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BARR

Technical Memorandum

To: Alex Smith, Enbridge Energy
From: Ryan Erickson and Kaitlin Johnson
Subject: 2019 Tank 24 Header Line Release Response
WI Spill #: 13795 ID 20190413NO16-1
Date: July 9, 2019
Project: 49161374.07 006

This memorandum summarizes the environmental response and assessment activities performed by Enbridge Energy (Enbridge) and Barr Engineering (Barr) following a crude oil release from the Tank 24 header line at Enbridge Superior Terminal (Terminal) in Superior, Wisconsin (Figure 1).

Background and Initial Response Actions

On April 3, 2019, Enbridge advanced line locate pothole excavations (potholes) to the southwest of Tank 9 that were associated with an infrastructure project to the west of the western Tank 9 containment basin corner (Figure 2). On April 4, Enbridge discovered free-product in one of the potholes next to a Tank 24 header line valve (Photos 1 and 2). Enbridge Environment and Pipe Line Maintenance (PLM) personnel were notified of the discovery.

PLM immediately responded to the site to determine whether the product was associated with a new or historical release. Given the depth of the buried infrastructure in that location and the potholes proximity to historical releases, it was not immediately clear whether the product was new or historical. Activities that were completed to determine this included:

- A product sample was collected by Barr and a fingerprint laboratory analysis was completed to determine if the product was weathered (i.e., historical release source) or not (i.e., new release). The lab results indicated that the product was not weathered.
- Product was recovered from the pothole by the PLM with a vacuum truck to determine if new product would return to the pothole. Product returned to the pothole.
- Three additional potholes were advanced around the initial pothole with product to delineate the extent of the impacts. Evidence of hydrocarbon impacts were identified in the other potholes.
- Soil around the buried pipeline infrastructure in the vicinity of the valve and impacted potholes was excavated and the pipelines were inspected.

After excavating the pipeline infrastructure, the PLM identified the crude oil source as a pinhole release from a Line 24 Header Line approximately 20 feet north of the initial pothole and 12 feet below ground surface (bgs). The PLM reported that approximately 84 gallons of crude oil had been released. Excavated soil with hydrocarbon impacts was stockpiled in the Terminal Soil Management Area (SMA) until offsite management was approved. Free-product was recovered with a vacuum truck and oil absorbent pads. Impacted water with a sheen removed from the excavation to facilitate infrastructure maintenance and remediation was containerized in a frac tank until offsite management was approved. Note that hydrocarbon impacts were not observed in the infrastructure project excavation to the north of the remedial excavation (Figure 2).

Enbridge Environment requested that Barr complete the following activities during and after release response and remedial excavation activity:

- document remediation activities and environmental conditions at the time of those activities,
- investigate whether the free-product was from a historical or new release by collecting fingerprint analysis samples and by completing a historical release file review,
- assist with the characterization and offsite management coordination of hydrocarbon-impacted soil and water,
- 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 header line release on April 13, 2019 and spill number #13795 ID 20190413NO16-1 was assigned to the site. The associated WDNR communication is provided in Attachment A.

A historical release file review was completed and multiple crude oil releases were identified (Figure 2). The releases that were considered potential contamination sources during the initial site evaluation included the *Tank 9 Pressure Line* release (Remediation and Redevelopment Tracking System (BRRTS) #0216552700; 2008 115-barrel (bbl) release) located approximately 90 feet to the northeast and the *Nemadji River* release (BRRTS# 0216513788; 2003 4,500-bbl release) that migrated through a ditch 40 feet to the west of the site.

Field Activities

Barr was on site April 4, 5, and 15, 2019 to observe site conditions and remedial actions and collect fingerprint and waste characterization analytical samples (Photos 1-4). Barr was on site May 31, 2019 to document conditions in the final remedial excavation through field observation and soil screening (Photos 5-7; Attachment B).

On April 4, Barr collected free-product sample *SUPTK9-Product-1* from the pothole 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 ATS laboratory report and an initial results-interpretation communication (email dated April 9, 2019) are provided in Attachment C.

On April 5, Barr collected waste characterization samples *TK9-Stockpile-1* and *TK9-Stockpile-2* from the impacted soil stockpile at the SMA for analysis of Benzene, Toluene, Ethyl Benzene, and Xylene (BTEX), toxicity characteristic leaching procedure (TCLP) 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.

On May 31, Barr 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 *TK24HL-B-1* was collected from the excavation bottom. 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. Barr also collected an impacted water sample *TK24HL-Frac-1* from the Frac tank staged south of the remedial excavation (Photo 7). The sample was submitted to ALS for analysis of DRO and Volatile Organic Compounds (VOCs). The laboratory report is provided in Attachment D.

Results

ATS laboratory analysts reviewed the analytical results from the free-product fingerprint sample *SUPTK9-Product-1* and their analysts indicated that the sample results exhibited "a slight to moderate degree of weathering" (Attachment C- email dated April 9, 2019).

The Tank 24 Header excavation was approximately 60 feet long (SW to NE) by 45 feet wide (SE to NW) and up to 16 feet deep (Photos 3, 4, 5; Figure 2; Attachment B). Reddish brown clay was observed in the excavation extents and stockpiles. Groundwater was observed approximately 10 feet below the road grade (6 feet below the bottom of the stormwater ditch).

Barr field screened eight soil samples from the final excavation sidewalls. Soil from 0 to 2 feet bgs had headspace readings between 0.2 and 1.5 parts per million (ppm) and no other evidence of hydrocarbon impacts was identified in the sidewalls. Soil from the excavation bottom (14-16 feet bgs; *B-1* through *B-7*) had headspace readings between 0.9 ppm and 71.9 ppm and some of the samples had a light to moderate hydrocarbon odor (Photos 5 and 6; Attachment B).

Analytical soil confirmation sample *TK24HL-B-1* was collected from the bottom excavation at 15 feet bgs to document conditions beneath the pipeline infrastructure where headspace detections exceeded 10 ppm (Photo 6; Figure 2; Attachment B). Analyte concentrations were below WDNR Industrial Direct Contact Residual Contaminant Levels (RCLs) and WDNR Groundwater RCLs with the exception of benzene (0.26 mg/kg) which exceeded the Groundwater RCL (0.0026 mg/kg). The laboratory results are summarized in Table 1 and the ALS laboratory report is provided in Attachment C.

Material Management

Soil Management

On April 5, Barr collected analytical waste characterization soil samples *TK9-Stockpile-1* and *TK9-Stockpile-2* from the contaminated soil stockpile at the SMA for laboratory analysis at ALS Laboratory (Photo 8). The sample was analyzed for DRO, BTEX, and TCLP 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 #19-037-I. A total of 740 tons of contaminated soil were hauled to the landfill from April 11 through June 17, 2019. The waste profile documents, the waste characterization laboratory report, and the landfill summary report are included in Attachment D.

Impacted Water Management

Groundwater with identified hydrocarbon impacts removed from the project excavation was containerized in one 21,000 gallon frac tank (Photo 7). Water from the frac tank was approved for disposal at the WLSSD Facility in Duluth, Minnesota on June 18, 2019. Water transport and disposal were still pending as of July 9, 2019. Available material management documentation is provided in Attachment D.

Receptor Survey

No direct contact risks were identified based on field observations and analytical sampling results. No impacts to surface water were identified and there is little risk of future surface water impacts based on the remedial actions that were completed. The groundwater pathway at the Superior Terminal is 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 and will conduct its next sampling event in the fall of 2019. The nearest downgradient monitoring well is MW-20 located 750 feet to the south of the release site (Figure 3). The nearest structures are slab-on-grade terminal buildings approximately 350 feet north of the site. The risk of hazardous vapor accumulation in those structures is low because they are above ground buildings with minimal human occupancy. Onsite employees are also required to wear four-gas detectors that would alert them to a potentially hazardous atmosphere.

Discussion and Conclusions

The 2019 Tank 24 Header release (WI SPILL #13795 ID 20190413NO16-1) was reported to the WDNR based on the estimated release volume (84 gallons). Based on field observations, the impacted soil associated with the header release was adequately addressed; however, residual soil impacts exceeding WDNR groundwater criteria was identified in the final excavation. There are no identified remaining risks to direct contact, surface water, or vapor intrusion and groundwater is monitored through the existing facility-wide groundwater monitoring program.

Based on this information, 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 8
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	Material Management Documentation

Site Photos

Photo 1

Photo 2

Photo 1: Line locate pothole excavation area the southwest of the Tank 9 containment basin berm. The pothole excavation is located just beyond the plywood in the center of the photo and a blue PVC pipe is sticking out of it. The three orange paint circles (one near the lower right of photo and two left of center) mark the locations of the planned contaminant extent delineation potholes. Photo taken facing north on April 4, 2019.
Photo 2: Pothole excavation containing free-product. Photo taken on April 4, 2019.

Photo 3

Photo 4

Photo 3: The Tank 24 Header Line excavation area. Tank 9 is visible in the upper right corner of the photo. Photo taken facing northeast on April 15, 2019.
Photo 4: The Tank 24 Header Line excavation with stained soil and hydrocarbon impacted water visible in the bottom right corner of the photo. Photo taken facing north on April 15, 2019.

Photo 5

Photo 6

Photo 5: Tank 24 Header Line remedial excavation. Photo taken facing northwest on May 31, 2019.
Photo 6: Bottom of the Tank 24 Header Line remedial excavation (16 feet bgs) near the release location where soil with residual hydrocarbon impacts was identified. Photo taken on May 31, 2019.

Photo 7

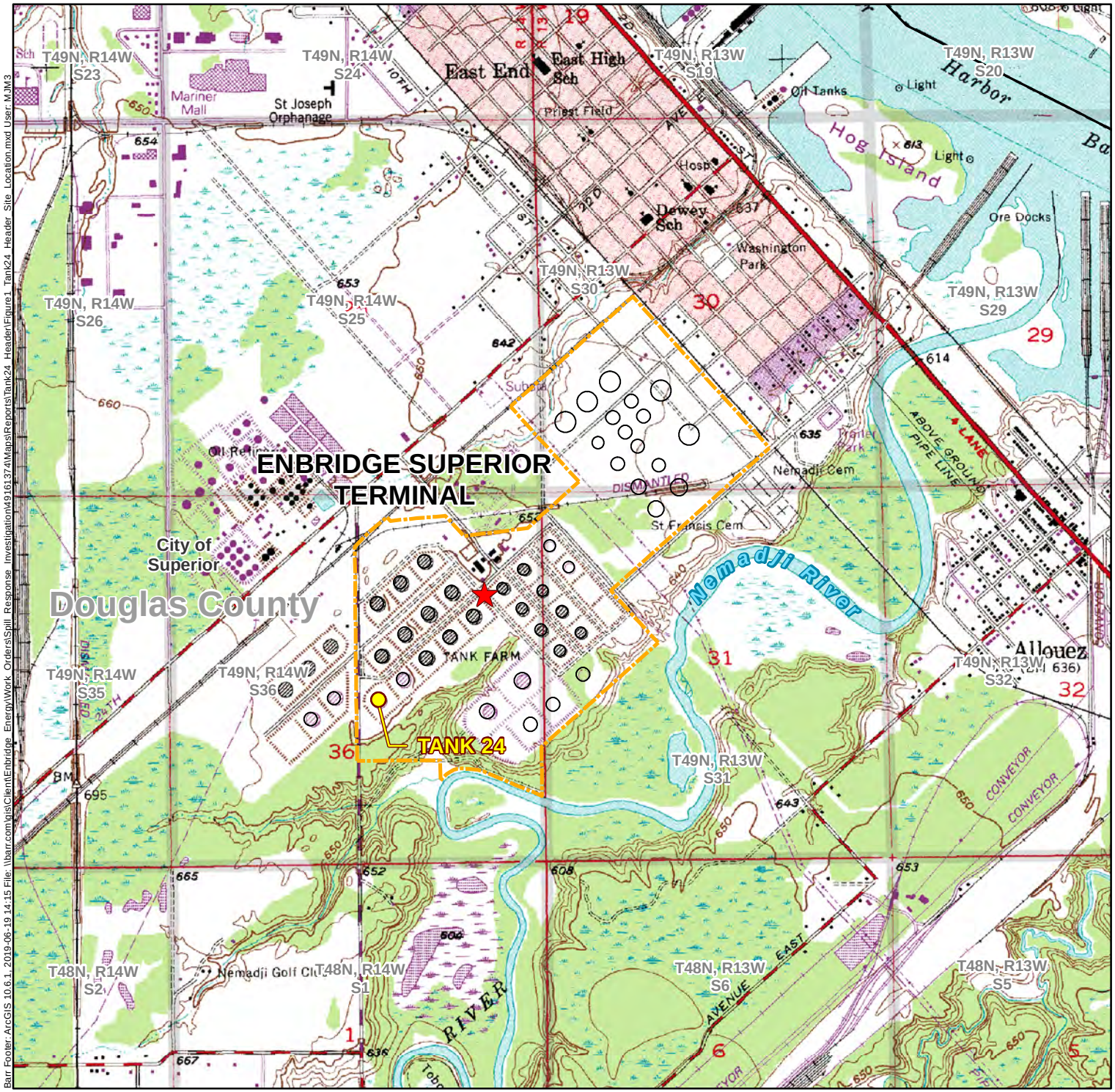
Photo 8

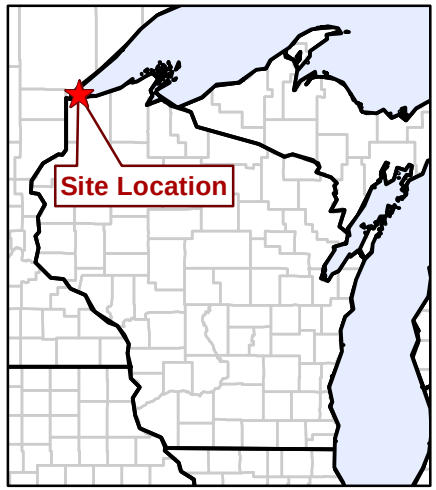
Photo 7: Frac tank staged south of remedial excavation that was used to containerize hydrocarbon impacted water. Photo taken facing southeast on May 31, 2019.
Photo 8: Hydrocarbon impacted soil stockpile in the Terminal SMA building. Photo taken on April 5, 2019.

Table 1
Soil Analytical Data Summary
2019 Tank 24 Header Line Release Response
Enbridge Energy Superior Terminal
Superior, WI

		Location	**TK 24 HL-B-1**
		Date	**5/31/2019**
Parameter	Wisconsin Groundwater RCLs, DF=1	Wisconsin Not to Exceed Direct Contact Industrial RCLs	
Effective Date	06/01/2018	06/01/2018	
Exceedance Key	**Bold**	No Exceedances	
General Parameters			
% Moisture			25
Volatile Organic Compounds			
1,2,4-Trimethylbenzene	0.6894 (1)	219	< 0.033
1,3,5-Trimethylbenzene	0.6894 (1)	182	< 0.053
Benzene	**0.0026**	7.07	**0.26**
Ethyl benzene	0.785	35.4	< 0.0096
Naphthalene	0.3291	24.1	< 0.11
Toluene	0.5536	818	0.17
Xylene, m & p	1.98 XYL	260 XYL	< 0.061
Xylene, o	1.98 XYL	434	< 0.018
Xylene, total	1.98	260	< 0.061

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





Site Location

Tank 24

Terminal Property Boundary

N

0 2,000 4,000

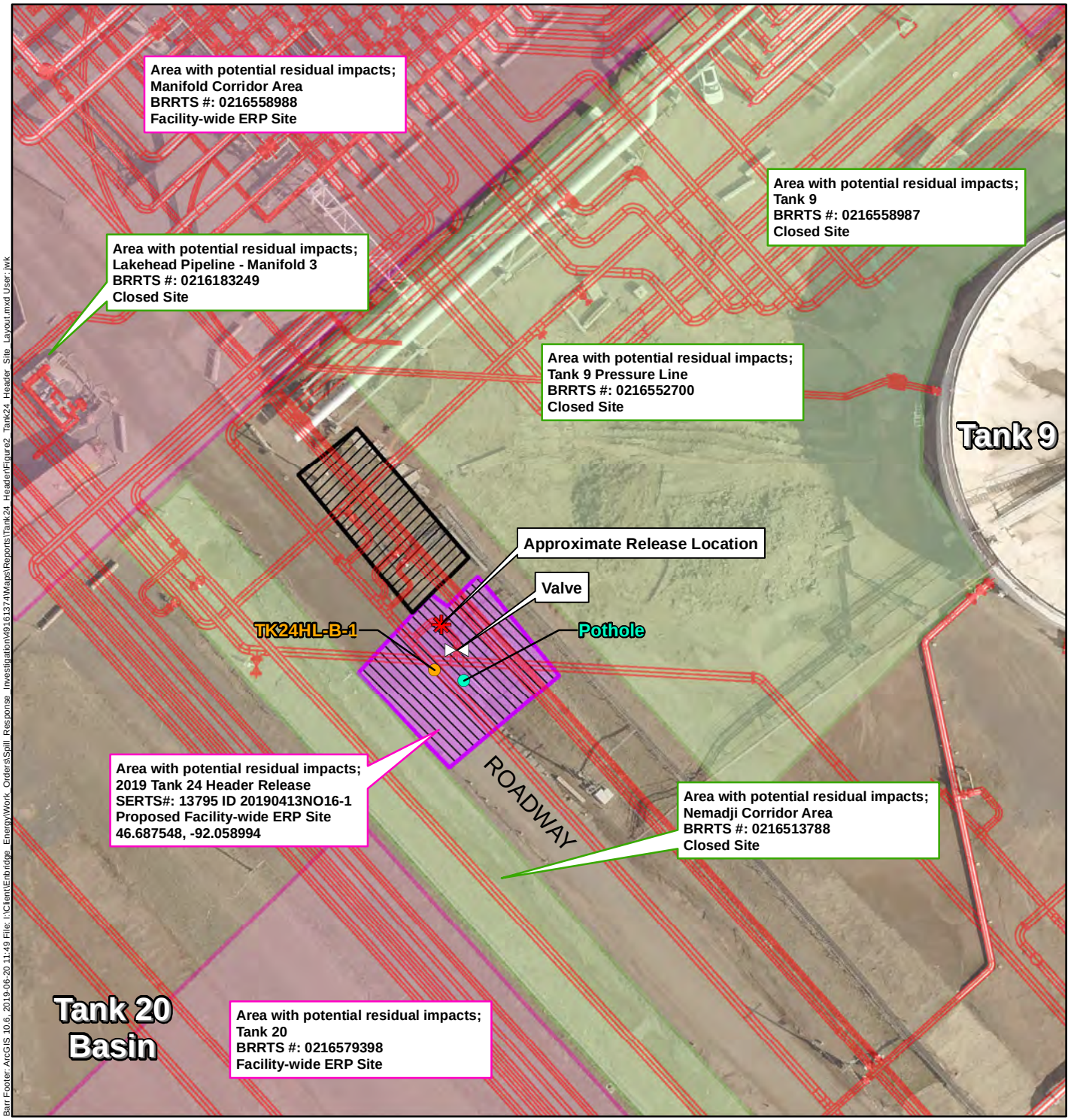
Feet

1 Inch = 2,000 Feet

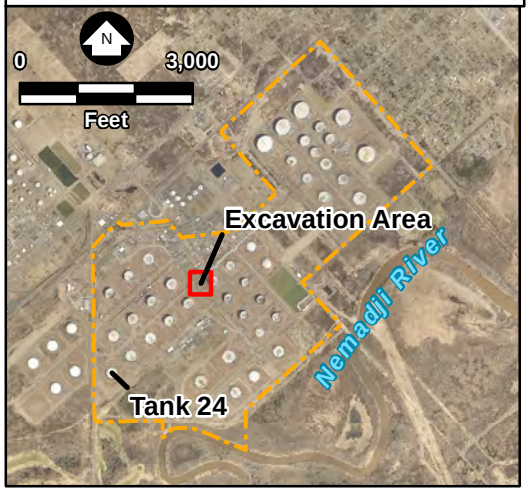
Figure 1

SITE LOCATION
TANK 24 HEADER
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE



ENBRIDGE SUPERIOR TERMINAL



- Approximate Release Location
- Analytical Sample Locations
- Pothole
- Infrastructure Project Excavation Area
- Remedial Excavation Area
- Area with Potential Residual Impacts
- Estimated extent of potential residual impacts - Facility-wide BRRTS sites
- Estimated extent of residual impacts - Closed BRRTS sites
- Valve
- Pipeline Infrastructure
- Terminal Property Boundary

