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BARR

Technical Memorandum

To: Nick Larabel, Enbridge Energy
From: Ryan Erickson
Subject: Enbridge Terminal - Line 1 PCV Historical Response
WDNR Facility-wide BRRTS #: 01-16-560657
WDNR Site BRRTS #: 02-16-589076 (ENBRIDGE TERMINAL – LINE 1 PCV)
Date: February 24, 2022
Project: 49161092.11 003 001
Site Coordinates: 46.689047°, -92.060601° (NAD83)

This memorandum summarizes the environmental response activities performed by Barr Engineering (Barr) at the request of Enbridge Energy (Enbridge) following the discovery of historical petroleum impacts near the Line 1 Pressure Control Valve (PCV) at the Enbridge Superior Terminal (Terminal) in Superior, Wisconsin (Figure 1).

Background

Between July and September 2021, Enbridge contractors conducted excavation activities in multiple locations in and near the Manifold Corridor Area and Nemadji Corridor for terminal infrastructure projects (Line 93 tie-in, Line 1 project (PCV and Trap); Figure 2). During excavation activities, petroleum-impacted soil and groundwater was encountered. When impacts were encountered, Enbridge personnel inspected the exposed infrastructure and no active releases were identified. Enbridge reviewed historical release documents and confirmed the impacts were in or near areas with previously identified historical petroleum impacts (Manifold Corridor Area, Line 2 MP1098.1, Nemadji Corridor) that had been reported to Wisconsin Department of Natural Resources (WDNR), as documented in the *Continuing Obligation Package* (Barr, 2019) and shown on Figure 2.

Enbridge requested that Barr complete the following activities:

- review the historical release information at the Terminal,
- assist with the characterization and offsite management coordination of impacted soil and water,
- field screen and sample soil from the final excavation extents to document the soil conditions, and
- prepare a memorandum summarizing the response actions and the excavation conditions upon the completion of project activities.

The WDNR was notified of the identification of historical petroleum impacts in the project excavation on July 20, 2021, and the existing Bureau of Remediation & Redevelopment Tracking System (BRRTS) number #02-16-577298 (Manifold Corridor Area) was referenced at the time based on the location of the impacts. Petroleum impacted material was also encountered to the north of the Manifold Corridor Area and in the Nemadji Corridor Area (BRRTS #: 02-16-513788). On January 7, 2022, the site closure pathway was discussed with the WDNR, and it was decided that a new BRRTS number should be set-up for the Line 1 PCV area (north of the Manifold Corridor) and that the associated summary report would also discuss the historical impacts that were encountered and remediated in the adjacent and existing BRRTS areas (Nemadji Corridor, Manifold Corridor). On January 10, 2021, a *Notification For Hazardous Substance Discharge* (Form 4400-225) was submitted to the WDNR and on January 18, 2022, the WDNR provided a

letter response indicating the site was assigned BRRTS # 02-16-589076. Note as stated above, the project area was located within multiple Terminal pipeline and building systems, so the *Line 1 PCV* site name is based on the proximity of that infrastructure to the newly established BRRTS area. Associated Enbridge and WDNR notification communications are provided in Attachment A.

Field Activities

During the summer and fall of 2021, Enbridge contractors performed work on the Line 93 Tie-in project (Photos 1, 2, 11) and the Line 1 projects (Photos 3 through 11; Figure 2). The project teams first encountered impacted material in July 2021. Excavation continued into September of 2021 and work in the excavations was completed in October of that year. The project contractors and inspectors identified and segregated petroleum-impacted soil and water based on visual and olfactory evidence (i.e., discoloration, sheen, crude oil, odor). All excavation activity was conducted with hydrovacuum trucks (hydrovacs) and soil with evidence of petroleum impacts was transported to the Terminal Soil Management Area (SMA) until off-site disposal could occur (Photo 12). Petroleum-impacted water removed from the excavation was pumped into an onsite frac tank until off-site disposal could occur (Photo 11). Additional details on the characterization, management and disposal of the impacted soil and water are provided in the *Material Management* section of this document.

On July 14 and 30, 2021 Barr was on site to observe ongoing project activities and assist with material characterization tasks required for disposal.

On September 10 and 20, 2021 Barr was on site to field screen and sample soil from accessible portions of the completed excavations where historical impacts had been observed to document environmental conditions per the WDNR-approved *Site Investigation and Response Action Plan* (SI/RAP; 2014). Soil samples were field screened for the presence of total organic vapors using a 10.6eV photoionization detector (PID). The samples were also visually assessed for the presence of other potential indicators of petroleum impacts such as odor, discoloration and sheen. The field screening sample locations and results were documented on site investigation field sampling and screening logs (Attachment B). Soil with headspace readings above 10 parts per million (ppm) and/or other evidence of hydrocarbon contamination (e.g., hydrocarbon odor, sheen, the presence of historical crude oil) was classified as impacted.

Analytical soil sample *MAN223-S-1* was collected from the location of previously observed impacts along the Line 93 Tie-in trench. Samples *MAN223-S-2, MAN223-S-3,* and *MAN223-S-4* were collected from the Line 1 Trap excavation in locations exhibiting residual impacts above the screening levels areas, as outlined below. Note that *MAN223* nomenclature was initially used in the sample names based on the assumption that the impacts would be tied to the Manifold Corridor Area BRRTS site. As stated above, the project and new BRRTS site were later associated with the Line 1 PCV for reporting purposes. The samples were submitted to ALS Environmental Laboratory (ALS) in Holland, Michigan for analysis of petroleum volatile organic compounds (PVOCs) and naphthalene. The sampling locations are shown in Figure 2 and Attachment B. The laboratory results are summarized in Table 1 and the laboratory reports are provided in Attachment C.

Results

The following results section is separated into the Line 93 Tie-in and the Line 1 Trap scopes of work.

LINE 93 TIE-IN EXCAVATION

The Line 93 Tie-in excavation was approximately 240 feet long (SW to NE), up to 30 feet wide (SE to NW), and up to 10 feet deep (Photos 1 and 2; Figure 2; Attachment B – Sheet 1). Steel sheet pile was present along most of the excavation sidewalls. Soil encountered in the excavation consisted of construction fill (sand) around buried infrastructure and clay soil away from the infrastructure. No groundwater was observed in the excavation at the time of the field assessment.

Based on communications with the inspector, the soil with petroleum impacts was found in fill around a buried pipeline (8 to 10 feet below ground surface, bgs) near the southeast end of the excavation. The inspector reported that no evidence of residual impacts was identified in this location upon completion of the project activities.

On September 10, 2021, Barr observed field conditions in the final excavation and collected three field screening sidewall soil samples from accessible, non-disturbed areas near the buried pipe where the impacts had been previously observed. No evidence of residual petroleum impacts were identified and that highest soil headspace reading was 0.6 ppm.

Analytical soil sample *MAN223-S-1* was collected from below the pipeline on the northwest side of the excavation. All PVOC + naphthalene analyte concentrations were below the laboratory reporting limits. The analytical results are summarized in Table 1, and the ALS laboratory report is provided in Attachment C.

LINE 1 EXCAVATION

The Line 1 Trap excavation was approximately 170 feet long (SE to NW), up to 70 feet wide (SW to NE), and up to 10 feet deep (Photos 3 through 10; Figure 2; Attachment B – Sheet 2). Soil encountered in the excavation consisted of construction fill around buried infrastructure (sand) and on the ground surface (gravel) and native clay soil away from the infrastructure. The southwestern half of the excavation had also been partially backfilled prior to the field assessment. Groundwater was observed in portions of the excavation at the time of the field assessment at approximately 8 feet bgs. The excavation was dewatered as needed to enable safe access.

Based on communications with the inspector, petroleum-impacted soil and groundwater was encountered in fill around buried infrastructure along the entire length of the excavation. The inspector reported that once the fill was removed, the presence of residual impacts dramatically decreased, including impacts on water in the excavation.

On September 20, 2021, Barr observed field conditions in the final excavation and collected 11 field screening sidewall soil samples from accessible, non-disturbed areas near the location of the previously identified petroleum impacts. Soil headspace readings only exceeded 10 ppm in samples S-3 (16.3 ppm) and S-8 (28.7 ppm). No soil with petroleum odor, sheen or discoloration was identified. An apparent petroleum sheen was observed on water in the north end of the excavation (Photos 9 and 10).

Three analytical soil samples were collected from excavation sidewalls. *MAN223-S-2* and *MAN223-S-4* were collected from the field screening locations with headspace readings greater than 10 ppm, as noted above. *MAN223-S-3* was collected from the northwest end of the excavation where the apparent sheen on groundwater was observed. All PVOC + naphthalene analyte concentrations were below the laboratory reporting limits. The analytical results are summarized in Table 1, and the ALS laboratory report is provided in Attachment C.

Upon completion of the project activities, both excavations were backfilled with clean fill.

Receptor Survey

No direct contact risks were identified based on the field screening and analytical sampling results and the use of clean backfill material. No impacts to surface water were identified and there is little risk of future surface water impacts based on the project remedial actions and the site's location within the Terminal where surface water conditions are monitored for evidence of potential impacts. No groundwater risks were identified based on the analytical sampling results from a review of ongoing facility-wide groundwater monitoring program data. Specifically, 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 the Terminal monitoring well network (shown on Figure 3) on an annual basis and provides the data to the WDNR. The nearest enclosed structures immediately adjacent to the excavation are pipeline operation buildings placed over pressure control valves and have limited human occupancy. The nearest regularly occupied building is the Terminal office building approximately 80 feet to the east. The risk of hazardous vapor accumulation in this structure is low due to the documented remedial efforts and soil conditions, the distance to the structure, and the slab-on-grade construction. Terminal employees are also required to wear four-gas detectors that would alert them to a potentially hazardous atmosphere.

Material Management

During the project excavation activities, soil with evidence of petroleum impacts was transported to the Terminal SMA (Photo 12) and hauled to the landfill once approval was granted. Barr collected soil characterization samples from the stockpile on July 14, July 19, and August 10, 2021, for laboratory analysis at ALS. The samples were analyzed for diesel range organics (DRO) and benzene, toluene, ethyl benzene, and xylenes (BTEX). The laboratory report and waste profile application were submitted to the VONCO V landfill in Duluth, Minnesota and the soil was assigned waste profile #21-065-I. Note that the volume of soil that was approved at the landfill was increased during the project when the additional characterization samples were submitted. A total of 2,720.21 tons of contaminated soil was hauled to the landfill between July 27 and November 5, 2021. The waste profile approval, landfill summary, and waste characterization laboratory reports are included in Attachment D.

Petroleum-impacted water removed from the excavation was pumped into a frac tank until water treatment disposal was granted. Barr collected water characterization sample *Manifold 223-Frac-1* on July 27, 2021, for laboratory analysis at ALS. The sample was analyzed for DRO and BTEX. The laboratory report and a water treatment request were sent to the Western Lake Superior Sanitary District (WLSSD) water treatment facility and the waste stream was approved on August 5, 2021. Approximately 70,400 gallons of water were managed at the treatment facility between August 11 and October 15, 2021. The water

treatment approval letter, disposal tracking ledger, and waste characterization laboratory report are included in Attachment D.

Conclusions

Evidence of historical petroleum impacts were identified during the Line 93 Tie-in and Line 1 projects conducted between July and October 2021. No active release was identified during the projects. Excavated soil with evidence of contamination was managed at a landfill and impacted water was managed a water treatment facility.

Based on the results of field screening and analytical sampling, no historically impacted soil was identified in the final excavations, and clean fill was used to backfill the excavations.

Based on the information in this report, the ongoing Terminal groundwater monitoring program, and Enbridge's environmental response procedures implemented at the facility, there appears to be little risk to potential direct contact, vapor, surface water and groundwater receptors from the impacted material that was removed and the conditions that were identified in the final excavation.

Per Wisconsin Statute NR708.09, Barr recommends that Enbridge submit this memo to the WDNR and request a No Further Response Action determination that states that no further remediation or investigation actions are required at this time. If residual contamination associated with this site is identified in the future, the WDNR will be notified, and site conditions will be documented and reported to the WDNR.

Reference

Barr Engineering Co. 2014, *Site Investigation and Response Action Plan Enbridge, Energy Superior Terminal (Facility-Wide)*. Prepared for Enbridge Energy, July 2014.

Barr, 2019. *Continuing Obligation Package*. Technical report submitted by Barr and Enbridge to the WDNR. July 24, 2019.

Attachments:

Site Photos	1 through 12
Table 1	Soil Analytical Data Summary
Figure 1	Site Location
Figure 2	Site Layout
Figure 3	Receptor Survey
Attachment A	WDNR Reporting Communications
Attachment B	Site Investigation Field Sampling and Screening Logs
Attachment C	Confirmation Soil Sample Laboratory Reports
Attachment D	Material Management Documents

Site Photos

Line 93 tie-in trench

Photo 1

Photo 2

Photo 1: Line 93 tie-in trench. The arrow points to the location where historical petroleum impacts were encountered and removed. Photo taken facing southwest on September 10, 2021.
Photo 2: Location where historical petroleum impacts were encountered and removed and where field screening samples and analytical sample *MAN223-S-1* were collected. Photo taken facing southwest on September 10, 2021.

Line 1 Excavation

Photo 3: Southeast end of final project excavation. The Line 1 trap is shown on the left side of the photo and the Line 2 trap and PCV building are on the right side of the photo. Photo taken facing northwest on September 20, 2021.
Photo 4: Southeast end of final project excavation. Location of analytical samples of *MAN223-S-2* and *MAN223-S-3*. Photo taken facing east on September 20, 2021.

Photo 5: Line 2 PCV building and final project excavation sidewall. Photo taken facing north on September 20, 2021.
Photo 6: Soil with historical petroleum impacts (i.e., sheen, odor) that was subsequently removed (see photo 5) in eastern sidewall immediately southwest of the Line 2 PCV building. Photo taken facing north on July 30, 2021.

Photo 7: Middle portion of the final project excavation. The Line 1 PCV infrastructure is beyond the black equipment rack. Photo taken facing northwest on September 20, 2021.
Photo 8: Northwest end of the final project excavation and the Line 1 PCV area (right side of photo). Photo taken facing northwest on September 20, 2021.

Photo 9

Photo 10

Photo 9: Northeast corner of the final project excavation and Line 1 PCV infrastructure (left side of photo). Photo taken facing northeast on September 20, 2021.
Photo 10: Apparent petroleum impacts on the water surface in the northeast corner of the final project excavation. Photo taken facing northeast on September 20, 2021.

Photo 11

Photo 12

Photo 11: Middle of Line 1 excavation south of Line 2 PCV building (foreground) and Line 93 Tie-in excavation (yellow arrow). Red frac tank used to containerize impacted water removed from project excavations is in the upper left corner. Photo taken facing southwest on September 10, 2021.
Photo 12: Impacted soil stockpile in the Terminal SMA building. Photo taken on July 16, 2021.

Table 1
Soil Analytical Data Summary
Enbridge Terminal - Line 1 PCV
Superior, WI

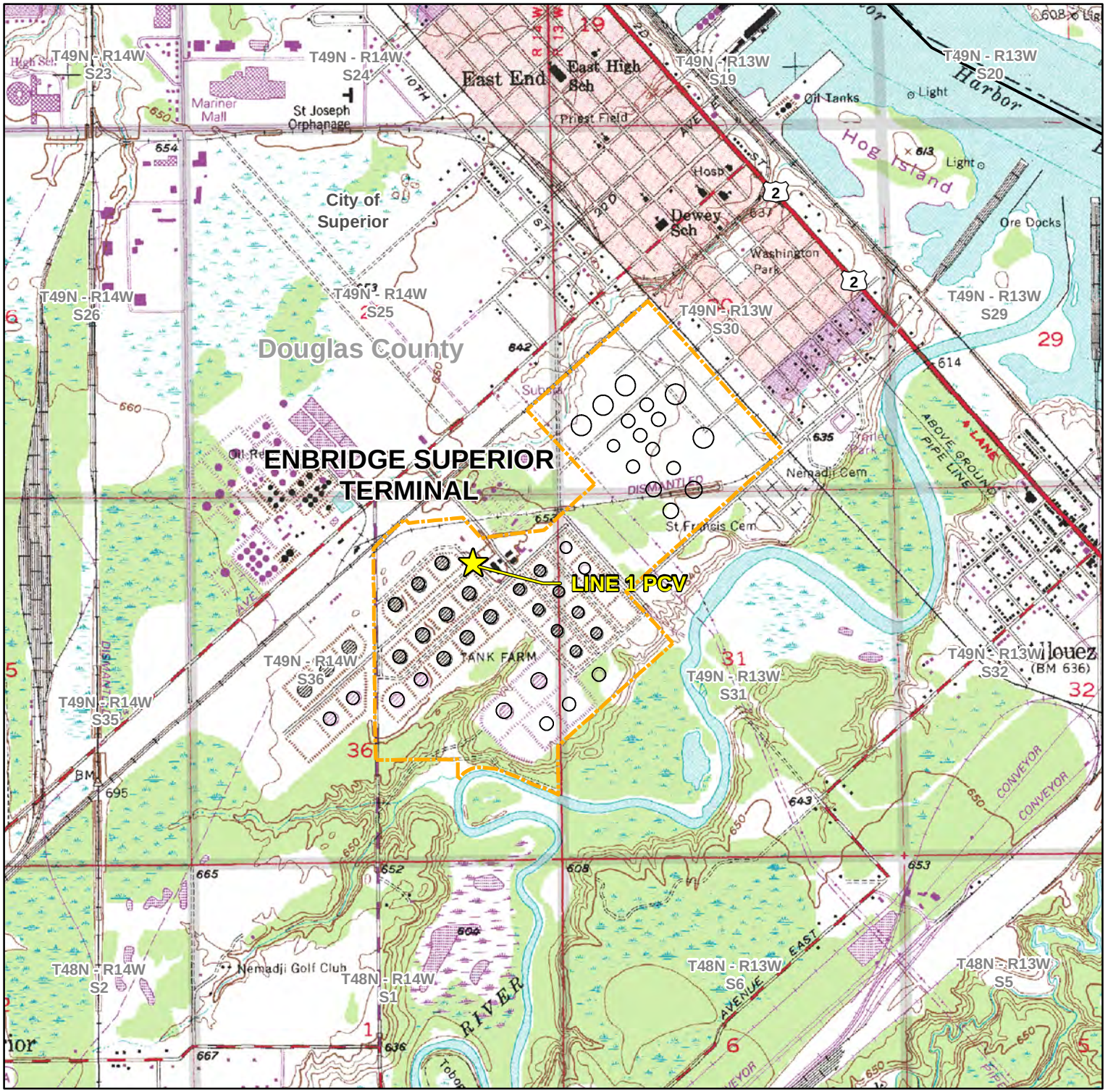
		Location	MAN223-S-1	MAN223-S-2	MAN223-S-3	MAN223-S-4
		Date	9/10/2021	9/20/2021	9/20/2021	9/20/2021
		Depth	10 ft	10 ft	8 ft	10 ft
Parameter	Units	Wisconsin Not to Exceed Direct Contact Industrial RCLs				
Last Updated		12/01/2018				
Exceedance Key		No Exceedances				
General Parameters						
% Moisture	%		18	30	19	27
Volatile Organic Compounds						
1,2,4-Trimethylbenzene	mg/kg	219	< 0.038 U	< 0.04 U	< 0.026 U	< 0.04 U
1,3,5-Trimethylbenzene	mg/kg	182	< 0.13 U	< 0.063 U	< 0.041 U	< 0.064 U
Benzene	mg/kg	7.07	< 0.038 U	< 0.026 U	< 0.017 U	< 0.027 U
Ethyl benzene	mg/kg	35.4	< 0.038 U	< 0.011 U	< 0.0075 U	< 0.012 U
Naphthalene	mg/kg	24.1	< 0.13 U	< 0.13 U	< 0.085 U	< 0.13 U
Toluene	mg/kg	818	< 0.038 U	< 0.015 U	< 0.0097 U	< 0.015 U
Xylene, m & p	mg/kg		< 0.076 U	< 0.072 U	< 0.047 U	< 0.073 U
Xylene, o	mg/kg	434	< 0.038 U	< 0.021 U	< 0.014 U	< 0.021 U
Xylene, total	mg/kg	260	< 0.11 U	< 0.072 U	< 0.047 U	< 0.073 U

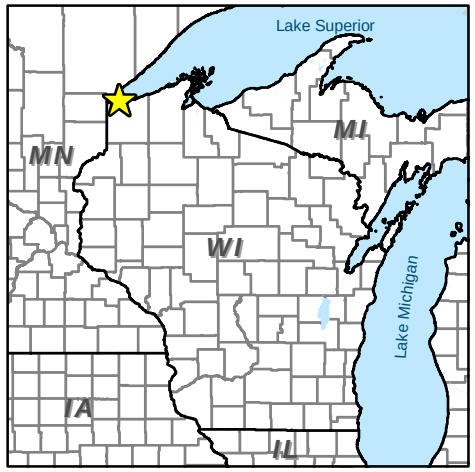
Note:
All values in mg/kg unless otherwise noted

Data Footnotes and Qualifiers

Barr Standard Footnotes and Qualifiers

U	The analyte was analyzed for, but was not detected.





Site Location

Terminal Property Boundary

N

0 2,000 4,000

Feet

1 Inch = 2,000 Feet

Figure 1

SITE LOCATION
LINE 1 PCV
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE

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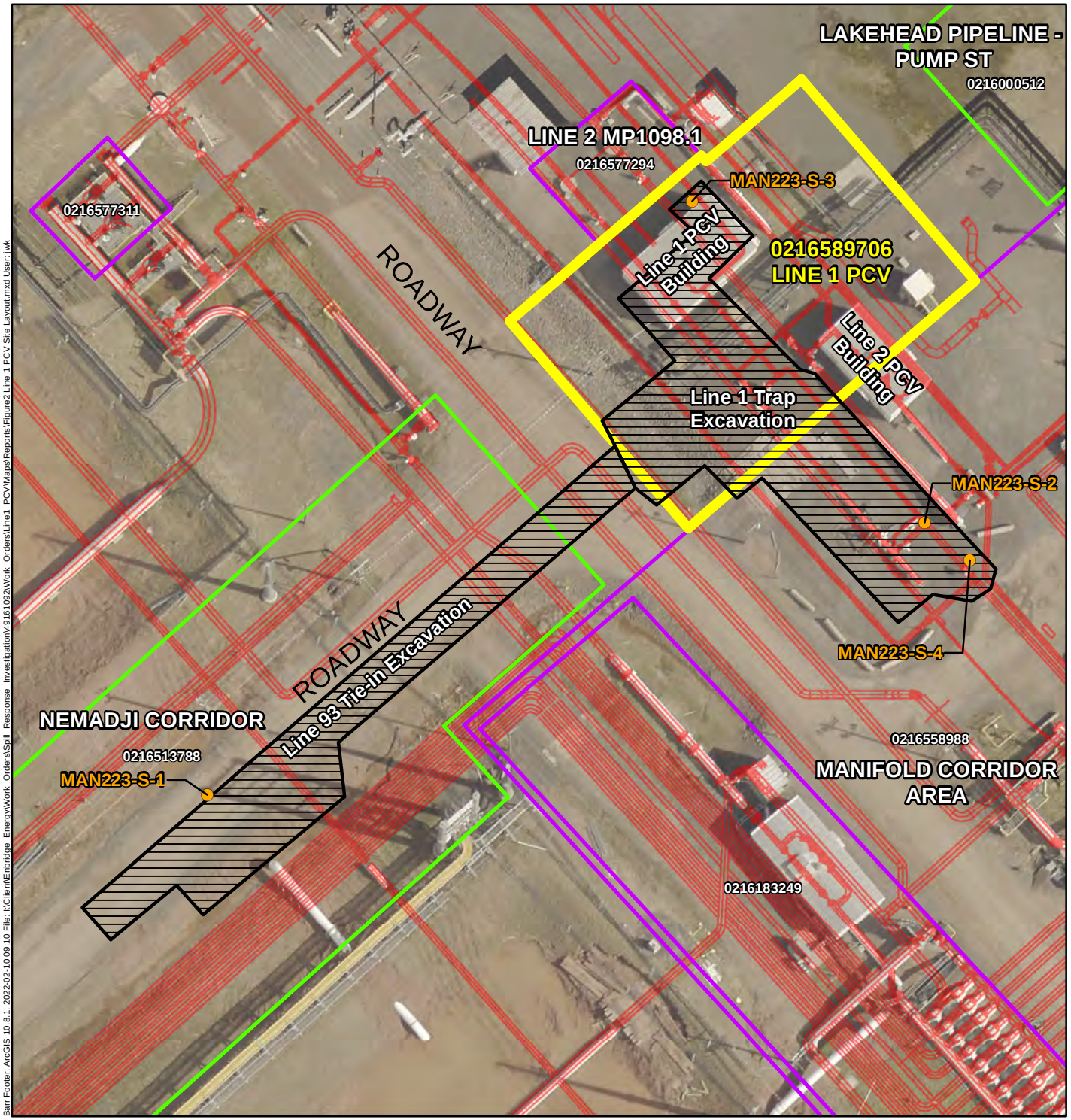
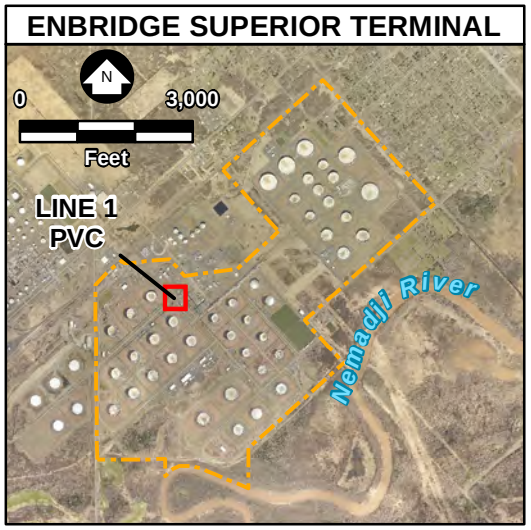




- Analytical Sample Locations
- Excavation Extents
- Proposed Line 1 PCV Area
- Closed-BRRTS Site
- Facility-wide Site
- Pipeline Infrastructure
- Terminal Property Boundary

N

0 50 100

Feet

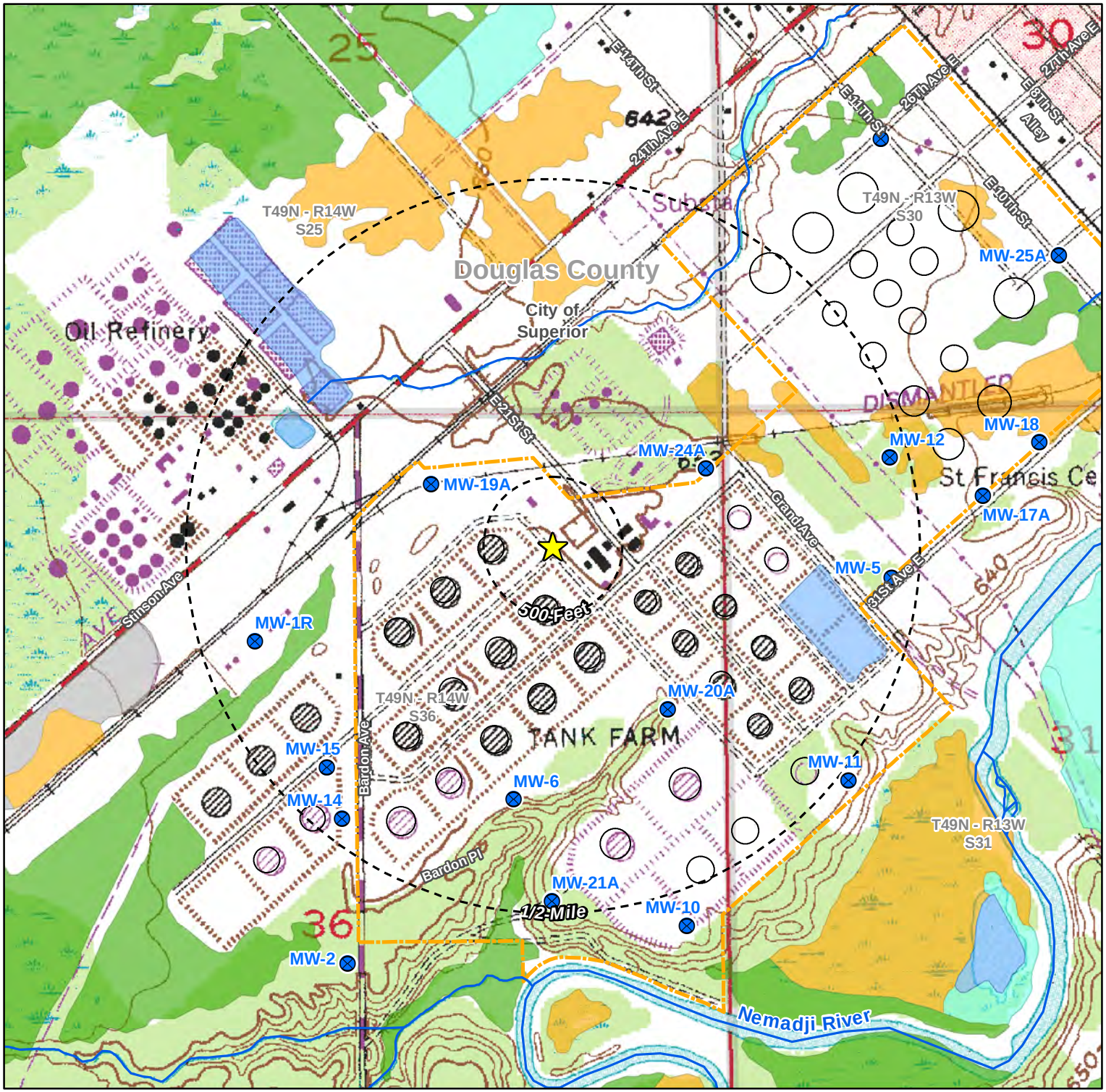
1 Inch = 50 Feet

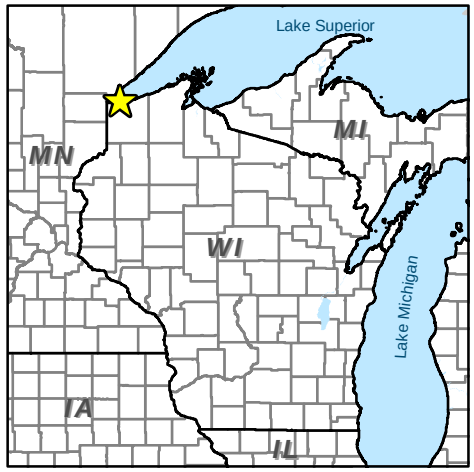
Douglas County Imagery Circa May, 2019

Figure 2

SITE LAYOUT
LINE 1 PCV
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE





- Site Location
- Enbridge Monitoring Well
- Terminal Property Boundary
- Watercourses

Wisconsin Wetland Inventory

- Emergent/wet meadow
- Filled/drained wetland
- Forested
- Open Water
- Scrub/Shrub

N

0 1,000 2,000

Feet

1 Inch = 1,000 Feet

Figure 3

RECEPTOR SURVEY
LINE 1 PCV
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE

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Attachment A

WDNR Reporting Communications

Email Notification (7/20/2021)

4400-225 Notification Form (1/10/2022)

Reported Contamination at Enbridge Energy – Line 1 PCV (1/18/2022)

From: Sager, John E - DNR <John.Sager@wisconsin.gov>
Sent: Tuesday, July 20, 2021 2:12 PM
To: Nick Larabel <nick.larabel@enbridge.com>
Subject: [External] RE: Superior Terminal Manifold Area

Nick,

Thank you for the summary. I will look out for Barr's Report.

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

John Sager
Hydrogeologist – Remediation and Redevelopment Program
Wisconsin Department of Natural Resources
1701 N. 4th St.
Superior, WI 54880
Phone: (715) 919-7239
john.sager@wisconsin.gov

From: Nick Larabel <nick.larabel@enbridge.com>
Sent: Tuesday, July 20, 2021 1:01 PM
To: Sager, John E - DNR <John.Sager@wisconsin.gov>
Subject: Superior Terminal Manifold Area

Hi John,

Late last week, historical residual crude oil impacted soil was identified in the manifold area near the office building (east of Tank 13 and 16) and adjacent to the Nemadji River corridor within the terminal. When reviewing the existing files, this appears to fall within the previously identified manifold area site (BRRTS 02-16-577298). I'm having our consultant, Barr assist with field work and reporting updates. However, no active release was identified in the project excavation, the location of the impacts is within the manifold Facility-wide area with potential residual impacts, impacted material will be removed by the contractor and managed at the Vonco V landfill in Duluth, and Barr will be collecting field screening samples from the final excavation extents (when completed).

Upon project completion, Barr will draft a short memo documenting the assessment and Enbridge's response actions, including providing the necessary ongoing WDNR reporting updates.

I wanted to send this out to let you know, but please let me know if you have any questions in the meantime.

Thanks,

Nick

Nicholas B. Larabel, PG, CPG
Environment Advisor, LP US Environment Operations
—
ENBRIDGE
TEL: 269-330-3872
455 Leggitt Road, Marshall, MI 49068

State of Wisconsin
Department of Natural Resources
PO Box 7921, Madison WI 53707-7921
dnr.wi.gov

Notification For Hazardous Substance Discharge (Non-Emergency Only)

Form 4400-225 (R 05/21)

Emergency Discharges / Spills should be reported via the 24-Hour Hotline: 1-800-943-0003

Notice: Hazardous substance discharges must be reported immediately according to s. 292.11 Wis. Stats. Non-emergency hazardous substance discharges may be reported by telefaxing or e-mailing a completed report to the Department, or calling or visiting a Department office in person. If you choose to notify the Department by telefax or by email, you should use this form to be sure that all necessary information is included. However, use of this form is not mandatory. Under s. 292.99, Wis. Stats., the penalty for violating the reporting requirements of ch. 292 Wis. Stats., shall be no less than $10 nor more than $5000 for each violation. Each day of continued violation is a separate offense. It is not the Department's intention to use any personally identifiable information from this form for any purpose other than program administration. However, information submitted on this form may also be made available to requesters under Wisconsin's Public Records Law (ss. 19.31 – 19.39, Wis. Stats.).

Confirmatory laboratory data should be included with this form, to assist the DNR in processing this Hazardous Substance Release Notification.

Complete this form. **TYPE or PRINT LEGIBLY.** NOTIFY appropriate DNR region (see next page) **IMMEDIATELY** upon discovery of a potential release from **(check one)**:

- ◯ Underground Petroleum Storage Tank System (additional information may be required for Item 6 below)
- ◯ Aboveground Petroleum Storage Tank System
- ◯ Dry Cleaner Facility
- ◉ Other - Describe: Pipeline Terminal

ATTN DNR: **R & R Program Associate**

Date DNR Notified: 07/20/2021

1. Discharge Reported By

Name	Firm	Phone Number (include area code)
Karl Beaster	Enbridge Energy	(218) 464-5623

Mailing Address	Email
11 East Superior Street, Suite 125, Duluth, MN 55802	karl.beaster@enbridge.com

2. Site Information

Name of site at which discharge occurred. Include local name of site/business, not responsible party name, unless a residence/vacant property.

ENBRIDGE TERMINAL - LINE 1 PCV

Location: Include street address, not PO Box. If no street address, describe as precisely as possible, i.e., 1/4 mile NW of CTHs 60 & 123 on E side of CTH 60.

2800 East 21st Street, Superior, WI 54880

Municipality: (City, Village, Township) Specify municipality in which the site is located, not mailing address/city.

Superior, WI

County	Legal Description:	WTM:
Douglas	NE ¼ of NE ¼ Section 36 , Town 49 N, Range 14 ◯ E ◉ W	X 362440 Y 692664

3. Responsible Party (RP) and/or RP Representative

Responsible Party Name: Business or owner name that is responsible for cleanup. If more than one, list all. Attach additional pages as necessary.

Enbridge Energy

☐ A local governmental unit claiming an exemption from state Spill Law and Solid Waste Management responsibilities for the discharge being reported, per Wis. Stat. §§ 292.11(9)(e) and 292.23, should: 1) check this box; 2) review DNR publication RR-055; and 3) provide documentation to DNR that demonstrates compliance with the statutory requirements of the liability exemptions. Local governmental units may also request a fee-based liability clarification letter from DNR by using DNR Form 4400-237.

Contact Person Name (if different)	Phone Number	Email
Enbridge - Karl Beaster	(218) 464-5623	karl.beaster@enbridge.com

Mailing Address	City	State	ZIP Code

Responsible Party Name: Business or owner name that is responsible for cleanup. If more than one, list all. Attach additional pages as necessary.

Contact Person Name (if different)	Phone Number	Email

Mailing Address	City	State	ZIP Code

(continued)

4. Hazardous Substance Information

Identify hazardous substance discharged (check all that apply):

- ☐ VOCs
  - ☐ PCE
  - ☐ TCE
  - ☐ Other Chlorinated
  - ☐ Diesel
  - ☐ Fuel Oil
  - ☐ Gasoline
  - ☐ Hydraulic Oil
  - ☐ Jet Fuel
- *(VOCs continued)*
  - ☐ Mineral Oil
  - ☐ Waste Oil
  - ☐ Petroleum-Unknown Type
  - ☐ 1,4-dioxane
- ☐ PAHs
- ☐ PCBs
- ☐ Cyanide
- ☐ Leachate
- ☐ Manure
- ☐ Metals
  - ☐ Arsenic
  - ☐ Chromium
  - ☐ Lead
  - ☐ Other: _____
- ☐ Pesticides: _____
- ☐ Fertilizer: _____
- ☐ RCRA Hazardous Waste: _____
- ☒ Other: Crude oil - historical
- ☐ Unknown

5. Impacts to the Environment Information

Enter "K" for known/confirmed or "P" for potential for all that apply.

- ____ Air Contamination
- ____ Co-mingled (Petroleum & Non-Petroleum)
- ____ Contamination in Fractured Bedrock
- ____ Contamination Within 1 Meter of Bedrock
- ____ Contaminated Private Well
- ____ Contaminated Public Well
- ____ Contamination in Right of Way
- ____ Fire Explosion Threat
- ____ Free Product
- K Groundwater Contamination
- ____ Off-Site Contamination
- ____ Sanitary Sewer Contamination
- ____ Storm Sewer Contamination
- ____ Sediment Contamination
- K Soil Contamination
- ____ Soil Gas Contamination
- ____ Sub-slab Vapor Contamination
- ____ Surface Water Contamination
- ____ Within 100 ft of Private Well
- ____ Within 1000 ft of Public Well

Other (specify): _____

Contamination was discovered as a result of:

☐ Tank closure assessment Date _____

☐ Site assessment Date _____

☒ Other - Describe: Infrastructure maintenance excavation Date 07/19/2021

Lab results: ☐ Lab results will be faxed upon receipt ☐ Lab results are attached

Additional Comments: Include a brief description of immediate actions taken to halt the release and contain or cleanup hazardous substances that have been discharged.

Enbridge encountered petroleum impacted soil and water during project excavation activity. No active release was identified therefore the impacts were interpreted to be historical and the WDNR was notified of the discovery (email). Soil around the infrastructure was removed and no evidence of contamination was identified in the final excavation through soil field screening and analytical sampling. All water with a sheen and soil with evidence contamination removed from the excavation has been disposed of at off-site facilities.

6. Federal Energy Act Requirements (Section 9002(d) of the Solid Waste Disposal Act (SWDA))

For all confirmed releases from USTs occurring after 9/30/2007 please provide the following information:

☒ Does not apply.

Source

- ☐ Tank
- ☐ Piping
- ☐ Dispenser
- ☐ Submersible Turbine Pump
- ☐ Delivery Problem
- ☐ Other (specify): _____

Cause

- ☐ Spill
- ☐ Overfill
- ☐ Corrosion
- ☐ Physical or Mechanical Damage
- ☐ Installation Problem
- ☐ Other (does not fit any of above)
- ☐ Unknown

Submit this completed form along with any associate lab results using the RR Program Submittal Portal, found on the DNR website at https://dnr.wisconsin.gov/topic/Brownfields/Submittal.html.

If you have any questions, please contact the appropriate regional Environmental Program Associate (EPA) listed under the "EPAs" tab at https://dnr.wisconsin.gov/topic/Brownfields/Contact.html.

State of Wisconsin
DEPARTMENT OF NATURAL RESOURCES
1701 North 4th Street
Superior WI 54880

Tony Evers, Governor
Preston D. Cole, Secretary
Telephone 608-266-2621
Toll Free 1-888-936-7463
TTY Access via relay - 711

January 18, 2022

Karl Beaster
Enbridge Energy
11 East Superior Street, Suite 125
Duluth, MN 55802
(sent via email only to karl.beaster@enbridge.com)

Subject: Reported Contamination at Enbridge Energy – Line 1 PCV,
2800 East 21st Street, Superior, WI 54880, WI
DNR BRRTS Activity # 02-16-589076
DNR FID # 816010580

Dear Mr. Beaster:

On , July 20, 2021 Enbridge Energy notified the Wisconsin Department of Natural Resources (DNR) that a hazardous substance discharge was detected at the site described above.

Information submitted to the DNR regarding this site indicates you are responsible for the discharge of a hazardous substance or other environmental pollution (hereafter referred to as "contamination") at the above-described site. "Site" refers to the property where the contamination occurred and any other property it has migrated to, as defined in Wisconsin Administrative Code ("Wis. Admin. Code") § NR 700.03 (56).

This letter explains how to initiate the investigation and cleanup of contamination of the site, and how to access further information and assistance from the DNR. The longer contamination is left in the environment, the farther it can spread and the more it may cost to clean up. Quick action may lessen damage to your property and neighboring properties and reduce your costs to investigate and clean up the contamination.

Legal Responsibilities:

Persons meeting the definition of "responsible party" under Wis. Admin. Code § NR 700.03 (51) must follow applicable law to address the discharge of a hazardous substance to the environment or other environmental pollution. Wisconsin Statutes ("Wis. Stat.") ch. 292 and Wis. Admin. Code chs. NR 700-799 provide specific requirements for undertaking appropriate response actions to address contamination, including requirements for emergency and interim actions, public information, site investigations, remedy selection, design and operation of remedial action systems, and case closure.

Special Vapor Intrusion Concern with Trichloroethylene:

Contamination that includes trichloroethylene ("TCE"), a chlorinated solvent and common degreaser, is of special concern from a human health perspective due to its potential for acute (short-term) health risks at relatively low concentrations in air. TCE is also a breakdown product of tetrachloroethylene ("PCE," also known as "Perc"), a historically common dry-cleaning chemical. Vapors can travel from contaminated soil or groundwater and along preferential pathways, such as within sewer lines, and enter occupied buildings. This is known as vapor intrusion (VI). Screening for VI must be conducted at every contaminated site in Wisconsin, as defined in Wis. Admin.

Code § 716.11 (5) (a). **However, when TCE is present, screening for VI should be made a priority and an interim action under Wis. Admin. Code § NR 708.11 may be necessary.** For an overview on VI, see *What is Vapor Intrusion?* (RR-892). For more information, go to dnr.wi.gov and search "vapor." Additional technical guidance on VI is available in *Addressing Vapor Intrusion at Remediation & Redevelopment Sites in Wisconsin*, (RR-800).

General Recommendations for Responsible Parties:

The DNR recommends that you:

1. Hire a Qualified Environmental Consultant

To ensure response actions you plan to undertake comply with Wisconsin law, you should hire an environmental consultant within **30 days,** by February 17, 2022, to meet the regulatory deadlines listed below. A delay in hiring an environmental consultant could result in you missing key submittal deadlines.

Hiring a consulting firm with staff that have the appropriate state of Wisconsin qualifications to supervise and certify the submittals is a critical component and necessary to meet your requirements. Further, an environmental consultant should be knowledgeable of Wisconsin's technical procedures and laws, and be able to answer questions regarding cleanup requirements. Required qualifications for environmental consultants are specified in Wis. Admin. Code ch. NR 712. See *Wis. Admin. Code ch. NR 712 Qualifications and Certifications* (RR-081), for more information.

2. Properly Submit Reports on Time with Required Information Included

Wisconsin law includes timeframes for submitting technical documents and conducting work, as well as specifications for what should be included in those submittals. This letter provides a general overview of the timeframes and first steps to take for site investigation and cleanup. For an overview of timing requirements, please refer to *NR 700 Process and Timeline Overview* (RR-967), *enclosed.*

The DNR developed the publication *Guidance for Electronic Submittals for the Remediation and Redevelopment Program* (RR-690), to assist responsible parties and consultants in properly submitting documents. Wis. Admin. Code § NR 700.11 (3g), and other specific provisions within Wis. Admin. Code ch. NR 700, outline the requirements for submittals, including electronic submittals.

3. Consider the Benefits of a Fee-based Technical Review of your Submittals

In-depth DNR review of technical reports and submittals is available for a fee. The Remediation and Redevelopment (RR) Program project managers are available throughout the process to answer general questions and provide general input as the site moves toward case closure. However, if you want a formal, written response from the DNR, a meeting with the DNR or both on a specific submittal, a review fee will be required in accordance with Wis. Admin. Code ch. NR 749. **Obtaining technical assistance from DNR project managers throughout the process is an effective way to prevent problems and delays at the end of the process when case closure is requested.** Forms, a fee schedule and further information on technical assistance is available at dnr.wi.gov by searching "brownfield fees."

Required Steps to Take and Documents to Submit:

The steps listed below serve as a general overview only — all mandatory steps and submittals specified in Wis. Admin. Code, chs. NR 700-799 must be met before the DNR can grant case closure, which is a determination by the DNR that no further cleanup is necessary at a site, as defined in Wis. Admin. Code § NR 700.03 (3m).

1. **Immediate Actions – NR 708.09**: The law requires you to take any immediate actions needed to halt and minimize harmful effects, unless you are otherwise directed by the DNR, and to submit documentation describing immediate actions and outcomes within 45 days after the initial hazardous substance discharge notification is given to the DNR. A final immediate action report should be submitted in accordance with Wis. Admin. Code § NR 708.09.

2. **Scoping and Work Plan Submittal – NR 716.07 and 716.09**: The law requires that you appropriately scope your site investigation and submit a work plan within **60 days of this notification,** by March 19, 2022, for completing a site investigation. The work plan must comply with the requirements in Wis. Admin. Code, chs. NR 700-799. For additional assistance, the DNR has extensive guidance on its website at dnr.wi.gov, search "site investigation scoping."

   Per Wis. Admin. Code § NR 716.07 and Wis. Admin. Code § NR 716.09, site investigation scoping and work plans should include an evaluation of the history of the site or facility, including industrial, commercial or other land uses that may have been associated with one or more hazardous substance discharges at the facility. In addition, an evaluation of the history of previous hazardous substance discharges or environmental pollution, the location of the site or facility, and its proximity to other sources of contamination must be included. Site investigation work plans should also include a sampling and analysis strategy to be used during field investigation that considers all information in the evaluation conducted under Wis. Admin. Code § NR 716.07. Emerging contaminants discharged to the environment, including perfluoroalkyl and polyfluoroalkyl substances (PFAS) and 1,4-dioxane, meet the definition of a hazardous substance or environmental pollution under Wis. Stat. § 292.01 and must be considered during site investigation scoping.

   Prior to and during a site investigation, you must evaluate whether any interim actions are needed to contain or stabilize a hazardous substance discharge or environmental pollution, pursuant to Wis. Admin. Code § NR 708.11. If you undertake an interim action (*e.g.*, free product removal), you must submit documentation of the action per Wis. Admin. Code § NR 708.15.

   As you develop the site investigation work plan, you must include an assessment of the vapor intrusion pathway. Wis. Admin. Code § NR 716.11 (5) outlines the requirements for when to evaluate for the presence of vapors in the sub-surface and in indoor air. The results and conclusions from the vapor assessment must be included in the Wis. Admin. Code § NR 716.15 site investigation report whether or not you elected to take vapor samples. *Addressing Vapor Intrusion at Remediation & Redevelopment Sites in Wisconsin* (RR-800), is available to help responsible parties and their consultants comply with these requirements.

3. **Field Investigation – NR 716.11**: Following submission of the work plan, the site investigation must be started within the timeframe provided under law. The timeframe varies depending on whether you are requesting the DNR's fee-based review of the work plan. If you do not request a fee-based review of the work plan, you must initiate the field investigation within 90 days of submitting the work plan, and you may proceed with the field investigation upon DNR notification to proceed; however, if the DNR has not responded within 30 days from submittal of the work plan, you may then proceed with the field investigation. If a fee and request for DNR review of the work plan is submitted, the field investigation must begin within 60 days after receiving DNR approval.

4. **Sample Results Notification Requirements – NR 716.14**: You must report sampling results to the DNR, owners, occupants and various other parties within 10 business days after receiving the sampling results, unless a different timeframe is approved by the DNR, in accordance with Wis. Admin. Code § NR 716.14.

5. **Site Investigation Report – NR 716.15**: Within 60 days after completion of the field investigation and receipt of the laboratory data, the law requires you to submit a Site Investigation Report (SIR) to the DNR. As

part of the SIR or in the Remedial Actions Options Report (RAOR), if there is soil contamination, the responsible party shall identify the current land use (*i.e.*, industrial or non-industrial) and zoning for the site or facility in accordance with Wis. Admin. Code § NR 720.05 (5). Also, as part of the SIR or in the RAOR, you must include any interim action report that may be required under Wis. Admin. Code § NR 708.15.

6. **Remedial Actions Options Report – NR 722**: Within 60 days after submitting the SIR, the law requires you to submit a RAOR. The selected remedy in the RAOR should include an evaluation of green and sustainable remediation criteria, as appropriate, as required by Wis. Admin. Code § NR 722.09 (2m). This may be submitted as part of a broader SIR.

7. **Remedial and Interim Action Design, Implementation, Operation, Maintenance and Monitoring Reports – NR 724**: Unless otherwise directed by the DNR, the responsible party shall submit all plans and reports required by Wis. Admin. Code ch. NR 724.

8. **Notification of Residual Contamination or Continuing Obligations – NR 725**: In situations where notification is required, the responsible party must provide a submittal(s) that confirms that continuing obligations have been identified and affected property owners have been notified by the responsible parties 30 days prior to case closure, as required by Wis. Admin. Code ch. NR 725 and § NR 726.13 (1) (d).

9. **Semi-Annual Reporting – NR 700.11**: Wis. Admin. Code § NR 700.11 (1) (a) requires responsible parties to submit semi-annual site progress reports to the DNR until case closure is granted. The reports summarize the work completed over six months and additional work planned to adequately complete the response action at the site. Consultants may submit these reports on behalf of responsible parties. These reports are due in January and July of each year. Please refer to DNR publication *NR 700 Semi-Annual Site Progress Report* (RR-082), for more information.

Submittals required under Wis. Admin. Code chs. NR 700-799

These documents, as applicable, must be submitted to the DNR prior to the responsible party requesting case closure, unless otherwise directed by the DNR:

- ☐ Ch. NR 708 reports and documentation for any immediate or interim actions.
- ☐ Ch. NR 712 professional certifications and signatures are included with applicable submittals.
- ☐ Ch. NR 716 work plan(s) and site investigation report.
- ☐ Ch. NR 722 remedial action options report (exception is for Dry Cleaners Environmental Response Fund sites), with the selected remedial action identified.
- ☐ Ch. NR 724 design, construction documentation, operation, maintenance and monitoring plans and reports, including vapor mitigation commissioning.
- ☐ Ch. NR 725 submittal(s) that confirms that continuing obligations have been identified and affected property owners have been notified by the responsible parties 30 days prior to requesting case closure.
- ☐ If requesting case closure, the Ch. NR 726 case closure form and documentation substantiating compliance with the NR 700 rule series.
- ☐ Ch. NR 749 fees have been paid, as applicable, including closure and database fees.
- ☐ Ch. NR 700 semi-annual site progress reports starting six months after notification.

Additional Information:

The DNR tracks information on all cleanup sites in a DNR database available at dnr.wi.gov, search "BOTW." The Bureau for Remediation and Redevelopment Tracking System (BRRTS) identification number for this site is listed at the top of this letter. You may view information related to your site on this database at any time.

All correspondence regarding this site should be directed to:

John Sager
Remediation and Redevelopment Program
Wisconsin Department of Natural Resources
1701North 4th Street
Superior, WI 54880
John.sager@wisconsin.gov

To speed up processing, your correspondence should reference the BRRTS and Facility Identification (FID) numbers (if assigned) listed at the top of this letter.

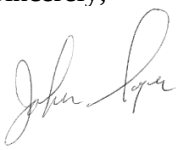
Submittals required under the NR 700 rule series should be sent to the DNR using the RR Program Submittal Portal at dnr.wi.gov, search "RR submittal portal" (https://dnr.wi.gov/topic/Brownfields/Submittal.html). Questions on using this portal can be directed to the contact below or to the environmental program associate (EPA) for the regional DNR office. Visit dnr.wi.gov, search "RR contacts" and select the EPA tab (https://dnr.wi.gov/topic/Brownfields/Contact.html).

Please visit the DNR's Remediation and Redevelopment Program web page at dnr.wi.gov, search "Brownfields" for information on selecting a consultant, seeking financial assistance, and understanding the investigation and cleanup process. Information regarding review fees, liability clarification letters, post-cleanup liability and more is also available.

If you have questions, please call me at (715) 919-7239 or john.sager@wisconsin.gov for more information.

Thank you for your cooperation.

Sincerely,



John Sager
Hydrogeologist - Remediation & Redevelopment Program
Northern Region

cc: Nicholas Larabel, Enbridge Energy
Ryan Erickson, Barr Engineering

Attachment B

Site Investigation Field Sampling and Screening Logs

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG - SHEET 1

Superior Terminal Line 93 Tie-in

Date: 9/10/2021

Sampler: REE

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

Background Headspace: 0.0 ppm

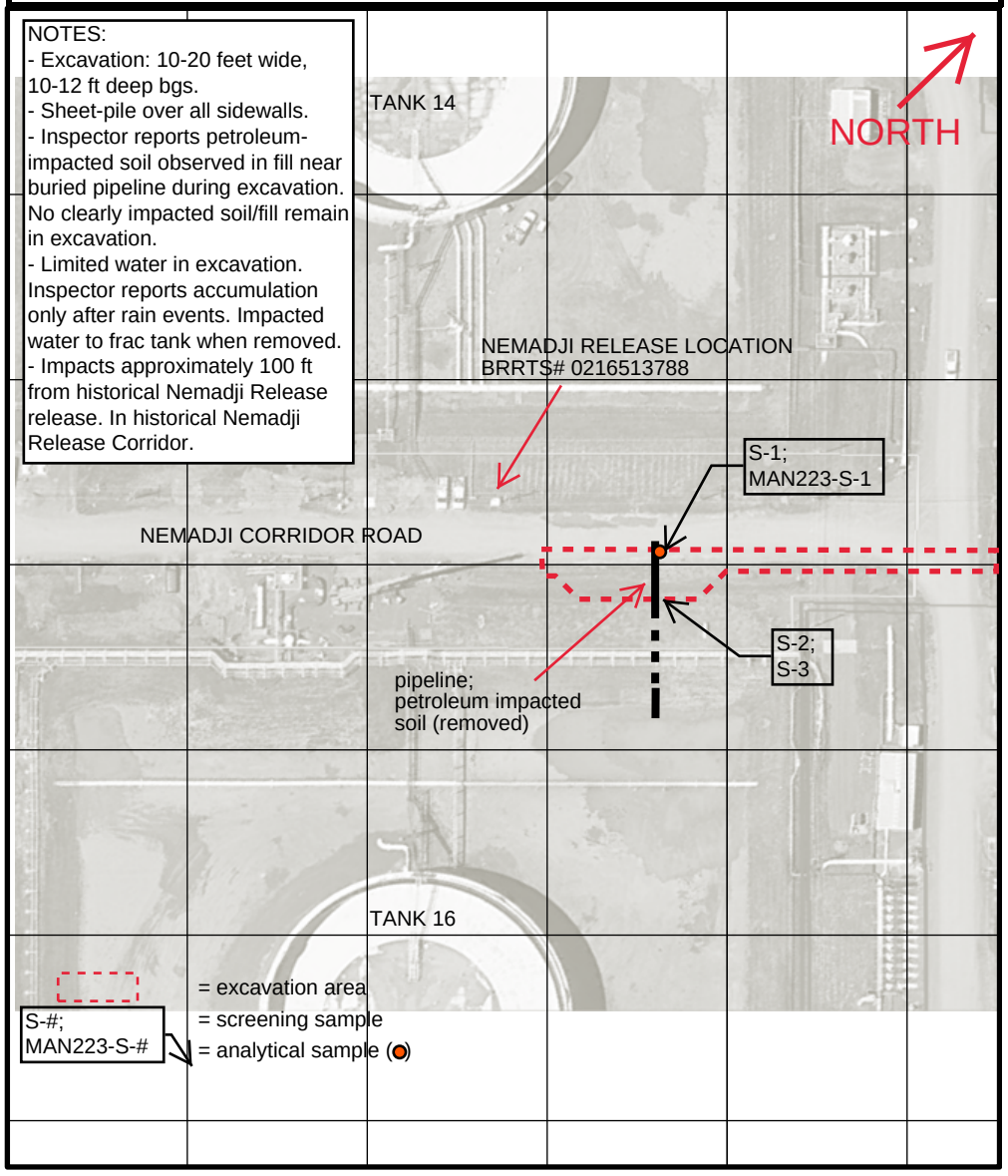
Calibration Time: 7:30

Sample Nomenclature *(Location - sample type - #):* M223-

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*	10	8:30	sand	brown	N/N	0.6
S-2	10		sand	brown	N/N	0.6
S-3	1	8:45	CL	red brown	N/N	0.1
* = analytical sample location						
ANALYTICAL SAMPLES						
MAN223-S-1 @ 10 FT BGS, 8:50						

SITE SKETCH: *north is up; excavation extents & depths, impacted areas, sample locations, borings, wells, structures, utilities, natural features…* ***1 inch/grid = 100 ft***



SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG - SHEET 2

Superior Terminal Line 1 Excavation

Date: 9/20/2021
Sampler: REE
Calibration Time: 7:30

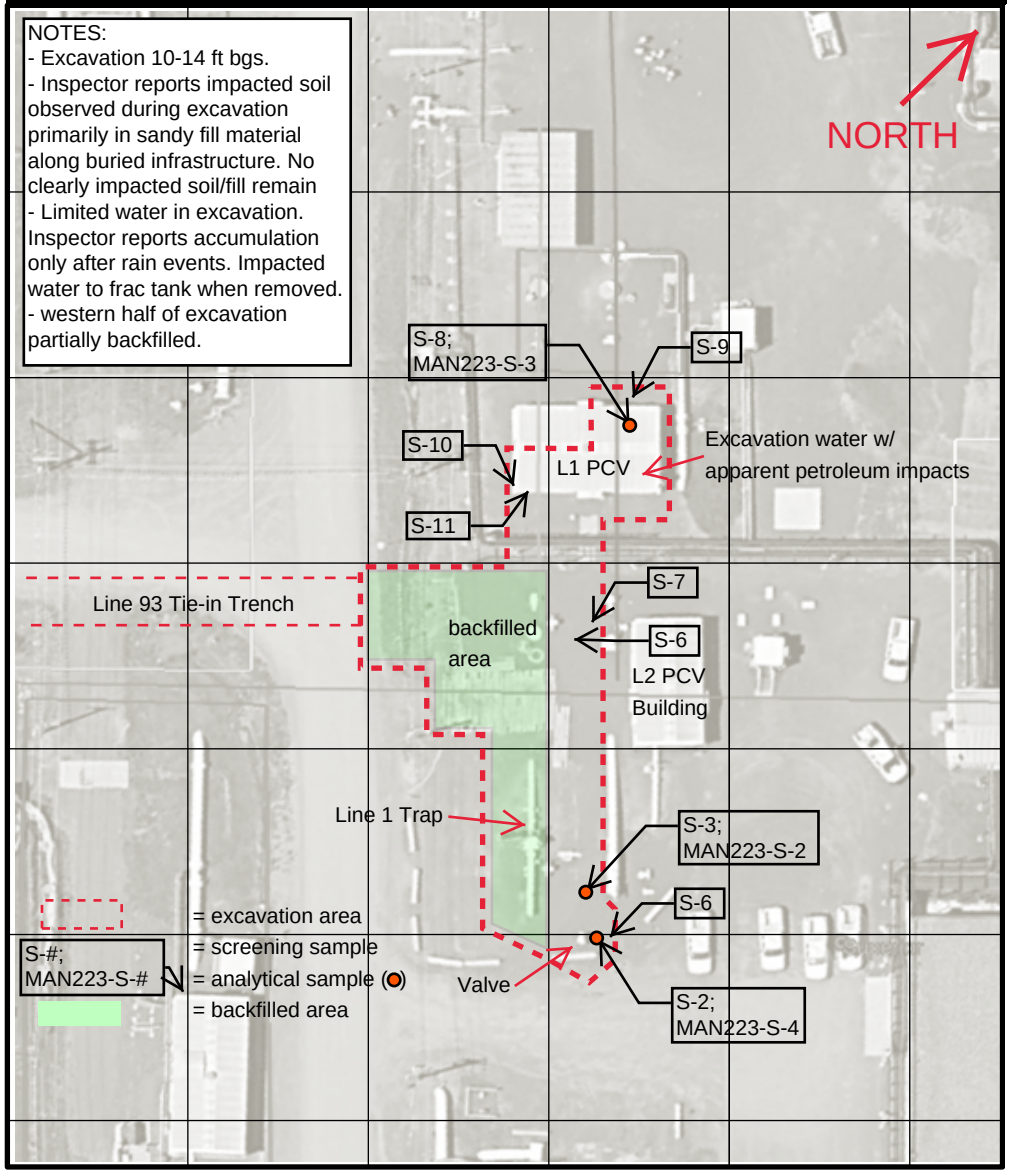
Equipment used: Photo *-ionization detector with* 10.6 *eV lamp* Background Headspace: 0.0 ppm

Sample Nomenclature *(Location - sample type - #):* M223-

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	3	8:45	CL	red brown	N/N	0.3
S-2*	10		CL	red brown	N/N	0.4
S-3*	10		CL	red brown	N/N	16.3
S-4	10		CL	red brown	N/N	0.8
S-5	2		sand fill	red brown	N/N	0.3
S-6	8		CL	red brown	N/N	0.4
S-7	2		sand fill	red brown	N/N	0.3
S-8*	8		CL	red brown	N/N	28.7
S-9	2		gravel	gray	N/N	0.5
S-10	2		CL	red brown	N/N	2.3
S-11	8		CL	red brown	N/N	2.2
* = analytical sample location						
ANALYTICAL SAMPLES						
MAN223-S-2 @ 10 FT BGS, 9:00						
MAN223-S-3 @ 8 FT BGS, 9:25						
MAN223-S-4 @ 10 FT BGS, 8:50						

SITE SKETCH: *north is up; excavation extents & depths, impacted areas, sample locations, borings, wells, structures, utilities, natural features…* ***1 inch/grid = 50ft***



Attachment C

Confirmation Soil Sample Laboratory Reports

22-Sep-2021

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

Re: **Manifold 223** Work Order: **21091402**

Dear Ryan,

ALS Environmental received 1 sample on 14-Sep-2021 09:30 AM 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,

Jodi Blouw

Electronically approved by: Jodi Blouw

Jodi Blouw

Report of Laboratory Analysis

ALS Group, USA

Date: 22-Sep-21

Client: Barr Engineering Company
Project: Manifold 223
Work Order: **21091402**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
21091402-01	MAN223-S-1	Soil		9/10/2021 08:50	9/14/2021 09:30	☐

ALS Group, USA

Date: 22-Sep-21

Client: Barr Engineering Company
Project: Manifold 223
WorkOrder: **21091402**

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

ALS Group, USA

Date: 22-Sep-21

Client: Barr Engineering Company
Project: Manifold 223
Work Order: 21091402

Case Narrative

Samples for the above noted Work Order were received on 09/14/2021. 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: *22-Sep-2021*

Client: Barr Engineering Company
Project: Manifold 223
Work Order: 21091402
Sample ID: MAN223-S-1
Lab ID: 21091402-01
Collection Date: 9/10/2021 08:50 AM
Matrix: SOIL

Analyses	Result	Qual	Report Limit	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS			**SW8260C**	Prep: SW5035A 9/15/21 15:12		Analyst: **HJ**
1,2,4-Trimethylbenzene	ND		38	µg/Kg-dry	1	9/17/2021 10:26 AM
1,3,5-Trimethylbenzene	ND		130	µg/Kg-dry	1	9/17/2021 10:26 AM
Benzene	ND		38	µg/Kg-dry	1	9/17/2021 10:26 AM
Ethylbenzene	ND		38	µg/Kg-dry	1	9/17/2021 10:26 AM
m,p-Xylene	ND		76	µg/Kg-dry	1	9/17/2021 10:26 AM
Naphthalene	ND		130	µg/Kg-dry	1	9/17/2021 10:26 AM
o-Xylene	ND		38	µg/Kg-dry	1	9/17/2021 10:26 AM
Toluene	ND		38	µg/Kg-dry	1	9/17/2021 10:26 AM
Xylenes, Total	ND		110	µg/Kg-dry	1	9/17/2021 10:26 AM
Surr: 1,2-Dichloroethane-d4	*102*		*70-130*	*%REC*	1	9/17/2021 10:26 AM
Surr: 4-Bromofluorobenzene	*100*		*70-130*	*%REC*	1	9/17/2021 10:26 AM
Surr: Dibromofluoromethane	*97.0*		*70-130*	*%REC*	1	9/17/2021 10:26 AM
Surr: Toluene-d8	*96.7*		*70-130*	*%REC*	1	9/17/2021 10:26 AM
MOISTURE			**SW3550C**			Analyst: **ALG**
Moisture	**18**		**0.10**	**% of sample**	1	9/15/2021 02:53 PM

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

QC BATCH REPORT

Batch ID: **183753** Instrument ID **VMS9** Method: **SW8260C**

MBLK Sample ID: **MBLK-183753-183753** Units: **µg/Kg-dry** Analysis Date: **9/15/2021 09:14 PM**

Client ID: Run ID: **VMS9_210915B** SeqNo: **7751254** Prep Date: **9/15/2021** DF: **1**

Analyte	Result	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	ND	30								
1,3,5-Trimethylbenzene	ND	100								
Benzene	ND	30								
Ethylbenzene	ND	30								
m,p-Xylene	ND	60								
Naphthalene	ND	100								
o-Xylene	ND	30								
Toluene	ND	30								
Xylenes, Total	ND	90								
Surr: 1,2-Dichloroethane-d4	*1034*	0	*1000*	0	*103*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*996*	0	*1000*	0	*99.6*	*70-130*	0			
Surr: Dibromofluoromethane	*972*	0	*1000*	0	*97.2*	*70-130*	0			
Surr: Toluene-d8	*1015*	0	*1000*	0	*102*	*70-130*	0			

LCS Sample ID: **LCS-183753-183753** Units: **µg/Kg-dry** Analysis Date: **9/15/2021 08:27 PM**

Client ID: Run ID: **VMS9_210915B** SeqNo: **7751252** Prep Date: **9/15/2021** DF: **1**

Analyte	Result	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	1006	30	1000	0	101	65-135	0			
1,3,5-Trimethylbenzene	1040	100	1000	0	104	65-135	0			
Benzene	979.5	30	1000	0	98	75-125	0			
Ethylbenzene	1001	30	1000	0	100	75-125	0			
m,p-Xylene	2019	60	2000	0	101	80-125	0			
Naphthalene	1022	100	1000	0	102	40-140	0			
o-Xylene	1004	30	1000	0	100	75-125	0			
Toluene	956.5	30	1000	0	95.6	70-125	0			
Xylenes, Total	3022	90	3000	0	101	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1056*	0	*1000*	0	*106*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1028*	0	*1000*	0	*103*	*70-130*	0			
Surr: Dibromofluoromethane	*982.5*	0	*1000*	0	*98.2*	*70-130*	0			
Surr: Toluene-d8	*978*	0	*1000*	0	*97.8*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 21091402
Project: Manifold 223

QC BATCH REPORT

Batch ID: **183753** Instrument ID **VMS9** Method: **SW8260C**

MS	Sample ID: **21091400-01A MS**				Units: **µg/Kg-dry**		Analysis Date: **9/16/2021 03:15 AM**			
Client ID:		Run ID: **VMS9_210915B**			SeqNo: **7752397**		Prep Date: **9/15/2021**		DF: **1**	
Analyte	Result	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	616	19	634.7	0	97.1	65-135	0			
1,3,5-Trimethylbenzene	627.1	63	634.7	0	98.8	65-135	0			
Benzene	604	19	634.7	0	95.2	75-125	0			
Ethylbenzene	615.1	19	634.7	0	96.9	75-125	0			
m,p-Xylene	1261	38	1270	0	99.3	80-125	0			
Naphthalene	612.8	63	634.7	0	96.5	40-140	0			
o-Xylene	600.2	19	634.7	0	94.5	75-125	0			
Toluene	598.6	19	634.7	0	94.3	70-125	0			
Xylenes, Total	1861	57	1904	0	97.7	75-125	0			
Surr: 1,2-Dichloroethane-d4	*663*	0	*634.7*	0	*104*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*649*	0	*634.7*	0	*102*	*70-130*	0			
Surr: Dibromofluoromethane	*650.9*	0	*634.7*	0	*103*	*70-130*	0			
Surr: Toluene-d8	*647.8*	0	*634.7*	0	*102*	*70-130*	0			

MSD	Sample ID: **21091400-01A MSD**				Units: **µg/Kg-dry**		Analysis Date: **9/16/2021 03:31 AM**			
Client ID:		Run ID: **VMS9_210915B**			SeqNo: **7751288**		Prep Date: **9/15/2021**		DF: **1**	
Analyte	Result	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	522.7	19	634.7	0	82.4	65-135	616	0	30	
1,3,5-Trimethylbenzene	541.8	63	634.7	0	85.3	65-135	627.1	0	30	
Benzene	511	19	634.7	0	80.5	75-125	604	0	30	
Ethylbenzene	525.9	19	634.7	0	82.9	75-125	615.1	0	30	
m,p-Xylene	1075	38	1270	0	84.6	80-125	1261	0	30	
Naphthalene	539.9	63	634.7	0	85	40-140	612.8	0	30	
o-Xylene	507.5	19	634.7	0	79.9	75-125	600.2	0	30	
Toluene	518	19	634.7	0	81.6	70-125	598.6	0	30	
Xylenes, Total	1582	57	1904	0	83.1	75-125	1861	0	30	
Surr: 1,2-Dichloroethane-d4	*673.5*	0	*634.7*	0	*106*	*70-130*	663	*0*	*30*	
Surr: 4-Bromofluorobenzene	*615.7*	0	*634.7*	0	*97*	*70-130*	649	*0*	*30*	
Surr: Dibromofluoromethane	*646.2*	0	*634.7*	0	*102*	*70-130*	650.9	*0*	*30*	
Surr: Toluene-d8	*642.7*	0	*634.7*	0	*101*	*70-130*	647.8	*0*	*30*	

The following samples were analyzed in this batch: 21091402-01A

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

Client: Barr Engineering Company
Work Order: 21091402
Project: Manifold 223

QC BATCH REPORT

Batch ID: **R326747** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R326747** Units: **% of sample** Analysis Date: **9/15/2021 02:53 PM**
Client ID: Run ID: **MOIST_210915B** SeqNo: **7750599** Prep Date: DF: **1**

Analyte	Result	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	ND	0.10								

LCS Sample ID: **LCS-R326747** Units: **% of sample** Analysis Date: **9/15/2021 02:53 PM**
Client ID: Run ID: **MOIST_210915B** SeqNo: **7750598** Prep Date: DF: **1**

Analyte	Result	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	99.97	0.10	100	0	100	98-102	0			

DUP Sample ID: **21091213-03B DUP** Units: **% of sample** Analysis Date: **9/15/2021 02:53 PM**
Client ID: Run ID: **MOIST_210915B** SeqNo: **7750581** Prep Date: DF: **1**

Analyte	Result	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	3.27	0.10	0	0	0	0-0	3.62	10.2	10	R

DUP Sample ID: **21091280-09A DUP** Units: **% of sample** Analysis Date: **9/15/2021 02:53 PM**
Client ID: Run ID: **MOIST_210915B** SeqNo: **7750584** Prep Date: DF: **1**

Analyte	Result	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	6.12	0.10	0	0	0	0-0	5.74	6.41	10	

The following samples were analyzed in this batch: 21091402-01B

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

21091402

Barr Engineering Co. Chain of Custody

Sample Origination State
☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ TX ☐ UT ☒ WI ☐ Other: _____

COC Number: № 589630
COC 1 of 1

REPORT TO	INVOICE TO
Company: BARR	Company: SAME
Address: Duluth	Address:
Address:	Address:
Name: Ryan Erickson	Name:
email: Rerickson@barr.com	email:
Copy to: BarrDM@barr.com	P.O.
Project Name: Manifold 223	Barr Project No: 49/61092.10 003 001

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

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

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	Analysis Requested: Water	Analysis Requested: Soil: PVOC's + Naphthalene	Analysis Requested: Soil: % Solids	Preservative Code	Field Filtered Y/N	Notes
1. MAN223-S-1	10	10	FT	9/10/21	8:50	S		3		2	1			PVOC's + Naphthalene
2.														
3.														
4.														JD
5.														
6.														
7.														
8.														IR1 4.0-C
9.														
10.														

BARR USE ONLY

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

Relinquished by	On Ice? Y N	Date	Time	Received by	Date	Time
[signature]	(Y) N	9/13/2021	11:12	FED EX		
FED EX	Y N	9/14/21	0930	[signature]		

Samples Shipped VIA: ☐ Ground Courier ☐ Air Carrier ☐ Sampler ☐ Other: _____

Air Bill Number: 4662 2966 5536

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

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

Distribution - White-Original: Accompanies Shipment to Laboratory; **Yellow Copy:** Include in Field Documents; **Scan and email:** a copy to BarrDM@barr.com for tracking and filing procedures

H:\RLG\STDFORMS\Chain of Custody Form 2015 RLG Rev. 01/30/2020

ALS Group, USA

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **14-Sep-21 09:30**

Work Order: **21091402** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 15-Sep-21 (Date) Reviewed by: Jodi Blouw (eSignature) 15-Sep-21 (Date)

Matrices: Soil

Carrier name: FedEx

Shipping container/cooler in good condition?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on shipping container/cooler?	Yes ☐	No ☐	Not Present ☑
Custody seals intact on sample bottles?	Yes ☐	No ☐	Not Present ☑
Chain of custody present?	Yes ☑	No ☐	
Chain of custody signed when relinquished and received?	Yes ☑	No ☐	
Chain of custody agrees with sample labels?	Yes ☑	No ☐	
Samples in proper container/bottle?	Yes ☑	No ☐	
Sample containers intact?	Yes ☑	No ☐	
Sufficient sample volume for indicated test?	Yes ☑	No ☐	
All samples received within holding time?	Yes ☑	No ☐	
Container/Temp Blank temperature in compliance?	Yes ☑	No ☐	
Sample(s) received on ice?	Yes ☑	No ☐	
Temperature(s)/Thermometer(s):	4.0/4.0 c	IR1	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	9/15/2021 1:29:11 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:

30-Sep-2021

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

Re: **Manifold 223** Work Order: **21092157**

Dear Ryan,

ALS Environmental received 3 samples on 22-Sep-2021 09:00 AM for the analyses presented in the following report.

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

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

The total number of pages in this report is 13.

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

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

Sincerely,

Jodi Blouw

Electronically approved by: Jodi Blouw

Jodi Blouw

Report of Laboratory Analysis

Certificate No: WI: 399084510

ALS Group, USA

Date: 30-Sep-21

Client: Barr Engineering Company
Project: Manifold 223
Work Order: **21092157**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
21092157-01	MAN223-S-4	Soil		9/20/2021	9/22/2021 09:00	☐
21092157-02	MAN223-S-2	Soil		9/20/2021 09:00	9/22/2021 09:00	☐
21092157-03	MAN223-S-3	Soil		9/20/2021 09:25	9/22/2021 09:00	☐

ALS Group, USA

Date: 30-Sep-21

Client: Barr Engineering Company
Project: Manifold 223
WorkOrder: **21092157**

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

ALS Group, USA

Date: 30-Sep-21

Client: Barr Engineering Company
Project: Manifold 223
Work Order: 21092157

Case Narrative

Samples for the above noted Work Order were received on 09/22/2021. 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: *30-Sep-21*

Client: Barr Engineering Company
Project: Manifold 223
Sample ID: MAN223-S-4
Collection Date: 9/20/2021

Work Order: 21092157
Lab ID: 21092157-01
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035A / 9/27/21		Analyst: **MF**
1,2,4-Trimethylbenzene	U		40	55	µg/Kg-dry	1	9/29/2021 14:43
1,3,5-Trimethylbenzene	U		64	180	µg/Kg-dry	1	9/29/2021 14:43
Benzene	U		27	55	µg/Kg-dry	1	9/29/2021 14:43
Ethylbenzene	U		12	55	µg/Kg-dry	1	9/29/2021 14:43
m,p-Xylene	U		73	110	µg/Kg-dry	1	9/29/2021 14:43
Naphthalene	U		130	180	µg/Kg-dry	1	9/29/2021 14:43
o-Xylene	U		21	55	µg/Kg-dry	1	9/29/2021 14:43
Toluene	U		15	55	µg/Kg-dry	1	9/29/2021 14:43
Xylenes, Total	U		73	160	µg/Kg-dry	1	9/29/2021 14:43
Surr: 1,2-Dichloroethane-d4	*98.2*			*70-130*	*%REC*	1	9/29/2021 14:43
Surr: 4-Bromofluorobenzene	*107*			*70-130*	*%REC*	1	9/29/2021 14:43
Surr: Dibromofluoromethane	*98.4*			*70-130*	*%REC*	1	9/29/2021 14:43
Surr: Toluene-d8	*100*			*70-130*	*%REC*	1	9/29/2021 14:43
MOISTURE		Method: **SW3550C**					Analyst: **ALG**
Moisture	**27**		**0.10**	**0.10**	**% of sample**	1	9/24/2021 14:19

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

ALS Group, USA

Date: *30-Sep-21*

Client: Barr Engineering Company
Project: Manifold 223
Sample ID: MAN223-S-2
Collection Date: 9/20/2021 09:00 AM

Work Order: 21092157
Lab ID: 21092157-02
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035A / 9/27/21		Analyst: **MF**
1,2,4-Trimethylbenzene	U		40	54	µg/Kg-dry	1	9/29/2021 15:02
1,3,5-Trimethylbenzene	U		63	180	µg/Kg-dry	1	9/29/2021 15:02
Benzene	U		26	54	µg/Kg-dry	1	9/29/2021 15:02
Ethylbenzene	U		11	54	µg/Kg-dry	1	9/29/2021 15:02
m,p-Xylene	U		72	110	µg/Kg-dry	1	9/29/2021 15:02
Naphthalene	U		130	180	µg/Kg-dry	1	9/29/2021 15:02
o-Xylene	U		21	54	µg/Kg-dry	1	9/29/2021 15:02
Toluene	U		15	54	µg/Kg-dry	1	9/29/2021 15:02
Xylenes, Total	U		72	160	µg/Kg-dry	1	9/29/2021 15:02
Surr: 1,2-Dichloroethane-d4	*97.3*			*70-130*	*%REC*	1	9/29/2021 15:02
Surr: 4-Bromofluorobenzene	*104*			*70-130*	*%REC*	1	9/29/2021 15:02
Surr: Dibromofluoromethane	*99.4*			*70-130*	*%REC*	1	9/29/2021 15:02
Surr: Toluene-d8	*100*			*70-130*	*%REC*	1	9/29/2021 15:02
MOISTURE		Method: **SW3550C**					Analyst: **ALG**
Moisture	**30**		**0.10**	**0.10**	**% of sample**	1	9/24/2021 14:19

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

ALS Group, USA

Date: *30-Sep-21*

Client: Barr Engineering Company
Project: Manifold 223
Sample ID: MAN223-S-3
Collection Date: 9/20/2021 09:25 AM

Work Order: 21092157
Lab ID: 21092157-03
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035A / 9/27/21		Analyst: **MF**
1,2,4-Trimethylbenzene	U		26	36	µg/Kg-dry	1	9/29/2021 15:21
1,3,5-Trimethylbenzene	U		41	120	µg/Kg-dry	1	9/29/2021 15:21
Benzene	U		17	36	µg/Kg-dry	1	9/29/2021 15:21
Ethylbenzene	U		7.5	36	µg/Kg-dry	1	9/29/2021 15:21
m,p-Xylene	U		47	71	µg/Kg-dry	1	9/29/2021 15:21
Naphthalene	U		85	120	µg/Kg-dry	1	9/29/2021 15:21
o-Xylene	U		14	36	µg/Kg-dry	1	9/29/2021 15:21
Toluene	U		9.7	36	µg/Kg-dry	1	9/29/2021 15:21
Xylenes, Total	U		47	110	µg/Kg-dry	1	9/29/2021 15:21
Surr: 1,2-Dichloroethane-d4	*96.1*			*70-130*	*%REC*	1	9/29/2021 15:21
Surr: 4-Bromofluorobenzene	*105*			*70-130*	*%REC*	1	9/29/2021 15:21
Surr: Dibromofluoromethane	*96.5*			*70-130*	*%REC*	1	9/29/2021 15:21
Surr: Toluene-d8	*98.1*			*70-130*	*%REC*	1	9/29/2021 15:21
MOISTURE		Method: **SW3550C**					Analyst: **ALG**
Moisture	**19**		**0.10**	**0.10**	**% of sample**	1	9/24/2021 14:19

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

ALS Group, USA

Date: 30-Sep-21

Client: Barr Engineering Company

Work Order: 21092157

Project: Manifold 223

QC BATCH REPORT

Batch ID: **184416** Instrument ID **VMS8** Method: **SW8260C**

MBLK Sample ID: **MBLK-184416-184416** Units: **µg/Kg-dry** Analysis Date: **9/28/2021 02:54 PM**

Client ID: Run ID: **VMS8_210928A** SeqNo: **7791931** Prep Date: **9/27/2021** 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	15	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	*995.5*	0	0	*1000*	0	*99.6*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1047*	0	0	*1000*	0	*105*	*70-130*	0			
Surr: Dibromofluoromethane	*981*	0	0	*1000*	0	*98.1*	*70-130*	0			
Surr: Toluene-d8	*975.5*	0	0	*1000*	0	*97.6*	*70-130*	0			

LCS Sample ID: **LCS-184416-184416** Units: **µg/Kg-dry** Analysis Date: **9/28/2021 01:40 PM**

Client ID: Run ID: **VMS8_210928A** SeqNo: **7791930** Prep Date: **9/27/2021** 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	956	22	30	1000	0	95.6	65-135	0			
1,3,5-Trimethylbenzene	1131	35	100	1000	0	113	65-135	0			
Benzene	1032	15	30	1000	0	103	75-125	0			
Ethylbenzene	1102	6.3	30	1000	0	110	75-125	0			
m,p-Xylene	2252	40	60	2000	0	113	80-125	0			
Naphthalene	1050	72	100	1000	0	105	40-140	0			
o-Xylene	1073	12	30	1000	0	107	75-125	0			
Toluene	1086	8.2	30	1000	0	109	70-125	0			
Xylenes, Total	3326	40	90	3000	0	111	75-125	0			
Surr: 1,2-Dichloroethane-d4	*959.5*	0	0	*1000*	0	*96*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1012*	0	0	*1000*	0	*101*	*70-130*	0			
Surr: Dibromofluoromethane	*986*	0	0	*1000*	0	*98.6*	*70-130*	0			
Surr: Toluene-d8	*1010*	0	0	*1000*	0	*101*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 21092157
Project: Manifold 223

QC BATCH REPORT

Batch ID: **184416** Instrument ID **VMS8** Method: **SW8260C**

MS Sample ID: **21092157-01A MS** Units: **µg/Kg-dry** Analysis Date: **9/29/2021 07:42 PM**
Client ID: **MAN223-S-4** Run ID: **VMS8_210929A** SeqNo: **7797536** Prep Date: **9/27/2021** 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	1747	40	55	1832	0	95.4	65-135	0			
1,3,5-Trimethylbenzene	2046	64	180	1832	0	112	65-135	0			
Benzene	1819	27	55	1832	0	99.3	75-125	0			
Ethylbenzene	1995	12	55	1832	0	109	75-125	0			
m,p-Xylene	4076	73	110	3664	0	111	80-125	0			
Naphthalene	1855	130	180	1832	0	101	40-140	0			
o-Xylene	1954	21	55	1832	0	107	75-125	0			
Toluene	1891	15	55	1832	0	103	70-125	0			
Xylenes, Total	6029	73	160	5495	0	110	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1828*	0	0	*1832*	0	*99.8*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1917*	0	0	*1832*	0	*105*	*70-130*	0			
Surr: Dibromofluoromethane	*1777*	0	0	*1832*	0	*97*	*70-130*	0			
Surr: Toluene-d8	*1858*	0	0	*1832*	0	*101*	*70-130*	0			

MSD Sample ID: **21092157-01A MSD** Units: **µg/Kg-dry** Analysis Date: **9/29/2021 08:00 PM**
Client ID: **MAN223-S-4** Run ID: **VMS8_210929A** SeqNo: **7797537** Prep Date: **9/27/2021** 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	1735	40	55	1832	0	94.7	65-135	1747	0.684	30	
1,3,5-Trimethylbenzene	2021	64	180	1832	0	110	65-135	2046	1.26	30	
Benzene	1841	27	55	1832	0	101	75-125	1819	1.2	30	
Ethylbenzene	1967	12	55	1832	0	107	75-125	1995	1.39	30	
m,p-Xylene	4006	73	110	3664	0	109	80-125	4076	1.72	30	
Naphthalene	1851	130	180	1832	0	101	40-140	1855	0.198	30	
o-Xylene	1907	21	55	1832	0	104	75-125	1954	2.42	30	
Toluene	1879	15	55	1832	0	103	70-125	1891	0.631	30	
Xylenes, Total	5913	73	160	5495	0	108	75-125	6029	1.95	30	
Surr: 1,2-Dichloroethane-d4	*1817*	0	0	*1832*	0	*99.2*	*70-130*	1828	*0.603*	*30*	
Surr: 4-Bromofluorobenzene	*1921*	0	0	*1832*	0	*105*	*70-130*	1917	*0.191*	*30*	
Surr: Dibromofluoromethane	*1833*	0	0	*1832*	0	*100*	*70-130*	1777	*3.1*	*30*	
Surr: Toluene-d8	*1819*	0	0	*1832*	0	*99.3*	*70-130*	1858	*2.14*	*30*	

The following samples were analyzed in this batch: 21092157-01A 21092157-02A 21092157-03A

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

Client: Barr Engineering Company
Work Order: 21092157
Project: Manifold 223

QC BATCH REPORT

Batch ID: **R327576** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R327576** Units: **% of sample** Analysis Date: **9/24/2021 02:19 PM**
Client ID: Run ID: **MOIST_210924C** SeqNo: **7784572** 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-R327576** Units: **% of sample** Analysis Date: **9/24/2021 02:19 PM**
Client ID: Run ID: **MOIST_210924C** SeqNo: **7784571** 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: **21091835-43B DUP** Units: **% of sample** Analysis Date: **9/24/2021 02:19 PM**
Client ID: Run ID: **MOIST_210924C** SeqNo: **7784550** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	6.68	0.1	0.10	0	0	0	0-0	6.54	2.12	10	

DUP Sample ID: **21091835-46A DUP** Units: **% of sample** Analysis Date: **9/24/2021 02:19 PM**
Client ID: Run ID: **MOIST_210924C** SeqNo: **7784554** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	6.37	0.1	0.10	0	0	0	0-0	6.46	1.4	10	

The following samples were analyzed in this batch: 21092157-01B 21092157-02B 21092157-03B

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

21092157

Barr Engineering Co. Chain of Custody

Sample Origination State
☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ TX ☐ UT ☒ WI ☐ Other: _____

COC Number: № 589624
COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr	Company: BARR
Address: Duluth	Address: SAME
Address:	Address:
Name: Ryan Erickson	Name:
email: REE@Barr.com	email:
Copy to: BarrDM@barr.com	P.O.
Project Name: Manfield 223	Barr Project No: 49161092.10 003 001

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

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

Location	Sample Depth Start	Stop	Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform MS/MSD Y / N	Total Number Of Containers	Analysis Requested: Water	Soil: F: PVOCs + Naphthalene	Soil: % Solids	Preservative Code	Field Filtered Y/N
1. MAN223-S-1	10	10	FT	9/20/21		S		3		2	1	PVOCs + Naphthalene - MTBEs	
2. MAN 223-S-2	10	10	↓	↓	900	↓		3		2	1	↓	
3. MAN 223-S-3	8	8	↓	↓	925	↓		3		2	1	↓	
4.													
5.													
6.													
7.													
8.													
9.													
10.												IR1 4.6°C	

JM

BARR USE ONLY	Relinquished by	On Ice? Y / N	Date	Time	Received by	Date	Time
Sampled by: REE	Ryan Erickson	Y	9/20/21	1200	FED EX		
Barr Proj. Manager: REE	Relinquished by: FED EX	On Ice? Y / N: N	Date: 9/22/21	Time: 0900	Received by:	Date	Time
Barr DQ Manager: JET							
Lab Name: ALS	Samples Shipped VIA: ☐ Ground Courier ☒ Air Carrier ☐ Sampler ☐ Other: _____				Air Bill Number:		
Lab Location: Holland, MI	Lab WO:	Temperature on Receipt (°C):			Custody Seal Intact? ☐ Y ☐ N ☐ None		

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

Distribution - White-Original: Accompanies Shipment to Laboratory; **Yellow Copy:** Include in Field Documents; **Scan and email:** a copy to BarrDM@barr.com for tracking and filing procedures

H:\RLG\STDFORMS\Chain of Custody Form 2015 RLG Rev. 01/30/2020

Barr Engineering Co. Chain of Custody

Sample Origination State: ☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ TX ☐ UT ☒ WI ☐ Other: _____

COC Number: № 589624

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr	Company: BARR
Address: Duluth	Address: SAME
Address:	Address:
Name: Ryan Erickson	Name:
email: REE@Barr.com	email:
Copy to: BarrDM@barr.com	P.O.
Project Name: Manfield 223	Barr Project No: 49161092.10 003 001

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

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

	Location	Sample Depth Start	Stop	Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform MS/MSD Y/N	Total Number Of Containers	Analysis Requested: Water	Soil: PVOCs + Naphthalene	Soil: % Solids	Preservative Code	Field Filtered Y/N
1	MAN223-S-4 (MAN223-S-1)	10	10	FT	9/20/21		S		3		Z	1	PVOCs + Naphthalene – MTBEs	
2	MAN 223-S-2	10	10	↓	↓	900	↓		3		Z	1	↓	
3	MAN 223-S-3	8	8	↓	↓	925	↓		3		Z	1	↓	
4														
5														
6														
7														
8														
9														
10														

JM

BARR USE ONLY

Sampled by: REE

Barr Proj Manager: REE

Barr DQ Manager: JET

Lab Name: ALS

Lab Location: Holland, MI

Relinquished by: [signature] On Ice? Y N: 8 Date: 9/20/21 Time: 1200

Relinquished by: [signature] On Ice? Y N: Date: Time:

Received by: Date: Time:

Received by: Date: Time:

Samples Shipped VIA: ☐ Sampler ☐ Ground Courier ☒ Air Carrier ☐ Other

Air Bill Number:

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

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

Distribution - **White-Original:** Accompanies Shipment to Laboratory, **Yellow Copy:** Include in Field Documents; **Scan and email:** a copy to BarrDM@barr.com for tracking and filing procedures

H:\RLG\STDFORMS\Chain of Custody Form 2015 RLG Rev 01/30/2020

ALS Group, USA

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **22-Sep-21 09:00**

Work Order: **21092157** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 23-Sep-21 (Date) Reviewed by: Jodi Blouw (eSignature) 24-Sep-21 (Date)

Matrices: Soil

Carrier name: FedEx

Shipping container/cooler in good condition?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on shipping container/cooler?	Yes ☐	No ☐	Not Present ☑
Custody seals intact on sample bottles?	Yes ☐	No ☐	Not Present ☑
Chain of custody present?	Yes ☑	No ☐	
Chain of custody signed when relinquished and received?	Yes ☑	No ☐	
Chain of custody agrees with sample labels?	Yes ☑	No ☐	
Samples in proper container/bottle?	Yes ☑	No ☐	
Sample containers intact?	Yes ☑	No ☐	
Sufficient sample volume for indicated test?	Yes ☑	No ☐	
All samples received within holding time?	Yes ☑	No ☐	
Container/Temp Blank temperature in compliance?	Yes ☑	No ☐	
Sample(s) received on ice?	Yes ☑	No ☐	
Temperature(s)/Thermometer(s):	4.6/4.6 c	IR1	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	9/23/2021 7:59:50 AM		
Water - VOA vials have zero headspace?	Yes ☐	No ☐	No VOA vials submitted ☑
Water - pH acceptable upon receipt?	Yes ☐	No ☐	N/A ☑
pH adjusted?	Yes ☐	No ☐	N/A ☑
pH adjusted by:	-		

Login Notes:

Client Contacted: Date Contacted: Person Contacted:

Contacted By: Regarding:

Comments:

CorrectiveAction:

Attachment D

Material Management Documents

Soil Documents

Water Documents

Soil 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

July 22, 2021

Enbridge Energy
Nick Larabel
1100 Louisiana Ave, Ste 3300
Houston, TX 77002

RE: Profile 21-065-I/ Hydrocarbon impacted soil

Nick,

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

The referenced waste must maintain consistency with what was originally submitted on the waste profile. Vonco V Waste Management Campus must be contacted immediately for any changes in material composition or process generation as further testing and analysis may apply. The term of the approval is 3 years and will expire on 6\29\2024.

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

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

21-065-I Terminal Manifold 223					
Date	**Ticket**	**Customer**	**Truck**	**Material**	**Tons**
07/27/2021	334283	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	23.35
07/27/2021	334285	001342 - Enbridge Energy	T94385W	Contaminated Soil - Tons	23.52
07/27/2021	334306	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	21.44
07/27/2021	334322	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	19.13
07/27/2021	334330	001342 - Enbridge Energy	T21989Z	Contaminated Soil - Tons	19.60
07/27/2021	334331	001342 - Enbridge Energy	T21989Z	Contaminated Soil - Tons	19.71
07/27/2021	334338	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	21.24
07/27/2021	334341	001342 - Enbridge Energy	T21989Z	Contaminated Soil - Tons	18.96
07/27/2021	334356	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	22.51
07/27/2021	334362	001342 - Enbridge Energy	T21989Z	Contaminated Soil - Tons	23.45
07/27/2021	334369	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	21.27
07/27/2021	334371	001342 - Enbridge Energy	T21989Z	Contaminated Soil - Tons	21.60
08/02/2021	334547	001342 - Enbridge Energy	T94385W	Contaminated Soil - Tons	31.92
08/02/2021	334548	001342 - Enbridge Energy	T94385W	Contaminated Soil - Tons	30.22
08/02/2021	334549	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	20.54
08/02/2021	334551	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	18.81
08/02/2021	334558	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	21.41
08/02/2021	334559	001342 - Enbridge Energy	T95725W	Contaminated Soil - Tons	20.77
08/02/2021	334560	001342 - Enbridge Energy	T94385W	Contaminated Soil - Tons	21.25
08/02/2021	334562	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	20.44
08/02/2021	334563	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	17.05
08/02/2021	334571	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	21.37
08/02/2021	334573	001342 - Enbridge Energy	T95725W	Contaminated Soil - Tons	18.67
08/02/2021	334577	001342 - Enbridge Energy	T94385W	Contaminated Soil - Tons	16.24
08/02/2021	334581	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	18.81
08/02/2021	334583	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	19.27
08/02/2021	334587	001342 - Enbridge Energy	T95725W	Contaminated Soil - Tons	19.24
08/02/2021	334588	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	25.00

08/02/2021	334591	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	23.49
08/02/2021	334593	001342 - Enbridge Energy	T94385W	Contaminated Soil - Tons	28.99
08/02/2021	334595	001342 - Enbridge Energy	T95725W	Contaminated Soil - Tons	24.33
08/02/2021	334603	001342 - Enbridge Energy	T95725W	Contaminated Soil - Tons	22.16
08/02/2021	334604	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	22.74
08/02/2021	334606	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	23.79
08/02/2021	334607	001342 - Enbridge Energy	T94385W	Contaminated Soil - Tons	19.30
08/02/2021	334608	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	20.44
08/02/2021	334611	001342 - Enbridge Energy	T95725W	Contaminated Soil - Tons	25.11
08/02/2021	334615	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	21.98
08/02/2021	334617	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	21.84
08/02/2021	334618	001342 - Enbridge Energy	T94385W	Contaminated Soil - Tons	25.99
08/02/2021	334619	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	20.77
08/03/2021	334677	001342 - Enbridge Energy	RB25340	Contaminated Soil - Tons	18.15
08/03/2021	334678	001342 - Enbridge Energy	RB32217	Contaminated Soil - Tons	16.34
08/03/2021	334689	001342 - Enbridge Energy	RB32913	Contaminated Soil - Tons	21.27
08/03/2021	334692	001342 - Enbridge Energy	RB32217	Contaminated Soil - Tons	16.47
08/03/2021	334696	001342 - Enbridge Energy	RB25340	Contaminated Soil - Tons	15.59
08/11/2021	334939	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	15.32
08/11/2021	334945	001342 - Enbridge Energy	T219897	Contaminated Soil - Tons	16.79
08/11/2021	334947	001342 - Enbridge Energy	T95469W	Contaminated Soil - Tons	15.33
08/11/2021	334954	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	21.85
08/11/2021	334966	001342 - Enbridge Energy	T219897	Contaminated Soil - Tons	24.36
08/11/2021	334967	001342 - Enbridge Energy	T95469W	Contaminated Soil - Tons	22.30
08/11/2021	334973	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	24.21
08/11/2021	334982	001342 - Enbridge Energy	T219897	Contaminated Soil - Tons	20.52
08/11/2021	334983	001342 - Enbridge Energy	T95469W	Contaminated Soil - Tons	18.07
08/11/2021	334989	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	23.59
08/11/2021	334997	001342 - Enbridge Energy	T21989Z	Contaminated Soil - Tons	27.45
08/11/2021	334998	001342 - Enbridge Energy	T95469W	Contaminated Soil - Tons	20.80
08/11/2021	335000	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	24.51
08/11/2021	335007	001342 - Enbridge Energy	T21989Z	Contaminated Soil - Tons	22.73
08/11/2021	335010	001342 - Enbridge Energy	T95469W	Contaminated Soil - Tons	18.78
08/11/2021	335015	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	18.60
08/20/2021	335350	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	21.90
08/20/2021	335351	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	23.50
08/20/2021	335352	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	22.45
08/20/2021	335357	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	25.27
08/20/2021	335359	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	26.12
08/20/2021	335361	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	23.31

08/20/2021	335370	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	21.93
08/20/2021	335373	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	23.69
08/20/2021	335374	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	21.67
08/20/2021	335375	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	24.34
08/20/2021	335377	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	23.13
08/20/2021	335379	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	28.51
08/20/2021	335382	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	28.49
08/20/2021	335384	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	19.74
08/20/2021	335385	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	21.64
08/20/2021	335390	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	20.29
08/20/2021	335392	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	24.76
08/20/2021	335393	001342 - Enbridge Energy	T78659W	Contaminated Soil - Tons	21.39
09/03/2021	335892	001342 - Enbridge Energy	T18529Z	Contaminated Soil - Tons	15.23
09/03/2021	335893	001342 - Enbridge Energy	R11847Z	Contaminated Soil - Tons	13.35
09/03/2021	335894	001342 - Enbridge Energy	T39449X	Contaminated Soil - Tons	13.52
09/03/2021	335895	001342 - Enbridge Energy	T87447X	Contaminated Soil - Tons	12.37
09/03/2021	335898	001342 - Enbridge Energy	T35273Z	Contaminated Soil - Tons	12.42
09/03/2021	335900	001342 - Enbridge Energy	R11847Z	Contaminated Soil - Tons	12.39
09/03/2021	335901	001342 - Enbridge Energy	T18529Z	Contaminated Soil - Tons	15.33
09/03/2021	335902	001342 - Enbridge Energy	T39449X	Contaminated Soil - Tons	13.09
09/03/2021	335903	001342 - Enbridge Energy	T87447X	Contaminated Soil - Tons	13.31
09/09/2021	335990	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	24.82
09/09/2021	335991	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	23.38
09/09/2021	335999	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	27.60
09/09/2021	336002	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	23.02
09/09/2021	336012	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	19.06
09/09/2021	336018	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	18.99
09/09/2021	336023	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	24.11
09/09/2021	336026	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	25.16
09/09/2021	336031	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	23.71
09/09/2021	336032	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	23.18
09/09/2021	336041	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	26.45
09/09/2021	336044	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	24.41
09/09/2021	336050	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	24.95
09/17/2021	336321	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	22.25
09/17/2021	336327	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	25.27
09/17/2021	336339	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	24.85
09/17/2021	336351	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	24.83
09/17/2021	336358	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	25.77
09/17/2021	336366	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	24.56

10/28/2021	337598	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	30.49
10/28/2021	337609	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	24.09
10/28/2021	337618	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	23.18
10/28/2021	337627	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	24.13
10/28/2021	337637	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	22.23
10/28/2021	337642	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	21.33
11/05/2021	337941	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	20.00
11/05/2021	337942	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	17.89
11/05/2021	337954	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	26.30
11/05/2021	337958	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	24.44
11/05/2021	337964	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	24.02
11/05/2021	337966	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	19.63
11/05/2021	337977	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	27.71
11/05/2021	337980	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	24.43
11/05/2021	337994	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	23.64
11/05/2021	337997	001342 - Enbridge Energy	T49903X	Contaminated Soil - Tons	20.66
11/05/2021	338010	001342 - Enbridge Energy	T36615Z	Contaminated Soil - Tons	25.78

Total Tons	**2,720.21**
Total Loads	**125**

21-Jul-2021

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

Re: **Manifold 223 Soil (49161092.09 003 001)** Work Order: **21071213**

Dear Jim,

ALS Environmental received 1 sample on 15-Jul-2021 08:45 AM 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 12.

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: Jodi Blouw

Ehrland Bosworth
Project Manager

Report of Laboratory Analysis

Certificate No: WI: 399084510

ALS Group, USA

Date: 21-Jul-21

Client: Barr Engineering Company
Project: Manifold 223 Soil (49161092.09 003 001)
Work Order: **21071213**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
21071213-01	Sup SMA Stockpile-3	Soil		7/14/2021 11:20	7/15/2021 08:45	☐

Client:	Barr Engineering Company
Project:	Manifold 223 Soil (49161092.09 003 001)
WorkOrder:	**21071213**

QUALIFIERS, ACRONYMS, UNITS

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

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

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

ALS Group, USA

Date: 21-Jul-21

Client:	Barr Engineering Company
Project:	Manifold 223 Soil (49161092.09 003 001)
Work Order:	21071213

Case Narrative

Samples for the above noted Work Order were received on 07/15/2021. 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: *21-Jul-21*

Client: Barr Engineering Company
Project: Manifold 223 Soil (49161092.09 003 001)
Sample ID: Sup SMA Stockpile-3
Collection Date: 7/14/2021 11:20 AM

Work Order: 21071213
Lab ID: 21071213-01
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
DIESEL RANGE ORGANICS BY GC-FID		Method: **PUBL-SW-141**			Prep: PUBL-SW-141 / 7/19/21		Analyst: **SJB**
DRO (C10-C28)	**230**		**3.4**	**35**	**mg/Kg-dry**	4	7/20/2021 19:16
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**					Analyst: **BG**
Benzene	U		48	160	µg/Kg-dry	1	7/17/2021 02:27
Ethylbenzene	U		21	70	µg/Kg-dry	1	7/17/2021 02:27
m,p-Xylene	U		130	440	µg/Kg-dry	1	7/17/2021 02:27
o-Xylene	U		38	130	µg/Kg-dry	1	7/17/2021 02:27
Toluene	U		27	90	µg/Kg-dry	1	7/17/2021 02:27
Xylenes, Total	U		130	440	µg/Kg-dry	1	7/17/2021 02:27
Surr: 1,2-Dichloroethane-d4	*95.2*			*70-130*	*%REC*	1	7/17/2021 02:27
Surr: 4-Bromofluorobenzene	*96.2*			*70-130*	*%REC*	1	7/17/2021 02:27
Surr: Dibromofluoromethane	*92.3*			*70-130*	*%REC*	1	7/17/2021 02:27
Surr: Toluene-d8	*98.9*			*70-130*	*%REC*	1	7/17/2021 02:27
MOISTURE		Method: **SW3550C**					Analyst: **CDG**
Moisture	**44**		**0.10**	**0.10**	**% of sample**	1	7/16/2021 14:41

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

QC BATCH REPORT

Batch ID: **180419** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK Sample ID: **DBLKS-180419-180419** Units: **mg/Kg** Analysis Date: **7/20/2021 05:19 AM**

Client ID: Run ID: **GC8_210719B** SeqNo: **7592867** Prep Date: **7/19/2021** 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: **DLCSS-180419-180419** Units: **mg/Kg** Analysis Date: **7/20/2021 05:56 AM**

Client ID: Run ID: **GC8_210719B** SeqNo: **7592868** Prep Date: **7/19/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	7.705	0.5	5.0	10	0	77	70-120	0			

LCSD Sample ID: **DLCSDS-180419-180419** Units: **mg/Kg** Analysis Date: **7/20/2021 06:33 AM**

Client ID: Run ID: **GC8_210719B** SeqNo: **7592869** Prep Date: **7/19/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	7	0.5	5.0	10	0	70	70-120	7.705	9.59	20	

The following samples were analyzed in this batch: 21071213-01B

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

Client: Barr Engineering Company
Work Order: 21071213
Project: Manifold 223 Soil (49161092.09 003 001)

QC BATCH REPORT

Batch ID: **180376w** Instrument ID **VMS9** Method: **SW8260C**

MBLK Sample ID: **MBLK-180376-180376w** Units: **µg/Kg-dry** Analysis Date: **7/16/2021 10:00 PM**
Client ID: Run ID: **VMS9_210716A** SeqNo: **7590238** Prep Date: **7/16/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	U	15	48								
Ethylbenzene	U	6.3	21								
m,p-Xylene	U	40	130								
o-Xylene	U	12	39								
Toluene	U	8.2	27								
Xylenes, Total	U	40	130								
Surr: 1,2-Dichloroethane-d4	*938.5*	0	0	*1000*	0	*93.8*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*951.5*	0	0	*1000*	0	*95.2*	*70-130*	0			
Surr: Dibromofluoromethane	*909.5*	0	0	*1000*	0	*91*	*70-130*	0			
Surr: Toluene-d8	*982*	0	0	*1000*	0	*98.2*	*70-130*	0			

LCS Sample ID: **LCS-180376-180376w** Units: **µg/Kg-dry** Analysis Date: **7/16/2021 09:14 PM**
Client ID: Run ID: **VMS9_210716A** SeqNo: **7590232** Prep Date: **7/16/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	951.5	15	48	1000	0	95.2	75-125	0			
Ethylbenzene	969	6.3	21	1000	0	96.9	75-125	0			
m,p-Xylene	1958	40	130	2000	0	97.9	80-125	0			
o-Xylene	985.5	12	39	1000	0	98.6	75-125	0			
Toluene	984	8.2	27	1000	0	98.4	70-125	0			
Xylenes, Total	2943	40	130	3000	0	98.1	75-125	0			
Surr: 1,2-Dichloroethane-d4	*942*	0	0	*1000*	0	*94.2*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*929.5*	0	0	*1000*	0	*93*	*70-130*	0			
Surr: Dibromofluoromethane	*956*	0	0	*1000*	0	*95.6*	*70-130*	0			
Surr: Toluene-d8	*1016*	0	0	*1000*	0	*102*	*70-130*	0			

MS Sample ID: **21071213-01A MS** Units: **µg/Kg-dry** Analysis Date: **7/17/2021 03:29 AM**
Client ID: **Sup SMA Stockpile-3** Run ID: **VMS9_210716A** SeqNo: **7590259** Prep Date: **7/16/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	3027	48	160	3301	0	91.7	75-125	0			
Ethylbenzene	3066	21	70	3301	0	92.9	75-125	0			
m,p-Xylene	6176	130	440	6602	0	93.6	80-125	0			
o-Xylene	3106	38	130	3301	0	94.1	75-125	0			
Toluene	3007	27	90	3301	0	91.1	70-125	0			
Xylenes, Total	9282	130	440	9902	0	93.7	75-125	0			
Surr: 1,2-Dichloroethane-d4	*3063*	0	0	*3301*	0	*92.8*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*3225*	0	0	*3301*	0	*97.7*	*70-130*	0			
Surr: Dibromofluoromethane	*3030*	0	0	*3301*	0	*91.8*	*70-130*	0			
Surr: Toluene-d8	*3248*	0	0	*3301*	0	*98.4*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 21071213
Project: Manifold 223 Soil (49161092.09 003 001)

QC BATCH REPORT

Batch ID: **180376w** Instrument ID **VMS9** Method: **SW8260C**

MSD Sample ID: **21071213-01A MSD** Units: **µg/Kg-dry** Analysis Date: **7/17/2021 03:45 AM**
Client ID: **Sup SMA Stockpile-3** Run ID: **VMS9_210716A** SeqNo: **7590260** Prep Date: **7/16/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	3132	48	160	3301	0	94.9	75-125	3027	3.43	30	
Ethylbenzene	3180	21	70	3301	0	96.3	75-125	3066	3.65	30	
m,p-Xylene	6460	130	440	6602	0	97.9	80-125	6176	4.49	30	
o-Xylene	3261	38	130	3301	0	98.8	75-125	3106	4.87	30	
Toluene	3132	27	90	3301	0	94.9	70-125	3007	4.09	30	
Xylenes, Total	9721	130	440	9902	0	98.2	75-125	9282	4.62	30	
Surr: 1,2-Dichloroethane-d4	*3111*	0	0	*3301*	0	*94.2*	*70-130*	3063	*1.55*	*30*	
Surr: 4-Bromofluorobenzene	*3296*	0	0	*3301*	0	*99.9*	*70-130*	3225	*2.18*	*30*	
Surr: Dibromofluoromethane	*3035*	0	0	*3301*	0	*92*	*70-130*	3030	*0.164*	*30*	
Surr: Toluene-d8	*3205*	0	0	*3301*	0	*97.1*	*70-130*	3248	*1.33*	*30*	

The following samples were analyzed in this batch: 21071213-01A

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

Client: Barr Engineering Company
Work Order: 21071213
Project: Manifold 223 Soil (49161092.09 003 001)

QC BATCH REPORT

Batch ID: **R322290** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R322290** Units: **% of sample** Analysis Date: **7/16/2021 02:41 PM**
Client ID: Run ID: **MOIST_210716C** SeqNo: **7588013** 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-R322290** Units: **% of sample** Analysis Date: **7/16/2021 02:41 PM**
Client ID: Run ID: **MOIST_210716C** SeqNo: **7588012** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	99.99	0.1	0.10	100	0	100	98-102	0			

DUP Sample ID: **21070739-06B DUP** Units: **% of sample** Analysis Date: **7/16/2021 02:41 PM**
Client ID: Run ID: **MOIST_210716C** SeqNo: **7587993** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	29.97	0.1	0.10	0	0	0	0-0	32.57	8.31	10	

DUP Sample ID: **21070739-17B DUP** Units: **% of sample** Analysis Date: **7/16/2021 02:41 PM**
Client ID: Run ID: **MOIST_210716C** SeqNo: **7588004** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	33.97	0.1	0.10	0	0	0	0-0	34.26	0.85	10	

The following samples were analyzed in this batch: 21071213-01C

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

21071213

BARR Barr Engineering Co. Chain of Custody

Sample Origination State
☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ TX ☐ UT ☒ WI ☐ Other: _____

COC Number: № 589712
COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company: Enbridge Energy
Address: 325 South Lake Ave.	Address: 119 North 25th St. East
Address: Duluth, MN	Address: Superior, WI 54880
Name: Kym Erickson	Name: Julie O'Brien
email: kerickson@barr.com	email:
Copy to: BarrDM@barr.com / Staraldsen@barr.com	
Project Name: SMA Soil mgt	Barr Project No: 49/61225.10 100 002

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 = Zn Acetate; K = 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 X N	Total Number Of Containers	Analysis Requested: Water	Soil: 40ml VOA Vials+MeoH	Soil: 4oz Amber jar- DRO	Soil: 4oz Plastic jar	Soil: % Solids	Preservative Code	Field Filtered Y/N	Comments
										F	A	A	A			
										-	-	-	-			
1. Sup SMA Stockpile-3				7/14/21	11:20	S	N	5		2	1	1	1			BTEX, DRO, moisture and Hold Sample
2.																
3.																
4.																
5.																
6.																
7.																
8.																
9.																
10.																

JW

BARR USE ONLY

Sampled by: JET
Barr Proj. Manager: Kym Erickson
Barr DQ Manager: JET
Lab Name: ALS Environmental
Lab Location: Holland, MI

Relinquished by: [signature] On Ice? (Y) N Date 7/14/21 Time 18:00 Received by: FedEx Date Time
Relinquished by: FedEx On Ice? (Y) N Date 7/15/21 Time 0845 Received by: [signature] Date Time

Samples Shipped VIA: ☐ Ground Courier ☒ Air Carrier ☐ Sampler ☐ Other: _____
Air Bill Number: 3.0% IB3
Lab WO: Temperature on Receipt (°C): Custody Seal Intact? ☐ Y ☐ N ☐ None

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

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Scan and email: a copy to BarrDM@barr.com for tracking and filing procedures

H:\RLG\STDFORMS\Chain of Custody Form 2015 RLG Rev. 01/30/2020

ALS Group, USA

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **15-Jul-21 08:45**

Work Order: **21071213** Received by: **KRW**

Checklist completed by Keith Wierenga (eSignature) 15-Jul-21 (Date) Reviewed by: Jodi Blouw (eSignature) 16-Jul-21 (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):	3.0/4.0 C	IR3	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	7/15/2021 2:03:05 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:

28-Jul-2021

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

Re: **Historical Contamination** Work Order: **21071815**

Dear Jim,

ALS Environmental received 1 sample on 22-Jul-2021 09:30 AM 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 12.

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,

Jodi Blouw

Electronically approved by: Jodi Blouw

Jodi Blouw

Report of Laboratory Analysis

ALS Group, USA

Date: 28-Jul-21

Client: Barr Engineering Company
Project: Historical Contamination
Work Order: **21071815**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
21071815-01	Line 1 Stockpile-1	Soil		7/19/2021 12:20	7/22/2021 09:30	☐

Client: Barr Engineering Company
Project: Historical Contamination
WorkOrder: **21071815**

QUALIFIERS, ACRONYMS, UNITS

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

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

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

ALS Group, USA

Date: 28-Jul-21

Client: Barr Engineering Company
Project: Historical Contamination
Work Order: 21071815

Case Narrative

Samples for the above noted Work Order were received on 07/22/2021. 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 other deviations or anomalies were noted.

Extractable Organics:
No other deviations or anomalies were noted.

Wet Chemistry:
Batch R322914, Method SW3550C, Sample 21071815-01C DUP: The RPD between the sample and its duplicate was out of control. The corresponding sample result should be considered estimated for this analyte.
No other deviations or anomalies were noted.

ALS Group, USA

Date: *28-Jul-21*

Client: Barr Engineering Company
Project: Historical Contamination
Sample ID: Line 1 Stockpile-1
Collection Date: 7/19/2021 12:20 PM

Work Order: 21071815
Lab ID: 21071815-01
Matrix: SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
DIESEL RANGE ORGANICS BY GC-FID		Method: **PUBL-SW-141**			Prep: PUBL-SW-141 / 7/26/21		Analyst: **SJB**
DRO (C10-C28)	**120**		**3.6**	**36**	**mg/Kg-dry**	5	7/26/2021 19:10
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035A / 7/23/21		Analyst: **MF**
Benzene	ND		27	56	µg/Kg-dry	1	7/27/2021 03:58
Ethylbenzene	ND		12	56	µg/Kg-dry	1	7/27/2021 03:58
m,p-Xylene	ND		75	110	µg/Kg-dry	1	7/27/2021 03:58
o-Xylene	ND		22	56	µg/Kg-dry	1	7/27/2021 03:58
Toluene	ND		15	56	µg/Kg-dry	1	7/27/2021 03:58
Xylenes, Total	ND		75	170	µg/Kg-dry	1	7/27/2021 03:58
Surr: 1,2-Dichloroethane-d4	*102*			*70-130*	*%REC*	1	7/27/2021 03:58
Surr: 4-Bromofluorobenzene	*107*			*70-130*	*%REC*	1	7/27/2021 03:58
Surr: Dibromofluoromethane	*97.6*			*70-130*	*%REC*	1	7/27/2021 03:58
Surr: Toluene-d8	*104*			*70-130*	*%REC*	1	7/27/2021 03:58
MOISTURE		Method: **SW3550C**					Analyst: **ALG**
Moisture	**32**		**0.10**	**0.10**	**% of sample**	1	7/26/2021 10:12

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

ALS Group, USA

Date: 28-Jul-21

Client: Barr Engineering Company

Work Order: 21071815

Project: Historical Contamination

QC BATCH REPORT

Batch ID: **180827** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK Sample ID: **DBLKS1-180827-180827** Units: **mg/Kg** Analysis Date: **7/26/2021 04:03 PM**

Client ID: Run ID: **GC8_210726A** SeqNo: **7610863** Prep Date: **7/26/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	0.9424	0.5	5.0								J

LCS Sample ID: **DLCSS1-180827-180827** Units: **mg/Kg** Analysis Date: **7/26/2021 04:41 PM**

Client ID: Run ID: **GC8_210726A** SeqNo: **7610864** Prep Date: **7/26/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	8.973	0.5	5.0	10	0	89.7	70-120	0			

LCSD Sample ID: **DLCSDS1-180827-180827** Units: **mg/Kg** Analysis Date: **7/26/2021 05:55 PM**

Client ID: Run ID: **GC8_210726A** SeqNo: **7610866** Prep Date: **7/26/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	8.134	0.5	5.0	10	0	81.3	70-120	8.973	9.81	20	

The following samples were analyzed in this batch: 21071815-01B

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

Client: Barr Engineering Company
Work Order: 21071815
Project: Historical Contamination

QC BATCH REPORT

Batch ID: **180749** Instrument ID **VMS9** Method: **SW8260C**

MBLK Sample ID: **MBLK-180749-180749** Units: **µg/Kg-dry** Analysis Date: **7/27/2021 03:47 PM**
Client ID: Run ID: **VMS9_210727A** SeqNo: **7615456** Prep Date: **7/23/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	ND	15	30								
Ethylbenzene	ND	6.3	30								
m,p-Xylene	ND	40	60								
o-Xylene	ND	12	30								
Toluene	ND	8.2	30								
Xylenes, Total	ND	40	90								
Surr: 1,2-Dichloroethane-d4	*999*	0	0	*1000*	0	*99.9*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*992.5*	0	0	*1000*	0	*99.2*	*70-130*	0			
Surr: Dibromofluoromethane	*966.5*	0	0	*1000*	0	*96.6*	*70-130*	0			
Surr: Toluene-d8	*1012*	0	0	*1000*	0	*101*	*70-130*	0			

LCS Sample ID: **LCS-180749-180749** Units: **µg/Kg-dry** Analysis Date: **7/27/2021 02:45 PM**
Client ID: Run ID: **VMS9_210727A** SeqNo: **7615453** Prep Date: **7/23/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	944.5	15	30	1000	0	94.4	75-125	0			
Ethylbenzene	943.5	6.3	30	1000	0	94.4	75-125	0			
m,p-Xylene	1962	40	60	2000	0	98.1	80-125	0			
o-Xylene	976	12	30	1000	0	97.6	75-125	0			
Toluene	947	8.2	30	1000	0	94.7	70-125	0			
Xylenes, Total	2938	40	90	3000	0	97.9	75-125	0			
Surr: 1,2-Dichloroethane-d4	*990*	0	0	*1000*	0	*99*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*936*	0	0	*1000*	0	*93.6*	*70-130*	0			
Surr: Dibromofluoromethane	*986*	0	0	*1000*	0	*98.6*	*70-130*	0			
Surr: Toluene-d8	*1002*	0	0	*1000*	0	*100*	*70-130*	0			

MS Sample ID: **21071815-01A MS** Units: **µg/Kg-dry** Analysis Date: **7/27/2021 09:44 AM**
Client ID: **Line 1 Stockpile-1** Run ID: **VMS9_210726B** SeqNo: **7612319** Prep Date: **7/23/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1963	27	56	1879	19.73	103	75-125	0			
Ethylbenzene	1957	12	56	1879	34.77	102	75-125	0			
m,p-Xylene	4012	75	110	3758	62.01	105	80-125	0			
o-Xylene	2005	22	56	1879	14.09	106	75-125	0			
Toluene	1937	15	56	1879	0	103	70-125	0			
Xylenes, Total	6017	75	170	5638	0	107	75-125	0			
Surr: 1,2-Dichloroethane-d4	*1918*	0	0	*1879*	0	*102*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1984*	0	0	*1879*	0	*106*	*70-130*	0			
Surr: Dibromofluoromethane	*1766*	0	0	*1879*	0	*94*	*70-130*	0			
Surr: Toluene-d8	*1948*	0	0	*1879*	0	*104*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 21071815
Project: Historical Contamination

QC BATCH REPORT

Batch ID: **180749** Instrument ID **VMS9** Method: **SW8260C**

MSD Sample ID: **21071815-01A MSD** Units: **µg/Kg-dry** Analysis Date: **7/27/2021 09:59 AM**

Client ID: **Line 1 Stockpile-1** Run ID: **VMS9_210726B** SeqNo: **7612320** Prep Date: **7/23/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1945	27	56	1879	19.73	102	75-125	1963	0.914	30	
Ethylbenzene	1968	12	56	1879	34.77	103	75-125	1957	0.527	30	
m,p-Xylene	3943	75	110	3758	62.01	103	80-125	4012	1.75	30	
o-Xylene	1986	22	56	1879	14.09	105	75-125	2005	0.942	30	
Toluene	1916	15	56	1879	0	102	70-125	1937	1.12	30	
Xylenes, Total	5929	75	170	5638	0	105	75-125	6017	1.48	30	
Surr: 1,2-Dichloroethane-d4	*1893*	0	0	*1879*	0	*101*	*70-130*	1918	*1.28*	*30*	
Surr: 4-Bromofluorobenzene	*2000*	0	0	*1879*	0	*106*	*70-130*	1984	*0.802*	*30*	
Surr: Dibromofluoromethane	*1877*	0	0	*1879*	0	*99.9*	*70-130*	1766	*6.09*	*30*	
Surr: Toluene-d8	*1977*	0	0	*1879*	0	*105*	*70-130*	1948	*1.48*	*30*	

The following samples were analyzed in this batch: 21071815-01A

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

Client: Barr Engineering Company
Work Order: 21071815
Project: Historical Contamination

QC BATCH REPORT

Batch ID: **R322914** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R322914** Units: **% of sample** Analysis Date: **7/26/2021 10:12 AM**
Client ID: Run ID: **MOIST_210726B** SeqNo: **7611259** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	ND	0.1	0.10								

LCS Sample ID: **LCS-R322914** Units: **% of sample** Analysis Date: **7/26/2021 10:12 AM**
Client ID: Run ID: **MOIST_210726B** SeqNo: **7611258** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	99.96	0.1	0.10	100	0	100	98-102	0			

DUP Sample ID: **21071815-01C DUP** Units: **% of sample** Analysis Date: **7/26/2021 10:12 AM**
Client ID: **Line 1 Stockpile-1** Run ID: **MOIST_210726B** SeqNo: **7611243** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	36.39	0.1	0.10	0	0	0	0-0	32.49	11.3	10	R

DUP Sample ID: **21071841-05A DUP** Units: **% of sample** Analysis Date: **7/26/2021 10:12 AM**
Client ID: Run ID: **MOIST_210726B** SeqNo: **7611252** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	13.54	0.1	0.10	0	0	0	0-0	13.56	0.148	10	

The following samples were analyzed in this batch: 21071815-01C

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

21071815

Barr Engineering Co. Chain of Custody

Sample Origination State: ☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ TX ☐ UT ☒ WI ☐ Other: _____

COC Number: № 589914

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company:
Address: 3255 Lake Ave	Address: Barr Engineering
Address: Duluth, MN	Name: — Same —
Name: Ryan Erickson	email:
email: rerickson@barr.com	Barr Project No: 49161092.09 003001
Copy to: BarrDM@barr.com, JTgualdsen@barr.com	
Project Name: Historical Contamination	

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 = Zn Acetate; K = Other

Location	Sample Depth Start	Stop	Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform MS/MSD Y/N	Total Number Of Containers	Analysis Requested: Water	Soil: 40ml VOA + MeOH	4oz Jar - DRO	4oz Jar - Hold sample	% Solids	Preservative Code	Field Filtered Y/N	Comments
1. Line 1 Stockpile-1		—		7/19/21	12:20	S	N	5		2	1	1		F	N	DRO, BTEX, % Moisture + Hold sample
2.																
3.																
4.																
5.																
6.																
7.																
8.																
9.																
10.																

ASAP TAT

JW

BARR USE ONLY

Sampled by: J. Tovaldsen

Barr Proj. Manager: Ryan Erickson

Barr DQ Manager: J. Tovaldsen

Lab Name: ALS

Lab Location: Holland, MI

Relinquished by: Jennifer Tovaldsen — On Ice? (Y) N — Date 7/19/21 — Time 14:30 — Received by: FEDEx — Date — Time

Relinquished by: FEDEx — On Ice? (Y) N — Date 7/22/21 — Time 0930 — Received by: — Date — Time

Samples Shipped VIA: ☐ Ground Courier ☒ Air Carrier ☐ Sampler ☐ Other: _____

Air Bill Number:

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

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

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Scan and email: a copy to BarrDM@barr.com for tracking and filing procedures

H:\RLG\STDFORMS\Chain of Custody Form 2015 RLG Rev. 01/30/2020

ALS Group, USA

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **22-Jul-21 09:30**

Work Order: **21071815** Received by: **KRW**

Checklist completed by Keith Wierenga (eSignature) 22-Jul-21 (Date) Reviewed by: Jodi Blouw (eSignature) 25-Jul-21 (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):	13.4/14.4 C	IR3	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	7/22/2021 12:18:08 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:

18-Aug-2021

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

Re: **Manifold - 223** Work Order: **21081019**

Dear Ryan,

ALS Environmental received 1 sample on 11-Aug-2021 04:00 PM 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 12.

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,

Jodi Blouw

Electronically approved by: Jodi Blouw

Jodi Blouw

Report of Laboratory Analysis

ALS Group, USA

Date: 18-Aug-21

Client: Barr Engineering Company
Project: Manifold - 223
Work Order: **21081019**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
21081019-01	Manifold 223 - Stockpile-2	Soil		8/10/2021 13:45	8/11/2021 16:00	☐

Client:	Barr Engineering Company
Project:	Manifold - 223
WorkOrder:	**21081019**

QUALIFIERS, ACRONYMS, UNITS

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

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

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

Client: Barr Engineering Company
Project: Manifold - 223
Work Order: 21081019

Case Narrative

Samples for the above noted Work Order were received on 08/11/2021. 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: *18-Aug-21*

Client: Barr Engineering Company
Project: Manifold - 223
Sample ID: Manifold 223 - Stockpile-2
Collection Date: 8/10/2021 01:45 PM

Work Order: 21081019
Lab ID: 21081019-01
Matrix: SOIL

Analyses	Result	Qual	MDL	Report Limit	Units	Dilution Factor	Date Analyzed
DIESEL RANGE ORGANICS BY GC-FID		Method: **PUBL-SW-141**			Prep: PUBL-SW-141 / 8/16/21		Analyst: **SJB**
DRO (C10-C28)	ND		0.66	6.7	mg/Kg-dry	1	8/17/2021 18:38
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**			Prep: SW5035A / 8/12/21		Analyst: **HJ**
Benzene	ND		18	38	µg/Kg-dry	1	8/16/2021 17:44
Ethylbenzene	ND		8.0	38	µg/Kg-dry	1	8/16/2021 17:44
m,p-Xylene	ND		50	75	µg/Kg-dry	1	8/16/2021 17:44
o-Xylene	ND		15	38	µg/Kg-dry	1	8/16/2021 17:44
Toluene	ND		10	38	µg/Kg-dry	1	8/16/2021 17:44
Xylenes, Total	ND		50	110	µg/Kg-dry	1	8/16/2021 17:44
Surr: 1,2-Dichloroethane-d4	*101*			*70-130*	*%REC*	1	8/16/2021 17:44
Surr: 4-Bromofluorobenzene	*96.9*			*70-130*	*%REC*	1	8/16/2021 17:44
Surr: Dibromofluoromethane	*96.9*			*70-130*	*%REC*	1	8/16/2021 17:44
Surr: Toluene-d8	*95.0*			*70-130*	*%REC*	1	8/16/2021 17:44
MOISTURE		Method: **SW3550C**					Analyst: **ALG**
Moisture	**28**		**0.10**	**0.10**	**% of sample**	1	8/12/2021 14:07

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

ALS Group, USA

Date: 18-Aug-21

Client: Barr Engineering Company

Work Order: 21081019

Project: Manifold - 223

QC BATCH REPORT

Batch ID: **182044** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK Sample ID: **DBLKS1-182044-182044** Units: **mg/Kg** Analysis Date: **8/17/2021 05:23 PM**

Client ID: Run ID: **GC8_210817A** SeqNo: **7674330** Prep Date: **8/16/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	ND	0.5	5.0								

LCS Sample ID: **DLCSS1-182044-182044** Units: **mg/Kg** Analysis Date: **8/17/2021 06:00 PM**

Client ID: Run ID: **GC8_210817A** SeqNo: **7674331** Prep Date: **8/16/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	8.09	0.5	5.0	10	0	80.9	70-120	0			

LCSD Sample ID: **DLCSDS1-182044-182044** Units: **mg/Kg** Analysis Date: **8/18/2021 07:03 AM**

Client ID: Run ID: **GC8_210817A** SeqNo: **7674352** Prep Date: **8/16/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	7.603	0.5	5.0	10	0	76	70-120	8.09	6.21	20	

The following samples were analyzed in this batch: 21081019-01B

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

Client: Barr Engineering Company
Work Order: 21081019
Project: Manifold - 223

QC BATCH REPORT

Batch ID: **181879** Instrument ID **VMS11** Method: **SW8260C**

MBLK Sample ID: **MBLK-181879-181879** Units: **µg/Kg-dry** Analysis Date: **8/16/2021 02:46 PM**
Client ID: Run ID: **VMS11_210816A** SeqNo: **7670503** Prep Date: **8/12/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	ND	15	30								
Ethylbenzene	ND	6.3	30								
m,p-Xylene	ND	40	60								
o-Xylene	ND	12	30								
Toluene	ND	8.2	30								
Xylenes, Total	ND	40	90								
Surr: 1,2-Dichloroethane-d4	*990*	0	0	*1000*	0	*99*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*992.5*	0	0	*1000*	0	*99.2*	*70-130*	0			
Surr: Dibromofluoromethane	*1000*	0	0	*1000*	0	*100*	*70-130*	0			
Surr: Toluene-d8	*960.5*	0	0	*1000*	0	*96*	*70-130*	0			

LCS Sample ID: **LCS-181879-181879** Units: **µg/Kg-dry** Analysis Date: **8/16/2021 01:40 PM**
Client ID: Run ID: **VMS11_210816A** SeqNo: **7670501** Prep Date: **8/12/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	972	15	30	1000	0	97.2	75-125	0			
Ethylbenzene	1011	6.3	30	1000	0	101	75-125	0			
m,p-Xylene	2031	40	60	2000	0	102	80-125	0			
o-Xylene	989	12	30	1000	0	98.9	75-125	0			
Toluene	992	8.2	30	1000	0	99.2	70-125	0			
Xylenes, Total	3020	40	90	3000	0	101	75-125	0			
Surr: 1,2-Dichloroethane-d4	*920.5*	0	0	*1000*	0	*92*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1014*	0	0	*1000*	0	*101*	*70-130*	0			
Surr: Dibromofluoromethane	*919.5*	0	0	*1000*	0	*92*	*70-130*	0			
Surr: Toluene-d8	*1010*	0	0	*1000*	0	*101*	*70-130*	0			

MS Sample ID: **21080953-01A MS** Units: **µg/Kg-dry** Analysis Date: **8/16/2021 09:48 PM**
Client ID: Run ID: **VMS11_210816A** SeqNo: **7670522** Prep Date: **8/12/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	1005	15	31	1026	0	97.9	75-125	0			
Ethylbenzene	931.3	6.5	31	1026	0	90.8	75-125	0			
m,p-Xylene	1874	41	62	2051	0	91.3	80-125	0			
o-Xylene	893.3	12	31	1026	0	87.1	75-125	0			
Toluene	948.2	8.4	31	1026	0	92.4	70-125	0			
Xylenes, Total	2767	41	92	3077	0	89.9	75-125	0			
Surr: 1,2-Dichloroethane-d4	*993.8*	0	0	*1026*	0	*96.9*	*70-130*	0			
Surr: 4-Bromofluorobenzene	*1074*	0	0	*1026*	0	*105*	*70-130*	0			
Surr: Dibromofluoromethane	*979*	0	0	*1026*	0	*95.4*	*70-130*	0			
Surr: Toluene-d8	*1008*	0	0	*1026*	0	*98.2*	*70-130*	0			

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

Client: Barr Engineering Company
Work Order: 21081019
Project: Manifold - 223

QC BATCH REPORT

Batch ID: **181879** Instrument ID **VMS11** Method: **SW8260C**

MSD Sample ID: **21080953-01A MSD** Units: **µg/Kg-dry** Analysis Date: **8/16/2021 10:10 PM**
Client ID: Run ID: **VMS11_210816A** SeqNo: **7670523** Prep Date: **8/12/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	813.8	15	31	1026	0	79.3	75-125	1005	21	30	
Ethylbenzene	733.8	6.5	31	1026	0	71.5	75-125	931.3	23.7	30	S
m,p-Xylene	1478	41	62	2051	0	72.1	80-125	1874	23.6	30	S
o-Xylene	709.2	12	31	1026	0	69.1	75-125	893.3	23	30	S
Toluene	749.7	8.4	31	1026	0	73.1	70-125	948.2	23.4	30	
Xylenes, Total	2187	41	92	3077	0	71.1	75-125	2767	23.4	30	S
Surr: 1,2-Dichloroethane-d4	*1018*	0	0	*1026*	0	*99.3*	*70-130*	993.8	*2.45*	*30*	
Surr: 4-Bromofluorobenzene	*1063*	0	0	*1026*	0	*104*	*70-130*	1074	*1.01*	*30*	
Surr: Dibromofluoromethane	*987.2*	0	0	*1026*	0	*96.2*	*70-130*	979	*0.835*	*30*	
Surr: Toluene-d8	*988.2*	0	0	*1026*	0	*96.3*	*70-130*	1008	*1.95*	*30*	

The following samples were analyzed in this batch: 21081019-01A

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

Client: Barr Engineering Company
Work Order: 21081019
Project: Manifold - 223

QC BATCH REPORT

Batch ID: **R324317** Instrument ID **MOIST** Method: **SW3550C**

MBLK Sample ID: **WBLKS-R324317** Units: **% of sample** Analysis Date: **8/12/2021 02:07 PM**
Client ID: Run ID: **MOIST_210812B** SeqNo: **7663381** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	ND	0.1	0.10								

LCS Sample ID: **LCS-R324317** Units: **% of sample** Analysis Date: **8/12/2021 02:07 PM**
Client ID: Run ID: **MOIST_210812B** SeqNo: **7663380** Prep Date: DF: **1**

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

DUP Sample ID: **21080735-01A DUP** Units: **% of sample** Analysis Date: **8/12/2021 02:07 PM**
Client ID: Run ID: **MOIST_210812B** SeqNo: **7663366** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	7.19	0.1	0.10	0	0	0	0-0	7.28	1.24	10	

DUP Sample ID: **21080834-01B DUP** Units: **% of sample** Analysis Date: **8/12/2021 02:07 PM**
Client ID: Run ID: **MOIST_210812B** SeqNo: **7663374** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	19.28	0.1	0.10	0	0	0	0-0	19.82	2.76	10	

The following samples were analyzed in this batch: 21081019-01C

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

21081019

Barr Engineering Co. Chain of Custody

Sample Origination State: ☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ TX ☐ UT ☒ WI ☐ Other: _____

COC Number: № 589587

COC 1 of 1

REPORT TO		INVOICE TO	
Company:	Barr Engineering	Company:	
Address:	325 S. Lake Ave	Address:	Same
Address:	Duluth, MN	Address:	
Name:	Ryan Erickson	Name:	Barr Engineering
email:	Rerickson@barr.com	email:	
Copy to:	BarrDM@barr.com / Jtorgldsen@barr.com	Barr Project No:	49161092.09 003 007
Project Name:	Manifold-223		

Location	Sample Depth Start	Sample Depth Stop	Sample Depth 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: VOA, VOA, 4 med-BTEX	Analysis Requested: Soil: 4 oz brat jars-DRO	Analysis Requested: Soil: % Solids	Preservative Code	Field Filtered Y/N	Comments
									F	A	A	-	-	
1. Manifold 223-Stockpile-2	—	—	—	8/10/21	13:45	S	N	5		2	2		1	BTEX, DRO, T. Moisture
2.														
3.														
4.														
5.														
6.														
7.														
8.														
9.														
10.														

ASAP TAT

JP

Matrix Code		Preservative Code	
GW =	Groundwater	A =	None
SW =	Surface Water	B =	HCl
WW =	Waste Water	C =	HNO_3
DW =	Drinking Water	D =	H_2SO_4
S =	Soil/Solid	E =	NaOH
SD =	Sediment	F =	MeOH
O =	Other	G =	$NaHSO_4$
		H =	$Na_2S_2O_3$
		I =	Ascorbic Acid
		J =	Zn Acetate
		K =	Other

BARR USE ONLY

Sampled by:	J. Torgldsen
Barr Proj. Manager:	Ryan Erickson
Barr DQ Manager:	JET
Lab Name:	ALS Environmental
Lab Location:	Holland MI

		On Ice?	Date	Time		Date	Time
Relinquished by:	Jtorgldsen	(Y) N	8/10/21	16:00	Received by:	FedEx	
Relinquished by:	FedEx	(Y) N	8/11/21	1600	Received by:		

Samples Shipped VIA: ☐ Ground Courier ☒ Air Carrier ☐ Sampler ☐ Other: _____

Air Bill Number: 3.0°C IR3

Lab WO: Temperature on Receipt (°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; **Scan and email:** a copy to BarrDM@barr.com for tracking and filing procedures

H:\RLG\STDFORMS\Chain of Custody Form 2015 RLG Rev. 01/30/2020

ALS Group, USA

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **11-Aug-21 16:00**

Work Order: **21081019** Received by: **KRW**

Checklist completed by Keith Wierenga (eSignature) 12-Aug-21 (Date) Reviewed by: Jodi Blouw (eSignature) 12-Aug-21 (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):	3.0/4.0 C	IR3	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	8/12/2021 8:12:02 AM		
Water - VOA vials have zero headspace?	Yes ☐	No ☐	No VOA vials submitted ☑
Water - pH acceptable upon receipt?	Yes ☐	No ☐	N/A ☑
pH adjusted?	Yes ☐	No ☐	N/A ☑
pH adjusted by:	-		

Login Notes:

Client Contacted: Date Contacted: Person Contacted:

Contacted By: Regarding:

Comments:

CorrectiveAction:

Water Documents

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

WLSSD

Western Lake Superior Sanitary District

Amended Letter of Approval

August 5, 2021

Nicholas Larabel, PG, CPG
Enbridge Energy
455 Leggitt Road
Marshall, MN 49068

Dear Mr. Larabel,

Based on data received August 2, 2021 and August 5, 2021, the Western Lake Superior Sanitary District (WLSSD) gives approval to Enbridge to discharge up to 100,000 gallons of petroleum-impacted wastewater at WLSSD. This approval letter is effective until November 30, 2021.

A hauler licensed to dispose of wastewater at WLSSD is required. Any petroleum on the water surface must be removed before water is transported. The hauler is required to leave a manifest in the drop box on Building 8, which states volume, date, time, description of water, and hauler. Please attempt to discharge during business hours 7am – 5pm.

The wastewater will be billed at the domestic rate of $1.9862/1000 gallons. There is one-time fee of $50 for the approval letter.

This approval letter does not release Enbridge or any consultant/contractor from any conditions/regulations set forth by MPCA or any other agency that regulated discharge. In addition, this approval does not release Enbridge or any consultant/contractor involved from any liabilities associated with conducting this discharge.

Thank you,

James Forsberg, Lab Leader
Western Lake Superior Sanitary District (WLSSD)
2626 Courtland St, Duluth, MN 55806
Direct 218-740-4853
james.forsberg@wlssd.com

WESTERN LAKE SUPERIOR SANITARY DISTRICT
LIQUID WASTE HAULER MONTHLY DISCHARGE REPORT

Company Name: NORTHLAND CONSTRUCTORS
Company Address: 4843 RICE LAKE RD - DULUTH MN, 55803
WLSSD Permit #: W127

Disposal Date at WLSSD	Disposal Time at WLSSD	Customer Name, Address and Phone	Customer Pick-up Date	Customer Pick-up Time	Gallons: Domestic Holding	Domestic Septic	Domestic Portable Toilet	Fats, Oils and Grease	Industrial Commercial Other
8/11/21	1315	ENBRIDGE SUPERIOR TERMINAL MAN-							2200
8/11/21	1515	6I FOLD 223/ LINE 1 / TRAP BOOSTER 1							2200
8/11/21	1710	(FIAT 2800)							2200
8/12/21	0900								2200
8/12/21	1100								2200
8/12/21	1300								2200
09/20/21	0900								2200
9/20/21	1005								2200
9/20/21	1125								2200
9/20/21	1320								2200
9/21/21	1050								2200
9/21/21	1200								2200
9/21/21	1310								2200
9/21/21	1435								2200
9/21/21	1530								2200
9/22/21	0905								2200
9/22/21	1035								2200
9/22/21	1155								2200
9/22/21	1315								2200
9/22/21	1445								2200
9/24/21	1305								2200
9/24/21	1420								2200
9/24/21	1545								2200
		CONTINUED ON NEXT PAGE							
			Totals:						50,600

~~Owner~~ HAULER
Signature: [signature] Date: 10/16/21

TOTALS BY MONTH:
AUGUST = 13,200 GALLONS
SEPTEMBER: 46,200 GALLONS
OCTOBER = 11,000 GALLONS

WESTERN LAKE SUPERIOR SANITARY DISTRICT
LIQUID WASTE HAULER MONTHLY DISCHARGE REPORT

Company Name: NORTHLAND CONSTRUCTORS
Company Address: 4843 RICE LAKE RD- DULUTH MN, 55803
WLSSD Permit #: W127

Disposal Date at WLSSD	Disposal Time at WLSSD	Customer Name, Address and Phone	Customer Pick-up Date	Customer Pick-up Time	Gallons				
					Domestic Holding	Domestic Septic	Domestic Portable Toilet	Fats, Oils and Grease	Industrial Commercial Other
9/29/21	0900	ENBRIDGE SUPERIOR TERMINAL MAN-							2200
9/29/21	1000	IFOLD 223 / LINE 1 / TRAP BOOSTER 1							2200
9/29/21	1125	(FIAT 2800)							2200
9/29/21	1230								2200
10/15/21	0850								2200
10/15/21	1005								2200
10/15/21	1110								2200
10/15/21	1210								2200
10/15/21	1325								2200
			Totals:						19,800

~~Owner~~ HAULER Signature: _____ Date: 10/16/21

30-Jul-2021

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

Re: **Manifold 223 Contaminated** Work Order: **21072323**

Dear Ryan,

ALS Environmental received 1 sample on 28-Jul-2021 09:00 AM 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,

Jodi Blouw

Electronically approved by: Jodi Blouw

Jodi Blouw

Report of Laboratory Analysis

Certificate No: WI: 399084510

ALS Group, USA

Date: 30-Jul-21

Client: Barr Engineering Company
Project: Manifold 223 Contaminated
Work Order: **21072323**

Work Order Sample Summary

Lab Samp ID	Client Sample ID	Matrix	Tag Number	Collection Date	Date Received	Hold
21072323-01	Manifold 223-Frac-1	Water		7/27/2021 11:15	7/28/2021 09:00	☐

ALS Group, USA

Date: 30-Jul-21

Client: Barr Engineering Company
Project: Manifold 223 Contaminated
WorkOrder: **21072323**

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: Manifold 223 Contaminated
Work Order: 21072323

Case Narrative

Samples for the above noted Work Order were received on 07/28/2021. 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: *30-Jul-21*

Client: Barr Engineering Company
Project: Manifold 223 Contaminated
Sample ID: Manifold 223-Frac-1
Collection Date: 7/27/2021 11:15 AM

Work Order: 21072323
Lab ID: 21072323-01
Matrix: WATER

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
DIESEL RANGE ORGANICS BY GC-FID		Method: **PUBL-SW-141**			Prep: PUBL-SW-141 / 7/29/21		Analyst: **SJB**
DRO (C10-C28)	ND		0.35	2.0	mg/L	1	7/30/2021 02:20
VOLATILE ORGANIC COMPOUNDS		Method: **SW8260C**					Analyst: **MF**
Benzene	**1.2**		**0.46**	**1.0**	**µg/L**	1	7/30/2021 07:14
Ethylbenzene	ND		0.34	1.0	µg/L	1	7/30/2021 07:14
m,p-Xylene	**4.4**		**0.81**	**2.0**	**µg/L**	1	7/30/2021 07:14
o-Xylene	ND		0.31	1.0	µg/L	1	7/30/2021 07:14
Toluene	ND		0.45	1.0	µg/L	1	7/30/2021 07:14
Xylenes, Total	**4.4**		**0.81**	**3.0**	**µg/L**	1	7/30/2021 07:14
Surr: 1,2-Dichloroethane-d4	*104*			*75-120*	*%REC*	1	7/30/2021 07:14
Surr: 4-Bromofluorobenzene	*96.7*			*80-110*	*%REC*	1	7/30/2021 07:14
Surr: Dibromofluoromethane	*100*			*85-115*	*%REC*	1	7/30/2021 07:14
Surr: Toluene-d8	*99.0*			*85-110*	*%REC*	1	7/30/2021 07:14

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

ALS Group, USA

Date: 30-Jul-21

Client: Barr Engineering Company

Work Order: 21072323

Project: Manifold 223 Contaminated

QC BATCH REPORT

Batch ID: **181056** Instrument ID **GC8** Method: **PUBL-SW-141**

MBLK Sample ID: **DBLKW1-181056-181056** Units: **mg/L** Analysis Date: **7/30/2021 01:06 AM**

Client ID: Run ID: **GC8_210729C** SeqNo: **7622139** Prep Date: **7/29/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	0.03913	0.017	0.10								J

LCS Sample ID: **DLCSW1-181056-181056** Units: **mg/L** Analysis Date: **7/30/2021 01:43 AM**

Client ID: Run ID: **GC8_210729C** SeqNo: **7622140** Prep Date: **7/29/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	0.1053	0.017	0.10	0.1	0	105	75-115	0			

LCSD Sample ID: **DLCSDW1-181056-181056** Units: **mg/L** Analysis Date: **7/30/2021 02:58 AM**

Client ID: Run ID: **GC8_210729C** SeqNo: **7622142** Prep Date: **7/29/2021** DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)	0.08722	0.017	0.10	0.1	0	87.2	75-115	0.1053	0	20	J

The following samples were analyzed in this batch: 21072323-01B

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

Client: Barr Engineering Company
Work Order: 21072323
Project: Manifold 223 Contaminated

QC BATCH REPORT

Batch ID: **R323201b** Instrument ID **VMS8** Method: **SW8260C**

MBLK Sample ID: **8V-BLKW2-210729-R323201b** Units: **µg/L** Analysis Date: **7/30/2021 02:37 AM**
Client ID: Run ID: **VMS8_210729C** SeqNo: **7622959** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	ND	0.46	1.0								
Ethylbenzene	ND	0.34	1.0								
m,p-Xylene	ND	0.81	2.0								
o-Xylene	ND	0.31	1.0								
Toluene	ND	0.45	1.0								
Xylenes, Total	ND	0.81	3.0								
Surr: 1,2-Dichloroethane-d4	*20.28*	0	0	*20*	0	*101*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*20.14*	0	0	*20*	0	*101*	*80-110*	0			
Surr: Dibromofluoromethane	*19.85*	0	0	*20*	0	*99.2*	*85-115*	0			
Surr: Toluene-d8	*19.47*	0	0	*20*	0	*97.4*	*85-110*	0			

LCS Sample ID: **8V-LCSW2-210729-R323201b** Units: **µg/L** Analysis Date: **7/30/2021 01:48 AM**
Client ID: Run ID: **VMS8_210729C** SeqNo: **7622957** Prep Date: DF: **1**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	19.39	0.46	1.0	20	0	97	70-130	0			
Ethylbenzene	19.31	0.34	1.0	20	0	96.6	76-123	0			
m,p-Xylene	37.89	0.81	2.0	40	0	94.7	75-130	0			
o-Xylene	18.93	0.31	1.0	20	0	94.6	76-127	0			
Toluene	20.41	0.45	1.0	20	0	102	76-125	0			
Xylenes, Total	56.82	0.81	3.0	60	0	94.7	76-127	0			
Surr: 1,2-Dichloroethane-d4	*19.8*	0	0	*20*	0	*99*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*20.69*	0	0	*20*	0	*103*	*80-110*	0			
Surr: Dibromofluoromethane	*19.9*	0	0	*20*	0	*99.5*	*85-115*	0			
Surr: Toluene-d8	*19.8*	0	0	*20*	0	*99*	*85-110*	0			

MS Sample ID: **21071979-11A MS** Units: **µg/L** Analysis Date: **7/30/2021 09:08 AM**
Client ID: Run ID: **VMS8_210729C** SeqNo: **7622995** Prep Date: DF: **50**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	12660	23	50	1000	12510	14.6	70-130	0			SEO
Ethylbenzene	1554	17	50	1000	533	102	76-123	0			
m,p-Xylene	5908	40	100	2000	3908	100	75-130	0			
o-Xylene	1134	16	50	1000	128.5	100	76-127	0			
Toluene	3606	22	50	1000	2657	94.9	76-125	0			
Xylenes, Total	7041	40	150	3000	4036	100	76-127	0			
Surr: 1,2-Dichloroethane-d4	*882.5*	0	0	*1000*	0	*88.2*	*75-120*	0			
Surr: 4-Bromofluorobenzene	*1002*	0	0	*1000*	0	*100*	*80-110*	0			
Surr: Dibromofluoromethane	*1014*	0	0	*1000*	0	*101*	*85-115*	0			
Surr: Toluene-d8	*994.5*	0	0	*1000*	0	*99.4*	*85-110*	0			

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

Client: Barr Engineering Company
Work Order: 21072323
Project: Manifold 223 Contaminated

QC BATCH REPORT

Batch ID: **R323201b** Instrument ID **VMS8** Method: **SW8260C**

MSD Sample ID: **21071979-11A MSD** Units: **µg/L** Analysis Date: **7/30/2021 09:25 AM**
Client ID: Run ID: **VMS8_210729C** SeqNo: **7622997** Prep Date: DF: **50**

Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Benzene	12790	23	50	1000	12510	28	70-130	12660	1.05	30	SEO
Ethylbenzene	1576	17	50	1000	533	104	76-123	1554	1.41	30	
m,p-Xylene	5840	40	100	2000	3908	96.6	75-130	5908	1.16	30	
o-Xylene	1150	16	50	1000	128.5	102	76-127	1134	1.4	30	
Toluene	3622	22	50	1000	2657	96.4	76-125	3606	0.429	30	
Xylenes, Total	6989	40	150	3000	4036	98.4	76-127	7041	0.741	30	
Surr: 1,2-Dichloroethane-d4	*876*	0	0	*1000*	0	*87.6*	*75-120*	882.5	*0.739*	*30*	
Surr: 4-Bromofluorobenzene	*972.5*	0	0	*1000*	0	*97.2*	*80-110*	1002	*2.94*	*30*	
Surr: Dibromofluoromethane	*1014*	0	0	*1000*	0	*101*	*85-115*	1014	*0.0493*	*30*	
Surr: Toluene-d8	*977.5*	0	0	*1000*	0	*97.8*	*85-110*	994.5	*1.72*	*30*	

The following samples were analyzed in this batch: 21072323-01A

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

21072323

Barr Engineering Co. Chain of Custody

Sample Origination State: ☐ CO ☐ MI ☐ MN ☐ MO ☐ ND ☐ TX ☐ UT ☒ WI ☐ Other: _____

COC Number: № 589923

COC 1 of 1

REPORT TO	INVOICE TO
Company: Barr Engineering	Company:
Address: 325 South Lake Ave.	Address: Barr Engineering
Address: Duluth, MN	Address:
Name: Ryan Erickson	Name: -Same-
email: rerickson@barr.com	email:
Copy to: BarrDM@barr.com / Jtaraldsen@barr.com	
Project Name: Manifold 223 Contamination	Barr Project No: 49161092.09 003001

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 = Zn Acetate; K = Other

Location	Sample Depth Start	Stop	Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform MS/MSD Y (N)	Total Number Of Containers	Analysis Requested: Water — 1000ml Amber +HCl - DRO	1000ml Amber - Hold	40ml vials with BTEX + HCl	125ml metals +HNO3	Soil — % Solids	Preservative Code	Field Filtered Y/N	Comments
									B	A	B	C				
1. Manifold 223 - FRAC-1	—			7/27/21	11:15	W	N	10	2	1	6	1				DRO, BTEX (Hold metals and 1000ml unpreserved sample)
2.																
3.																
4.																
5.																
6.																
7.																
8.																
9.																
10.																

ASAP TAT JM

IR3 2.1°C pH<2

BARR USE ONLY							
Sampled by: J. Taraldsen	Relinquished by:	On Ice? Y (N)	Date 7/27/21	Time 17:15	Received by: FED EX	Date	Time
Barr Proj. Manager: Ryan Erickson	Relinquished by: FED EX	On Ice? Y N	Date 7/28/21	Time 0900	Received by:	Date	Time
Barr DQ Manager: Jim Taraldsen	Samples Shipped VIA: ☐ Ground Courier ☒ Air Carrier				Air Bill Number:		
Lab Name: MLS Environmental	☐ Sampler ☐ Other: _____						
Lab Location: Holland, MI	Lab WO:	Temperature on Receipt (°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; Scan and email: a copy to BarrDM@barr.com for tracking and filing procedures

H:\RLG\STD\FORMS\Chain of Custody Form 2015 RLG Rev. 01/30/2020

ALS Group, USA

Sample Receipt Checklist

Client Name: **BARRENG-MN** Date/Time Received: **28-Jul-21 09:00**

Work Order: **21072323** Received by: **DS**

Checklist completed by Diane Shaw (eSignature) 28-Jul-21 (Date) Reviewed by: Jodi Blouw (eSignature) 29-Jul-21 (Date)

Matrices: Water

Carrier name: FedEx

Shipping container/cooler in good condition?	Yes ☑	No ☐	Not Present ☐
Custody seals intact on shipping container/cooler?	Yes ☐	No ☐	Not Present ☑
Custody seals intact on sample bottles?	Yes ☐	No ☐	Not Present ☑
Chain of custody present?	Yes ☑	No ☐	
Chain of custody signed when relinquished and received?	Yes ☑	No ☐	
Chain of custody agrees with sample labels?	Yes ☑	No ☐	
Samples in proper container/bottle?	Yes ☑	No ☐	
Sample containers intact?	Yes ☑	No ☐	
Sufficient sample volume for indicated test?	Yes ☑	No ☐	
All samples received within holding time?	Yes ☑	No ☐	
Container/Temp Blank temperature in compliance?	Yes ☑	No ☐	
Sample(s) received on ice?	Yes ☑	No ☐	
Temperature(s)/Thermometer(s):	2.1/3.1 c	IR3	
Cooler(s)/Kit(s):			
Date/Time sample(s) sent to storage:	7/28/2021 11:27:28 AM		
Water - VOA vials have zero headspace?	Yes ☑	No ☐	No VOA vials submitted ☐
Water - pH acceptable upon receipt?	Yes ☑	No ☐	N/A ☐
pH adjusted?	Yes ☐	No ☑	N/A ☐
pH adjusted by:	-		

Login Notes:

Client Contacted: Date Contacted: Person Contacted:

Contacted By: Regarding:

Comments:

CorrectiveAction: