field observations and the existing facility-wide groundwater monitoring program, Barr believes that the WDNR will transfer the spill site to the Superior Terminal Facility-wide BRRTS # 02-16-560657.

If this memorandum and site classification/closure pathway are approved by the WDNR, an *Enbridge Superior Terminal Facility-Wide Continuing Obligations GIS Registry Update* will be prepared and submitted to the WDNR.

Attachments:

Site Photos	1 through 9
Table 1	Soil Analytical Data Summary
Figure 1	Site Location
Figure 2	Site Layout
Figure 3	Receptor Survey
Attachment A	WDNR Release Reporting Communication
Attachment B	Site Investigation Field Sampling and Screening Logs
Attachment C	ATS and ALS Laboratory Reports
Attachment D	Waste Management Documentation

Site Photos

2/28/2019 release response surface soil scrape

Photo 1

Photo 1: The initial Tank 22 mixer release remedial surficial soil scrape excavation. The bottom of the mixer is shown in the top left corner of photo. Free-product is present on water within the excavation. Plastic sheeting (top right corner) was placed over the excavation after the initial remedial response. Photo taken by the PLM facing northeast on February 28, 2019.

3/7/2019 remedial excavation, product recovery and sump installation

Photo 2

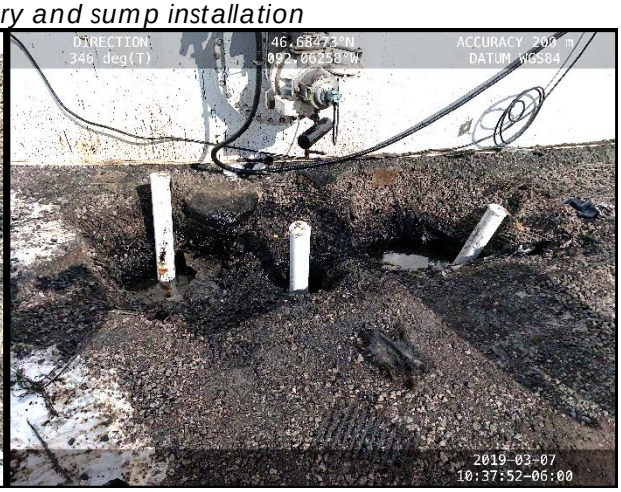


Photo 3

Photo 2: The mixer release area on the south side of Tank 22. The bottom of the mixer is shown in the top of photo. Shallow remedial excavations containing free-product are below the mixer. Photo taken facing north on March 7, 2019.
Photo 3: Tank 22 remedial excavations beneath the mixer (top of photo). Shallow remedial excavation containing free-product and three PVC product-recovery sump pipes are below the mixer. Photo taken facing north on March 7, 2019.

3/14/2019 product recovery

Photo 4

Photo 5

Photo 4: The Tank 22 remediation area. The current excavation and sumps are covered in plastic. The orange cone (right side of the photo) is over the southern product recovery sump. Oil absorbent boom is around the excavations. Photo taken facing east on March 14, 2019.
Photo 5: Free-product on water within the excavation. Photo taken on March 14, 2019.

5/2/2019 remedial excavation, environmental analysis and backfilling

Photo 6

Photo 7

Photo 6: Tank 22 remedial excavation. The *Sump-5* white PVC free-product recovery sump is shown to the right of the tank mixer. Photo taken facing east on May 2, 2019.
Photo 7: Tank 22 remedial excavation. Photo taken facing west on May 2, 2019.

Photo 8

Photo 9

Photo 8: Eastern test pit located approximately 20 feet east of the mixer. The test pit was excavated to better define contaminant extents. Photo taken facing west on May 2, 2019.
Photo 9: Eastern test pit with a water pump hose and oil absorbent pads. Photo taken on May 2, 2019.

Table 1
Soil Analytical Data Summary
2019 Tank 22 Mixer Release Response
Enbridge Energy Superior Terminal
Superior, WI

		Location	**TK22-S-1**
		Date	**5/02/2019**
		Depth	**2 ft**
Parameter	Wisconsin Groundwater RCLs, DF=1	Wisconsin Not to Exceed Direct Contact Industrial RCLs	
Effective Date	06/01/2018	06/01/2018	
Exceedance Key	No Exceed	No Exceed	
General Parameters			
% Moisture			25
Volatile Organic Compounds			
1,2,4-Trimethylbenzene	0.6894 (1)	219	0.0031 j
1,3,5-Trimethylbenzene	0.6894 (1)	182	< 0.0040
Benzene	0.0026	7.07	< 0.0022
Ethyl benzene	0.785	35.4	< 0.0028
Naphthalene	0.3291	24.1	< 0.0036
Toluene	0.5536	818	< 0.0036
Xylene, m & p	1.98 XYL	260 XYL	< 0.0062
Xylene, o	1.98 XYL	434	< 0.0051
Xylene, total	1.98	260	< 0.011

Note:
All units in mg/kg unless otherwise noted
RCL - Residual Contaminant Level
DF - Dilution Factor

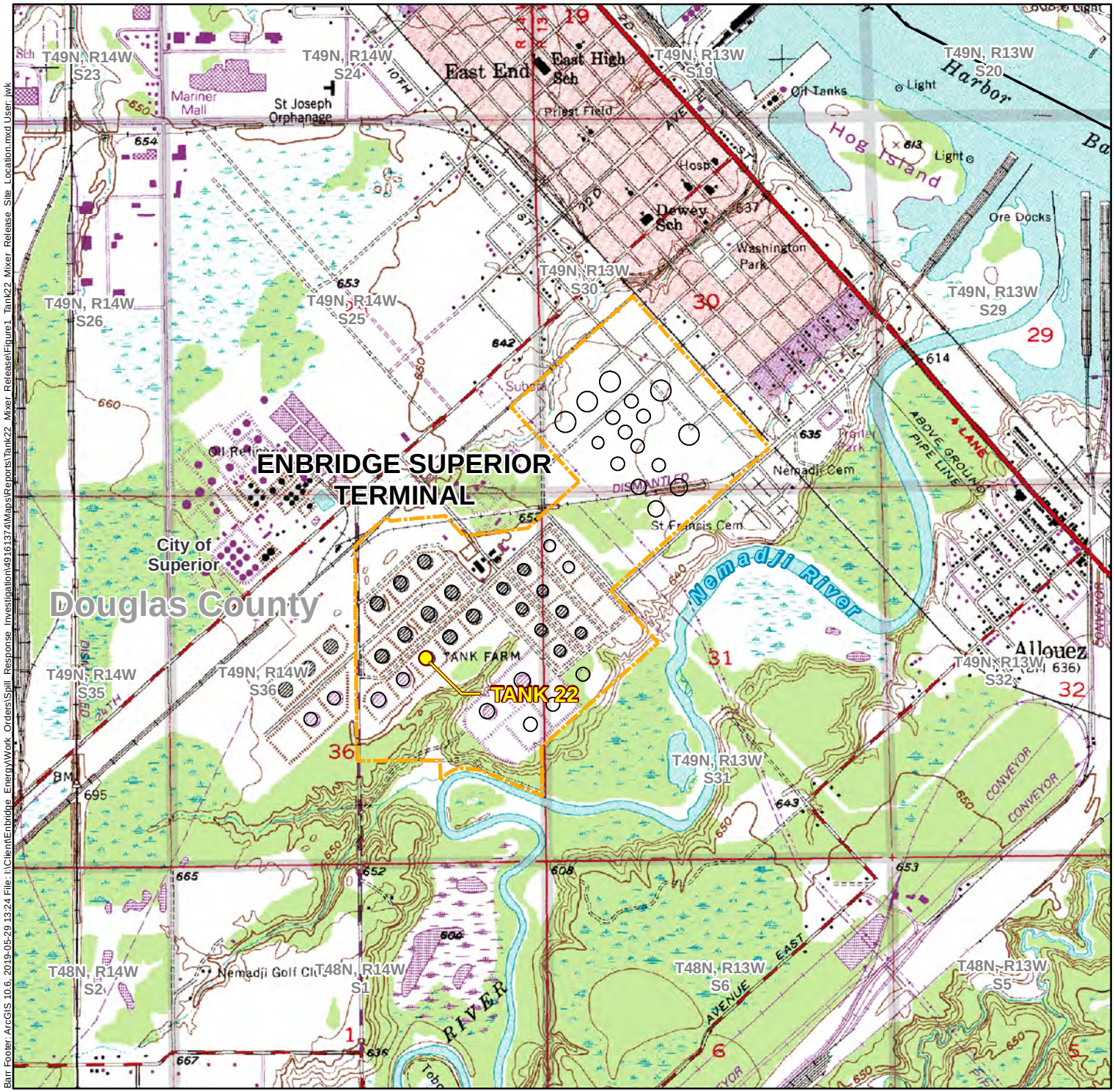
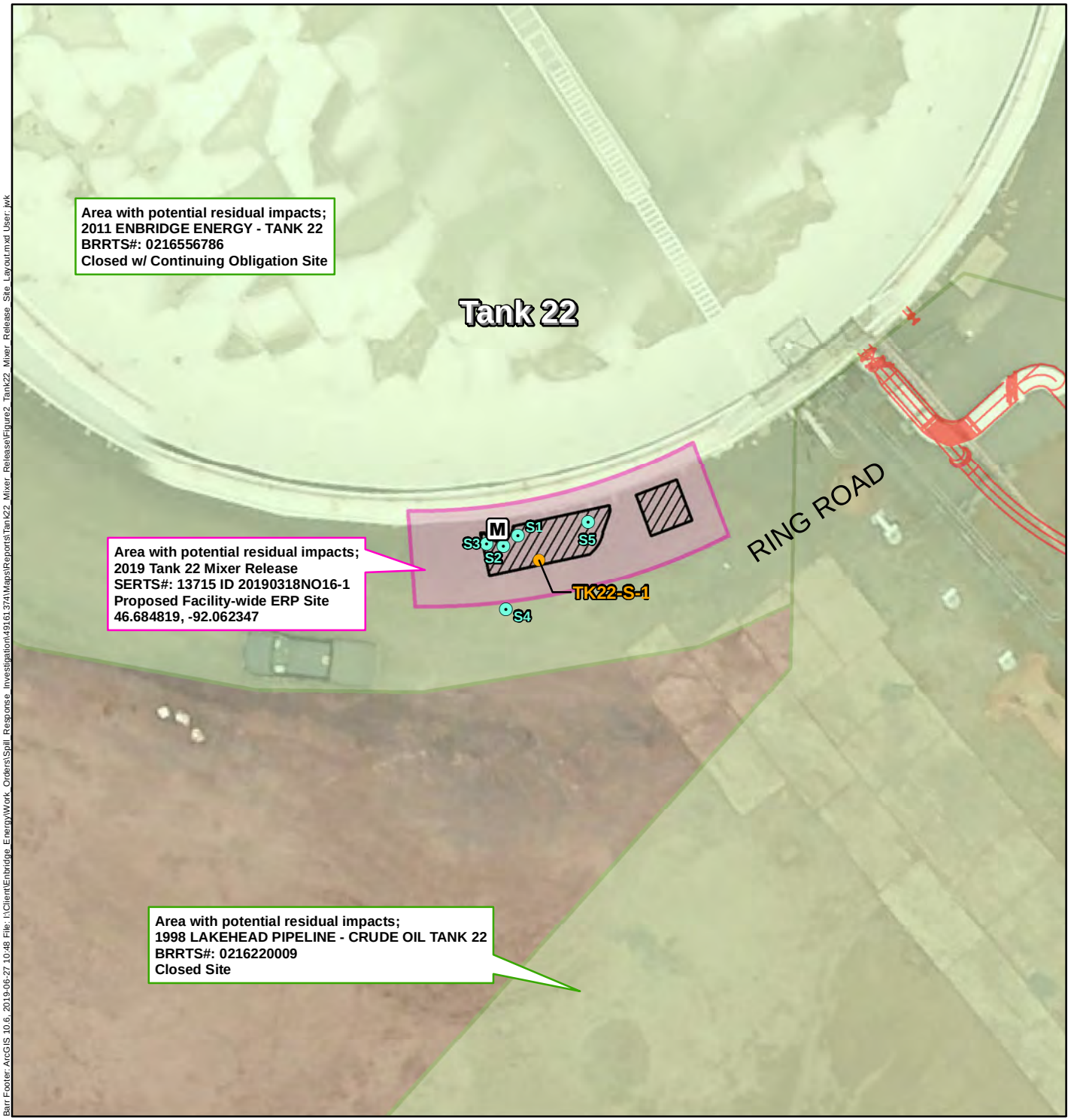


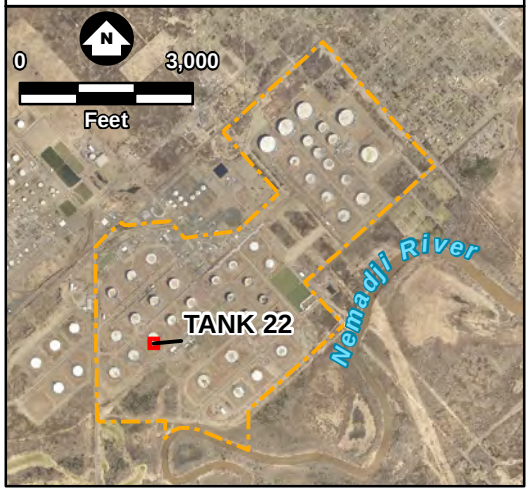
Figure 1

SITE LOCATION
TANK 22 MIXER RELEASE
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE



ENBRIDGE SUPERIOR TERMINAL



- Analytical Sample Locations
- Mixer Door
- Sump Locations
- Excavation Extents
- Area with Potential Residual Impacts
- Estimated extent of residual impacts - Closed BRRTS sites
- Pipeline Infrastructure
- Terminal Property Boundary

N

0 20 40

Feet

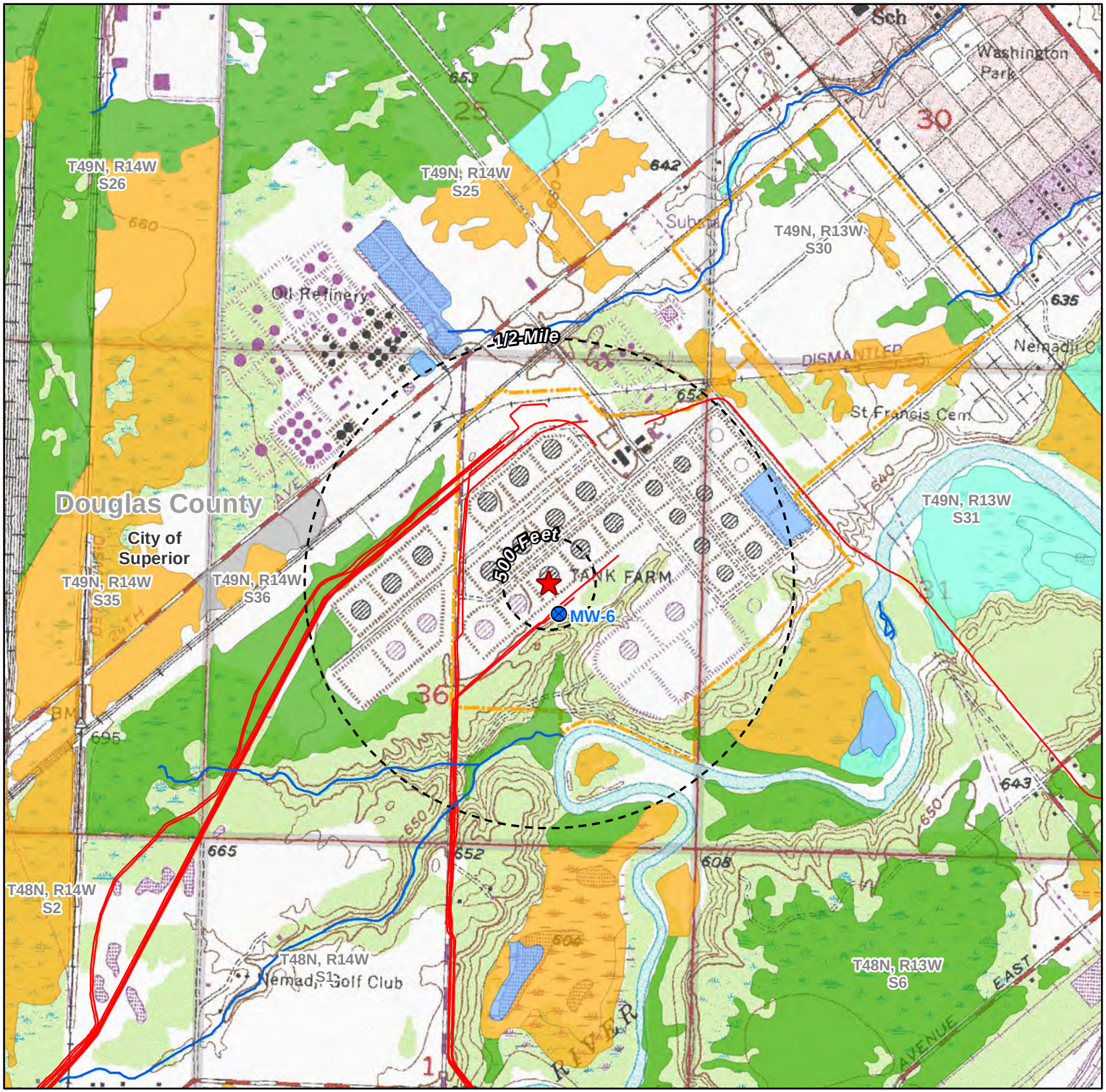
1 Inch = 20 Feet

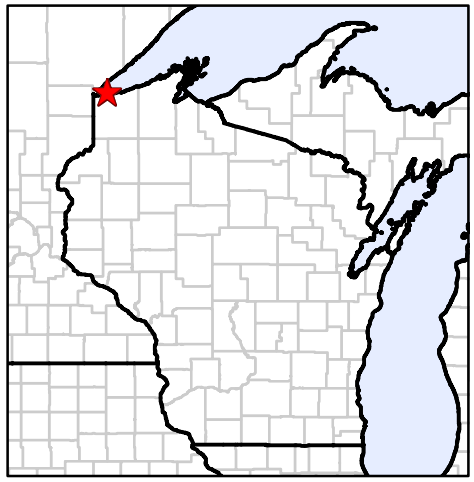
Douglas County Imagery Circa May, 2016

Figure 2

SITE LAYOUT
TANK 22 MIXER RELEASE
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 22 MIXER RELEASE
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE

Barr Footer: ArcGIS 10.6, 2019-05-29 13:36 File: I:\Client\Enbridge_Energy\Work_Orders\Spill_Response_Investigation\49161374\Maps\Reports\Tank22_Mixer_Release\Figure3_Tank22_Mixer_Release_Receptor.mxd User: jwk

Attachment A

WDNR Release Reporting Communication

From: Rahn, Matthew W - DNR
To: Alex Smith
Subject: [External] FW: WI SPILL #13715 ID 20190318NO16-1 - CRUDE OIL [CRUDE OIL]
Date: Tuesday, March 19, 2019 11:06:10 AM

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

We are committed to service excellence.
Visit our survey at http://dnr.wi.gov/customersurvey to evaluate how I did.

-----Original Message-----
From: meagan.welke@wisconsin.gov <meagan.welke@wisconsin.gov>
Sent: Monday, March 18, 2019 5:34 PM
To: Rahn, Matthew W - DNR <Matthew.Rahn@wisconsin.gov>
Subject: WI SPILL #13715 ID 20190318NO16-1 - CRUDE OIL [CRUDE OIL]

SERTS ID:
20190318NO16-1

Reported:
03/18/2019 17:21

Occurred:
02/27/2019 15:30

Reported by:
TERRI PICKTON
COMPLIANCE ADVISOR
ENBRIDGE PIPELINES
theresa.pickton@enbridge.com
(715) 718-1208

Location:
NO REGION
DOUGLAS COUNTY
SUPERIOR, CITY OF
ENBRIDGE TERMINAL
20000 E 21ST ST

Responsible Party:
ENBRIDGE PIPELINES
(715) 817-8322

RP Contact:
ALEX SMITH
ENBRIDGE PIPELINES
ENVIRONMENTAL ADVISER
(715) 817-8322

Substance:

CRUDE OIL [CRUDE OIL]
Released Amt: 6.5 Gal
Recovered Amt: 6.5 Gal
(Amounts are often estimated)

Cause:
EQUIPMENT FAILURE

Cause Description:
MECHANICAL SEAL ON TANK MIXER LEAK. ON 2/27 3.9 GALLONS WERE RELEASED. WHEN EXCAVATION WAS OCCURRING ON 3/18 ADDITIONAL PRODUCT WAS FOUND. A TOTAL OF 6.5 GALLONS WERE RELEASED.

Environmental Impact:
SPILLED ONTO SOIL. NO WATERWAYS AFFECTED.

Cleanup:
SOIL REMOVED. EXCAVATION AROUND MIXER IS BEING COMPLETED. RP CONTACT WILL KNOW MORE.

Notified TREVOR BANNISTER DNR OCSC at 17:31 by Voicemail

Submitted by:
MEAGAN WELKE
(800) 943-0003
meagan.welke@wisconsin.gov

Sent to:
andrew.savagian@wisconsin.gov
anita.smith@wisconsin.gov
brian.satula@wisconsin.gov
christopher.saari@wisconsin.gov
curtis.hedman@dhs.wisconsin.gov
danielle.wincentsen@wisconsin.gov
david.neste@wisconsin.gov
dmawemdutyofficer@wisconsin.gov
dnrledo@wisconsin.gov
dnrlehotline@wisconsin.gov
jason.lowery@wisconsin.gov
jessica.maloney@dhs.wisconsin.gov
john.sager@wisconsin.gov
josie.schultz@wisconsin.gov
kkesler@douglascountywi.org
kleist.andrew@epa.gov
kondreck.robert@epa.gov
matthew.rahn@wisconsin.gov
matthewa.thompson@wisconsin.gov
michael.schmoller@wisconsin.gov
patrick.collins@wisconsin.gov
philip.richard@wisconsin.gov
randy.books@wisconsin.gov
richard.joslin@wisconsin.gov
riley.neumann@wisconsin.gov
robert.thiboldeaux@dhs.wisconsin.gov
roxanne.chronert@wisconsin.gov
roy.irving@dhs.wisconsin.gov
ryan.wozniak@dhs.wisconsin.gov

stephen.ales@wisconsin.gov
stephend.mueller@wisconsin.gov
theresa.pickton@enbridge.com
trevora.bannister@wisconsin.gov

Attachment B

Site Investigation Field Sampling and Screening Logs

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Date: 3/7/2018

Location: *Milepost or Facility* Superior Terminal Tank 22 Response (49161092.07 300 006) Sampler: MDH2

Equipment used: N/A *-ionization detector with* N/A *eV lamp* Background Headspace: N/A ppm Calibration Time: _____

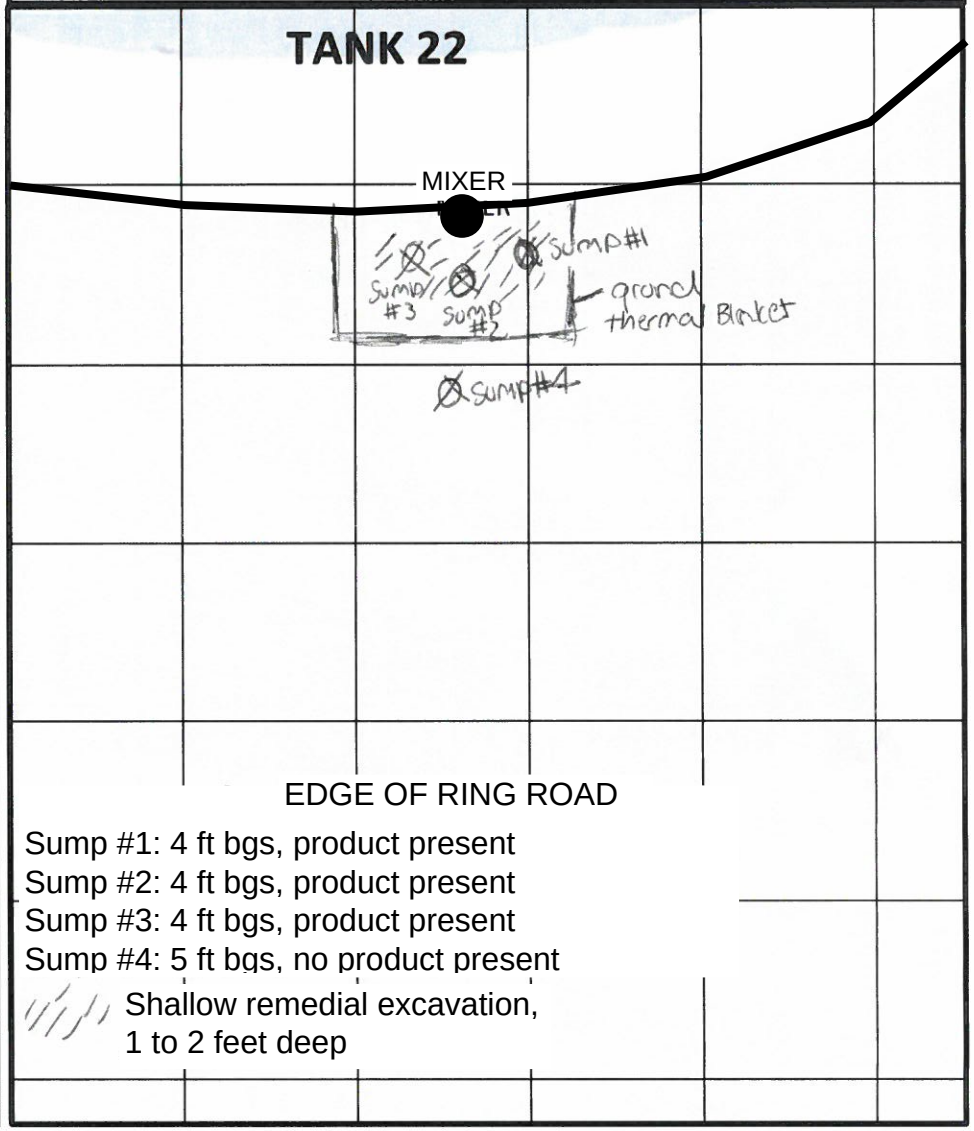
Sample Nomenclature *(Location - sample type - #):* TK22-

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: R-1	4	16:30	CL	Reddish brown	Petroleum/ Rainbow	275
	0-1.5		gravel fill		Product	–
	1.5-1.7		gravel fill / ICE		Product	–
	1.7-3		gravel		spotty product	–
	3-5		clay		N/N	–

GENERAL RING ROAD STRATIGRAPHY IN SUMPS

SITE SKETCH: *north is up; excavation extents & depths, impacted areas, sample locations, borings, wells, structures, utilities, natural features…* ***1 inch/grid = 10 FEET***



Sump #1: 4 ft bgs, product present
Sump #2: 4 ft bgs, product present
Sump #3: 4 ft bgs, product present
Sump #4: 5 ft bgs, no product present

Shallow remedial excavation, 1 to 2 feet deep

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Date: 5/11/19

Location: *Milepost or Facility* Superior Terminal Tank 22 Response (49161092.07 300 006)

Sampler: MAG

Equipment used:_____ *-ionization detector with* _____ *eV lamp*

Background Headspace:_____ppm

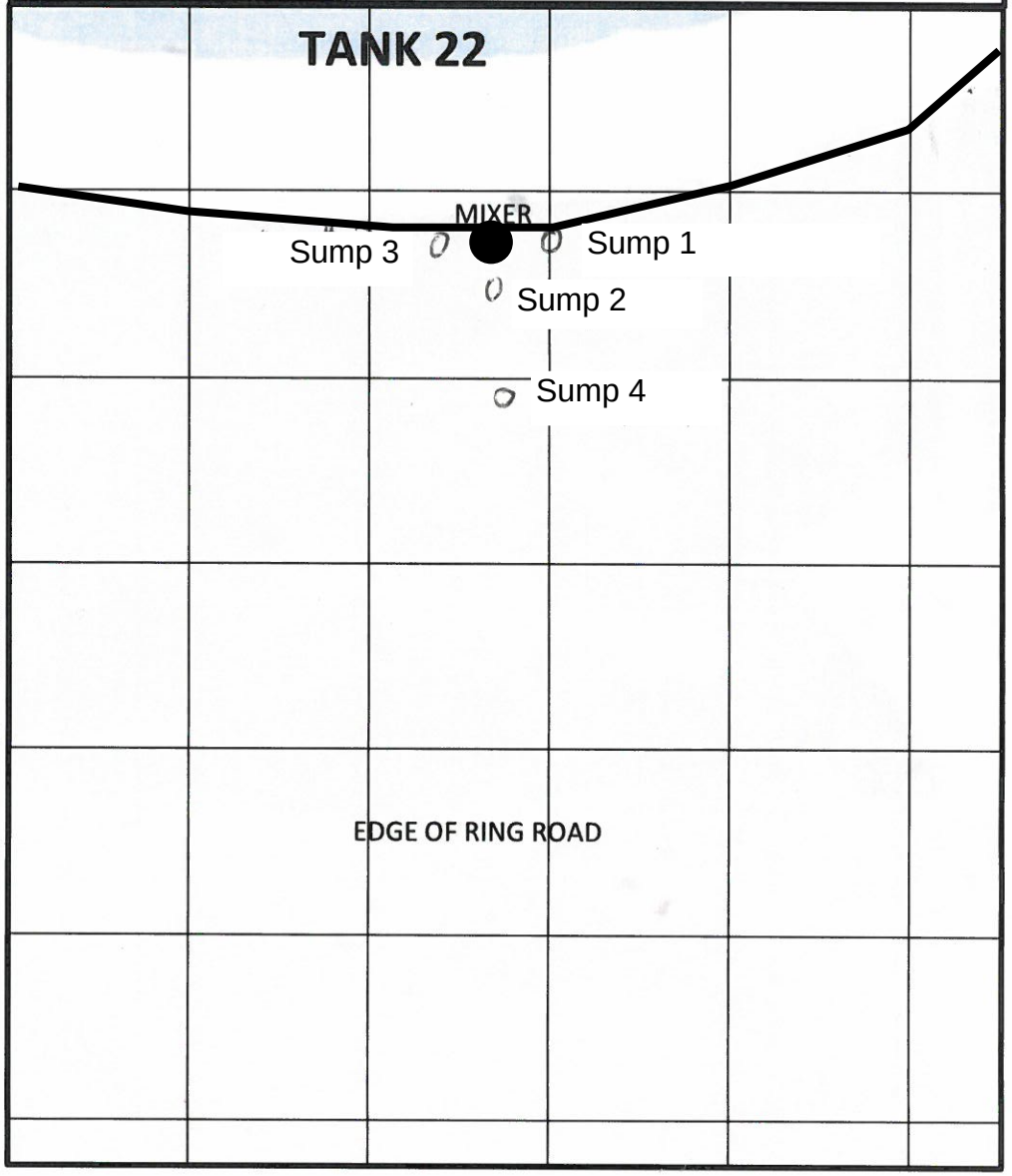
Calibration Time: _____

Sample Nomenclature *(Location - sample type - #):* TK22-

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: R-1	4	16:30	CL	Reddish brown	Petroleum/ Rainbow	275
Sump	DTW	DTP	Time			
1	1.68	—	1435			
2	1.96	—	↓			
3	1.86	—	↓			
4	4.62'	~~1435~~	1435			
	DTW	Depth	to water			
	DTP	Depth	to product			

SITE SKETCH: *north is up; excavation extents & depths, impacted areas, sample locations, borings, wells, structures, utilities, natural features...* ***1 inch/grid = 10 FEET***



SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Date: 3/14/19

Location: *Milepost or Facility* Superior Terminal Tank 22 Response (49161092.07 300 006)

Sampler: MAJ

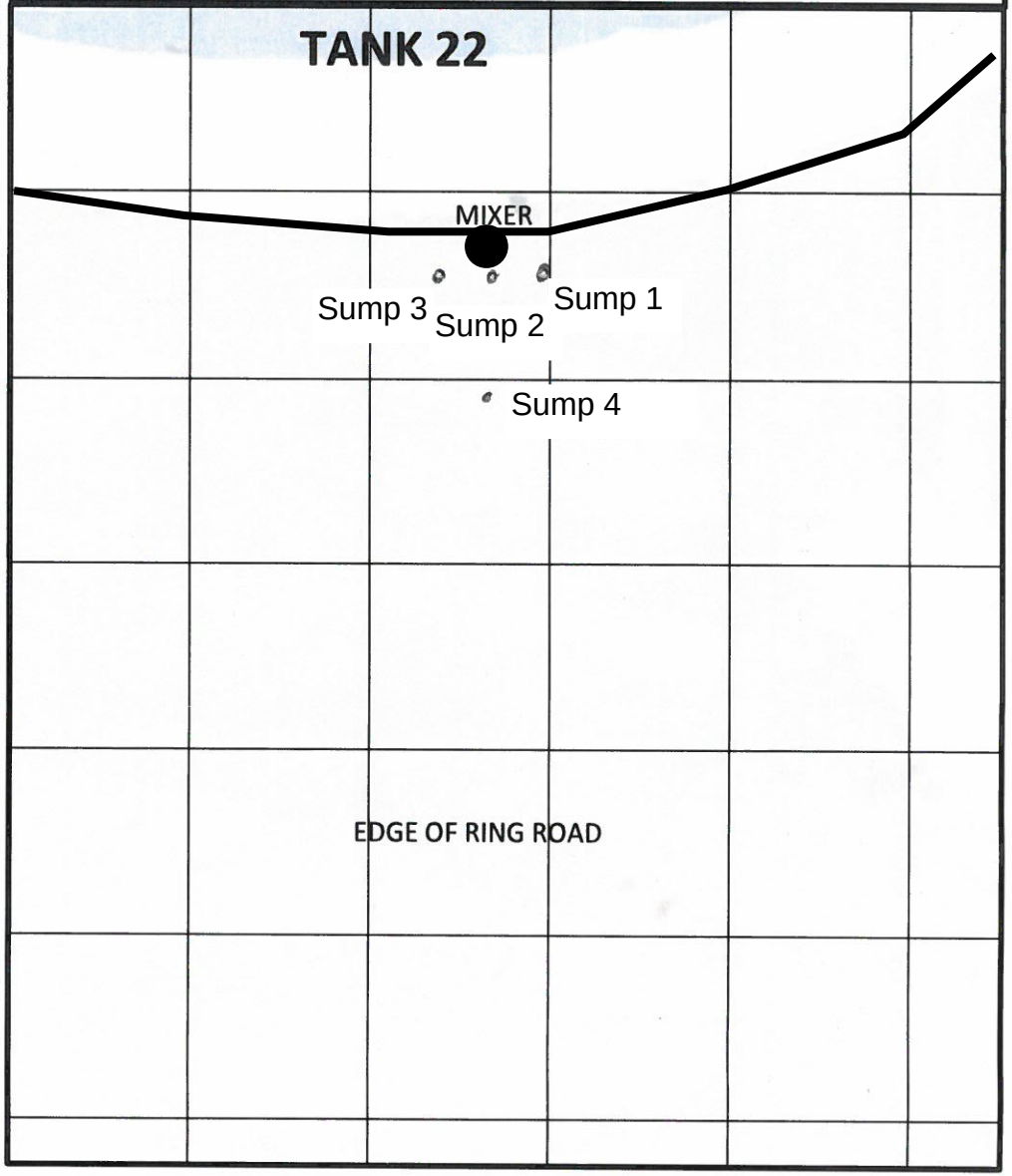
Equipment used: — *-ionization detector with* — *eV lamp* Background Headspace: — ppm Calibration Time: —

Sample Nomenclature *(Location - sample type - #):* TK22-

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: R-1	*4*	*16:30*	*CL*	*Reddish brown*	*Petroleum/ Rainbow*	*275*
	DTW	DTP	PT	Time		
Sump 1	1.51	—	—	1520		
Sump 2	1.58	—	—			
Sump 3	1.45	—	—			
Sump 4	2.75'	—	—	1515		
	DTW	Depth	to water			
	DTP	Depth	to product			
	PT	Product	thickness			

SITE SKETCH: *north is up; excavation extents & depths, impacted areas, sample locations, borings, wells, structures, utilities, natural features…* ***1 inch/grid = 10 FEET***



100ppm = 101.3ppm

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Location: *Milepost or Facility* Superior Terminal Tank 22 Response (4916/092.07300006)

Equipment used: photo *-ionization detector with* 10.8 *eV lamp* Background Headspace: 0.0 ppm

Sample Nomenclature *(Location - sample type - #):* TK22-

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

Date: 5/2/19

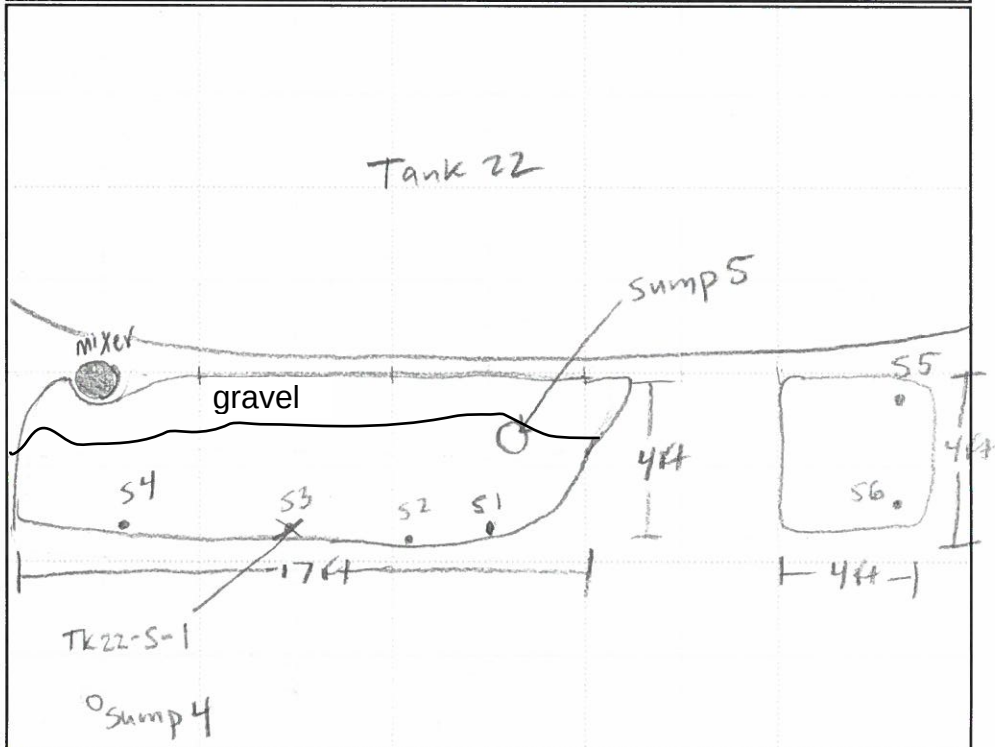
Sampler: KMJS

Calibration Time: 0740

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
S1	2	1108	CL	reddish brown	N/N	0.0
S2	2	1109				0.3
S3	2	1110				1.6
S4	2	1112				1.1
S5	2	1115				0.2
S6	3	1116				0.1
TK-22-S-1	2	1210				–

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



Residual contamination found on north side along Tank 22 in gravel area
Road bed material found on North site to 3 ft
Road bed material found on South side to 1 ft with clay beneath
Eastern test pit was excavated to delineate impacts, no impacts observed

Attachment C

ATS and ALS Laboratory Reports

ATS Laboratory Report for Product Fingerprint Analysis

DATA PACKAGE - LEVEL II

ATS Project
BENB.T01

Prepared for:
Mr. Ryan Erickson
Barr Engineering Company
325 South Lake Avenue, Suite 700
Duluth, MN 55802

Sample Delivery Groups (SDGs):
0308191

Prepared By:
Ann Arbor Technical Services, Inc.
290 South Wagner Road
Ann Arbor, MI 48103

Issued: March 14, 2019

LABORATORY OPERATIONS
SAMPLE DELIVERY GROUP (SDG) CASE NARRATIVE

ATS Project Number: BENB-T01
Report Date: 3/14/19
SDG / SRF Number: 0308191-B

Case Narrative Summary

This case narrative applies to the following one sample that was received by Ann Arbor Technical Services, Inc. (ATS) on 3/8/19, and associated matrix-specific QA/QC:

Samples

Client Sample Identification	Laboratory Sample ID	Requested Turn Around Time	Matrix
Tank-22-Product-1 3/7/19	0308191-1	Rush	Oily Liquid

Matrix Specific QC

Client Sample Identification	Laboratory Sample ID	Matrix
Tank-22-Product-1 3/7/19 Matrix Spike	0308191-1 MS	Oily Liquid
Tank-22-Product-1 3/7/19 Matrix Spike Duplicate	0308191-1 MSD	Oily Liquid

Upon receipt, samples were scheduled for the following analyses:

- Total and Saturated Hydrocarbon Analysis by modified US EPA method 8015 (8015-MOD)

Sample Receipt and Chain of Custody Records

Samples were delivered directly to ATS by commercial carrier. Samples were received in coolers, on ice, with proper chain of custody records included. Sample condition and anomalies, if any, are presented in the "Chain of Custody and Sample Receipt Documentation" section of this DVP.

Data Review and Approval

All data contained in this report have been generated in accordance with guidelines provided in the referenced standard test method, and are consistent with detailed procedures described in a written standard operating procedure (SOP) specific to the ATS Laboratory, as required by USEPA. All data are peer and management reviewed to ensure compliance with the above referenced SOP's and project specifications. In addition all data conform to the laboratory's Quality Assurance / Quality Control Manuals.

Consultants in Chemistry & Environmental Science
290 South Wagner Road, Ann Arbor, Michigan 48103 Tel 734/995-0995 Fax 734/995-3731

Data Qualifications, Specifications, and Technical Narration

The following are qualifier descriptions that may be used throughout this SDG and are presented with their associated samples in each SDG section as appropriate.

- "E" – exceeds the calibration range of the method
- "D" – result taken from sample dilution
- "J" – concentration reported between the laboratory / instrument determined method detection limit (MDL) and the practical quantitation limit (PQL)
- "B" – analyte concentration in method blank exceeds reporting limit
- "U" – analyte not detected above MDL
- "*" – indicates analyte has exceeded batch or sample specific QA/QC control limits
- "M" – indicates matrix interference

A single QA/QC batch is defined as no more than 20 samples excluding method blanks (MB, LRB), fortified blanks (BS, LFB, LCS), matrix spikes (MS, SPK), and duplicates whether spiked or native (MSD, SPK DUP, DUP, LR).

Data Deliverables

All data deliverables are generated to be in compliance with USEPA R5 EDD format and loaded directly into the ATS GeoPortal project geodatabase software. This allows for real time integration and review by project management as the chemistry data pertains to soils information, site mapping, etc. Subsequent EDD formats were exported from the GeoPortal database based on client request. This data package constitutes a level II package. There were no hardcopy data summary sheets generated for this project.

_____ / March 14, 2019

Mark T. DeLong (Quality Assurance Coordinator)

_____ / March 14, 2019

Philip B. Simon (Laboratory Director)

CHAIN OF CUSTODY RECORDS

and

SAMPLE RECEIPT DOCUMENTATION

ATS Project Number: BENB-T01

ATS SDG(s): 0308191

Prepared By:
Ann Arbor Technical Services, Inc.
290 South Wagner Road
Ann Arbor, MI 48103

290 South Wagner Road
Ann Arbor, Michigan 48103
Tel. 734/995-0995 Fax. 734/995-3731
Michigan Laboratory ID: 9604
Wisconsin Laboratory ID: 998321720

ANN ARBOR TECHNICAL SERVICES, INC.
SAMPLE RECEIPT FORM (rev 072610)

Project Identification And General Sample Information

ATS Project Number: BENB-101
Number of Sample Locations: 1
Date: 3/8/19
Date Range: 3/7/19
Time: 10:30
Matrix *(choose those that apply)*: Water | Soil | Air | Industrial Product | Extract | Drinking Water | Sediment | Other (explain): O.L
SRF Number: 0308191
ATS Quote / Contract Number if applicable:
Choose One: Weekly | Monthly | Quarterly | Semi-Annual | Annual | N/A
Client PO Number:
Turn Around *(choose one)*: Standard | Rush | 4-7 Days | Other (explain): | On Hold
Analyst:

Sample Delivery Information

Delivered by *(choose one)*:
ATS — Name:
Commercial Carrier — Name: FED EX
Client Representative — Name:
Firm: BANN ENGINEERING
Address:
Telephone:
Email:

Chain of Custody Information

ATS COC *(choose one)*: Yes | No
Completed By *(choose one)*: ATS | Client
Client COC *(choose one)*: Yes | No
Completed By *(choose one)*: ATS | Client
Internal COC Completed *(choose one)*: Yes | No | N/A
Complete By *(choose one)*: ATS — Name:
Custody Seals *(choose one)*: None | Present / Intact | Present / Not Intact — Seal Number:

COC Matches Sample Labels: *Choose One:* Yes | No — Unknown
Anomaly Form Filled Out: *Choose One:* Yes | No
COC Matches Sample Labels: *Choose One:* Yes | No
Anomaly Form Filled Out: *Choose One:* Yes | No

Sample Receipt Information

Packaging *(choose those that apply)*: Cooler (s) | Cardboard | Hazardous | Other (explain):
Thermal Preservation:
At Time of Collection By: ATS | Client | N/A

	Container #1	Container #2	Container #3	Container #4	Container #5
Temperature Upon Receipt (°C or "On Ice"):	Ice				

Chemical Preservation By:
Pre-Preserved: ATS | Client | N/A — *See Chemical Preservation Verification Form*
At Time of Collection By: ATS | Client | N/A — *See Chemical Preservation Verification Form*
Upon Sample Receipt: ATS | Client | N/A — *See Chemical Preservation Verification Form*
ATS Sampling Containers *(choose one)*: Yes | No
Sample Integrity *(choose those that apply)*: Intact | Broken | Leaking | Headspace
Limited Volume *(choose one)*: Yes | No

Anomaly Form Filled Out: *Choose One:* Yes | No — See photo

Storage Location, Retention, And Disposition Information:

Ambient: #110
Cold Room:
Frozen:
VOC:
Sample Retention *(choose those that apply)*: 6 months | 1 year | Other (explain): Return
Sample Disposition *(choose one)*: Disposal | Return to Client

Subcontract Information (If applicable):

Laboratory:
Contact Name:
ATS PO Number:
ATS Contract Number:
Analyses:

SRF Distribution

Scanned

ATS Project Manager: By (Initials):
ATS Accounting: Date:
By (Initials):
Date:

Barr Engineering Co. Chain of Custody

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

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

COC Number: 58025

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr	Company:
Address: 325 S Lake	Address:
Name: ~~Matthew Haro~~ Ryan Erickson	Name:
email: RErickson@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: Tank 22 Response	Barr Project No: 49161092.07

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: Finger printing	Soil: % Solids	Preservative Code	Field Filtered Y/N
1. Tank22-product-1	–	–	–	03/07/19	9:25	O			A			N
2.												
3.												
4.												
5.												
6.												
7.												
8.												
9.												
10.												

Finger printing analysis

BARR USE ONLY	Relinquished by	On Ice? Y N	Date	Time	Received by	Date	Time
Sampled by: Matthew Haro	Matthew Haro	Y	03/07/19	16:00		3/8/19	10:30
Barr Proj. Manager: Ryan Erickson							
Barr DQ Manager: Jamestjet	Relinquished by:	On Ice? Y N	Date	Time	Received by:	Date	Time
Lab Name: ATS							
Lab Location: Ann Arbor							

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): 6.5 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.

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

Barr Engineering Co. Chain of Custody

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

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

COC Number: 58025

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr	Company:
Address: 325 S Lake	Address:
Name: ~~Matthew Hora~~ Ryan Erickson	Name:
email: REricksson@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: Tank 22 Response	Barr Project No: 49161092.07

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: Fingerprinting	Soil: % Solids	Preservative Code	Field Filtered Y/N
1. Tank22-product-1	–	–	–	03/07/19	9:25	O			A			N
2.												
3. Lab ID												
4. 0308191-1												
5.												
6.												
7.												
8.												
9.												
10.												

Fingerprinting Analysis

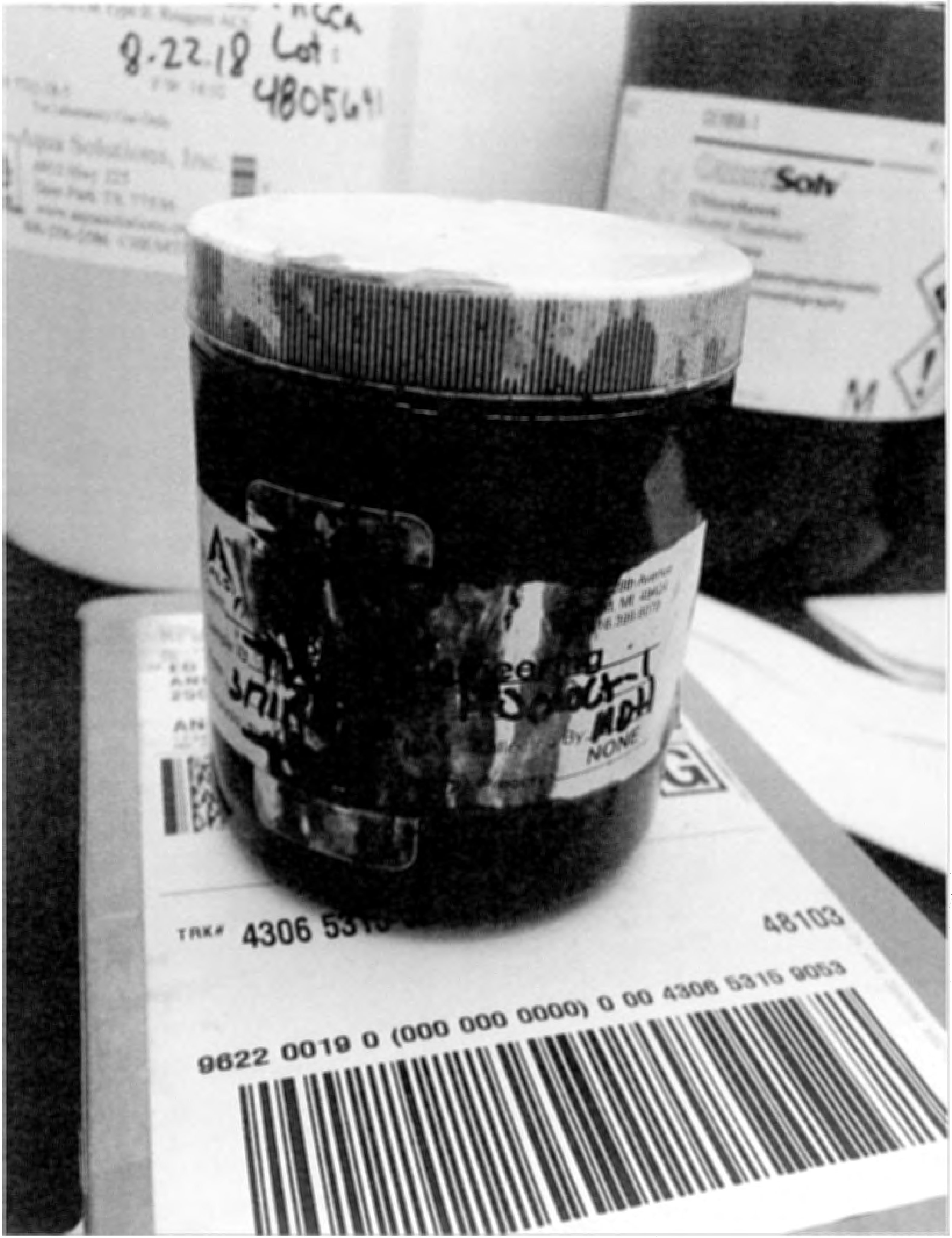
COPY COPY

BARR USE ONLY	Relinquished by	On Ice? Y N	Date	Time	Received by	Date	Time
Sampled by: Matthew Hora	Matthew Hora	Y	03/07/19	16:00			
Barr Proj. Manager: Ryan Erickson	Relinquished by:	On Ice? Y N	Date	Time	Received by:	Date	Time
Barr DQ Manager: Tamesa Jet							
Lab Name: ATS	Samples Shipped VIA: ☐ Courier ☐ Federal Express ☐ Sampler ☐ Other:				Air Bill Number:		
Lab Location: Ann Arbor	Lab WO:				Temperature on Receipt (°C): Yes	Custody Seal Intact? ☐ Y ☐ N ☐ None	

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

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

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


8.22.18
Lot
Solv
NONE
TRK# 4306 531
48103
9622 0019 0 (000 000 0000) 0 00 4306 5316 9053

0005B

100

Express US Airbill

FedEx Tracking Number 8139 1094 8349

Form ID No. 0215

Recipient's Copy

1 From

Date 3/7/19

Sender's Name Matthew Harre Phone 218 529-8200

Company BARR ENGINEERING

Address 325 S LAKE AVE STE 700

Dept./Floor/Suite/Room

City DULUTH State MN ZIP 55802-2325

2 Your Internal Billing Reference

3 To

Recipient's Name ATS Phone 734 761 4724

Company ATS

Address 705 zeeb rd

We cannot deliver to P.O. boxes or P.O. ZIP codes.

Dept./Floor/Suite/Room

Address

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

City Ann Arbor State MI ZIP 48103

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

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

0132158893

8139 1094 8349

4 Express Package Service * To most locations.

Packages up to 150 lbs.
For packages over 150 lbs., use the FedEx Express Freight US Airbill.

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.
- ☑ 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 ☑ 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.

☐ 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.

☐ No ☐ Yes — As per attached Shipper's Declaration. ☐ Yes — Shipper's Declaration not required. ☐ Dry Ice — Dry ice, 9, UN 1845 ____ x ____ kg

Restrictions apply for dangerous goods — see the current FedEx Service Guide. ☐ Cargo Aircraft Only

7 Payment *Bill to:*

Enter FedEx Acct. No. or Credit Card No. below. Obtain recip. Acct. No. ☐

☑ Sender Acct. No. in Section 1 will be billed. ☐ Recipient ☐ Third Party ☐ Credit Card ☐ Cash/Check

Total Packages ____ Total Weight ____ lbs. Credit Card Auth. ____

†Our liability is limited to US$100 unless you declare a higher value. See the current FedEx Service Guide for details.

611

5/15 • Part #163134 • ©1994–2015 FedEx • PRINTED IN U.S.A. SRM

fedex.com 1.800.GoFedEx 1.800.463.3339

fedex.com 1.800.GoFedEx 1.800.463.3339

FedEx TRK# 0215 8139 1094 8349

FRI - 08 MAR 10:30A
PRIORITY OVERNIGHT

48103
MI-US
DTW

XH ARBA

FID 5115555 07MAR19 DLHA 553C1/46D3/0C8A

ORGANIC ANALYSIS

TOTAL AND SATURATED HYDROCARBON ANALYSIS
USEPA METHOD SW8015-M

ATS Project Number: BENB-T01

ATS SDG: 0308191-B

Prepared By:
Ann Arbor Technical Services, Inc.
290 South Wagner Road
Ann Arbor, MI 48103

LABORATORY OPERATIONS
SAMPLE DELIVERY GROUP (SDG) CASE NARRATIVE

ATS Project Number: BENB-T01
ATS SDG: 0308191-B
Analysis Method: 8015-MOD

Sample Preparation

The sample was received in a glass container packed in a zip-lock bag and bubble wrap over-pack bag. Approximately 200 mg of the oil layer was drawn off with a capillary pipette and diluted for analysis in dichloromethane according to the ATS SOP for waste dilution. The resultant extract was then cleaned with silica gel and copper.

Anomalies Noted:

- **None**

Sample Analysis

Residue Upon Evaporation (RUE): RUE was determined by drying and weighing a 20uL portion of the pre-cleaned extract on a microbalance.

Total and Saturated Hydrocarbons: Samples were analyzed by GC/FID in accordance with US EPA method 8015-MOD. An initial calibration with at least five levels was used to quantitate the individual saturated hydrocarbons. Concentrations were reported to the method detection limit (MDL). Samples were reported on a wet weight basis.

Anomalies Noted:

- **None**

Calibration Verification

Method calibration was verified through the running of a mid-level calibration verification standard at a minimum of every 20 samples. All verification solutions and standards met the criteria with the following exceptions:

- **None**

Low system background was demonstrated through the analysis of instrument blanks at a minimum of every 20 samples. All instrument blanks met the acceptance criteria with the following exceptions:

- **None**

Consultants in Chemistry & Environmental Science
290 South Wagner Road, Ann Arbor, Michigan 48103 Tel 734/995-0995 Fax 734/995-3731

Internal Standards and Surrogates

Internal standards areas and retention times met the acceptance criteria with the following exceptions:

- **None**

Surrogate recoveries were within acceptance limits except for samples listed below:

Lab Sample ID	Constituent	Percent Recovery	Acceptance Limits
0308191-1	Pyrene-d10	178.5	50-150%
0308191-1 MS	Pyrene-d10	201.0	50-150%
0308191-1 MSD	Pyrene-d10	213.5	50-150%

- **An interfering compound co-eluting with Pyrene-D10 caused high recoveries of this surrogate in these samples.**

Laboratory Reagent Blanks

A laboratory reagent blank (LRB) was analyzed as part of the QA/QC batch. The LRB met the acceptance criteria with the following exceptions:

- **None**

Laboratory Fortified Blanks

A laboratory fortified blank (LFB) was analyzed as part of the QA/QC batch. The LFB met the acceptance criteria with the following exceptions:

- **None**

Matrix Spikes

A matrix spike (MS) and matrix spike duplicate (MSD) was analyzed as part of the QA/QC batch. The MS/MSD's met the acceptance criteria with the following exceptions:

Lab Sample ID	Constituent	Percent Recovery	Acceptance Limits
0308191-1 MS	n-Dotriacontane (C32)	143	70-130%
0308191-1 MS	n-Hentriacontane (C31)	69.7	70-130%
0308191-1 MS	n-Tritriacontane (C33)	133	70-130%
0308191-1 MSD	2,6,10,14-TETRAMETHYL PENTADECANE	66.1	70-130%
0308191-1 MSD	n-Dotriacontane (C32)	133	70-130%
0308191-1 MSD	n-Heptadecane (C17)	69.2	70-130%
0308191-1 MSD	n-Heptatriacontane (C37)	131	70-130%
0308191-1 MSD	n-Hexacosane (C26)	132	70-130%

- **In addition, for some compounds percent recovery was not reportable due to low spike levels relative to native concentrations in these MS/MSD samples**

Matrix Replicates

A matrix spike (MS) and matrix spike duplicate (MSD) was analyzed as part of the QA/QC batch. The replicates met the acceptance criteria with the following exceptions:

Lab Sample ID	Constituent	Percent Difference	Acceptance Limits
0308191-1 MS/MSD	n-Heptacosane (C27)	24.4	<20%
0308191-1 MS/MSD	n-Nonadecane (C19)	20.1	<20%
0308191-1 MS/MSD	n-Nonane (C9)	22.9	<20%
0308191-1 MS/MSD	n-Pentatriacontane (C35)	26.0	<20%
0308191-1 MS/MSD	n-Tetradecane (C14)	21.9	<20%
0308191-1 MS/MSD	n-Undecane (C11)	22.3	<20%

Sample Extract Dilutions

Samples containing compounds at concentrations above the initial calibration curve were diluted and reanalyzed for those compounds. The following sample extracts were diluted:

- **None**

_____ / March 14, 2019

Mark T. DeLong (Quality Assurance Coordinator)

_____ / March 14, 2019

Philip B. Simon (Laboratory Director)

ANN ARBOR TECHNICAL SERVICES, INC.

Laboratory Sample ID Summary

ATS Project Number	SDG Number	Analytical Method	Field Sample Identification	Laboratory Sample Identification
BENB-T01	0308191-B	SW8015M	Tank-22-Product-1	0308191-1

Total and Saturated Hydrocarbon Analysis
Data Summary Sheet

ATS Project Number	BENB-T01	**Percent Moisture**	NA
ATS SDG Number	0308191-B	**Preparation Date**	03/11/2019
Client Sample ID	Tank-22-Product-1	**Analysis Date:**	03/11/2019
Laboratory Sample ID	0308191-1	**Instrument**	3800 FID
Matrix	Oily Liquid	**Subsample (g)**	0.2068
Sample Date	03/07/2019	**Final Volume (mL)**	20
Analytical Method (USEPA)	SW8015M	**Dilution Factor**	1
Preparation Method (USEPA)	USEPA Method 3580A	**Basis**	WET
QC Batch Number	QCORG0311191-B	**Units**	mg/kg

Parameter	Chemical Identifier	Result	MDL	PQL	Qual
Hydrocarbons					
n-Nonane (C9)	111-84-2	2680	11.7	39.1	
n-Decane (C10)	124-18-5	2220	4.9	16.3	
n-Undecane (C11)	1120-21-4	2370	4.5	15.1	
n-Dodecane (C12)	112-40-3	1470	9.9	32.9	
n-Tridecane (C13)	629-50-5	1710	6.9	23.1	
2,6,10 Trimethyldodecane (1380)	3891-98-3	209	13.6	45.4	
n-Tetradecane (C14)	629-59-4	1810	13.6	45.4	
2,6,10 Trimethyltridecane (1470)	3891-99-4	562	10.9	36.3	
n-Pentadecane (C15)	629-62-9	677	10.9	36.3	
n-Hexadecane (C16)	544-76-3	942	10.6	35.2	
Norpristane (1650)	3892-00-0	593	12.1	40.3	
n-Heptadecane (C17)	629-78-7	780	12.1	40.3	
Pristane	1921-70-6	156	14.4	48.1	
n-Octadecane (C18)	593-45-3	853	7.3	24.5	
Phytane	638-36-8	182	11	36.7	
n-Nonadecane (C19)	629-92-5	560	8.6	28.5	
n-Eicosane (C20)	112-95-8	620	10.9	36.3	
n-Heneicosane (C21)	629-94-7	562	10.8	35.9	
n-Docosane (C22)	629-97-0	669	15.5	51.8	
n-Tricosane (C23)	638-67-5	292	13.2	43.8	
n-Tetracosane (C24)	646-31-1	270	13.8	46	
n-Pentacosane (C25)	629-99-2	635	11.4	38	
n-Hexacosane (C26)	630-01-3	287	9	30	
n-Heptacosane (C27)	593-49-7	319	10.1	33.5	
n-Octacosane (C28)	630-02-4	587	12.2	40.7	
n-Nonacosane (C29)	630-03-5	860	12.4	41.4	
n-Triacontane (C30)	638-68-6	391	14	46.6	
n-Hentriacontane (C31)	630-04-6	906	15.2	50.7	
n-Dotriacontane (C32)	544-85-4	174	14.9	49.5	
n-Tritriacontane (C33)	630-05-7	279	13.6	45.5	
n-Tetratriacontane (C34)	14167-59-0	333	16.1	53.7	
n-Pentatriacontane (C35)	630-07-9	253	16.7	55.8	
n-Hexatriacontane (C36)	630-06-8	179	20.4	67.8	
n-Heptatriacontane (C37)	7194-84-5	92.6	16.2	54.1	
n-Octatriacontane (C38)	7194-85-6	ND	23	76.7	U
n-Nonatriacontane (C39)	7194-86-7	88.7	21.5	71.5	
n-Tetracontane (C40)	4181-95-7	92.6	21.5	71.8	
Total Saturated Hydrocarbons (C9-C44)	TSHC9-C44	25700	1100	3600	
Total Resolvable Hydrocarbons (C9-C44)	TOTRESHC	93900	1100	3600	
Total Petroleum Hydrocarbons (C9-C44)	C9-C44	752000	1100	3600	
Residue Upon Evaporation	RUE	815534	-	-	
n-Heptadecane / Pristane Ratio	-	5.0	-	-	
n-Octadecane / Phytane Ratio	-	4.7	-	-	

Surrogate Recoveries			LCL	UCL	
2-Fluorobiphenyl	321-60-8	130.5	30	150	
Pyrene-D10	1718-52-1	178.5	50	150	

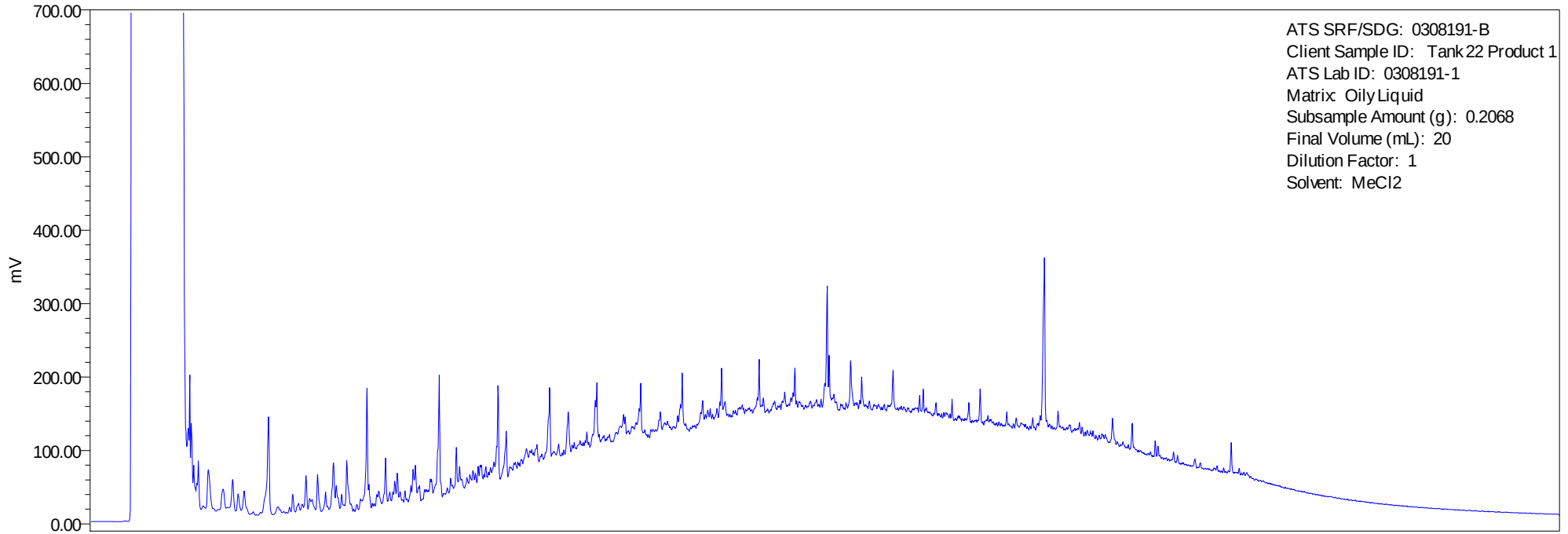
Comments

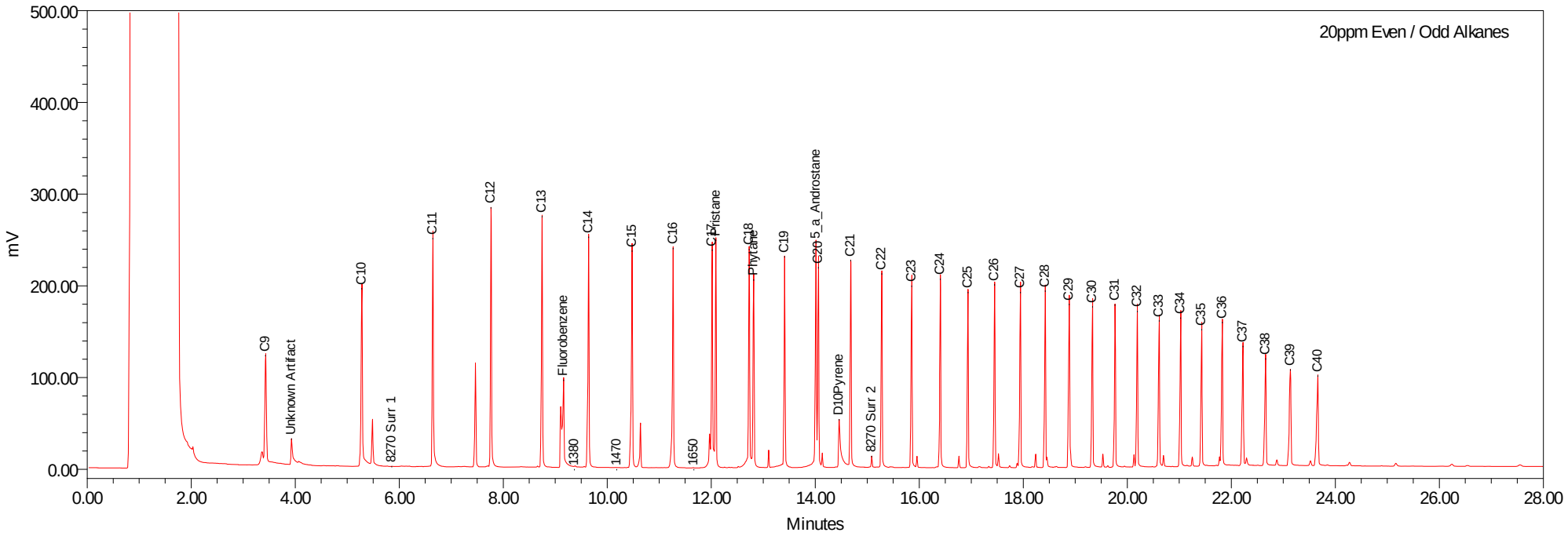
Matrix interference precluded lower detection limit.
All calculations performed prior to rounding.
MDL/PQL values assume 100% solids content.
nc = Not calculated.

Ann Arbor Technical Services, Inc.
290 South Wagner Road
Ann Arbor, Michigan 48103

Office: 734-995-0995
Fax: 734-995-3731

#BENB-T01 TPH/SHC Chromatogram





ANN ARBOR TECHNICAL SERVICES, INC.

Surrogate Recovery Summary

SW8015M

Sample Identification	Field Sample Identification	Instrument ID	Analysis Date	Percent Recovery 2-Fluorobiphenyl	Flag	Pyrene-D10	Flag
0308191-1	Tank-22-Product-1	3800 FID	03/11/2019	130.5		178.5	*
0308191-1 MS	Tank-22-Product-1 MS	3800 FID	03/11/2019	116.0		201.0	*
0308191-1 MSD	Tank-22-Product-1 MSD	3800 FID	03/12/2019	94.0		213.5	*
LFB-1 3/11/19	LFB-1 3/11/19	3800 FID	03/11/2019	68.5		115.5	
LRB-1 3/11/19	LRB-1 3/11/19	3800 FID	03/11/2019	86.4		138.0	

QA/QC Limits	
2-Fluorobiphenyl	30-150
Pyrene-D10	50-150

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Laboratory Reagent Blank (LRB) / Method Blank (MB)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Result	Units	Basis	Method Detection Limit	Reporting Detection Limit	Comments
LRB-1 3/11/19	03/11/2019	19:56:07	2,6,10 Trimethyldodecane (1380)	3891-98-3		mg/kg	WET	13.6	45.4	
LRB-1 3/11/19	03/11/2019	19:56:07	2,6,10 Trimethyltridecane (1470)	3891-99-4		mg/kg	WET	10.9	36.3	
LRB-1 3/11/19	03/11/2019	19:56:07	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6		mg/kg	WET	14.4	48.1	
LRB-1 3/11/19	03/11/2019	19:56:07	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8		mg/kg	WET	11	36.7	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Decane (C10)	124-18-5		mg/kg	WET	4.9	16.3	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Docosane (C22)	629-97-0		mg/kg	WET	15.5	51.8	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Dodecane (C12)	112-40-3		mg/kg	WET	9.9	32.9	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Dotriacontane (C32)	544-85-4		mg/kg	WET	14.9	49.5	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Eicosane (C20)	112-95-8		mg/kg	WET	10.9	36.3	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Heneicosane (C21)	629-94-7		mg/kg	WET	10.8	35.9	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Hentriacontane (C31)	630-04-6		mg/kg	WET	15.2	50.7	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Heptacosane (C27)	593-49-7		mg/kg	WET	10.1	33.5	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Heptadecane (C17)	629-78-7		mg/kg	WET	12.1	40.3	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Heptatriacontane (C37)	7194-84-5		mg/kg	WET	16.2	54.1	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Hexacosane (C26)	630-01-3		mg/kg	WET	9	30	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Hexadecane (C16)	544-76-3		mg/kg	WET	10.6	35.2	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Hexatriacontane (C36)	630-06-8		mg/kg	WET	20.4	67.8	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Nonacosane (C29)	630-03-5		mg/kg	WET	12.4	41.4	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Nonadecane (C19)	629-92-5		mg/kg	WET	8.6	28.5	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Nonane (C9)	111-84-2		mg/kg	WET	11.7	39.1	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Nonatriacontane (C39)	7194-86-7		mg/kg	WET	21.5	71.5	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Octacosane (C28)	630-02-4		mg/kg	WET	12.2	40.7	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Octadecane (C18)	593-45-3		mg/kg	WET	7.3	24.5	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Octatriacontane (C38)	7194-85-6		mg/kg	WET	23	76.7	
LRB-1 3/11/19	03/11/2019	19:56:07	Norpristane (1650)	3892-00-0		mg/kg	WET	12.1	40.3	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Laboratory Reagent Blank (LRB) / Method Blank (MB)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Result	Units	Basis	Method Detection Limit	Reporting Detection Limit	Comments
LRB-1 3/11/19	03/11/2019	19:56:07	n-Pentacosane (C25)	629-99-2		mg/kg	WET	11.4	38	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Pentadecane (C15)	629-62-9		mg/kg	WET	10.9	36.3	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Pentatriacontane (C35)	630-07-9		mg/kg	WET	16.7	55.8	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Tetracontane (C40)	4181-95-7		mg/kg	WET	21.5	71.8	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Tetracosane (C24)	646-31-1		mg/kg	WET	13.8	46	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Tetradecane (C14)	629-59-4		mg/kg	WET	13.6	45.4	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Tetratriacontane (C34)	14167-59-0		mg/kg	WET	16.1	53.7	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Triacontane (C30)	638-68-6		mg/kg	WET	14	46.6	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Tricosane (C23)	638-67-5		mg/kg	WET	13.2	43.8	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Tridecane (C13)	629-50-5		mg/kg	WET	6.9	23.1	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Tritriacontane (C33)	630-05-7		mg/kg	WET	13.6	45.5	
LRB-1 3/11/19	03/11/2019	19:56:07	n-Undecane (C11)	1120-21-4		mg/kg	WET	4.5	15.1	
LRB-1 3/11/19	03/11/2019	19:56:07	Total Petroleum Hydrocarbons (C9-C44)	C9-C44		mg/kg	WET	1100	3600	
LRB-1 3/11/19	03/11/2019	19:56:07	Total Resolvable Hydrocarbons	TOTRESHC		mg/kg	WET	1100	3600	
LRB-1 3/11/19	03/11/2019	19:56:07	Total Saturated Hydrocarbons (C9-C44)	TSHC9-C44		mg/kg	WET	1100	3600	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY ACCURACY SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Laboratory Fortified Blank (LFB) / Laboratory Control Sample (LCS)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Sample Concentration	Spike Added	Measured Concentration	Units	Basis	Percent Recovery	LCL	UCL	Comments
LFB-1 3/11/19	03/11/2019	20:30:39	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6		200	170	mg/kg	WET	85.2	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8		200	182	mg/kg	WET	91.0	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Decane (C10)	124-18-5		200	219	mg/kg	WET	110	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Docosane (C22)	629-97-0		200	209	mg/kg	WET	105	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Dodecane (C12)	112-40-3		200	245	mg/kg	WET	123	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Dotriacontane (C32)	544-85-4		200	228	mg/kg	WET	114	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Eicosane (C20)	112-95-8		200	231	mg/kg	WET	116	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Heneicosane (C21)	629-94-7		200	193	mg/kg	WET	96.5	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Hentriacontane (C31)	630-04-6		200	257	mg/kg	WET	128	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Heptacosane (C27)	593-49-7		200	179	mg/kg	WET	89.5	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Heptadecane (C17)	629-78-7		200	214	mg/kg	WET	107	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Heptatriacontane (C37)	7194-84-5		200	209	mg/kg	WET	105	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Hexacosane (C26)	630-01-3		200	223	mg/kg	WET	111	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Hexadecane (C16)	544-76-3		200	198	mg/kg	WET	98.8	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Hexatriacontane (C36)	630-06-8		200	178	mg/kg	WET	89.2	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Nonacosane (C29)	630-03-5		200	232	mg/kg	WET	116	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Nonadecane (C19)	629-92-5		200	188	mg/kg	WET	94.1	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Nonane (C9)	111-84-2		200	235	mg/kg	WET	118	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Nonatriacontane (C39)	7194-86-7		200	182	mg/kg	WET	91.1	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Octacosane (C28)	630-02-4		200	246	mg/kg	WET	123	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Octadecane (C18)	593-45-3		200	233	mg/kg	WET	117	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Octatriacontane (C38)	7194-85-6		200	201	mg/kg	WET	100	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Pentacosane (C25)	629-99-2		200	224	mg/kg	WET	112	70	130	

Comments
Calculations performed prior to rounding.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY ACCURACY SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Laboratory Fortified Blank (LFB) / Laboratory Control Sample (LCS)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Sample Concentration	Spike Added	Measured Concentration	Units	Basis	Percent Recovery	LCL	UCL	Comments
LFB-1 3/11/19	03/11/2019	20:30:39	n-Pentadecane (C15)	629-62-9		200	199	mg/kg	WET	99.6	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Pentatriacontane (C35)	630-07-9		200	195	mg/kg	WET	97.4	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Tetracontane (C40)	4181-95-7		200	172	mg/kg	WET	86.1	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Tetracosane (C24)	646-31-1		200	217	mg/kg	WET	108	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Tetradecane (C14)	629-59-4		200	241	mg/kg	WET	120	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Tetratriacontane (C34)	14167-59-0		200	208	mg/kg	WET	104	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Triacontane (C30)	638-68-6		200	257	mg/kg	WET	129	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Tricosane (C23)	638-67-5		200	211	mg/kg	WET	105	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Tridecane (C13)	629-50-5		200	254	mg/kg	WET	127	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Tritriacontane (C33)	630-05-7		200	218	mg/kg	WET	109	70	130	
LFB-1 3/11/19	03/11/2019	20:30:39	n-Undecane (C11)	1120-21-4		200	224	mg/kg	WET	112	70	130	

Comments
Calculations performed prior to rounding.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY ACCURACY SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Matrix Spike (MS)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Sample Concentration	Spike Added	Measured Concentration	Units	Basis	Percent Recovery	LCL	UCL	Comments
0308191-1 MS	03/11/2019	23:22:32	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6	156	941	850	mg/kg	WET	73.8	70	130	
0308191-1 MS	03/11/2019	23:22:32	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8	182	941	1090	mg/kg	WET	96.6	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Decane (C10)	124-18-5	2220	941	NR	mg/kg	WET	nc	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Docosane (C22)	629-97-0	669	941	1780	mg/kg	WET	118	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Dodecane (C12)	112-40-3	1470	941	NR	mg/kg	WET	nc	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Dotriacontane (C32)	544-85-4	174	941	1520	mg/kg	WET	143	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Eicosane (C20)	112-95-8	620	941	1820	mg/kg	WET	128	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Heneicosane (C21)	629-94-7	562	941	1740	mg/kg	WET	126	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Hentriacontane (C31)	630-04-6	906	941	1560	mg/kg	WET	69.7	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Heptacosane (C27)	593-49-7	319	941	1350	mg/kg	WET	110	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Heptadecane (C17)	629-78-7	780	941	1550	mg/kg	WET	82.2	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Heptatriacontane (C37)	7194-84-5	92.6	941	1240	mg/kg	WET	122	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Hexacosane (C26)	630-01-3	287	941	1440	mg/kg	WET	122	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Hexadecane (C16)	544-76-3	942	941	1730	mg/kg	WET	83.8	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Hexatriacontane (C36)	630-06-8	179	941	1380	mg/kg	WET	128	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Nonacosane (C29)	630-03-5	860	941	1700	mg/kg	WET	89.2	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Nonadecane (C19)	629-92-5	560	941	1510	mg/kg	WET	101	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Nonane (C9)	111-84-2	2680	941	NR	mg/kg	WET	nc	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Nonatriacontane (C39)	7194-86-7	88.7	941	1080	mg/kg	WET	106	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Octacosane (C28)	630-02-4	587	941	1680	mg/kg	WET	116	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Octadecane (C18)	593-45-3	853	941	1800	mg/kg	WET	101	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Octatriacontane (C38)	7194-85-6		941	1150	mg/kg	WET	122	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Pentacosane (C25)	629-99-2	635	941	1660	mg/kg	WET	108	70	130	

Comments
Calculations performed prior to rounding.
NR - Not Reportable due to inadequate spiking levels.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY ACCURACY SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Matrix Spike (MS)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Sample Concentration	Spike Added	Measured Concentration	Units	Basis	Percent Recovery	LCL	UCL	Comments
0308191-1 MS	03/11/2019	23:22:32	n-Pentadecane (C15)	629-62-9	677	941	1470	mg/kg	WET	84.5	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Pentatriacontane (C35)	630-07-9	253	941	1430	mg/kg	WET	125	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Tetracontane (C40)	4181-95-7	92.6	941	1010	mg/kg	WET	97.4	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Tetracosane (C24)	646-31-1	270	941	1370	mg/kg	WET	117	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Tetradecane (C14)	629-59-4	1810	941	NR	mg/kg	WET	nc	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Tetratriacontane (C34)	14167-59-0	333	941	1560	mg/kg	WET	130	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Triacontane (C30)	638-68-6	391	941	1600	mg/kg	WET	129	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Tricosane (C23)	638-67-5	292	941	1510	mg/kg	WET	129	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Tridecane (C13)	629-50-5	1710	941	NR	mg/kg	WET	nc	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Tritriacontane (C33)	630-05-7	279	941	1530	mg/kg	WET	133	70	130	
0308191-1 MS	03/11/2019	23:22:32	n-Undecane (C11)	1120-21-4	2370	941	NR	mg/kg	WET	nc	70	130	

Comments
Calculations performed prior to rounding.
NR - Not Reportable due to inadequate spiking levels.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY ACCURACY SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Matrix Spike Duplicate (MSD)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Sample Concentration	Spike Added	Measured Concentration	Units	Basis	Percent Recovery	LCL	UCL	Comments
0308191-1 MSD	03/12/2019	00:31:22	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6	156	918	762	mg/kg	WET	66.1	70	130	
0308191-1 MSD	03/12/2019	00:31:22	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8	182	918	938	mg/kg	WET	82.4	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Decane (C10)	124-18-5	2220	918	NR	mg/kg	WET	nc	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Docosane (C22)	629-97-0	669	918	1720	mg/kg	WET	115	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Dodecane (C12)	112-40-3	1470	918	NR	mg/kg	WET	nc	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Dotriacontane (C32)	544-85-4	174	918	1400	mg/kg	WET	133	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Eicosane (C20)	112-95-8	620	918	1680	mg/kg	WET	116	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Heneicosane (C21)	629-94-7	562	918	1520	mg/kg	WET	104	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Hentriacontane (C31)	630-04-6	906	918	1610	mg/kg	WET	77.1	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Heptacosane (C27)	593-49-7	319	918	1060	mg/kg	WET	80.3	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Heptadecane (C17)	629-78-7	780	918	1420	mg/kg	WET	69.2	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Heptatriacontane (C37)	7194-84-5	92.6	918	1300	mg/kg	WET	131	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Hexacosane (C26)	630-01-3	287	918	1500	mg/kg	WET	132	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Hexadecane (C16)	544-76-3	942	918	1920	mg/kg	WET	107	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Hexatriacontane (C36)	630-06-8	179	918	1160	mg/kg	WET	107	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Nonacosane (C29)	630-03-5	860	918	1510	mg/kg	WET	70.3	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Nonadecane (C19)	629-92-5	560	918	1240	mg/kg	WET	73.8	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Nonane (C9)	111-84-2	2680	918	NR	mg/kg	WET	nc	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Nonatriacontane (C39)	7194-86-7	88.7	918	1180	mg/kg	WET	119	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Octacosane (C28)	630-02-4	587	918	1600	mg/kg	WET	110	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Octadecane (C18)	593-45-3	853	918	1580	mg/kg	WET	79.4	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Octatriacontane (C38)	7194-85-6		918	1120	mg/kg	WET	122	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Pentacosane (C25)	629-99-2	635	918	1480	mg/kg	WET	92.3	70	130	

Comments
Calculations performed prior to rounding.
NR - Not Reportable due to inadequate spiking levels.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY ACCURACY SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Matrix Spike Duplicate (MSD)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Sample Concentration	Spike Added	Measured Concentration	Units	Basis	Percent Recovery	LCL	UCL	Comments
0308191-1 MSD	03/12/2019	00:31:22	n-Pentadecane (C15)	629-62-9	677	918	1550	mg/kg	WET	95.0	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Pentatriacontane (C35)	630-07-9	253	918	1100	mg/kg	WET	92.1	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Tetracontane (C40)	4181-95-7	92.6	918	1000	mg/kg	WET	99.1	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Tetracosane (C24)	646-31-1	270	918	1390	mg/kg	WET	122	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Tetradecane (C14)	629-59-4	1810	918	NR	mg/kg	WET	nc	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Tetratriacontane (C34)	14167-59-0	333	918	1310	mg/kg	WET	107	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Triacontane (C30)	638-68-6	391	918	1530	mg/kg	WET	123	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Tricosane (C23)	638-67-5	292	918	1460	mg/kg	WET	127	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Tridecane (C13)	629-50-5	1710	918	NR	mg/kg	WET	nc	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Tritriacontane (C33)	630-05-7	279	918	1270	mg/kg	WET	107	70	130	
0308191-1 MSD	03/12/2019	00:31:22	n-Undecane (C11)	1120-21-4	2370	918	NR	mg/kg	WET	nc	70	130	

Comments
Calculations performed prior to rounding.
NR - Not Reportable due to inadequate spiking levels.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY PRECISION SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Result	Mean	Units	Basis	RPD	Control Limit	Comments
0308191-1 MS	03/11/2019	23:22:32	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6	850		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6	762	806	mg/kg	WET	10.8	20	
0308191-1 MS	03/11/2019	23:22:32	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8	1090		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8	938	1010	mg/kg	WET	15.0	20	
0308191-1 MS	03/11/2019	23:22:32	n-Decane (C10)	124-18-5	3320		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Decane (C10)	124-18-5	3220	3270	mg/kg	WET	3.19	20	
0308191-1 MS	03/11/2019	23:22:32	n-Docosane (C22)	629-97-0	1780		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Docosane (C22)	629-97-0	1720	1750	mg/kg	WET	3.28	20	
0308191-1 MS	03/11/2019	23:22:32	n-Dodecane (C12)	112-40-3	2500		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Dodecane (C12)	112-40-3	2060	2280	mg/kg	WET	19.6	20	
0308191-1 MS	03/11/2019	23:22:32	n-Dotriacontane (C32)	544-85-4	1520		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Dotriacontane (C32)	544-85-4	1400	1460	mg/kg	WET	8.69	20	
0308191-1 MS	03/11/2019	23:22:32	n-Eicosane (C20)	112-95-8	1820		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Eicosane (C20)	112-95-8	1680	1750	mg/kg	WET	7.84	20	
0308191-1 MS	03/11/2019	23:22:32	n-Heneicosane (C21)	629-94-7	1740		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Heneicosane (C21)	629-94-7	1520	1630	mg/kg	WET	14.0	20	
0308191-1 MS	03/11/2019	23:22:32	n-Hentriacontane (C31)	630-04-6	1560		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Hentriacontane (C31)	630-04-6	1610	1590	mg/kg	WET	3.28	20	
0308191-1 MS	03/11/2019	23:22:32	n-Heptacosane (C27)	593-49-7	1350		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Heptacosane (C27)	593-49-7	1060	1200	mg/kg	WET	24.4	20	
0308191-1 MS	03/11/2019	23:22:32	n-Heptadecane (C17)	629-78-7	1550		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Heptadecane (C17)	629-78-7	1420	1480	mg/kg	WET	9.32	20	
0308191-1 MS	03/11/2019	23:22:32	n-Heptatriacontane (C37)	7194-84-5	1240		mg/kg	WET			

Comments
Calculations performed prior to rounding.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY PRECISION SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Result	Mean	Units	Basis	RPD	Control Limit	Comments
0308191-1 MSD	03/12/2019	00:31:22	n-Heptatriacontane (C37)	7194-84-5	1300	1270	mg/kg	WET	4.80	20	
0308191-1 MS	03/11/2019	23:22:32	n-Hexacosane (C26)	630-01-3	1440		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Hexacosane (C26)	630-01-3	1500	1470	mg/kg	WET	4.24	20	
0308191-1 MS	03/11/2019	23:22:32	n-Hexadecane (C16)	544-76-3	1730		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Hexadecane (C16)	544-76-3	1920	1830	mg/kg	WET	10.6	20	
0308191-1 MS	03/11/2019	23:22:32	n-Hexatriacontane (C36)	630-06-8	1380		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Hexatriacontane (C36)	630-06-8	1160	1270	mg/kg	WET	17.0	20	
0308191-1 MS	03/11/2019	23:22:32	n-Nonacosane (C29)	630-03-5	1700		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Nonacosane (C29)	630-03-5	1510	1600	mg/kg	WET	12.1	20	
0308191-1 MS	03/11/2019	23:22:32	n-Nonadecane (C19)	629-92-5	1510		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Nonadecane (C19)	629-92-5	1240	1380	mg/kg	WET	20.1	20	
0308191-1 MS	03/11/2019	23:22:32	n-Nonane (C9)	111-84-2	3810		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Nonane (C9)	111-84-2	3030	3420	mg/kg	WET	22.9	20	
0308191-1 MS	03/11/2019	23:22:32	n-Nonatriacontane (C39)	7194-86-7	1080		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Nonatriacontane (C39)	7194-86-7	1180	1130	mg/kg	WET	8.45	20	
0308191-1 MS	03/11/2019	23:22:32	n-Octacosane (C28)	630-02-4	1680		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Octacosane (C28)	630-02-4	1600	1640	mg/kg	WET	4.73	20	
0308191-1 MS	03/11/2019	23:22:32	n-Octadecane (C18)	593-45-3	1800		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Octadecane (C18)	593-45-3	1580	1690	mg/kg	WET	13.0	20	
0308191-1 MS	03/11/2019	23:22:32	n-Octatriacontane (C38)	7194-85-6	1150		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Octatriacontane (C38)	7194-85-6	1120	1140	mg/kg	WET	2.29	20	
0308191-1 MS	03/11/2019	23:22:32	n-Pentacosane (C25)	629-99-2	1660		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Pentacosane (C25)	629-99-2	1480	1570	mg/kg	WET	11.0	20	

Comments
Calculations performed prior to rounding.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY PRECISION SUMMARY

Method: SW8015M
QA/QC Batch Number: QCORG0311191-B
SDG 0308191-B
Project Number: BENB-T01
Report Date: 3/14/2019

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Lab Sample ID	Analysis Date	Analysis Time	Chemical Name	CAS	Result	Mean	Units	Basis	RPD	Control Limit	Comments
0308191-1 MS	03/11/2019	23:22:32	n-Pentadecane (C15)	629-62-9	1470		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Pentadecane (C15)	629-62-9	1550	1510	mg/kg	WET	5.05	20	
0308191-1 MS	03/11/2019	23:22:32	n-Pentatriacontane (C35)	630-07-9	1430		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Pentatriacontane (C35)	630-07-9	1100	1260	mg/kg	WET	26.0	20	
0308191-1 MS	03/11/2019	23:22:32	n-Tetracontane (C40)	4181-95-7	1010		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Tetracontane (C40)	4181-95-7	1000	1010	mg/kg	WET	0.689	20	
0308191-1 MS	03/11/2019	23:22:32	n-Tetracosane (C24)	646-31-1	1370		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Tetracosane (C24)	646-31-1	1390	1380	mg/kg	WET	1.68	20	
0308191-1 MS	03/11/2019	23:22:32	n-Tetradecane (C14)	629-59-4	2800		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Tetradecane (C14)	629-59-4	2250	2520	mg/kg	WET	21.9	20	
0308191-1 MS	03/11/2019	23:22:32	n-Tetratriacontane (C34)	14167-59-0	1560		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Tetratriacontane (C34)	14167-59-0	1310	1440	mg/kg	WET	17.2	20	
0308191-1 MS	03/11/2019	23:22:32	n-Triacontane (C30)	638-68-6	1600		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Triacontane (C30)	638-68-6	1530	1560	mg/kg	WET	5.05	20	
0308191-1 MS	03/11/2019	23:22:32	n-Tricosane (C23)	638-67-5	1510		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Tricosane (C23)	638-67-5	1460	1480	mg/kg	WET	3.15	20	
0308191-1 MS	03/11/2019	23:22:32	n-Tridecane (C13)	629-50-5	2990		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Tridecane (C13)	629-50-5	2530	2760	mg/kg	WET	16.7	20	
0308191-1 MS	03/11/2019	23:22:32	n-Tritriacontane (C33)	630-05-7	1530		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Tritriacontane (C33)	630-05-7	1270	1400	mg/kg	WET	19.0	20	
0308191-1 MS	03/11/2019	23:22:32	n-Undecane (C11)	1120-21-4	3520		mg/kg	WET			
0308191-1 MSD	03/12/2019	00:31:22	n-Undecane (C11)	1120-21-4	2810	3160	mg/kg	WET	22.3	20	

Comments
Calculations performed prior to rounding.

Ryan E. Erickson

From: Peter Simon <Peter.Simon@annarbortechnicalservices.com>
Sent: Thursday, March 14, 2019 2:56 PM
To: Ryan E. Erickson; James E. Taraldsen
Cc: Philip Simon; Sarah Stubblefield; Alex.Smith@enbridge.com
Subject: RE: Tank 22 Product for Fingerprint Analysis
Attachments: SDG 0308191 Level II.pdf

Ryan,

Attached is the USEPA Level II data report for the oils sample we received March 8, 2019 associated with the Enbridge Tank 22 project in Superior Wisconsin. A quick look at the SHC profile and chromatogram for the sample strongly suggests a fresh crude oil with limited weathering (if any).

Peter

Peter M. Simon | **Sr. Project Manager**
Office. +734.995.0995 • Fax. +734.995.3731 • Mobile. +734.368.4724
Email. Peter.Simon@AnnArborTechnicalServices.com

Ann Arbor Technical Services, Inc.
290 South Wagner Road | Ann Arbor, Michigan 48103
United States of America
www.AnnArborTechnicalServices.com

Consultants in Chemistry & Environmental Science


Please consider the environment before printing this email.

From: Ryan E. Erickson <RErickson@barr.com>
Sent: Monday, March 11, 2019 5:57 PM
To: Peter Simon <Peter.Simon@annarbortechnicalservices.com>; James E. Taraldsen <JTaraldsen@barr.com>
Cc: Philip Simon <Philip.Simon@annarbortechnicalservices.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>; Alex.Smith@enbridge.com
Subject: RE: Tank 22 Product for Fingerprint Analysis

Hey Peter,
I am just checking on the estimated analysis completion timeline so that we can keep an eye out for it. Thanks for the info.

Ryan E. Erickson, PG

Senior Geologist
Duluth, MN office: 218.529.7112
fax: 218.529.8202
cell: 612.418.0166
rerickson@barr.com
www.barr.com

From: Peter Simon <Peter.Simon@annarbortechnicalservices.com>
Sent: Friday, March 08, 2019 10:48 AM
To: James E. Taraldsen <JTaraldsen@barr.com>
Cc: Ryan E. Erickson <RErickson@barr.com>; Philip Simon <Philip.Simon@annarbortechnicalservices.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>
Subject: RE: Tank 22 Product for Fingerprint Analysis

Hi Jim,

The Tank 22 product samples was received intact this morning.

We have not yet received the recovered/soil/oil sample. I'll let you know when that arrives.

Peter

From: James E. Taraldsen <JTaraldsen@barr.com>
Sent: Friday, March 8, 2019 9:25 AM
To: Peter Simon <Peter.Simon@annarbortechnicalservices.com>
Cc: Ryan E. Erickson <RErickson@barr.com>; Philip Simon <Philip.Simon@annarbortechnicalservices.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>
Subject: RE: Tank 22 Product for Fingerprint Analysis

Hi Peter,

Thanks for the update.

Jim

James E. Taraldsen

Senior Data Quality Specialist
Duluth, MN office: 218.529.7138
JTaraldsen@barr.com
www.barr.com

From: Peter Simon <Peter.Simon@annarbortechnicalservices.com>
Sent: Friday, March 8, 2019 4:44 AM
To: James E. Taraldsen <JTaraldsen@barr.com>
Cc: Ryan E. Erickson <RErickson@barr.com>; Philip Simon <Philip.Simon@annarbortechnicalservices.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>
Subject: Re: Tank 22 Product for Fingerprint Analysis

James and Ryan,

Thank you for the communication and coordinating the sample collection/shipping. We will look for the samples this morning and let you know when they arrive.

I will check with the labs current schedule and get back to you today with a projected schedule for data deliverables based on the scope you have requested and our lab schedule.

The scope as of right now includes the analysis of total and saturated hydrocarbons and n-alkanes by USEPA 8015m on two samples plus two QA/QC samples (tank 22 and recovered sample plus a matrix spike and matrix spike duplicate which are required by the method for qc).

Thanks

Peter

From: James E. Taraldsen <jtaraldsen@barr.com>
Sent: Thursday, March 7, 2019 10:27 PM
To: Peter Simon
Cc: Ryan E. Erickson
Subject: Tank 22 Product for Fingerprint Analysis

Hi Peter,

The product sample collected from Tank 22 was shipped this afternoon, and you will receive the sample in the morning. As Ryan mentioned in an earlier email, we would like the sample fingerprint analyzed to determine if the product is weathered, indicating an old release, or if this is a recent release. We would like this information as soon as possible. Please let us know if you have any questions when you have received the sample. Thanks!!!!

Jim

James E. Taraldsen

Senior Data Quality Specialist
Duluth, MN office: 218.529.7138
JTaraldsen@barr.com
www.barr.com

ALS Laboratory Report for Confirmation Soil Sample

08-May-2019

Jim Taraldsen
Barr Engineering Company
4300 Market Pointe Drive
Suite 200
Minneapolis, MN 55435

Re: **ENB Tank 22 Response (49161374.07)** Work Order: **19050225**

Dear Jim,

ALS Environmental received 1 sample on 03-May-2019 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 10.

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: 08-May-19

Client: Barr Engineering Company
Project: ENB Tank 22 Response (49161374.07)
Work Order: **19050225**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
19050225-01	TK22-S-1	Soil		5/2/2019 12:10	5/3/2019 09:00	☐

ALS Group, USA

Date: 08-May-19

Client: Barr Engineering Company
Project: ENB Tank 22 Response (49161374.07)
WorkOrder: **19050225**

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: ENB Tank 22 Response (49161374.07)
Work Order: 19050225

Case Narrative

Samples for the above noted Work Order were received on 05/03/19. 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: *08-May-19*

Client: Barr Engineering Company
Project: ENB Tank 22 Response (49161374.07)
Sample ID: TK22-S-1
Collection Date: 5/2/2019 12:10 PM

Work Order: 19050225
Lab ID: 19050225-01
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 5/3/19		Analyst: **SHW**
1,2,4-Trimethylbenzene	**3.1**	J	**2.4**	**13**	**µg/Kg-dry**	1	5/7/2019 07:40
1,3,5-Trimethylbenzene	U		4.0	13	µg/Kg-dry	1	5/7/2019 07:40
Benzene	U		2.2	13	µg/Kg-dry	1	5/7/2019 07:40
Ethylbenzene	U		2.8	13	µg/Kg-dry	1	5/7/2019 07:40
m,p-Xylene	U		6.2	26	µg/Kg-dry	1	5/7/2019 07:40
Naphthalene	U		3.6	44	µg/Kg-dry	1	5/7/2019 07:40
o-Xylene	U		5.1	13	µg/Kg-dry	1	5/7/2019 07:40
Toluene	U		3.6	13	µg/Kg-dry	1	5/7/2019 07:40
Xylenes, Total	U		11	39	µg/Kg-dry	1	5/7/2019 07:40
Surr: 1,2-Dichloroethane-d4	*107*			*70-130*	*%REC*	1	5/7/2019 07:40
Surr: 4-Bromofluorobenzene	*102*			*70-130*	*%REC*	1	5/7/2019 07:40
Surr: Dibromofluoromethane	*93.2*			*70-130*	*%REC*	1	5/7/2019 07:40
Surr: Toluene-d8	*96.9*			*70-130*	*%REC*	1	5/7/2019 07:40
MOISTURE		Method: **SW3550C**					Analyst: **KTP**
Moisture	**25**		**0.10**	**0.10**	**% of sample**	1	5/7/2019 14:25

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

ALS Group, USA

Date: 08-May-19

Client: Barr Engineering Company

QC BATCH REPORT

Work Order: 19050225

Project: ENB Tank 22 Response (49161374.07)

Batch ID: **135549** Instrument ID **VMS9** Method: **SW8260C**

MBLK Sample ID: **MBLK-135549-135549** Units: **µg/Kg-dry** Analysis Date: **5/6/2019 12:24 PM**

Client ID: Run ID: **VMS9_190506A** SeqNo: **5644330** Prep Date: **5/3/2019** 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	22	30								
1,3,5-Trimethylbenzene	U	35	100								
Benzene	U	5.1	30								
Ethylbenzene	U	6.3	30								
m,p-Xylene	U	40	60								
Naphthalene	U	72	100								
o-Xylene	U	12	30								
Toluene	U	8.2	30								
Xylenes, Total	U	40	90								
Surr: 1,2-Dichloroethane-d4	*976.5*	0	0	*1000*	0	*97.6*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*968.5*	0	0	*1000*	0	*96.8*	*70-130*	0			
Surr: Dibromofluoromethane	*906*	0	0	*1000*	0	*90.6*	*70-130*	0			
Surr: Toluene-d8	*956*	0	0	*1000*	0	*95.6*	*70-130*	0			

LCS Sample ID: **LCS-135549-135549** Units: **µg/Kg-dry** Analysis Date: **5/6/2019 11:37 AM**

Client ID: Run ID: **VMS9_190506A** SeqNo: **5644329** Prep Date: **5/3/2019** 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	916	22	30	1000	0	91.6	65-135	0			
1,3,5-Trimethylbenzene	989	35	100	1000	0	98.9	65-135	0			
Benzene	1002	5.1	30	1000	0	100	75-125	0			
Ethylbenzene	999.5	6.3	30	1000	0	100	75-125	0			
m,p-Xylene	1972	40	60	2000	0	98.6	80-125	0			
Naphthalene	937	72	100	1000	0	93.7	40-140	0			
o-Xylene	992	12	30	1000	0	99.2	75-125	0			
Toluene	984.5	8.2	30	1000	0	98.4	70-125	0			
Xylenes, Total	2964	40	90	3000	0	98.8	75-125	0			
Surr: 1,2-Dichloroethane-d4	*966*	0	0	*1000*	0	*96.6*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*977.5*	0	0	*1000*	0	*97.8*	*70-130*	0			
Surr: Dibromofluoromethane	*1016*	0	0	*1000*	0	*102*	*70-130*	0			
Surr: Toluene-d8	*1008*	0	0	*1000*	0	*101*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 19050225
Project: ENB Tank 22 Response (49161374.07)

QC BATCH REPORT

Batch ID: **135549** Instrument ID **VMS9** Method: **SW8260C**

MS Sample ID: **19050227-02A MS** Units: **µg/Kg-dry** Analysis Date: **5/7/2019 02:31 PM**
Client ID: Run ID: **VMS9_190507A** SeqNo: **5645337** Prep Date: **5/3/2019** 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	1149	23	31	1027	0	112	65-135	0			
1,3,5-Trimethylbenzene	1240	36	100	1027	0	121	65-135	0			
Benzene	1187	5.3	31	1027	0	116	75-125	0			
Ethylbenzene	1187	6.5	31	1027	0	116	75-125	0			
m,p-Xylene	2409	41	62	2053	0	117	80-125	0			
Naphthalene	1130	74	100	1027	0	110	40-140	0			
o-Xylene	1181	12	31	1027	0	115	75-125	0			
Toluene	1148	8.4	31	1027	0	112	70-125	0			
Xylenes, Total	3590	41	92	3080	0	117	75-125	0			
Surr: 1,2-Dichloroethane-d4	*990.2*	0	0	*1027*	0	*96.4*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1025*	0	0	*1027*	0	*99.8*	*70-130*	0			
Surr: Dibromofluoromethane	*997.4*	0	0	*1027*	0	*97.2*	*70-130*	0			
Surr: Toluene-d8	*969.7*	0	0	*1027*	0	*94.4*	*70-130*	0			

MSD Sample ID: **19050227-02A MSD** Units: **µg/Kg-dry** Analysis Date: **5/7/2019 02:46 PM**
Client ID: Run ID: **VMS9_190507A** SeqNo: **5645338** Prep Date: **5/3/2019** 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	1037	22	30	990.1	0	105	65-135	1149	10.3	30	
1,3,5-Trimethylbenzene	1127	35	99	990.1	0	114	65-135	1240	9.51	30	
Benzene	1090	5.1	30	990.1	0	110	75-125	1187	8.59	30	
Ethylbenzene	1105	6.3	30	990.1	0	112	75-125	1187	7.19	30	
m,p-Xylene	2185	40	59	1980	0	110	80-125	2409	9.77	30	
Naphthalene	1090	71	99	990.1	0	110	40-140	1130	3.63	30	
o-Xylene	1088	11	30	990.1	0	110	75-125	1181	8.2	30	
Toluene	1084	8.1	30	990.1	0	109	70-125	1148	5.75	30	
Xylenes, Total	3273	40	89	2970	0	110	75-125	3590	9.25	30	
Surr: 1,2-Dichloroethane-d4	*923.3*	0	0	*990.1*	0	*93.2*	*70-130*	990.2	*7*	*30*	
Surr: 4-Bromofluorobenzene	*958.4*	0	0	*990.1*	0	*96.8*	*70-130*	1025	*6.68*	*30*	
Surr: Dibromofluoromethane	*932.2*	0	0	*990.1*	0	*94.2*	*70-130*	997.4	*6.76*	*30*	
Surr: Toluene-d8	*945*	0	0	*990.1*	0	*95.4*	*70-130*	969.7	*2.58*	*30*	

The following samples were analyzed in this batch: 19050225-01A

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

Client: Barr Engineering Company
Work Order: 19050225
Project: ENB Tank 22 Response (49161374.07)

QC BATCH REPORT

Batch ID: **R259998** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R259998** Units: **% of sample** Analysis Date: **5/7/2019 02:25 PM**
Client ID: Run ID: **MOIST_190507B** SeqNo: **5646042** 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	0.1	0.10								

LCS Sample ID: **LCS-R259998** Units: **% of sample** Analysis Date: **5/7/2019 02:25 PM**
Client ID: Run ID: **MOIST_190507B** SeqNo: **5646040** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	99.98	0.1	0.10	100	0	100	98-102	0			

DUP Sample ID: **19050227-02B DUP** Units: **% of sample** Analysis Date: **5/7/2019 02:25 PM**
Client ID: Run ID: **MOIST_190507B** SeqNo: **5646011** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	8.77	0.1	0.10	0	0	0	0-0	8.81	0.455	10	

DUP Sample ID: **19050227-03B DUP** Units: **% of sample** Analysis Date: **5/7/2019 02:25 PM**
Client ID: Run ID: **MOIST_190507B** SeqNo: **5646017** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	8.84	0.1	0.10	0	0	0	0-0	9.01	1.9	10	

The following samples were analyzed in this batch: 19050225-01B

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

19050225

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: 57837
COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company: Same
Address: 325 S. Lake Ave Duluth MN	Address:
Name: Ryan Ericksen	Name:
email: reee@barr.com	email:
Copy to: datamgt@barr.com	P.O. —
Project Name: ENB Tank 22 respare	Barr Project No: 49161374.07 005 100

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: PVOC + Naphthalene	Soil: % Solids	Preservative Code	Field Filtered Y/N
										F	A		
										N	N		
1. TK22-S-1	2	2	ft	05/02/2019	1210	S	N	3		2	1	PVOC+Naphthalene, % moisture	
2.													
3.													
4.													
5.													
6.													
7.													
8.													
9.													
10.													

BARR USE ONLY

Sampled by: KMJ3
Barr Proj. Manager: REE
Barr DQ Manager: JET
Lab Name: A15
Lab Location: Holland, MI

Relinquished by: [signature] — On Ice? Y ☒ N ☐ — Date 5/2/19 — Time 1500 — Received by: FED EX — Date — Time
Relinquished by: FED EX — On Ice? Y ☐ N ☐ — Date 5/3/19 — Time 0900 — Received by: [signature] — Date — Time

Samples Shipped VIA: ☐ Courier ☐ Federal Express ☐ Sampler ☐ Other: _____
Air Bill Number: EB

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.

SR2 4.4°c

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: **03-May-19 09:00**

Work Order: **19050225** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 03-May-19 (Date) Reviewed by: Ehrland Bosworth (eSignature) 03-May-19 (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):	4.4/4.4 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	5/3/2019 10:24:02 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

Waste Management Documentation

Chris Guillemette
Vice President
1100 West Gary Street
Duluth, MN 55808

Office: 218.626.3830
Mobile: 612.221.0785
Fax: 218.626.4874
CGuillemette@VoncoUSA.com

March 12, 2019

Enbridge Energy
Alex Smith
2800 E 21st Street
Superior, WI 54880

RE: 19-017-I/Tank 22 (Contaminated Soil)

Alex,

Please be advised that the above described waste material is acceptable for up to **500/yards** disposal at the Vonco V Waste Management Campus Facility in Duluth, MN. The waste material is acceptable per Vonco V (SW-536) Minnesota Pollution Control Agency 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 3\7\2022.

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: 612-221-0785.

We look forward to working with you,

Vonco V, LLC
Vice President

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 - Nemadji Corridor
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	715-395-3836
Fax	

C. Description of Waste

Name of Waste: Tank 22 - Soil

Process Generating Waste: Hydrocarbon contaminated soil.

Estimated Volume: 100 CY

Frequency: One time

Physical State: Solid (soil) Color: Reddish brown Free Liquids: No

Flash Point (°F): Not applicable 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: 3/7/2019 Sample I.D.: TK22-Stockpile-1

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 Advisor

Print Name: Alex Smith Date: 3/12/2019

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 _____

12-Mar-2019

Jim Taraldsen
Barr Engineering Company
4300 Market Pointe Drive
Suite 200
Minneapolis, MN 55435

Re: **Tank 22 Response (49161092.07)** Work Order: **1903418**

Dear Jim,

ALS Environmental received 3 samples on 08-Mar-2019 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 14.

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: 12-Mar-19

Client: Barr Engineering Company
Project: Tank 22 Response (49161092.07)
Work Order: **1903418**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
1903418-01	TK22-Stockpile-1	Soil		3/7/2019 12:34	3/8/2019 10:00	☐
1903418-02	Duplicate	Soil		3/7/2019 12:34	3/8/2019 10:00	☐
1903418-03	Trip Blank	Soil		3/7/2019	3/8/2019 10:00	☐

Client: Barr Engineering Company
Project: Tank 22 Response (49161092.07)
WorkOrder: **1903418**

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

ALS Group, USA

Date: 12-Mar-19

Client: Barr Engineering Company
Project: Tank 22 Response (49161092.07)
Work Order: 1903418

Case Narrative

Samples for the above noted Work Order were received on 03/08/19. 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: *12-Mar-19*

Client: Barr Engineering Company
Project: Tank 22 Response (49161092.07)
Sample ID: TK22-Stockpile-1
Collection Date: 3/7/2019 12:34 PM

Work Order: 1903418
Lab ID: 1903418-01
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 / 3/11/19		Analyst: **RP**
DRO (C10-C28)	**3,600**		**10**	**100**	**mg/Kg-dry**	10	3/12/2019 02:05
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 3/11/19		Analyst: **WH**
Benzene	**130**		**17**	**98**	**µg/Kg-dry**	1	3/11/2019 14:37
Ethylbenzene	**380**		**21**	**98**	**µg/Kg-dry**	1	3/11/2019 14:37
m,p-Xylene	**1,700**		**47**	**200**	**µg/Kg-dry**	1	3/11/2019 14:37
o-Xylene	**710**		**38**	**98**	**µg/Kg-dry**	1	3/11/2019 14:37
Toluene	**860**		**27**	**98**	**µg/Kg-dry**	1	3/11/2019 14:37
Xylenes, Total	**2,400**		**85**	**300**	**µg/Kg-dry**	1	3/11/2019 14:37
Surr: 1,2-Dichloroethane-d4	*97.0*			*70-130*	*%REC*	1	3/11/2019 14:37
Surr: 4-Bromofluorobenzene	*96.9*			*70-130*	*%REC*	1	3/11/2019 14:37
Surr: Dibromofluoromethane	*94.2*			*70-130*	*%REC*	1	3/11/2019 14:37
Surr: Toluene-d8	*99.2*			*70-130*	*%REC*	1	3/11/2019 14:37
MOISTURE		Method: **SW3550C**					Analyst: **KTP**
Moisture	**52**		**0.10**	**0.10**	**% of sample**	1	3/8/2019 14:49

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

ALS Group, USA

Date: *12-Mar-19*

Client: Barr Engineering Company
Project: Tank 22 Response (49161092.07)
Sample ID: Duplicate
Collection Date: 3/7/2019 12:34 PM

Work Order: 1903418
Lab ID: 1903418-02
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 / 3/11/19		Analyst: **RP**
DRO (C10-C28)	**7,200**		**9.3**	**94**	**mg/Kg-dry**	10	3/12/2019 02:34
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 3/11/19		Analyst: **WH**
Benzene	**130**		**14**	**85**	**µg/Kg-dry**	1	3/11/2019 14:21
Ethylbenzene	**480**		**18**	**85**	**µg/Kg-dry**	1	3/11/2019 14:21
m,p-Xylene	**2,200**		**40**	**170**	**µg/Kg-dry**	1	3/11/2019 14:21
o-Xylene	**910**		**33**	**85**	**µg/Kg-dry**	1	3/11/2019 14:21
Toluene	**1,000**		**23**	**85**	**µg/Kg-dry**	1	3/11/2019 14:21
Xylenes, Total	**3,100**		**73**	**250**	**µg/Kg-dry**	1	3/11/2019 14:21
Surr: 1,2-Dichloroethane-d4	*96.6*			*70-130*	*%REC*	1	3/11/2019 14:21
Surr: 4-Bromofluorobenzene	*98.4*			*70-130*	*%REC*	1	3/11/2019 14:21
Surr: Dibromofluoromethane	*94.2*			*70-130*	*%REC*	1	3/11/2019 14:21
Surr: Toluene-d8	*99.1*			*70-130*	*%REC*	1	3/11/2019 14:21
MOISTURE		Method: **SW3550C**					Analyst: **KTP**
Moisture	**47**		**0.10**	**0.10**	**% of sample**	1	3/8/2019 14:49

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

ALS Group, USA

Date: *12-Mar-19*

Client: Barr Engineering Company
Project: Tank 22 Response (49161092.07)
Sample ID: Trip Blank
Collection Date: 3/7/2019

Work Order: 1903418
Lab ID: 1903418-03
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 3/11/19		Analyst: **WH**
Benzene	U		5.1	30	µg/Kg-dry	1	3/11/2019 14:06
Ethylbenzene	U		6.3	30	µg/Kg-dry	1	3/11/2019 14:06
m,p-Xylene	**27**	J	**14**	**60**	**µg/Kg-dry**	1	3/11/2019 14:06
o-Xylene	U		12	30	µg/Kg-dry	1	3/11/2019 14:06
Toluene	U		8.2	30	µg/Kg-dry	1	3/11/2019 14:06
Xylenes, Total	**27**	J	**26**	**90**	**µg/Kg-dry**	1	3/11/2019 14:06
Surr: 1,2-Dichloroethane-d4	*96.0*			*70-130*	*%REC*	1	3/11/2019 14:06
Surr: 4-Bromofluorobenzene	*99.9*			*70-130*	*%REC*	1	3/11/2019 14:06
Surr: Dibromofluoromethane	*87.5*			*70-130*	*%REC*	1	3/11/2019 14:06
Surr: Toluene-d8	*99.5*			*70-130*	*%REC*	1	3/11/2019 14:06

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

ALS Group, USA

Date: 12-Mar-19

Client: Barr Engineering Company

Work Order: 1903418

Project: Tank 22 Response (49161092.07)

QC BATCH REPORT

Batch ID: **132927** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK	Sample ID: **DBLKS1-132927-132927**				Units: **mg/Kg**			Analysis Date: **3/12/2019 01:36 AM**			
Client ID:	Run ID: **GC8_190311A**				SeqNo: **5554341**			Prep Date: **3/11/2019**		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: **DLCSS1-132927-132927**				Units: **mg/Kg**			Analysis Date: **3/12/2019 01:07 AM**			
Client ID:	Run ID: **GC8_190311A**				SeqNo: **5554340**			Prep Date: **3/11/2019**		DF: **1**	
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	7.462	0.5	5.0	10	0	74.6	70-120	0			

LCSD	Sample ID: **DLCSDS1-132927-132927**				Units: **mg/Kg**			Analysis Date: **3/12/2019 03:03 AM**			
Client ID:	Run ID: **GC8_190311A**				SeqNo: **5554344**			Prep Date: **3/11/2019**		DF: **1**	
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	8.466	0.5	5.0	10	0	84.7	70-120	7.462	12.6	20	

The following samples were analyzed in this batch: 1903418-01C 1903418-02C

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

Client: Barr Engineering Company
Work Order: 1903418
Project: Tank 22 Response (49161092.07)

QC BATCH REPORT

Batch ID: **132984** Instrument ID **VMS7** Method: **SW8260C**

MBLK Sample ID: **MBLK-132984-132984** Units: **µg/Kg-dry** Analysis Date: **3/11/2019 11:44 AM**
Client ID: Run ID: **VMS7_190311A** SeqNo: **5554631** Prep Date: **3/11/2019** 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								
Ethylbenzene	U	6.3	30								
m,p-Xylene	U	14	60								
o-Xylene	U	12	30								
Toluene	U	8.2	30								
Xylenes, Total	U	26	90								
Surr: 1,2-Dichloroethane-d4	*957.5*	0	0	*1000*	0	*95.8*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*964.5*	0	0	*1000*	0	*96.4*	*70-130*	0			
Surr: Dibromofluoromethane	*917*	0	0	*1000*	0	*91.7*	*70-130*	0			
Surr: Toluene-d8	*985*	0	0	*1000*	0	*98.5*	*70-130*	0			

LCS Sample ID: **LCS-132984-132984** Units: **µg/Kg-dry** Analysis Date: **3/11/2019 10:58 AM**
Client ID: Run ID: **VMS7_190311A** SeqNo: **5554630** Prep Date: **3/11/2019** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	930.5	5.1	30	1000	0	93	75-125	0			
Ethylbenzene	881.5	6.3	30	1000	0	88.2	75-125	0			
m,p-Xylene	1822	14	60	2000	0	91.1	80-125	0			
o-Xylene	893	12	30	1000	0	89.3	75-125	0			
Toluene	912	8.2	30	1000	0	91.2	70-125	0			
Xylenes, Total	2716	26	90	3000	0	90.5	75-125	0			
Surr: 1,2-Dichloroethane-d4	*943*	0	0	*1000*	0	*94.3*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*997*	0	0	*1000*	0	*99.7*	*70-130*	0			
Surr: Dibromofluoromethane	*980.5*	0	0	*1000*	0	*98*	*70-130*	0			
Surr: Toluene-d8	*1000*	0	0	*1000*	0	*100*	*70-130*	0			

MS Sample ID: **1903470-01A MS** Units: **µg/Kg-dry** Analysis Date: **3/11/2019 07:00 PM**
Client ID: Run ID: **VMS7_190311A** SeqNo: **5554656** Prep Date: **3/11/2019** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	2298	8.7	51	1703	160.1	126	75-125	0			S
Ethylbenzene	2109	11	51	1703	32.35	122	75-125	0			
m,p-Xylene	4384	24	100	3405	364.4	118	80-125	0			
o-Xylene	2056	20	51	1703	54.49	118	75-125	0			
Toluene	2489	14	51	1703	487.8	118	70-125	0			
Xylenes, Total	6440	44	150	5108	414	118	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1651*	0	0	*1703*	0	*97*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1710*	0	0	*1703*	0	*100*	*70-130*	0			
Surr: Dibromofluoromethane	*1641*	0	0	*1703*	0	*96.4*	*70-130*	0			
Surr: Toluene-d8	*1697*	0	0	*1703*	0	*99.6*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 1903418
Project: Tank 22 Response (49161092.07)

QC BATCH REPORT

Batch ID: **132984** Instrument ID **VMS7** Method: **SW8260C**

MSD Sample ID: **1903470-01A MSD** Units: **µg/Kg-dry** Analysis Date: **3/11/2019 07:15 PM**
Client ID: Run ID: **VMS7_190311A** SeqNo: **5554657** Prep Date: **3/11/2019** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	2332	8.7	51	1703	160.1	128	75-125	2298	1.47	30	S
Ethylbenzene	2144	11	51	1703	32.35	124	75-125	2109	1.64	30	
m,p-Xylene	4581	24	100	3405	364.4	124	80-125	4384	4.41	30	
o-Xylene	2126	20	51	1703	54.49	122	75-125	2056	3.34	30	
Toluene	2623	14	51	1703	487.8	125	70-125	2489	5.23	30	S
Xylenes, Total	6707	44	150	5108	414	123	75-125	6440	4.07	30	
Surr: 1,2-Dichloroethane-d4	*1624*	0	0	*1703*	0	*95.4*	*70-130*	1651	*1.66*	*30*	
Surr: 4-Bromofluorobenzene	*1689*	0	0	*1703*	0	*99.2*	*70-130*	1710	*1.25*	*30*	
Surr: Dibromofluoromethane	*1623*	0	0	*1703*	0	*95.3*	*70-130*	1641	*1.15*	*30*	
Surr: Toluene-d8	*1691*	0	0	*1703*	0	*99.3*	*70-130*	1697	*0.352*	*30*	

The following samples were analyzed in this batch: 1903418-01A 1903418-02A 1903418-03A

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

Client: Barr Engineering Company
Work Order: 1903418
Project: Tank 22 Response (49161092.07)

QC BATCH REPORT

Batch ID: **R256220** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R256220** Units: **% of sample** Analysis Date: **3/8/2019 02:49 PM**
Client ID: Run ID: **MOIST_190308B** SeqNo: **5552933** 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	0.1	0.10								

LCS Sample ID: **LCS-R256220** Units: **% of sample** Analysis Date: **3/8/2019 02:49 PM**
Client ID: Run ID: **MOIST_190308B** SeqNo: **5552931** 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	0.1	0.10	100	0	100	98-102	0			

DUP Sample ID: **1903414-01B DUP** Units: **% of sample** Analysis Date: **3/8/2019 02:49 PM**
Client ID: Run ID: **MOIST_190308B** SeqNo: **5552922** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	21.44	0.1	0.10	0	0	0	0-0	20.88	2.65	10	

DUP Sample ID: **1903418-02B DUP** Units: **% of sample** Analysis Date: **3/8/2019 02:49 PM**
Client ID: **Duplicate** Run ID: **MOIST_190308B** SeqNo: **5552930** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	45.04	0.1	0.10	0	0	0	0-0	46.79	3.81	10	

The following samples were analyzed in this batch: 1903418-01B 1903418-02B

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

1903418

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: 58924
COC 1 of ____

REPORT TO	INVOICE TO
Company: Barr	Company:
Address: 325 Blake Ave	Address:
Name: Ryan Erickson	Name:
email: RErickson@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: Tank 22 response	Barr Project No: 4916/092.07 003 006

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: DRO	Soil: BTEX	Soil: % Solids
Preservative Code										A	A	
Field Filtered Y/N										N	N	
1. TK22-Stockpile-1	-	-	-	03/07/2019	12:34	S		3		1	2	
2. Duplicate	-		-	-	-	-						
3.												
4.												
5.												
6.												
7.												
8.												
9.												
10.												

BARR USE ONLY

Sampled by: Matthew Harris
Barr Proj. Manager: Ryan Erickson
Barr DQ Manager: James Get
Lab Name: ALS Environment
Lab Location: Holland, MI

Relinquished by: Matthew Harris — On Ice? (Y) N — Date 03/07/19 — Time 16:00 — Received by: FED EX — Date — Time
Relinquished by: FED EX — On Ice? Y N — Date 03/8/19 — Time 1000 — Received by: — Date — Time

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 ____ (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: **08-Mar-19 10:00**

Work Order: **1903418** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 08-Mar-19 (Date) Reviewed by: Ehrland Bosworth (eSignature) 08-Mar-19 (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):	4.2/4.2 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	3/8/2019 1:53:02 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:

Vonco V Waste Management Campus
1100 West Gary Street
Duluth, MN 55808
Permit: SW 536

19-017-I Nemadji Corridor					
Date	**Ticket**	**Customer**	**Truck**	**Material**	**Tons**
03/29/2019	307839	001342 - Enbridge Pipelines LLC	T87447X	Contaminated Soil Tons	15.92
03/29/2019	307840	001342 - Enbridge Pipelines LLC	T87447X	Contaminated Soil Tons	14.74
03/29/2019	307841	001342 - Enbridge Pipelines LLC	S39858W	Contaminated Soil Tons	15.82
				Total Tons	**46.48**
				Total Loads	**3**