N

0 50 100

Feet

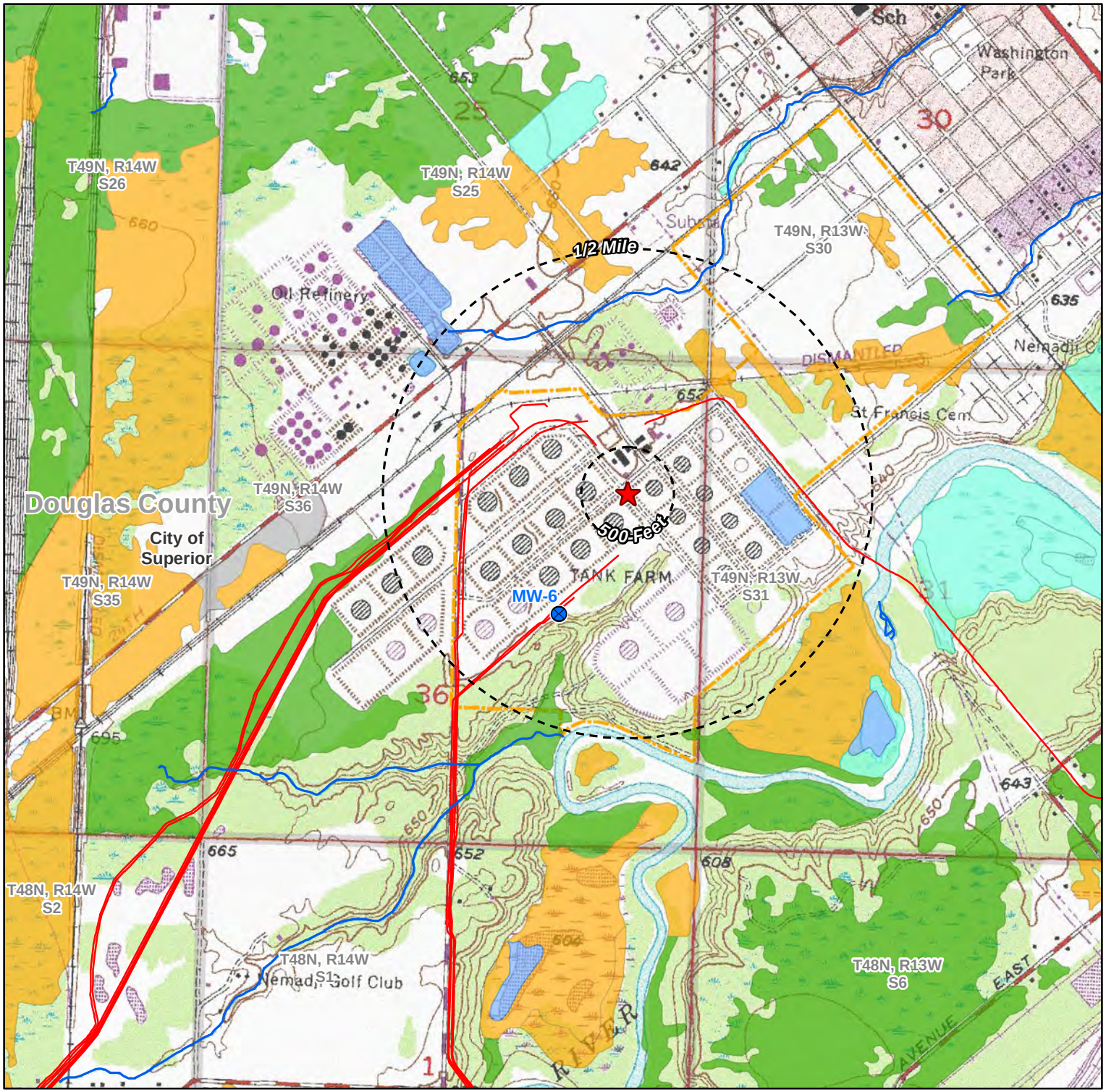
1 Inch = 50 Feet

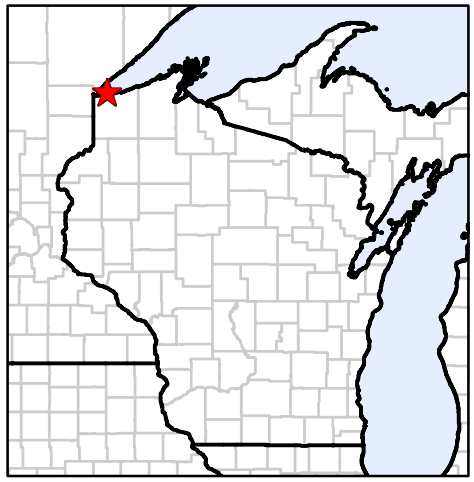
Douglas County Imagery Circa May, 2016

Figure 2

SITE LAYOUT
TANK 24 HEADER
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 24 HEADER
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE

Barr Footer: ArcGIS 10.6.1, 2019-06-19 14:13 File: \\barr.com\gis\Client\Enbridge_Energy\Work_Orders\Spill_Response_Investigation\49161374\Maps\Reports\Tank24_Header\Figure3_Tank24_Header_Release_Receptor.mxd User: MJM3

Attachment A

WDNR Release Reporting Communication

Kaitlin M. Johnson

From: nicholas.ramos@wisconsin.gov
Sent: Saturday, April 13, 2019 1:32 PM
To: Alex Smith
Subject: [External] WI SPILL #13795 ID 20190413NO16-1 - CRUDE OIL [CRUDE OIL]

SERTS ID:
20190413NO16-1

Reported:
04/13/2019 13:25

Occurred:
04/13/2019 12:30

Reported by:
ALEX SMITH
ENVIRONMENTAL ADVISOR
ENBRIDGE ENERGY
alex.smith@enbridge.com
(715) 817-8322

Location:
NO REGION
DOUGLAS COUNTY
SUPERIOR, CITY OF
ENBRIDGE TERMINAL
2800 21ST ST E

Responsible Party:
ENBRIDGE

Substance:
CRUDE OIL [CRUDE OIL]
Released Amt: 84 Gal
Recovered Amt: 84 Gal
(Amounts are often estimated)

Cause:
EQUIPMENT FAILURE

Cause Description:
LEAK IN PIPE.

Environmental Impact:
SPILLED ONTO SOIL AND GROUNDWATER.

Cleanup:
OIL VACUUMED UP AND AFFECTED SOIL EXCAVATED.

Notified OCSC JASON LOWERY at 13:29 by Email

Submitted by:
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Attachment B

Site Investigation Field Sampling and Screening Logs

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Location: *Milepost or Facility* Superior Terminal Tank 24 Header Line

Equipment used: Photo -ionization detector with 10.6 eV lamp

Background Headspace: 0.0 ppm

Date: 5/31/19

Sampler: PLL

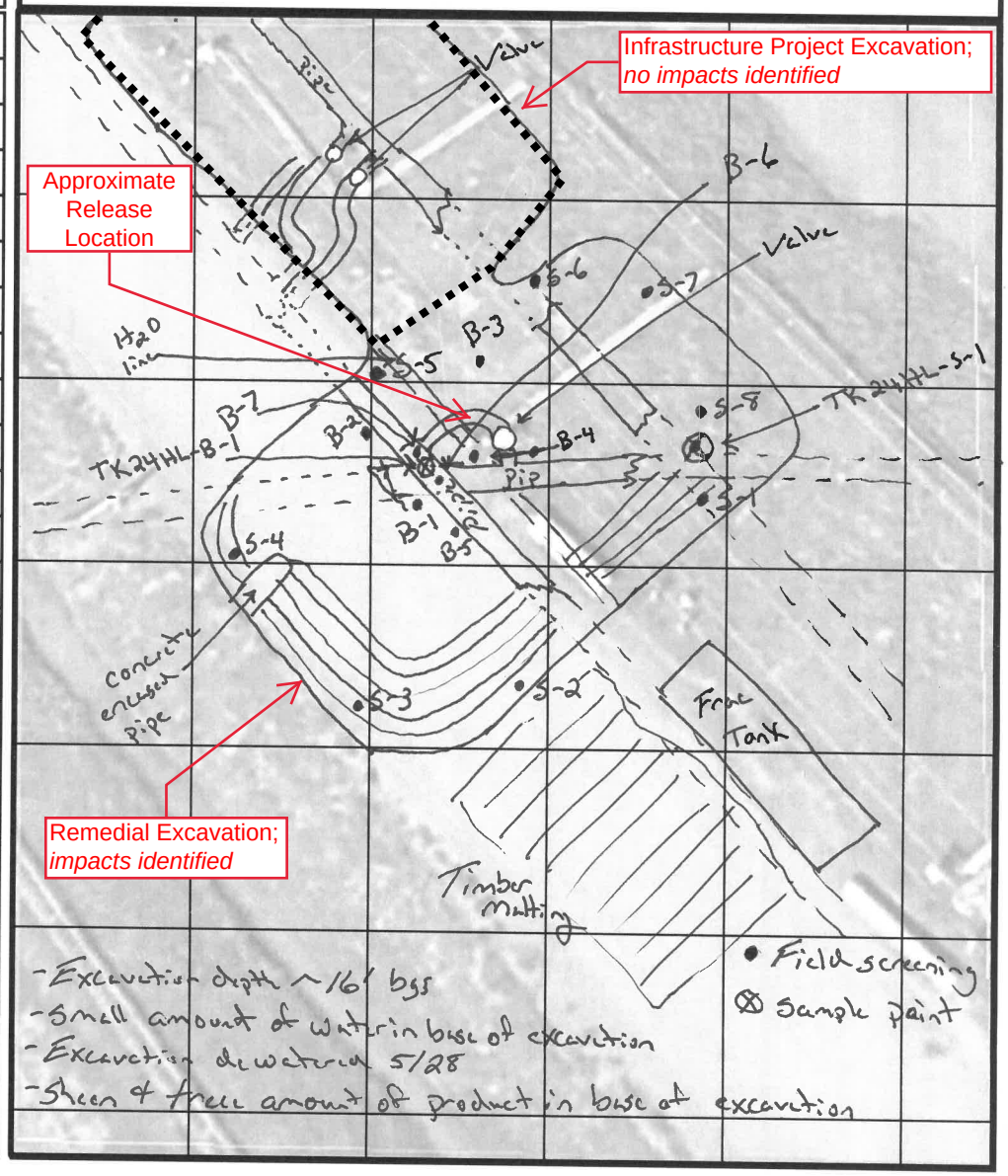
Calibration Time: 06:45

Sample Nomenclature *(Location - sample type - #)*: TK24HL-

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
S-1	0-2'	10:30	CH	Reddish brown	None/None	0.5
S-2				Reddish brown/ dark brown		1.5
S-3				Reddish brown		0.2
S-4			CH 80% SW 20%			0.4
S-5			CH 90% SW 10%			0.4
S-6			CH		Earthy/None	0.3
S-7			CH 90% organic 10%			0.3
S-8			CH 90% SW 20%			0.2
B-1	14-16'	10:40	CH	Reddish brown	None/None	1.9
B-2					Slight petro/None	13.2
B-3					None/None	1.4
B-4					Med petro/None	71.9
B-5		11:10			None/None	0.9
B-6					None/None	8.2
B-7					Slight petro/None	21.4
TK24HL-Free		11:45				
TK24HL-B-1 @ 15' bgs		11:35				
TK24HL-S-1 @ 2' bgs		11:30				

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



Attachment C

ATS and ALS Laboratory Reports

DATA PACKAGE - LEVEL II

ATS Project
BENB.T02

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

Sample Delivery Groups (SDGs):
0405191

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

Issued: April 10, 2019

Updated: April 15, 2019

LABORATORY OPERATIONS
SAMPLE DELIVERY GROUP (SDG) CASE NARRATIVE

ATS Project Number: BENB-T02
Report Date: 4/10/19 – Revised 4/5/19
SDG / SRF Number: 0405191-B

Case Narrative Summary

This case narrative applies to the following sample that was received by Ann Arbor Technical Services, Inc. (ATS) on 4/5/19, and associated matrix-specific QA/QC:

Samples

Client Sample Identification	Laboratory Sample ID	Requested Turn Around Time	Matrix
SUP TK9-PRODUCT-1 4/4/19	0405191-1	Rush	Product

Matrix Specific QC

Client Sample Identification	Laboratory Sample ID	Matrix
SUP TK9-PRODUCT-1 4/4/19 Matrix Spike	0405191-1 MS	Product
SUP TK9-PRODUCT-1 4/4/19 Matrix Spike Duplicate	0405191-1 MSD	Product

Upon receipt, samples were scheduled for the following analyses:

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

Subsequent analysis included:

- Alkylated PAH's Analysis by modified USEPA method 8270-MOD
- Biomarker Analysis by modified USEPA method 8270-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.

_____/ April 15, 2019

Mark T. DeLong (Quality Assurance Coordinator)

_____/ April 15, 2019

Philip B. Simon (Laboratory Director)

CHAIN OF CUSTODY RECORDS

and

SAMPLE RECEIPT DOCUMENTATION

ATS Project Number: BENB-T02

ATS SDG(s): 0405191

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

ATS ANN ARBOR TECHNICAL SERVICES, INC.
200 South Wagner Road
Ann Arbor, Michigan 48103
Tel. 734/995-0995 Fax. 734/995-3731
Michigan Laboratory ID: 9004
Wisconsin Laboratory ID: 998321720

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

Project Identification And General Sample Information

ATS Project Number: B2NB-702
Date: 4/5/19
Time: 1000
SRF Number: 0405191
ATS Quote / Contract Number if applicable:
Client PO Number:
Turn Around *(choose one)*: Standard | Rush | ___ Days | Other (explain): | On Hold
Analyst: DMS

Number of Sample Locations: 1
Date Range: 4/4/19

Matrix *(choose those that apply)*: Water | Soil | Air | Industrial Product | Extract | Drinking Water | Sediment | Other (explain): O.L = CRUDE ?

Choose One: Weekly | Monthly | Quarterly | Semi-Annual | Annual | N/A

Sample Delivery Information

Delivered by *(choose one)*:

ATS — Name:
Commercial Carrier — Name: Fedex
Client Representative — Name:
Firm:
Address:
Telephone:
Email:

Chain of Custody Information

ATS COC *(choose one)*:	Yes	No		COC Matches Sample Labels: *Choose One:*	Yes	No
Completed By *(choose one)*:	ATS	Client		Anomaly Form Filled Out: *Choose One:*	Yes	No
Client COC *(choose one)*:	Yes	No		COC Matches Sample Labels: *Choose One:*	Yes	No
Completed By *(choose one)*:	ATS	Client		Anomaly Form Filled Out: *Choose One:*	Yes	No
Internal COC Completed *(choose one)*:	Yes	No	N/A	Listed On COC		
Complete By *(choose one)*:	ATS	Name:				
Custody Seals *(choose one)*:	None	Present / Intact	Present / Not Intact	Seal Number:		

Sample Receipt Information

Packaging *(choose those that apply)*: Cooler (s) | Cardboard | Hazardous | Other (explain):

Thermal Preservation:				Temperature Upon Receipt ("C or "On Ice"):	Container #1	Container #2	Container #3	Container #4	Container #5
At Time of Collection By:	ATS	Client	N/A		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	Anomaly Form Filled Out: *Choose One:*	Yes / No
Limited Volume *(choose one)*:	Yes	No				

Storage Location, Retention, And Disposition Information:

Ambient: 110
Cold Room:
Frozen:
VOC:
Sample Retention *(choose those that apply)*: 6 months | 1 year | Other (explain): VIA client
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

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: **57770**

COC 1 of 1

REPORT TO		INVOICE TO	
Company:	Barr	Company:	~~BARR~~ Enbridge
Address:		Address:	
Name:		Name:	
email:	JET@BARR.COM	email:	Alex.Smith@enbridge.com
Copy to:	datamgt@barr.com	P.O.	
Project Name:	Superior Tank 9 Valve	Barr Project No:	49161092.07 003 006

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

Analysis Requested: Water | Soil | % Solids

Location	Sample Depth Start	Sample Depth 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
1. SUP TK9-PRODUCT-1	—	—	—	4/4/2019	1330	O product		
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								

Preservative Code
Field Filtered Y/N

Fingerprint Analysis
Hydrocarbon Analysis
US EPA Method (8015-MOD)

ASAP TAT

ATS LAB ID 0405191-1

Pace Analytical Services, Inc.
315 Chestnut Street
PO Box 1212
Virginia, MN 55792
(218) 735-6700

Pace Analytical
www.pacelabs.com

Client:
Client Sample ID: SUP TK9-PRODUCT-1
Date Collected: 4/4/2019 Time: 1330
Collected by: REE
Analysis: [illegible]

BARR USE ONLY	
Sampled by:	REE (Ryan)
Barr Proj. Manager:	REE
Barr DQ Manager:	JET (Jim)
Lab Name:	ATS
Lab Location:	Ann Arbor

Relinquished by:	On Ice?	Date	Time	Received by:	Date	Time
[signature]	Y	4/4/19	16:30	[signature]	4/4/19	16:45
				[signature]	4/5/19	1000

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

Air Bill Number:

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

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

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

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

FedEx Express Package US Airbill

FedEx Tracking Number 8139 1094 8213

Form ID No. 0215

Recipient's Copy

1 From

Date: 4/4/19

Sender's Name: Jim Taraldsen Phone: 218 529-8200

Company: BARR ENGINEERING

Address: 325 S LAKE AVE STE 700 Dept./Floor/Suite/Room

City: DULUTH State: MN ZIP: 55802-2323

2 Your Internal Billing Reference

49161092.07 003 006

3 To

Recipient's Name: Peter Simon Phone: 734 368 4771

Company: ATS

Address: 790 South Wagner Road Dept./Floor/Suite/Room

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

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

Address:

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

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

City: Ann Arbor State: MI ZIP: 48103

0132158893

8139 1094 8213

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

☐ Cargo Aircraft Only

Restrictions apply for dangerous goods — see the current FedEx Service Guide.

7 Payment *Bill to:*

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

☒ Sender — Acct. No. in Section 1 will be billed. ☐ Recipient ☐ Third Party ☐ Credit Card ☐ Cash/Check ☐ Obtain recip. Acct. No.

Total Packages: 1 Total Weight: 5 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

fedex.com 1.800.GoFedEx 1.800.463.3339

ORGANIC ANALYSIS

TOTAL AND SATURATED HYDROCARBON ANALYSIS
USEPA METHOD SW8015-M

ATS Project Number: BENB-T02

ATS SDG: 0405191-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-T02
ATS SDG: 0405191-B
Analysis Method: 8015-MOD

Sample Preparation

The sample was received in two 40mL glass containers 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:

- **None**

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
0405191-1 MS	n-Pentatriacontane (C35)	133	70-130%
0405191-1 MS	n-Tetradecane (C14)	68.4	70-130%
0405191-1 MSD	n-Pentatriacontane (C35)	140	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:

- **None**

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**

_____ / April 10, 2019

Mark T. DeLong (Quality Assurance Coordinator)

_____ / April 10, 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-T02	0405191-B	SW8015M	SUP TK9-PRODUCT-1	0405191-1

Total and Saturated Hydrocarbon Analysis
Data Summary Sheet

ATS Project Number	BENB-T02	**Percent Moisture**	NA
ATS SDG Number	0405191-B	**Preparation Date**	04/08/2019
Client Sample ID	SUP TK9-PRODUCT-1	**Analysis Date:**	04/08/2019
Laboratory Sample ID	0405191-1	**Instrument**	3800 FID
Matrix	Product	**Subsample (g)**	0.243
Sample Date	04/04/2019	**Final Volume (mL)**	20
Analytical Method (USEPA)	SW8015M	**Dilution Factor**	1
Preparation Method (USEPA)	USEPA Method 3580A	**Basis**	WET
QC Batch Number	QCORG0408191-B	**Units**	mg/kg

Parameter	Chemical Identifier	Result	MDL	PQL	Qual
Hydrocarbons					
n-Nonane (C9)	111-84-2	3040	11.7	39.1	
n-Decane (C10)	124-18-5	1340	4.9	16.3	
n-Undecane (C11)	1120-21-4	1120	4.5	15.1	
n-Dodecane (C12)	112-40-3	589	9.9	32.9	
n-Tridecane (C13)	629-50-5	501	6.9	23.1	
2,6,10 Trimethyldodecane (1380)	3891-98-3	50.4	13.6	45.4	
n-Tetradecane (C14)	629-59-4	608	13.6	45.4	
2,6,10 Trimethyltridecane (1470)	3891-99-4	454	10.9	36.3	
n-Pentadecane (C15)	629-62-9	399	10.9	36.3	
n-Hexadecane (C16)	544-76-3	315	10.6	35.2	
Norpristane (1650)	3892-00-0	282	12.1	40.3	
n-Heptadecane (C17)	629-78-7	216	12.1	40.3	
Pristane	1921-70-6	172	14.4	48.1	
n-Octadecane (C18)	593-45-3	227	7.3	24.5	
Phytane	638-36-8	171	11	36.7	
n-Nonadecane (C19)	629-92-5	246	8.6	28.5	
n-Eicosane (C20)	112-95-8	411	10.9	36.3	
n-Heneicosane (C21)	629-94-7	326	10.8	35.9	
n-Docosane (C22)	629-97-0	409	15.5	51.8	
n-Tricosane (C23)	638-67-5	257	13.2	43.8	
n-Tetracosane (C24)	646-31-1	164	13.8	46	
n-Pentacosane (C25)	629-99-2	445	11.4	38	
n-Hexacosane (C26)	630-01-3	221	9	30	
n-Heptacosane (C27)	593-49-7	214	10.1	33.5	
n-Octacosane (C28)	630-02-4	296	12.2	40.7	
n-Nonacosane (C29)	630-03-5	99.4	12.4	41.4	
n-Triacontane (C30)	638-68-6	136	14	46.6	
n-Hentriacontane (C31)	630-04-6	58.8	15.2	50.7	
n-Dotriacontane (C32)	544-85-4	133	14.9	49.5	
n-Tritriacontane (C33)	630-05-7	191	13.6	45.5	
n-Tetratriacontane (C34)	14167-59-0	221	16.1	53.7	
n-Pentatriacontane (C35)	630-07-9	106	16.7	55.8	
n-Hexatriacontane (C36)	630-06-8	47.2	20.4	67.8	
n-Heptatriacontane (C37)	7194-84-5	70.6	16.2	54.1	
n-Octatriacontane (C38)	7194-85-6	35.5	23	76.7	
n-Nonatriacontane (C39)	7194-86-7	30.8	21.5	71.5	
n-Tetracontane (C40)	4181-95-7	45.7	21.5	71.8	
Total Saturated Hydrocarbons (C9-C44)	TSHC9-C44	13700	1100	3600	
Total Resolvable Hydrocarbons (C9-C44)	TOTRESHC	38100	1100	3600	
Total Petroleum Hydrocarbons (C9-C44)	C9-C44	444000	1100	3600	
Residue Upon Evaporation	RUE	829000	-	-	
n-Heptadecane / Pristane Ratio	-	1.25	-	-	
n-Octadecane / Phytane Ratio	-	1.32	-	-	

Surrogate Recoveries			LCL	UCL
2-Fluorobiphenyl	321-60-8	69.6	30	150
Pyrene-D10	1718-52-1	118	50	150

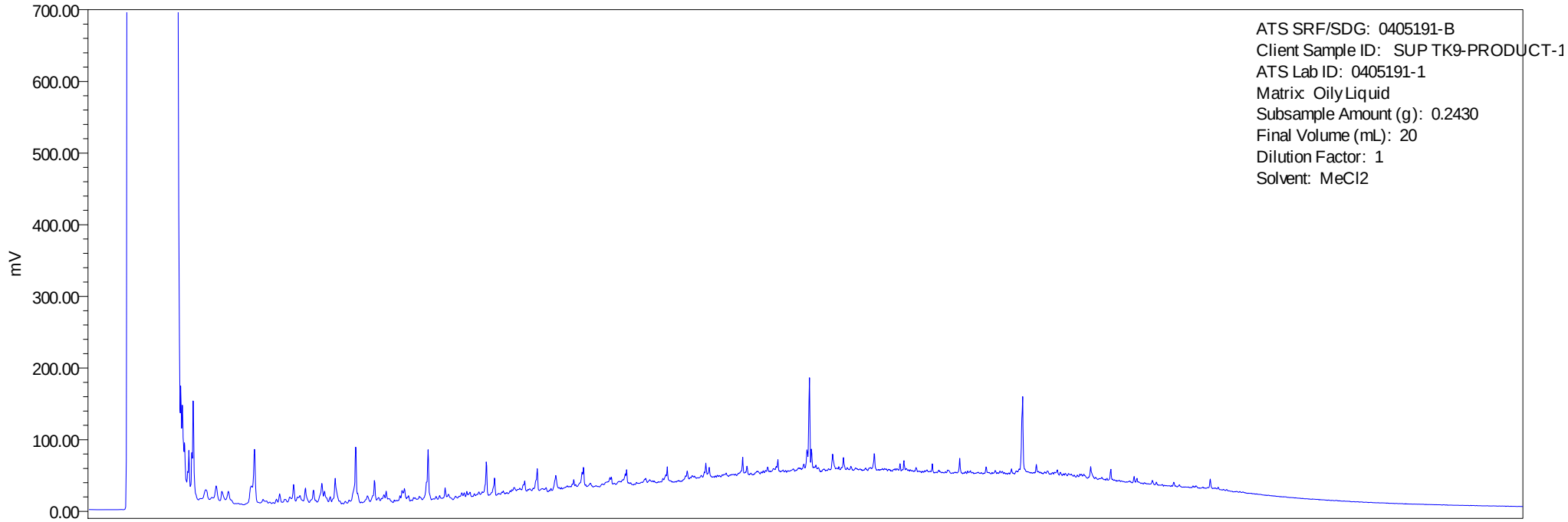
Comments

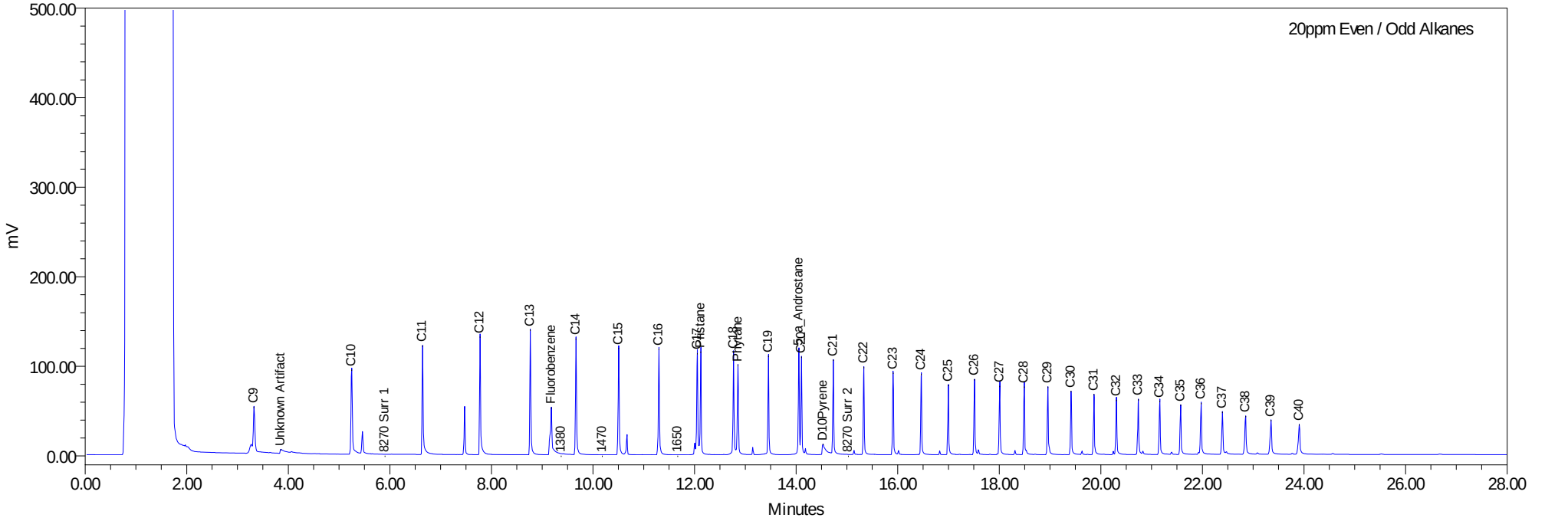
Matrix interference precluded lower detection limit.
All calculations performed prior to rounding.
MDL/PQL values assume 100% solids content.
nc = Not calculated.

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

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

#BENB-T02 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
0405191-1	SUP TK9-PRODUCT-1	3800 FID	04/08/2019	69.6		118	
0405191-1 MS	SUP TK9-PRODUCT-1 MS	3800 FID	04/08/2019	55.5		113	
0405191-1 MSD	SUP TK9-PRODUCT-1 MSD	3800 FID	04/09/2019	69.7		135	
LFB-1 4/8/19	LFB-1 4/8/19	3800 FID	04/08/2019	71.5		56.9	
LRB-1 4/8/19	LRB-1 4/8/19	3800 FID	04/08/2019	70.2		97.1	

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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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 4/8/19	04/08/2019	19:22:43	2,6,10 Trimethyldodecane (1380)	3891-98-3		mg/kg	WET	13.6	45.4	
LRB-1 4/8/19	04/08/2019	19:22:43	2,6,10 Trimethyltridecane (1470)	3891-99-4		mg/kg	WET	10.9	36.3	
LRB-1 4/8/19	04/08/2019	19:22:43	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6		mg/kg	WET	14.4	48.1	
LRB-1 4/8/19	04/08/2019	19:22:43	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8		mg/kg	WET	11	36.7	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Decane (C10)	124-18-5		mg/kg	WET	4.9	16.3	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Docosane (C22)	629-97-0		mg/kg	WET	15.5	51.8	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Dodecane (C12)	112-40-3		mg/kg	WET	9.9	32.9	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Dotriacontane (C32)	544-85-4		mg/kg	WET	14.9	49.5	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Eicosane (C20)	112-95-8		mg/kg	WET	10.9	36.3	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Heneicosane (C21)	629-94-7		mg/kg	WET	10.8	35.9	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Hentriacontane (C31)	630-04-6		mg/kg	WET	15.2	50.7	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Heptacosane (C27)	593-49-7		mg/kg	WET	10.1	33.5	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Heptadecane (C17)	629-78-7		mg/kg	WET	12.1	40.3	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Heptatriacontane (C37)	7194-84-5		mg/kg	WET	16.2	54.1	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Hexacosane (C26)	630-01-3		mg/kg	WET	9	30	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Hexadecane (C16)	544-76-3		mg/kg	WET	10.6	35.2	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Hexatriacontane (C36)	630-06-8		mg/kg	WET	20.4	67.8	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Nonacosane (C29)	630-03-5		mg/kg	WET	12.4	41.4	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Nonadecane (C19)	629-92-5		mg/kg	WET	8.6	28.5	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Nonane (C9)	111-84-2		mg/kg	WET	11.7	39.1	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Nonatriacontane (C39)	7194-86-7		mg/kg	WET	21.5	71.5	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Octacosane (C28)	630-02-4		mg/kg	WET	12.2	40.7	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Octadecane (C18)	593-45-3		mg/kg	WET	7.3	24.5	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Octatriacontane (C38)	7194-85-6		mg/kg	WET	23	76.7	
LRB-1 4/8/19	04/08/2019	19:22:43	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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 4/8/19	04/08/2019	19:22:43	n-Pentacosane (C25)	629-99-2		mg/kg	WET	11.4	38	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Pentadecane (C15)	629-62-9		mg/kg	WET	10.9	36.3	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Pentatriacontane (C35)	630-07-9		mg/kg	WET	16.7	55.8	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Tetracontane (C40)	4181-95-7		mg/kg	WET	21.5	71.8	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Tetracosane (C24)	646-31-1		mg/kg	WET	13.8	46	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Tetradecane (C14)	629-59-4		mg/kg	WET	13.6	45.4	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Tetratriacontane (C34)	14167-59-0		mg/kg	WET	16.1	53.7	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Triacontane (C30)	638-68-6		mg/kg	WET	14	46.6	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Tricosane (C23)	638-67-5		mg/kg	WET	13.2	43.8	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Tridecane (C13)	629-50-5		mg/kg	WET	6.9	23.1	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Tritriacontane (C33)	630-05-7		mg/kg	WET	13.6	45.5	
LRB-1 4/8/19	04/08/2019	19:22:43	n-Undecane (C11)	1120-21-4		mg/kg	WET	4.5	15.1	
LRB-1 4/8/19	04/08/2019	19:22:43	Total Petroleum Hydrocarbons (C9-C44)	C9-C44		mg/kg	WET	1100	3600	
LRB-1 4/8/19	04/08/2019	19:22:43	Total Resolvable Hydrocarbons	TOTRESHC		mg/kg	WET	1100	3600	
LRB-1 4/8/19	04/08/2019	19:22:43	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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 4/8/19	04/08/2019	19:58:52	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6		200	149	mg/kg	WET	74.7	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8		200	153	mg/kg	WET	76.3	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Decane (C10)	124-18-5		200	146	mg/kg	WET	73.1	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Docosane (C22)	629-97-0		200	171	mg/kg	WET	85.5	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Dodecane (C12)	112-40-3		200	162	mg/kg	WET	81.0	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Dotriacontane (C32)	544-85-4		200	159	mg/kg	WET	79.7	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Eicosane (C20)	112-95-8		200	199	mg/kg	WET	99.5	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Heneicosane (C21)	629-94-7		200	162	mg/kg	WET	80.8	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Hentriacontane (C31)	630-04-6		200	161	mg/kg	WET	80.5	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Heptacosane (C27)	593-49-7		200	173	mg/kg	WET	86.5	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Heptadecane (C17)	629-78-7		200	169	mg/kg	WET	84.6	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Heptatriacontane (C37)	7194-84-5		200	172	mg/kg	WET	86.2	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Hexacosane (C26)	630-01-3		200	163	mg/kg	WET	81.5	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Hexadecane (C16)	544-76-3		200	149	mg/kg	WET	74.6	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Hexatriacontane (C36)	630-06-8		200	146	mg/kg	WET	73.0	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Nonacosane (C29)	630-03-5		200	160	mg/kg	WET	80.1	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Nonadecane (C19)	629-92-5		200	158	mg/kg	WET	78.8	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Nonane (C9)	111-84-2		200	260	mg/kg	WET	130	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Nonatriacontane (C39)	7194-86-7		200	150	mg/kg	WET	75.2	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Octacosane (C28)	630-02-4		200	175	mg/kg	WET	87.5	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Octadecane (C18)	593-45-3		200	173	mg/kg	WET	86.7	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Octatriacontane (C38)	7194-85-6		200	150	mg/kg	WET	75.1	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Pentacosane (C25)	629-99-2		200	209	mg/kg	WET	104	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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 4/8/19	04/08/2019	19:58:52	n-Pentadecane (C15)	629-62-9		200	147	mg/kg	WET	73.4	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Pentatriacontane (C35)	630-07-9		200	158	mg/kg	WET	79.0	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Tetracontane (C40)	4181-95-7		200	153	mg/kg	WET	76.3	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Tetracosane (C24)	646-31-1		200	176	mg/kg	WET	88.1	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Tetradecane (C14)	629-59-4		200	154	mg/kg	WET	77.2	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Tetratriacontane (C34)	14167-59-0		200	142	mg/kg	WET	70.8	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Triacontane (C30)	638-68-6		200	176	mg/kg	WET	88.2	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Tricosane (C23)	638-67-5		200	174	mg/kg	WET	87.1	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Tridecane (C13)	629-50-5		200	150	mg/kg	WET	74.8	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Tritriacontane (C33)	630-05-7		200	148	mg/kg	WET	74.1	70	130	
LFB-1 4/8/19	04/08/2019	19:58:52	n-Undecane (C11)	1120-21-4		200	155	mg/kg	WET	77.5	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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
0405191-1 MS	04/08/2019	23:00:40	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6	172	876	887	mg/kg	WET	81.6	70	130	
0405191-1 MS	04/08/2019	23:00:40	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8	171	876	942	mg/kg	WET	88.0	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Decane (C10)	124-18-5	1340	876	NR	mg/kg	WET	nc	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Docosane (C22)	629-97-0	409	876	1160	mg/kg	WET	86.3	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Dodecane (C12)	112-40-3	589	876	1350	mg/kg	WET	86.8	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Dotriacontane (C32)	544-85-4	133	876	1010	mg/kg	WET	100	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Eicosane (C20)	112-95-8	411	876	1140	mg/kg	WET	82.7	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Heneicosane (C21)	629-94-7	326	876	1030	mg/kg	WET	80.9	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Hentriacontane (C31)	630-04-6	58.8	876	883	mg/kg	WET	94.2	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Heptacosane (C27)	593-49-7	214	876	1060	mg/kg	WET	96.7	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Heptadecane (C17)	629-78-7	216	876	866	mg/kg	WET	74.2	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Heptatriacontane (C37)	7194-84-5	70.6	876	1150	mg/kg	WET	123	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Hexacosane (C26)	630-01-3	221	876	955	mg/kg	WET	83.9	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Hexadecane (C16)	544-76-3	315	876	967	mg/kg	WET	74.5	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Hexatriacontane (C36)	630-06-8	47.2	876	1080	mg/kg	WET	118	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Nonacosane (C29)	630-03-5	99.4	876	882	mg/kg	WET	89.3	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Nonadecane (C19)	629-92-5	246	876	918	mg/kg	WET	76.7	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Nonane (C9)	111-84-2	3040	876	NR	mg/kg	WET	nc	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Nonatriacontane (C39)	7194-86-7	30.8	876	1020	mg/kg	WET	113	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Octacosane (C28)	630-02-4	296	876	1110	mg/kg	WET	93.5	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Octadecane (C18)	593-45-3	227	876	982	mg/kg	WET	86.2	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Octatriacontane (C38)	7194-85-6	35.5	876	1030	mg/kg	WET	113	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Pentacosane (C25)	629-99-2	445	876	1200	mg/kg	WET	85.9	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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
0405191-1 MS	04/08/2019	23:00:40	n-Pentadecane (C15)	629-62-9	399	876	1060	mg/kg	WET	76.1	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Pentatriacontane (C35)	630-07-9	106	876	1270	mg/kg	WET	133	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Tetracontane (C40)	4181-95-7	45.7	876	1100	mg/kg	WET	121	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Tetracosane (C24)	646-31-1	164	876	958	mg/kg	WET	90.7	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Tetradecane (C14)	629-59-4	608	876	1210	mg/kg	WET	68.4	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Tetratriacontane (C34)	14167-59-0	221	876	1000	mg/kg	WET	89.4	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Triacontane (C30)	638-68-6	136	876	979	mg/kg	WET	96.2	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Tricosane (C23)	638-67-5	257	876	1060	mg/kg	WET	92.0	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Tridecane (C13)	629-50-5	501	876	1200	mg/kg	WET	79.8	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Tritriacontane (C33)	630-05-7	191	876	1050	mg/kg	WET	98.0	70	130	
0405191-1 MS	04/08/2019	23:00:40	n-Undecane (C11)	1120-21-4	1120	876	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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
0405191-1 MSD	04/09/2019	00:13:08	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6	172	851	898	mg/kg	WET	85.3	70	130	
0405191-1 MSD	04/09/2019	00:13:08	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8	171	851	941	mg/kg	WET	90.4	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Decane (C10)	124-18-5	1340	851	NR	mg/kg	WET	nc	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Docosane (C22)	629-97-0	409	851	1180	mg/kg	WET	90.2	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Dodecane (C12)	112-40-3	589	851	1260	mg/kg	WET	78.9	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Dotriacontane (C32)	544-85-4	133	851	1080	mg/kg	WET	112	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Eicosane (C20)	112-95-8	411	851	1170	mg/kg	WET	88.7	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Heneicosane (C21)	629-94-7	326	851	1060	mg/kg	WET	86.4	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Hentriacontane (C31)	630-04-6	58.8	851	873	mg/kg	WET	95.6	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Heptacosane (C27)	593-49-7	214	851	1060	mg/kg	WET	99.8	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Heptadecane (C17)	629-78-7	216	851	853	mg/kg	WET	74.8	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Heptatriacontane (C37)	7194-84-5	70.6	851	1090	mg/kg	WET	119	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Hexacosane (C26)	630-01-3	221	851	1030	mg/kg	WET	94.9	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Hexadecane (C16)	544-76-3	315	851	1050	mg/kg	WET	86.1	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Hexatriacontane (C36)	630-06-8	47.2	851	1120	mg/kg	WET	126	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Nonacosane (C29)	630-03-5	99.4	851	902	mg/kg	WET	94.3	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Nonadecane (C19)	629-92-5	246	851	903	mg/kg	WET	77.2	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Nonane (C9)	111-84-2	3040	851	NR	mg/kg	WET	nc	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Nonatriacontane (C39)	7194-86-7	30.8	851	975	mg/kg	WET	111	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Octacosane (C28)	630-02-4	296	851	1060	mg/kg	WET	89.8	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Octadecane (C18)	593-45-3	227	851	963	mg/kg	WET	86.5	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Octatriacontane (C38)	7194-85-6	35.5	851	983	mg/kg	WET	111	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Pentacosane (C25)	629-99-2	445	851	1250	mg/kg	WET	94.5	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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
0405191-1 MSD	04/09/2019	00:13:08	n-Pentadecane (C15)	629-62-9	399	851	1180	mg/kg	WET	92.4	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Pentatriacontane (C35)	630-07-9	106	851	1300	mg/kg	WET	140	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Tetracontane (C40)	4181-95-7	45.7	851	985	mg/kg	WET	110	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Tetracosane (C24)	646-31-1	164	851	952	mg/kg	WET	92.6	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Tetradecane (C14)	629-59-4	608	851	1410	mg/kg	WET	94.6	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Tetratriacontane (C34)	14167-59-0	221	851	1040	mg/kg	WET	96.2	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Triacontane (C30)	638-68-6	136	851	1070	mg/kg	WET	109	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Tricosane (C23)	638-67-5	257	851	1110	mg/kg	WET	101	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Tridecane (C13)	629-50-5	501	851	1300	mg/kg	WET	94.3	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Tritriacontane (C33)	630-05-7	191	851	1050	mg/kg	WET	101	70	130	
0405191-1 MSD	04/09/2019	00:13:08	n-Undecane (C11)	1120-21-4	1120	851	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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
0405191-1 MS	04/08/2019	23:00:40	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6	887		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	2,6,10,14-TETRAMETHYL PENTADECANE	1921-70-6	898	893	mg/kg	WET	1.28	20	
0405191-1 MS	04/08/2019	23:00:40	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8	942		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	2,6,10,14-TETRAMETHYLHEXADECANE	638-36-8	941	941	mg/kg	WET	0.134	20	
0405191-1 MS	04/08/2019	23:00:40	n-Decane (C10)	124-18-5	1740		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Decane (C10)	124-18-5	1620	1680	mg/kg	WET	7.64	20	
0405191-1 MS	04/08/2019	23:00:40	n-Docosane (C22)	629-97-0	1160		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Docosane (C22)	629-97-0	1180	1170	mg/kg	WET	1.04	20	
0405191-1 MS	04/08/2019	23:00:40	n-Dodecane (C12)	112-40-3	1350		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Dodecane (C12)	112-40-3	1260	1300	mg/kg	WET	6.80	20	
0405191-1 MS	04/08/2019	23:00:40	n-Dotriacontane (C32)	544-85-4	1010		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Dotriacontane (C32)	544-85-4	1080	1050	mg/kg	WET	6.87	20	
0405191-1 MS	04/08/2019	23:00:40	n-Eicosane (C20)	112-95-8	1140		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Eicosane (C20)	112-95-8	1170	1150	mg/kg	WET	2.65	20	
0405191-1 MS	04/08/2019	23:00:40	n-Heneicosane (C21)	629-94-7	1030		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Heneicosane (C21)	629-94-7	1060	1050	mg/kg	WET	2.61	20	
0405191-1 MS	04/08/2019	23:00:40	n-Hentriacontane (C31)	630-04-6	883		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Hentriacontane (C31)	630-04-6	873	878	mg/kg	WET	1.23	20	
0405191-1 MS	04/08/2019	23:00:40	n-Heptacosane (C27)	593-49-7	1060		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Heptacosane (C27)	593-49-7	1060	1060	mg/kg	WET	0.207	20	
0405191-1 MS	04/08/2019	23:00:40	n-Heptadecane (C17)	629-78-7	866		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Heptadecane (C17)	629-78-7	853	860	mg/kg	WET	1.53	20	
0405191-1 MS	04/08/2019	23:00:40	n-Heptatriacontane (C37)	7194-84-5	1150		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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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
0405191-1 MSD	04/09/2019	00:13:08	n-Heptatriacontane (C37)	7194-84-5	1090	1120	mg/kg	WET	5.71	20	
0405191-1 MS	04/08/2019	23:00:40	n-Hexacosane (C26)	630-01-3	955		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Hexacosane (C26)	630-01-3	1030	992	mg/kg	WET	7.39	20	
0405191-1 MS	04/08/2019	23:00:40	n-Hexadecane (C16)	544-76-3	967		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Hexadecane (C16)	544-76-3	1050	1010	mg/kg	WET	8.06	20	
0405191-1 MS	04/08/2019	23:00:40	n-Hexatriacontane (C36)	630-06-8	1080		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Hexatriacontane (C36)	630-06-8	1120	1100	mg/kg	WET	3.17	20	
0405191-1 MS	04/08/2019	23:00:40	n-Nonacosane (C29)	630-03-5	882		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Nonacosane (C29)	630-03-5	902	892	mg/kg	WET	2.25	20	
0405191-1 MS	04/08/2019	23:00:40	n-Nonadecane (C19)	629-92-5	918		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Nonadecane (C19)	629-92-5	903	911	mg/kg	WET	1.59	20	
0405191-1 MS	04/08/2019	23:00:40	n-Nonane (C9)	111-84-2	3210		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Nonane (C9)	111-84-2	2990	3100	mg/kg	WET	7.04	20	
0405191-1 MS	04/08/2019	23:00:40	n-Nonatriacontane (C39)	7194-86-7	1020		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Nonatriacontane (C39)	7194-86-7	975	1000	mg/kg	WET	4.90	20	
0405191-1 MS	04/08/2019	23:00:40	n-Octacosane (C28)	630-02-4	1110		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Octacosane (C28)	630-02-4	1060	1090	mg/kg	WET	5.00	20	
0405191-1 MS	04/08/2019	23:00:40	n-Octadecane (C18)	593-45-3	982		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Octadecane (C18)	593-45-3	963	972	mg/kg	WET	1.93	20	
0405191-1 MS	04/08/2019	23:00:40	n-Octatriacontane (C38)	7194-85-6	1030		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Octatriacontane (C38)	7194-85-6	983	1010	mg/kg	WET	4.52	20	
0405191-1 MS	04/08/2019	23:00:40	n-Pentacosane (C25)	629-99-2	1200		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Pentacosane (C25)	629-99-2	1250	1220	mg/kg	WET	4.24	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: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/10/2019

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
0405191-1 MS	04/08/2019	23:00:40	n-Pentadecane (C15)	629-62-9	1060		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Pentadecane (C15)	629-62-9	1180	1120	mg/kg	WET	10.7	20	
0405191-1 MS	04/08/2019	23:00:40	n-Pentatriacontane (C35)	630-07-9	1270		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Pentatriacontane (C35)	630-07-9	1300	1280	mg/kg	WET	2.13	20	
0405191-1 MS	04/08/2019	23:00:40	n-Tetracontane (C40)	4181-95-7	1100		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Tetracontane (C40)	4181-95-7	985	1040	mg/kg	WET	11.1	20	
0405191-1 MS	04/08/2019	23:00:40	n-Tetracosane (C24)	646-31-1	958		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Tetracosane (C24)	646-31-1	952	955	mg/kg	WET	0.562	20	
0405191-1 MS	04/08/2019	23:00:40	n-Tetradecane (C14)	629-59-4	1210		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Tetradecane (C14)	629-59-4	1410	1310	mg/kg	WET	15.8	20	
0405191-1 MS	04/08/2019	23:00:40	n-Tetratriacontane (C34)	14167-59-0	1000		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Tetratriacontane (C34)	14167-59-0	1040	1020	mg/kg	WET	3.55	20	
0405191-1 MS	04/08/2019	23:00:40	n-Triacontane (C30)	638-68-6	979		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Triacontane (C30)	638-68-6	1070	1020	mg/kg	WET	8.51	20	
0405191-1 MS	04/08/2019	23:00:40	n-Tricosane (C23)	638-67-5	1060		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Tricosane (C23)	638-67-5	1110	1090	mg/kg	WET	4.74	20	
0405191-1 MS	04/08/2019	23:00:40	n-Tridecane (C13)	629-50-5	1200		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Tridecane (C13)	629-50-5	1300	1250	mg/kg	WET	8.35	20	
0405191-1 MS	04/08/2019	23:00:40	n-Tritriacontane (C33)	630-05-7	1050		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Tritriacontane (C33)	630-05-7	1050	1050	mg/kg	WET	0.235	20	
0405191-1 MS	04/08/2019	23:00:40	n-Undecane (C11)	1120-21-4	1580		mg/kg	WET			
0405191-1 MSD	04/09/2019	00:13:08	n-Undecane (C11)	1120-21-4	1490	1540	mg/kg	WET	5.40	20	

Comments
Calculations performed prior to rounding.

ORGANIC ANALYSIS

POLYNUCLEAR AROMATIC HYDROCARBON
and
BIOMARKER ANALYSIS
USEPA METHOD SW8270-M

ATS Project Number: BENB-T02

ATS SDG: 0405191-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-T02
ATS SDG: 0405191-B
Analysis Method: 8270M

Sample Preparation

The sample was received in two 40mL glass containers 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

Samples were analyzed by full scan GC/MS. An initial calibration with at least five levels was used to quantitate the individual compounds. Triterpane concentrations were calculated using the response factor for Hopane, and sterane concentrations were calculated using the response factor for Cholane. Alkyl PAH homologue groups were calculated using the response factor of the parent PAH compound (or compounds). Concentrations were reported to the method detection limit (MDL). MDLs for triterpanes, steranes, and alkyl PAH groups are based on Hopane, Cholane, and parent PAHs respectively. Samples were reported on a wet weight basis.

Anomalies Noted:

- **None**

Calibration Verification

Method calibration and instrument suitability was verified through the running of a tuning solution and a mid-level calibration verification standard at a minimum of every 24 hours. 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 12 hours. All instrument blanks met the acceptance criteria with the following exceptions:

- **None**

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Internal Standards and Surrogates

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

- **None**

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

- **None**

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
0405191-1 MS	Benzo(g,h,i)perylene	127	50-125%
0405191-1 MS	Fluoranthene	144	50-125%
0405191-1 MSD	Benzo(a)anthracene	138	50-125%
0405191-1 MSD	Fluoranthene	149	50-125%

- **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
0405191-1 MS / MSD	Benzo(a)anthracene	31.0	<30%
0405191-1 MS / MSD	Benzo(g,h,i)perylene	42.4	<30%

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**

_____/ April 15, 2019

Mark T. DeLong (Quality Assurance Coordinator)

_____/ April 15, 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-T02	0405191-B	SW8270M	SUP TK9-PRODUCT-1	0405191-1

Polynuclear Aromatic Hydrocarbons Analysis
Data Summary Sheet

ATS Project Number	BENB-T02	**Percent Moisture**	NA
ATS SDG Number	0405191-B	**Preparation Date**	04/08/2019
Client Sample ID	SUP TK9-PRODUCT-1	**Analysis Date:**	04/11/2019
Laboratory Sample ID	0405191-1	**Instrument**	2100T
Matrix	Product	**Subsample (g)**	0.243
Sample Date	04/04/2019	**Final Volume (mL)**	20
Analytical Method (USEPA)	SW8270M	**Dilution Factor**	1
Preparation Method (USEPA)	USEPA Method 3580A	**Basis**	WET
QC Batch Number	QCORG0408191-B	**Units**	mg/kg

Parameter	Chemical Identifier	Result	MDL	PQL	Qual
2 Ring Compounds					
cis/trans-Decalin	CTDecalin	80.1	0.66	2.2	
C1-Decalins	DecalinsC1	268	0.66	2.2	
C2-Decalins	DecalinsC2	350	0.66	2.2	
C3-Decalins	DecalinsC3	361	0.66	2.2	
C4-Decalins	DecalinsC4	214	0.66	2.2	
Naphthalene	91-20-3	23.8	0.6	2	
C1-Naphthalenes	NPHC1	55.1	0.6	2	
1-Methylnaphthalene	90-12-0	41.6	0.6	2	
2-Methylnaphthalene	91-57-6	66.0	0.6	2	
C2-Naphthalenes	NPHC2	192	0.6	2	
2,6-Dimethylnaphthalene	581-42-0	82.7	0.6	2	
C3-Naphthalenes	NPHC3	250	0.6	2	
2,3,5-Trimethylnaphthalene	2245-38-7	27.2	0.6	2	
C4-Naphthalenes	NPHC4	237	0.6	2	
Benzo(b)thiophene	95-15-8	ND	0.66	2.2	U
C1-Benzo(b)thiophenes	BZTPHC1	19.1	0.66	2.2	
C2-Benzo(b)thiophenes	BZTPHC2	41.9	0.66	2.2	
C3-Benzo(b)thiophenes	BZTPHC3	84.0	0.66	2.2	
C4-Benzo(b)thiophenes	BZTPHC4	138	0.66	2.2	
Biphenyl	92-52-4	4.86	0.29	1	
3 Ring Compounds					
Fluorene	86-73-7	ND	0.5	1.7	U
C1-Fluorenes	FLUORC1	57.4	0.5	1.7	
1-Methylfluorene	1730-37-6	24.0	0.5	1.7	
C2-Fluorenes	FLUORC2	186	0.5	1.7	
C3-Fluorenes	FLUORC3	204	0.5	1.7	
Dibenzothiophene	132-65-0	18.5	0.44	1.5	
C1-Dibenzothiophenes	DBZTPC1	58.4	0.44	1.5	
1-Methyldibenzothiophene	31317-07-4	14.5	0.44	1.5	
2/3-Methyldibenzothiophene	DBZTPM2M3	16.8	0.44	1.5	
4-Methyldibenzothiophene	7372-88-5	35.4	0.44	1.5	
C2-Dibenzothiophenes	DBZTPC2	183	0.44	1.5	
4,6-Dimethyldibenzothiophene	1207-12-1	20.7	0.44	1.5	
C3-Dibenzothiophenes	DBZTPC3	339	0.44	1.5	
2,3,7-Trimethyldibenzothiophene	153524-16-4	12.9	0.44	1.5	
2,4,6-Trimethyldibenzothiophene	185493-79-7	11.9	0.44	1.5	
2-n-Propyldibenzothiophene	147792-30-1	51.6	0.44	1.5	
4-Ethyl-6-methyldibenzothiophene	132034-90-3	23.7	0.44	1.5	
4-n-Propyldibenzothiophene	132034-86-7	ND	0.44	1.5	U
C4-Dibenzothiophenes	DBZTPC4	298	0.44	1.5	
2-n-Butyldibenzothiophene	147792-31-2	9.22	0.44	1.5	
4,6-Diethyldibenzothiophene	132034-91-4	4.77	0.44	1.5	
4-n-Butyldibenzothiophene	147792-33-4	12.4	0.44	1.5	
2-n-Pentyldibenzothiophene	147793-32-3	15.5	0.44	1.5	
4-n-Pentyldibenzothiophene	147792-34-5	9.88	0.44	1.5	

Surrogate Recoveries			LCL	UCL
2-Fluorobiphenyl (Surr)	321-60-8	63.5	30	150
5B (H) Cholane (Surr)	5BCHOLANE (S)	109	50	150
Pyrene-D10 (SURR)	1718-52-1	128	50	150

Parameter	Chemical Identifier	Result	MDL	PQL	Qual
3 Ring Compounds Cont.					
Phenanthrene	85-01-8	40.7	0.28	0.9	
Anthracene	120-12-7	ND	0.08	0.3	U
C1-Phenanthrenes / Anthracenes	PHANC1	109	0.18	0.6	
2-Methylanthracene	613-12-7	3.13	0.08	0.3	
1-Methylphenanthrene	832-69-9	26.0	0.18	0.6	
2-Methylphenanthrene	2531-84-2	35.6	0.18	0.6	
3-Methylphenanthrene	832-71-3	18.1	0.18	0.6	
9/4-Methylphenanthrene	4/9-methylphen	27.6	0.18	0.6	
C2-Phenanthrenes / Anthracenes	PHANC2	185	0.18	0.6	
3,6-Dimethylphenanthrene	1576-67-6	20.8	0.18	0.6	
C3-Phenanthrenes / Anthracenes	PHANC3	198	0.18	0.6	
1,2,4 / 1,2,9-Trimethylphenanthrene	23189-64-2 / 146448-88-6	11.1	0.18	0.6	
1,2,8-Trimethylphenanthrene	20291-75-2	24.4	0.18	0.6	
1,3,4-Trimethylphenanthrene	66271-45-2	22.3	0.18	0.6	
2,6,9-Trimethylphenanthrene	66271-32-7	52.6	0.18	0.6	
C4-Phenanthrenes / Anthracenes	PHANC4	206	0.18	0.6	
1,2,6,9-Tetramethylphenanthrene	204256-39-3	8.40	0.18	0.6	
9-n-Butylphenanthrene	10394-57-7	ND	0.18	0.6	U
Retene (7-Isopropyl-1-methylphenanthrene)	483-65-8	9.47	0.18	0.6	
Acenaphthene	83-32-9	ND	0.32	1.1	U
Acenaphthylene	208-96-8	ND	0.23	0.8	U
Carbazole	86-74-8	ND	0.57	1.9	U
Dibenzofuran	132-64-9	4.20	0.15	0.5	
4 Ring Compounds					
Fluoranthene	206-44-0	5.76	0.29	1	
Pyrene	129-00-0	21.4	0.3	1	
C1-Fluoranthenes / Pyrenes	FLPYC1	216	0.3	1	
2-Methylfluoranthene	33543-31-6	27.0	0.29	1	
C2-Fluoranthenes / Pyrenes	FLPYC2	ND	0.3	1	U
C3-Fluoranthenes / Pyrenes	FLPYC3	ND	0.3	1	U
C4-Fluoranthenes / Pyrenes	FLPYC4	259	0.3	1	
Naphthobenzothiophene-1,2D	205-43-6	ND	0.26	0.9	U
Naphthobenzothiophene-2,1D	239-35-0	ND	0.26	0.9	U
Naphthobenzothiophene-2,3D	243-46-9	ND	0.26	0.9	U
Total Naphthobenzothiophenes	NAPHTHBZOTHPH	ND	0.26	0.9	U
C1-Naphthobenzothiophenes	NBZTPC1	ND	0.26	0.9	U
C2-Naphthobenzothiophenes	NBZTPC2	428	0.26	0.9	
C3-Naphthobenzothiophenes	NBZTPC3	136	0.26	0.9	
C4-Naphthobenzothiophenes	NBZTPC4	ND	0.26	0.9	U
Chrysene/Triphenylene	CHRYTRIPHEN	ND	0.56	1.9	U
C1-Chrysenes	BZACHRYSC1	ND	0.56	1.9	U
6-Methylchrysene	1705-85-7	ND	0.56	1.9	U
C2-Chrysenes	BZACHRYSC2	ND	0.56	1.9	U
C3-Chrysenes	BZACHRYSC3	ND	0.56	1.9	U
C4-Chrysenes	BZACHRYSC4	ND	0.56	1.9	U
Benz[a]anthracene	56-55-3	ND	0.29	1	U
Benzo(b)fluorene	243-17-4	ND	0.29	1	U
5 Ring Compounds					
Benzo[b]fluoranthene	205-99-2	ND	0.86	2.9	U
Benzo[J]+[k]fluoranthene	BJFBKF	ND	1.03	3.4	U
Benzo[a]pyrene	50-32-8	ND	0.48	1.6	U
Benzo[e]pyrene	192-97-2	ND	0.48	1.6	U
Perylene	198-55-0	ND	0.31	1	U
6 Ring Compounds					
Benzo[g,h,i]perylene	191-24-2	ND	0.38	1.3	U
Dibenz[a,h]anthracene	53-70-3	ND	0.44	1.5	U
Indeno[1,2,3-cd]pyrene	193-39-5	ND	0.33	1.1	U

Comments

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

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Terpane and Sterane Analysis
Data Summary Sheet

ATS Project Number	BENB-T02	**Percent Moisture**	NA
ATS SDG Number	0405191-B	**Preparation Date**	04/08/2019
Client Sample ID	SUP TK9-PRODUCT-1	**Analysis Date:**	04/11/2019
Laboratory Sample ID	0405191-1	**Instrument**	2100T
Matrix	Product	**Subsample (g)**	0.243
Sample Date	04/04/2019	**Final Volume (mL)**	20
Analytical Method (USEPA)	SW8270M	**Dilution Factor**	1
Preparation Method (USEPA)	USEPA Method 3580A	**Basis**	WET
QC Batch Number	QCORG0408191-B	**Units**	mg/kg

Parameter	Chemical Identifier	Result	MDL	PQL	Qual
Terpanes					
C23 Tricyclic Terpane	C23-TRITERP	352	0.99	3.3	
C24 Tricyclic Terpane	C24-TRITERP	147	0.99	3.3	
C25 Tricyclic Terpane	C25-TRITERP	143	0.99	3.3	
C24 Tetracyclic Terpane	C24-TETRATERP	39.9	0.99	3.3	
C26 Tricyclic Terpane-22S	C26-TRITERP22S	ND	0.99	3.3	U
C26 Tricyclic Terpane-22R	C26-TRITERP22R	166	0.99	3.3	
C28 Tricyclic Terpane-22S	C28-TRITERP22S	87.9	0.99	3.3	
C28 Tricyclic Terpane-22R	C28-TRITERP22R	45.2	0.99	3.3	
C29 Tricyclic Terpane-22S	C29-TRITERP22S	81.4	0.99	3.3	
C29 Tricyclic Terpane-22R	C29-TRITERP22R	55.9	0.99	3.3	
18a-22,29,30-Trisnorneohopane-TS	TRISNORNEOHP01	37.2	0.29	1	
C30 Tricyclic Terpane-22S	C30-TRITERP22S	88.6	0.99	3.3	
C30 Tricyclic Terpane-22R	C30-TRITERP22R	45.2	0.99	3.3	
17a(H)-22,29,30-Trisnorhopane-TM	53584-59-1	174	0.99	3.3	
17a/b,21b/a 28,30-Bisnorhopane	BISNORHOPANE01	95.0	0.29	1	
17a(H),21b(H)-25-Norhopane	NORHOPANE01	35.1	0.29	1	
30-Norhopane (T15)	53584-60-4	416	0.99	3.3	
18a(H)-30-Norneohopane-C29Ts	NORNEOHPANE01	ND	0.99	3.3	U
17a(H)-Diahopane	DIAHOPANE01	11.9	0.99	3.3	
30-Normoretane	3258-87-5	37.4	0.99	3.3	
18a(H)&18b(H)-Oleananes	OLEANANE01	ND	0.99	3.3	U
Hopane	13849-96-2	214	0.99	3.3	
Moretane	1176-44-9	18.0	0.99	3.3	
30-Homohopane-22S	60305-23-6	206	0.99	3.3	
30-Homohopane-22R	60305-22-8	175	0.99	3.3	
T22a-Gammacerane/C32-diahopane	GAMACERDIAHP01	106	0.99	3.3	
30,31-Bishomohopane-22S	BISNORHOPANE02	126	0.99	3.3	
30,31-Bishomohopane-22R	BISNORHOPANE03	56.5	0.99	3.3	
30,31-Trishomohopane-22S	TRISHOMOHOP01	5.51	0.99	3.3	
30,31-Trishomohopane-22R	TRISHOMOHOP02	103	0.99	3.3	
Tetrakishomohopane-22S	TETRAKSHOMHP01	40.7	0.99	3.3	
Tetrakishomohopane-22R	TETRAKSHOMHP02	66.7	0.99	3.3	
Pentakishomohopane-22S	PENTAKSHOMHP01	69.9	0.99	3.3	
Pentakishomohopane-22R	PENTAKSHOMHP02	37.6	0.99	3.3	

Parameter	Chemical Identifier	Result	MDL	PQL	Qual
Steranes					
13b(H),17a(H)-20S-Diacholestane	DIACHOLESTAN01	79.2	0.29	1	
13b(H),17a(H)-20R-Diacholestane	82079-08-1	ND	0.29	1	U
13b,17a-20S-Methyldiacholestane	METHYLDIACHO01	23.0	0.29	1	
14a(H),17a(H)-20S-Chloestane/13b(H),17a(H)-20S-Ethyldiacholestane (S12)	CHOLETHDIACH01	69.5	0.29	1	
14a(H),17a(H)-20R-Chloestane/13b(H),17a(H)-20R-Ethyldiacholestane (S17)	CHOLETHDIACH02	68.1	0.29	1	
Unknown Sterane (S18)	UNKSTERANE01	13.3	0.29	1	
13a,17b-20S-Ethyldiacholestane	ETHDIACHOL02	30.3	0.29	1	
14a,17a-20S-Methylcholestane	METHDIACHOLE02	49.1	0.29	1	
14a,17a-20R-Methylcholestane	METHDIACHOLE03	ND	0.29	1	U
14a(H),17a(H)-20S-Ethylcholestane	ETHCHOLE01	47.0	0.29	1	
14a(H),17a(H)-20R-Ethylcholestane	ETHCHOLE02	58.2	0.29	1	
14b(H),17b(H)-20R-Cholestane	CHOLESTANE03	22.1	0.29	1	
14b(H),17b(H)-20S-Cholestane	CHOLESTANE04	48.1	0.29	1	
14b,17b-20R-Methylcholestane	METHCHOLE01	ND	0.29	1	U
14b,17b-20S-Methylcholestane	METHCHOLE02	ND	0.29	1	U
14b(H),17b(H)-20R-Ethylcholestane	ETHCHOLE03	46.5	0.29	1	
14b(H),17b(H)-20S-Ethylcholestane	ETHCHOLE04	7.82	0.29	1	
C26,20R+C27,20S Triaromatic Steroid	TRIAROMROID01	187	0.29	1	
C28,20S-Triaromatic Steroid	TRIAROMROID02	167	0.29	1	
C27,20R Triaromatic Steroid (C)	TRIAROMROID03	132	0.29	1	
C28,20R Triaromatic Steroid (C)	TRIAROMROID04	140	0.29	1	

Surrogate Recoveries			LCL	UCL
2-Fluorobiphenyl (Surr)	321-60-8	63.5	30	150
5B (H) Cholane (Surr)	5BCHOLANE (S)	109	50	150
Pyrene-D10 (SURR)	1718-52-1	128	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.

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ANN ARBOR TECHNICAL SERVICES, INC.

Surrogate Recovery Summary

SW8270M

Sample Identification	Field Sample Identification	Instrument ID	Analysis Date	Percent Recovery					
				2-FLUOROBIPHENYL	Flag	PYRENE-D10	Flag	5B(H)Cholane	Flag
0405191-1	SUP TK9-PRODUCT-1	2100T	04/11/2019	63.5		128		109	
0405191-1 MS	SUP TK9-PRODUCT-1 MS	2100T	04/11/2019	48.8		87.5		121	
0405191-1 MSD	SUP TK9-PRODUCT-1 MSD	2100T	04/11/2019	58.5		122		128	
LFB-1 4/8/19	LFB-1 4/8/19	2100T	04/11/2019	67.5		75.5		95.5	
LRB-1 4/8/19	LRB-1 4/8/19	2100T	04/11/2019	60.8		68.2		85.0	

	QA/QC Limits
2-FLUOROBIPHENYL	30-150
PYRENE-D10	50-150
5B(H)Cholane	50-150

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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 4/8/19	04/11/2019	18:45:09	1,2,4/1,2,9-Trimethylphenanthrene	23189-64-2 / 146448-88-6		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	1,2,6,9-Tetramethylphenanthrene	204256-39-3		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	1,2,8-Trimethylphenanthrene	20291-75-2		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	1,3,4-Trimethylphenanthrene	66271-45-2		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	13a,17b-20S-Ethyldiacholestane	ETHDIACHOL02		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	13b(H),17a(H)-20R-Diacholestane	82079-08-1		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	13b(H),17a(H)-20S-Diacholestane	DIACHOLESTAN01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	13b,17a-20S-Methyldiacholestane	METHYLDIACHO01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14a(H),17a(H)-20R-Cholestane/13b(H),17a(H)-20R-Ethyldiacholestane (S17)	CHOLETHDIACH02		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14a(H),17a(H)-20R-Ethylcholestane	ETHCHOLE02		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14a(H),17a(H)-20S-Cholestane/13b(H),17a(H)-20S-Ethyldiacholestane (S12)	CHOLETHDIACH01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14a(H),17a(H)-20S-Ethylcholestane	ETHCHOLE01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14a,17a-20R-Methylcholestane	METHDIACHOLE03		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14a,17a-20S-Methylcholestane	METHDIACHOLE02		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14b(H),17b(H)-20R-Cholestane	CHOLESTANE03		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14b(H),17b(H)-20R-Ethylcholestane	ETHCHOLE03		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14b(H),17b(H)-20S-Cholestane	CHOLESTANE04		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14b(H),17b(H)-20S-Ethylcholestane	ETHCHOLE04		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14b,17b-20R-Methylcholestane	METHCHOLE01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	14b,17b-20S-Methylcholestane	METHCHOLE02		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	17a(H),21b(H)-25-Norhopane	NORHOPANE01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	17a(H)-22,29,30-Trisnorhopane-TM	53584-59-1		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	17a(H)-Diahopane	DIAHOPANE01		mg/kg	WET	0.99	3.3	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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 4/8/19	04/11/2019	18:45:09	17a/b,21b/a 28,30-Bisnorhopane	BISNORHOPANE01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	18a(H)&18b(H)-Oleananes	OLEANANE01		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	18a(H)-30-Norneohopane-C29Ts	NORNEOHPANE01		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	18a-22,29,30-Trisnorneohopane-TS	TRISNORNEOHP01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	1-Methyldibenzothiophene	31317-07-4		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	1-Methylfluorene	1730-37-6		mg/kg	WET	0.5	1.7	
LRB-1 4/8/19	04/11/2019	18:45:09	1-Methylnaphthalene	90-12-0		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	1-Methylphenanthrene	832-69-9		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	2,3,5-Trimethylnaphthalene	2245-38-7		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	2,3,7-Trimethyldibenzothiophene	153524-16-4		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	2,4,6-Trimethyldibenzothiophene	185493-79-7		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	2,4,7/2,4,8-Trimethyldibenzothiophene	216983-03-8 / 1210-52-2		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	2,6,9-Trimethylphenanthrene	66271-32-7		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	2,6-Dimethylnaphthalene	581-42-0		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	2/3-Methyldibenzothiophene	DBZTPM2M3		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	2-Methylanthracene	613-12-7		mg/kg	WET	0.08	0.3	
LRB-1 4/8/19	04/11/2019	18:45:09	2-Methylfluoranthene	33543-31-6		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	2-Methylnaphthalene	91-57-6		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	2-Methylphenanthrene	2531-84-2		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	2-n-Butyldibenzothiophene	147792-31-2		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	2-n-Pentyldibenzothiophene	147793-32-3		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	2-n-Propyldibenzothiophene	147792-30-1		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	3,6-DIMETHYLPHENANTHRENE	1576-67-6		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	30,31-Bishomohopane-22R	BISNORHOPANE03		mg/kg	WET	0.99	3.3	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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 4/8/19	04/11/2019	18:45:09	30,31-Bishomohopane-22S	BISNORHOPANE02		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	30,31-Trishomohopane-22R	TRISHOMOHOP02		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	30,31-Trishomohopane-22S	TRISHOMOHOP01		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	30-Homohopane-22R	60305-22-8		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	30-Homohopane-22S	60305-23-6		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	30-Norhopane	53584-60-4		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	30-Normoretane	3258-87-5		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	3-Methylphenanthrene	832-71-3		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	4,6-Diethyldibenzothiophene	132034-91-4		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	4,6-Dimethyldibenzothiophene	1207-12-1		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	4-Ethyl-6-methyldibenzothiophene	132034-90-3		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	4-Methyldibenzothiophene	7372-88-5		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	4-n-Butyldibenzothiophene	147792-33-4		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	4-n-Pentyldibenzothiophene	147792-34-5		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	4-n-Propyldibenzothiophene	132034-86-7		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	6-Methylchrysene	1705-85-7		mg/kg	WET	0.56	1.9	
LRB-1 4/8/19	04/11/2019	18:45:09	9/4-Methylphenanthrene	4/9-methylphen		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	9-n-Butylphenanthrene	10394-57-7		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	Acenaphthene	83-32-9		mg/kg	WET	0.32	1.1	
LRB-1 4/8/19	04/11/2019	18:45:09	Acenaphthylene	208-96-8		mg/kg	WET	0.23	0.8	
LRB-1 4/8/19	04/11/2019	18:45:09	Anthracene	120-12-7		mg/kg	WET	0.08	0.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Benzo(a)anthracene	56-55-3		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	Benzo(a)pyrene	50-32-8		mg/kg	WET	0.48	1.6	
LRB-1 4/8/19	04/11/2019	18:45:09	Benzo(b)fluoranthene	205-99-2		mg/kg	WET	0.86	2.9	
LRB-1 4/8/19	04/11/2019	18:45:09	Benzo(b)fluorene	243-17-4		mg/kg	WET	0.29	1	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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 4/8/19	04/11/2019	18:45:09	Benzo(b)thiophene	95-15-8		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	Benzo(e)pyrene	192-97-2		mg/kg	WET	0.48	1.6	
LRB-1 4/8/19	04/11/2019	18:45:09	Benzo(g,h,i)perylene	191-24-2		mg/kg	WET	0.38	1.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Benzo[j]fluoranthene/Benzo[k]fluoranthene	BJFBKF		mg/kg	WET	1.03	3.4	
LRB-1 4/8/19	04/11/2019	18:45:09	Biphenyl	92-52-4		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Benzo(b)thiophenes	BZTPHC1		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Chrysenes	BZACHRYSC1		mg/kg	WET	0.56	1.9	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Decalins	DecalinsC1		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Dibenzothiophenes	DBZTPC1		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Fluoranthenes/Pyrenes	FLPYC1		mg/kg	WET	0.3	1	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Fluorenes	FLUORC1		mg/kg	WET	0.5	1.7	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Naphthalenes	NPHC1		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Naphthobenzothiophenes	NBZTPC1		mg/kg	WET	0.26	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	C1-Phenanthrenes/Anthracenes	PHANC1		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	C23 Tricyclic Terpane	C23-TRITERP		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C24 Tetracyclic Terpane	C24-TETRATERP		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C24 Tricyclic Terpane	C24-TRITERP		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C25 Tricyclic Terpane	C25-TRITERP		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C26 Tricyclic Terpane-22R	C26-TRITERP22R		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C26 Tricyclic Terpane-22S	C26-TRITERP22S		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C26,20R- +C27,20S- triaromatic steroid	TRIAROMROID01		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	C27,20R-triaromatic steroid	TRIAROMROID03		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	C28 Tricyclic Terpane-22R	C28-TRITERP22R		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C28 Tricyclic Terpane-22S	C28-TRITERP22S		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C28,20R-triaromatic steroid	TRIAROMROID04		mg/kg	WET	0.29	1	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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 4/8/19	04/11/2019	18:45:09	C28,20S-triaromatic steroid	TRIAROMROID02		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	C29 Tricyclic Terpane-22R	C29-TRITERP22R		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C29 Tricyclic Terpane-22S	C29-TRITERP22S		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Benzo(b)thiophenes	BZTPHC2		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Chrysenes	BZACHRYSC2		mg/kg	WET	0.56	1.9	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Decalins	DecalinsC2		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Dibenzothiophenes	DBZTPC2		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Fluoranthenes/Pyrenes	FLPYC2		mg/kg	WET	0.3	1	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Fluorenes	FLUORC2		mg/kg	WET	0.5	1.7	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Naphthalenes	NPHC2		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Naphthobenzothiophenes	NBZTPC2		mg/kg	WET	0.26	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	C2-Phenanthrenes/Anthracenes	PHANC2		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	C30 Tricyclic Terpane-22R	C30-TRITERP22R		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C30 Tricyclic Terpane-22S	C30-TRITERP22S		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Benzo(b)thiophenes	BZTPHC3		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Chrysenes	BZACHRYSC3		mg/kg	WET	0.56	1.9	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Decalins	DecalinsC3		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Dibenzothiophenes	DBZTPC3		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Fluoranthenes/Pyrenes	FLPYC3		mg/kg	WET	0.3	1	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Fluorenes	FLUORC3		mg/kg	WET	0.5	1.7	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Naphthalenes	NPHC3		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Naphthobenzothiophenes	NBZTPC3		mg/kg	WET	0.26	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	C3-Phenanthrenes/Anthracenes	PHANC3		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	C4-Benzo(b)thiophenes	BZTPHC4		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	C4-Chrysenes	BZACHRYSC4		mg/kg	WET	0.56	1.9	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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 4/8/19	04/11/2019	18:45:09	C4-Decalins	DecalinsC4		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	C4-Dibenzothiophenes	DBZTPC4		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	C4-Fluoranthenes/Pyrenes	FLPYC4		mg/kg	WET	0.3	1	
LRB-1 4/8/19	04/11/2019	18:45:09	C4-Naphthalenes	NPHC4		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	C4-Naphthobenzothiophenes	NBZTPC4		mg/kg	WET	0.26	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	C4-Phenanthrenes/Anthracenes	PHANC4		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	Carbazole	86-74-8		mg/kg	WET	0.57	1.9	
LRB-1 4/8/19	04/11/2019	18:45:09	Chrysene/Triphenylene	CHRYTRIPHEN		mg/kg	WET	0.56	1.9	
LRB-1 4/8/19	04/11/2019	18:45:09	cis/trans Decalin	CTDECALIN		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	Cis-Decalin	493-01-6		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	Dibenzo(a,h)anthracene	53-70-3		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	Dibenzofuran	132-64-9		mg/kg	WET	0.15	0.5	
LRB-1 4/8/19	04/11/2019	18:45:09	Dibenzothiophene	132-65-0		mg/kg	WET	0.44	1.5	
LRB-1 4/8/19	04/11/2019	18:45:09	Fluoranthene	206-44-0		mg/kg	WET	0.29	1	
LRB-1 4/8/19	04/11/2019	18:45:09	Fluorene	86-73-7		mg/kg	WET	0.5	1.7	
LRB-1 4/8/19	04/11/2019	18:45:09	Hopane	13849-96-2		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Indeno(1,2,3-c,d)pyrene	193-39-5		mg/kg	WET	0.33	1.1	
LRB-1 4/8/19	04/11/2019	18:45:09	Moretane	1176-44-9		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Naphthalene	91-20-3		mg/kg	WET	0.6	2	
LRB-1 4/8/19	04/11/2019	18:45:09	Naphthobenzothiophene-1,2-D	205-43-6		mg/kg	WET	0.26	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	Naphthobenzothiophene-2,1-D	239-35-0		mg/kg	WET	0.26	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	Naphthobenzothiophene-2,3-D	243-46-9		mg/kg	WET	0.26	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	Pentakishomohopane-22R	PENTAKSHOMHP02		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Pentakishomohopane-22S	PENTAKSHOMHP01		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Perylene	198-55-0		mg/kg	WET	0.31	1	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY BLANK SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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 4/8/19	04/11/2019	18:45:09	Phenanthrene	85-01-8		mg/kg	WET	0.28	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	Pyrene	129-00-0		mg/kg	WET	0.3	1	
LRB-1 4/8/19	04/11/2019	18:45:09	Retene	483-65-8		mg/kg	WET	0.18	0.6	
LRB-1 4/8/19	04/11/2019	18:45:09	T22a-Gammacerane/C32-diahopane	GAMACERDIAHP01		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Tetrakishomohopane-22R	TETRAKSHOMHP02		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Tetrakishomohopane-22S	TETRAKSHOMHP01		mg/kg	WET	0.99	3.3	
LRB-1 4/8/19	04/11/2019	18:45:09	Total Naphthobenzothiophenes	NAPHTHBZOTHPH		mg/kg	WET	0.26	0.9	
LRB-1 4/8/19	04/11/2019	18:45:09	trans-Decalin	493-02-7		mg/kg	WET	0.66	2.2	
LRB-1 4/8/19	04/11/2019	18:45:09	Unknown Sterane (S18)	UNKSTERANE01		mg/kg	WET	0.29	1	

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY ACCURACY SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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 4/8/19	04/11/2019	19:22:43	Acenaphthene	83-32-9		10.0	10.0	mg/kg	WET	100	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Acenaphthylene	208-96-8		10.0	9.62	mg/kg	WET	96.2	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Anthracene	120-12-7		10.0	7.50	mg/kg	WET	75.0	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Benzo(a)anthracene	56-55-3		10.0	10.9	mg/kg	WET	109	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Benzo(a)pyrene	50-32-8		10.0	10.5	mg/kg	WET	105	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Benzo(b)fluoranthene	205-99-2		10.0	9.44	mg/kg	WET	94.4	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Benzo(g,h,i)perylene	191-24-2		10.0	11.2	mg/kg	WET	112	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Benzo[j]fluoranthene/Benzo[k]fluoranthene	BJFBKF		10.0	10.8	mg/kg	WET	108	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Chrysene/Triphenylene	CHRYTRIPHEN		10.0	10.3	mg/kg	WET	103	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Dibenzo(a,h)anthracene	53-70-3		10.0	8.10	mg/kg	WET	81.0	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Dibenzothiophene	132-65-0		10.0	9.12	mg/kg	WET	91.2	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Fluoranthene	206-44-0		10.0	11.2	mg/kg	WET	112	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Fluorene	86-73-7		10.0	9.02	mg/kg	WET	90.2	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Hopane	13849-96-2		10.0	9.68	mg/kg	WET	96.8	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Indeno(1,2,3-c,d)pyrene	193-39-5		10.0	9.08	mg/kg	WET	90.8	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Naphthalene	91-20-3		10.0	11.0	mg/kg	WET	110	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Naphthobenzothiophene-2,1-D	239-35-0		10.0	7.08	mg/kg	WET	70.8	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Phenanthrene	85-01-8		10.0	9.36	mg/kg	WET	93.6	50	125	
LFB-1 4/8/19	04/11/2019	19:22:43	Pyrene	129-00-0		10.0	11.0	mg/kg	WET	110	50	125	

Comments
Calculations performed prior to rounding.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY ACCURACY SUMMARY

Method: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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
0405191-1 MS	04/11/2019	22:30:26	Acenaphthene	83-32-9		43.8	50.6	mg/kg	WET	116	50	125	
0405191-1 MS	04/11/2019	22:30:26	Acenaphthylene	208-96-8		43.8	33.0	mg/kg	WET	75.4	50	125	
0405191-1 MS	04/11/2019	22:30:26	Anthracene	120-12-7		43.8	54.8	mg/kg	WET	125	50	125	
0405191-1 MS	04/11/2019	22:30:26	Benzo(a)anthracene	56-55-3		43.8	42.8	mg/kg	WET	97.8	50	125	
0405191-1 MS	04/11/2019	22:30:26	Benzo(a)pyrene	50-32-8		43.8	37.1	mg/kg	WET	84.8	50	125	
0405191-1 MS	04/11/2019	22:30:26	Benzo(b)fluoranthene	205-99-2		43.8	39.4	mg/kg	WET	90.0	50	125	
0405191-1 MS	04/11/2019	22:30:26	Benzo(g,h,i)perylene	191-24-2		43.8	55.5	mg/kg	WET	127	50	125	
0405191-1 MS	04/11/2019	22:30:26	Benzo[j]fluoranthene/Benzo[k]fluoranthene	BJFBKF		43.8	47.6	mg/kg	WET	109	50	125	
0405191-1 MS	04/11/2019	22:30:26	Chrysene/Triphenylene	CHRYTRIPHEN		43.8	53.7	mg/kg	WET	123	50	125	
0405191-1 MS	04/11/2019	22:30:26	Dibenzo(a,h)anthracene	53-70-3		43.8	24.8	mg/kg	WET	56.6	50	125	
0405191-1 MS	04/11/2019	22:30:26	Dibenzothiophene	132-65-0	18.5	43.8	45.4	mg/kg	WET	61.5	50	125	
0405191-1 MS	04/11/2019	22:30:26	Fluoranthene	206-44-0	5.76	43.8	68.8	mg/kg	WET	144	50	125	
0405191-1 MS	04/11/2019	22:30:26	Fluorene	86-73-7		43.8	53.7	mg/kg	WET	123	50	125	
0405191-1 MS	04/11/2019	22:30:26	Hopane	13849-96-2	214	43.8	NR	mg/kg	WET	nc	50	125	
0405191-1 MS	04/11/2019	22:30:26	Indeno(1,2,3-c,d)pyrene	193-39-5		43.8	37.0	mg/kg	WET	84.4	50	125	
0405191-1 MS	04/11/2019	22:30:26	Naphthalene	91-20-3	23.8	43.8	74.1	mg/kg	WET	115	50	125	
0405191-1 MS	04/11/2019	22:30:26	Naphthobenzothiophene-2,1-D	239-35-0		43.8	46.9	mg/kg	WET	107	50	125	
0405191-1 MS	04/11/2019	22:30:26	Phenanthrene	85-01-8	40.7	43.8	94.4	mg/kg	WET	123	50	125	
0405191-1 MS	04/11/2019	22:30:26	Pyrene	129-00-0	21.4	43.8	53.6	mg/kg	WET	73.5	50	125	

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: SW8270M
QA/QC Batch Number: QCORG0408191-B
SDG 0405191-B
Project Number: BENB-T02
Report Date: 4/15/2019

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
0405191-1 MSD	04/11/2019	23:45:23	Acenaphthene	83-32-9		42.6	37.8	mg/kg	WET	88.8	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Acenaphthylene	208-96-8		42.6	43.4	mg/kg	WET	102	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Anthracene	120-12-7		42.6	40.5	mg/kg	WET	95.2	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Benzo(a)anthracene	56-55-3		42.6	58.6	mg/kg	WET	138	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Benzo(a)pyrene	50-32-8		42.6	36.0	mg/kg	WET	84.6	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Benzo(b)fluoranthene	205-99-2		42.6	29.6	mg/kg	WET	69.6	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Benzo(g,h,i)perylene	191-24-2		42.6	36.1	mg/kg	WET	84.8	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Benzo[j]fluoranthene/Benzo[k]fluoranthene	BJFBKF		42.6	36.6	mg/kg	WET	86.0	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Chrysene/Triphenylene	CHRYTRIPHEN		42.6	44.0	mg/kg	WET	103	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Dibenzo(a,h)anthracene	53-70-3		42.6	26.6	mg/kg	WET	62.4	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Dibenzothiophene	132-65-0	18.5	42.6	58.4	mg/kg	WET	93.7	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Fluoranthene	206-44-0	5.76	42.6	69.3	mg/kg	WET	149	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Fluorene	86-73-7		42.6	51.3	mg/kg	WET	121	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Hopane	13849-96-2	214	42.6	NR	mg/kg	WET	nc	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Indeno(1,2,3-c,d)pyrene	193-39-5		42.6	31.3	mg/kg	WET	73.6	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Naphthalene	91-20-3	23.8	42.6	61.0	mg/kg	WET	87.5	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Naphthobenzothiophene-2,1-D	239-35-0		42.6	53.4	mg/kg	WET	125	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Phenanthrene	85-01-8	40.7	42.6	82.2	mg/kg	WET	97.7	50	125	
0405191-1 MSD	04/11/2019	23:45:23	Pyrene	129-00-0	21.4	42.6	50.6	mg/kg	WET	68.5	50	125	

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:	SW8270M
QA/QC Batch Number:	QCORG0408191-B
SDG	0405191-B
Project Number:	BENB-T02
Report Date:	4/15/2019

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
0405191-1 MS	04/11/2019	22:30:26	Acenaphthene	83-32-9	50.6		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Acenaphthene	83-32-9	37.8	44.2	mg/kg	WET	29.0	30	
0405191-1 MS	04/11/2019	22:30:26	Acenaphthylene	208-96-8	33.0		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Acenaphthylene	208-96-8	43.4	38.2	mg/kg	WET	27.2	30	
0405191-1 MS	04/11/2019	22:30:26	Anthracene	120-12-7	54.8		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Anthracene	120-12-7	40.5	47.7	mg/kg	WET	30.0	30	
0405191-1 MS	04/11/2019	22:30:26	Benzo(a)anthracene	56-55-3	42.8		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Benzo(a)anthracene	56-55-3	58.6	50.7	mg/kg	WET	31.0	30	
0405191-1 MS	04/11/2019	22:30:26	Benzo(a)pyrene	50-32-8	37.1		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Benzo(a)pyrene	50-32-8	36.0	36.6	mg/kg	WET	3.08	30	
0405191-1 MS	04/11/2019	22:30:26	Benzo(b)fluoranthene	205-99-2	39.4		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Benzo(b)fluoranthene	205-99-2	29.6	34.5	mg/kg	WET	28.4	30	
0405191-1 MS	04/11/2019	22:30:26	Benzo(g,h,i)perylene	191-24-2	55.5		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Benzo(g,h,i)perylene	191-24-2	36.1	45.8	mg/kg	WET	42.4	30	
0405191-1 MS	04/11/2019	22:30:26	Benzo[j]fluoranthene/Benzo[k]fluoranthene	BJFBKF	47.6		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Benzo[j]fluoranthene/Benzo[k]fluoranthene	BJFBKF	36.6	42.1	mg/kg	WET	26.2	30	
0405191-1 MS	04/11/2019	22:30:26	Chrysene/Triphenylene	CHRYTRIPHEN	53.7		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Chrysene/Triphenylene	CHRYTRIPHEN	44.0	48.8	mg/kg	WET	19.8	30	
0405191-1 MS	04/11/2019	22:30:26	Dibenzo(a,h)anthracene	53-70-3	24.8		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Dibenzo(a,h)anthracene	53-70-3	26.6	25.7	mg/kg	WET	6.90	30	
0405191-1 MS	04/11/2019	22:30:26	Dibenzothiophene	132-65-0	45.4		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Dibenzothiophene	132-65-0	58.4	51.9	mg/kg	WET	24.9	30	
0405191-1 MS	04/11/2019	22:30:26	Fluoranthene	206-44-0	68.8		mg/kg	WET			

Comments

Calculations performed prior to rounding.

ANN ARBOR TECHNICAL SERVICES, INC.

QUALITY ASSURANCE / QUALITY CONTROL SUMMARY

LABORATORY PRECISION SUMMARY

Method:	SW8270M
QA/QC Batch Number:	QCORG0408191-B
SDG	0405191-B
Project Number:	BENB-T02
Report Date:	4/15/2019

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
0405191-1 MSD	04/11/2019	23:45:23	Fluoranthene	206-44-0	69.3	69.1	mg/kg	WET	0.652	30	
0405191-1 MS	04/11/2019	22:30:26	Fluorene	86-73-7	53.7		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Fluorene	86-73-7	51.3	52.5	mg/kg	WET	4.49	30	
0405191-1 MS	04/11/2019	22:30:26	Hopane	13849-96-2	221		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Hopane	13849-96-2	225	223	mg/kg	WET	1.95	30	
0405191-1 MS	04/11/2019	22:30:26	Indeno(1,2,3-c,d)pyrene	193-39-5	37.0		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Indeno(1,2,3-c,d)pyrene	193-39-5	31.3	34.1	mg/kg	WET	16.5	30	
0405191-1 MS	04/11/2019	22:30:26	Naphthalene	91-20-3	74.1		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Naphthalene	91-20-3	61.0	67.6	mg/kg	WET	19.3	30	
0405191-1 MS	04/11/2019	22:30:26	Naphthobenzothiophene-2,1-D	239-35-0	46.9		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Naphthobenzothiophene-2,1-D	239-35-0	53.4	50.1	mg/kg	WET	12.8	30	
0405191-1 MS	04/11/2019	22:30:26	Phenanthrene	85-01-8	94.4		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Phenanthrene	85-01-8	82.2	88.3	mg/kg	WET	13.8	30	
0405191-1 MS	04/11/2019	22:30:26	Pyrene	129-00-0	53.6		mg/kg	WET			
0405191-1 MSD	04/11/2019	23:45:23	Pyrene	129-00-0	50.6	52.1	mg/kg	WET	5.83	30	

Comments

Calculations performed prior to rounding.

From:	James E. Taraldsen
To:	Margaret S. Treanor
Subject:	ATS Lab Report 0405191 -- FW: [External] RE: Enbridge Tank 9 Product (49/16-1092)
Date:	Monday, April 15, 2019 2:07:55 PM
Attachments:	SDG 0405191 Level II.pdf image001.png

Here you go.

Jim

James E. Taraldsen

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

From: Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>
Sent: Wednesday, April 10, 2019 3:24 PM
To: Philip Simon <Philip.Simon@annarbortechnicalservices.com>; Alex Smith <alex.smith@enbridge.com>
Cc: ryanerickson04@gmail.com; James E. Taraldsen <JTaraldsen@barr.com>; Peter Simon <Peter.Simon@annarbortechnicalservices.com>; ATS Clerical <ATSClerical@annarbortechnicalservices.com>
Subject: RE: [External] RE: Enbridge Tank 9 Product

Team.

Attached, please find one file consisting of the analytical results from one product sample received by ATS on 4/5/19. Just let us know if you have any questions.

Thanks.

-Sarah-

Sarah Stubblefield | Senior Chemist / Laboratory Manager
Tel: 734.995.0995 | Fax: 734.995.3731 | Cell: 734.368.4730
Email. Sarah.Stubblefield@AnnArborTechnicalServices.com

Ann Arbor Technical Services, Inc.
290 South Wagner Road | Ann Arbor, Michigan 48103
Web. **AnnArborTechnicalServices.com**

From: Philip Simon
Sent: Wednesday, April 10, 2019 4:17 PM
To: Alex Smith <alex.smith@enbridge.com>

Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen <jtaraldsen@barr.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>; Peter Simon <Peter.Simon@annarbortechnicalservices.com>
Subject: RE: [External] RE: Enbridge Tank 9 Product

Good afternoon Alex,

I am reviewing the report now. It should go out within the next15 minutes.

Philip

From: Alex Smith <alex.smith@enbridge.com>
Sent: Wednesday, April 10, 2019 4:06 PM
To: Philip Simon <Philip.Simon@annarbortechnicalservices.com>
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen <jtaraldsen@barr.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>; Peter Simon <Peter.Simon@annarbortechnicalservices.com>
Subject: RE: [External] RE: Enbridge Tank 9 Product

Good Afternoon Philip,

I was curious if you have seen the final results from the pristine and phytane ratios or have the draft report? Sorry to be a pest, trying to direct the project and we are running into some head scratchers.

Thanks for all the help,
Alex

From: Philip Simon [mailto:Philip.Simon@annarbortechnicalservices.com]
Sent: Tuesday, April 09, 2019 2:58 PM
To: Alex Smith
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen; Sarah Stubblefield; Peter Simon
Subject: RE: [External] RE: Enbridge Tank 9 Product

Alex,

The dilutions from last nights were valid and no further running in needed to issue the report, which will go during the day tomorrow.

From the chromatograms we would estimate the crude has a "slight to moderate" degree of weathering. A more quantitative estimate of weathering will come from the pristane and phytane ratios, which will be in the formal report tomorrow.

Phil

From: Alex Smith <alex.smith@enbridge.com>
Sent: Tuesday, April 09, 2019 2:56 PM

To: Philip Simon <Philip.Simon@annarbortechnicalservices.com>
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen <jtaraldsen@barr.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>; Peter Simon <Peter.Simon@annarbortechnicalservices.com>
Subject: RE: [External] RE: Enbridge Tank 9 Product

Good Afternoon Phil,

Thank you for the detailed feedback and yes I would appreciate a update end of today/tomorrow morning on any results observed.

Thanks,

Alex Smith
Environmental Advisor, S&R Environment Operations
—

ENBRIDGE
TEL: 715-395-3836| FAX: 832-325-5511 | CELL: 715-817-8322
119 North 25th Street East, Superior, WI 54880

enbridge.com
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From: Philip Simon [mailto:Philip.Simon@annarbortechnicalservices.com]
Sent: Tuesday, April 09, 2019 1:13 PM
To: Alex Smith
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen; Sarah Stubblefield; Peter Simon
Subject: RE: [External] RE: Enbridge Tank 9 Product

Alex,

Your sample was received Friday morning last week, and was processed immediately. The prep for this analysis takes 48 hours, at a minimum.

This sample was chromatographed last night. We are reviewing these runs now. Assuming the dilutions were right, we should have some feedback for you on the degree of aging by the end of the afternoon today.

If additional dilutions needed, they will be run tonight. Our goal is to have the SHC/TPH report out to you by the close of business tomorrow.

I will get back to you by the end of the afternoon today to see what we can tell you about aging from the chromatograms generated last night.

Phil

Philip B. Simon
President

ANN ARBOR TECHNICAL SERVICES, INC.

From: Alex Smith <alex.smith@enbridge.com>
Sent: Tuesday, April 09, 2019 1:30 PM
To: Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>; Peter Simon <Peter.Simon@annarbortechnicalservices.com>; Philip Simon <Philip.Simon@annarbortechnicalservices.com>
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen <jtaraldsen@barr.com>
Subject: RE: [External] RE: Enbridge Tank 9 Product

Good Afternoon Sarah,

I was curious if ATS had an update on the determining weathering characteristics for the crude oil sample received last week? Or when we could expect initial results?

I always appreciate the full report but an email with initial findings/results would help direct our activities.

Thank you,

Alex Smith
Environmental Advisor, S&R Environment Operations
—

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From: Sarah Stubblefield [mailto:Sarah.Stubblefield@annarbortechnicalservices.com]
Sent: Friday, April 05, 2019 1:07 PM
To: Peter Simon; Alex Smith; Philip Simon
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen
Subject: RE: [External] RE: Enbridge Tank 9 Product

The sample (two VOA's) was received this morning in good condition.

-Sarah_

Sarah Stubblefield | Senior Chemist / Laboratory Manager
Tel: 734.995.0995 | Fax: 734.995.3731 | Cell: 734.368.4730
Email. Sarah.Stubblefield@AnnArborTechnicalServices.com

Ann Arbor Technical Services, Inc.
290 South Wagner Road | Ann Arbor, Michigan 48103
Web. **AnnArborTechnicalServices.com**

From: Peter Simon
Sent: Friday, April 05, 2019 9:56 AM

To: Alex Smith <alex.smith@enbridge.com>; Philip Simon <Philip.Simon@annarbortechnicalservices.com>
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen <jtaraldsen@barr.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>
Subject: Re: [External] RE: Enbridge Tank 9 Product

Great! Thanks Alex.
Peter

Peter SIMON

From: Alex Smith <alex.smith@enbridge.com>
Sent: Friday, April 5, 2019 9:55 AM
To: Peter Simon; Philip Simon
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen; Sarah Stubblefield
Subject: RE: [External] RE: Enbridge Tank 9 Product

Hi Peter,

It can be directly billed to me. Electronic bill works too.

Thanks,

Alex Smith
Environmental Advisor, S&R Environment Operations
—

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119 North 25th Street East, Superior, WI 54880

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From: Peter Simon [mailto:Peter.Simon@annarbortechnicalservices.com]
Sent: Friday, April 05, 2019 7:53 AM
To: Alex Smith; Philip Simon
Cc: ryanerickson04@gmail.com; BARR James E. Taraldsen; Sarah Stubblefield
Subject: Re: [External] RE: Enbridge Tank 9 Product

Alex.

How do you want this billed? Direct to you/Enbridge?

Peter

Peter SIMON

From: Alex Smith <alex.smith@enbridge.com>
Sent: Friday, April 5, 2019 8:15 AM
To: Philip Simon
Cc: Peter Simon; ryanerickson04@gmail.com; BARR James E. Taraldsen; Sarah Stubblefield
Subject: Re: [External] RE: Enbridge Tank 9 Product

Morning Philip,

Sounds great and thanks for the update.

Thanks,

Alex Smith

On Apr 5, 2019, at 7:06 AM, Philip Simon <Philip.Simon@annarbortechnicalservices.com> wrote:

> Peter and Ryan,
>
> I checked with Sarah this morning and we can process this sample as soon as it arrives.
>
> Phil
>
> ---
>
> **From:** Peter Simon <Peter.Simon@annarbortechnicalservices.com>
> **Sent:** Thursday, April 04, 2019 5:15 PM
> **To:** ryanerickson04@gmail.com
> **Cc:** ENB Alex Smith <alex.smith@enbridge.com>; BARR James E. Taraldsen <jtaraldsen@barr.com>; Sarah Stubblefield <Sarah.Stubblefield@annarbortechnicalservices.com>; Philip Simon <Philip.Simon@annarbortechnicalservices.com>
> **Subject:** Re: Enbridge Tank 9 Product
>
> Ryan
>
> I will touch base with the lab in the morning and give you feedback based on the current lab schedule.
>
> Peter
>
> Peter SIMON
>
> ---
>
> **From:** ryanerickson04@gmail.com
> **Sent:** Thursday, April 4, 2019 5:08 PM

To: Peter Simon
Cc: ENB Alex Smith; BARR James E. Taraldsen
Subject: Enbridge Tank 9 Product

Peter,
Another product sample is headed your way and should arrive tomorrow. Looking for same fingerprint analysis as we did on the recent Tank 22 project. ASAP TAT unless Alex corrects me.

Let me know if you have any questions.

Ryan Erickson

Sent from my iPhone

---

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06-Jun-2019

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

Re: **TK24 Head Line (49161374.07)** Work Order: **19060060**

Dear Ryan,

ALS Environmental received 1 sample on 01-Jun-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: 06-Jun-19

Client: Barr Engineering Company
Project: TK24 Head Line (49161374.07)
Work Order: **19060060**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
19060060-01	TK 24 HL-B-1	Soil		5/31/2019 11:35	6/1/2019 09:30	☐

ALS Group, USA

Date: 06-Jun-19

Client: Barr Engineering Company
Project: TK24 Head Line (49161374.07)
WorkOrder: **19060060**

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: TK24 Head Line (49161374.07)
Work Order: 19060060

Case Narrative

Samples for the above noted Work Order were received on 06/01/19. 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: *06-Jun-19*

Client: Barr Engineering Company
Project: TK24 Head Line (49161374.07)
Sample ID: TK 24 HL-B-1
Collection Date: 5/31/2019 11:35 AM

Work Order: 19060060
Lab ID: 19060060-01
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 6/4/19		Analyst: **WH**
1,2,4-Trimethylbenzene	U		33	45	µg/Kg-dry	1	6/5/2019 08:04
1,3,5-Trimethylbenzene	U		53	150	µg/Kg-dry	1	6/5/2019 08:04
Benzene	**260**		**7.8**	**45**	**µg/Kg-dry**	1	6/5/2019 08:04
Ethylbenzene	U		9.6	45	µg/Kg-dry	1	6/5/2019 08:04
m,p-Xylene	U		61	91	µg/Kg-dry	1	6/5/2019 08:04
Naphthalene	U		110	150	µg/Kg-dry	1	6/5/2019 08:04
o-Xylene	U		18	45	µg/Kg-dry	1	6/5/2019 08:04
Toluene	**170**		**12**	**45**	**µg/Kg-dry**	1	6/5/2019 08:04
Xylenes, Total	U		61	140	µg/Kg-dry	1	6/5/2019 08:04
Surr: 1,2-Dichloroethane-d4	*97.0*			*70-130*	*%REC*	1	6/5/2019 08:04
Surr: 4-Bromofluorobenzene	*99.8*			*70-130*	*%REC*	1	6/5/2019 08:04
Surr: Dibromofluoromethane	*96.5*			*70-130*	*%REC*	1	6/5/2019 08:04
Surr: Toluene-d8	*99.4*			*70-130*	*%REC*	1	6/5/2019 08:04
MOISTURE		Method: **SW3550C**					Analyst: **KTP**
Moisture	**25**		**0.10**	**0.10**	**% of sample**	1	6/4/2019 08:39

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

Batch ID: **137069** Instrument ID **VMS8** Method: **SW8260C**

MBLK Sample ID: **MBLK-137069-137069** Units: **µg/Kg-dry** Analysis Date: **6/5/2019 07:48 AM**

Client ID: Run ID: **VMS8_190604B** SeqNo: **5696589** Prep Date: **6/4/2019** 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	*1038*	0	0	*1000*	0	*104*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1002*	0	0	*1000*	0	*100*	*70-130*	0			
Surr: Dibromofluoromethane	*950*	0	0	*1000*	0	*95*	*70-130*	0			
Surr: Toluene-d8	*979.5*	0	0	*1000*	0	*98*	*70-130*	0			

LCS Sample ID: **LCS-137069-137069** Units: **µg/Kg-dry** Analysis Date: **6/5/2019 01:21 PM**

Client ID: Run ID: **VMS8_190604B** SeqNo: **5696594** Prep Date: **6/4/2019** 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	972	22	30	1000	0	97.2	65-135	0			
1,3,5-Trimethylbenzene	888.5	35	100	1000	0	88.8	65-135	0			
Benzene	948.5	5.1	30	1000	0	94.8	75-125	0			
Ethylbenzene	902.5	6.3	30	1000	0	90.2	75-125	0			
m,p-Xylene	1729	40	60	2000	0	86.4	80-125	0			
Naphthalene	835.5	72	100	1000	0	83.6	40-140	0			
o-Xylene	996	12	30	1000	0	99.6	75-125	0			
Toluene	834.5	8.2	30	1000	0	83.4	70-125	0			
Xylenes, Total	2725	40	90	3000	0	90.8	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1006*	0	0	*1000*	0	*101*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1012*	0	0	*1000*	0	*101*	*70-130*	0			
Surr: Dibromofluoromethane	*1040*	0	0	*1000*	0	*104*	*70-130*	0			
Surr: Toluene-d8	*983*	0	0	*1000*	0	*98.3*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 19060060
Project: TK24 Head Line (49161374.07)

QC BATCH REPORT

Batch ID: **137069** Instrument ID **VMS8** Method: **SW8260C**

MS Sample ID: **19060060-01A MS** Units: **µg/Kg-dry** Analysis Date: **6/5/2019 01:38 PM**
Client ID: **TK 24 HL-B-1** Run ID: **VMS8_190604B** SeqNo: **5696595** Prep Date: **6/4/2019** 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	1620	36	49	1650	0	98.2	65-135	0			
1,3,5-Trimethylbenzene	1502	58	160	1650	0	91	65-135	0			
Benzene	1576	8.5	49	1650	256.9	80	75-125	0			
Ethylbenzene	1481	10	49	1650	0	89.8	75-125	0			
m,p-Xylene	2913	66	99	3299	0	88.3	80-125	0			
Naphthalene	1415	120	160	1650	0	85.8	40-140	0			
o-Xylene	1674	19	49	1650	0	102	75-125	0			
Toluene	1414	14	49	1650	166.7	75.6	70-125	0			
Xylenes, Total	4587	66	150	4949	0	92.7	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1617*	0	0	*1650*	0	*98*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1693*	0	0	*1650*	0	*103*	*70-130*	0			
Surr: Dibromofluoromethane	*1659*	0	0	*1650*	0	*101*	*70-130*	0			
Surr: Toluene-d8	*1617*	0	0	*1650*	0	*98*	*70-130*	0			

MSD Sample ID: **19060060-01A MSD** Units: **µg/Kg-dry** Analysis Date: **6/5/2019 01:55 PM**
Client ID: **TK 24 HL-B-1** Run ID: **VMS8_190604B** SeqNo: **5696596** Prep Date: **6/4/2019** 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	1644	37	50	1672	0	98.3	65-135	1620	1.45	30	
1,3,5-Trimethylbenzene	1514	59	170	1672	0	90.6	65-135	1502	0.802	30	
Benzene	1679	8.6	50	1672	256.9	85	75-125	1576	6.3	30	
Ethylbenzene	1490	11	50	1672	0	89.1	75-125	1481	0.57	30	
m,p-Xylene	2947	67	100	3344	0	88.1	80-125	2913	1.15	30	
Naphthalene	1419	120	170	1672	0	84.8	40-140	1415	0.239	30	
o-Xylene	1722	19	50	1672	0	103	75-125	1674	2.82	30	
Toluene	1480	14	50	1672	166.7	78.5	70-125	1414	4.51	30	
Xylenes, Total	4669	67	150	5016	0	93.1	75-125	4587	1.77	30	
Surr: 1,2-Dichloroethane-d4	*1675*	0	0	*1672*	0	*100*	*70-130*	1617	*3.47*	*30*	
Surr: 4-Bromofluorobenzene	*1672*	0	0	*1672*	0	*100*	*70-130*	1693	*1.26*	*30*	
Surr: Dibromofluoromethane	*1685*	0	0	*1672*	0	*101*	*70-130*	1659	*1.55*	*30*	
Surr: Toluene-d8	*1610*	0	0	*1672*	0	*96.3*	*70-130*	1617	*0.397*	*30*	

The following samples were analyzed in this batch: 19060060-01A

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

Client: Barr Engineering Company
Work Order: 19060060
Project: TK24 Head Line (49161374.07)

QC BATCH REPORT

Batch ID: **R261954** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R261954** Units: **% of sample** Analysis Date: **6/4/2019 08:39 AM**
Client ID: Run ID: **MOIST_190604A** SeqNo: **5695540** 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-R261954** Units: **% of sample** Analysis Date: **6/4/2019 08:39 AM**
Client ID: Run ID: **MOIST_190604A** SeqNo: **5695539** 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: **19051983-01B DUP** Units: **% of sample** Analysis Date: **6/4/2019 08:39 AM**
Client ID: Run ID: **MOIST_190604A** SeqNo: **5695518** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	5.97	0.1	0.10	0	0	0	0-0	5.94	0.504	10	

DUP Sample ID: **19060060-01B DUP** Units: **% of sample** Analysis Date: **6/4/2019 08:39 AM**
Client ID: **TK 24 HL-B-1** Run ID: **MOIST_190604A** SeqNo: **5695538** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	24.66	0.1	0.10	0	0	0	0-0	24.93	1.09	10	

The following samples were analyzed in this batch: 19060060-01B

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

19060060

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: 58101
COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr	Company:
Address: 325 S. Lake Ave, Ste 700, Duluth MN 55802	Address: SAME
Name: Ryan Erickson	Name:
email: RErickson@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: TK 24 Head Line	Barr Project No: 49161374.07 006 100

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: PVOCs + Naphalene	Soil: Moisture	Soil: % Solids	Preservative Code	Field Filtered Y/N
										F	A			
										N	N			
1. TK 24 HL-B-1	–	–	–	05/31/2019	11:35	S	N	3		2	1			PVOCs + Naphalene and Moisture.
2.														
3.														
4.														
5.														
6.														
7.														
8.														
9.														
10.														

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

BARR USE ONLY

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

Relinquished by: [signature] — On Ice? Y (0) N — Date: 5/31/19 — Time: 13:30 — Received by: FED EX — Date — Time

Relinquished by: FED EX — On Ice? Y N — Date: 6/1/19 — Time: 0930 — Received by: [signature] — Date — Time

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

Air Bill Number: 68

Lab WO: Temperature on Receipt (°C): SR2 3.0°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: **01-Jun-19 09:30**

Work Order: **19060060** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 03-Jun-19 (Date) Reviewed by: Ehrland Bosworth (eSignature) 03-Jun-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):	3.0/3.0 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	6/3/2019 12:19:14 PM		
Water - VOA vials have zero headspace?	Yes ☐	No ☐	No VOA vials submitted ☑
Water - pH acceptable upon receipt?	Yes ☐	No ☐	N/A ☑
pH adjusted?	Yes ☐	No ☐	N/A ☑
pH adjusted by:	-		

Login Notes:

Client Contacted: Date Contacted: Person Contacted:

Contacted By: Regarding:

Comments:

CorrectiveAction:

Attachment D

Material Management Documentation

Soil Management Documents

Cassidy Potter
Sales Representative
1100 West Gary Street
Duluth, MN 55808

Office: 218.626.3867
Mobile: 218.395.0315
Fax: 218.626.1009
CPotter@VoncoUSA.com

April 10, 2019

Enbridge Energy
Alex Smith
1100 Louisiana Ave Ste 3300
Houston TX, 77002

RE: Profile 19-037-I/Contaminated Soil

Alex,

Please be advised that the above described waste material is acceptable up to **1000 cubic yards** for disposal at the Vonco V Waste Management Campus Facility in Duluth, MN. The waste material is acceptable per Vonco V Duluth, LLC (SW-536) Minnesota Pollution Control Agency approved Industrial Solid Waste Management Plan.

The referenced waste must maintain consistency with what was originally submitted on the waste profile. Vonco V Waste Management Campus must be contacted immediately for any changes in material composition or process generation as further testing and analysis may apply. The term of the approval is 3 years and will expire on 4\5\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: (218)-395-0315.

We look forward to working with you,

Cassidy Potter
Vonco V Duluth, LLC

VONCO V, LLC.

Industrial Waste Profile Sheet

PROFILE# _____

Designated Facility: Vonco V, LLC.

Permit #536

A. Generator, Waste Site Location

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

B. Billing

Name	Enbridge Energy
Site Address	1100 Louisiana Ave, Ste 3300
City, State, Zip	Houston, TX 77002
Contact	Alex Smith
Phone	715-395-3836
Fax	

C. Description of Waste

Name of Waste: Tank 9 Ditch - Soil

Estimated Volume: 1,000 CY

Frequency: One time

Process Generating Waste: Hydrocarbon contaminated soil from project excavation.

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: 4/5/2019 Sample I.D.: TK9-Stockpile-1, TK9-Stockpile-2

F. Generator Certifications

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

Generator's Signature _____ Title: Environmental Advisor

Print Name: Alex Smith Date: 10 April 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 _____

10-Apr-2019

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

Re: **TK9 Response (49161092.07)** Work Order: **1904459**

Dear Jim,

ALS Environmental received 5 samples on 06-Apr-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 17.

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: 10-Apr-19

Client: Barr Engineering Company
Project: TK9 Response (49161092.07)
Work Order: **1904459**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
1904459-01	TK9_Stockpile_1	Soil		4/5/2019 11:30	4/6/2019 10:30	☐
1904459-02	TK9_Stockpile_1	Tclp Extract		4/5/2019 11:30	4/6/2019 10:30	☐
1904459-03	TK9_Stockpile_2	Soil		4/5/2019 11:45	4/6/2019 10:30	☐
1904459-04	TK9_Stockpile_2	Tclp Extract		4/5/2019 11:45	4/6/2019 10:30	☐
1904459-05	Trip Blank	Soil		4/5/2019	4/6/2019 10:30	☐

Client: Barr Engineering Company
Project: TK9 Response (49161092.07)
WorkOrder: **1904459**

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
µg/L	Micrograms per Liter
mg/Kg-dry	Milligrams per Kilogram Dry Weight

Client: Barr Engineering Company
Project: TK9 Response (49161092.07)
Work Order: 1904459

Case Narrative

Samples for the above noted Work Order were received on 04/06/19. 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: *10-Apr-19*

Client: Barr Engineering Company
Project: TK9 Response (49161092.07)
Sample ID: TK9_Stockpile_1
Collection Date: 4/5/2019 11:30 AM

Work Order: 1904459
Lab ID: 1904459-01
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 / 4/8/19		Analyst: **KB**
DRO (C10-C28)	**72**		**0.69**	**6.9**	**mg/Kg-dry**	1	4/10/2019 02:23
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 4/8/19		Analyst: **PM**
Benzene	**1,100**		**9.0**	**53**	**µg/Kg-dry**	1	4/8/2019 16:24
Ethylbenzene	**810**		**11**	**53**	**µg/Kg-dry**	1	4/8/2019 16:24
m,p-Xylene	**4,200**		**25**	**110**	**µg/Kg-dry**	1	4/8/2019 16:24
o-Xylene	**990**		**20**	**53**	**µg/Kg-dry**	1	4/8/2019 16:24
Toluene	**3,000**		**14**	**53**	**µg/Kg-dry**	1	4/8/2019 16:24
Xylenes, Total	**5,200**		**45**	**160**	**µg/Kg-dry**	1	4/8/2019 16:24
Surr: 1,2-Dichloroethane-d4	*94.7*			*70-130*	*%REC*	1	4/8/2019 16:24
Surr: 4-Bromofluorobenzene	*102*			*70-130*	*%REC*	1	4/8/2019 16:24
Surr: Dibromofluoromethane	*91.4*			*70-130*	*%REC*	1	4/8/2019 16:24
Surr: Toluene-d8	*99.8*			*70-130*	*%REC*	1	4/8/2019 16:24
MOISTURE		Method: **SW3550C**					Analyst: **KTP**
Moisture	**34**		**0.10**	**0.10**	**% of sample**	1	4/8/2019 16:37

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

ALS Group, USA

Date: *10-Apr-19*

Client: Barr Engineering Company
Project: TK9 Response (49161092.07)
Sample ID: TK9_Stockpile_1
Collection Date: 4/5/2019 11:30 AM

Work Order: 1904459
Lab ID: 1904459-02
Matrix: TCLP EXTRACT

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**					Analyst: **BG**
Benzene	U		8.4	20	µg/L	20	4/9/2019 15:39
Ethylbenzene	U		5.8	20	µg/L	20	4/9/2019 15:39
m,p-Xylene	**35**	J	**11**	**40**	**µg/L**	20	4/9/2019 15:39
o-Xylene	U		3.8	20	µg/L	20	4/9/2019 15:39
Toluene	**23**		**6.4**	**20**	**µg/L**	20	4/9/2019 15:39
Xylenes, Total	**35**	J	**15**	**60**	**µg/L**	20	4/9/2019 15:39
Surr: 1,2-Dichloroethane-d4	*93.2*			*75-120*	*%REC*	20	4/9/2019 15:39
Surr: 4-Bromofluorobenzene	*99.8*			*80-110*	*%REC*	20	4/9/2019 15:39
Surr: Dibromofluoromethane	*91.1*			*85-115*	*%REC*	20	4/9/2019 15:39
Surr: Toluene-d8	*96.7*			*85-110*	*%REC*	20	4/9/2019 15:39

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

ALS Group, USA

Date: *10-Apr-19*

Client: Barr Engineering Company
Project: TK9 Response (49161092.07)
Sample ID: TK9_Stockpile_2
Collection Date: 4/5/2019 11:45 AM

Work Order: 1904459
Lab ID: 1904459-03
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 / 4/8/19		Analyst: **KB**
DRO (C10-C28)	**140**		**1.2**	**12**	**mg/Kg-dry**	1	4/10/2019 02:52
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 4/8/19		Analyst: **PM**
Benzene	**750**		**23**	**140**	**µg/Kg-dry**	1	4/8/2019 16:08
Ethylbenzene	**500**		**29**	**140**	**µg/Kg-dry**	1	4/8/2019 16:08
m,p-Xylene	**3,000**		**64**	**270**	**µg/Kg-dry**	1	4/8/2019 16:08
o-Xylene	**880**		**52**	**140**	**µg/Kg-dry**	1	4/8/2019 16:08
Toluene	**4,100**		**37**	**140**	**µg/Kg-dry**	1	4/8/2019 16:08
Xylenes, Total	**3,900**		**120**	**410**	**µg/Kg-dry**	1	4/8/2019 16:08
Surr: 1,2-Dichloroethane-d4	*98.5*			*70-130*	*%REC*	1	4/8/2019 16:08
Surr: 4-Bromofluorobenzene	*97.1*			*70-130*	*%REC*	1	4/8/2019 16:08
Surr: Dibromofluoromethane	*89.0*			*70-130*	*%REC*	1	4/8/2019 16:08
Surr: Toluene-d8	*99.0*			*70-130*	*%REC*	1	4/8/2019 16:08
MOISTURE		Method: **SW3550C**					Analyst: **KTP**
Moisture	**64**		**0.10**	**0.10**	**% of sample**	1	4/8/2019 16:37

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

ALS Group, USA

Date: *10-Apr-19*

Client: Barr Engineering Company
Project: TK9 Response (49161092.07)
Sample ID: TK9_Stockpile_2
Collection Date: 4/5/2019 11:45 AM

Work Order: 1904459
Lab ID: 1904459-04
Matrix: TCLP EXTRACT

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**					Analyst: **BG**
Benzene	U		8.4	20	µg/L	20	4/9/2019 16:01
Ethylbenzene	U		5.8	20	µg/L	20	4/9/2019 16:01
m,p-Xylene	**34**	J	**11**	**40**	**µg/L**	20	4/9/2019 16:01
o-Xylene	U		3.8	20	µg/L	20	4/9/2019 16:01
Toluene	**44**		**6.4**	**20**	**µg/L**	20	4/9/2019 16:01
Xylenes, Total	**34**	J	**15**	**60**	**µg/L**	20	4/9/2019 16:01
Surr: 1,2-Dichloroethane-d4	*94.0*			*75-120*	*%REC*	20	4/9/2019 16:01
Surr: 4-Bromofluorobenzene	*98.1*			*80-110*	*%REC*	20	4/9/2019 16:01
Surr: Dibromofluoromethane	*91.6*			*85-115*	*%REC*	20	4/9/2019 16:01
Surr: Toluene-d8	*97.4*			*85-110*	*%REC*	20	4/9/2019 16:01

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

ALS Group, USA

Date: *10-Apr-19*

Client: Barr Engineering Company
Project: TK9 Response (49161092.07)
Sample ID: Trip Blank
Collection Date: 4/5/2019

Work Order: 1904459
Lab ID: 1904459-05
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035 / 4/8/19		Analyst: **PM**
Benzene	U		5.1	30	µg/Kg-dry	1	4/8/2019 15:38
Ethylbenzene	U		6.3	30	µg/Kg-dry	1	4/8/2019 15:38
m,p-Xylene	**38**	J	**14**	**60**	**µg/Kg-dry**	1	4/8/2019 15:38
o-Xylene	U		12	30	µg/Kg-dry	1	4/8/2019 15:38
Toluene	U		8.2	30	µg/Kg-dry	1	4/8/2019 15:38
Xylenes, Total	**38**	J	**26**	**90**	**µg/Kg-dry**	1	4/8/2019 15:38
Surr: 1,2-Dichloroethane-d4	*96.9*			*70-130*	*%REC*	1	4/8/2019 15:38
Surr: 4-Bromofluorobenzene	*99.6*			*70-130*	*%REC*	1	4/8/2019 15:38
Surr: Dibromofluoromethane	*89.4*			*70-130*	*%REC*	1	4/8/2019 15:38
Surr: Toluene-d8	*93.6*			*70-130*	*%REC*	1	4/8/2019 15:38

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

ALS Group, USA

Date: 10-Apr-19

Client: Barr Engineering Company

QC BATCH REPORT

Work Order: 1904459

Project: TK9 Response (49161092.07)

Batch ID: **134272** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK	Sample ID: **DBLKS1-134272-134272**				Units: **mg/Kg**			Analysis Date: **4/10/2019 01:54 AM**			
Client ID:	Run ID: **GC8_190409B**				SeqNo: **5599041**			Prep Date: **4/8/2019**		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: **DLCSS1-134272-134272**				Units: **mg/Kg**			Analysis Date: **4/10/2019 01:25 AM**			
Client ID:	Run ID: **GC8_190409B**				SeqNo: **5599040**			Prep Date: **4/8/2019**		DF: **1**	
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	10.02	0.5	5.0	10	0	100	70-120	0			

LCSD	Sample ID: **DLCSDS1-134272-134272**				Units: **mg/Kg**			Analysis Date: **4/10/2019 03:21 AM**			
Client ID:	Run ID: **GC8_190409B**				SeqNo: **5599049**			Prep Date: **4/8/2019**		DF: **1**	
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	9.562	0.5	5.0	10	0	95.6	70-120	10.02	4.68	20	

The following samples were analyzed in this batch: 1904459-01C 1904459-03C

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

Client: Barr Engineering Company
Work Order: 1904459
Project: TK9 Response (49161092.07)

QC BATCH REPORT

Batch ID: **134275** Instrument ID **VMS9** Method: **SW8260C**

MBLK Sample ID: **MBLK-134275-134275** Units: **µg/Kg-dry** Analysis Date: **4/8/2019 01:19 PM**
Client ID: Run ID: **VMS9_190408A** SeqNo: **5597325** Prep Date: **4/8/2019** 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	*980*	0	0	*1000*	0	*98*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*969*	0	0	*1000*	0	*96.9*	*70-130*	0			
Surr: Dibromofluoromethane	*921*	0	0	*1000*	0	*92.1*	*70-130*	0			
Surr: Toluene-d8	*974*	0	0	*1000*	0	*97.4*	*70-130*	0			

LCS Sample ID: **LCS-134275-134275** Units: **µg/Kg-dry** Analysis Date: **4/8/2019 12:33 PM**
Client ID: Run ID: **VMS9_190408A** SeqNo: **5597320** Prep Date: **4/8/2019** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	893.5	5.1	30	1000	0	89.4	75-125	0			
Ethylbenzene	961	6.3	30	1000	0	96.1	75-125	0			
m,p-Xylene	1744	14	60	2000	0	87.2	80-125	0			
o-Xylene	877	12	30	1000	0	87.7	75-125	0			
Toluene	919.5	8.2	30	1000	0	92	70-125	0			
Xylenes, Total	2621	26	90	3000	0	87.4	75-125	0			
Surr: 1,2-Dichloroethane-d4	*966.5*	0	0	*1000*	0	*96.6*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1050*	0	0	*1000*	0	*105*	*70-130*	0			
Surr: Dibromofluoromethane	*1063*	0	0	*1000*	0	*106*	*70-130*	0			
Surr: Toluene-d8	*1014*	0	0	*1000*	0	*101*	*70-130*	0			

The following samples were analyzed in this batch: 1904459-01A 1904459-03A 1904459-05A

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

Client: Barr Engineering Company
Work Order: 1904459
Project: TK9 Response (49161092.07)

QC BATCH REPORT

Batch ID: **R258138a** Instrument ID **VMS11** Method: **SW8260C**

MBLK Sample ID: **VBLKW1-190409-R258138a** Units: **µg/L** Analysis Date: **4/9/2019 12:40 PM**
Client ID: Run ID: **VMS11_190409A** SeqNo: **5598524** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	U	0.42	1.0								
Ethylbenzene	U	0.29	1.0								
m,p-Xylene	U	0.53	2.0								
o-Xylene	U	0.19	1.0								
Toluene	U	0.32	1.0								
Xylenes, Total	U	0.74	3.0								
Surr: 1,2-Dichloroethane-d4	*18.7*	0	0	*20*	0	*93.5*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*19.51*	0	0	*20*	0	*97.6*	*80-110*	0			
Surr: Dibromofluoromethane	*18.54*	0	0	*20*	0	*92.7*	*85-115*	0			
Surr: Toluene-d8	*19.07*	0	0	*20*	0	*95.4*	*85-110*	0			

LCS Sample ID: **VLCSW1-190409-R258138a** Units: **µg/L** Analysis Date: **4/9/2019 11:34 AM**
Client ID: Run ID: **VMS11_190409A** SeqNo: **5598522** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	20.77	0.42	1.0	20	0	104	85-125	0			
Ethylbenzene	18.84	0.29	1.0	20	0	94.2	76-123	0			
m,p-Xylene	38.15	0.53	2.0	40	0	95.4	75-130	0			
o-Xylene	18.69	0.19	1.0	20	0	93.4	76-127	0			
Toluene	19.38	0.32	1.0	20	0	96.9	76-125	0			
Xylenes, Total	56.84	0.74	3.0	60	0	94.7	76-127	0			
Surr: 1,2-Dichloroethane-d4	*18.94*	0	0	*20*	0	*94.7*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*19.67*	0	0	*20*	0	*98.4*	*80-110*	0			
Surr: Dibromofluoromethane	*20.29*	0	0	*20*	0	*101*	*85-115*	0			
Surr: Toluene-d8	*19.43*	0	0	*20*	0	*97.2*	*85-110*	0			

MS Sample ID: **1904394-01C MS** Units: **µg/L** Analysis Date: **4/9/2019 08:52 PM**
Client ID: Run ID: **VMS11_190409A** SeqNo: **5599193** Prep Date: DF: **100**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1432	42	100	1500	0	95.5	85-125	0			
Ethylbenzene	1272	29	100	1500	0	84.8	76-123	0			
m,p-Xylene	2538	53	200	3000	13	84.2	75-130	0			
o-Xylene	1273	19	100	1500	0	84.9	76-127	0			
Toluene	1318	32	100	1500	0	87.9	76-125	0			
Xylenes, Total	3811	74	300	4500	0	84.7	76-127	0			
Surr: 1,2-Dichloroethane-d4	*1849*	0	0	*2000*	0	*92.4*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*2081*	0	0	*2000*	0	*104*	*80-110*	0			
Surr: Dibromofluoromethane	*2002*	0	0	*2000*	0	*100*	*85-115*	0			
Surr: Toluene-d8	*1956*	0	0	*2000*	0	*97.8*	*85-110*	0			

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

Client: Barr Engineering Company
Work Order: 1904459
Project: TK9 Response (49161092.07)

QC BATCH REPORT

Batch ID: **R258138a** Instrument ID **VMS11** Method: **SW8260C**

MSD Sample ID: **1904394-01C MSD** Units: **µg/L** Analysis Date: **4/9/2019 09:14 PM**
Client ID: Run ID: **VMS11_190409A** SeqNo: **5599194** Prep Date: DF: **100**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1521	42	100	1500	0	101	85-125	1432	6.03	30	
Ethylbenzene	1345	29	100	1500	0	89.7	76-123	1272	5.58	30	
m,p-Xylene	2681	53	200	3000	13	88.9	75-130	2538	5.48	30	
o-Xylene	1351	19	100	1500	0	90.1	76-127	1273	5.95	30	
Toluene	1384	32	100	1500	0	92.3	76-125	1318	4.89	30	
Xylenes, Total	4032	74	300	4500	0	89.6	76-127	3811	5.64	30	
Surr: 1,2-Dichloroethane-d4	*1864*	0	0	*2000*	0	*93.2*	*75-120*	1849	*0.808*	*30*	
Surr: 4-Bromofluorobenzene	*2088*	0	0	*2000*	0	*104*	*80-110*	2081	*0.336*	*30*	
Surr: Dibromofluoromethane	*2001*	0	0	*2000*	0	*100*	*85-115*	2002	*0.05*	*30*	
Surr: Toluene-d8	*1945*	0	0	*2000*	0	*97.2*	*85-110*	1956	*0.564*	*30*	

The following samples were analyzed in this batch: 1904459-02A 1904459-04A

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

Client: Barr Engineering Company
Work Order: 1904459
Project: TK9 Response (49161092.07)

QC BATCH REPORT

Batch ID: **R258122** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R258122** Units: **% of sample** Analysis Date: **4/8/2019 04:37 PM**
Client ID: Run ID: **MOIST_190408B** SeqNo: **5597247** 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-R258122** Units: **% of sample** Analysis Date: **4/8/2019 04:37 PM**
Client ID: Run ID: **MOIST_190408B** SeqNo: **5597245** 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: **1904442-06B DUP** Units: **% of sample** Analysis Date: **4/8/2019 04:37 PM**
Client ID: Run ID: **MOIST_190408B** SeqNo: **5597226** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	37.28	0.1	0.10	0	0	0	0-0	37.79	1.36	10	

DUP Sample ID: **1904459-01B DUP** Units: **% of sample** Analysis Date: **4/8/2019 04:37 PM**
Client ID: **TK9_Stockpile_1** Run ID: **MOIST_190408B** SeqNo: **5597240** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	34.38	0.1	0.10	0	0	0	0-0	34.42	0.116	10	

The following samples were analyzed in this batch: 1904459-01B 1904459-03B

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

1709434

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: 57771
COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company: Same
Address: 325 S Lake Ave	Address:
Name: Ryan Erickson	Name:
email: REricksson@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: TK9 Response	Barr Project No: 49161092.07 003 006

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	Water	Soil: Tared amber 4oz	Soil: amber 4oz	Soil: VOA	Soil: % Solids	Preservative Code	Field Filtered Y/(N)	
										A	A	F	A			
										1	1	1	1			
1. TK9_Stockpile_1	—			4/5/19	1130	S	N	6		1	2	2	1			DRO, BTEX,
2. TK9_Stockpile_2	—			"	1145	S	N	6		1	2	2	1			TCLP BTEX
3. Trip Blank	—			—	—		N	1				1				
4.																Rush TAT
5.																
6.																
7.																
8.																
9.																
10.																

BARR USE ONLY	
Sampled by:	MAB
Barr Proj. Manager:	REE
Barr DQ Manager:	JET
Lab Name:	ALS
Lab Location:	Holland, MI

Relinquished by: [signature] On Ice? (Y) N Date 4/5/19 Time 1400 Received by: FED EX Date Time

Relinquished by: FED EX On Ice? Y N Date 4/6/19 Time 1030 Received by: [signature] Date Time

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

Air Bill Number: 7748 9812 1882

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

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

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

SR2 3.6°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: **06-Apr-19 10:30**

Work Order: **1904459** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 08-Apr-19 (Date) Reviewed by: Ehrland Bosworth (eSignature) 08-Apr-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):	3.6/3.6 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	4/8/2019 1:37:37 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

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

19-037-I Sup Terminal 21st Street					
Date	**Ticket**	**Customer**	**Truck**	**Material**	**Tons**
04/11/2019	308068	001342 - Enbridge Pipelines LLC	T39449X	Contaminated Soil Tons	16.95
04/11/2019	308070	001342 - Enbridge Pipelines LLC	S19589X	Contaminated Soil Tons	12.59
04/11/2019	308074	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	13.70
04/11/2019	308075	001342 - Enbridge Pipelines LLC	T39449X	Contaminated Soil Tons	16.68
04/11/2019	308078	001342 - Enbridge Pipelines LLC	S19589X	Contaminated Soil Tons	14.76
04/11/2019	308080	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	13.70
04/15/2019	308096	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	15.85
04/15/2019	308104	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	16.20
04/15/2019	308106	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	19.40
04/15/2019	308110	001342 - Enbridge Pipelines LLC	S19589X	Contaminated Soil Tons	17.15
04/15/2019	308111	001342 - Enbridge Pipelines LLC	S39858W	Contaminated Soil Tons	18.64
04/15/2019	308114	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	15.70
04/15/2019	308115	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	20.28
04/15/2019	308119	001342 - Enbridge Pipelines LLC	S19589X	Contaminated Soil Tons	17.32
04/15/2019	308120	001342 - Enbridge Pipelines LLC	S39858W	Contaminated Soil Tons	15.79
04/15/2019	308121	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	16.98
04/15/2019	308123	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	18.05
04/15/2019	308129	001342 - Enbridge Pipelines LLC	S19589X	Contaminated Soil Tons	15.96
04/15/2019	308130	001342 - Enbridge Pipelines LLC	S39858W	Contaminated Soil Tons	11.91
04/15/2019	308131	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	17.07
04/15/2019	308132	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	16.96
04/16/2019	308136	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	13.38
04/16/2019	308138	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	15.30
04/16/2019	308139	001342 - Enbridge Pipelines LLC	S36746W	Contaminated Soil Tons	14.70
04/16/2019	308143	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	14.55
04/16/2019	308144	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	15.22
04/16/2019	308145	001342 - Enbridge Pipelines LLC	S36746W	Contaminated Soil Tons	14.71
04/16/2019	308148	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	15.49
04/16/2019	308150	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	15.68
04/16/2019	308152	001342 - Enbridge Pipelines LLC	S36746W	Contaminated Soil Tons	15.63
04/16/2019	308156	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	15.47
04/16/2019	308158	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	13.18

04/16/2019	308159	001342 - Enbridge Pipelines LLC	S36746W	Contaminated Soil Tons	13.87
04/16/2019	308167	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	13.30
04/16/2019	308168	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	12.62
04/16/2019	308171	001342 - Enbridge Pipelines LLC	S36746W	Contaminated Soil Tons	12.86
04/16/2019	308175	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	13.94
04/16/2019	308177	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	14.91
04/16/2019	308178	001342 - Enbridge Pipelines LLC	S36746W	Contaminated Soil Tons	13.24
04/16/2019	308180	001342 - Enbridge Pipelines LLC	T38099W	Contaminated Soil Tons	13.09
04/16/2019	308182	001342 - Enbridge Pipelines LLC	S36747W	Contaminated Soil Tons	14.02
04/17/2019	308183	001342 - Enbridge Pipelines LLC	S36746W	Contaminated Soil Tons	12.02
04/17/2019	308184	001342 - Enbridge Pipelines LLC	S39858W	Contaminated Soil Tons	11.79
04/17/2019	308187	001342 - Enbridge Pipelines LLC	S36746W	Contaminated Soil Tons	13.66
06/17/2019	309933	001342 - Enbridge Pipelines LLC	T39759X	Contaminated Soil Tons	23.36
06/17/2019	309934	001342 - Enbridge Pipelines LLC	T95469W	Contaminated Soil Tons	20.68
06/17/2019	309946	001342 - Enbridge Pipelines LLC	T39759X	Contaminated Soil Tons	18.99
06/17/2019	309947	001342 - Enbridge Pipelines LLC	T95469W	Contaminated Soil Tons	12.70
				Total Tons	**740**
				Total Loads	**48**

Water Management Documents

2626 Courtland Street
Duluth, MN 55806-1894
phone 218.722.3336
fax 218.727.7471
www.wlssd.com

Western Lake Superior Sanitary District

June 18, 2019

Enbridge
Attention: Mr. Alex Smith, Environmental Advisor
119 North 25th Street East
Superior, WI 54880

Re: WLSSD Discharge Approval (**Superior Terminal Tank 24 Header Line Project Excavation Wastewater**)

Dear Mr. Smith:

Based on the analytical information provided on June 4, 2019, the WLSSD approves the discharge of up to **20,000 gallons of Excavation Wastewater from Superior Terminal Tank 24 Header Line Project** provided there is no visiual sign of petroleum oil, grease, or other petroleum related products. This contaminated water is to be disposed of at the WLSSD's main treatment facility which is located at 2626 Courtland Street in Duluth.

This is a one time only approval for the wastewater described. It does not release **Enbridge** from any conditions/regulations set forth by the MPCA and/or any other agency that regulates the waste being discharged. In addition, this approval does not release **Enbridge or any consultant/contractor** involved from any subsequent liabilities associated with conducting this discharge. Wastewater must be transported and disposed by a hauler permitted by WLSSD.

Disposal during a significant rainstorm may be denied because of high flows. A copy of this letter of approval is to accompany each load and is to be disposed of and given to the process control operator. **Please attempt to discharge at our facility between 7:00 a.m. and 5:00 p.m. If you are unable to discharge at that time please call the procees control operator (218) 722-3336 ext. 301 with you estimated time of arrival.**

If there are any questions, please contact me at (218) 740-4814.

Sincerely,

Julie Macor

Julie Macor
Lab Lead Chemist

05-Jun-2019

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

Re: **TK24 Head Line (49161374.07)** Work Order: **19060055**

Dear Ryan,

ALS Environmental received 2 samples on 01-Jun-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 11.

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

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

Sincerely,

Ehrland Bosworth

Electronically approved by: Ehrland Bosworth

Ehrland Bosworth
Project Manager

Report of Laboratory Analysis

Certificate No: WI: 399084510

ALS Group, USA

Date: 05-Jun-19

Client: Barr Engineering Company
Project: TK24 Head Line (49161374.07)
Work Order: **19060055**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
19060055-01	TK24 HL-Frac-1	Wastewater		5/31/2019 11:45	6/1/2019 09:30	☐
19060055-02	Trip Blank	Water		5/31/2019	6/1/2019 09:30	☐

ALS Group, USA

Date: 05-Jun-19

Client: Barr Engineering Company
Project: TK24 Head Line (49161374.07)
WorkOrder: **19060055**

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
µg/L	Micrograms per Liter
mg/L	Milligrams per Liter

Client: Barr Engineering Company
Project: TK24 Head Line (49161374.07)
Work Order: 19060055

Case Narrative

Samples for the above noted Work Order were received on 06/01/19. 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.

ALS Group, USA

Date: *05-Jun-19*

Client: Barr Engineering Company
Project: TK24 Head Line (49161374.07)
Sample ID: TK24 HL-Frac-1
Collection Date: 5/31/2019 11:45 AM

Work Order: 19060055
Lab ID: 19060055-01
Matrix: WASTEWATER

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
DIESEL RANGE ORGANICS BY GC-FID		Method: **PUBL-SW-141**			Prep: PUBL-SW-141 / 6/4/19		Analyst: **DWJ**
DRO (C10-C28)	**6.5**		**0.17**	**1.0**	**mg/L**	10	6/4/2019 15:50
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**					Analyst: **JEB**
Benzene	**82**		**0.46**	**1.0**	**µg/L**	1	6/4/2019 18:38
Ethylbenzene	**6.8**		**0.34**	**1.0**	**µg/L**	1	6/4/2019 18:38
m,p-Xylene	**53**		**0.81**	**2.0**	**µg/L**	1	6/4/2019 18:38
o-Xylene	**29**		**0.31**	**1.0**	**µg/L**	1	6/4/2019 18:38
Toluene	**84**		**0.45**	**1.0**	**µg/L**	1	6/4/2019 18:38
Xylenes, Total	**81**		**0.81**	**3.0**	**µg/L**	1	6/4/2019 18:38
Surr: 1,2-Dichloroethane-d4	*111*			*75-120*	*%REC*	1	6/4/2019 18:38
Surr: 4-Bromofluorobenzene	*97.2*			*80-110*	*%REC*	1	6/4/2019 18:38
Surr: Dibromofluoromethane	*98.7*			*85-115*	*%REC*	1	6/4/2019 18:38
Surr: Toluene-d8	*108*			*85-110*	*%REC*	1	6/4/2019 18:38

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

ALS Group, USA

Date: *05-Jun-19*

Client: Barr Engineering Company
Project: TK24 Head Line (49161374.07)
Sample ID: Trip Blank
Collection Date: 5/31/2019

Work Order: 19060055
Lab ID: 19060055-02
Matrix: WATER

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**					Analyst: **JEB**
Benzene	U		0.46	1.0	µg/L	1	6/4/2019 18:12
Ethylbenzene	U		0.34	1.0	µg/L	1	6/4/2019 18:12
m,p-Xylene	U		0.81	2.0	µg/L	1	6/4/2019 18:12
o-Xylene	U		0.31	1.0	µg/L	1	6/4/2019 18:12
Toluene	U		0.45	1.0	µg/L	1	6/4/2019 18:12
Xylenes, Total	U		0.81	3.0	µg/L	1	6/4/2019 18:12
Surr: 1,2-Dichloroethane-d4	*110*			*75-120*	*%REC*	1	6/4/2019 18:12
Surr: 4-Bromofluorobenzene	*95.4*			*80-110*	*%REC*	1	6/4/2019 18:12
Surr: Dibromofluoromethane	*96.4*			*85-115*	*%REC*	1	6/4/2019 18:12
Surr: Toluene-d8	*107*			*85-110*	*%REC*	1	6/4/2019 18:12

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

ALS Group, USA

Date: 05-Jun-19

Client: Barr Engineering Company

Work Order: 19060055

Project: TK24 Head Line (49161374.07)

QC BATCH REPORT

Batch ID: **137018** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK Sample ID: **DBLKW1-137018-137018** Units: **mg/L** Analysis Date: **6/4/2019 02:22 PM**

Client ID: Run ID: **GC8_190604A** SeqNo: **5694587** Prep Date: **6/4/2019** 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	0.017	0.10								

LCS Sample ID: **DLCSW1-137018-137018** Units: **mg/L** Analysis Date: **6/4/2019 02:51 PM**

Client ID: Run ID: **GC8_190604A** SeqNo: **5694588** Prep Date: **6/4/2019** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	0.0811	0.017	0.10	0.1	0	81.1	75-115	0			J

LCSD Sample ID: **DLCSDW1-137018-137018** Units: **mg/L** Analysis Date: **6/4/2019 04:19 PM**

Client ID: Run ID: **GC8_190604A** SeqNo: **5694591** Prep Date: **6/4/2019** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	0.08668	0.017	0.10	0.1	0	86.7	75-115	0.0811	0	20	J

The following samples were analyzed in this batch: 19060055-01B

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

Client: Barr Engineering Company
Work Order: 19060055
Project: TK24 Head Line (49161374.07)

QC BATCH REPORT

Batch ID: **R261901** Instrument ID **VMS11** Method: **SW8260C**

MBLK Sample ID: **VBLKW1-190604-R261901** Units: **µg/L** Analysis Date: **6/4/2019 02:40 PM**
Client ID: Run ID: **VMS11_190604A** SeqNo: **5695326** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	U	0.46	1.0								
Ethylbenzene	U	0.34	1.0								
m,p-Xylene	U	0.81	2.0								
o-Xylene	U	0.31	1.0								
Toluene	U	0.45	1.0								
Xylenes, Total	U	0.81	3.0								
Surr: 1,2-Dichloroethane-d4	*22.25*	0	0	*20*	0	*111*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*19.61*	0	0	*20*	0	*98*	*80-110*	0			
Surr: Dibromofluoromethane	*20.33*	0	0	*20*	0	*102*	*85-115*	0			
Surr: Toluene-d8	*21.45*	0	0	*20*	0	*107*	*85-110*	0			

LCS Sample ID: **VLCSW1-190604-R261901** Units: **µg/L** Analysis Date: **6/4/2019 01:55 PM**
Client ID: Run ID: **VMS11_190604A** SeqNo: **5695325** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	17.22	0.46	1.0	20	0	86.1	85-125	0			
Ethylbenzene	18.51	0.34	1.0	20	0	92.6	76-123	0			
m,p-Xylene	37.49	0.81	2.0	40	0	93.7	75-130	0			
o-Xylene	18.86	0.31	1.0	20	0	94.3	76-127	0			
Toluene	18.06	0.45	1.0	20	0	90.3	76-125	0			
Xylenes, Total	56.35	0.81	3.0	60	0	93.9	76-127	0			
Surr: 1,2-Dichloroethane-d4	*21.91*	0	0	*20*	0	*110*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*19.56*	0	0	*20*	0	*97.8*	*80-110*	0			
Surr: Dibromofluoromethane	*21.62*	0	0	*20*	0	*108*	*85-115*	0			
Surr: Toluene-d8	*21.21*	0	0	*20*	0	*106*	*85-110*	0			

MS Sample ID: **19051639-02B MS** Units: **µg/L** Analysis Date: **6/4/2019 11:04 PM**
Client ID: Run ID: **VMS11_190604A** SeqNo: **5695332** Prep Date: DF: **10**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	166.2	4.6	10	200	0	83.1	85-125	0			S
Ethylbenzene	186.4	3.4	10	200	0	93.2	76-123	0			
m,p-Xylene	379.7	8.1	20	400	0	94.9	75-130	0			
o-Xylene	189.5	3.1	10	200	0	94.8	76-127	0			
Toluene	177.6	4.5	10	200	0	88.8	76-125	0			
Xylenes, Total	569.2	8.1	30	600	0	94.9	76-127	0			
Surr: 1,2-Dichloroethane-d4	*217*	0	0	*200*	0	*108*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*198.9*	0	0	*200*	0	*99.4*	*80-110*	0			
Surr: Dibromofluoromethane	*205.9*	0	0	*200*	0	*103*	*85-115*	0			
Surr: Toluene-d8	*211.8*	0	0	*200*	0	*106*	*85-110*	0			

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

Client: Barr Engineering Company
Work Order: 19060055
Project: TK24 Head Line (49161374.07)

QC BATCH REPORT

Batch ID: **R261901** Instrument ID **VMS11** Method: **SW8260C**

MSD Sample ID: **19051639-02B MSD** Units: **µg/L** Analysis Date: **6/4/2019 11:26 PM**

Client ID: Run ID: **VMS11_190604A** SeqNo: **5695333** Prep Date: DF: **10**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	169.9	4.6	10	200	0	85	85-125	166.2	2.2	30	S
Ethylbenzene	188.8	3.4	10	200	0	94.4	76-123	186.4	1.28	30	
m,p-Xylene	387.1	8.1	20	400	0	96.8	75-130	379.7	1.93	30	
o-Xylene	195.2	3.1	10	200	0	97.6	76-127	189.5	2.96	30	
Toluene	181.7	4.5	10	200	0	90.8	76-125	177.6	2.28	30	
Xylenes, Total	582.3	8.1	30	600	0	97	76-127	569.2	2.28	30	
Surr: 1,2-Dichloroethane-d4	*216.6*	0	0	*200*	0	*108*	*75-120*	217	*0.185*	*30*	
Surr: 4-Bromofluorobenzene	*194.6*	0	0	*200*	0	*97.3*	*80-110*	198.9	*2.19*	*30*	
Surr: Dibromofluoromethane	*207.6*	0	0	*200*	0	*104*	*85-115*	205.9	*0.822*	*30*	
Surr: Toluene-d8	*213.8*	0	0	*200*	0	*107*	*85-110*	211.8	*0.94*	*30*	

The following samples were analyzed in this batch:

19060055-01A	19060055-02A

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

14060055

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: **58007**

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr	Company:
Address: 325 S. Lake Ave, Ste 700, Duluth MN 55802	Address: SAME
Name: Ryan Erickson	Name:
email: RErickson@barr.com	email:
Copy to: datamgt@barr.com	P.O.
Project Name: TK24 Head Line	Barr Project No: 49161374.07 006 100

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 DRO	Water BTEX	Soil	% Solids	Preservative Code	Field Filtered Y/N
1. TK24 HL-Frac-1	—	—	—	05/31/2019	11:45	WW	N	5	2	3			DRO & BTEX	
2.														
3.														
4.														
5.														
6.														
7.														
8.														
9.														
10. Trip Blank	—	—	—	—	—	—	N	1						

Preservative Code: B B

Field Filtered Y/N: N N

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

BARR USE ONLY

Sampled by: PLL

Barr Proj. Manager: REE

Barr DQ Manager: JET

Lab Name: ALS

Lab Location: Holland, MI

Relinquished by: _____ On Ice? (Y) N Date 5/31/19 Time 13:30 Received by: FED EX Date ____ Time ____

Relinquished by: FED EX On Ice? Y N Date 6/1/19 Time 0930 Received by: _____ Date ____ Time ____

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

Air Bill Number: FB

Lab WO: Temperature on Receipt (°C): SRZ 3.0°C Custody Seal Intact? ☐ Y ☐ N ☐ None

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

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

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: **01-Jun-19 09:30**

Work Order: **19060055** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 03-Jun-19 (Date) Reviewed by: Ehrland Bosworth (eSignature) 03-Jun-19 (Date)

Matrices: Wastewater

Carrier name: FedEx

Shipping container/cooler in good condition?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on shipping container/cooler?	Yes ☐	No ☐	Not Present ☑
Custody seals intact on sample bottles?	Yes ☐	No ☐	Not Present ☑
Chain of custody present?	Yes ☑	No ☐	
Chain of custody signed when relinquished and received?	Yes ☑	No ☐	
Chain of custody agrees with sample labels?	Yes ☑	No ☐	
Samples in proper container/bottle?	Yes ☑	No ☐	
Sample containers intact?	Yes ☑	No ☐	
Sufficient sample volume for indicated test?	Yes ☑	No ☐	
All samples received within holding time?	Yes ☑	No ☐	
Container/Temp Blank temperature in compliance?	Yes ☑	No ☐	
Sample(s) received on ice?	Yes ☑	No ☐	
Temperature(s)/Thermometer(s):	3.0/3.0 c	SR2	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	6/3/2019 12:02:46 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: