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# Technical Memorandum

**To:** Karl Beaster, Enbridge Energy
**From:** Ryan Erickson and Chris Goscinak
**Subject:** Superior Terminal Pipe Rack Associated Projects - Historical Crude Oil Impacts
**Date:** February 5, 2014
**Project:** 49161238

This memorandum summarizes the field screening, analytical sampling and waste management assistance conducted by Barr Engineering (Barr) at the request of Enbridge Energy (Enbridge) in the response to the discovery of historical, crude oil impacted soil and water during projects associated with the Enbridge Superior Terminal (Terminal) pipe rack construction project in Superior, Wisconsin (Figure 1).

## Background and Response Activities

Projects associated with the construction of the Enbridge Superior Terminal pipe rack structure (Figure 2) were completed between 2012 and 2014. The purpose of the pipe rack is to support aboveground piping infrastructure associated with newly constructed tanks located in the northeast corner of the terminal. The completed pipe rack structure will be approximately 1,500 feet long by 40 feet wide by 40 feet high. Additional projects associated with the pipe rack construction included the expansion of the central manifold and improvements to the surrounding terminal infrastructure (i.e. waterlines, hydrants, structures).

Crude oil impacted soil and/or water was encountered by Enbridge contractors in five pipe rack-associated project excavations (Figure 3) between the August 2012 and August 2013. The impacted excavations are identified in this memo as: geotechnical investigation hydrovac borings, water valve replacement excavation, fire hydrant replacement excavation, pipe rack footing excavations, and the central manifold expansion excavation. Enbridge Environment was notified by the contractor when crude oil impacts were encountered.

Enbridge requested that Barr complete the following activities during the pipe rack project:

- identify historical crude oil releases in the pipe rack project area that are documented in the Wisconsin Department of Natural Resources (WDNR) Bureau for Remediation and Redevelopment Tracking System (BRRTS) database
- propose a modified pipe rack project contaminated excavation sampling plan to WDNR
- assess the environmental site conditions
- segregate excavated crude oil impacted soil from unimpacted soil
- identify crude oil impacted water, if present
- assist with the off-site disposal coordination and documentation of contaminated soil and water
- document the residual crude oil impacts left in place, when applicable

Enbridge indicated that the crude oil impacts discovered during the pipe rack project were likely historical based on the location and characteristics of the contaminated soil. Barr checked the WDNR BRRTS database and six sites were identified near the pipe rack project location; however, none of the BRRTS sites are within the immediate footprint of the pipe rack project. Each BRRTS site has received closure. BRRTS site activity numbers and closure dates are listed below and the site locations are shown on Figure 2:

- WDNR BRRTS Activity #02-16-000512 - Lakehead Pipeline – Pump St: A 1988 release of 174 barrels of crude oil. The site was closed on December 1, 1997;
- WDNR BRRTS Activity #02-16-000161 - Murphy Oil – Lakehead Tank Fac: A 1990 release of 1 barrel of crude oil. The site was closed on March 9, 1995;
- WDNR BRRTS Activity #02-16-183249 - Lakehead Pipeline – Manifold 3: A 1998 crude oil release. The site was closed on April 15, 2004;
- WDNR BRRTS Activity #02-16-552700 - Enbridge Energy – Tank 9 Pressure Line: A 2008 release of 115 barrels of crude oil. The site was closed on November 18, 2008;
- WDNR BRRTS Activity #02-16-558987 - Enbridge Energy – Tank 9: Historical hydrocarbon impacts discovered in a maintenance excavation in 2012. The site was closed on September 4, 2012;
- WDNR BRRTS Activity #02-16-558988 – Office Excavation: Historical hydrocarbon impacts discovered in a construction excavation in 2012. The site was closed on September 4, 2012.

Given the large scope and extended schedule of the pipe rack project, Barr prepared a modified soil sampling plan (Attachment A; Memo dated August 12, 2013) for Enbridge for submittal to the WDNR. Enbridge submitted the modified sampling plan revisions to the WDNR on August 23, 2013 and the WDNR approved the modifications on August 26, 2013 (Attachment A; email dated August 26, 2013). All soil sampling events described in this memo occurred prior to the final approval of the modified sampling plan and the actual laboratory parameters analyzed may vary from what is described in the modified soil sampling plan proposal.

## Field Methods

Barr was onsite as needed to provide environmental assistance during the pipe rack construction activities in 2012 and 2013. Barr field screened excavated soil for the presence of organic vapors using a photoionization detector (PID) and documented other potential indicators of crude oil impacts such as odor, discoloration and sheen (Attachment B). Excavated soil with PID headspace readings greater than 10 parts per million (ppm), or other evidence of crude oil impacts, was segregated and transported to the Superior Terminal Soil Management Area (SMA) (Figure 2) for storage until it could be characterized and approved for off-site disposal.

In addition to field screening soil, Barr field screened water present within the excavation for the presence of a sheen or free-product. If a petroleum sheen or free-product were observed on water within the excavation, the water was considered contaminated and it was containerized until it could be characterized and approved for off-site disposal.

After construction excavation activities were completed, Barr collected field screening soil samples from the sidewalls and base of excavation, when possible, to identify whether residual soil impacts were present. Residual soil impacts were considered present if a headspace greater than 10 ppm was identified. If residual impacts were identified, and the impacted soil could not be excavated, analytical soil samples were typically collected from the sidewall to document residual soil impacts. Soil samples were submitted to Pace Analytical for some or all of the following laboratory analyses: petroleum volatile organic compounds (PVOCs), diesel range organics (DRO), and polycyclic aromatic hydrocarbons (PAH). Analytical results from the sidewall samples were input into the WDNR Web Calculator (Table 1) to compare analyte detections to groundwater residual contaminant levels (RCL) and industrial direct contact RCL and to determine whether the soil passes the Cumulative Hazard Index criteria described in

WDNR guidance document PUB-RR-890. Soil sample locations are shown on Figure 3. Field screening data and excavation documentation laboratory reports are provided in Attachment B and C, respectively.

## Results

Excavation, field screening and excavation documentation analytical sampling activities at the identified contaminated locations are described below. The project sites and analytical sampling locations are shown on Figure 3 and field screening locations, results and analytical sampling locations are included in Attachment B. Analytical sampling results are summarized in Table 1 and the laboratory reports are included in Attachment C.

*Geotechnical Investigation Hydrovac Borings- August and October 2012*

Crude oil impacted soil with a hydrocarbon sheen and odor was encountered within multiple hydrovac boring excavations near the western corner of the Tank 5 containment basin (LHB Investigation, Barr Investigation) (Photos 1 and 2). The hydrovac borings were advanced to a depth of approximately 10 feet below ground surface (bgs) to identify the locations of buried infrastructure prior to the advancement of deeper geotechnical borings. The contaminated soil removed from the hydrovac borings was stockpiled in the SMA until it could be characterized and approved for off-site disposal. Field screening and analytical soil sample collection from the hydrovac boring extents was not possible due to the excavation size.

*Water Valve Replacement Excavation – November 2012*

Crude oil impacted soil with a hydrocarbon sheen and odor and water with a sheen were encountered in a water valve replacement/maintenance excavation near the southeast corner of the Terminal office building (Photos 3 and 4). The maintenance work was completed prior to the construction of an office trailer that would be used by Enbridge employees. The excavation extents were approximately 8 feet long by 8 feet wide by 6 feet deep (Attachment B). Impacted soil within the excavation was observed between approximately 4 feet and 6 feet bgs and had elevated headspace detections (>150 ppm), a sheen and hydrocarbon odor. Hydrocarbon impacts were not identified in the soil between 0 and 4 feet bgs. The contaminated soil excavated for the water valve replacement was stockpiled in the SMA until it could be characterized and approved for off-site disposal. Hydrocarbon impacted water was pumped into a tanker until it could be characterized and approved for off-site disposal.

Analytical soil samples *Office Valve S-1* and *Office Valve S-2* were collected from the excavation sidewalls for laboratory analysis of DRO and PVOCs to document residual soil impacts left in place. Analyte concentrations in both samples were below the groundwater RCL and the industrial direct contact RCL and passed the Cumulative Hazard Index criteria.

After the water valve was replaced, the excavation was backfilled with clean fill and a terminal office trailer was constructed at the location of the completed water valve replacement excavation (Photo 5).

*Fire Hydrant Replacement Excavation – November 2012*

Crude oil impacted soil with a hydrocarbon sheen and odor were encountered in a fire hydrant replacement excavation (Photos 6 and 7). The fire hydrant was being replaced prior to the start of nearby pipe rack construction activity. The excavation extents were approximately 10 feet long by 8 feet wide by 7 feet deep. Impacted soil within the excavation was identified between approximately 3 feet and 5 feet bgs and had elevated headspace detections (203 ppm), a hydrocarbon sheen and odor. Hydrocarbon impacts were not identified between 0 and 3 feet bgs. The contaminated soil removed from the fire hydrant excavation was stockpiled in the SMA until it could be characterized and approved for off-site disposal. Analytical samples could not be collected from the excavation due to the presence of terminal infrastructure.

The excavation was backfilled with clean fill after the fire hydrant was replaced.

*Pipe Rack Footing Excavations – July 2013*

Crude oil impacted soil was encountered in three pipe rack-structure footing excavations between terminal crude oil storage Tanks 5 and 9 (Photos 8 and 9). Each excavation was approximately 8 feet long by 8 feet wide by 8 feet deep. Impacted soil within the excavation was identified at approximately 4 feet bgs and had elevated headspace detections between 18.4 ppm and 199 ppm, dark discoloration and a hydrocarbon sheen and odor. The contaminated soil removed from the pipe rack footing excavations was stockpiled in the SMA until it could be characterized and approved for off-site disposal.

Analytical soil samples *2013 Pipe Rack-S-1, 2013 Pipe Rack-S-2* and *2013 Pipe Rack-S-3* were collected from the excavation sidewalls for laboratory analysis of PVOC's to document residual soil impacts left in place. A PAH analysis were also run on *2013 Pipe Rack-S-3*. Analyte concentrations in *2013 Pipe Rack-*

*S-3* exceeded the WDNR groundwater RCL for xylene (3.4 mg/kg), 1,2,4-Trimethyl benzene (5.4 mg/kg), 1,3,5-Trimethyl benzene (3.5 mg/kg) and Chrysene (0.291 mg/kg). Analyte concentrations from all three samples were below the industrial direct contact RCL and passed the Cumulative Hazard Index criteria.

Upon completion of the pipe rack footing construction, the excavations were backfilled with clean fill in preparation for the pipe rack structure construction activities (Photos 10 and 11).

*Central Manifold Expansion Excavation, July – August 2013*

Crude oil impacted soil and water were encountered within the central manifold expansion excavation (Photos 12 and 13). The excavation was approximately 100 feet long by 100 feet wide by 6 feet deep. Impacted soil within the excavation was identified along buried manifold infrastructure approximately 4 feet to 6 feet bgs. Crude oil impacted soil was identified by its dark discoloration and hydrocarbon odor. Based on visual observations, it appeared that most of the crude oil impacted soil was removed during construction excavation activities. The contaminated soil removed from the central manifold expansion excavation was stockpiled in the SMA until it could be characterized and approved for off-site disposal. Barr field screened the sidewalls of the final manifold excavation on August 1, 2013 and only one field screening point exceeded 10 ppm (sample R-15 with a headspace of 26.8 ppm). The excavation bottom could not be field screened because a new 2-foot thick concrete foundation had been installed in the base of the excavation (Photo 14).

Analytical sample *2013 Pipe Rack-S-4* was collected from the excavation sidewall, near field screening point R-15, for laboratory analysis of PVOCs and PAHs to document residual hydrocarbon impacts were left in place (Figure 3; Attachment B). Analyte concentrations of Chrysene (0.126 mg/kg) exceeded the groundwater RCL. All other analyte concentrations were below the groundwater RCL and the industrial direct contact RCL and passed the Cumulative Hazard Index criteria.

Upon completion of the central manifold expansion construction activities, the area around the new manifold structure was backfilled with clean fill.

## Discussion

Analyte concentrations detected in the Superior Terminal pipe rack associated project excavations were below the industrial direct contact RCL and passed the Cumulative Hazard Index criteria (Table 1). Analyte concentrations were below the groundwater RCL, with the following exceptions: the pipe rack footing soil sample *2013 Pipe Rack-S-3* exceeded groundwater RCL's for xylene (3.4 mg/kg), 1,2,4-trimethylbenzene (5.4 mg/kg), 1,3,5-trimethylbenzene (3.5 mg/kg) and chrysene (0.291 mg/kg) and the central manifold soil sample *2013 Pipe Rack-S-4* exceeded the groundwater RCL for chrysene (0.126 mg/kg).

Further excavation of the hydrocarbon soil was limited due to the presence of the terminal infrastructure. Following the completion of the above projects, the excavations were backfilled with clean fill and no hydrocarbon impacted soil is exposed at the ground surface.

## Waste Disposal Coordination and Documentation

Barr collected seven analytical waste characterization samples (*Pipe Rock, Office Water-Stockpile-1, 2013 PIPERACK STOCKPILE 1, 2013 Pipe Rack-stockpile-2, 2013 PIPE RACK STOCKPILE-3, 2013 PIPE RACK Stockpile 4*) from crude oil impacted stockpiles associated with the pipe rack project for laboratory analysis at Legend Technical Services and Pace Analytical Services. The samples were analyzed for DRO and benzene, toluene, ethylbenzene, and xylenes (BTEX). Two waste profile applications were submitted with the associated laboratory results to the Shamrock Landfill near Cloquet, Minnesota for the different waste streams. A total of 93.09 tons of impacted soil from the geotechnical investigation hydrovac borings, the water valve replacement excavation and the fire hydrant replacement excavation was accepted under waste profile #CL12-0067 in November and December of 2012. A total of 776.58 tons of soil from the central manifold excavation and pipe rack foundation excavations was accepted under waste profile #CL13-0027 between June and August of 2013. Waste characterization sample laboratory reports, Shamrock Landfill Waste Profile documentation and landfill hauling summaries are included in Attachment D.

Hydrocarbon impacted water with a rainbow sheen was removed from project excavations (the water valve replacement excavation and the central manifold expansion excavation) and containerized in tanker trailers. Analytical water samples *Hydrant Water-Waste-1* and *Pipe Rack Water-1* were collected from their respective waste streams and submitted to Legend Technical Services for laboratory analysis of DRO and BTEX. The laboratory report was submitted as part of a water disposal request to the Western

Lake Superior Sanitary District (WLSSD) water treatment facility in Duluth, Minnesota. The water treatment request was approved on November 15, 2012 (water valve replacement excavation) and August 12, 2013 (central manifold excavation). The project contractor facilitated the transport and disposal of the water. The WLSSD water disposal approval letters and the associated laboratory reports are included in Attachment D.

## Conclusions and Recommendations

Crude oil impacted soil and water were encountered during excavation activities associated with the construction of the Superior Terminal pipe rack structure. The contaminated soil was excavated to the extent possible; however, some hydrocarbon impacted soil was left in place due to the presence of terminal infrastructure. Analyte concentrations in the residual hydrocarbon impacted soil did not exceed industrial direct contact RCLs, passed the Cumulative Hazard Index Criteria and has been covered with clean fill and/or permanent terminal infrastructure. The presence of backfill and infrastructure and employee awareness will prevent direct contact exposure.

The groundwater pathway for the Superior Terminal is currently being reviewed by the WDNR on a case by case site-wide basis. If the WDNR agrees that the risk to the groundwater pathway associated with these historical releases can be addressed using the site-wide approach, no further response action for groundwater or documentation for the WDNR will be required. Assuming a site-wide GIS registry is established for the terminal, the figures and tables attached to this memo can be used to update the registry.

## Attachments:

| | |
|---|---|
| Photos | 1 through 14 |
| Figure 1 | Pipe Rack Site Location |
| Figure 2 | Pipe Rack Project Site Layout |
| Figure 3 | Pipe Rack Project Sample Locations |
| Table 1 | Soil Analytical Data Summary |
| Attachment A | WDNR Documentation and Communications |
| Attachment B | Enbridge Site Investigation Field Sampling and Screening Logs |
| Attachment C | Pace Analytical Laboratory Reports for Excavation Soil Samples |
| Attachment D | Waste Disposal Documentation |

## Photos:

**Photo 1**

**Photo 2**

**Photo 1**: Hydrovac excavation locations associated with the LHB geotechnical borings in the Tank 5 containment basin. Photo taken facing east.
**Photo 2:** Hydrovac excavation associated with the LHB geotechnical borings in the Tank 5 containment basin.

**Photo 3**

**Photo 4**

**Photo 3**: Water valve replacement excavation. Photo taken facing southwest.
**Photo 4**: Water valve replacement excavation. The replaced water valve is shown in the center of the photo.

**Photo 5**: The Superior Terminal office trailer constructed after the completion of the water valve replacement excavation. Photo taken facing west.

**Photo 6**

**Photo 7**

**Photo 6**: Fire hydrant replacement excavation. Photo taken facing northeast.
**Photo 7**: Fire hydrant replacement excavation. Contaminated soil was encountered in the excavation and was left in place due to inaccessibility resulting from the presence of terminal infrastructure.

**Photo 8**

**Photo 9**

**Photo 8**: Pipe rack footing excavation facing northeast.
**Photo 9**: Pipe rack footing excavation facing north.


**Photo 10**


**Photo 11**

**Photo 10**: The pipe rack frame located in the area where the crude oil impacts were discovered during the geotechnical investigations and the pipe rack footing excavations. Tank 5 is shown behind the pipe rack frame. Photo taken facing east.
**Photo 11**: The pipe rack frame. Photo taken facing northeast from near fire hydrant replacement excavation location.

**Photo 12**

**Photo 13**

**Photo 12**: Crude oil impacted soil and water in the central manifold expansion excavation. Photo taken facing northwest.
**Photo 13**: Central manifold expansion excavation water with a sheen.

**Photo 14**: The concrete base of the central manifold. Photo taken facing north.

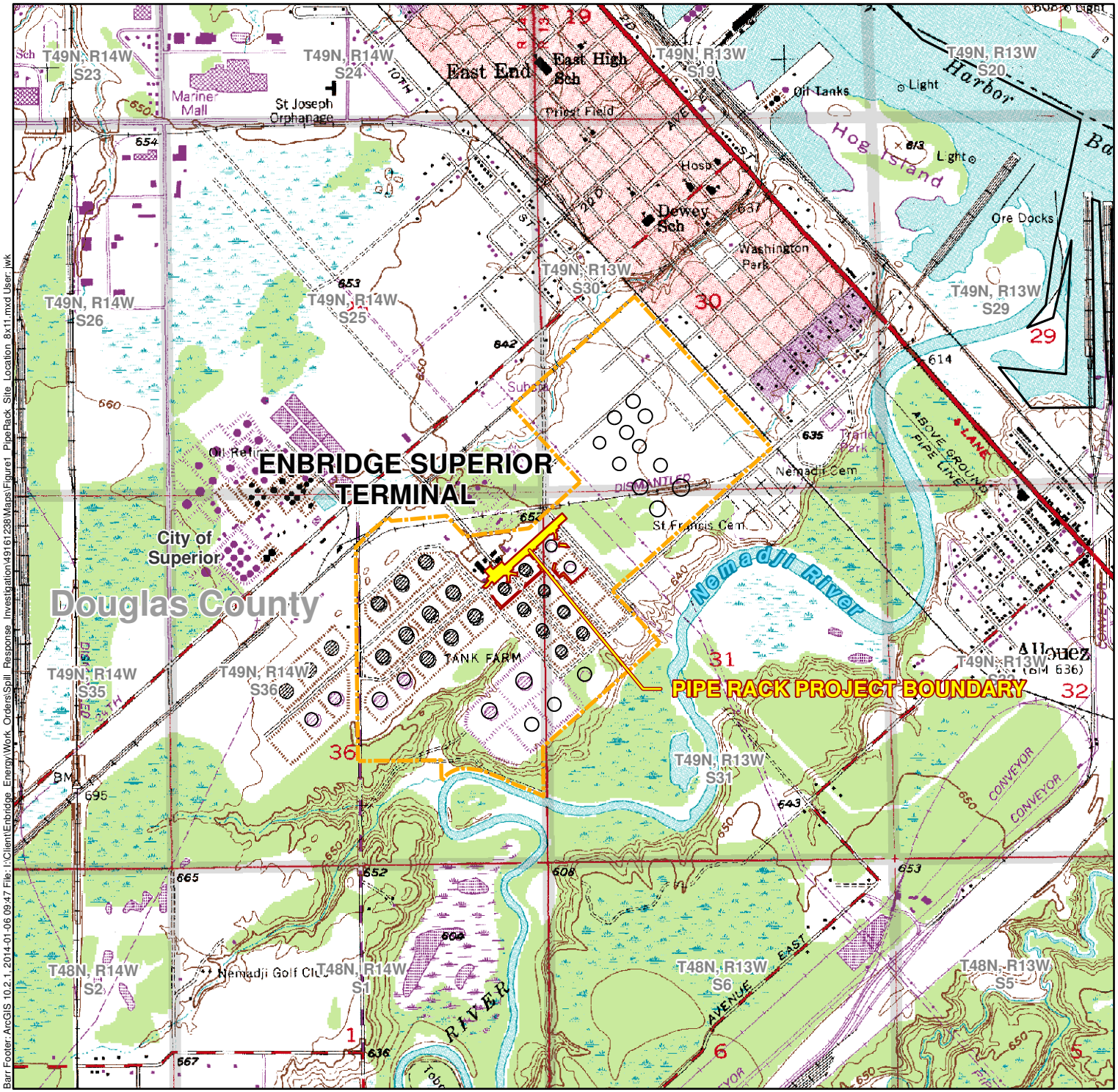
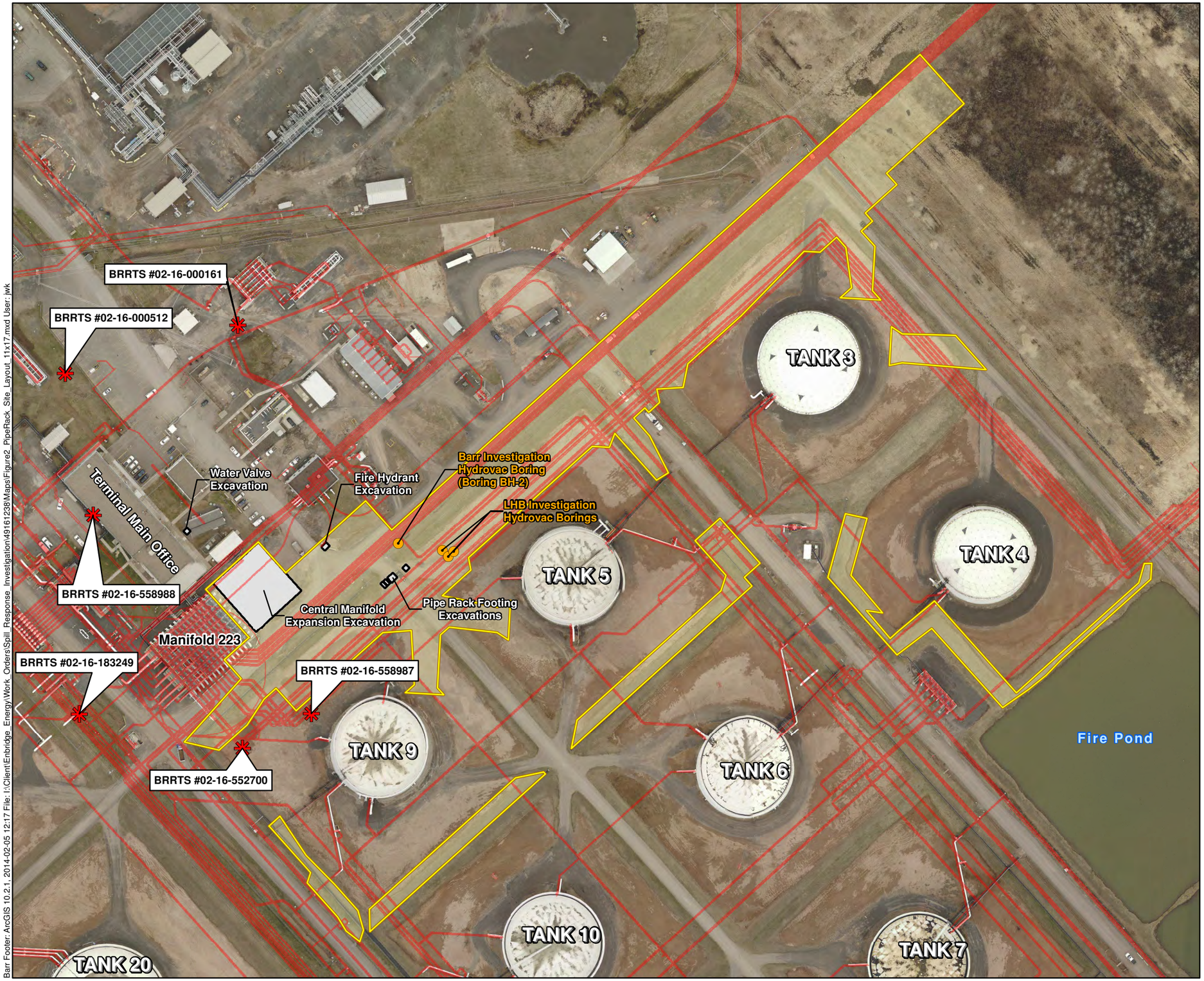



Figure 1

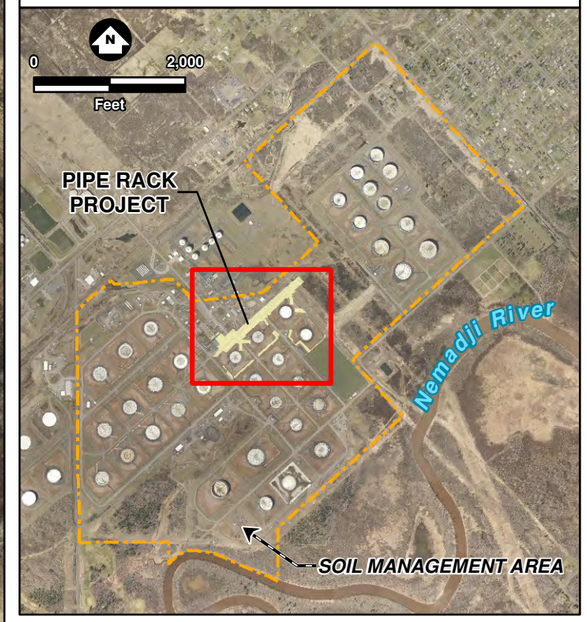
**PIPE RACK SITE LOCATION**
**SUPERIOR TERMINAL**
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE




Barr Footer: ArcGIS 10.2.1, 2014-02-05 12:17 File: I:\Client\Enbridge_Energy\Work_Orders\Spill_Response_Investigation\49161238\Maps\Figure2_PipeRack_Site_Layout_11x17.mxd User: jwk

## ENBRIDGE SUPERIOR TERMINAL




- WDNR BRRTS Site Locations
- Geotechnical Investigation Hydrovac Borings with Crude Oil Impacts
- Excavation Extents
- Pipe Rack Project Boundary
- Pipeline Infrastructure
- Terminal Property Boundary

N

0 150 300

Feet

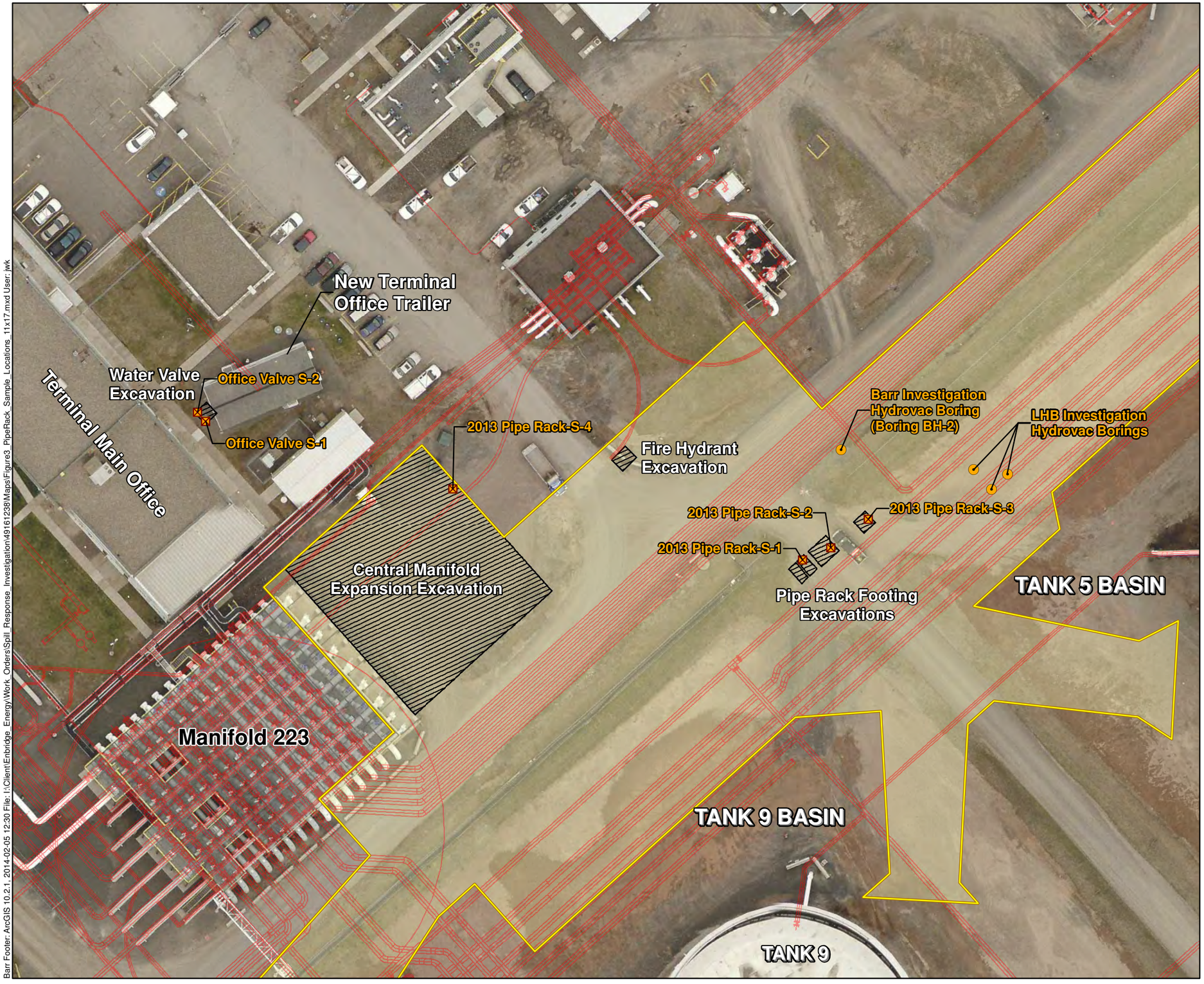
1 Inch = 150 Feet

*Douglas County Imagery Circa May, 2013*

Figure 2

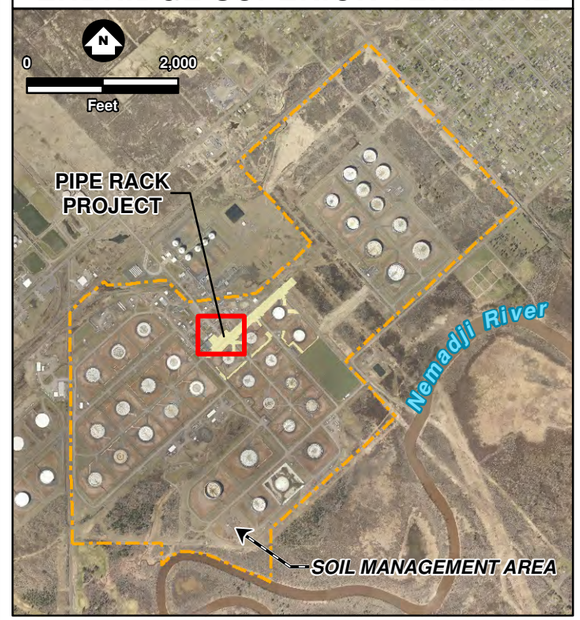
**PIPE RACK PROJECT SITE LAYOUT**
**SUPERIOR TERMINAL**
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE






## ENBRIDGE SUPERIOR TERMINAL




- Sample Locations
- Geotechnical Investigation Hydrovac Borings with Crude Oil Impacts
- Excavation Extents
- Pipe Rack Project Boundary
- Pipeline Infrastructure
- Terminal Property Boundary

N

0 50 100

Feet

1 Inch = 50 Feet

*Douglas County Imagery Circa May, 2013*

Figure 3

**PIPE RACK PROJECT SAMPLE LOCATIONS**
**SUPERIOR TERMINAL**
Enbridge Energy, L.P.
Superior, Wisconsin

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# Table 1
## Soil Analytical Data Summary
## Pipe Rack Associated Projects
## Enbridge Energy Terminal - Superior, Wisconsin
## Units, mg/kg (unless otherwise noted)

| | | Parameter | Moisture | Benzene | Ethyl benzene | Toluene | Xylene, total | 1,2,4-Trimethyl benzene | 1,3,5-Trimethyl benzene | Diesel Range Organics | Acenaphthene | Acenaphthylene | Anthracene | Benz(a) anthracene | Benzo(a) pyrene | Benzo(b) fluoranthene | Benzo(g,h,i) perylene | Benzo(k) fluoranthene |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | **Effective Date** | **Exceedance Key** | | | | | | | | | | | | | | | | |
| **Groundwater RCL** | | **Bold** | | 0.0051 | 0.785 | 0.5536 | **1.97 XYL** | **1.3793 TR** | **1.3793 TR** | | | | 196.7442 | | 0.47 | 0.48 | | |
| **Industrial Direct Contact RCL** | 05/01/2012 | No Exceed | | 7.41 | 37 | 818 | 258 | 219 | 182 | | 33000 | 487 | 100000 | 2.11 | 0.211 | 2.11 | | 21.1 |
| **Location** | **Date** | **Depth (ft)** | | | | | | | | | | | | | | | | |
| Office Valve-S-1 | 11/15/2012 | 1 | 24.1 % | < 0.067 | < 0.067 | < 0.067 | < 0.20 | < 0.067 | < 0.067 | < 12.3 | -- | -- | -- | -- | -- | -- | -- | -- |
| Office Valve-S-2 | 11/15/2012 | 6 | 32.7 % | < 0.078 | < 0.078 | < 0.078 | < 0.23 | 0.56 | 0.22 | 222 | -- | -- | -- | -- | -- | -- | -- | -- |
| 2013 Pipe Rack-S-1 | 7/09/2013 | 4 | 24.0 % | < 0.071 | < 0.071 | < 0.071 | < 0.21 | < 0.071 | < 0.071 | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 2013 Pipe Rack-S-2 | 7/09/2013 | 4 | 21.8 % | < 0.15 * | 0.22 * | < 0.15 * | 1.2 * | 0.85 * | 0.58 * | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 2013 Pipe Rack-S-3 | 7/09/2013 | 4 | 20.4 % | < 0.63 * | < 0.63 * | < 0.63 * | **3.4** * | **5.4** * | **3.5** * | -- | 0.0707 * | 0.0688 * | < 0.0628 * | 0.163 * | 0.147 * | 0.207 * | 0.0901 * | 0.0729 * |
| 2013 Pipe Rack-S-4 | 8/01/2013 | 3.5 | 26.4 % | < 0.065 | < 0.065 | < 0.065 | < 0.20 | < 0.065 | < 0.065 | -- | < 0.0135 | < 0.0135 | 0.0385 | 0.117 | 0.0910 | 0.122 | 0.0458 | 0.0483 |

[1]WDNR RCL Determinations based on guidance criteria described in WDNR document PUB-RR-890. Hazard index is based a cumulative direct contact standard.

TR - Based on 1,2,4-Trimethylbenzene and 1,3,5-Trimethylbenzene combined.

XYL - Based on Xylenes (m-, o-, p- combined).

* - Estimated value, QA/QC criteria not met.

**Table 1**
**Soil Analytical Data Summary**
**Pipe Rack Associated Projects**
**Enbridge Energy Terminal - Superior, Wisconsin**
**Units, mg/kg (unless otherwise noted)**

| | | Parameter | Chrysene | Dibenz(a,h) anthracene | Fluoranthene | Fluorene | Indeno(1,2,3-cd) pyrene | Naphthalene | Phenanthrene | Pyrene | WDNR RCL Determinations[1] | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | Exceedance Count | Hazard Index | Cumulative Cancer Risk | Pass or Fail |
| | **Effective Date** | **Exceedance Key** | | | | | | | | | | | | |
| **Groundwater RCL** | | **Bold** | **0.0725** | | 44.4089 | 7.4074 | | 0.3294 | | 27.2362 | | | | |
| **Industrial Direct Contact RCL** | 05/01/2012 | No Exceed | 211 | 0.211 | 22000 | 22000 | 2.11 | 26 | 115 | 16500 | 0 | 1.0 | 0.00001 | Pass |
| **Location** | **Date** | **Depth (ft)** | | | | | | | | | | | | |
| Office Valve-S-1 | 11/15/2012 | 1 | -- | -- | -- | -- | -- | -- | -- | -- | 0 | 0.0003 | 1.1E-08 | Pass |
| Office Valve-S-2 | 11/15/2012 | 6 | -- | -- | -- | -- | -- | -- | -- | -- | 0 | 0.0017 | 1.3E-08 | Pass |
| 2013 Pipe Rack-S-1 | 7/09/2013 | 4 | -- | -- | -- | -- | -- | -- | -- | -- | 0 | 0.0004 | 1.2E-08 | Pass |
| 2013 Pipe Rack-S-2 | 7/09/2013 | 4 | -- | -- | -- | -- | -- | -- | -- | -- | 0 | 0.0029 | 2.6E-08 | Pass |
| 2013 Pipe Rack-S-3 | 7/09/2013 | 4 | **0.291** * | < 0.0628 * | 0.317 * | 0.409 * | 0.0769 * | 0.251 * | 0.662 * | 0.311 * | 0 | 0.0169 | 1.3E-06 | Pass |
| 2013 Pipe Rack-S-4 | 8/01/2013 | 3.5 | **0.126** | 0.0144 | 0.232 | < 0.0135 | 0.0405 | < 0.0135 | 0.113 | 0.226 | 0 | 0.0004 | 6.5E-07 | Pass |

[1]WDNR RCL Determinations based on guidance criteria described in WDNR document PUB-RR-890. Hazard index is based a cumulative direct contact standard.

TR - Based on 1,2,4-Trimethylbenzene and 1,3,5-Trimethylbenzene combined.

XYL - Based on Xylenes (m-, o-, p- combined).

* - Estimated value, QA/QC criteria not met.

# Attachment A

## WDNR Documentation and Communications

*2013 Pipe Rack Construction - Proposed Soil Sampling Plan*
*WDNR approval communication for Proposed Soil Sampling Plan*

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# Technical Memorandum

**To:** Karl Beaster, Enbridge Energy
**From:** Ryan Erickson
**Subject:** 2013 Pipe Rack Construction - Proposed Soil Sampling Plan
**Date:** August 12, 2013
**Project:** 49161092 300 018

This purpose of this memorandum is to propose a modified contaminated soil sampling plan for the Enbridge Superior Terminal 2013 pipe rack construction project at the Enbridge Superior Terminal. The objective of the plan is to meet Wisconsin Department of Natural Resources (WDNR) requirements and be protective of human health and the environment, while taking into account the project's extensive size, scope and schedule.

## Background

The pipe rack project involves the construction of an approximately 1,400 feet long by 100 feet wide aboveground structure at the Superior Terminal (Figures 1 and 2). Construction of the structure will take place in 2013 and 2014. Hundreds of excavations will be completed within the project footprint and will range in size from one foot diameter hydrovac borings to 90 foot by 90 foot construction excavations. Many of these excavations will be in close proximity to each other and may even overlap during different construction phases. No existing WDNR Bureau of Remediation and Redevelopment Tracking System (BRRTS) sites were identified within the project footprint; however, historically contaminated soil with a hydrocarbon odor, discoloration, and/or sheen has been discovered in some of the construction excavations. The initial field screening results from these encounters will be reported to the WDNR under a separate memo as discussed below.

## Objectives

Due to the nature of the historical contamination and size and scope of the project, Enbridge will request that the WDNR allow the contamination discovered during the course of this project to be handled and combined as a single WDNR release site. This will eliminate the need to report each contaminated excavation individually and allow a pragmatic approach to sampling and documentation over a large project area. The project area would be defined as shown in Figure 2.

To accomplish this goal, the following pipe rack project-specific sampling plan has been developed for WDNR approval. The sampling plan attempts to adequately document the contaminated soil discovered during the pipe rack project while taking into account the project's size, scope and schedule.

## Proposed Sampling Plan

Enbridge will continue to document and manage contaminated soil discovered in the pipe rack excavations by completing the following tasks:

- Document the location of all contaminated excavations.
- Field screen soil to determine the vertical and horizontal extent of impacts within excavations. Field screening will include: using a photoionization detector to identify volatile organic headspace; and identifying the presence of hydrocarbon odor, discoloration and sheen.
- Soil containing free-product will be excavated as feasible, including when contaminated soil is present outside the excavation limits required for construction purposes.
- Excavated contaminated soil with a headspace of greater than 10 parts per million (ppm), or obvious hydrocarbon impacts, will be segregated and disposed of off-site at an approved landfill facility.

The current soil sampling plan described in the proposed WDNR-Enbridge negotiated agreement includes collecting excavation soil sidewall samples and base of excavation samples if headspace readings are greater than 10 ppm. Soil analytical samples are proposed to be collected from the following locations: soil sidewall samples from each exposed sidewall at 0 to 4 feet below ground surface (bgs) for petroleum volatile organic compound (PVOC) analysis; a single base of excavation sample for PVOC analysis; and one polycyclic aromatic hydrocarbon (PAH) sample from the excavation sidewall sample with the highest headspace detection . Enbridge proposes to modify the contaminated excavation sampling plan in the following ways:

- Enbridge would overlay a 25 foot grid over the pipe rack construction footprint (Figure 2).
- When contaminated soil is discovered (as defined by a headspace greater than 10 ppm), soil field screening samples will used to delineate the extent of contaminated soil within the excavation.
- Regardless of the number of small excavations completed within a 25 foot by 25 foot grid, only one representative sidewall and one bottom analytical sample would be collected from each grid

cell when residual contamination (headspace greater than 10 ppm) is present and cannot be excavated. The samples collected will be analyzed for PVOC and PAH. Sidewall samples will be collected from 0 to 4 feet bgs.

- Analytical samples would be used to document residual contamination within each grid area.
- If field screening identifies either an apparently different contaminant source or a greater level of contamination than was previously identified within a sampled grid, an additional PVOC and PAH sample will be collected from the excavation at that time.

## Reporting

Enbridge will submit the following documents at the completion of the pipe rack construction project:

- A summary of the excavations where impacts were identified
- Site figures identifying the location of the contaminated excavations and the analytical samples collected.
- Excavation field screening notes containing the field screening data.
- Analytical soil sample tables and laboratory reports.
- Waste disposal documentation.

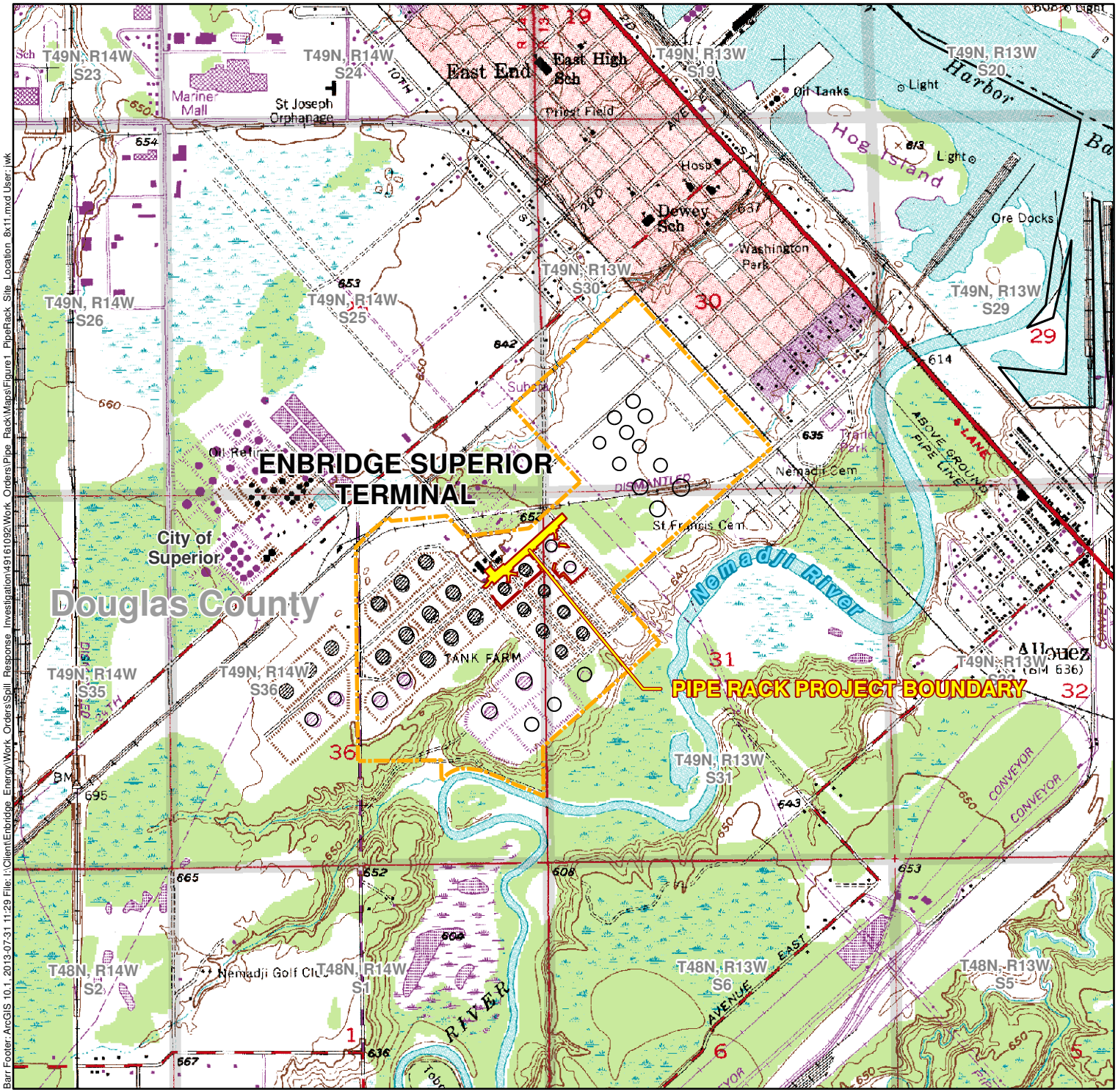
## Site Visit

Enbridge has offered that if the WDNR is interested in a terminal site visit to learn more about the project, they can contact Karl Beaster at Enbridge to make arrangements.

**Attachments:**

Figure 1 Pipe Rack Site Location Map

Figure 2 Pipe Rack Project Modified Sampling Plan





Pipe Rack Project Boundary

Terminal Property Boundary

N

0 2,000 4,000

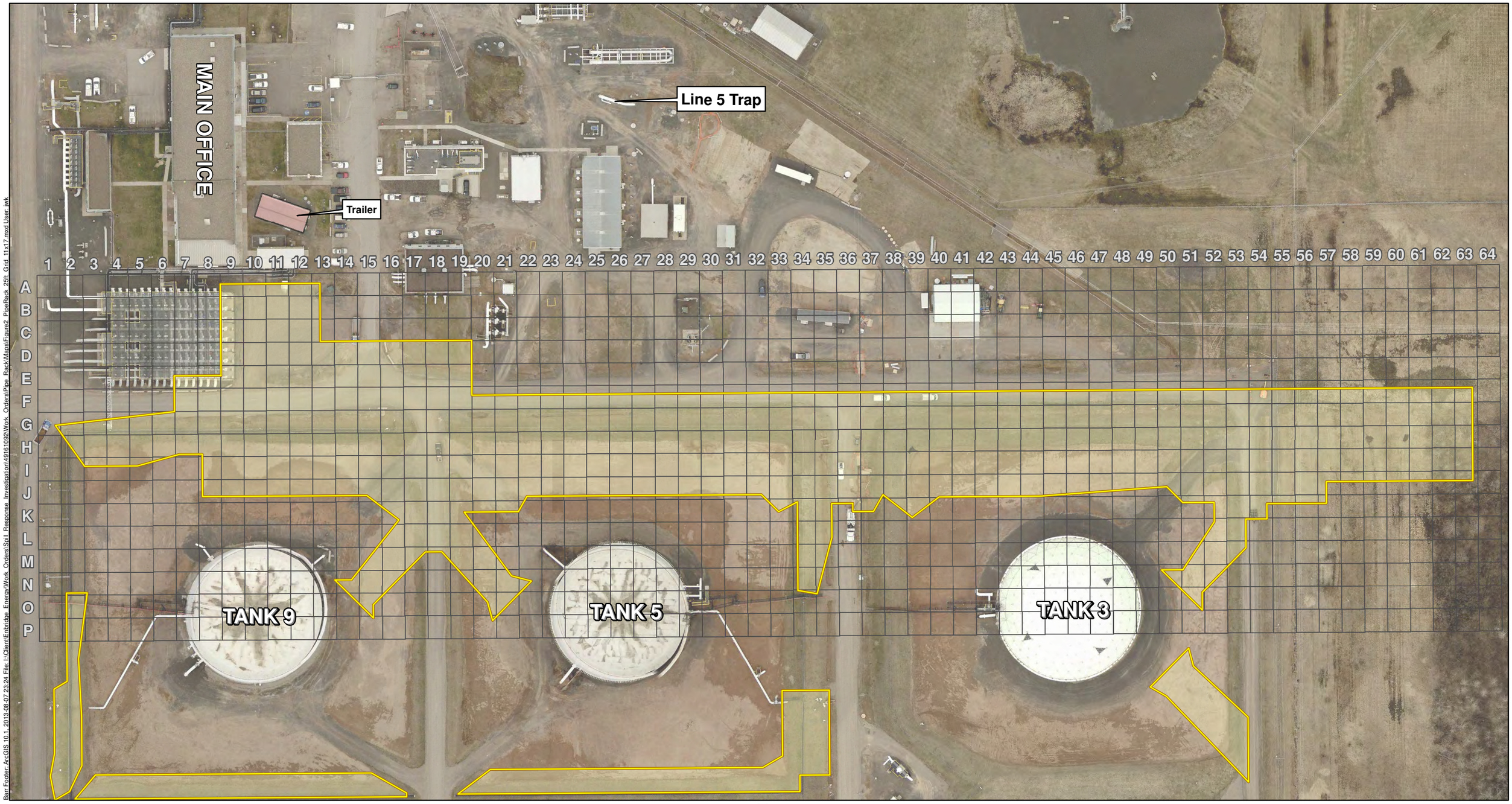
Feet

1 Inch = 2,000 Feet

Figure 1

**PIPE RACK SITE LOCATION**
**SUPERIOR TERMINAL**
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE




Pipe Rack Project Boundaries

25-Foot Reference Grid

Trailer

0 100 200

Feet

1 Inch = 100 Feet

*Douglas County Aerial Photography Circa April/May, 2013*

Figure 2

**PIPE RACK PROJECT**
**MODIFIED SAMPLING PLAN**
**SUPERIOR TERMINAL**
Enbridge Energy, L.P.
Superior, Wisconsin

## Ryan E. Erickson

**From:** Karl Beaster <Karl.Beaster@enbridge.com>
**Sent:** Wednesday, September 04, 2013 9:30 AM
**To:** Ryan E. Erickson
**Subject:** FW: Pipe Rack Project Sampling Plan Proposal

FYI – we can discuss at 10 and think about a good time to do a site visit with Erin.

Karl F. Beaster, PG
Environmental Analyst II, LP Environment

ENBRIDGE
TEL: 715-398-4754 | CELL: 715-718-1040
1320 Grand Ave, Superior, WI 54880
www.enbridgeUS.com



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**From:** Endsley, Erin A - DNR [mailto:Erin.Endsley@wisconsin.gov]
**Sent:** Monday, August 26, 2013 4:01 PM
**To:** Karl Beaster
**Subject:** RE: Pipe Rack Project Sampling Plan Proposal

Hello Karl –

Thanks for the info on the pipe rack project. Here are my thoughts.

Given the size of the project, I agree that it makes sense to handle sampling and reporting requirements in a comprehensive manner. We will not expect you to submit separate notifications or reports if impacts are discovered during site construction work. Please submit any necessary data at the completion of the project. However, we won't be able to make a determination on how to handle any contamination that is discovered until we look at the data. If we simply see a lot of low-level impacts throughout the area, I think we can treat it as one BRRTS activity. If we see a pattern that indicates that there are one or more discrete areas that represent larger releases, we will likely have to assign separate BRRTS activity numbers. This is because we have to treat separate releases as separate activities. There could also be complications if there are areas with structural impediments. Like I said, though, I think we should wait until we have all of the sampling data in hand at the end of the excavation, and then we can have a discussion on how to address any contamination that is encountered. I'll be as flexible as possible in how we approach this; simplifying things will make it easier on my end, as well.

I also agree with the proposed sampling plan on a conceptual level. It's hard for me to visualize, as I imagine the plan on paper will look quite different in reality, given the various sizes and locations of excavations that will be done. There are also a lot of complicating factors. I think this is an area that would benefit from some further discussion during a site visit. In the meantime, here are some thoughts:

- Analyze for PVOC+N; hold off on PAH sampling unless the initial sampling indicates a larger release may have occurred. I think we want to be strategic with when and where we analyze for PAHs.
- We might not need sampling results from every grid area, depending on the size of the area with impacts. In other words, for larger areas, we could look at reducing the number of samples over a given area.
- During site work, we should stay in communication so that we are in agreement on an approach if it is warranted to increase or decrease the amount of sampling, depending upon what is encountered. I think we can allow for some flexibility in the sampling plan, if we have open communication during the construction.

I would also be interested in a site visit to learn more about the project, just let me know when would be a good time. We can have more discussion at that time on the best approach to take with the project. I do appreciate the advanced planning and communication on this project.

I also hope to get back to you on a couple other items this week, including the no further action status for the line 5 pig trap and some follow up on the negotiated agreement submittal. I'll put each in separate emails.

Thanks!

Erin

---

**From:** Karl Beaster [mailto:Karl.Beaster@enbridge.com]
**Sent:** Friday, August 23, 2013 12:20 PM
**To:** Endsley, Erin A - DNR
**Subject:** Pipe Rack Project Sampling Plan Proposal

Erin – please review the attached sampling plan for the Enbridge Superior Terminal Pipe Rack Project, as previously discussed. We look forward to any final comments and/or your approval.
Thanks!
Karl

Karl F. Beaster, PG
Environmental Analyst II, LP Environment

ENBRIDGE
TEL: 715-398-4754 | CELL: 715-718-1040
1320 Grand Ave, Superior, WI 54880
www.enbridgeUS.com

**Integrity. Safety. Respect.**

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* * * * * * * * * * * * * * * * * * * * * * * * * IMPORTANT NOTICE* * * * * * * * * * * * * * * * * * * * * * * * *
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# Attachment B

## Enbridge Site Investigation Field Sampling and Screening Logs

*11/5/12 and 11/15/12 – Water Valve Replacement*
*11/5/12 and 11/7/12 – Fire Hydrant Replacement*
*7/9/13 – Pipe Rack Footing Excavations*
*8/1/13 – Central Manifold Excavation*

# Figure 4

**ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG**

Location: *Milepost or Facility* Superior Terminal/Water Valve Replacement Excavation

Date: 11/5/12 11/15/12

Sampler: RGG

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

Background Headspace: — ppm

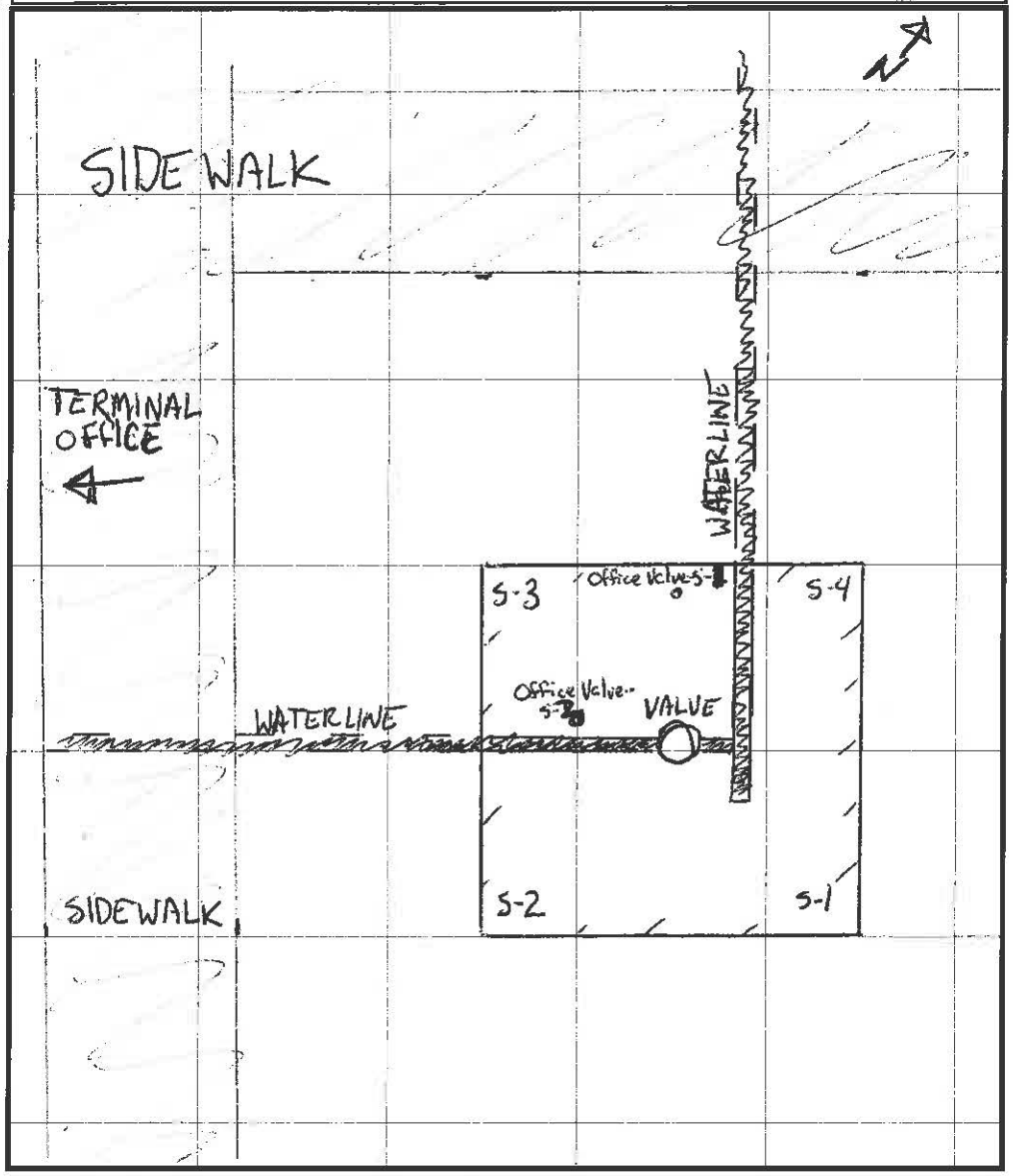
Calibration Time: -

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

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

| Sample ID | Depth (ft) | Time (military) | Soil Type (USCS) | Color/ Discolor | Odor/ Sheen | Headspace Reading (ppm) |
|---|---|---|---|---|---|---|
| Example R-1 | 4 | 16:30 | CL | Reddish brown | Petroleum/ Rainbow | 275 |
| S-1 | 3 | 1310 11/5 | CL | Reddish Brown | N /N | 1.1 |
| S-2 | 3 | 1312 11/5 | ↓ | ↓ | N/N | .8 |
| S-3 | 2 | 1314 11/5 | ↓ | ↓ | N/N | .6 |
| S-4 | 4 | 1316 11/5 | ↓ | ↓ | N/N | 1.1 |
| Office Valve-S-1 | 1 | 1015 11/15 | ↓ | ↓ | N/N | 1.0 |
| Office Valve-S-2 | 6 | 1030 11/15 | ↓ | ↓ | Petroleum/Y | X150 |

***SITE SKETCH:*** ~~*north is up*~~*; excavation extents and depths, sample locations, structures, utilities, boring locations, wells, natural features...* ***1 inch/grid = 4 FEET***



# Figure 3

**ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG**

Date: 11/5/2012 11/7/2012

Location: *Milepost or Facility* Superior Termin. | Fire Hydrant Excavation

Sampler: REE

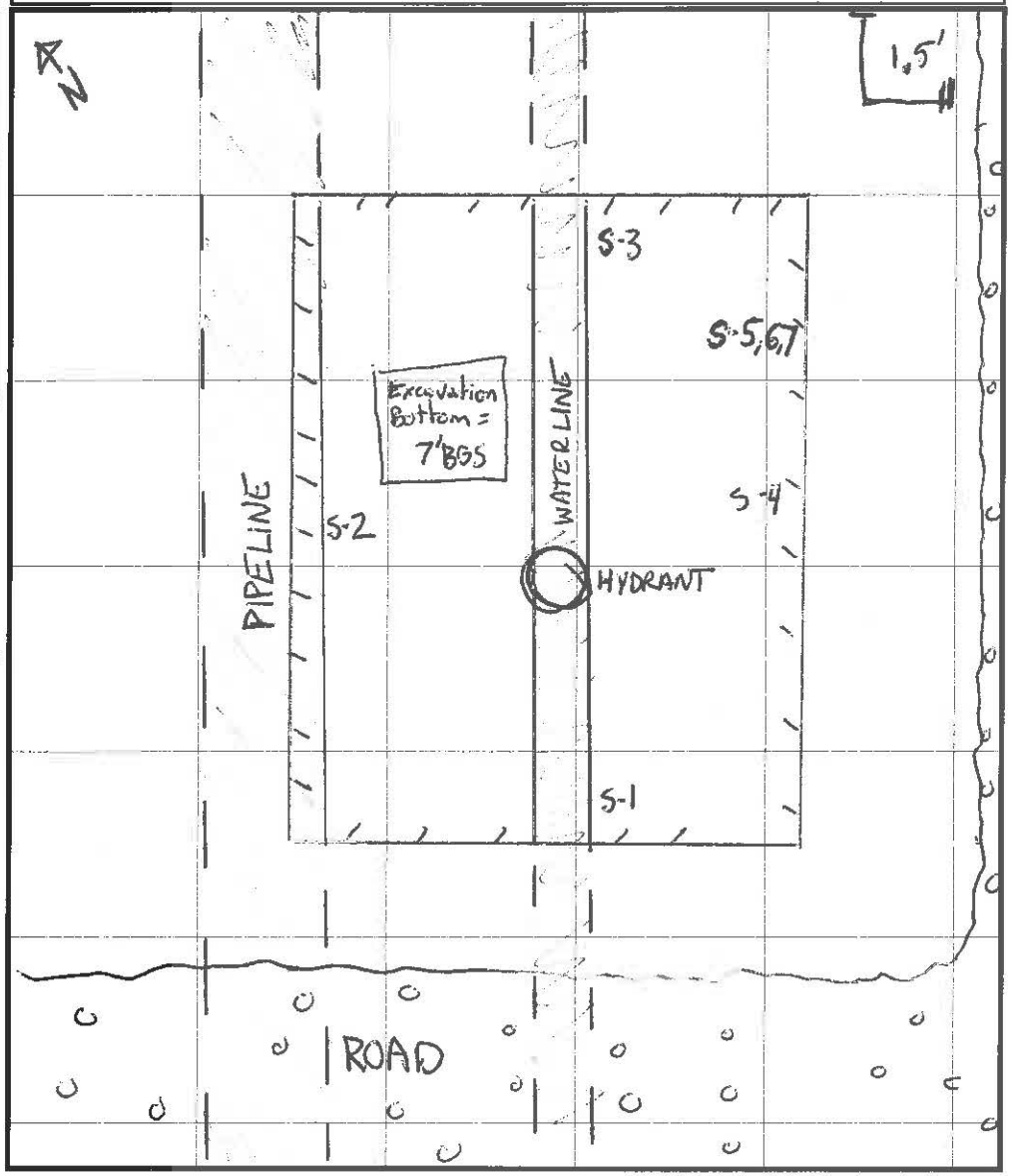
Equipment used: PID *-ionization detector with* 10.6 *eV lamp*  Background Headspace: – ppm  Calibration Time: –

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

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

| Sample ID | Depth (ft) | Date/ 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 | 1 | 11/5 1030 | CL/GP | Red | N/- | .7 |
| S-2 | 1 | 11/5 1035 | CL | | N/- | .4 |
| S-3 | 1 | 11/5 1040 | CL | | N/- | .8 |
| S-4 | 1 | 11/5 1045 | CL | | N/- | 1.2 |
| S-5 | 2 | 11/7 900 | CL | | N/- | .1 |
| S-6 | 3 | 11/7 905 | CL | Discolored Grayish | Petroleum odor/ | 203 |
| S-7 | 6 | 11/7 910 | CL | | N/- | 15.8 |

***SITE SKETCH:*** ~~*north is up*~~*; excavation extents and depths, sample locations, structures, utilities, boring locations, wells, natural features...* ***1 inch/grid =*** 3 ***FEET***



**ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG**

Date: 7/9/13

Location: *Milepost or Facility* Pipe Rack Structure Footing Excavations

Sampler: CGZ

Equipment used: Photo -*ionization detector with* 10.6 *eV lamp* Background Headspace: 1.0 ppm

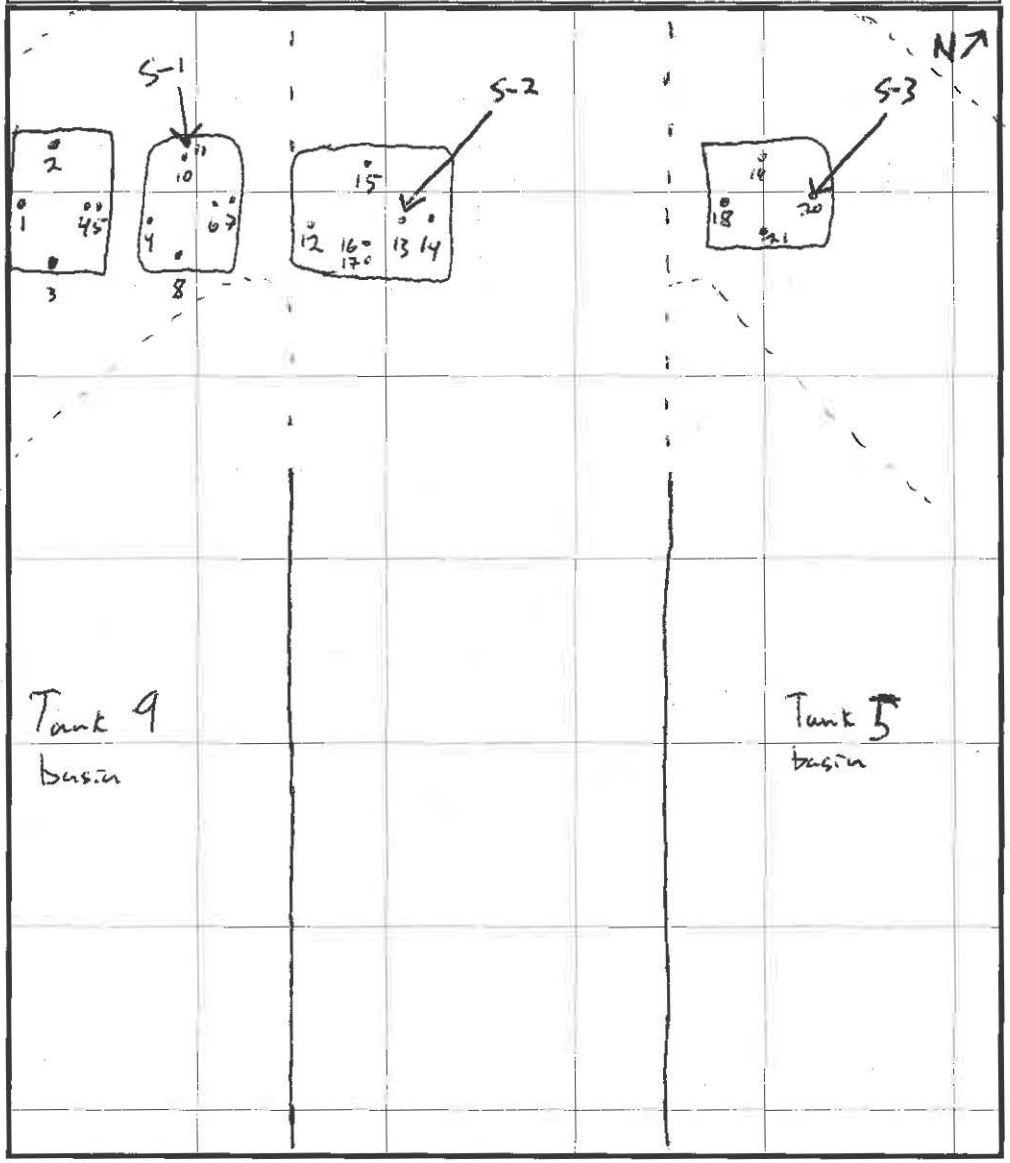
Calibration Time: 845

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

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

| Sample ID | Depth (ft) | Time (military) | Soil Type (USCS) | Color/ Discolor | Odor/ Sheen | Headspace Reading (ppm) |
|---|---|---|---|---|---|---|
| Example R-1 | 4 | 16:30 | CL | Reddish brown | Petroleum/ Rainbow | 275 |
| R-1 | 5 | 1700 | CL | Red brown | none/none | 2.0 |
| R-2 | 5 | ↓ | ↓ | ↓ | ↓ | 2.1 |
| R-3 | 5 | ↓ | ↓ | ↓ | ↓ | 2.2 |
| R-4 | 6 | ↓ | ↓ | ↓ | ↓ | 2.7 |
| R-5 | 4 | ↓ | ↓ | ↓ | ↓ | 2.2 |
| R-6 | 4 | 1710 | CL | Red brown | none/none | 4.4 |
| R-7 | 6 | ↓ | ↓ | ↓ | ↓ | 2.7 |
| R-8 | 4 | ↓ | ↓ | ↓ | ↓ | 4.8 |
| R-9 | 4 | ↓ | ↓ | ↓ | ↓ | 2.2 |
| R-10 | 6 | ↓ | ↓ | discolored | Strong Petroleum/Sheen | 199 |
| R-11 Analyzed S-1 | 4 | ↓ | ↓ | Slightly discolored | Slight odor/no sheen | 18.4 |
| R-12 | 4' | 1720 | CL | Red brown | none/none | 4.2 |
| R-13 Analyzed S-2 | 4' | ↓ | ↓ | discolored | Slight odor/ Sheen | 52.8 |
| R-14 | 6' | ↓ | ↓ | Slightly discolored | ↓ | 46.7 |
| R-15 | 4' | ↓ | ↓ | discolored | Strong odor/ rainbow sheen | 102.5 |
| R-16 | 6' | ↓ | ↓ | ↓ | ↓ | 119 |
| R-17 | 4' | ↓ | ↓ | Red brown | none/none | 3.8 |
| R-18 | 4' | 1730 | CL | Red brown | none/none | 2.9 |
| R-19 | 4' | ↓ | ↓ | ↓ | ↓ | 5.0 |
| R-20 Analyzed S-3 | 4' | ↓ | ↓ | discolored | Strong odor/ Sheen | 87.5 |
| R-21 | 4' | ↓ | ↓ | Slightly discolored | Slight odor, slight sheen | 21.9 |
| S-1 | 4' | 1800 | CL | Slightly discolored | Slight odor/ no sheen | |
| S-2 | 4' | 1805 | CL | discolored | Slight odor/ Sheen | |
| S-3 | 4' | 1810 | CL | discolored | Strong odor/ Sheen | |

***SITE SKETCH:*** ~~*north is up*~~*; excavation extents and depths, sample locations, structures, utilities, boring locations, wells, natural features...* ***1 inch/grid = 10' FEET***



**ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG**

Location: *Milepost or Facility* Terminal Central Manifold Expansion Excavation

Date: 8/1/13

Sampler: CSG / RGG

Equipment used: Photo -*ionization detector with* 10.6 *eV lamp* Background Headspace: -1 ppm

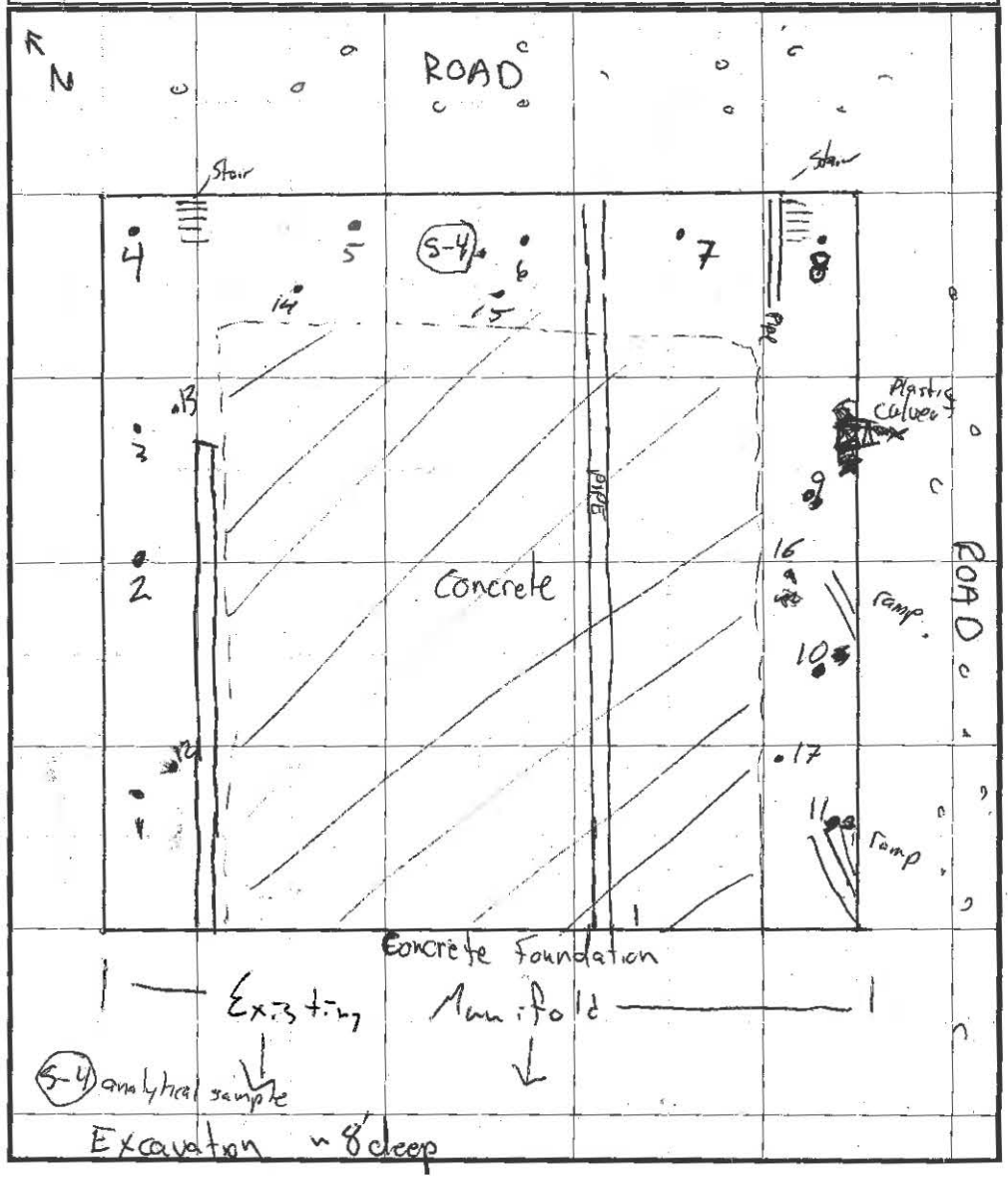
Calibration Time: 10:45

Sample Nomenclature *(Location - sample type - #)*: 2013 Pipe Rack -

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 |
| R-1 | 3 | 1200 | CL | Red brown | none | 0.0 |
| R-2 | 2 | 1205 | CL | Red brown | none | 0.0 |
| R-3 | 2 | 1205 | CL | Red brown (slightly lighter) | none | 0.1 |
| R-4 | 3 | 1210 | CL | Red brown | none | 0.2 |
| R-5 | 2 | 1210 | CL | Red brown | none | 0.1 |
| R-6 | 2 | 1215 | CL | Red brown | none | 0.1 |
| R-7 | 3 | 1217 | CL | Red Brown | N/N | 0.2 |
| R-8 | 2 | 1219 | CL | Red Brown | N/N | 0.2 |
| R-9 | 3 | 1221 | CL | Red Brown | N/N | 0.1 |
| R-10 | 3 | 1222 | CL | Red Brown | N/N | 0.1 |
| R-11 | 2 | 1224 | CL | Red Brown | N/N | 0.2 |
| R-12 | 5 | 1245 | CL | Red Brown | N/N | 0.6 |
| R-13 | 6' | 1247 | CL | Red Brown | N/N | 0.4 |
| R-14 | 6' | 1250 | CL | Red brown | N/N | 0.5 |
| R-15 | 6 | 1252 | CL | Red brown | N/Film | 26.8 |
| R-16 | 7' | 1254 | CL | Red brown | None | 0.5 |
| R-17 | 7' | 1256 | CL | Red brown | None | 0.6 |
| | | | | | | |
| S-4 | 3.5 | 1310 | CL | Red brown | None | 0.4 |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |

***SITE SKETCH:*** *north is up; excavation extents and depths, sample locations, structures, utilities, boring locations, wells, natural features...* ***1 inch/grid = 25 FEET***



# Attachment C

## Pace Analytical Laboratory Reports for Excavation Soil Samples

*Pace Analytical (Water Valve Replacement)*
*Pace Analytical (Pipe Rack Footings)*
*Pace Analytical (Central Manifold Excavation)*

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

November 28, 2012

Andrea Nord
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

RE: Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on November 16, 2012. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

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

Sincerely,

Andrea Opland

andrea.opland@pacelabs.com
Project Manager

Enclosures

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## CERTIFICATIONS

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

**Minnesota Certification IDs**

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Hawaii Certification #Pace
Idaho Certification #: MN00064
Illinois Certification #: 200011
Kansas Certification #: E-10167
Louisiana Certification #: 03086
Louisiana Certification #: LA080009
Maine Certification #: 2007029
Maryland Certification #: 322
Michigan DEQ Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT CERT0092
Nebraska Certification #: Pace
Nevada Certification #: MN_00064
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
North Dakota Certification #: R-036A
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Utah Certification #: MN00064
Virginia/DCLS Certification #: 002521
Virginia/VELAP Certification #: 460163
Washington Certification #: C754
West Virginia Certification #: 382
Wisconsin Certification #: 999407970

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## SAMPLE SUMMARY

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|---|---|---|---|---|
| **10212795001** | **Office Valve-S-1** | Solid | 11/15/12 10:15 | 11/16/12 09:35 |
| **10212795002** | **Office Valve-S-2** | Solid | 11/15/12 10:30 | 11/16/12 09:35 |

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1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## SAMPLE ANALYTE COUNT

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|---|---|---|---|---|---|
| **10212795001** | **Office Valve-S-1** | WI MOD DRO | MT | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| **10212795002** | **Office Valve-S-2** | WI MOD DRO | MT | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# PROJECT NARRATIVE

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

**Method:** **WI MOD DRO**
**Description:** WIDRO GCS
**Client:** Barr Engineering
**Date:** November 28, 2012

**General Information:**
2 samples were analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with WI MOD DRO with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Additional Comments:**

Analyte Comments:

QC Batch: OEXT/20308

T6: High boiling point hydrocarbons are present in the sample.

- Office Valve-S-2 (Lab ID: 10212795002)
  - Diesel Range Organics

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## PROJECT NARRATIVE

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

**Method:** **WI MOD GRO**
**Description:** WIGRO GCV
**Client:** Barr Engineering
**Date:** November 28, 2012

**General Information:**
2 samples were analyzed for WI MOD GRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with TPH GRO/PVOC WI ext. with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Additional Comments:**

This data package has been reviewed for quality and completeness and is approved for release.

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## ANALYTICAL RESULTS

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

**Sample: Office Valve-S-1** **Lab ID: 10212795001** Collected: 11/15/12 10:15 Received: 11/16/12 09:35 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIDRO GCS** | Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | | |
| Diesel Range Organics | **<12.3** | mg/kg | 12.3 | 1.4 | 1 | 11/19/12 13:33 | 11/20/12 09:12 | | |
| ***Surrogates*** | | | | | | | | | |
| n-Triacontane (S) | 85 | % | 50-150 | | 1 | 11/19/12 13:33 | 11/20/12 09:12 | | |
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| Benzene | **<0.067** | mg/kg | 0.067 | 0.0080 | 1 | 11/18/12 20:51 | 11/19/12 08:50 | 71-43-2 | |
| Ethylbenzene | **<0.067** | mg/kg | 0.067 | 0.011 | 1 | 11/18/12 20:51 | 11/19/12 08:50 | 100-41-4 | |
| Toluene | **<0.067** | mg/kg | 0.067 | 0.0080 | 1 | 11/18/12 20:51 | 11/19/12 08:50 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | **<0.067** | mg/kg | 0.067 | 0.0094 | 1 | 11/18/12 20:51 | 11/19/12 08:50 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | **<0.067** | mg/kg | 0.067 | 0.015 | 1 | 11/18/12 20:51 | 11/19/12 08:50 | 108-67-8 | |
| Xylene (Total) | **<0.20** | mg/kg | 0.20 | 0.021 | 1 | 11/18/12 20:51 | 11/19/12 08:50 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 100 | % | 80-125 | | 1 | 11/18/12 20:51 | 11/19/12 08:50 | 98-08-8 | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **24.1** | % | 0.10 | 0.10 | 1 | | 11/20/12 00:00 | | |

**Sample: Office Valve-S-2** **Lab ID: 10212795002** Collected: 11/15/12 10:30 Received: 11/16/12 09:35 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIDRO GCS** | Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | | |
| Diesel Range Organics | **222** | mg/kg | 13.9 | 1.5 | 1 | 11/19/12 13:33 | 11/20/12 09:19 | | T6 |
| ***Surrogates*** | | | | | | | | | |
| n-Triacontane (S) | 87 | % | 50-150 | | 1 | 11/19/12 13:33 | 11/20/12 09:19 | | |
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| Benzene | **<0.078** | mg/kg | 0.078 | 0.0093 | 1 | 11/18/12 20:51 | 11/19/12 09:10 | 71-43-2 | |
| Ethylbenzene | **<0.078** | mg/kg | 0.078 | 0.012 | 1 | 11/18/12 20:51 | 11/19/12 09:10 | 100-41-4 | |
| Toluene | **<0.078** | mg/kg | 0.078 | 0.0093 | 1 | 11/18/12 20:51 | 11/19/12 09:10 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | **0.56** | mg/kg | 0.078 | 0.011 | 1 | 11/18/12 20:51 | 11/19/12 09:10 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | **0.22** | mg/kg | 0.078 | 0.017 | 1 | 11/18/12 20:51 | 11/19/12 09:10 | 108-67-8 | |
| Xylene (Total) | **<0.23** | mg/kg | 0.23 | 0.025 | 1 | 11/18/12 20:51 | 11/19/12 09:10 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 96 | % | 80-125 | | 1 | 11/18/12 20:51 | 11/19/12 09:10 | 98-08-8 | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **32.7** | % | 0.10 | 0.10 | 1 | | 11/20/12 00:00 | | |

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Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

QC Batch: GCV/10047 Analysis Method: WI MOD GRO
QC Batch Method: TPH GRO/PVOC WI ext. Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10212795001, 10212795002

METHOD BLANK: 1335984 Matrix: Solid
Associated Lab Samples: 10212795001, 10212795002

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 11/19/12 05:03 | |
| 1,3,5-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 11/19/12 05:03 | |
| Benzene | mg/kg | <0.050 | 0.050 | 11/19/12 05:03 | |
| Ethylbenzene | mg/kg | <0.050 | 0.050 | 11/19/12 05:03 | |
| Toluene | mg/kg | <0.050 | 0.050 | 11/19/12 05:03 | |
| Xylene (Total) | mg/kg | <0.15 | 0.15 | 11/19/12 05:03 | |
| a,a,a-Trifluorotoluene (S) | % | 99 | 80-125 | 11/19/12 05:03 | |

LABORATORY CONTROL SAMPLE & LCSD: 1335985 1335986

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | 5 | 4.2 | 4.7 | 84 | 95 | 80-120 | 12 | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | 5 | 4.3 | 4.8 | 86 | 97 | 80-120 | 12 | 20 | |
| Benzene | mg/kg | 5 | 4.1 | 4.7 | 82 | 93 | 80-120 | 13 | 20 | |
| Ethylbenzene | mg/kg | 5 | 4.2 | 4.8 | 85 | 96 | 80-120 | 12 | 20 | |
| Toluene | mg/kg | 5 | 4.2 | 4.7 | 83 | 94 | 80-120 | 12 | 20 | |
| Xylene (Total) | mg/kg | 15 | 12.8 | 14.4 | 86 | 96 | 80-120 | 12 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 97 | 97 | 80-125 | | | |

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 1335987 1335988

| Parameter | Units | 1214887001 Result | MS Spike Conc. | MSD Spike Conc. | MS Result | MSD Result | MS % Rec | MSD % Rec | % Rec Limits | RPD | Max RPD | Qual |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | 0.15 | 5.6 | 5.7 | 5.5 | 5.5 | 96 | 92 | 80-120 | .6 | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | 0.094 | 5.6 | 5.7 | 5.6 | 5.6 | 99 | 95 | 80-120 | .9 | 20 | |
| Benzene | mg/kg | ND | 5.6 | 5.7 | 5.4 | 5.3 | 95 | 92 | 80-120 | 1 | 20 | |
| Ethylbenzene | mg/kg | 0.072 | 5.6 | 5.7 | 5.6 | 5.5 | 98 | 95 | 80-120 | .2 | 20 | |
| Toluene | mg/kg | 0.077 | 5.6 | 5.7 | 5.5 | 5.4 | 96 | 93 | 80-120 | .9 | 20 | |
| Xylene (Total) | mg/kg | 0.41 | 16.8 | 17.3 | 16.6 | 16.8 | 97 | 95 | 80-120 | .8 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | | | 96 | 95 | 80-125 | | | |

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## QUALITY CONTROL DATA

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

QC Batch: MPRP/36530
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10212795001, 10212795002

SAMPLE DUPLICATE: 1337110

| Parameter | Units | 10212530007 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 8.5 | 8.5 | .6 | 30 | |

SAMPLE DUPLICATE: 1337301

| Parameter | Units | 10212794005 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 23.4 | 23.9 | 2 | 30 | |

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## QUALITY CONTROL DATA

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

QC Batch: OEXT/20308 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10212795001, 10212795002

METHOD BLANK: 1336473 Matrix: Solid
Associated Lab Samples: 10212795001, 10212795002

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | <10.0 | 10.0 | 11/20/12 06:50 | |
| n-Triacontane (S) | % | 74 | 50-150 | 11/20/12 06:50 | |

LABORATORY CONTROL SAMPLE & LCSD: 1336474 1336475

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | 80 | 68.7 | 75.4 | 86 | 94 | 70-120 | 9 | 20 | |
| n-Triacontane (S) | % | | | | 80 | 82 | 50-150 | | | |

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# QUALIFIERS

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

## DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

## LABORATORIES

| | |
|---|---|
| PASI-M | Pace Analytical Services - Minneapolis |

## ANALYTE QUALIFIERS

| | |
|---|---|
| T6 | High boiling point hydrocarbons are present in the sample. |

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## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092 Ebridge Water Hydrant
Pace Project No.: 10212795

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|---|---|---|---|---|---|
| **10212795001** | **Office Valve-S-1** | WI MOD DRO | OEXT/20308 | WI MOD DRO | GCSV/10471 |
| **10212795002** | **Office Valve-S-2** | WI MOD DRO | OEXT/20308 | WI MOD DRO | GCSV/10471 |
| **10212795001** | **Office Valve-S-1** | TPH GRO/PVOC WI ext. | GCV/10047 | WI MOD GRO | GCV/10052 |
| **10212795002** | **Office Valve-S-2** | TPH GRO/PVOC WI ext. | GCV/10047 | WI MOD GRO | GCV/10052 |
| **10212795001** | **Office Valve-S-1** | ASTM D2974 | MPRP/36530 | | |
| **10212795002** | **Office Valve-S-2** | ASTM D2974 | MPRP/36530 | | |

# Chain of Custody

1130 10/2795

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

COC 1 of 1

Project Number: 49161092

Project Name: ~~Formant~~ Enbridge Water Hydrant + Valves

Sample Origination State WI (use two letter postal state abbreviation)

COC Number: № 40043

Project Manager: REE

Project QC Contact: ADN

Sampled by: REE

Laboratory: PACE

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Type: Grab | Type: Comp. | Type: QC | Number of Containers/Preservative: Water | Number of Containers/Preservative: Soil | Total Number Of Containers | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | VOCs (tared MeOH) #1 | GRO, PVOC (tared MeOH) #1 | DRO (tared unpreserved) | Metals (unpreserved) | SVOCs (unpreserved) #2 | % Solids (plastic vial, unpres.) | |
| 1. Office Valve - S-1 | 1 | 1 | ft | 11/15/12 | 1015 | | | | | | | | X | X | | | X | 3 | DRO, ~~BTEX~~, Moisture PVOC - MTBE |
| 2. Office Valve - S-2 | 6 | 6 | | ↓ | 1030 | | | | | | | | X | X | | | X | 3 | ↓ |
| 3. | | | | | | | | | | | | | | | | | | | |
| 4. | | | | | | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | | | | | | TAT by 11/27/12 |
| 6. | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | | | | | | |

Common Parameter/Container - Preservation Key

*#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List*
*#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs*
*#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate*
*#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN*

| Relinquished By: | On Ice? Y N | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| | (Y) N | 11/15/12 | 1130 | TN / Pace | 11/16/12 | 935 |
| Relinquished By: | On Ice? Y N | Date | Time | Received by: | Date | Time |
| | Y N | | | | | |

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☒ Sampler ☐ Other: _____

Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:\RLG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

| Pace Analytical® | Document Name: **Sample Condition Upon Receipt Form** | Document Revised: 13Nov2012 Page 1 of 1 |
|---|---|---|
| | Document No.: **F-MN-L-213-rev.05** | Issuing Authority: Pace Minnesota Quality Office |

**Sample Condition Upon Receipt**

**Client Name:** Barr

**Project #:** WO# : 10212795

10212795

**Courier:** ☒ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other: _____

**Tracking Number:** 7940 8673 3444

**Custody Seal on Cooler/Box Present?** ☒ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No

**Optional:** Proj. Due Date: Proj. Name:

**Packing Material:** ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: _____ **Temp Blank?** ☒ Yes ☐ No

**Thermometer Used:** ☐ B88A912167504 ☒ 80512447 **Type of Ice:** ☒ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

**Cooler Temp Read (°C):** 1.9 **Cooler Temp Corrected (°C):** 2.1 **Biological Tissue Frozen?** ☐ Yes ☐ No

Temp should be above freezing to 6°C **Date and Initials of Person Examining Contents:** 11/16/12

| | | | | Comments: |
|---|---|---|---|---|
| Chain of Custody Present? | ☒ Yes | ☐ No | ☐ N/A | 1. |
| Chain of Custody Filled Out? | ☒ Yes | ☐ No | ☐ N/A | 2. |
| Chain of Custody Relinquished? | ☒ Yes | ☐ No | ☐ N/A | 3. |
| Sampler Name and/or Signature on COC? | ☒ Yes | ☐ No | ☐ N/A | 4. |
| Samples Arrived within Hold Time? | ☒ Yes | ☐ No | ☐ N/A | 5. |
| **Short Hold Time Analysis (<72 hr)?** | ☐ Yes | ☒ No | ☐ N/A | 6. |
| **Rush Turn Around Time Requested?** | ☐ Yes | ☒ No | ☐ N/A | 7. |
| Sufficient Volume? | ☒ Yes | ☐ No | ☐ N/A | 8. |
| Correct Containers Used? | ☒ Yes | ☐ No | ☐ N/A | 9. |
| -Pace Containers Used? | ☐ Yes | ☒ No | ☐ N/A | |
| Containers Intact? | ☒ Yes | ☐ No | ☐ N/A | 10. |
| Filtered Volume Received for Dissolved Tests? | ☐ Yes | ☐ No | ☒ N/A | 11. |
| Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: S2 | ☒ Yes | ☐ No | ☐ N/A | 12. |
| All containers needing acid/base preservation have been checked? Noncompliances are noted in 13. | ☐ Yes | ☐ No | ☒ N/A | 13. ☐ $HNO_3$ ☐ $H_2SO_4$ ☐ NaOH ☐ HCl |
| All containers needing preservation are found to be in compliance with EPA recommendation? ($HNO_3$, $H_2SO_4$, HCl<2; NaOH>12) | ☐ Yes | ☐ No | ☒ N/A | Sample # |
| Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water) | ☐ Yes | ☒ No | | Initial when completed: Lot # of added preservative: |
| Headspace in VOA Vials ( >6mm)? | ☐ Yes | ☐ No | ☒ N/A | 14. |
| Trip Blank Present? | ☐ Yes | ☒ No | ☐ N/A | 15. |
| Trip Blank Custody Seals Present? | ☐ Yes | ☒ No | ☐ N/A | |
| Pace Trip Blank Lot # (if purchased): | | | | |

**CLIENT NOTIFICATION/RESOLUTION** **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

**Project Manager Review:** CMD **Date:** 11-19-12

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e out of hold, incorrect preservative, out of temp, incorrect containers)

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
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December 06, 2013

Andrea Nord
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

RE: Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on July 13, 2013. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

This report was revised to change the sample IDs to indicate 4.0-4.0 foot depth per client request.

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

Sincerely,

Aaron Fredrikson for
Andrea Opland
andrea.opland@pacelabs.com
Project Manager

Enclosures

**Pace Analytical Services, Inc.**
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Minneapolis, MN 55414
(612)607-1700

## CERTIFICATIONS

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

**Minnesota Certification IDs**

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alabama Dept of Environmental Management #40770
Alaska Certification #: UST-078
Alaska Certification #MN00064
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 5 #WD-15J
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Hawaii Certification #Pace
Idaho Certification #: MN00064
Illinois Certification #: 200011
Indiana Certification#C-MN-01
Iowa Certification #: 368
Kansas Certification #: E-10167
Kentucky Dept of Envi. Protection - DW #90062
Louisiana Certification #: 03086
Louisiana Certification #: LA080009
Maine Certification #: 2007029
Maryland Certification #: 322
Michigan DEQ Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT CERT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Utah Certification #: MN00064
Virginia/DCLS Certification #: 002521
Virginia/VELAP Certification #: 460163
Washington Certification #: C754
West Virginia Certification #: 382
Wisconsin Certification #: 999407970

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## SAMPLE SUMMARY

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|---|---|---|---|---|
| **10235240001** | **2013-PIPERACK-S-1_4.0-4.0 ft** | Solid | 07/09/13 18:00 | 07/13/13 08:15 |
| **10235240002** | **2013-PIPERACK-S-2_4.0-4.0 ft** | Solid | 07/09/13 18:05 | 07/13/13 08:15 |
| **10235240003** | **2013-PIPERACK-S-3_4.0-4.0 ft** | Solid | 07/09/13 18:10 | 07/13/13 08:15 |

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## SAMPLE ANALYTE COUNT

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|---|---|---|---|---|---|
| **10235240001** | **2013-PIPERACK-S-1_4.0-4.0 ft** | WI MOD GRO | LLC | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| **10235240002** | **2013-PIPERACK-S-2_4.0-4.0 ft** | WI MOD GRO | LLC | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| **10235240003** | **2013-PIPERACK-S-3_4.0-4.0 ft** | WI MOD GRO | KT1 | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| | | EPA 8270 by SIM | JMZ | 18 | PASI-M |

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## PROJECT NARRATIVE

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

**Method:** **WI MOD GRO**
**Description:** WIGRO GCV
**Client:** Barr Engineering
**Date:** December 06, 2013

**General Information:**
3 samples were analyzed for WI MOD GRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with TPH GRO/PVOC WI ext. with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank, where applicable, with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Duplicate Sample:**
All duplicate sample results were within method acceptance criteria with any exceptions noted below.

**Additional Comments:**

Analyte Comments:

QC Batch: GCV/11054

1M: Surrogate recovery outside laboratory control limits due to matrix interferences.

- 2013-PIPERACK-S-2_4.0-4.0 ft (Lab ID: 10235240002)
  - a,a,a-Trifluorotoluene (S)
- 2013-PIPERACK-S-3_4.0-4.0 ft (Lab ID: 10235240003)
  - a,a,a-Trifluorotoluene (S)

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## PROJECT NARRATIVE

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

**Method:** **WI MOD GRO**
**Description:** WIGRO GCV
**Client:** Barr Engineering
**Date:** December 06, 2013

Analyte Comments:

QC Batch: GCV/11054

D3: Sample was diluted due to the presence of high levels of non-target analytes or other matrix interference.

- 2013-PIPERACK-S-2_4.0-4.0 ft (Lab ID: 10235240002)
  - a,a,a-Trifluorotoluene (S)
- 2013-PIPERACK-S-3_4.0-4.0 ft (Lab ID: 10235240003)
  - a,a,a-Trifluorotoluene (S)

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# PROJECT NARRATIVE

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

**Method:** **EPA 8270 by SIM**
**Description:** 8270 MSSV PAH by SIM
**Client:** Barr Engineering
**Date:** December 06, 2013

**General Information:**
1 sample was analyzed for EPA 8270 by SIM. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with EPA 3550 with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank, where applicable, with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

QC Batch: OEXT/22333

A matrix spike and/or matrix spike duplicate (MS/MSD) were performed on the following sample(s): 10235240003

M1: Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.

- MS (Lab ID: 1481002)
  - Acenaphthene
  - Acenaphthylene
  - Benzo(b)fluoranthene
  - Chrysene
  - Fluorene
  - Naphthalene
  - Phenanthrene
- MSD (Lab ID: 1481003)
  - Acenaphthene
  - Benzo(k)fluoranthene

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# PROJECT NARRATIVE

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

**Method:** **EPA 8270 by SIM**
**Description:** 8270 MSSV PAH by SIM
**Client:** Barr Engineering
**Date:** December 06, 2013

QC Batch: OEXT/22333

A matrix spike and/or matrix spike duplicate (MS/MSD) were performed on the following sample(s): 10235240003

M1: Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.

- Chrysene
- Fluoranthene
- Fluorene
- Naphthalene
- Phenanthrene

R1: RPD value was outside control limits.

- MSD (Lab ID: 1481003)
  - Acenaphthene
  - Acenaphthylene
  - Fluorene
  - Naphthalene
  - Phenanthrene

**Additional Comments:**

This data package has been reviewed for quality and completeness and is approved for release.

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## ANALYTICAL RESULTS

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

**Sample: 2013-PIPERACK-S-1_4.0-4.0 ft** **Lab ID: 10235240001** Collected: 07/09/13 18:00 Received: 07/13/13 08:15 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| Benzene | **<0.071** | mg/kg | 0.071 | 0.011 | 1 | 07/15/13 10:17 | 07/16/13 08:17 | 71-43-2 | |
| Ethylbenzene | **<0.071** | mg/kg | 0.071 | 0.010 | 1 | 07/15/13 10:17 | 07/16/13 08:17 | 100-41-4 | |
| Toluene | **<0.071** | mg/kg | 0.071 | 0.0071 | 1 | 07/15/13 10:17 | 07/16/13 08:17 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | **<0.071** | mg/kg | 0.071 | 0.011 | 1 | 07/15/13 10:17 | 07/16/13 08:17 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | **<0.071** | mg/kg | 0.071 | 0.010 | 1 | 07/15/13 10:17 | 07/16/13 08:17 | 108-67-8 | |
| Xylene (Total) | **<0.21** | mg/kg | 0.21 | 0.024 | 1 | 07/15/13 10:17 | 07/16/13 08:17 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 98 | % | 80-125 | | 1 | 07/15/13 10:17 | 07/16/13 08:17 | 98-08-8 | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **24.0** | % | 0.10 | 0.10 | 1 | | 07/17/13 00:00 | | |

**Sample: 2013-PIPERACK-S-2_4.0-4.0 ft** **Lab ID: 10235240002** Collected: 07/09/13 18:05 Received: 07/13/13 08:15 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| Benzene | **<0.15** | mg/kg | 0.15 | 0.024 | 2 | 07/16/13 10:41 | 07/17/13 15:43 | 71-43-2 | |
| Ethylbenzene | **0.22** | mg/kg | 0.15 | 0.021 | 2 | 07/16/13 10:41 | 07/17/13 15:43 | 100-41-4 | |
| Toluene | **<0.15** | mg/kg | 0.15 | 0.015 | 2 | 07/16/13 10:41 | 07/17/13 15:43 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | **0.85** | mg/kg | 0.15 | 0.024 | 2 | 07/16/13 10:41 | 07/17/13 15:43 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | **0.58** | mg/kg | 0.15 | 0.021 | 2 | 07/16/13 10:41 | 07/17/13 15:43 | 108-67-8 | |
| Xylene (Total) | **1.2** | mg/kg | 0.45 | 0.051 | 2 | 07/16/13 10:41 | 07/17/13 15:43 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 51 | % | 80-125 | | 2 | 07/16/13 10:41 | 07/17/13 15:43 | 98-08-8 | 1M,D3 |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **21.8** | % | 0.10 | 0.10 | 1 | | 07/17/13 00:00 | | |

**Sample: 2013-PIPERACK-S-3_4.0-4.0 ft** **Lab ID: 10235240003** Collected: 07/09/13 18:10 Received: 07/13/13 08:15 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| Benzene | **<0.63** | mg/kg | 0.63 | 0.10 | 10 | 07/16/13 10:41 | 07/23/13 07:39 | 71-43-2 | |
| Ethylbenzene | **<0.63** | mg/kg | 0.63 | 0.089 | 10 | 07/16/13 10:41 | 07/23/13 07:39 | 100-41-4 | |
| Toluene | **<0.63** | mg/kg | 0.63 | 0.063 | 10 | 07/16/13 10:41 | 07/23/13 07:39 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | **5.4** | mg/kg | 0.63 | 0.10 | 10 | 07/16/13 10:41 | 07/23/13 07:39 | 95-63-6 | |

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

**Sample: 2013-PIPERACK-S-3_4.0-4.0 ft** **Lab ID: 10235240003** Collected: 07/09/13 18:10 Received: 07/13/13 08:15 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| 1,3,5-Trimethylbenzene | **3.5** | mg/kg | 0.63 | 0.089 | 10 | 07/16/13 10:41 | 07/23/13 07:39 | 108-67-8 | |
| Xylene (Total) | **3.4** | mg/kg | 1.9 | 0.22 | 10 | 07/16/13 10:41 | 07/23/13 07:39 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 70 | % | 80-125 | | 10 | 07/16/13 10:41 | 07/23/13 07:39 | 98-08-8 | 1M,D3 |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **20.4** | % | 0.10 | 0.10 | 1 | | 07/17/13 00:00 | | |
| **8270 MSSV PAH by SIM** | Analytical Method: EPA 8270 by SIM Preparation Method: EPA 3550 | | | | | | | | |
| Acenaphthene | **70.7** | ug/kg | 62.8 | 6.3 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 83-32-9 | M1,R1 |
| Acenaphthylene | **68.8** | ug/kg | 62.8 | 8.6 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 208-96-8 | M1,R1 |
| Anthracene | **<62.8** | ug/kg | 62.8 | 31.4 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 120-12-7 | |
| Benzo(a)anthracene | **163** | ug/kg | 62.8 | 31.4 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 56-55-3 | |
| Benzo(a)pyrene | **147** | ug/kg | 62.8 | 31.4 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 50-32-8 | |
| Benzo(b)fluoranthene | **207** | ug/kg | 62.8 | 3.6 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 205-99-2 | M1 |
| Benzo(g,h,i)perylene | **90.1** | ug/kg | 62.8 | 31.4 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 191-24-2 | |
| Benzo(k)fluoranthene | **72.9** | ug/kg | 62.8 | 5.5 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 207-08-9 | M1 |
| Chrysene | **291** | ug/kg | 62.8 | 4.8 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 218-01-9 | M1 |
| Dibenz(a,h)anthracene | **<62.8** | ug/kg | 62.8 | 31.4 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 53-70-3 | |
| Fluoranthene | **317** | ug/kg | 62.8 | 4.5 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 206-44-0 | M1 |
| Fluorene | **409** | ug/kg | 62.8 | 7.7 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 86-73-7 | M1,R1 |
| Indeno(1,2,3-cd)pyrene | **76.9** | ug/kg | 62.8 | 31.4 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 193-39-5 | |
| Naphthalene | **251** | ug/kg | 62.8 | 7.8 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 91-20-3 | M1,R1 |
| Phenanthrene | **662** | ug/kg | 62.8 | 4.1 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 85-01-8 | M1,R1 |
| Pyrene | **311** | ug/kg | 62.8 | 3.8 | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 129-00-0 | |
| ***Surrogates*** | | | | | | | | | |
| 2-Fluorobiphenyl (S) | 83 | % | 51-125 | | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 321-60-8 | |
| Terphenyl-d14 (S) | 88 | % | 57-125 | | 5 | 07/18/13 07:32 | 07/23/13 01:37 | 1718-51-0 | |

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

QC Batch: GCV/11048 Analysis Method: WI MOD GRO
QC Batch Method: TPH GRO/PVOC WI ext. Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10235240001

METHOD BLANK: 1478093 Matrix: Solid
Associated Lab Samples: 10235240001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 07/16/13 01:04 | |
| 1,3,5-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 07/16/13 01:04 | |
| Benzene | mg/kg | <0.050 | 0.050 | 07/16/13 01:04 | |
| Ethylbenzene | mg/kg | <0.050 | 0.050 | 07/16/13 01:04 | |
| Toluene | mg/kg | <0.050 | 0.050 | 07/16/13 01:04 | |
| Xylene (Total) | mg/kg | <0.15 | 0.15 | 07/16/13 01:04 | |
| a,a,a-Trifluorotoluene (S) | % | 100 | 80-125 | 07/16/13 01:04 | |

LABORATORY CONTROL SAMPLE & LCSD: 1478094 1478095

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | 5 | 4.4 | 4.4 | 87 | 88 | 80-120 | .7 | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | 5 | 4.4 | 4.4 | 87 | 88 | 80-120 | .7 | 20 | |
| Benzene | mg/kg | 5 | 4.1 | 4.3 | 83 | 87 | 80-120 | 5 | 20 | |
| Ethylbenzene | mg/kg | 5 | 4.4 | 4.6 | 88 | 91 | 80-120 | 3 | 20 | |
| Toluene | mg/kg | 5 | 4.3 | 4.5 | 86 | 89 | 80-120 | 4 | 20 | |
| Xylene (Total) | mg/kg | 15 | 13.3 | 13.6 | 88 | 91 | 80-120 | 3 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 98 | 99 | 80-125 | | | |

MATRIX SPIKE SAMPLE: 1478096

| Parameter | Units | 10234814001 Result | Spike Conc. | MS Result | MS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | ND | 6 | 6.6 | 110 | 80-120 | |
| 1,3,5-Trimethylbenzene | mg/kg | ND | 6 | 6.6 | 110 | 80-120 | |
| Benzene | mg/kg | ND | 6 | 6.3 | 104 | 80-120 | |
| Ethylbenzene | mg/kg | ND | 6 | 6.7 | 111 | 80-120 | |
| Toluene | mg/kg | ND | 6 | 6.5 | 107 | 80-120 | |
| Xylene (Total) | mg/kg | ND | 18.2 | 20.2 | 111 | 80-120 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 99 | 80-125 | |

SAMPLE DUPLICATE: 1478097

| Parameter | Units | 10234814002 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | ND | <0.059 | | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | ND | <0.059 | | 20 | |
| Benzene | mg/kg | ND | <0.059 | | 20 | |
| Ethylbenzene | mg/kg | ND | <0.059 | | 20 | |
| Toluene | mg/kg | ND | <0.059 | | 20 | |

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

SAMPLE DUPLICATE: 1478097

| Parameter | Units | 10234814002 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Xylene (Total) | mg/kg | ND | <0.18 | | 20 | |
| a,a,a-Trifluorotoluene (S) | % | 98 | 100 | 2 | | |

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

QC Batch: GCV/11054 Analysis Method: WI MOD GRO
QC Batch Method: TPH GRO/PVOC WI ext. Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10235240002, 10235240003

METHOD BLANK: 1478787 Matrix: Solid
Associated Lab Samples: 10235240002, 10235240003

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 07/16/13 17:19 | |
| 1,3,5-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 07/16/13 17:19 | |
| Benzene | mg/kg | <0.050 | 0.050 | 07/16/13 17:19 | |
| Ethylbenzene | mg/kg | <0.050 | 0.050 | 07/16/13 17:19 | |
| Toluene | mg/kg | <0.050 | 0.050 | 07/16/13 17:19 | |
| Xylene (Total) | mg/kg | <0.15 | 0.15 | 07/16/13 17:19 | |
| a,a,a-Trifluorotoluene (S) | % | 102 | 80-125 | 07/16/13 17:19 | |

LABORATORY CONTROL SAMPLE & LCSD: 1478788 1478789

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | 5 | 4.3 | 4.5 | 86 | 91 | 80-120 | 5 | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | 5 | 4.3 | 4.5 | 86 | 90 | 80-120 | 4 | 20 | |
| Benzene | mg/kg | 5 | 4.1 | 4.3 | 82 | 85 | 80-120 | 4 | 20 | |
| Ethylbenzene | mg/kg | 5 | 4.4 | 4.5 | 88 | 91 | 80-120 | 3 | 20 | |
| Toluene | mg/kg | 5 | 4.3 | 4.4 | 85 | 89 | 80-120 | 4 | 20 | |
| Xylene (Total) | mg/kg | 15 | 13.1 | 13.8 | 88 | 92 | 80-120 | 5 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 101 | 99 | 80-125 | | | |

MATRIX SPIKE SAMPLE: 1478790

| Parameter | Units | 10235310002 Result | Spike Conc. | MS Result | MS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | ND | 6.3 | 6.2 | 98 | 80-120 | |
| 1,3,5-Trimethylbenzene | mg/kg | ND | 6.3 | 6.2 | 98 | 80-120 | |
| Benzene | mg/kg | ND | 6.3 | 6.0 | 94 | 80-120 | |
| Ethylbenzene | mg/kg | ND | 6.3 | 6.3 | 100 | 80-120 | |
| Toluene | mg/kg | ND | 6.3 | 6.1 | 97 | 80-120 | |
| Xylene (Total) | mg/kg | ND | 18.9 | 18.9 | 99 | 80-120 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 98 | 80-125 | |

SAMPLE DUPLICATE: 1478791

| Parameter | Units | 10235310003 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | ND | <0.051 | | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | ND | <0.051 | | 20 | |
| Benzene | mg/kg | ND | <0.051 | | 20 | |
| Ethylbenzene | mg/kg | ND | <0.051 | | 20 | |
| Toluene | mg/kg | ND | <0.051 | | 20 | |

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

SAMPLE DUPLICATE: 1478791

| Parameter | Units | 10235310003 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Xylene (Total) | mg/kg | ND | <0.15 | | 20 | |
| a,a,a-Trifluorotoluene (S) | % | 99 | 98 | 17 | | |

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

QC Batch: MPRP/40608 Analysis Method: ASTM D2974
QC Batch Method: ASTM D2974 Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10235240001, 10235240002, 10235240003

SAMPLE DUPLICATE: 1480064

| Parameter | Units | 10234875007 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | | 13.2 | | | |

SAMPLE DUPLICATE: 1480065

| Parameter | Units | 10235202001 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 12.3 | 15.7 | 24 | 30 | |

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

QC Batch: OEXT/22333
QC Batch Method: EPA 3550
Analysis Method: EPA 8270 by SIM
Analysis Description: 8270 Solid PAH by SIM MSSV
Associated Lab Samples: 10235240003

METHOD BLANK: 1481000 Matrix: Solid
Associated Lab Samples: 10235240003

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Acenaphthene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Acenaphthylene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Anthracene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Benzo(a)anthracene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Benzo(a)pyrene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Benzo(b)fluoranthene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Benzo(g,h,i)perylene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Benzo(k)fluoranthene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Chrysene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Dibenz(a,h)anthracene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Fluoranthene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Fluorene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Indeno(1,2,3-cd)pyrene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Naphthalene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Phenanthrene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| Pyrene | ug/kg | <10.0 | 10.0 | 07/22/13 18:19 | |
| 2-Fluorobiphenyl (S) | % | 76 | 51-125 | 07/22/13 18:19 | |
| Terphenyl-d14 (S) | % | 88 | 57-125 | 07/22/13 18:19 | |

LABORATORY CONTROL SAMPLE: 1481001

| Parameter | Units | Spike Conc. | LCS Result | LCS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|
| Acenaphthene | ug/kg | 33.3 | 22.7 | 68 | 45-125 | |
| Acenaphthylene | ug/kg | 33.3 | 21.7 | 65 | 45-125 | |
| Anthracene | ug/kg | 33.3 | 25.5 | 77 | 53-125 | |
| Benzo(a)anthracene | ug/kg | 33.3 | 26.7 | 80 | 56-125 | |
| Benzo(a)pyrene | ug/kg | 33.3 | 28.9 | 87 | 55-125 | |
| Benzo(b)fluoranthene | ug/kg | 33.3 | 27.9 | 84 | 59-125 | |
| Benzo(g,h,i)perylene | ug/kg | 33.3 | 26.6 | 80 | 54-125 | |
| Benzo(k)fluoranthene | ug/kg | 33.3 | 29.0 | 87 | 52-125 | |
| Chrysene | ug/kg | 33.3 | 28.3 | 85 | 54-125 | |
| Dibenz(a,h)anthracene | ug/kg | 33.3 | 26.5 | 79 | 53-125 | |
| Fluoranthene | ug/kg | 33.3 | 28.4 | 85 | 60-125 | |
| Fluorene | ug/kg | 33.3 | 25.0 | 75 | 52-125 | |
| Indeno(1,2,3-cd)pyrene | ug/kg | 33.3 | 26.6 | 80 | 55-125 | |
| Naphthalene | ug/kg | 33.3 | 21.1 | 63 | 40-125 | |
| Phenanthrene | ug/kg | 33.3 | 25.9 | 78 | 50-125 | |
| Pyrene | ug/kg | 33.3 | 29.2 | 88 | 59-125 | |
| 2-Fluorobiphenyl (S) | % | | | 70 | 51-125 | |
| Terphenyl-d14 (S) | % | | | 87 | 57-125 | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 1481002 1481003

| Parameter | Units | 10235240003 Result | MS Spike Conc. | MSD Spike Conc. | MS Result | MSD Result | MS % Rec | MSD % Rec | % Rec Limits | RPD | Max RPD | Qual |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Acenaphthene | ug/kg | 70.7 | 41.6 | 41.6 | 79.9 | 126 | 22 | 133 | 30-129 | 45 | 30 | M1,R1 |
| Acenaphthylene | ug/kg | 68.8 | 41.6 | 41.6 | 73.6 | 111 | 12 | 101 | 30-150 | 40 | 30 | M1,R1 |
| Anthracene | ug/kg | <62.8 | 41.6 | 41.6 | 103 | 114 | 96 | 125 | 30-150 | 11 | 30 | |
| Benzo(a)anthracene | ug/kg | 163 | 41.6 | 41.6 | 188 | 189 | 60 | 62 | 30-150 | .5 | 30 | |
| Benzo(a)pyrene | ug/kg | 147 | 41.6 | 41.6 | 168 | 202 | 50 | 131 | 30-150 | 18 | 30 | |
| Benzo(b)fluoranthene | ug/kg | 207 | 41.6 | 41.6 | 209 | 257 | 4 | 120 | 30-150 | 21 | 30 | M1 |
| Benzo(g,h,i)perylene | ug/kg | 90.1 | 41.6 | 41.6 | 104 | 110 | 33 | 48 | 30-150 | 5 | 30 | |
| Benzo(k)fluoranthene | ug/kg | 72.9 | 41.6 | 41.6 | 128 | 139 | 132 | 159 | 30-150 | 8 | 30 | M1 |
| Chrysene | ug/kg | 291 | 41.6 | 41.6 | 304 | 362 | 29 | 169 | 30-150 | 17 | 30 | M1 |
| Dibenz(a,h)anthracene | ug/kg | <62.8 | 41.6 | 41.6 | <62.4 | <62.4 | 131 | 146 | 30-150 | | 30 | |
| Fluoranthene | ug/kg | 317 | 41.6 | 41.6 | 356 | 406 | 93 | 215 | 30-150 | 13 | 30 | M1 |
| Fluorene | ug/kg | 409 | 41.6 | 41.6 | 362 | 583 | -114 | 419 | 36-125 | 47 | 30 | M1,R1 |
| Indeno(1,2,3-cd)pyrene | ug/kg | 76.9 | 41.6 | 41.6 | 97.7 | 102 | 50 | 60 | 30-150 | 4 | 30 | |
| Naphthalene | ug/kg | 251 | 41.6 | 41.6 | 231 | 361 | -46 | 265 | 30-150 | 44 | 30 | M1,R1 |
| Phenanthrene | ug/kg | 662 | 41.6 | 41.6 | 585 | 930 | -186 | 644 | 30-150 | 46 | 30 | M1,R1 |
| Pyrene | ug/kg | 311 | 41.6 | 41.6 | 326 | 373 | 38 | 150 | 30-150 | 13 | 30 | |
| 2-Fluorobiphenyl (S) | % | | | | | | 73 | 81 | 51-125 | | | |
| Terphenyl-d14 (S) | % | | | | | | 82 | 81 | 57-125 | | | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# QUALIFIERS

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

## DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

## LABORATORIES

| | |
|---|---|
| PASI-M | Pace Analytical Services - Minneapolis |

## ANALYTE QUALIFIERS

| | |
|---|---|
| 1M | Surrogate recovery outside laboratory control limits due to matrix interferences. |
| D3 | Sample was diluted due to the presence of high levels of non-target analytes or other matrix interference. |
| M1 | Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery. |
| R1 | RPD value was outside control limits. |

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092 ENBRIDGE ENVIRONMEN_R
Pace Project No.: 10235240

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|---|---|---|---|---|---|
| **10235240001** | **2013-PIPERACK-S-1_4.0-4.0 ft** | TPH GRO/PVOC WI ext. | GCV/11048 | WI MOD GRO | GCV/11050 |
| **10235240002** | **2013-PIPERACK-S-2_4.0-4.0 ft** | TPH GRO/PVOC WI ext. | GCV/11054 | WI MOD GRO | GCV/11056 |
| **10235240003** | **2013-PIPERACK-S-3_4.0-4.0 ft** | TPH GRO/PVOC WI ext. | GCV/11054 | WI MOD GRO | GCV/11056 |
| **10235240001** | **2013-PIPERACK-S-1_4.0-4.0 ft** | ASTM D2974 | MPRP/40608 | | |
| **10235240002** | **2013-PIPERACK-S-2_4.0-4.0 ft** | ASTM D2974 | MPRP/40608 | | |
| **10235240003** | **2013-PIPERACK-S-3_4.0-4.0 ft** | ASTM D2974 | MPRP/40608 | | |
| **10235240003** | **2013-PIPERACK-S-3_4.0-4.0 ft** | EPA 3550 | OEXT/22333 | EPA 8270 by SIM | MSSV/9515 |

# Chain of Custody

BARR

4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

1128

10235240

Project Number: 49161092

Project Name: Enbridge Environmental Support

Sample Origination State: WI (use two letter postal state abbreviation)

COC Number: № 40103

COC 1 of 1

Project Manager: REE

Project QC Contact: AAN

Sampled by: CJG2

Laboratory: Pace

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Type: Grab | Type: Comp. | Type: QC | Number of Containers/Preservative: Water: VOCs (HCl) #1 | SVOCs (unpreserved) #2 | Dissolved Metals ($HNO_3$) | Total Metals ($HNO_3$) | General (unpreserved) #3 | Diesel Range Organics (HCl) | Nutrients ($H_2SO_4$) #4 | Soil: VOCs (tared MeOH) #1 | GRO, BTEX (tared MeOH) #1 | DRO (tared unpreserved) | Metals (unpreserved) | SVOCs (unpreserved) #2 | % Solids (plastic vial, unpres.) | PVOC (-MTBE) | PAH | Total Number Of Containers | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. 2013-Piperack-S-1 | 4 | 4 | Ft | 7/9/13 | 1800 | | X | X | | | | | | | | | | | | | | | X | X | | 2 | PVOC-MTBE, % solids |
| 2. 2013-Piperack-S-2 | 4 | 4 | Ft | 7/9/13 | 1805 | | X | X | | | | | | | | | | | | | | | X | X | | 2 | For all samples |
| 3. 2013-Piperack-S-3 | 4 | 4 | Ft | 7/9/13 | 1810 | | X | X | | | | | | | | | | | | | | | X | X | X | 3 | * also PAH |
| 4. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | | | | | | | | | Normal turnaround |
| 8. | | | | | | | | | | | | | | | | | | | | | | | | | | | time |
| 9. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| Relinquished By: | On Ice? Y N | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| | Y N | 7/12/13 | 1345 | T=2.4 | 7/13/13 | 815 |
| Relinquished By: | On Ice? Y N | Date | Time | Received by: | Date | Time |
| | | | | | | |

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other: Dropped off @ Pace Duluth

Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:\RLG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

| Pace Analytical® | Document Name: Sample Condition Upon Receipt Form | Document Revised: 28Jan2013 Page 1 of 1 |
|---|---|---|
| | Document No.: F-MN-L-213-rev.06 | Issuing Authority: Pace Minnesota Quality Office |

**Sample Condition Upon Receipt**

**Client Name:** Bare

**Project #:** WO# : 10235240

10235240

**Courier:** ☒ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other: _____

**Tracking Number:** 7962 7031 8584

**Custody Seal on Cooler/Box Present?** ☒ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No

**Optional:** Proj. Due Date: Proj. Name:

**Packing Material:** ☒ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other: _____ **Temp Blank?** ☒ Yes ☐ No

**Thermom. Used:** ☒ B88A912167504 ☐ 80512447 ☐ 72337080 **Type of Ice:** ☒ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

**Cooler Temp Read (°C):** 2.4 **Cooler Temp Corrected (°C):** 2.4 **Biological Tissue Frozen?** ☐ Yes ☐ No

Temp should be above freezing to 6°C **Correction Factor:** 0.0 **Date and Initials of Person Examining Contents:** AMS 7/15/13

**Comments:**

| | | | | |
|---|---|---|---|---|
| Chain of Custody Present? | ☒ Yes | ☐ No | ☐ N/A | 1. |
| Chain of Custody Filled Out? | ☒ Yes | ☐ No | ☐ N/A | 2. |
| Chain of Custody Relinquished? | ☒ Yes | ☐ No | ☐ N/A | 3. |
| Sampler Name and/or Signature on COC? | ☒ Yes | ☐ No | ☐ N/A | 4. |
| Samples Arrived within Hold Time? | ☒ Yes | ☐ No | ☐ N/A | 5. |
| Short Hold Time Analysis (<72 hr)? | ☐ Yes | ☒ No | ☐ N/A | 6. |
| Rush Turn Around Time Requested? | ☐ Yes | ☒ No | ☐ N/A | 7. |
| Sufficient Volume? | ☒ Yes | ☐ No | ☐ N/A | 8. |
| Correct Containers Used? | ☒ Yes | ☐ No | ☐ N/A | 9. |
| -Pace Containers Used? | ☒ Yes | ☐ No | ☐ N/A | |
| Containers Intact? | ☒ Yes | ☐ No | ☐ N/A | 10. |
| Filtered Volume Received for Dissolved Tests? | ☐ Yes | ☐ No | ☒ N/A | 11. |
| Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL | ☒ Yes | ☐ No | ☐ N/A | 12. |
| All containers needing acid/base preservation have been checked? Noncompliances are noted in 13. | ☐ Yes | ☐ No | ☒ N/A | 13. ☐ $HNO_3$ ☐ $H_2SO_4$ ☐ NaOH ☐ HCl |
| All containers needing preservation are found to be in compliance with EPA recommendation? ($HNO_3$, $H_2SO_4$, HCl<2; NaOH>12) | ☐ Yes | ☐ No | ☒ N/A | Sample # |
| Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water) | ☐ Yes | ☒ No | | Initial when completed: Lot # of added preservative: |
| Headspace in VOA Vials ( >6mm)? | ☐ Yes | ☐ No | ☒ N/A | 14. |
| Trip Blank Present? | ☐ Yes | ☒ No | ☐ N/A | 15. |
| Trip Blank Custody Seals Present? | ☐ Yes | ☒ No | ☐ N/A | |
| Pace Trip Blank Lot # (if purchased): | | | | |

**CLIENT NOTIFICATION/RESOLUTION** **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

**Project Manager Review:** _____ **Date:** 7/15/13

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e out of hold, incorrect preservative, out of temp, incorrect containers)

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

August 12, 2013

Andrea Nord
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

RE: Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on August 02, 2013. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

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

Sincerely,

Andrea Opland

andrea.opland@pacelabs.com
Project Manager

Enclosures

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# CERTIFICATIONS

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

**Minnesota Certification IDs**

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Hawaii Certification #Pace
Idaho Certification #: MN00064
Illinois Certification #: 200011
Kansas Certification #: E-10167
Louisiana Certification #: 03086
Louisiana Certification #: LA080009
Maine Certification #: 2007029
Maryland Certification #: 322
Michigan DEQ Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT CERT0092
Nebraska Certification #: Pace
Nevada Certification #: MN_00064
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Utah Certification #: MN00064
Virginia/DCLS Certification #: 002521
Virginia/VELAP Certification #: 460163
Washington Certification #: C754
West Virginia Certification #: 382
Wisconsin Certification #: 999407970

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## SAMPLE SUMMARY

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|---|---|---|---|---|
| **10237460001** | **2013 Pipe Rack-S-4** | Solid | 08/01/13 13:10 | 08/02/13 09:35 |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## SAMPLE ANALYTE COUNT

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|---|---|---|---|---|---|
| **10237460001** | **2013 Pipe Rack-S-4** | WI MOD GRO | LLC | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| | | EPA 8270 by SIM | AJP | 18 | PASI-M |

**Pace Analytical Services, Inc.**
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(612)607-1700

# PROJECT NARRATIVE

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

**Method:** **WI MOD GRO**
**Description:** WIGRO GCV
**Client:** Barr Engineering
**Date:** August 12, 2013

**General Information:**
1 sample was analyzed for WI MOD GRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with TPH GRO/PVOC WI ext. with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Duplicate Sample:**
All duplicate sample results were within method acceptance criteria with any exceptions noted below.

**Additional Comments:**

Pace Analytical Services, Inc.
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Minneapolis, MN 55414
(612)607-1700

# PROJECT NARRATIVE

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

**Method:** **EPA 8270 by SIM**
**Description:** 8270 MSSV PAH by SIM
**Client:** Barr Engineering
**Date:** August 12, 2013

**General Information:**
1 sample was analyzed for EPA 8270 by SIM. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with EPA 3550 with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

QC Batch: OEXT/22549

A matrix spike and matrix spike duplicate (MS/MSD) were performed on the following sample(s): 10236157027

M1: Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.

- MS (Lab ID: 1494945)
  - Acenaphthene
  - Acenaphthylene
  - Anthracene
  - Chrysene
  - Fluoranthene
  - Fluorene
  - Naphthalene
  - Phenanthrene
  - Pyrene
- MSD (Lab ID: 1494946)

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# PROJECT NARRATIVE

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

**Method:** **EPA 8270 by SIM**
**Description:** 8270 MSSV PAH by SIM
**Client:** Barr Engineering
**Date:** August 12, 2013

QC Batch: OEXT/22549

A matrix spike and matrix spike duplicate (MS/MSD) were performed on the following sample(s): 10236157027

M1: Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.

- Acenaphthene
- Acenaphthylene
- Anthracene
- Chrysene
- Fluoranthene
- Fluorene
- Naphthalene
- Phenanthrene
- Pyrene

R1: RPD value was outside control limits.

- MSD (Lab ID: 1494946)
  - Naphthalene

**Additional Comments:**

Analyte Comments:

QC Batch: OEXT/22549

1M: MS/MSD do not match parent sample or any other sample in batch. This could be due to soil sample not being homogenized.

- MS (Lab ID: 1494945)
  - 2-Fluorobiphenyl (S)
- MSD (Lab ID: 1494946)
  - 2-Fluorobiphenyl (S)

E: Analyte concentration exceeded the calibration range. The reported result is estimated.

- MS (Lab ID: 1494945)
  - Fluoranthene
  - Naphthalene
  - Phenanthrene
- MSD (Lab ID: 1494946)
  - Fluoranthene
  - Naphthalene
  - Phenanthrene

This data package has been reviewed for quality and completeness and is approved for release.

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## ANALYTICAL RESULTS

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

**Sample: 2013 Pipe Rack-S-4** **Lab ID: 10237460001** Collected: 08/01/13 13:10 Received: 08/02/13 09:35 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| Benzene | **<0.065** | mg/kg | 0.065 | 0.010 | 1 | 08/07/13 10:23 | 08/08/13 02:56 | 71-43-2 | |
| Ethylbenzene | **<0.065** | mg/kg | 0.065 | 0.0091 | 1 | 08/07/13 10:23 | 08/08/13 02:56 | 100-41-4 | |
| Toluene | **<0.065** | mg/kg | 0.065 | 0.0065 | 1 | 08/07/13 10:23 | 08/08/13 02:56 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | **<0.065** | mg/kg | 0.065 | 0.010 | 1 | 08/07/13 10:23 | 08/08/13 02:56 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | **<0.065** | mg/kg | 0.065 | 0.0091 | 1 | 08/07/13 10:23 | 08/08/13 02:56 | 108-67-8 | |
| Xylene (Total) | **<0.20** | mg/kg | 0.20 | 0.022 | 1 | 08/07/13 10:23 | 08/08/13 02:56 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 103 | % | 80-125 | | 1 | 08/07/13 10:23 | 08/08/13 02:56 | 98-08-8 | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **26.4** | % | 0.10 | 0.10 | 1 | | 08/06/13 00:00 | | |
| **8270 MSSV PAH by SIM** | Analytical Method: EPA 8270 by SIM Preparation Method: EPA 3550 | | | | | | | | |
| Acenaphthene | **<13.5** | ug/kg | 13.5 | 1.4 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 83-32-9 | |
| Acenaphthylene | **<13.5** | ug/kg | 13.5 | 1.9 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 208-96-8 | |
| Anthracene | **38.5** | ug/kg | 13.5 | 6.8 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 120-12-7 | |
| Benzo(a)anthracene | **117** | ug/kg | 13.5 | 6.8 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 56-55-3 | |
| Benzo(a)pyrene | **91.0** | ug/kg | 13.5 | 6.8 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 50-32-8 | |
| Benzo(b)fluoranthene | **122** | ug/kg | 13.5 | 0.77 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 205-99-2 | |
| Benzo(g,h,i)perylene | **45.8** | ug/kg | 13.5 | 6.8 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 191-24-2 | |
| Benzo(k)fluoranthene | **48.3** | ug/kg | 13.5 | 1.2 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 207-08-9 | |
| Chrysene | **126** | ug/kg | 13.5 | 1.0 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 218-01-9 | |
| Dibenz(a,h)anthracene | **14.4** | ug/kg | 13.5 | 6.8 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 53-70-3 | |
| Fluoranthene | **232** | ug/kg | 13.5 | 0.97 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 206-44-0 | |
| Fluorene | **<13.5** | ug/kg | 13.5 | 1.7 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 86-73-7 | |
| Indeno(1,2,3-cd)pyrene | **40.5** | ug/kg | 13.5 | 6.8 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 193-39-5 | |
| Naphthalene | **<13.5** | ug/kg | 13.5 | 1.7 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 91-20-3 | |
| Phenanthrene | **113** | ug/kg | 13.5 | 0.88 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 85-01-8 | |
| Pyrene | **226** | ug/kg | 13.5 | 0.81 | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 129-00-0 | |
| ***Surrogates*** | | | | | | | | | |
| 2-Fluorobiphenyl (S) | 71 | % | 51-125 | | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 321-60-8 | |
| Terphenyl-d14 (S) | 87 | % | 57-125 | | 1 | 08/06/13 08:01 | 08/07/13 12:12 | 1718-51-0 | |

## REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# QUALITY CONTROL DATA

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

QC Batch: GCV/11170
QC Batch Method: TPH GRO/PVOC WI ext.
Analysis Method: WI MOD GRO
Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10237460001

METHOD BLANK: 1496342 Matrix: Solid
Associated Lab Samples: 10237460001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 08/08/13 00:20 | |
| 1,3,5-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 08/08/13 00:20 | |
| Benzene | mg/kg | <0.050 | 0.050 | 08/08/13 00:20 | |
| Ethylbenzene | mg/kg | <0.050 | 0.050 | 08/08/13 00:20 | |
| Toluene | mg/kg | <0.050 | 0.050 | 08/08/13 00:20 | |
| Xylene (Total) | mg/kg | <0.15 | 0.15 | 08/08/13 00:20 | |
| a,a,a-Trifluorotoluene (S) | % | 103 | 80-125 | 08/08/13 00:20 | |

LABORATORY CONTROL SAMPLE & LCSD: 1496343 1496344

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | 5 | 4.7 | 4.9 | 93 | 98 | 80-120 | 5 | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | 5 | 4.6 | 4.9 | 93 | 98 | 80-120 | 5 | 20 | |
| Benzene | mg/kg | 5 | 4.4 | 4.7 | 87 | 95 | 80-120 | 9 | 20 | |
| Ethylbenzene | mg/kg | 5 | 4.5 | 4.9 | 89 | 98 | 80-120 | 9 | 20 | |
| Toluene | mg/kg | 5 | 4.4 | 4.9 | 89 | 97 | 80-120 | 9 | 20 | |
| Xylene (Total) | mg/kg | 15 | 14.0 | 15.0 | 93 | 100 | 80-120 | 7 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 98 | 98 | 80-125 | | | |

MATRIX SPIKE SAMPLE: 1496345

| Parameter | Units | 10237643003 Result | Spike Conc. | MS Result | MS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | ND | 5.7 | 6.0 | 105 | 80-120 | |
| 1,3,5-Trimethylbenzene | mg/kg | ND | 5.7 | 6.0 | 104 | 80-120 | |
| Benzene | mg/kg | ND | 5.7 | 5.6 | 98 | 80-120 | |
| Ethylbenzene | mg/kg | ND | 5.7 | 5.8 | 101 | 80-120 | |
| Toluene | mg/kg | ND | 5.7 | 5.7 | 100 | 80-120 | |
| Xylene (Total) | mg/kg | ND | 17.2 | 17.9 | 104 | 80-120 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 99 | 80-125 | |

SAMPLE DUPLICATE: 1496346

| Parameter | Units | 10237643005 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| 1,2,4-Trimethylbenzene | mg/kg | ND | <0.057 | | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | ND | <0.057 | | 20 | |
| Benzene | mg/kg | ND | <0.057 | | 20 | |
| Ethylbenzene | mg/kg | ND | <0.057 | | 20 | |
| Toluene | mg/kg | ND | <0.057 | | 20 | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

SAMPLE DUPLICATE: 1496346

| Parameter | Units | 10237643005 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Xylene (Total) | mg/kg | ND | <0.17 | | 20 | |
| a,a,a-Trifluorotoluene (S) | % | 102 | 102 | 4 | | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

QC Batch: MPRP/41098
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10237460001

SAMPLE DUPLICATE: 1495745

| Parameter | Units | 10237537005 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 14.6 | 14.3 | 2 | 30 | |

SAMPLE DUPLICATE: 1495746

| Parameter | Units | 10237460001 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 26.4 | 26.3 | .2 | 30 | |

## REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

QC Batch: OEXT/22549
QC Batch Method: EPA 3550
Analysis Method: EPA 8270 by SIM
Analysis Description: 8270 Solid PAH by SIM MSSV
Associated Lab Samples: 10237460001

METHOD BLANK: 1494943 Matrix: Solid
Associated Lab Samples: 10237460001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Acenaphthene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Acenaphthylene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Anthracene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Benzo(a)anthracene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Benzo(a)pyrene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Benzo(b)fluoranthene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Benzo(g,h,i)perylene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Benzo(k)fluoranthene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Chrysene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Dibenz(a,h)anthracene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Fluoranthene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Fluorene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Indeno(1,2,3-cd)pyrene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Naphthalene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Phenanthrene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| Pyrene | ug/kg | <10.0 | 10.0 | 08/07/13 09:35 | |
| 2-Fluorobiphenyl (S) | % | 73 | 51-125 | 08/07/13 09:35 | |
| Terphenyl-d14 (S) | % | 87 | 57-125 | 08/07/13 09:35 | |

LABORATORY CONTROL SAMPLE: 1494944

| Parameter | Units | Spike Conc. | LCS Result | LCS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|
| Acenaphthene | ug/kg | 33.3 | 21.8 | 65 | 45-125 | |
| Acenaphthylene | ug/kg | 33.3 | 20.4 | 61 | 45-125 | |
| Anthracene | ug/kg | 33.3 | 27.9 | 84 | 53-125 | |
| Benzo(a)anthracene | ug/kg | 33.3 | 21.9 | 66 | 56-125 | |
| Benzo(a)pyrene | ug/kg | 33.3 | 30.9 | 93 | 55-125 | |
| Benzo(b)fluoranthene | ug/kg | 33.3 | 24.5 | 73 | 59-125 | |
| Benzo(g,h,i)perylene | ug/kg | 33.3 | 26.7 | 80 | 54-125 | |
| Benzo(k)fluoranthene | ug/kg | 33.3 | 35.8 | 108 | 52-125 | |
| Chrysene | ug/kg | 33.3 | 35.6 | 107 | 54-125 | |
| Dibenz(a,h)anthracene | ug/kg | 33.3 | 24.9 | 75 | 53-125 | |
| Fluoranthene | ug/kg | 33.3 | 27.5 | 82 | 60-125 | |
| Fluorene | ug/kg | 33.3 | 24.2 | 73 | 52-125 | |
| Indeno(1,2,3-cd)pyrene | ug/kg | 33.3 | 25.4 | 76 | 55-125 | |
| Naphthalene | ug/kg | 33.3 | 18.1 | 54 | 40-125 | |
| Phenanthrene | ug/kg | 33.3 | 23.2 | 70 | 50-125 | |
| Pyrene | ug/kg | 33.3 | 29.7 | 89 | 59-125 | |
| 2-Fluorobiphenyl (S) | % | | | 63 | 51-125 | |
| Terphenyl-d14 (S) | % | | | 90 | 57-125 | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 1494945 1494946

| Parameter | Units | 10236157027 Result | MS Spike Conc. | MSD Spike Conc. | MS Result | MSD Result | MS % Rec | MSD % Rec | % Rec Limits | RPD | Max RPD | Qual |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Acenaphthene | ug/kg | ND | 33.3 | 33.3 | 122 | 93.5 | 366 | 281 | 30-129 | 26 | 30 | M1 |
| Acenaphthylene | ug/kg | ND | 33.3 | 33.3 | 55.5 | 50.3 | 166 | 151 | 30-150 | 10 | 30 | M1 |
| Anthracene | ug/kg | ND | 33.3 | 33.3 | 55.6 | 59.2 | 167 | 178 | 30-150 | 6 | 30 | M1 |
| Benzo(a)anthracene | ug/kg | ND | 33.3 | 33.3 | 43.7 | 47.0 | 131 | 141 | 30-150 | 7 | 30 | |
| Benzo(a)pyrene | ug/kg | ND | 33.3 | 33.3 | 34.1 | 35.3 | 102 | 106 | 30-150 | 4 | 30 | |
| Benzo(b)fluoranthene | ug/kg | ND | 33.3 | 33.3 | 36.0 | 38.4 | 108 | 115 | 30-150 | 7 | 30 | |
| Benzo(g,h,i)perylene | ug/kg | ND | 33.3 | 33.3 | 29.5 | 29.9 | 88 | 90 | 30-150 | 1 | 30 | |
| Benzo(k)fluoranthene | ug/kg | ND | 33.3 | 33.3 | 39.9 | 41.3 | 120 | 124 | 30-150 | 3 | 30 | |
| Chrysene | ug/kg | ND | 33.3 | 33.3 | 74.6 | 81.6 | 224 | 245 | 30-150 | 9 | 30 | M1 |
| Dibenz(a,h)anthracene | ug/kg | ND | 33.3 | 33.3 | 27.6 | 28.9 | 83 | 87 | 30-150 | 4 | 30 | |
| Fluoranthene | ug/kg | ND | 33.3 | 33.3 | 350 | 400 | 1050 | 1200 | 30-150 | 13 | 30 | E,M1 |
| Fluorene | ug/kg | ND | 33.3 | 33.3 | 103 | 112 | 309 | 337 | 36-125 | 9 | 30 | M1 |
| Indeno(1,2,3-cd)pyrene | ug/kg | ND | 33.3 | 33.3 | 28.4 | 29.2 | 85 | 88 | 30-150 | 3 | 30 | |
| Naphthalene | ug/kg | ND | 33.3 | 33.3 | 499 | 365 | 1500 | 1090 | 30-150 | 31 | 30 | E,M1, R1 |
| Phenanthrene | ug/kg | ND | 33.3 | 33.3 | 613 | 679 | 1840 | 2040 | 30-150 | 10 | 30 | E,M1 |
| Pyrene | ug/kg | ND | 33.3 | 33.3 | 235 | 265 | 704 | 794 | 30-150 | 12 | 30 | M1 |
| 2-Fluorobiphenyl (S) | % | | | | | | 80 | 81 | 51-125 | | | 1M |
| Terphenyl-d14 (S) | % | | | | | | 96 | 98 | 57-125 | | | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# QUALIFIERS

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

## DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

## LABORATORIES

| | |
|---|---|
| PASI-M | Pace Analytical Services - Minneapolis |

## ANALYTE QUALIFIERS

| | |
|---|---|
| 1M | MS/MSD do not match parent sample or any other sample in batch. This could be due to soil sample not being homogenized. |
| E | Analyte concentration exceeded the calibration range. The reported result is estimated. |
| M1 | Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery. |
| R1 | RPD value was outside control limits. |

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092.02 003 018 Enbridge
Pace Project No.: 10237460

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|---|---|---|---|---|---|
| **10237460001** | **2013 Pipe Rack-S-4** | TPH GRO/PVOC WI ext. | GCV/11170 | WI MOD GRO | GCV/11171 |
| **10237460001** | **2013 Pipe Rack-S-4** | ASTM D2974 | MPRP/41098 | | |
| **10237460001** | **2013 Pipe Rack-S-4** | EPA 3550 | OEXT/22549 | EPA 8270 by SIM | MSSV/9593 |

# Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

1124

10237460

Project Number: 49/6 1092.02 003 018

Project Name: Enbridge Pipe Rack

Sample Origination State: W I (use two letter postal state abbreviation)

COC Number: № 40513

COC 1 of 1

Project Manager: REE

Project QC Contact: AAN

Sampled by: CJG2

Laboratory: Pace

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Type: Grab | Type: Comp. | Type: QC | Number of Containers/Preservative — Water: VOCs (HCl) #1 | SVOCs (unpreserved) #2 | Dissolved Metals ($HNO_3$) | Total Metals ($HNO_3$) | General (unpreserved) #3 | Diesel Range Organics (HCl) | Nutrients ($H_2SO_4$) #4 | Soil: VOCs (tared MeOH) #1 | GRO, BTEX (tared MeOH) #1 | DRO (tared unpreserved) | Metals (unpreserved) | SVOCs (unpreserved) #2 | % Solids (plastic vial, unpres.) | PAH | PIOC-MTBE | Total Number Of Containers | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. 2013 Pipe Rack-S-4 | 3.5 | 3.5 | Ft | 8/1/13 | 1310 | | X | X | | | | | | | | | | | | | | | X | 1 | 1 | 3 | PIOC-MTBE, PAH moisture 001 |
| 2. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 3. | | | | | | | | | | | | | | | | | | | | | | | | | | | Normal TAT |
| 4. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Common Parameter/Container - Preservation Key

#1 Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| CJG2 | (Y) N | 8/1/13 | 1420 | | 8/1/13 | 1420 |
| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
| | (Y) N | 8/1/13 | 1700 | by Fed Ex | 8-2-13 | 935 |

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other: Dropped off @ Pace Duluth

Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:RLG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

| Pace Analytical | Document Name: **Sample Condition Upon Receipt Form** | Document Revised: 28Jan2013 Page 1 of 1 |
|---|---|---|
| | Document No.: **F-MN-L-213-rev.06** | Issuing Authority: Pace Minnesota Quality Office |

**Sample Condition Upon Receipt**

**Client Name:** BARR

**Project #:** WO# : 10237460

10237460

**Courier:** ☒ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☒ ~~Pace~~ 8213 ☐ Other:

**Tracking Number:** (96) 2422 9470 0355 1500 1546

**Custody Seal on Cooler/Box Present?** ☒ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

**Packing Material:** ☒ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☒ Yes ☐ No

**Thermom. Used:** ☒ B88A912167504 ☐ 80512447 ☐ 72337080 **Type of Ice:** ☒ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

**Cooler Temp Read (°C):** 2.6 **Cooler Temp Corrected (°C):** 2.8 **Biological Tissue Frozen?** ☐ Yes ☐ No

Temp should be above freezing to 6°C **Correction Factor:** +.2 **Date and Initials of Person Examining Contents:** 8-2-13/LS

| | | | | **Comments:** |
|---|---|---|---|---|
| Chain of Custody Present? | ☒ Yes | ☐ No | ☐ N/A | 1. |
| Chain of Custody Filled Out? | ☒ Yes | ☐ No | ☐ N/A | 2. |
| Chain of Custody Relinquished? | ☒ Yes | ☐ No | ☐ N/A | 3. |
| Sampler Name and/or Signature on COC? | ☒ Yes | ☐ No | ☐ N/A | 4. |
| Samples Arrived within Hold Time? | ☒ Yes | ☐ No | ☐ N/A | 5. |
| **Short Hold Time Analysis (<72 hr)?** | ☐ Yes | ☒ No | ☐ N/A | 6. |
| **Rush Turn Around Time Requested?** | ☐ Yes | ☒ No | ☐ N/A | 7. |
| Sufficient Volume? | ☒ Yes | ☐ No | ☐ N/A | 8. |
| Correct Containers Used? | ☒ Yes | ☐ No | ☐ N/A | 9. |
| -Pace Containers Used? | ☒ Yes | ☐ No | ☐ N/A | |
| Containers Intact? | ☒ Yes | ☐ No | ☐ N/A | 10. |
| Filtered Volume Received for Dissolved Tests? | ☐ Yes | ☐ No | ☒ N/A | 11. |
| Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL | ☒ Yes | ☐ No | ☐ N/A | 12. |
| All containers needing acid/base preservation have been checked? Noncompliances are noted in 13. | ☐ Yes | ☐ No | ☒ N/A | 13. ☐ $HNO_3$ ☐ $H_2SO_4$ ☐ NaOH ☐ HCl |
| All containers needing preservation are found to be in compliance with EPA recommendation? ($HNO_3$, $H_2SO_4$, HCl<2; NaOH>12) | ☐ Yes | ☐ No | ☒ N/A | Sample # |
| Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water) | ☐ Yes | ☒ No | | Initial when completed: Lot # of added preservative: |
| Headspace in VOA Vials ( >6mm)? | ☐ Yes | ☐ No | ☒ N/A | 14. |
| Trip Blank Present? | ☐ Yes | ☐ No | ☒ N/A | 15. |
| Trip Blank Custody Seals Present? | ☐ Yes | ☐ No | ☒ N/A | |
| Pace Trip Blank Lot # (if purchased): | | | | |

**CLIENT NOTIFICATION/RESOLUTION** **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

**Project Manager Review:** Date: 8/5/13

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e out of hold, incorrect preservative, out of temp, incorrect containers)

# Attachment D

## Waste Disposal Documentation

*Shamrock Landfill Documentation (Profile #CL12-0067) (8/27/2012)*
*Geotechnical Investigation Hydrovac Borings, Water Valve Replacement Excavation and Fire Hydrant Replacement Excavation*

*WLSSD Documentation (11/15/2012)*
*Water Valve Replacement Excavation*

*Shamrock Landfill Documentation (Profile #CL13-0027) (6/14/2013)*
*Pipe Rack Footing Excavations and Central Manifold Excavation*

*WLSSD Documentation (8/12/2013)*
*Pipe Rack Footing Excavation and Central Manifold Excavation*

# Waste Profile Sheet

Shamrock Landfill

| P.O. Number | Customer Code | SKB Representative Jon Penheiter | CL |
|---|---|---|---|

**I. Generator Information**

| | | | |
|---|---|---|---|
| Generator Name: Enbridge Pipelines Limited Partnership, LLC | | Generator EPA ID Number | SIC Code |
| Generator Location: Enbridge Superior Terminal -Pipe Rack Boring | County: Douglas | Generator Contact: Paul Turner | |
| | | Phone: 715-398-4752 | Fax: 715-398-3223 |
| Generator Mailing Address (if different: 1320 Grand Ave, Superior, WI 54880 | | Generator Email Address: paul.turner@enbridge.com | |
| Bill To Name & Address: Enbridge Energy, 1100 Louisiana Ave, STE. 3300, Houston, TX 77002 | Bill To #: | Billing Contact: Paul Turner | |
| | | Phone: 715-398-9192 | Fax: 715-398-3223 |
| Invoice Contact: | | Billing Email Address: paul.turner@enbridge.com | |

**II. Waste Generation Information**

| | | |
|---|---|---|
| Waste Name: Pipe Rack Boring | Estimated rate of waste generation: 10<br>☐ Lbs. ☐ tons ☒ cy ☐ drums | ☒ one time<br>☐ yearly |
| Generator Facility Operations and/or Site History: Enbridge Pipeline Terminal | | |
| Describe the generating process or source of contaminated soil/debris and/or waste: Pipeline Terminal Activities | | |

**III. Waste Composition and Constituents (list all known)**

| | Actual Range % | ppm |
|---|---|---|
| Crude contaminated soil | 100 | |
| | | |
| | | |
| | | |

**IV. Waste Properties**

| Physical state: | Free Liquids: | pH Range: | Flash point: | Color: | Odor (describe): |
|---|---|---|---|---|---|
| ☒ Solid ☐ Liquid<br>☐ Sludge ☐ Gas | ☐ Yes ☒ No<br>Content _____% | ☐ <2 ☐ 2-4<br>☐ 5-8 ☐ 8-12.4<br>☐ >12.5 | ☐ ≤ 140ºF<br>☐ > 140ºF to < 200ºF<br>☐ > 200ºF | Brown | petroleum odor |

**V. Waste Classification**

| | | | |
|---|---|---|---|
| Waste stream properties (answer ALL questions) | | Does this waste contain absorbents? | ☐ Yes ☒ No |
| Does this waste stream contain any D, F, K, U or P listed as hazardous waste, either in pure form, as a mixture, or treatment residue? | ☐ Yes ☒ No | Is this waste lethal (by Minn. Rules 7045.0131 Subp. 6)? | ☐ Yes ☒ No |
| Does this waste stream contain PCB material<br>If yes, concentration: _____ppm | ☐ Yes ☒ No | Is this waste recyclable? | ☐ Yes ☒ No |
| | | Is this waste explosive? | ☐ Yes ☒ No |
| Does this waste stream contain fuming acids? | ☐ Yes ☒ No | Is this waste infectious? | ☐ Yes ☒ No |
| Does this waste contain asbestos? | ☐ Yes ☒ No | Is this putrescible waste? | ☐ Yes ☒ No |
| Does this waste contain oxidizers? | ☐ Yes ☒ No | Is this waste demolition debris? | ☐ Yes ☒ No |
| Does this waste contain radioactive material? | ☐ Yes ☒ No | Is this waste sewer sludge? | ☐ Yes ☒ No |

**Please attach any available information or analytical test results that have previously been performed on this waste that substantiates these determinations. Include MSDS's and any information from other agencies (i.e., MPCA, USEPA)**

**VI. Shipping Information**

| | | | |
|---|---|---|---|
| Proper DOT Shipping Name (per CFR 172.101) where applicable | | | |
| Reportable Quantity | DOT Hazard Class | UN/NA Number | Packing Group |
| Method of packaging: ☐ drums (size _____)<br>☒ Bulk Solids ☐ boxes (size _____) | | Method of shipment<br>☐ Roll-off ☒ End dump ☐ Rail ☐ Other (Specify) _____ | |

**VII. Certification of Non Hazardous Waste & Approval Conditions**

I hereby certify and warrant, on behalf of the generator and myself that, to the best of my knowledge and belief, the information contained herein is accurate, and true and that the waste is nonhazardous as defined in Title 42, Unites States Code Section 6903, Minnesota Statute Section 116.06, Subdivision 13, and/or any rules adopted by the Minnesota Pollution Control Agency under Minnesota Statute Section 116.07.

I understand that any approval is no longer valid if there are any changes in the process generating the waste or there have been changes in the composition of the waste. Therefore, if the composition of the waste stream changes or potentially changes, I or someone representing the generator, will immediately

generator, hereby agree to fully indemnify SKB Environmental for any damages and/or costs incurred as a result

e.

| Paul Turner | Environmental Analyst | |
|---|---|---|
| Printed Name | Title | Date |

Version 2011

SKB ENVIRONMENTAL

Shamrock Landfill

August 27, 2012

Paul Turner
Enbridge Pipelines Limited Partnership, LLC
Central Square Office
1320 Grand Ave
Superior, WI 54880

RE: CL12-0067 Crude Contaminated Soil (Pipe Rack Boring)

Dear Mr. Turner,

This agreement will confirm the price and length of service for disposal and /or transportation of your non-hazardous industrial material at our facility. This agreement is for the term of the Waste Approval granted by SKB and is for all services ordered and performance initiated within such period and does include the disposal surcharge fees which you are obligated to pay as of the date of this agreement. SKB may incur additional costs including but not limited to increases in state and local taxes. SKB may pass these costs on to the customer only after notification to the Customer. This agreement grants SKB the exclusive right to dispose of the referenced waste for the term of this agreement. This agreement shall automatically renew thereafter for an additional term of 24 months "Renewal Term" unless either party provides the other party written notification of termination at least 90 days prior to the termination of the then-existing term. SKB will notify the customer prior to the expiration of the agreement of any rate changes prior to the start of the Renewal Term.

Payment and thirty (30) days. Interest will be charged at a rate of 1 ½% per month (18% annually) on any unpaid balance after the date of the invoice. In the event Customer terminates this Agreement prior to its expiration as a result of a breach by SKB or SKB terminates this agreement for Customer's breach (including non-payment), Customer agrees to pay to SKB as liquidated damages a sum calculated as follows: (1) if the remaining term under this agreement is six or more months Customer shall pay its average monthly charges multiplied by six; (2) if the remaining term under this agreement is less than six months Customer shall pay its average monthly charges multiplied by the number of months remaining in the term. Customer expressly acknowledges that in the event of an unauthorized termination of this agreement the anticipated loss to SKB in such event is estimated to be the amount set forth in the foregoing liquidated damages provision and such estimated value is reasonable and is not imposed as a penalty.

These prices are based on an approved waste stream composition. In the event that a non-conforming waste is received, you will be notified of additional charges, when applicable.

To accept this agreement, please sign one copy and return it to our Rosemount, MN office at SKB Rosemount, 13425 Courthouse Blvd, Rosemount, MN 55068 or Via Fax at 651/438-1549 or email to jonp@skbinc.com.

SKB Shamrock Landfill

Jon Penheiter

Customer ACCEPTED BY: (name, position) _____

DATE: _____

WASTE APPROVAL Period: 8/27/2012 to 8/9/2014

8/28/2012 Environmental Analyst

SKB ENVIRONMENTAL

Shamrock Landfill

## Bill To Customer

Enbridge Pipelines Limited Partnership, LLC
Central Square Office
1320 Grand Ave
Superior, WI 54880

## Service For Generator

Enbridge Pipelines Limited Partnership, LLC
2800 East 21st St
Superior, WI 54880

## Disposal

Waste Description: Crude Contaminated Soil (Pipe Rack Boring)

Estimated Volume: 10 YARDS / ONE TIME ONLY

Disposal Method: Secure Non-Hazardous Landfill

Treatment Method: None Expected For Conforming Waste

## Pricing

| | | | |
|---|---|---|---|
| Disposal | $19.00 | Per Ton | Crude Contaminated Soil (Pipe Rack Boring) |

SKB ENVIRONMENTAL

Shamrock Landfill

# Notification of Waste Acceptance



**CUSTOMER INFORMATION**

EPA ID#: WID981092133
Enbridge Pipelines Limited Partnership,
Enbridge Superior Terminal

2800 East 21st St
Superior, WI 54880
Contact: Paul Turner
Phone: (715) 398-4752

**INVOICE INFORMATION**

Bill #: 2133
Enbridge Pipelines Limited Partnership,
Central Square Office

1320 Grand Ave
Superior, WI 54880
Contact: Paul Turner
Phone: (715) 398-4752

Profile Sheet #:
Waste Stream #: CL12-0067
Waste Name: Crude Contaminated Soil (Pipe Rack Boring)

Thank you for selecting SKB SHAMROCK LANDFILL for your waste management requirements. Your waste stream has been reviewed and is acceptable for management at our facility based on the information provided in the profile sheet number listed above and conditions below. Our facility has the necessary permits to allow the storage, treatment, or disposal of this waste. The above referenced acceptance number should be listed on all shipping documents and correspondence. Please retain these documents for your records and future reference.

To schedule a shipment, or should you have any questions, please contact the facility at (218) 878-0112.

**ACCEPTANCE INFORMATION**

The waste stream identified by the reference above is acceptable for disposal.
The anticipated frequency of shipment is 10 YARDS / ONE TIME ONLY

This waste is acceptable for delivery beginning on 8/27/2012 thru 8/9/2014 at which time the material will need to be reanalyzed and recertified.

**PCB Statement:** The Minnesota Pollution Control Agency encourages generators of non-hazardous PCB waste to voluntarily manage the waste as hazardous waste or to seek an alternative to land disposal such as incineration

**Spill Reporting Reminder:** Proper County and MPCA spill reporting procedures must be followed.

**Empty Container Statement:** Each shipment containing empty containers must be accompanied with a completed 'EMPTY CONTAINER CERTIFICATION FORM'.

**Free Liquid Statement:** Free liquids will not be placed in cells at SKB Shamrock Landfill. Free liquids must be solidified either prior to shipment to SKB Shamrock Landfill or at SKB Shamrock Landfill.

**Shipping Requirements** A NON-HAZARDOUS certificate is required to be on file, certifying the waste is non-hazardous as specified per 40 CFR 261.4. The shipment must be accompanied with an SKB Shamrock Landfill manifest.

## WASTE STREAM ANALYSIS INFORMATION

| | |
|---|---|
| Waste Name: | Crude Contaminated Soil (Pipe Rack Boring) |
| Physical State: | Solid |
| Process Producing Waste: | pipeline terminal activities |

## PRE-ACCEPTANCE SAMPLE RESULTS

| | | | |
|---|---|---|---|
| Color: | | Physical State: | |
| Dust Present: | 0 | Free Liquids: | 0 |
| Paint Filter Test: | 0 | Odor: | |
| Flash Point Range: | | Density: | |
| Radioactive?: | 0 | Water Reactivity: | 0 |
| pH Range: | | React to Acid: | 0 |
| React to Base: | 0 | % Moisture: | |
| OVM Sniff: | | Sulfide: | |
| Oxidizers: | 0 | Cyanide: | |
| Reacts with Air: | 0 | | |

This analysis is solely for use by SKB Shamrock Landfill employees for the purpose of determining waste acceptability. No other claims are made or implied.

## COMMENTS

## AUTHORIZATION

Approval: _____ Date: 8/28/12

SKB CLOQUET

REPORT NAME: **Tons Each Load By WSID**
DESCRIPTION: **Tonnage for EACH LOAD, grouped by customer**
DATE RANGE: **01/01/2012 to 12/05/2012**
PRINTED ON (DATE): **Wednesday, December 05, 2012**

## ENBS1

Enbridge Pipelines Limited Partnership,
2800 East 21st St
Superior WI 54880

| LOAD # | MANIFEST | ARRIVED | WASTE STREAM | WASTE NAME | CELL | SPOT. | LIFT | TONS |
|---|---|---|---|---|---|---|---|---|
| 5762 (A) | 10873 | 11/12/2012 | CL12-0067 | Crude Contaminated Soil (Pipe Rac | 1A | T34 | 1170 | 12.24 |
| 5765 (A) | 10874 | 11/12/2012 | CL12-0067 | Crude Contaminated Soil (Pipe Rac | 1A | T34 | 1170 | 14.56 |
| 5766 (A) | 10875 | 11/12/2012 | CL12-0067 | Crude Contaminated Soil (Pipe Rac | 1A | T34 | 1170 | 9.93 |
| 6189 (A) | 5298 | 12/4/2012 | CL12-0067 | Crude Contaminated Soil (Pipe Rac | 2A | Y41 | 1160 | 13.92 |
| 6190 (A) | 5297 | 12/4/2012 | CL12-0067 | Crude Contaminated Soil (Pipe Rac | 2A | Y41 | 1160 | 13.60 |
| 6194 (A) | 5299 | 12/4/2012 | CL12-0067 | Crude Contaminated Soil (Pipe Rac | 2A | Y41 | 1160 | 15.43 |
| 6198 (A) | 5296 | 12/4/2012 | CL12-0067 | Crude Contaminated Soil (Pipe Rac | 2A | Y41 | 1160 | 13.41 |
| | | | | **Total # of Loads: 7** | | | **Total Tons:** | **93.09** |

**Grand Total (Tons): 93.09**
**Grand Total (Loads): 7**

LEGEND
Technical Services, Inc.
www.legend-group.com

88 Empire Drive
St Paul, MN 55103
Tel: 651-642-1150
Fax: 651-642-1239

August 15, 2012

**REVISION**

Ms. Andrea Nord
Barr Engineering Co.
4700 W 77th St
Minneapolis, MN 55435

Work Order Number: 1203709
RE: 49161172

This is a revised report. The details of the revision are listed in the case narrative on the following page.

Enclosed are the results of analyses for samples received by the laboratory on 08/10/12. If you have any questions concerning this report, please feel free to contact me.

All samples will be retained by LEGEND, unless consumed in the analysis, for 30 days from the date of this report and then discarded unless other arrangements are made.

WI Certification #998022410

Prepared by,
LEGEND TECHNICAL SERVICES, INC

Bach Pham
Client Manager I
bpham@legend-group.com

Tyler Jones
Chemist I
tjones@legend-group.com

LEGEND Technical Services, Inc.
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Fax: 651-642-1239

| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161172 | |
| 4700 W 77th St | Project Number: | 49161172 Pipe Rock | Work Order #: 1203709 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/15/12 |

## ANALYTICAL REPORT FOR SAMPLES

| Sample ID | Laboratory ID | Matrix | Date Sampled | Date Received |
|---|---|---|---|---|
| Pipe Rock | 1203709-01 | Soil | 08/09/12 14:00 | 08/10/12 08:45 |
| Trip Blank | 1203709-02 | Methanol | 08/09/12 00:00 | 08/10/12 08:45 |

**Shipping Container Information**

**Default Cooler** Temperature (°C): 6.8

Received on ice: Yes
Temperature blank was present
Received on ice pack: No
Received on melt water: No
Ambient: No
Acceptable (IH/ISO only): No
Custody seals: No

**Case Narrative:**

This report was revised on August 15, 2012 to attach the DRO chromatogram for the sample. This report supercedes the report dated August 14, 2012.

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| | | | |
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| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/15/12 |

## DRO/8015B
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **Pipe Rock (1203709-01) Soil Sampled: 08/09/12 14:00 Received: 08/10/12 8:45** | | | | | | | | | | |
| **Diesel Range Organics** | **120** | 14 | 2.3 | mg/kg dry | 1 | B2H1312 | 08/13/12 | 08/13/12 | WI(95) DRO | L1 |
| *Surrogate: Triacontane (C-30)* | *86.5* | | | *70-130 %* | | " | " | " | " | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161172 | |
| 4700 W 77th St | Project Number: | 49161172 Pipe Rock | Work Order #: 1203709 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/15/12 |

## WI(95) GRO/8015B
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **Pipe Rock (1203709-01) Soil Sampled: 08/09/12 14:00 Received: 08/10/12 8:45** | | | | | | | | | | |
| Benzene | <0.036 | 0.036 | 0.0054 | mg/kg dry | 1 | B2H1306 | 08/13/12 | 08/13/12 | WI(95) GRO | |
| Ethylbenzene | <0.036 | 0.036 | 0.0067 | mg/kg dry | 1 | " | " | " | " | |
| Toluene | <0.036 | 0.036 | 0.0034 | mg/kg dry | 1 | " | " | " | " | |
| Xylenes (total) | <0.11 | 0.11 | 0.017 | mg/kg dry | 1 | " | " | " | " | |
| *Surrogate: 4-Fluorochlorobenzene* | *98.2* | | | *80-150 %* | | " | " | " | " | |
| **Trip Blank (1203709-02) Methanol Sampled: 08/09/12 00:00 Received: 08/10/12 8:45** | | | | | | | | | | |
| Benzene | <0.025 | 0.025 | 0.0038 | mg/kg wet | 1 | B2H1306 | 08/13/12 | 08/13/12 | WI(95) GRO | |
| Ethylbenzene | <0.025 | 0.025 | 0.0047 | mg/kg wet | 1 | " | " | " | " | |
| Toluene | <0.025 | 0.025 | 0.0024 | mg/kg wet | 1 | " | " | " | " | |
| Xylenes (total) | <0.075 | 0.075 | 0.012 | mg/kg wet | 1 | " | " | " | " | |
| *Surrogate: 4-Fluorochlorobenzene* | *94.3* | | | *80-150 %* | | " | " | " | " | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161172 | |
| 4700 W 77th St | Project Number: | 49161172 Pipe Rock | Work Order #: 1203709 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/15/12 |

## PERCENT SOLIDS
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **Pipe Rock (1203709-01) Soil Sampled: 08/09/12 14:00 Received: 08/10/12 8:45** | | | | | | | | | | |
| **% Solids** | **65** | | | % | 1 | B2H1408 | 08/14/12 | 08/14/12 | % calculation | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161172 | |
| 4700 W 77th St | Project Number: | 49161172 Pipe Rock | Work Order #: 1203709 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/15/12 |

## DRO/8015B - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B2H1312 - Sonication (Wisc DRO)** | | | | | | | | | | | |
| **Blank (B2H1312-BLK1)** | | | | | Prepared & Analyzed: 08/13/12 | | | | | | |
| Diesel Range Organics | < 8.0 | 8.0 | 1.3 | mg/kg wet | | | | | | | |
| *Surrogate: Triacontane (C-30)* | *11.6* | | | *mg/kg wet* | *16.0* | | *72.7* | *70-130* | | | |
| **LCS (B2H1312-BS1)** | | | | | Prepared & Analyzed: 08/13/12 | | | | | | |
| Diesel Range Organics | 50.7 | 8.0 | 1.3 | mg/kg wet | 64.0 | | 79.2 | 70-120 | | | |
| *Surrogate: Triacontane (C-30)* | *12.4* | | | *mg/kg wet* | *16.0* | | *77.8* | *70-130* | | | |
| **LCS Dup (B2H1312-BSD1)** | | | | | Prepared: 08/13/12 Analyzed: 08/14/12 | | | | | | |
| Diesel Range Organics | 51.5 | 8.0 | 1.3 | mg/kg wet | 64.0 | | 80.4 | 70-120 | 1.59 | 20 | |
| *Surrogate: Triacontane (C-30)* | *13.6* | | | *mg/kg wet* | *16.0* | | *85.3* | *70-130* | | | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161172 | |
| 4700 W 77th St | Project Number: | 49161172 Pipe Rock | Work Order #: 1203709 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/15/12 |

## WI(95) GRO/8015B - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B2H1306 - EPA 5035 Soil (Purge and Trap)** | | | | | | | | | | | |
| **Blank (B2H1306-BLK1)** | | | | | Prepared & Analyzed: 08/13/12 | | | | | | |
| Benzene | < 0.025 | 0.025 | 0.0038 | mg/kg wet | | | | | | | |
| Ethylbenzene | < 0.025 | 0.025 | 0.0047 | mg/kg wet | | | | | | | |
| Toluene | < 0.025 | 0.025 | 0.0024 | mg/kg wet | | | | | | | |
| Xylenes (total) | < 0.075 | 0.075 | 0.012 | mg/kg wet | | | | | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *23.0* | | | *ug/L* | *25.0* | | *91.9* | *80-150* | | | |
| **LCS (B2H1306-BS1)** | | | | | Prepared & Analyzed: 08/13/12 | | | | | | |
| Benzene | 92.6 | | | ug/L | 100 | | 92.6 | 80-120 | | | |
| Ethylbenzene | 95.9 | | | ug/L | 100 | | 95.9 | 80-120 | | | |
| Toluene | 93.1 | | | ug/L | 100 | | 93.1 | 80-120 | | | |
| Xylenes (total) | 288 | | | ug/L | 300 | | 95.9 | 80-120 | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *24.0* | | | *ug/L* | *25.0* | | *96.1* | *80-150* | | | |
| **LCS Dup (B2H1306-BSD1)** | | | | | Prepared & Analyzed: 08/13/12 | | | | | | |
| Benzene | 95.1 | | | ug/L | 100 | | 95.1 | 80-120 | 2.72 | 20 | |
| Ethylbenzene | 98.5 | | | ug/L | 100 | | 98.5 | 80-120 | 2.77 | 20 | |
| Toluene | 96.1 | | | ug/L | 100 | | 96.1 | 80-120 | 3.17 | 20 | |
| Xylenes (total) | 301 | | | ug/L | 300 | | 100 | 80-120 | 4.59 | 20 | |
| *Surrogate: 4-Fluorochlorobenzene* | *25.0* | | | *ug/L* | *25.0* | | *100* | *80-150* | | | |
| **Matrix Spike (B2H1306-MS1)** | | **Source: 1203710-01** | | | Prepared & Analyzed: 08/13/12 | | | | | | |
| Benzene | 94.1 | | | ug/L | 100 | < | 94.1 | 80-120 | | | |
| Ethylbenzene | 97.7 | | | ug/L | 100 | 0.134 | 97.5 | 80-120 | | | |
| Toluene | 95.5 | | | ug/L | 100 | 0.139 | 95.4 | 80-120 | | | |
| Xylenes (total) | 293 | | | ug/L | 300 | < | 97.7 | 80-120 | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *23.7* | | | *ug/L* | *25.0* | | *94.7* | *80-150* | | | |

LEGEND Technical Services, Inc.
www.legend-group.com

88 Empire Drive
St Paul, MN 55103
Tel: 651-642-1150
Fax: 651-642-1239

| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161172 | |
| 4700 W 77th St | Project Number: | 49161172 Pipe Rock | Work Order #: 1203709 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/15/12 |

## PERCENT SOLIDS - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B2H1408 - General Preparation** | | | | | | | | | | | |
| **Duplicate (B2H1408-DUP1)** | | **Source: 1203714-02** | | | Prepared & Analyzed: 08/14/12 | | | | | | |
| % Solids | 91.0 | | | % | | 91.0 | | | 0.00 | 20 | |
| **Duplicate (B2H1408-DUP2)** | | **Source: 1203714-04** | | | Prepared & Analyzed: 08/14/12 | | | | | | |
| % Solids | 93.0 | | | % | | 94.0 | | | 1.07 | 20 | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161172 | |
| 4700 W 77th St | Project Number: | 49161172 Pipe Rock | Work Order #: 1203709 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/15/12 |

## Notes and Definitions

| | |
|---|---|
| L1 | Results in the diesel organics range are primarily due to overlap from a heavy oil range product. |
| < | Less than value listed |
| dry | Sample results reported on a dry weight basis |
| NA | Not applicable. The %RPD is not calculated from values less than the reporting limit. |
| MDL | Method Detection Limit |
| RL | Reporting Limit |
| RPD | Relative Percent Difference |
| LCS | Laboratory Control Spike = Blank Spike (BS) = Laboratory Fortified Blank (LFB) |
| MS | Matrix Spike = Laboratory Fortified Matrix (LFM) |

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1203709

Enbridge

**BARR** **Chain of Custody**

4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number: 49/16-1172

Project Name: Pipe Rack Geotechnical

Sample Origination State WI (use two letter postal state abbreviation)

COC Number: № 35266

COC 1 of 1

Project Manager: REE

Project QC Contact: AAN

Sampled by: BSL2

Laboratory: Legend

Number of Containers/Preservative

Water: VOCs (HCl) #1; SVOCs (unpreserved) #2; Dissolved Metals ($HNO_3$); Total Metals ($HNO_3$); General (unpreserved)#3; Diesel Range Organics (HCl); Nutrients ($H_2SO_4$) #4

Soil: VOCs (tared MeOH) #1; GRO, BTEX (tared MeOH) #1; DRO (tared unpreserved); Metals (unpreserved); SVOCs (unpreserved) #2; % Solids (plastic vial, unpres.); BTEX

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Type: Grab | Type: Comp. | Type: QC | Total Number Of Containers | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. Pipe Rack | – | – | – | 8/9/12 | 1400 | | X | | X | | 3 | Rush Analysis |
| 2. Trip Blank 8/10/12 #1 | | | | | | | | | | | | BTEX, DRO |
| 3. | | | | | | | | | | | | |
| 4. | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | |
| 6. | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | |

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| | Y N | 8/9/12 | 1430 | | | |
| Relinquished By: | On Ice? Y N | Date | Time | Received by: | Date | Time |
| | | | | | 8/10/12 | 8:45 |

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

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:\RLG\STD-FORMS\Chain Of Custody Form 2009 RLG Rev. 08/01/09

Fedex 6.8°c

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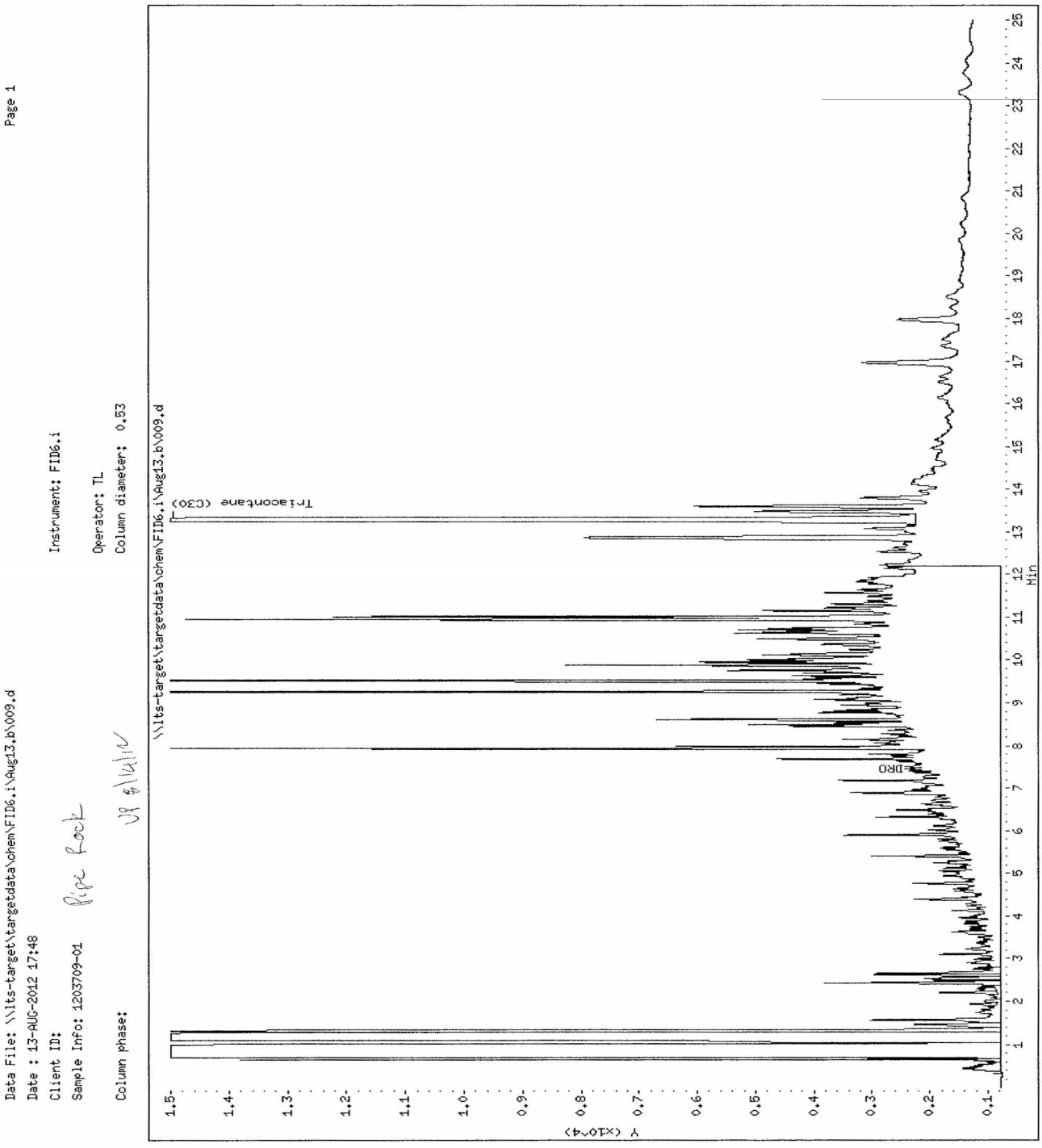

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

November 08, 2012

Andrea Nord
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

RE: Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on November 06, 2012. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

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

Sincerely,

Carol Davy for
Andrea Opland
andrea.opland@pacelabs.com
Project Manager

Enclosures

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## CERTIFICATIONS

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

**Minnesota Certification IDs**

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Hawaii Certification #Pace
Idaho Certification #: MN00064
Illinois Certification #: 200011
Kansas Certification #: E-10167
Louisiana Certification #: 03086
Louisiana Certification #: LA080009
Maine Certification #: 2007029
Maryland Certification #: 322
Michigan DEQ Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT CERT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
North Dakota Certification #: R-036A
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Utah Certification #: MN00064
Virginia/DCLS Certification #: 002521
Virginia/VELAP Certification #: 460163
Washington Certification #: C754
West Virginia Certification #: 382
Wisconsin Certification #: 999407970

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## SAMPLE SUMMARY

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|---|---|---|---|---|
| **10211407001** | **Office Water- Stockpile-1** | Solid | 11/05/12 13:30 | 11/06/12 09:00 |
| **10211407002** | **Line 5 Trap- Stockpile-1** | Solid | 11/05/12 09:30 | 11/06/12 09:00 |

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## SAMPLE ANALYTE COUNT

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|---|---|---|---|---|---|
| **10211407001** | **Office Water- Stockpile-1** | WI MOD DRO | MT | 2 | PASI-M |
| | | ASTM D2974 | LLC | 1 | PASI-M |
| | | EPA 8260 | CNC | 8 | PASI-M |
| **10211407002** | **Line 5 Trap- Stockpile-1** | WI MOD DRO | MT | 2 | PASI-M |
| | | ASTM D2974 | LLC | 1 | PASI-M |
| | | EPA 8260 | CNC | 8 | PASI-M |

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# PROJECT NARRATIVE

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

**Method:** **WI MOD DRO**
**Description:** WIDRO GCS
**Client:** Barr Engineering
**Date:** November 08, 2012

**General Information:**
2 samples were analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with WI MOD DRO with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Additional Comments:**

Analyte Comments:

QC Batch: OEXT/20186

T6: High boiling point hydrocarbons are present in the sample.

- Line 5 Trap- Stockpile-1 (Lab ID: 10211407002)
  - Diesel Range Organics
- Office Water- Stockpile-1 (Lab ID: 10211407001)
  - Diesel Range Organics

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# PROJECT NARRATIVE

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

**Method:** **EPA 8260**
**Description:** 8260 MSV UST
**Client:** Barr Engineering
**Date:** November 08, 2012

**General Information:**
2 samples were analyzed for EPA 8260. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with EPA 5035/5030B with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Duplicate Sample:**
All duplicate sample results were within method acceptance criteria with any exceptions noted below.

**Additional Comments:**

This data package has been reviewed for quality and completeness and is approved for release.

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## ANALYTICAL RESULTS

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

**Sample: Office Water- Stockpile-1** **Lab ID: 10211407001** Collected: 11/05/12 13:30 Received: 11/06/12 09:00 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIDRO GCS** | Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | | |
| Diesel Range Organics | **17.9** | mg/kg | 13.3 | 1.5 | 1 | 11/06/12 13:37 | 11/08/12 12:29 | | T6 |
| ***Surrogates*** | | | | | | | | | |
| n-Triacontane (S) | 93 | % | 50-150 | | 1 | 11/06/12 13:37 | 11/08/12 12:29 | | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **27.7** | % | 0.10 | 0.10 | 1 | | 11/07/12 00:00 | | |
| **8260 MSV UST** | Analytical Method: EPA 8260 Preparation Method: EPA 5035/5030B | | | | | | | | |
| Benzene | **<26.4** | ug/kg | 26.4 | 6.2 | 1 | 11/06/12 13:49 | 11/07/12 11:33 | 71-43-2 | |
| Ethylbenzene | **<66.0** | ug/kg | 66.0 | 5.5 | 1 | 11/06/12 13:49 | 11/07/12 11:33 | 100-41-4 | |
| Toluene | **<66.0** | ug/kg | 66.0 | 10 | 1 | 11/06/12 13:49 | 11/07/12 11:33 | 108-88-3 | |
| Xylene (Total) | **<198** | ug/kg | 198 | 21.9 | 1 | 11/06/12 13:49 | 11/07/12 11:33 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| Dibromofluoromethane (S) | 97 | % | 55-127 | | 1 | 11/06/12 13:49 | 11/07/12 11:33 | 1868-53-7 | |
| 1,2-Dichloroethane-d4 (S) | 94 | % | 49-125 | | 1 | 11/06/12 13:49 | 11/07/12 11:33 | 17060-07-0 | |
| Toluene-d8 (S) | 100 | % | 56-131 | | 1 | 11/06/12 13:49 | 11/07/12 11:33 | 2037-26-5 | |
| 4-Bromofluorobenzene (S) | 104 | % | 53-128 | | 1 | 11/06/12 13:49 | 11/07/12 11:33 | 460-00-4 | |

**Sample: Line 5 Trap- Stockpile-1** **Lab ID: 10211407002** Collected: 11/05/12 09:30 Received: 11/06/12 09:00 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIDRO GCS** | Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | | |
| Diesel Range Organics | **179** | mg/kg | 72.6 | 8.0 | 5 | 11/06/12 13:37 | 11/08/12 11:53 | | T6 |
| ***Surrogates*** | | | | | | | | | |
| n-Triacontane (S) | 90 | % | 50-150 | | 5 | 11/06/12 13:37 | 11/08/12 11:53 | | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **38.0** | % | 0.10 | 0.10 | 1 | | 11/07/12 00:00 | | |
| **8260 MSV UST** | Analytical Method: EPA 8260 Preparation Method: EPA 5035/5030B | | | | | | | | |
| Benzene | **<31.2** | ug/kg | 31.2 | 7.3 | 1 | 11/06/12 13:49 | 11/07/12 11:49 | 71-43-2 | |
| Ethylbenzene | **<77.9** | ug/kg | 77.9 | 6.5 | 1 | 11/06/12 13:49 | 11/07/12 11:49 | 100-41-4 | |
| Toluene | **<77.9** | ug/kg | 77.9 | 11.8 | 1 | 11/06/12 13:49 | 11/07/12 11:49 | 108-88-3 | |
| Xylene (Total) | **<234** | ug/kg | 234 | 25.9 | 1 | 11/06/12 13:49 | 11/07/12 11:49 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| Dibromofluoromethane (S) | 98 | % | 55-127 | | 1 | 11/06/12 13:49 | 11/07/12 11:49 | 1868-53-7 | |
| 1,2-Dichloroethane-d4 (S) | 97 | % | 49-125 | | 1 | 11/06/12 13:49 | 11/07/12 11:49 | 17060-07-0 | |
| Toluene-d8 (S) | 101 | % | 56-131 | | 1 | 11/06/12 13:49 | 11/07/12 11:49 | 2037-26-5 | |
| 4-Bromofluorobenzene (S) | 105 | % | 53-128 | | 1 | 11/06/12 13:49 | 11/07/12 11:49 | 460-00-4 | |

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## QUALITY CONTROL DATA

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

QC Batch: MPRP/36281
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10211407001, 10211407002

SAMPLE DUPLICATE: 1327715

| Parameter | Units | 10211304001 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 83.1 | 82.9 | .3 | 30 | |

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## QUALITY CONTROL DATA

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

QC Batch: MSV/22006 Analysis Method: EPA 8260
QC Batch Method: EPA 5035/5030B Analysis Description: 8260 MSV UST
Associated Lab Samples: 10211407001, 10211407002

METHOD BLANK: 1327485 Matrix: Solid
Associated Lab Samples: 10211407001, 10211407002

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Benzene | ug/kg | <20.0 | 20.0 | 11/07/12 10:59 | |
| Ethylbenzene | ug/kg | <50.0 | 50.0 | 11/07/12 10:59 | |
| Toluene | ug/kg | <50.0 | 50.0 | 11/07/12 10:59 | |
| Xylene (Total) | ug/kg | <150 | 150 | 11/07/12 10:59 | |
| 1,2-Dichloroethane-d4 (S) | % | 97 | 49-125 | 11/07/12 10:59 | |
| 4-Bromofluorobenzene (S) | % | 103 | 53-128 | 11/07/12 10:59 | |
| Dibromofluoromethane (S) | % | 99 | 55-127 | 11/07/12 10:59 | |
| Toluene-d8 (S) | % | 100 | 56-131 | 11/07/12 10:59 | |

LABORATORY CONTROL SAMPLE: 1327486

| Parameter | Units | Spike Conc. | LCS Result | LCS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|
| Benzene | ug/kg | 1000 | 773 | 77 | 74-126 | |
| Ethylbenzene | ug/kg | 1000 | 795 | 80 | 74-127 | |
| Toluene | ug/kg | 1000 | 796 | 80 | 75-125 | |
| Xylene (Total) | ug/kg | 3000 | 2440 | 81 | 75-126 | |
| 1,2-Dichloroethane-d4 (S) | % | | | 96 | 49-125 | |
| 4-Bromofluorobenzene (S) | % | | | 100 | 53-128 | |
| Dibromofluoromethane (S) | % | | | 96 | 55-127 | |
| Toluene-d8 (S) | % | | | 101 | 56-131 | |

MATRIX SPIKE SAMPLE: 1327487

| Parameter | Units | 10211407001 Result | Spike Conc. | MS Result | MS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|---|
| Benzene | ug/kg | <26.4 | 1380 | 1210 | 88 | 62-144 | |
| Ethylbenzene | ug/kg | <66.0 | 1380 | 1260 | 92 | 65-146 | |
| Toluene | ug/kg | <66.0 | 1380 | 1270 | 92 | 59-145 | |
| Xylene (Total) | ug/kg | <198 | 4130 | 3870 | 94 | 65-146 | |
| 1,2-Dichloroethane-d4 (S) | % | | | | 93 | 49-125 | |
| 4-Bromofluorobenzene (S) | % | | | | 102 | 53-128 | |
| Dibromofluoromethane (S) | % | | | | 98 | 55-127 | |
| Toluene-d8 (S) | % | | | | 102 | 56-131 | |

SAMPLE DUPLICATE: 1327488

| Parameter | Units | 10211407002 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Benzene | ug/kg | <31.2 | <32.1 | | 30 | |
| Ethylbenzene | ug/kg | <77.9 | <80.2 | | 30 | |



**REPORT OF LABORATORY ANALYSIS**

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
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(612)607-1700

## QUALITY CONTROL DATA

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

SAMPLE DUPLICATE: 1327488

| Parameter | Units | 10211407002 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Toluene | ug/kg | <77.9 | <80.2 | | 30 | |
| Xylene (Total) | ug/kg | <234 | <241 | | 30 | |
| 1,2-Dichloroethane-d4 (S) | % | 97 | 96 | .9 | | |
| 4-Bromofluorobenzene (S) | % | 105 | 104 | 3 | | |
| Dibromofluoromethane (S) | % | 98 | 98 | 3 | | |
| Toluene-d8 (S) | % | 101 | 100 | 2 | | |

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## QUALITY CONTROL DATA

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

QC Batch: OEXT/20186 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10211407001, 10211407002

METHOD BLANK: 1327464 Matrix: Solid
Associated Lab Samples: 10211407001, 10211407002

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | <10.0 | 10.0 | 11/08/12 09:28 | |
| n-Triacontane (S) | % | 80 | 50-150 | 11/08/12 09:28 | |

LABORATORY CONTROL SAMPLE & LCSD: 1327465 1327466

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | 80 | 63.7 | 64.9 | 80 | 81 | 70-120 | 2 | 20 | |
| n-Triacontane (S) | % | | | | 81 | 81 | 50-150 | | | |

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# QUALIFIERS

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

## DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

## LABORATORIES

| | |
|---|---|
| PASI-M | Pace Analytical Services - Minneapolis |

## ANALYTE QUALIFIERS

| | |
|---|---|
| T6 | High boiling point hydrocarbons are present in the sample. |

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: Enbridge Terminal Hydrant Exca
Pace Project No.: 10211407

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|---|---|---|---|---|---|
| **10211407001** | **Office Water- Stockpile-1** | WI MOD DRO | OEXT/20186 | WI MOD DRO | GCSV/10406 |
| **10211407002** | **Line 5 Trap- Stockpile-1** | WI MOD DRO | OEXT/20186 | WI MOD DRO | GCSV/10406 |
| **10211407001** | **Office Water- Stockpile-1** | ASTM D2974 | MPRP/36281 | | |
| **10211407002** | **Line 5 Trap- Stockpile-1** | ASTM D2974 | MPRP/36281 | | |
| **10211407001** | **Office Water- Stockpile-1** | EPA 5035/5030B | MSV/22006 | EPA 8260 | MSV/22007 |
| **10211407002** | **Line 5 Trap- Stockpile-1** | EPA 5035/5030B | MSV/22006 | EPA 8260 | MSV/22007 |

Report Date: 08-Nov-2012 12:46

Pace Analytical Services

WI Dept of Nat. Resources- WIDRO

Data file : \\192.168.10.12\chem\10gcs5.i\110812dro.b\313F0038.D
Lab Smp Id: 10211407001
Inj Date  : 08-NOV-2012 12:29
Operator  : MT  Inst ID: 10gcs5.i
Smp Info  : 10211407001
Misc Info : 10406
Comment   : C10-C28 DRO
Method    : \\192.168.10.12\chem\10gcs5.i\110812dro.b\WDRO5-102312.m
Meth Date : 08-Nov-2012 12:21 mthao  Quant Type: ESTD
Cal Date  : 23-OCT-2012 11:08  Cal File: 297F0018.D
Als bottle: 31
Dil Factor: 1.00000
Integrator: HP Genie  Compound Sublist: all.sub
Target Version:  4.14
Processing Host: 10VOA3

Concentration Formula: Amt * DF * Uf * Vt/(Ws * Vi*(100-M)/100) * CpndVariable

| Name | Value | Description |
|---|---|---|

| | | |
|---|---|---|
| DF | 1.000 | Dilution Factor |
| Uf | 1.000 | Correction factor |
| Vt | 1.000 | Volume of final extract (mL) |
| Ws | 25.000 | Weight of sample extracted (g) |
| Vi | 1.000 | Volume injected (uL) |
| M | 0.00000 | % Moisture |

Cpnd Variable Local Compound Variable

| | | | | | | CONCENTRATIONS | |
|---|---|---|---|---|---|---|---|
| | | | | | | ON-COLUMN | FINAL |
| Compounds | RT | EXP RT | DLT RT | RESPONSE | | (ug/mL) | (mg/kg) |
| S 2 DRO | 1.380 | -2.590 | | 44501246 | | 335.642 | 13.4 |
| $ 5 n-Triacontane (S) | 2.663 | 2.680 | -0.017 | 11645738 | | 116.617 | 4.66(aM) |

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

M - Compound response manually integrated.

Data File: \\192.168.10.12\chem\10gcs5.i\110812dro.b/313F0038.D

Report Date: 11/08/2012

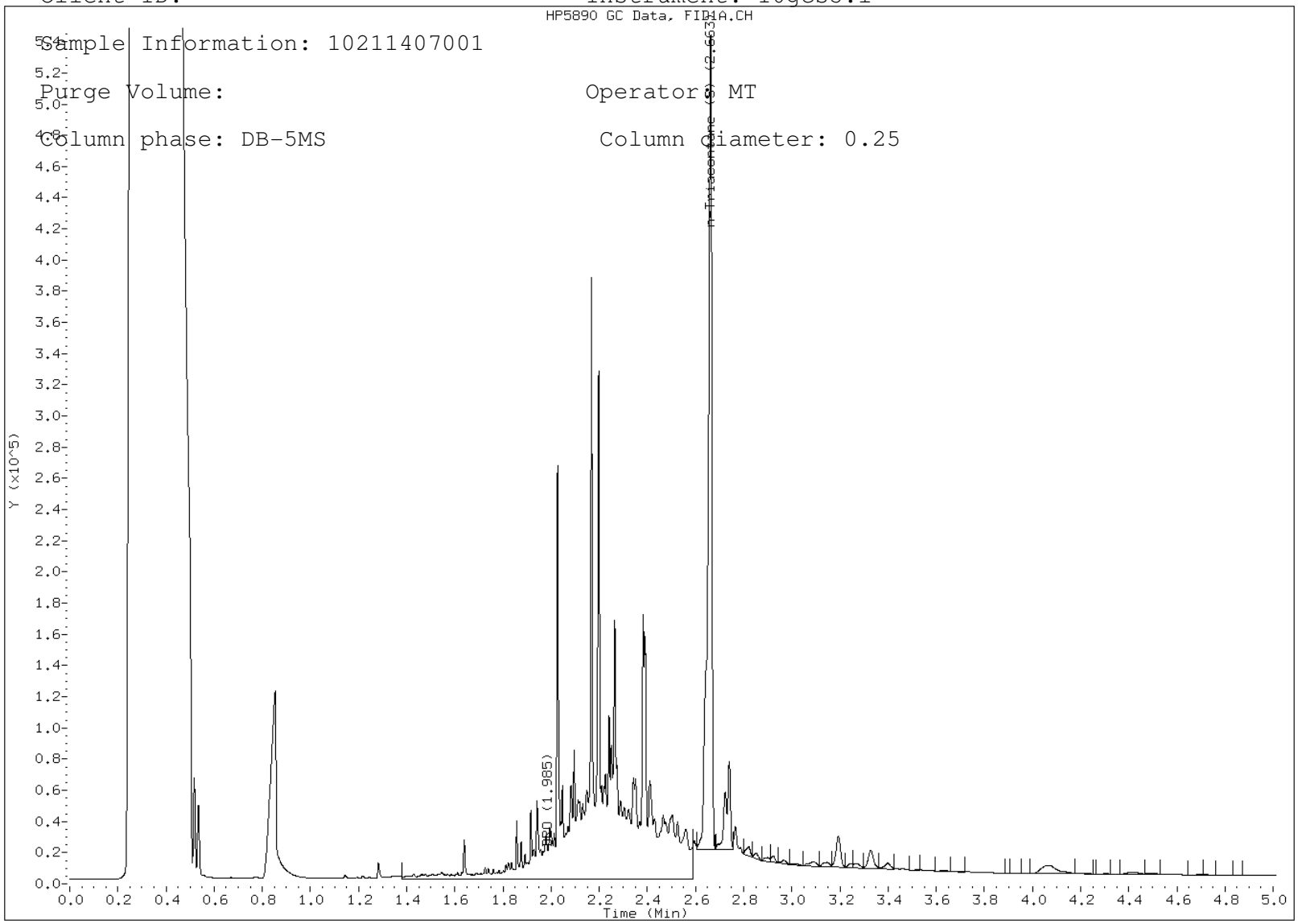
Sample ID: 10211407001

Client ID: Instrument: 10gcs5.i

Sample Information: 10211407001

Purge Volume: Operator: MT

Column phase: DB-5MS Column Diameter: 0.25



Report Date: 08-Nov-2012 12:08

Pace Analytical Services

WI Dept of Nat. Resources- WIDRO

Data file : \\192.168.10.12\chem\10gcs5.i\110812dro.b\313F0033.D
Lab Smp Id: 10211407002
Inj Date  : 08-NOV-2012 11:53
Operator  : MT  Inst ID: 10gcs5.i
Smp Info  : 10211407002,5
Misc Info : 10406
Comment   : C10-C28 DRO
Method    : \\192.168.10.12\chem\10gcs5.i\110812dro.b\WDRO5-102312.m
Meth Date : 08-Nov-2012 09:45 mthao  Quant Type: ESTD
Cal Date  : 23-OCT-2012 11:08  Cal File: 297F0018.D
Als bottle: 30
Dil Factor: 5.00000
Integrator: HP Genie  Compound Sublist: all.sub
Target Version:  4.14
Processing Host: 10VOA3

Concentration Formula: Amt * DF * Uf * Vt/(Ws * Vi*(100-M)/100) * CpndVariable

| Name | Value | Description |
|---|---|---|

| | | |
|---|---|---|
| DF | 5.000 | Dilution Factor |
| Uf | 1.000 | Correction factor |
| Vt | 1.000 | Volume of final extract (mL) |
| Ws | 25.000 | Weight of sample extracted (g) |
| Vi | 1.000 | Volume injected (uL) |
| M | 0.00000 | % Moisture |

Cpnd Variable Local Compound Variable

| | | | | | | CONCENTRATIONS | |
|---|---|---|---|---|---|---|---|
| | | | | | | ON-COLUMN | FINAL |
| Compounds | RT | EXP RT | DLT RT | RESPONSE | | (ug/mL) | (mg/kg) |
| S 2 DRO | 1.380 | -2.590 | | 81899597 | | 615.835 | 123 |
| $ 5 n-Triacontane (S) | 2.651 | 2.680 | -0.029 | 2273278 | | 22.5057 | 4.50(aM) |

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

M - Compound response manually integrated.

Data File: \\192.168.10.12\chem\10gcs5.i\110812dro.b/313F0033.D

Report Date: 11/08/2012

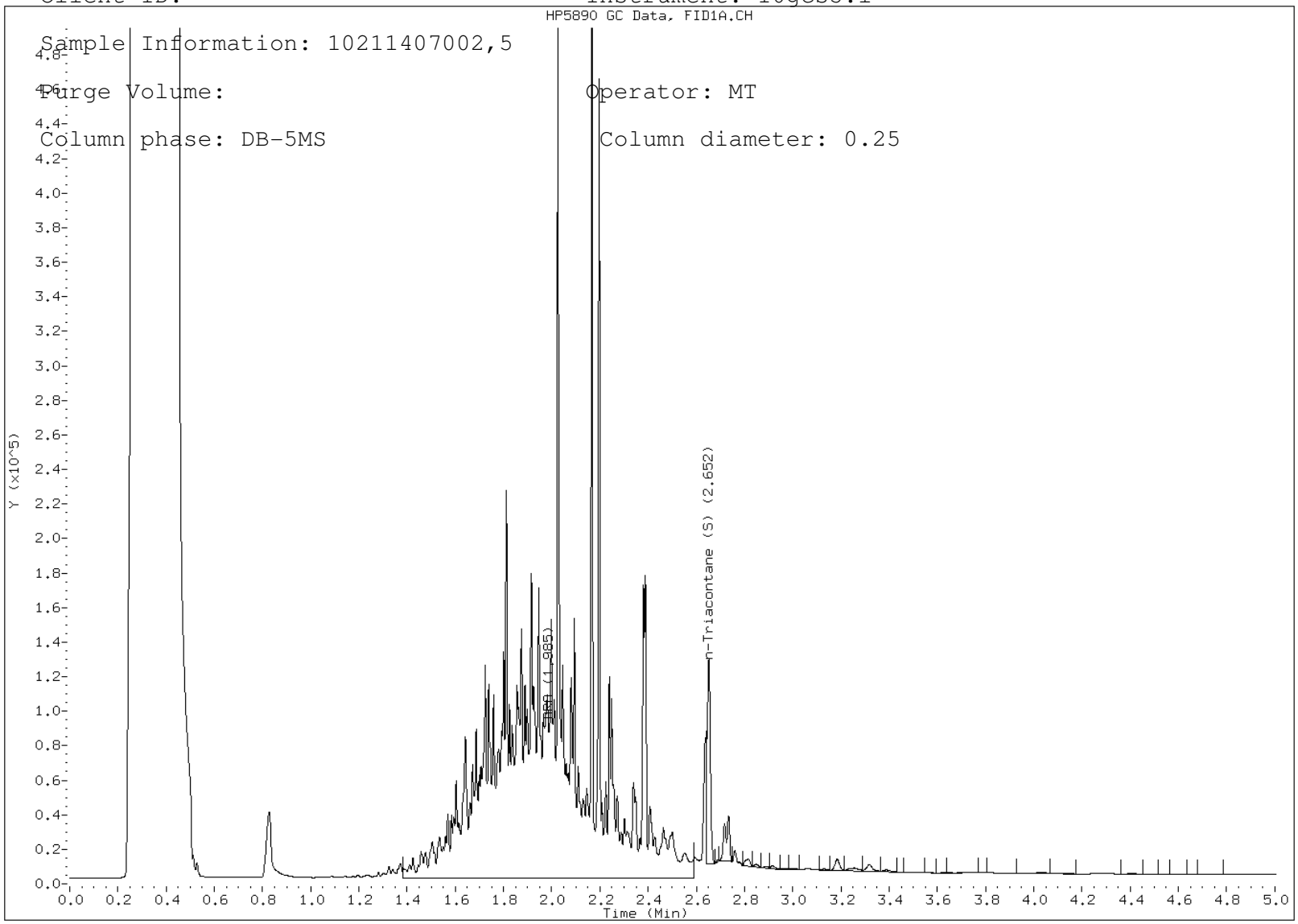
Sample ID: 10211407002

Client ID: Instrument: 10gcs5.i

Sample Information: 10211407002,5

Purge Volume: Operator: MT

Column phase: DB-5MS Column diameter: 0.25



11-6-2012 1132

RUSH

10211407

# Chain of Custody

BARR

4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

COC 1 of 1

Project Number: 49161092 RESP 017

Project Name: Enbridge Terminal Hydrant Excavations

Sample Origination State WI (use two letter postal state abbreviation)

COC Number: № 35329

Project Manager: Ryan Erickson REE@barr.com

Project QC Contact: Andrea Nord ADN@barr.com

Sampled by: REE

Laboratory: ~~Test~~ Pace

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Type: Grab | Type: Comp. | Type: QC | Number of Containers/Preservative — Water: VOCs (HCl) #1 | SVOCs (unpreserved) #2 | Dissolved Metals ($HNO_3$) | Total Metals ($HNO_3$) | General (unpreserved) #3 | Diesel Range Organics (HCl) | Nutrients ($H_2SO_4$) #4 | Soil: VOCs (tared MeOH) #1 | GRO, BTEX (tared MeOH) #1 | DRO (tared unpreserved) | Metals (unpreserved) | SVOCs (unpreserved) #2 | % Solids (plastic vial, unpres.) | Total Number Of Containers | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. Office Water - Stockpile-1 | — | | | 11/5/12 | 1330 | | X | X | | | | | | | | | | | X | X | | | X | 4 | BTEX, DRO, moisture |
| 2. Line 5 Trap - stockpile - 1 | — | | | 11/5/12 | 930 | | X | X | | | | | | | | | | | X | X | | | X | 4 | ↓ |
| 3. | | | | | | | | | | | | | | | | | | | | | | | | | |
| 4. | | | | | | | | | | | | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | | | | | | | | | | | | ASAP TAT |
| 6. | | | | | | | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | | | | | | | | | | | | |

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| | Y N | 11/5/12 | 1430 | CSI Express | 11-6-12 | 900 |
| Relinquished By: | On Ice? Y N | Date | Time | Received by: | Date | Time |

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☒ Sampler ☐ Other: _____

Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:RLG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

20 of 21

| Pace Analytical | Document Name: Sample Condition Upon Receipt Form | Document Revised: 22Aug2012 Page 1 of 1 |
|---|---|---|
| | Document No.: F-MN-L-213-rev.04 | Issuing Authority: Pace Minnesota Quality Office |

**Sample Condition Upon Receipt**

**Client Name:** Barr

**Project #:** WO# : 10211407

10211407

**Courier:** ☒ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other:

**Tracking Number:** 7940 0376 6011 9

**Custody Seal on Cooler/Box Present?** ☐ Yes ☒ No **Seals Intact?** ☐ Yes ☒ No **Optional:** Proj. Due Date: Proj. Name:

**Packing Material:** ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☒ Yes ☐ No

**Thermometer Used:** ☒ 888A912167504 ☐ 80512447 **Type of Ice:** ☒ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has beg

**Cooler Temperature:** 2.3 **Biological Tissue Frozen?** ☐ Yes ☐ No **Date and Initials of Person Examining Contents:** SL 11.6.12

Temp should be above freezing to 6°C

| | | | | Comments: |
|---|---|---|---|---|
| Chain of Custody Present? | ☒ Yes | ☐ No | ☐ N/A | 1. |
| Chain of Custody Filled Out? | ☒ Yes | ☐ No | ☐ N/A | 2. |
| Chain of Custody Relinquished? | ☒ Yes | ☐ No | ☐ N/A | 3. |
| Sampler Name and/or Signature on COC? | ☒ Yes | ☐ No | ☐ N/A | 4. |
| Samples Arrived within Hold Time? | ☐ Yes | ☒ No | ☐ N/A | 5. |
| Short Hold Time Analysis (<72 hr)? | ☐ Yes | ☒ No | ☐ N/A | 6. |
| Rush Turn Around Time Requested? | ☒ Yes | ☐ No | ☐ N/A | 7. |
| Sufficient Volume? | ☒ Yes | ☐ No | ☐ N/A | 8. |
| Correct Containers Used? | ☒ Yes | ☐ No | ☐ N/A | 9. |
| -Pace Containers Used? | ☒ Yes | ☐ No | ☐ N/A | |
| Containers Intact? | ☒ Yes | ☐ No | ☐ N/A | 10. |
| Filtered Volume Received for Dissolved Tests? | ☐ Yes | ☒ No | ☐ N/A | 11. |
| Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL | ☒ Yes | ☐ No | ☐ N/A | 12. |
| All containers needing acid/base preservation have been checked? Noncompliances are noted in 13. | ☐ Yes | ☒ No | ☐ N/A | 13. ☐ $HNO_3$ ☐ $H_2SO_4$ ☐ NaOH ☐ HCl |
| All containers needing preservation are found to be in compliance with EPA recommendation? ($HNO_3$, $H_2SO_4$, HCl<2; NaOH>12) | ☐ Yes | ☒ No | ☐ N/A | Sample # |
| Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water) | ☐ Yes | ☒ No | | Initial when completed: Lot # of added preservative: |
| Headspace in VOA Vials ( >6mm)? | ☐ Yes | ☒ No | ☐ N/A | 14. |
| Trip Blank Present? | ☐ Yes | ☒ No | ☐ N/A | 15. |
| Trip Blank Custody Seals Present? | ☐ Yes | ☒ No | ☐ N/A | |
| Pace Trip Blank Lot # (if purchased): | | | | |

**CLIENT NOTIFICATION/RESOLUTION** **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

**Project Manager Review:** CKD **Date:** 11-6-12

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e out hold, incorrect preservative, out of temp, incorrect containers)

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

**Western Lake Superior Sanitary District**

November 15, 2012

Ryan Erickson
Barr Enginerring
332 W. Superior Street, Suite 600
Duluth, MN 55802

Re: WLSSD Discharge Approval (Enbridge Groundwater Clean-up)

Dear Mr. Erickson:

Based on the analytical information provided on **11/15/2012**, the WLSSD approves the discharge of **approximately 6000 gallons of contaminated water from an Enbridge pipeline ground water clean-up site** provided there is no visual sign of the petroleum oil, grease or other petroleum related products. This contaminated water is to be disposed of at the WLSSD's main treatment facility, which is located at 2626 Courtland in Duluth.

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

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

If there are any questions, please contact me at (218) 740-4815.

Sincerely,

Tim Tuominen
Chemist

LEGEND
Technical Services, Inc.
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88 Empire Drive
St Paul, MN 55103
Tel: 651-642-1150
Fax: 651-642-1239

November 12, 2012

Ms. Andrea Nord
Barr Engineering Co.
4700 W 77th St
Minneapolis, MN 55435

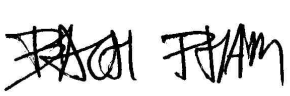
Work Order Number: 1205308
RE: 49161092

Enclosed are the results of analyses for samples received by the laboratory on 11/08/12. If you have any questions concerning this report, please feel free to contact me.

All samples will be retained by LEGEND, unless consumed in the analysis, for 30 days from the date of this report and then discarded unless other arrangements are made.

WI Certification #998022410

Prepared by,
LEGEND TECHNICAL SERVICES, INC



Bach Pham
Client Manager II
bpham@legend-group.com

Tyler Jones
Chemist I
tjones@legend-group.com

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Fax: 651-642-1239

| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092.01 RESP 017 | Work Order #: 1205308 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 11/12/12 |

## ANALYTICAL REPORT FOR SAMPLES

| Sample ID | Laboratory ID | Matrix | Date Sampled | Date Received |
|---|---|---|---|---|
| Hydrant Water-Waste-1 | 1205308-01 | Water | 11/07/12 10:00 | 11/08/12 10:05 |
| Trip Blank | 1205308-02 | Water | 11/07/12 00:00 | 11/08/12 10:05 |

**Shipping Container Information**

**Default Cooler** Temperature (°C):

Received on ice: Yes
Temperature blank was not present
Received on ice pack: No
Received on melt water: No
Ambient: No
Acceptable (IH/ISO only): No
Custody seals: No

**Case Narrative:**

Recovery of the DRO surrogate for the sample was not available due to sample dilution required from high analyte concentration.

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Fax: 651-642-1239

| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092.01 RESP 017 | Work Order #: 1205308 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 11/12/12 |

## DRO/8015B
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **Hydrant Water-Waste-1 (1205308-01) Water** | **Sampled: 11/07/12 10:00** | | | **Received: 11/08/12 10:05** | | | | | | |
| **Diesel Range Organics** | **15000** | 1100 | 220 | ug/L | 10 | B2K0803 | 11/08/12 | 11/09/12 | WI(95) DRO | PH2 |
| *Surrogate: Triacontane (C-30)* | | | | *70-130 %* | | " | " | " | " | *D-1* |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092.01 RESP 017 | Work Order #: 1205308 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 11/12/12 |

## WI(95) GRO/8015B
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **Hydrant Water-Waste-1 (1205308-01) Water Sampled: 11/07/12 10:00 Received: 11/08/12 10:05** | | | | | | | | | | |
| **Benzene** | **1.7** | 1.0 | 0.11 | ug/L | 1 | B2K0811 | 11/08/12 | 11/09/12 | WI(95) GRO | |
| **Ethylbenzene** | **3.2** | 1.0 | 0.095 | ug/L | 1 | " | " | " | " | |
| **Gasoline range organics** | **510** | 100 | 11 | ug/L | 1 | " | " | " | " | H |
| **Toluene** | **2.3** | 1.0 | 0.16 | ug/L | 1 | " | " | " | " | |
| **Xylenes (total)** | **5.8** | 3.0 | 0.19 | ug/L | 1 | " | " | " | " | |
| *Surrogate: 4-Fluorochlorobenzene* | *97.2* | | | *80-150 %* | | " | " | " | " | |
| **Trip Blank (1205308-02) Water Sampled: 11/07/12 00:00 Received: 11/08/12 10:05** | | | | | | | | | | |
| Benzene | <1.0 | 1.0 | 0.11 | ug/L | 1 | B2K0811 | 11/08/12 | 11/08/12 | WI(95) GRO | |
| Ethylbenzene | <1.0 | 1.0 | 0.095 | ug/L | 1 | " | " | " | " | |
| Gasoline range organics | <100 | 100 | 11 | ug/L | 1 | " | " | " | " | |
| Toluene | <1.0 | 1.0 | 0.16 | ug/L | 1 | " | " | " | " | |
| Xylenes (total) | <3.0 | 3.0 | 0.19 | ug/L | 1 | " | " | " | " | |
| *Surrogate: 4-Fluorochlorobenzene* | *97.7* | | | *80-150 %* | | " | " | " | " | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092.01 RESP 017 | Work Order #: 1205308 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 11/12/12 |

## DRO/8015B - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B2K0803 - EPA 3510C (Sep Funnel)** | | | | | | | | | | | |
| **Blank (B2K0803-BLK1)** | | | | | Prepared: 11/08/12 | | Analyzed: 11/09/12 | | | | |
| Diesel Range Organics | < 100 | 100 | 20 | ug/L | | | | | | | |
| *Surrogate: Triacontane (C-30)* | *364* | | | *ug/L* | *400* | | *90.9* | *70-130* | | | |
| **LCS (B2K0803-BS1)** | | | | | Prepared: 11/08/12 | | Analyzed: 11/09/12 | | | | |
| Diesel Range Organics | 1620 | 100 | 20 | ug/L | 1600 | | 101 | 75-115 | | | |
| *Surrogate: Triacontane (C-30)* | *383* | | | *ug/L* | *400* | | *95.6* | *70-130* | | | |
| **LCS Dup (B2K0803-BSD1)** | | | | | Prepared: 11/08/12 | | Analyzed: 11/09/12 | | | | |
| Diesel Range Organics | 1610 | 100 | 20 | ug/L | 1600 | | 100 | 75-115 | 0.643 | 20 | |
| *Surrogate: Triacontane (C-30)* | *379* | | | *ug/L* | *400* | | *94.8* | *70-130* | | | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092.01 RESP 017 | Work Order #: 1205308 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 11/12/12 |

## WI(95) GRO/8015B - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B2K0811 - EPA 5030 Water (Purge and Trap)** | | | | | | | | | | | |
| **Blank (B2K0811-BLK1)** | | | | | Prepared & Analyzed: 11/08/12 | | | | | | |
| Benzene | < 1.0 | 1.0 | 0.11 | ug/L | | | | | | | |
| Ethylbenzene | < 1.0 | 1.0 | 0.095 | ug/L | | | | | | | |
| Gasoline range organics | < 100 | 100 | 11 | ug/L | | | | | | | |
| Toluene | < 1.0 | 1.0 | 0.16 | ug/L | | | | | | | |
| Xylenes (total) | < 3.0 | 3.0 | 0.19 | ug/L | | | | | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *26.7* | | | *ug/L* | *25.0* | | *107* | *80-150* | | | |
| **LCS (B2K0811-BS1)** | | | | | Prepared & Analyzed: 11/08/12 | | | | | | |
| Benzene | 99.4 | 1.0 | 0.11 | ug/L | 100 | | 99.4 | 80-120 | | | |
| Ethylbenzene | 92.9 | 1.0 | 0.095 | ug/L | 100 | | 92.9 | 80-120 | | | |
| Gasoline range organics | 852 | 100 | 11 | ug/L | 1000 | | 85.2 | 80-120 | | | |
| Toluene | 93.1 | 1.0 | 0.16 | ug/L | 100 | | 93.1 | 80-120 | | | |
| Xylenes (total) | 287 | 3.0 | 0.19 | ug/L | 300 | | 95.7 | 80-120 | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *23.6* | | | *ug/L* | *25.0* | | *94.2* | *80-150* | | | |
| **LCS Dup (B2K0811-BSD1)** | | | | | Prepared: 11/08/12 Analyzed: 11/09/12 | | | | | | |
| Benzene | 107 | 1.0 | 0.11 | ug/L | 100 | | 107 | 80-120 | 7.08 | 20 | |
| Ethylbenzene | 105 | 1.0 | 0.095 | ug/L | 100 | | 105 | 80-120 | 12.7 | 20 | |
| Gasoline range organics | 918 | 100 | 11 | ug/L | 1000 | | 91.8 | 80-120 | 7.48 | 20 | |
| Toluene | 106 | 1.0 | 0.16 | ug/L | 100 | | 106 | 80-120 | 12.9 | 20 | |
| Xylenes (total) | 321 | 3.0 | 0.19 | ug/L | 300 | | 107 | 80-120 | 11.3 | 20 | |
| *Surrogate: 4-Fluorochlorobenzene* | *25.8* | | | *ug/L* | *25.0* | | *103* | *80-150* | | | |
| **Duplicate (B2K0811-DUP1)** | | **Source: 1205320-02** | | | Prepared & Analyzed: 11/08/12 | | | | | | |
| Gasoline range organics | < 100 | 100 | 11 | ug/L | | <100 | | | NA | 20 | |
| *Surrogate: 4-Fluorochlorobenzene* | *25.6* | | | *ug/L* | *25.0* | | *102* | *80-150* | | | |
| **Matrix Spike (B2K0811-MS1)** | | **Source: 1205320-01** | | | Prepared: 11/08/12 Analyzed: 11/09/12 | | | | | | |
| Benzene | 107 | 1.0 | 0.11 | ug/L | 100 | <1.0 | 107 | 80-120 | | | |
| Ethylbenzene | 103 | 1.0 | 0.095 | ug/L | 100 | <1.0 | 103 | 80-120 | | | |
| Toluene | 103 | 1.0 | 0.16 | ug/L | 100 | <1.0 | 103 | 80-120 | | | |
| Xylenes (total) | 314 | 3.0 | 0.19 | ug/L | 300 | <3.0 | 105 | 80-120 | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *25.4* | | | *ug/L* | *25.0* | | *101* | *80-150* | | | |

LEGEND Technical Services, Inc.
www.legend-group.com

88 Empire Drive
St Paul, MN 55103
Tel: 651-642-1150
Fax: 651-642-1239

| | | | | |
|---|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | | |
| 4700 W 77th St | Project Number: | 49161092.01 RESP 017 | Work Order #: | 1205308 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: | 11/12/12 |

## Notes and Definitions

| | |
|---|---|
| PH2 | Insufficient preservative to reduce the sample pH to less than 2. |
| H | Results in the gasoline range contain hydrocarbons less volatile than GRO. |
| D-1 | The surrogate recovery for this sample is not available due to sample dilution required from high analyte concentration and/or matrix interferences. |
| < | Less than value listed |
| dry | Sample results reported on a dry weight basis |
| NA | Not applicable. The %RPD is not calculated from values less than the reporting limit. |
| MDL | Method Detection Limit |
| RL | Reporting Limit |
| RPD | Relative Percent Difference |
| LCS | Laboratory Control Spike = Blank Spike (BS) = Laboratory Fortified Blank (LFB) |
| MS | Matrix Spike = Laboratory Fortified Matrix (LFM) |

Shamrock Landfill

# Waste Profile Sheet

Shamrock Landfill

| P.O. Number | Customer Code | SKB Representative | CL |
|---|---|---|---|
| | | | |

## I. Generator Information

| | | | |
|---|---|---|---|
| Generator Name: Enbridge Pipelines Limited Partnership, LLC | | Generator EPA ID Number | SIC Code |
| Generator Location: Enbridge Superior Terminal - Pipe Rack Borings | County: Douglas | Generator Contact: Alex Smith | |
| | | Phone: 715-398-4795 | Fax: 832-325-5511 |
| Generator Mailing Address (if different: 1320 Grand Ave, Superior, WI 54880 | | Generator Email Address: alex.smith@enbridge.com | |
| Bill To Name & Address: Enbridge Energy, 1100 Louisiana Ave, STE. 3300, Houston, TX 77002 | Bill To #: | Billing Contact: Alex Smith | |
| | | Phone: 715-398-4795 | Fax: 832-325-5511 |
| Invoice Contact: | | Billing Email Address: alex.smith@enbridge.com | |

## II. Waste Generation Information

| | | |
|---|---|---|
| Waste Name: Crude oil contaminated soil - Pipe Rack Borings | Estimated rate of waste generation: 50 ☐ Lbs. ☐ tons ☒ cy ☐ drums | ☒ one time ☐ yearly |
| Generator Facility Operations and/or Site History: Enbridge Pipeline Terminal | | |
| Describe the generating process or source of contaminated soil/debris and/or waste: Pipeline Terminal Activities | | |

## III. Waste Composition and Constituents (list all known)

| | Actual Range % | ppm |
|---|---|---|
| Crude contaminated soil | 100 | |
| | | |
| | | |
| | | |

## IV. Waste Properties

| Physical state: | Free Liquids: | pH Range: | Flash point: | Color: | Odor (describe): |
|---|---|---|---|---|---|
| ☒ Solid ☐ Liquid ☐ Sludge ☐ Gas | ☐ Yes ☒ No Content _____% | ☐ <2 ☐ 2-4 ☐ 5-8 ☐ 8-12.4 ☐ >12.5 | ☐ ≤ 140°F ☐ > 140°F to < 200°F ☐ > 200°F | Brown | petroleum odor |

## V. Waste Classification

| Waste stream properties (answer ALL questions) | | | Does this waste contain absorbents? | ☐ Yes | ☒ No |
|---|---|---|---|---|---|
| Does this waste stream contain any D, F, K, U or P listed as hazardous waste, either in pure form, as a mixture, or treatment residue? | ☐ Yes | ☒ No | Is this waste lethal (by Minn. Rules 7045.0131 Subp. 6)? | ☐ Yes | ☒ No |
| Does this waste stream contain PCB material | ☐ Yes | ☒ No | Is this waste recyclable? | ☐ Yes | ☒ No |
| If yes, concentration: _____ppm | | | Is this waste explosive? | ☐ Yes | ☒ No |
| Does this waste stream contain fuming acids? | ☐ Yes | ☒ No | Is this waste infectious? | ☐ Yes | ☒ No |
| Does this waste contain asbestos? | ☐ Yes | ☒ No | Is this putrescible waste? | ☐ Yes | ☒ No |
| Does this waste contain oxidizers? | ☐ Yes | ☒ No | Is this waste demolition debris? | ☐ Yes | ☒ No |
| Does this waste contain radioactive material? | ☐ Yes | ☒ No | Is this waste sewer sludge? | ☐ Yes | ☒ No |

**Please attach any available information or analytical test results that have previously been performed on this waste that substantiates these determinations. Include MSDS's and any information from other agencies (i.e., MPCA, USEPA)**

## VI. Shipping Information

| Proper DOT Shipping Name (per CFR 172.101) where applicable | | | |
|---|---|---|---|
| Reportable Quantity | DOT Hazard Class | UN/NA Number | Packing Group |
| Method of packaging: ☐ drums (size _____) ☒ Bulk Solids ☐ boxes (size _____) | | Method of shipment ☐ Roll-off ☒ End dump ☐ Rail ☐ Other (Specify) _____ | |

## VII. Certification of Non Hazardous Waste & Approval Conditions

I hereby certify and warrant, on behalf of the generator and myself that, to the best of my knowledge and belief, the information contained herein is accurate, and true and that the waste is nonhazardous as defined in Title 42, Unites States Code Section 6903, Minnesota Statute Section 116.06, Subdivision 13, and/or any rules adopted by the Minnesota Pollution Control Agency under Minnesota Statute Section 116.07.

I understand that any approval is no longer valid if there are any changes in the process generating the waste or there have been changes in the composition of the waste. Therefore, if the composition of the waste stream changes or potentially changes, I or someone representing the generator, will immediately notify SKB Environmental. I, on behalf of the generator, hereby agree to fully indemnify SKB Environmental for any damages and/or costs incurred as a result of this certification being inaccurate or untrue.

| Signature | Printed Name | Title | Date |
|---|---|---|---|
| | Alex Smith | Environmental Analyst | 6-13-2013 |

Shamrock Landfill

June 14, 2013

Alex Smith
Enbridge Pipelines Limited Partnership, LLC
Accounts Payable
1100 Louisiana Ave, Ste 3300
Houston, TX 77002

RE: CL13-0027 Crude Contaminated Soil - Pipe Rack Borings

Dear Smith,

This agreement will confirm the price and length of service for disposal and /or transportation of your non-hazardous industrial material at our facility. This agreement is for the term of the Waste Approval granted by Shamrock Landfill and is for all services ordered and performance initiated within such period and does include the disposal surcharge fees which you are obligated to pay as of the date of this agreement. Shamrock Landfill may incur additional costs including but not limited to increases in state and local taxes. Shamrock Landfill may pass these costs on to the customer only after notification to the Customer. This agreement grants Shamrock Landfill the exclusive right to dispose of the referenced waste for the term of this agreement. This agreement shall automatically renew thereafter for an additional term of 24 months "Renewal Term" unless either party gives the other party written notification of termination at least 90 days prior to the termination of the then-existing term. Shamrock Landfill will notify the customer prior to the expiration of the agreement of any rate changes prior to the start of the Renewal Term.

Payment and terms are net thirty (30) days. Interest will be charged at a rate of 1 ½% per month (18% annually) on any unpaid balance 30 days after the date of the invoice. In the event Customer terminates this Agreement prior to its expiration other than as a result of a breach by Shamrock Landfill or Shamrock Landfill terminates this agreement for Customer's breach (including nonpayment) Customer agrees to pay to Shamrock Landfill as liquidated damages a sum calculated as follows: (1) if the remaining term under this agreement is six or more months Customer shall pay its average monthly charges multiplied by six: or (2) if the remaining term under this agreement is less than six months Customer shall pay its average monthly charge multiplied by the number of months remaining in the term. Customer expressly acknowledges that in the event of an unauthorized termination of this agreement the anticipated loss to Shamrock Landfill in such event is estimated to be the amount set forth in the foregoing liquidated damages provision and such estimated value is reasonable and is not imposed as a penalty.

These prices are based on an approved waste stream composition. In the event that a non-conforming waste is received, you will be notified of additional charges, when applicable.

To accept this agreement, please sign one copy and return it to our St. Paul, MN office at Shamrock Landfill, 251 Starkey St., St. Paul, MN 55107 or Via Fax at 651-223-8197 or email to sopstad@skbinc.com.

Shamrock Landfill

Steve Opstad

Customer ACCEPTED BY: (name, position) Alex Smith Environmental Analyst

DATE: 21 June 2013

WASTE APPROVAL Period: 6/24/2013 to 5/31/2015

P.O. Box 338 • Esko, MN 55733-0338
Main: 218.878.0112 • Fax: 218.879.2120

Shamrock Landfill

**Bill To Customer**

Enbridge Pipelines Limited Partnership, LLC
Accounts Payable
1100 Louisiana Ave, Ste 3300
Houston, TX 77002

**Service For Generator**

Enbridge Pipelines Limited Partnership, LLC
2800 East 21st St
Superior, WI 54880

## Disposal

Waste Description: Crude Contaminated Soil - Pipe Rack Borings

Estimated Volume: 50 YARDS / ONE TIME ONLY

Disposal Method: Secure Non-Hazardous Landfill

Treatment Method: None Expected For Conforming Waste

## Pricing

| | | | |
|---|---|---|---|
| Disposal | $16.00 | Per Ton | Crude Contaminated Soil - Pipe Rack Borings |

Shamrock Landfill

# Notification of Waste Acceptance



**CUSTOMER INFORMATION**

EPA ID#: WID981092133
Enbridge Pipelines Limited Partnership,
Enbridge Superior Terminal

2800 East 21st St
Superior, WI 54880
Contact: Alex Smith
Phone: (715) 398-4795

**INVOICE INFORMATION**

Bill #: 2133
Enbridge Pipelines Limited Partnership,
Accounts Payable

1100 Louisiana Ave, Ste 3300
Houston, TX 77002
Contact: Alex Smith
Phone: (715) 398-4795

Profile Sheet #:
Waste Stream #: CL13-0027
Waste Name: Crude Contaminated Soil - Pipe Rack Borings

Thank you for selecting SHAMROCK LANDFILL for your waste management requirements. Your waste stream has been reviewed and is acceptable for management at our facility based on the information provided in the profile sheet number listed above and conditions below. Our facility has the necessary permits to allow the storage, treatment, or disposal of this waste. The above referenced acceptance number should be listed on all shipping documents and correspondence. Please retain these documents for your records and future reference.

To schedule a shipment, or should you have any questions, please contact the facility at (218) 878-0112.

**ACCEPTANCE INFORMATION**

The waste stream identified by the reference above is acceptable for disposal.
The anticipated frequency of shipment is 50 YARDS / ONE TIME ONLY

This waste is acceptable for delivery beginning on 6/24/2013 thru 5/31/2015 at which time the material will need to be reanalyzed and recertified.

**PCB Statement:** The Minnesota Pollution Control Agency encourages generators of non-hazardous PCB waste to voluntarily manage the waste as hazardous waste or to seek an alternative to land disposal such as incineration

**Spill Reporting Reminder:** Proper County and MPCA spill reporting procedures must be followed.

**Empty Container Statement:** Each shipment containing empty containers must be accompanied with a completed 'EMPTY CONTAINER CERTIFICATION FORM'.

**Free Liquid Statement:** Free liquids will not be placed in cells at Shamrock Landfill. Free liquids must be solidified either prior to shipment to Shamrock Landfill or at Shamrock Landfill.

**Shipping Requirements** A NON-HAZARDOUS certificate is required to be on file, certifying the waste is non-hazardous as specified per 40 CFR 261.4. The shipment must be accompanied with an Shamrock Landfill manifest.

Shamrock Landfill



## WASTE STREAM ANALYSIS INFORMATION

| | |
|---|---|
| Waste Name: | Crude Contaminated Soil - Pipe Rack Borings |
| Physical State: | Solid |
| Process Producing Waste: | pipeline terminal activities |

## PRE-ACCEPTANCE SAMPLE RESULTS

| | | | |
|---|---|---|---|
| Color: | | Physical State: | |
| Dust Present: | 0 | Free Liquids: | 0 |
| Paint Filter Test: | 0 | Odor: | |
| Flash Point Range: | | Density: | |
| Radioactive?: | 0 | Water Reactivity: | 0 |
| pH Range: | | React to Acid: | 0 |
| React to Base: | 0 | % Moisture: | |
| OVM Sniff: | | Sulfide: | |
| Oxidizers: | 0 | Cyanide: | |
| Reacts with Air: | 0 | | |

This analysis is solely for use by Shamrock Landfill employees for the purpose of determining waste acceptability. No other claims are made or implied.

## COMMENTS

## AUTHORIZATION

Approval: _____ Date: 6/17/13

P.O. Box 338 • Esko, MN 55733-0338
Main: 218.878.0112 • Fax: 218.879.2120

SKB CLOQUET

REPORT NAME: **Tons Each Load By WSID**
DESCRIPTION: **Tonnage for EACH LOAD, grouped by customer**
DATE RANGE: **01/01/2013 to 01/16/2014**
PRINTED ON (DATE): **Thursday, January 16, 2014**

## ENBS1

Enbridge Pipelines Limited Partnership,
2800 East 21st St
Superior WI 54880

| LOAD # | MANIFEST | ARRIVED | WASTE STREAM | WASTE NAME | CELL | SPOT | LIFT | TONS |
|---|---|---|---|---|---|---|---|---|
| 10825 (A) | 10421 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 9.65 |
| 10826 (A) | 10420 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 11.04 |
| 10827 (A) | 10419 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 12.75 |
| 10830 (A) | 10418 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 15.50 |
| 10832 (A) | 10417 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 14.84 |
| 10833 (A) | 10416 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 14.69 |
| 10835 (A) | 10415 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 15.77 |
| 10838 (A) | 10414 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 14.98 |
| 10839 (A) | 10413 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 14.04 |
| 10840 (A) | 10412 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 15.40 |
| 10842 (A) | 10411 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 16.18 |
| 10846 (A) | 10410 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 15.84 |
| 10847 (A) | 10409 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 15.32 |
| 10848 (A) | 10408 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 16.83 |
| 10851 (A) | 10407 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.22 |
| 10853 (A) | 10406 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.22 |
| 10854 (A) | 10405 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.91 |
| 10855 (A) | 10404 | 6/26/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.49 |
| 10857 (A) | 10403 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 18.79 |
| 10859 (A) | 10402 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 18.40 |
| 10861 (A) | 10401 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.63 |
| 10866 (A) | 10400 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.61 |
| 10867 (A) | 10399 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.77 |
| 10868 (A) | 10398 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 19.76 |
| 10874 (A) | 10397 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 18.03 |
| 10876 (A) | 10396 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 21.46 |
| 10877 (A) | 10395 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.99 |
| 10879 (A) | 10394 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 0.00 |
| 10880 (A) | 10393 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 17.13 |
| 10882 (A) | 10392 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 15.87 |
| 10887 (A) | 10391 | 6/27/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | Q45 | 1160 | 13.22 |
| 11156 (A) | 10390 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 15.52 |
| 11158 (A) | 16418 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 13.47 |
| 11159 (A) | 16417 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 16.40 |
| 11161 (A) | 16416 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 15.37 |
| 11171 (A) | 16415 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 18.62 |
| 11172 (A) | 16414 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 15.53 |
| 11174 (A) | 16413 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 18.47 |
| 11175 (A) | 16412 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 18.53 |
| 11182 (A) | 16408 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 17.84 |
| 11183 (A) | 16409 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 19.12 |
| 11184 (A) | 16410 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 20.11 |
| 11187 (A) | 16411 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 15.90 |
| 11191 (A) | 16407 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 14.68 |
| 11194 (A) | 16406 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 15.72 |
| 11205 (A) | 16405 | 7/11/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | P45 | 1175 | 16.09 |
| 11885 (A) | 16403 | 8/5/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | T45 | 1175 | 12.82 |
| 11887 (A) | 16402 | 8/5/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | T45 | 1175 | 7.58 |
| 11891 (A) | 16401 | 8/5/2013 | CL13-0027 | Crude Contaminated Soil - Pipe Rac | 2A | T45 | 1175 | 8.48 |

| | | |
|---|---|---|
| Total # of Loads: 49 | Total Tons: | 766.58 |

| | |
|---|---|
| Grand Total (Tons): | 766.58 |
| Grand Total (Loads): | 49 |

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

June 24, 2013

Andrea Nord
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

RE: Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on May 30, 2013. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

This report was revised on June 24, 2013 to add BTEX by WIGRO.

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

Sincerely,

Andrea Opland

andrea.opland@pacelabs.com
Project Manager

Enclosures

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# CERTIFICATIONS

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

**Minnesota Certification IDs**

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Hawaii Certification #Pace
Idaho Certification #: MN00064
Illinois Certification #: 200011
Kansas Certification #: E-10167
Louisiana Certification #: 03086
Louisiana Certification #: LA080009
Maine Certification #: 2007029
Maryland Certification #: 322
Michigan DEQ Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT CERT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
North Dakota Certification #: R-036A
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Utah Certification #: MN00064
Virginia/DCLS Certification #: 002521
Virginia/VELAP Certification #: 460163
Washington Certification #: C754
West Virginia Certification #: 382
Wisconsin Certification #: 999407970

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## SAMPLE SUMMARY

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|---|---|---|---|---|
| **10230368001** | **2013 PIPERACK STOCKPILE 1** | Solid | 05/29/13 09:00 | 05/30/13 09:50 |

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## SAMPLE ANALYTE COUNT

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|---|---|---|---|---|---|
| **10230368001** | **2013 PIPERACK STOCKPILE 1** | WI MOD DRO | MT | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 6 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |

## REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# PROJECT NARRATIVE

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

**Method:** **WI MOD DRO**
**Description:** WIDRO GCS
**Client:** Barr Engineering
**Date:** June 24, 2013

**General Information:**
1 sample was analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with WI MOD DRO with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Additional Comments:**

Analyte Comments:

QC Batch: OEXT/21878

T6: High boiling point hydrocarbons are present in the sample.

- 2013 PIPERACK STOCKPILE 1 (Lab ID: 10230368001)
  - Diesel Range Organics

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# PROJECT NARRATIVE

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

**Method:** **WI MOD GRO**
**Description:** WIGRO GCV
**Client:** Barr Engineering
**Date:** June 24, 2013

**General Information:**
1 sample was analyzed for WI MOD GRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with TPH GRO/PVOC WI ext. with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Additional Comments:**

This data package has been reviewed for quality and completeness and is approved for release.

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(612)607-1700

# ANALYTICAL RESULTS

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

**Sample: 2013 PIPERACK STOCKPILE 1** **Lab ID: 10230368001** Collected: 05/29/13 09:00 Received: 05/30/13 09:50 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|
| **WIDRO GCS** | Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | |
| Diesel Range Organics | **553** | mg/kg | 309 | 10 | 06/04/13 08:58 | 06/06/13 19:45 | | T6 |
| ***Surrogates*** | | | | | | | | |
| n-Triacontane (S) | 139 | % | 50-150 | 10 | 06/04/13 08:58 | 06/06/13 19:45 | 638-68-6 | |
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | |
| Benzene | **<0.20** | mg/kg | 0.20 | 1 | 06/02/13 14:59 | 06/03/13 09:04 | 71-43-2 | |
| Ethylbenzene | **<0.20** | mg/kg | 0.20 | 1 | 06/02/13 14:59 | 06/03/13 09:04 | 100-41-4 | |
| Gasoline Range Organics | **<19.7** | mg/kg | 19.7 | 1 | 06/02/13 14:59 | 06/03/13 09:04 | | |
| Toluene | **<0.20** | mg/kg | 0.20 | 1 | 06/02/13 14:59 | 06/03/13 09:04 | 108-88-3 | |
| Xylene (Total) | **<0.59** | mg/kg | 0.59 | 1 | 06/02/13 14:59 | 06/03/13 09:04 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | .1 | % | 80-125 | 1 | 06/02/13 14:59 | 06/03/13 09:04 | 98-08-8 | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | |
| Percent Moisture | **54.2** | % | 0.10 | 1 | | 05/31/13 00:00 | | |

## REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# QUALITY CONTROL DATA

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

QC Batch: GCV/10850
QC Batch Method: TPH GRO/PVOC WI ext.
Analysis Method: WI MOD GRO
Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10230368001

METHOD BLANK: 1446147 Matrix: Solid
Associated Lab Samples: 10230368001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Benzene | mg/kg | <0.050 | 0.050 | 06/03/13 06:28 | |
| Ethylbenzene | mg/kg | <0.050 | 0.050 | 06/03/13 06:28 | |
| Gasoline Range Organics | mg/kg | <5.0 | 5.0 | 06/03/13 06:28 | |
| Toluene | mg/kg | <0.050 | 0.050 | 06/03/13 06:28 | |
| Xylene (Total) | mg/kg | <0.15 | 0.15 | 06/03/13 06:28 | |
| a,a,a-Trifluorotoluene (S) | % | .1 | 80-125 | 06/03/13 06:28 | |

LABORATORY CONTROL SAMPLE & LCSD: 1446148 1446149

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| Benzene | mg/kg | 5 | 4.6 | 4.3 | 91 | 86 | 80-120 | 5 | 20 | |
| Ethylbenzene | mg/kg | 5 | 5.0 | 4.5 | 100 | 91 | 80-120 | 9 | 20 | |
| Gasoline Range Organics | mg/kg | 50 | 48.3 | 50.8 | 97 | 102 | 80-120 | 5 | 20 | |
| Toluene | mg/kg | 5 | 4.8 | 4.5 | 96 | 89 | 80-120 | 7 | 20 | |
| Xylene (Total) | mg/kg | 15 | 15.2 | 13.6 | 101 | 91 | 80-120 | 11 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | .1 | .1 | 80-125 | | | |

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 1446150 1446151

| Parameter | Units | 10230021005 Result | MS Spike Conc. | MSD Spike Conc. | MS Result | MSD Result | MS % Rec | MSD % Rec | % Rec Limits | RPD | Max RPD | Qual |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Benzene | mg/kg | ND | 5.3 | 5.5 | 5.2 | 5.3 | 99 | 96 | 80-120 | 2 | 20 | |
| Ethylbenzene | mg/kg | ND | 5.3 | 5.5 | 5.7 | 5.8 | 108 | 105 | 80-120 | 2 | 20 | |
| Gasoline Range Organics | mg/kg | ND | 52.5 | 55.1 | 59.6 | 63.0 | 113 | 114 | 80-120 | 6 | 20 | |
| Toluene | mg/kg | ND | 5.3 | 5.5 | 5.5 | 5.6 | 105 | 101 | 80-120 | 1 | 20 | |
| Xylene (Total) | mg/kg | ND | 15.8 | 16.5 | 17.3 | 17.8 | 110 | 107 | 80-120 | 2 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | | | .1 | .1 | 80-125 | | | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

QC Batch: MPRP/39573
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10230368001

SAMPLE DUPLICATE: 1444836

| Parameter | Units | 10230154008 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 2.8 | 3.4 | 19 | 30 | |

SAMPLE DUPLICATE: 1445361

| Parameter | Units | 10230166003 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 14.7 | 16.0 | 9 | 30 | |

## REPORT OF LABORATORY ANALYSIS

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Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

QC Batch: OEXT/21878 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10230368001

METHOD BLANK: 1446957 Matrix: Solid
Associated Lab Samples: 10230368001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | <10.0 | 10.0 | 06/06/13 17:12 | |
| n-Triacontane (S) | % | 82 | 50-150 | 06/06/13 17:12 | |

LABORATORY CONTROL SAMPLE & LCSD: 1446958 1446959

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | 80 | 57.8 | 60.5 | 72 | 76 | 70-120 | 5 | 20 | |
| n-Triacontane (S) | % | | | | 83 | 83 | 50-150 | | | |

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(612)607-1700

# QUALIFIERS

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

## DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

## LABORATORIES

| | |
|---|---|
| PASI-M | Pace Analytical Services - Minneapolis |

## ANALYTE QUALIFIERS

| | |
|---|---|
| T6 | High boiling point hydrocarbons are present in the sample. |

**Pace Analytical Services, Inc.**
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## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092.02 003 018 2013 Rev
Pace Project No.: 10230368

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|---|---|---|---|---|---|
| **10230368001** | **2013 PIPERACK STOCKPILE 1** | WI MOD DRO | OEXT/21878 | WI MOD DRO | GCSV/11446 |
| **10230368001** | **2013 PIPERACK STOCKPILE 1** | TPH GRO/PVOC WI ext. | GCV/10850 | WI MOD GRO | GCV/10851 |
| **10230368001** | **2013 PIPERACK STOCKPILE 1** | ASTM D2974 | MPRP/39573 | | |

1562367

# Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

COC 1 of 1

Project Number: 4916 1092.02 003 018

Project Name: 2013 piperack (tank five)

Sample Origination State WI (use two letter postal state abbreviation)

COC Number: № 40534

Project Manager: REE

Project QC Contact: AAN

Sampled by: MAB/BJL2

Laboratory: Pace

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Matrix: | Type: Grab | Type: Comp. | Type: QC | Number of Containers/Preservative — Water: VOCs (HCl) #1 | SVOCs (unpreserved) #2 | Dissolved Metals ($HNO_3$) | Total Metals ($HNO_3$) | General (unpreserved) #3 | Diesel Range Organics (HCl) | Nutrients ($H_2SO_4$) #4 | | | Soil: VOCs (tared MeOH) #1 | GRO, BTEX (tared MeOH) #1 | DRO (tared unpreserved) | Metals (unpreserved) | SVOCs (unpreserved) #2 | % Solids (plastic vial, unpres.) | | | Total Number Of Containers | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. 2013 PipeRack stockpile 1 | | | | 05/29/13 | 0900 | | X | | X | | | | | | | | | | | | | X | X | | | X | | | 6 | GRO, DRO, %solids. Hold 3 containers |
| 2. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 3. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 4. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| | (Y) N | 5/29/13 | 1100 | | 5/29 | 11:00 |
| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
| | (Y) N | 5/29/13 | 1700 | | 5/30/13 | 0950 |

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☒ Other: FedEx Ground

Air Bill Number: T=1.8

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:RLG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

| Pace Analytical | Document Name: **Sample Condition Upon Receipt Form** | Document Revised: 28Jan2013 Page 1 of 1 |
|---|---|---|
| | Document No.: **F-MN-L-213-rev.06** | Issuing Authority: Pace Minnesota Quality Office |

**Sample Condition Upon Receipt**

**Client Name:** Barr **Project #:** WO#: 10230368

**Courier:** ☒ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other:

**Tracking Number:** 947035551500 1853

**Custody Seal on Cooler/Box Present?** ☒ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

**Packing Material:** ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☒ Yes ☐ No

**Thermom. Used:** ☐ B88A912167504 ☐ 80512447 ☒ 72337080 **Type of Ice:** ☒ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

**Cooler Temp Read (°C):** 1.6 **Cooler Temp Corrected (°C):** 1.8 **Biological Tissue Frozen?** ☐ Yes ☐ No

Temp should be above freezing to 6°C **Correction Factor:** +.2 **Date and Initials of Person Examining Contents:** 5/30/13

| | | | | Comments: |
|---|---|---|---|---|
| Chain of Custody Present? | ☒ Yes | ☐ No | ☐ N/A | 1. |
| Chain of Custody Filled Out? | ☒ Yes | ☐ No | ☐ N/A | 2. |
| Chain of Custody Relinquished? | ☒ Yes | ☐ No | ☐ N/A | 3. |
| Sampler Name and/or Signature on COC? | ☒ Yes | ☐ No | ☐ N/A | 4. |
| Samples Arrived within Hold Time? | ☒ Yes | ☐ No | ☐ N/A | 5. |
| **Short Hold Time Analysis (<72 hr)?** | ☐ Yes | ☒ No | ☐ N/A | 6. |
| **Rush Turn Around Time Requested?** | ☐ Yes | ☒ No | ☐ N/A | 7. |
| Sufficient Volume? | ☒ Yes | ☐ No | ☐ N/A | 8. |
| Correct Containers Used? | ☒ Yes | ☐ No | ☐ N/A | 9. |
| -Pace Containers Used? | ☐ Yes | ☒ No | ☐ N/A | |
| Containers Intact? | ☒ Yes | ☐ No | ☐ N/A | 10. |
| Filtered Volume Received for Dissolved Tests? | ☐ Yes | ☐ No | ☒ N/A | 11. |
| Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL | ☒ Yes | ☐ No | ☐ N/A | 12. |
| All containers needing acid/base preservation have been checked? Noncompliances are noted in 13. | ☐ Yes | ☐ No | ☒ N/A | 13. ☐ $HNO_3$ ☐ $H_2SO_4$ ☐ NaOH ☐ HCl |
| All containers needing preservation are found to be in compliance with EPA recommendation? ($HNO_3$, $H_2SO_4$, HCl<2; NaOH>12) | ☐ Yes | ☐ No | ☒ N/A | Sample # |
| Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water) | ☐ Yes | ☒ No | | Initial when completed: Lot # of added preservative: |
| Headspace in VOA Vials ( >6mm)? | ☐ Yes | ☐ No | ☒ N/A | 14. |
| Trip Blank Present? | ☐ Yes | ☐ No | ☒ N/A | 15. |
| Trip Blank Custody Seals Present? | ☐ Yes | ☐ No | ☒ N/A | |
| Pace Trip Blank Lot # (if purchased): | | | | |

**CLIENT NOTIFICATION/RESOLUTION** **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

**Project Manager Review:** Date: 5/30/13

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e out of hold, incorrect preservative, out of temp, incorrect containers)

LEGEND
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Tel: 651-642-1150
Fax: 651-642-1239

June 06, 2013

Ms. Andrea Nord
Barr Engineering Co.
4700 W 77th St
Minneapolis, MN 55435

Work Order Number: 1302479
RE: 49161092

Enclosed are the results of analyses for samples received by the laboratory on 06/04/13. If you have any questions concerning this report, please feel free to contact me.

All samples will be retained by LEGEND, unless consumed in the analysis, for 30 days from the date of this report and then discarded unless other arrangements are made.

WI Certification #998022410

Prepared by,
LEGEND TECHNICAL SERVICES, INC

Bach Pham
Client Manager II
bpham@legend-group.com

Tyler Jones
Chemist I
tjones@legend-group.com

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 | Work Order #: 1302479 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 06/06/13 |

## ANALYTICAL REPORT FOR SAMPLES

| Sample ID | Laboratory ID | Matrix | Date Sampled | Date Received |
|---|---|---|---|---|
| 2013 Pipe Rack-stockpile-2 | 1302479-01 | Soil | 05/31/13 09:15 | 06/04/13 09:30 |

**Shipping Container Information**

**Default Cooler** Temperature (°C): 5.9

Received on ice: Yes
Temperature blank was present
Received on ice pack: No
Received on melt water: No
Ambient: No
Acceptable (IH/ISO only): No
Custody seals: No

**Case Narrative:**

LEGEND Technical Services, Inc.
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Tel: 651-642-1150
Fax: 651-642-1239

| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 | Work Order #: 1302479 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 06/06/13 |

## DRO/8015D
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **2013 Pipe Rack-stockpile-2 (1302479-01) Soil Sampled: 05/31/13 09:15 Received: 06/04/13 9:30** | | | | | | | | | | |
| **Diesel Range Organics** | **340** | 15 | 1.8 | mg/kg dry | 1 | B3F0503 | 06/05/13 | 06/05/13 | WI(95) DRO | |
| *Surrogate: Triacontane (C-30)* | *81.3* | | | *70-130 %* | | " | " | " | " | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 | Work Order #: 1302479 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 06/06/13 |

## WI(95) GRO/8015D
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **2013 Pipe Rack-stockpile-2 (1302479-01) Soil Sampled: 05/31/13 09:15 Received: 06/04/13 9:30** | | | | | | | | | | |
| Benzene | <0.045 | 0.045 | 0.0055 | mg/kg dry | 1 | B3F0412 | 06/04/13 | 06/04/13 | WI(95) GRO | |
| Ethylbenzene | <0.045 | 0.045 | 0.0038 | mg/kg dry | 1 | " | " | " | " | |
| Toluene | <0.045 | 0.045 | 0.0048 | mg/kg dry | 1 | " | " | " | " | |
| Xylenes (total) | <0.13 | 0.13 | 0.014 | mg/kg dry | 1 | " | " | " | " | |
| *Surrogate: 4-Fluorochlorobenzene* | *102* | | | *80-150 %* | | " | " | " | " | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 | Work Order #: 1302479 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 06/06/13 |

## PERCENT SOLIDS
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **2013 Pipe Rack-stockpile-2 (1302479-01) Soil Sampled: 05/31/13 09:15 Received: 06/04/13 9:30** | | | | | | | | | | |
| **% Solids** | **56** | | | % | 1 | B3F0609 | 06/06/13 | 06/06/13 | % calculation | |

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Tel: 651-642-1150
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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 | Work Order #: 1302479 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 06/06/13 |

## DRO/8015D - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B3F0503 - Sonication (Wisc DRO)** | | | | | | | | | | | |
| **Blank (B3F0503-BLK1)** | | | | | Prepared & Analyzed: 06/05/13 | | | | | | |
| Diesel Range Organics | < 6.7 | 6.7 | 0.78 | mg/kg wet | | | | | | | |
| *Surrogate: Triacontane (C-30)* | *11.3* | | | *mg/kg wet* | *13.3* | | *84.5* | *70-130* | | | |
| **LCS (B3F0503-BS1)** | | | | | Prepared & Analyzed: 06/05/13 | | | | | | |
| Diesel Range Organics | 60.1 | 8.0 | 0.93 | mg/kg wet | 64.0 | | 93.9 | 70-120 | | | |
| *Surrogate: Triacontane (C-30)* | *14.5* | | | *mg/kg wet* | *16.0* | | *90.9* | *70-130* | | | |
| **LCS Dup (B3F0503-BSD1)** | | | | | Prepared: 06/05/13 Analyzed: 06/06/13 | | | | | | |
| Diesel Range Organics | 61.4 | 8.0 | 0.93 | mg/kg wet | 64.0 | | 95.9 | 70-120 | 2.03 | 20 | |
| *Surrogate: Triacontane (C-30)* | *15.2* | | | *mg/kg wet* | *16.0* | | *95.0* | *70-130* | | | |

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www.legend-group.com

88 Empire Drive
St Paul, MN 55103
Tel: 651-642-1150
Fax: 651-642-1239

| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 | Work Order #: 1302479 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 06/06/13 |

## WI(95) GRO/8015D - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B3F0412 - EPA 5035 Soil (Purge and Trap)** | | | | | | | | | | | |
| **Blank (B3F0412-BLK1)** | | | | | Prepared & Analyzed: 06/04/13 | | | | | | |
| Benzene | < 0.025 | 0.025 | 0.0031 | mg/kg wet | | | | | | | |
| Ethylbenzene | < 0.025 | 0.025 | 0.0022 | mg/kg wet | | | | | | | |
| Toluene | < 0.025 | 0.025 | 0.0027 | mg/kg wet | | | | | | | |
| Xylenes (total) | < 0.075 | 0.075 | 0.0080 | mg/kg wet | | | | | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *24.9* | | | *ug/L* | *25.0* | | *99.6* | *80-150* | | | |
| **LCS (B3F0412-BS1)** | | | | | Prepared & Analyzed: 06/04/13 | | | | | | |
| Benzene | 99.8 | | | ug/L | 100 | | 99.8 | 80-120 | | | |
| Ethylbenzene | 103 | | | ug/L | 100 | | 103 | 80-120 | | | |
| Toluene | 103 | | | ug/L | 100 | | 103 | 80-120 | | | |
| Xylenes (total) | 313 | | | ug/L | 300 | | 104 | 80-120 | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *25.2* | | | *ug/L* | *25.0* | | *101* | *80-150* | | | |
| **LCS Dup (B3F0412-BSD1)** | | | | | Prepared & Analyzed: 06/04/13 | | | | | | |
| Benzene | 100 | | | ug/L | 100 | | 100 | 80-120 | 0.669 | 20 | |
| Ethylbenzene | 101 | | | ug/L | 100 | | 101 | 80-120 | 1.32 | 20 | |
| Toluene | 102 | | | ug/L | 100 | | 102 | 80-120 | 0.516 | 20 | |
| Xylenes (total) | 313 | | | ug/L | 300 | | 104 | 80-120 | 0.0386 | 20 | |
| *Surrogate: 4-Fluorochlorobenzene* | *26.0* | | | *ug/L* | *25.0* | | *104* | *80-150* | | | |
| **Matrix Spike (B3F0412-MS1)** | | **Source: 1302479-01** | | | Prepared & Analyzed: 06/04/13 | | | | | | |
| Benzene | 101 | | | ug/L | 100 | < | 101 | 80-120 | | | |
| Ethylbenzene | 104 | | | ug/L | 100 | 0.274 | 104 | 80-120 | | | |
| Toluene | 103 | | | ug/L | 100 | 0.116 | 103 | 80-120 | | | |
| Xylenes (total) | 323 | | | ug/L | 300 | 0.195 | 108 | 80-120 | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *26.6* | | | *ug/L* | *25.0* | | *106* | *80-150* | | | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 | Work Order #: 1302479 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 06/06/13 |

## PERCENT SOLIDS - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B3F0609 - General Preparation** | | | | | | | | | | | |
| **Duplicate (B3F0609-DUP1)** | | **Source: 1302421-05** | | | Prepared & Analyzed: 06/06/13 | | | | | | |
| % Solids | 93.0 | | | % | | 92.0 | | | 1.08 | 20 | |
| **Duplicate (B3F0609-DUP2)** | | **Source: 1302421-15** | | | Prepared & Analyzed: 06/06/13 | | | | | | |
| % Solids | 89.0 | | | % | | 89.0 | | | 0.00 | 20 | |
| **Duplicate (B3F0609-DUP3)** | | **Source: 1302482-01** | | | Prepared & Analyzed: 06/06/13 | | | | | | |
| % Solids | 92.0 | | | % | | 92.0 | | | 0.00 | 20 | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 | Work Order #: 1302479 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 06/06/13 |

## Notes and Definitions

| | |
|---|---|
| < | Less than value listed |
| dry | Sample results reported on a dry weight basis |
| NA | Not applicable. The %RPD is not calculated from values less than the reporting limit. |
| MDL | Method Detection Limit |
| RL | Reporting Limit |
| RPD | Relative Percent Difference |
| LCS | Laboratory Control Spike = Blank Spike (BS) = Laboratory Fortified Blank (LFB) |
| MS | Matrix Spike = Laboratory Fortified Matrix (LFM) |

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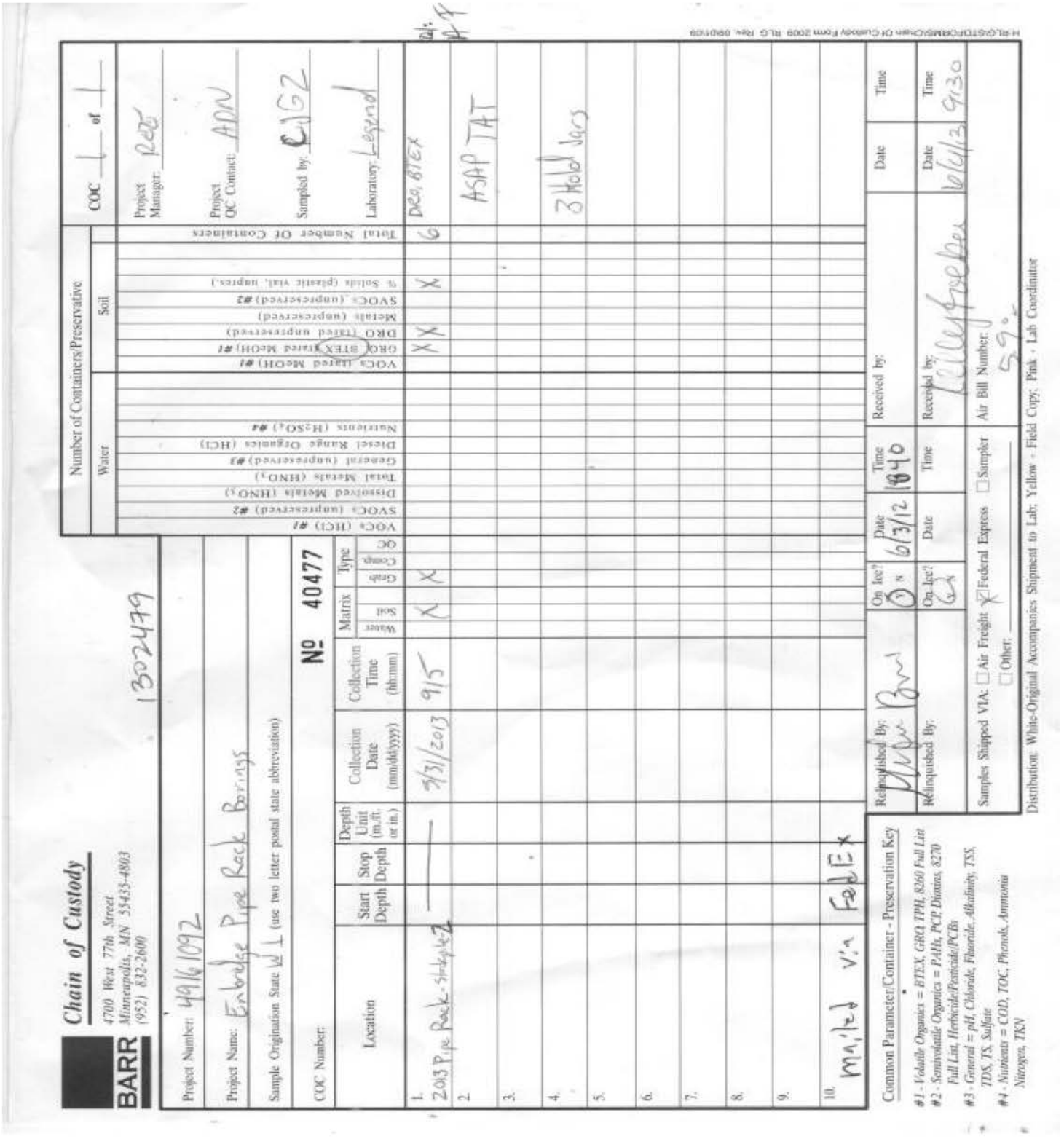

Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

1302479

Project Number: 49161092
Project Name: Enbridge Pipe Rack Berms
Sample Origination State: WI (use two letter postal state abbreviation)
COC Number: № 40477

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Waste | Matrix: Soil | Type: Grab | Type: Comp. | QC | VOCs (HCl) #1 | SVOCs (unpreserved) #2 | Dissolved Metals ($HNO_3$) | Total Metals ($HNO_3$) | Diesel Range Organics (HCl) | General (unpreserved) #3 | Nutrients ($H_2SO_4$) #4 | Water: VOCs (Jarred MeOH) #1 | Water: GRO (BTEX) (Jarred MeOH) #1 | Water: DRO (Jarred unpreserved) | Water: Metals (unpreserved) | Soil: SVOCs (unpreserved) #2 | Soil: % Solids (plastic vial, unpres.) | Total Number Of Containers |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. 203 Pipe Rack-Stockpile | — | — | — | 9/31/2013 | 915 | | X | X | | | | | | | | | | | X | X | | | X | 6 |
| 2. | | | | | | | | | | | | | | | | | | | | | | | | |
| 3. | | | | | | | | | | | | | | | | | | | | | | | | |
| 4. | | | | | | | | | | | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | | | | | | | | | | | |
| 6. | | | | | | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | | | | | | | | | | | |
| 10. MA/Fed Ex | | | | | | | | | | | | | | | | | | | | | | | | |

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| | Y N | 6/3/12 | 1040 | | | |
| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
| | Y N | | | | 6/6/13 | 9:30 |

Samples Shipped VIA: ☐ Air Freight ☒ Federal Express ☐ Sampler ☐ Other: _____ Air Bill Number: 59-

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:\LG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09012009

Project Manager: Reed
Project QC Contact: ADV
Sampled by: EJG2
Laboratory: Legend
Deg, BTEX
ASAP TAT
3 Hold Jars

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

July 18, 2013

Andrea Nord
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

RE: Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on June 21, 2013. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

This report was revised on July 18, 2013 to correct the project ID.

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

Sincerely,

Andrea Opland

andrea.opland@pacelabs.com
Project Manager

Enclosures

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# CERTIFICATIONS

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

**Minnesota Certification IDs**

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Hawaii Certification #Pace
Idaho Certification #: MN00064
Illinois Certification #: 200011
Kansas Certification #: E-10167
Louisiana Certification #: 03086
Louisiana Certification #: LA080009
Maine Certification #: 2007029
Maryland Certification #: 322
Michigan DEQ Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT CERT0092
Nebraska Certification #: Pace
Nevada Certification #: MN_00064
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Utah Certification #: MN00064
Virginia/DCLS Certification #: 002521
Virginia/VELAP Certification #: 460163
Washington Certification #: C754
West Virginia Certification #: 382
Wisconsin Certification #: 999407970

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## SAMPLE SUMMARY

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|---|---|---|---|---|
| **10232975001** | **2013 PIPE RACK STOCKPILE-3** | Solid | 06/19/13 16:30 | 06/21/13 08:45 |

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## SAMPLE ANALYTE COUNT

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|---|---|---|---|---|---|
| **10232975001** | **2013 PIPE RACK STOCKPILE-3** | WI MOD DRO | KL1 | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 5 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |

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# PROJECT NARRATIVE

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

**Method:** **WI MOD DRO**
**Description:** WIDRO GCS
**Client:** Barr Engineering
**Date:** July 18, 2013

**General Information:**
1 sample was analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with WI MOD DRO with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Additional Comments:**

Analyte Comments:

QC Batch: OEXT/22079

T6: High boiling point hydrocarbons are present in the sample.

- 2013 PIPE RACK STOCKPILE-3 (Lab ID: 10232975001)
  - Diesel Range Organics

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# PROJECT NARRATIVE

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

**Method:** **WI MOD GRO**
**Description:** WIGRO GCV
**Client:** Barr Engineering
**Date:** July 18, 2013

**General Information:**
1 sample was analyzed for WI MOD GRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with TPH GRO/PVOC WI ext. with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Duplicate Sample:**
All duplicate sample results were within method acceptance criteria with any exceptions noted below.

**Additional Comments:**

This data package has been reviewed for quality and completeness and is approved for release.

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## ANALYTICAL RESULTS

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

**Sample: 2013 PIPE RACK STOCKPILE-3** **Lab ID: 10232975001** Collected: 06/19/13 16:30 Received: 06/21/13 08:45 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIDRO GCS** | Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | | |
| Diesel Range Organics | **22.7** | mg/kg | 11.6 | 1.3 | 1 | 06/24/13 13:22 | 06/27/13 04:09 | | T6 |
| ***Surrogates*** | | | | | | | | | |
| n-Triacontane (S) | 80 | % | 50-150 | | 1 | 06/24/13 13:22 | 06/27/13 04:09 | 638-68-6 | |
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| Benzene | **<0.065** | mg/kg | 0.065 | 0.010 | 1 | 06/21/13 14:23 | 06/27/13 03:31 | 71-43-2 | |
| Ethylbenzene | **<0.065** | mg/kg | 0.065 | 0.0090 | 1 | 06/21/13 14:23 | 06/27/13 03:31 | 100-41-4 | |
| Toluene | **<0.065** | mg/kg | 0.065 | 0.0065 | 1 | 06/21/13 14:23 | 06/27/13 03:31 | 108-88-3 | |
| Xylene (Total) | **<0.19** | mg/kg | 0.19 | 0.022 | 1 | 06/21/13 14:23 | 06/27/13 03:31 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 93 | % | 80-125 | | 1 | 06/21/13 14:23 | 06/27/13 03:31 | 98-08-8 | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **17.0** | % | 0.10 | 0.10 | 1 | | 06/24/13 00:00 | | |

## REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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# QUALITY CONTROL DATA

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

QC Batch: GCV/10946
QC Batch Method: TPH GRO/PVOC WI ext.
Analysis Method: WI MOD GRO
Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10232975001

METHOD BLANK: 1462894 Matrix: Solid
Associated Lab Samples: 10232975001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Benzene | mg/kg | <0.050 | 0.050 | 06/25/13 14:49 | |
| Ethylbenzene | mg/kg | <0.050 | 0.050 | 06/25/13 14:49 | |
| Toluene | mg/kg | <0.050 | 0.050 | 06/25/13 14:49 | |
| Xylene (Total) | mg/kg | <0.15 | 0.15 | 06/25/13 14:49 | |
| a,a,a-Trifluorotoluene (S) | % | 99 | 80-125 | 06/25/13 14:49 | |

LABORATORY CONTROL SAMPLE & LCSD: 1462895 1462896

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| Benzene | mg/kg | 5 | 4.6 | 4.5 | 92 | 90 | 80-120 | 2 | 20 | |
| Ethylbenzene | mg/kg | 5 | 5.0 | 4.9 | 100 | 97 | 80-120 | 3 | 20 | |
| Toluene | mg/kg | 5 | 4.8 | 4.7 | 97 | 95 | 80-120 | 2 | 20 | |
| Xylene (Total) | mg/kg | 15 | 15.4 | 15.0 | 103 | 100 | 80-120 | 2 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 97 | 96 | 80-125 | | | |

MATRIX SPIKE SAMPLE: 1462897

| Parameter | Units | 10232665002 Result | Spike Conc. | MS Result | MS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|---|
| Benzene | mg/kg | ND | 5.6 | 4.7 | 84 | 80-120 | |
| Ethylbenzene | mg/kg | ND | 5.6 | 5.1 | 91 | 80-120 | |
| Toluene | mg/kg | ND | 5.6 | 5.0 | 88 | 80-120 | |
| Xylene (Total) | mg/kg | ND | 16.8 | 16.1 | 95 | 80-120 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 97 | 80-125 | |

SAMPLE DUPLICATE: 1462898

| Parameter | Units | 10232665003 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Benzene | mg/kg | ND | <0.058 | | 20 | |
| Ethylbenzene | mg/kg | ND | <0.058 | | 20 | |
| Toluene | mg/kg | ND | <0.058 | | 20 | |
| Xylene (Total) | mg/kg | ND | <0.17 | | 20 | |
| a,a,a-Trifluorotoluene (S) | % | 99 | 97 | 3 | | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

QC Batch: MPRP/40144
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10232975001

SAMPLE DUPLICATE: 1464138

| Parameter | Units | 10233018020 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 11.1 | 11.1 | .1 | 30 | |

SAMPLE DUPLICATE: 1464251

| Parameter | Units | 10232975001 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 17.0 | 18.7 | 9 | 30 | |

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

QC Batch: OEXT/22079 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10232975001

METHOD BLANK: 1464085 Matrix: Solid
Associated Lab Samples: 10232975001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | <10.0 | 10.0 | 06/27/13 01:50 | |
| n-Triacontane (S) | % | 80 | 50-150 | 06/27/13 01:50 | |

LABORATORY CONTROL SAMPLE & LCSD: 1464086 1464087

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | 80 | 71.5 | 67.3 | 89 | 84 | 70-120 | 6 | 20 | |
| n-Triacontane (S) | % | | | | 85 | 85 | 50-150 | | | |

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# QUALIFIERS

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

## DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

## LABORATORIES

| | |
|---|---|
| PASI-M | Pace Analytical Services - Minneapolis |

## ANALYTE QUALIFIERS

| | |
|---|---|
| T6 | High boiling point hydrocarbons are present in the sample. |

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092 PIPE RACK REV
Pace Project No.: 10232975

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|---|---|---|---|---|---|
| **10232975001** | **2013 PIPE RACK STOCKPILE-3** | WI MOD DRO | OEXT/22079 | WI MOD DRO | GCSV/11575 |
| **10232975001** | **2013 PIPE RACK STOCKPILE-3** | TPH GRO/PVOC WI ext. | GCV/10946 | WI MOD GRO | GCV/10947 |
| **10232975001** | **2013 PIPE RACK STOCKPILE-3** | ASTM D2974 | MPRP/40144 | | |

# Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

(131)

Project Number: 49161092

Project Name: Enbridge Piperack Excavation

Sample Origination State WI (use two letter postal state abbreviation)

COC Number: № 40481

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Type: Grab | Type: Comp. | Type: QC | Water: VOCs (HCl) #1 | Water: SVOCs (unpreserved) #2 | Water: Dissolved Metals ($HNO_3$) | Water: Total Metals ($HNO_3$) | Water: General (unpreserved) #3 | Water: Diesel Range Organics (HCl) | Water: Nutrients ($H_2SO_4$) #4 | Soil: VOCs (tared MeOH) #1 | Soil: GRO, BTEX (tared MeOH) #1 | Soil: DRO (tared unpreserved) | Soil: Metals (unpreserved) | Soil: SVOCs (unpreserved) #2 | Soil: % Solids (plastic vial, unpres.) | Total Number Of Containers |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. 2013 Pipe Rack Stockpile-3 | | | | 06/19/2013 | 16:30 | | X | X | | | | | | | | | | | X | X | | | X | 6 |
| 2. | | | | | | | | | | | | | | | | | | | | | | | | |
| 3. | | | | | | | | | | | | | | | | | | | | | | | | |
| 4. | | | | | | | | | | | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | | | | | | | | | | | |
| 6. | | | | | | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | | | | | | | | | | | |

Number of Containers/Preservative: Water / Soil

COC 1 of 1

Project Manager: REF

Project QC Contact: ADN

Sampled by: LEN

Laboratory: Legend

WI DRO, BTEX 00

3 Hold Jars

5-Day TAT

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| | (Y) N | 6/20/13 | 1230 | | 6/20/13 | 1230 |
| Relinquished By: | On Ice? | Date | Time | Received by: | Date | Time |
| | (Y) N | 6/20/13 | 1700 | PACE T=4.5 | 6/21/13 | 0845 |

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☒ Sampler ☐ Other: _____

Air Bill Number:

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H:\RLG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

| Pace Analytical® | Document Name: Sample Condition Upon Receipt Form | Document Revised: 28Jan2013 Page 1 of 1 |
|---|---|---|
| | Document No.: F-MN-L-213-rev.06 | Issuing Authority: Pace Minnesota Quality Office |

**Sample Condition Upon Receipt**

**Client Name:** BARR

**Project #:** WO#: 10232975

10232975

**Courier:** ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other: _____

**Tracking Number:** 9470855 1500133

**Custody Seal on Cooler/Box Present?** ☑ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No

**Optional:** Proj. Due Date: Proj. Name:

**Packing Material:** ☑ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other: _____ **Temp Blank?** ☐ Yes ☒ No

**Thermom. Used:** ☒ B88A912167504 ☐ 80512447 ☐ 72337080 **Type of Ice:** ☐ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

**Cooler Temp Read (°C):** 4.5 **Cooler Temp Corrected (°C):** 4.5 **Biological Tissue Frozen?** ☐ Yes ☐ No

Temp should be above freezing to 6°C **Correction Factor:** 0.0 **Date and Initials of Person Examining Contents:** 6/21/13

**Comments:**

| | | | | |
|---|---|---|---|---|
| Chain of Custody Present? | ☒ Yes | ☐ No | ☐ N/A | 1. |
| Chain of Custody Filled Out? | ☒ Yes | ☐ No | ☐ N/A | 2. |
| Chain of Custody Relinquished? | ☒ Yes | ☐ No | ☐ N/A | 3. |
| Sampler Name and/or Signature on COC? | ☐ Yes | ☒ No | ☐ N/A | 4. |
| Samples Arrived within Hold Time? | ☒ Yes | ☐ No | ☐ N/A | 5. |
| **Short Hold Time Analysis (<72 hr)?** | ☐ Yes | ☒ No | ☐ N/A | 6. |
| **Rush Turn Around Time Requested?** | ☐ Yes | ☒ No | ☐ N/A | 7. |
| Sufficient Volume? | ☒ Yes | ☐ No | ☐ N/A | 8. |
| Correct Containers Used? | ☒ Yes | ☐ No | ☐ N/A | 9. |
| -Pace Containers Used? | ☐ Yes | ☒ No | ☐ N/A | |
| Containers Intact? | ☒ Yes | ☐ No | ☐ N/A | 10. |
| Filtered Volume Received for Dissolved Tests? | ☐ Yes | ☐ No | ☒ N/A | 11. |
| Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL | ☐ Yes | ☒ No | ☐ N/A | 12. No Time on Smpls 16:30 |
| All containers needing acid/base preservation have been checked? Noncompliances are noted in 13. | ☐ Yes | ☐ No | ☒ N/A | 13. ☐ $HNO_3$ ☐ $H_2SO_4$ ☐ NaOH ☐ HCl Sample # |
| All containers needing preservation are found to be in compliance with EPA recommendation? ($HNO_3$, $H_2SO_4$, HCl<2; NaOH>12) | ☐ Yes | ☐ No | ☒ N/A | |
| Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water) | ☐ Yes | ☒ No | | Initial when completed: Lot # of added preservative: |
| Headspace in VOA Vials ( >6mm)? | ☐ Yes | ☐ No | ☒ N/A | 14. |
| Trip Blank Present? | ☐ Yes | ☐ No | ☒ N/A | 15. |
| Trip Blank Custody Seals Present? | ☐ Yes | ☐ No | ☒ N/A | |
| Pace Trip Blank Lot # (if purchased): | | | | |

**CLIENT NOTIFICATION/RESOLUTION** **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

**Project Manager Review:** _____ **Date:** 6/21/13

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e out of hold, incorrect preservative, out of temp, incorrect containers)

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

July 18, 2013

Andrea Nord
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

RE: Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on June 26, 2013. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

This report was revised on July 18, 2013 to correct the sample ID and project ID.

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

Sincerely,

Andrea Opland

andrea.opland@pacelabs.com
Project Manager

Enclosures

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# CERTIFICATIONS

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

**Minnesota Certification IDs**

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Hawaii Certification #Pace
Idaho Certification #: MN00064
Illinois Certification #: 200011
Kansas Certification #: E-10167
Louisiana Certification #: 03086
Louisiana Certification #: LA080009
Maine Certification #: 2007029
Maryland Certification #: 322
Michigan DEQ Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT CERT0092
Nebraska Certification #: Pace
Nevada Certification #: MN_00064
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Utah Certification #: MN00064
Virginia/DCLS Certification #: 002521
Virginia/VELAP Certification #: 460163
Washington Certification #: C754
West Virginia Certification #: 382
Wisconsin Certification #: 999407970

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## SAMPLE SUMMARY

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|---|---|---|---|---|
| **10233409001** | **2013 PIPE RACK Stockpile 4** | Solid | 06/25/13 10:00 | 06/26/13 09:20 |

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## SAMPLE ANALYTE COUNT

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|---|---|---|---|---|---|
| **10233409001** | **2013 PIPE RACK Stockpile 4** | WI MOD DRO | JRH | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 5 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |

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# PROJECT NARRATIVE

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

**Method:** **WI MOD DRO**
**Description:** WIDRO GCS
**Client:** Barr Engineering
**Date:** July 18, 2013

**General Information:**
1 sample was analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with WI MOD DRO with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

**Additional Comments:**

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# PROJECT NARRATIVE

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

**Method:** **WI MOD GRO**
**Description:** WIGRO GCV
**Client:** Barr Engineering
**Date:** July 18, 2013

**General Information:**
1 sample was analyzed for WI MOD GRO. All samples were received in acceptable condition with any exceptions noted below.

**Hold Time:**
The samples were analyzed within the method required hold times with any exceptions noted below.

**Sample Preparation:**
The samples were prepared in accordance with TPH GRO/PVOC WI ext. with any exceptions noted below.

**Initial Calibrations (including MS Tune as applicable):**
All criteria were within method requirements with any exceptions noted below.

**Continuing Calibration:**
All criteria were within method requirements with any exceptions noted below.

**Internal Standards:**
All internal standards were within QC limits with any exceptions noted below.

**Surrogates:**
All surrogates were within QC limits with any exceptions noted below.

QC Batch: GCV/10970

S0: Surrogate recovery outside laboratory control limits.

- MS (Lab ID: 1467280)
  - a,a,a-Trifluorotoluene (S)

**Method Blank:**
All analytes were below the report limit in the method blank with any exceptions noted below.

**Laboratory Control Spike:**
All laboratory control spike compounds were within QC limits with any exceptions noted below.

**Matrix Spikes:**
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

QC Batch: GCV/10970

A matrix spike and matrix spike duplicate (MS/MSD) were performed on the following sample(s): 10233318001

M1: Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.

- MS (Lab ID: 1467280)
  - Benzene
  - Ethylbenzene
  - Toluene

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# PROJECT NARRATIVE

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

**Method:** **WI MOD GRO**
**Description:** WIGRO GCV
**Client:** Barr Engineering
**Date:** July 18, 2013

**Duplicate Sample:**
All duplicate sample results were within method acceptance criteria with any exceptions noted below.

QC Batch: GCV/10970

D6: The relative percent difference (RPD) between the sample and sample duplicate exceeded laboratory control limits.

- DUP (Lab ID: 1467281)
  - Benzene
  - Ethylbenzene
  - Toluene

**Additional Comments:**

Analyte Comments:

QC Batch: GCV/10970

1M: Surrogate recovery outside laboratory control limits due to matrix interferences.

- MS (Lab ID: 1467280)
  - a,a,a-Trifluorotoluene (S)

This data package has been reviewed for quality and completeness and is approved for release.

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# ANALYTICAL RESULTS

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

**Sample: 2013 PIPE RACK Stockpile 4** **Lab ID: 10233409001** Collected: 06/25/13 10:00 Received: 06/26/13 09:20 Matrix: Solid

***Results reported on a "dry-weight" basis***

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---|---|---|---|---|---|---|---|---|
| **WIDRO GCS** | Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | | |
| Diesel Range Organics | **<17.5** | mg/kg | 17.5 | 3.8 | 1 | 06/26/13 11:37 | 07/01/13 11:16 | | |
| ***Surrogates*** | | | | | | | | | |
| n-Triacontane (S) | 89 | % | 50-150 | | 1 | 06/26/13 11:37 | 07/01/13 11:16 | 638-68-6 | |
| **WIGRO GCV** | Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | |
| Benzene | **<0.083** | mg/kg | 0.083 | 0.013 | 1 | 06/27/13 18:07 | 06/29/13 05:03 | 71-43-2 | |
| Ethylbenzene | **<0.083** | mg/kg | 0.083 | 0.012 | 1 | 06/27/13 18:07 | 06/29/13 05:03 | 100-41-4 | |
| Toluene | **<0.083** | mg/kg | 0.083 | 0.0083 | 1 | 06/27/13 18:07 | 06/29/13 05:03 | 108-88-3 | |
| Xylene (Total) | **<0.25** | mg/kg | 0.25 | 0.028 | 1 | 06/27/13 18:07 | 06/29/13 05:03 | 1330-20-7 | |
| ***Surrogates*** | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 97 | % | 80-125 | | 1 | 06/27/13 18:07 | 06/29/13 05:03 | 98-08-8 | |
| **Dry Weight** | Analytical Method: ASTM D2974 | | | | | | | | |
| Percent Moisture | **31.3** | % | 0.10 | 0.10 | 1 | | 06/27/13 00:00 | | |

## REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

# QUALITY CONTROL DATA

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

QC Batch: GCV/10970
QC Batch Method: TPH GRO/PVOC WI ext.
Associated Lab Samples: 10233409001

Analysis Method: WI MOD GRO
Analysis Description: WIGRO Solid GCV

METHOD BLANK: 1467277 Matrix: Solid
Associated Lab Samples: 10233409001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Benzene | mg/kg | <0.050 | 0.050 | 06/28/13 22:33 | |
| Ethylbenzene | mg/kg | <0.050 | 0.050 | 06/28/13 22:33 | |
| Toluene | mg/kg | <0.050 | 0.050 | 06/28/13 22:33 | |
| Xylene (Total) | mg/kg | <0.15 | 0.15 | 06/28/13 22:33 | |
| a,a,a-Trifluorotoluene (S) | % | 105 | 80-125 | 06/28/13 22:33 | |

LABORATORY CONTROL SAMPLE & LCSD: 1467278 1467279

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| Benzene | mg/kg | 5 | 4.3 | 4.3 | 86 | 87 | 80-120 | .3 | 20 | |
| Ethylbenzene | mg/kg | 5 | 4.7 | 4.7 | 93 | 94 | 80-120 | .8 | 20 | |
| Toluene | mg/kg | 5 | 4.5 | 4.6 | 90 | 91 | 80-120 | .9 | 20 | |
| Xylene (Total) | mg/kg | 15 | 14.2 | 14.4 | 95 | 96 | 80-120 | .8 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 97 | 96 | 80-125 | | | |

MATRIX SPIKE SAMPLE: 1467280

| Parameter | Units | 10233318001 Result | Spike Conc. | MS Result | MS % Rec | % Rec Limits | Qualifiers |
|---|---|---|---|---|---|---|---|
| Benzene | mg/kg | 13.6 | 5.8 | 23.3 | 168 | 80-120 | M1 |
| Ethylbenzene | mg/kg | 46.4 | 5.8 | 63.1 | 289 | 80-120 | M1 |
| Toluene | mg/kg | 192 | 5.8 | 251 | 1040 | 80-120 | M1 |
| Xylene (Total) | mg/kg | 245 | 17.3 | 321 | 437 | 80-120 | ES |
| a,a,a-Trifluorotoluene (S) | % | | | | 126 | 80-125 | 1M,S0 |

SAMPLE DUPLICATE: 1467281

| Parameter | Units | 10233318002 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Benzene | mg/kg | 1.8 | 12.4 | 150 | 20 | D6 |
| Ethylbenzene | mg/kg | 9.9 | 45.1 | 128 | 20 | D6 |
| Toluene | mg/kg | 30.2 | 169 | 139 | 20 | D6 |
| Xylene (Total) | mg/kg | 56.7 | 246 | 125 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | 102 | 116 | 8 | | |

## REPORT OF LABORATORY ANALYSIS

**Pace Analytical Services, Inc.**
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(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

QC Batch: MPRP/40224
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10233409001

SAMPLE DUPLICATE: 1466398

| Parameter | Units | 10233409001 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 31.3 | 30.8 | 2 | 30 | |

SAMPLE DUPLICATE: 1466399

| Parameter | Units | 10232880001 Result | Dup Result | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|
| Percent Moisture | % | 15.5 | 17.4 | 12 | 30 | |

## REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
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(612)607-1700

## QUALITY CONTROL DATA

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

QC Batch: OEXT/22101 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10233409001

METHOD BLANK: 1465539 Matrix: Solid
Associated Lab Samples: 10233409001

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | <10.0 | 10.0 | 07/01/13 09:59 | |
| n-Triacontane (S) | % | 83 | 50-150 | 07/01/13 09:59 | |

LABORATORY CONTROL SAMPLE & LCSD: 1465540 1465541

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|---|---|---|---|---|---|---|---|---|---|---|
| Diesel Range Organics | mg/kg | 80 | 69.1 | 65.7 | 86 | 82 | 70-120 | 5 | 20 | |
| n-Triacontane (S) | % | | | | 92 | 84 | 50-150 | | | |

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# QUALIFIERS

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

## DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

## LABORATORIES

| | |
|---|---|
| PASI-M | Pace Analytical Services - Minneapolis |

## ANALYTE QUALIFIERS

| | |
|---|---|
| 1M | Surrogate recovery outside laboratory control limits due to matrix interferences. |
| D6 | The relative percent difference (RPD) between the sample and sample duplicate exceeded laboratory control limits. |
| ES | The reported result is estimated because one or more of the constituent results are qualified as such. |
| M1 | Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery. |
| S0 | Surrogate recovery outside laboratory control limits. |

**Pace Analytical Services, Inc.**
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092.02003018 PIPE RACK RE
Pace Project No.: 10233409

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|---|---|---|---|---|---|
| **10233409001** | **2013 PIPE RACK Stockpile 4** | WI MOD DRO | OEXT/22101 | WI MOD DRO | GCSV/11588 |
| **10233409001** | **2013 PIPE RACK Stockpile 4** | TPH GRO/PVOC WI ext. | GCV/10970 | WI MOD GRO | GCV/10971 |
| **10233409001** | **2013 PIPE RACK Stockpile 4** | ASTM D2974 | MPRP/40224 | | |

# Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

AA 6-26-13 1125
RUSH 10233909

COC 1 of 1

Project Number: 49161092.02 003 018

Project Name: Pipe Rack Construction

Sample Origination State WI (use two letter postal state abbreviation)

COC Number: № 40101

Project Manager: REE

Project QC Contact: AAN

Sampled by: CJG2

Laboratory: Pace

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Matrix: | Type: Grab | Type: Comp. | Type: QC | Number of Containers/Preservative — Water: VOCs (HCl) #1 | SVOCs (unpreserved) #2 | Dissolved Metals ($HNO_3$) | Total Metals ($HNO_3$) | General (unpreserved) #3 | Diesel Range Organics (HCl) | Nutrients ($H_2SO_4$) #4 | | | Soil: VOCs (tared MeOH) #1 | ~~DRO~~ BTEX (tared MeOH) #1 | DRO (tared unpreserved) | Metals (unpreserved) | SVOCs (unpreserved) #2 | % Solids (plastic vial, unpres.) | 3 Extra Jars - Hold | | Total Number Of Containers | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. 2013 Pipe Rack Stockpile-4 | - | - | - | 6/25/13 | 1000 | | X | | X | | | | | | | | | | | | 1 | 1 | | | 1 | 1 | | 6 | BTEX, DRO, % solids |
| 2. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | 3 Extra Jars - Hold |
| 3. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 4. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | 5 day TAT |
| 6. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Common Parameter/Container - Preservation Key

#1 Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| Relinquished By: | On Ice? (Y / N) | Date | Time | Received by: | Date | Time |
|---|---|---|---|---|---|---|
| | Y | 6/25/13 | 1200 | | 6/25/13 | 12:10 |
| Relinquished By: | On Ice? (Y / N) | Date | Time | Received by: | Date | Time |
| | Y | 6/25/13 | 16:30 | AA / Pace | 6/26/13 | 9:20 |

Samples Shipped VIA: ☐ Air Freight ☒ Federal Express ☐ Sampler ☐ Other: Priority overnight (courier @ Pace 2 Work)

Air Bill Number:

T = 3.1

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator



H:RLG\STDFORMS\Chain Of Custody Form 2009 RLG Rev. 09\01\09

| Pace Analytical | Document Name: **Sample Condition Upon Receipt Form** | Document Revised: 28Jan2013 Page 1 of 1 |
|---|---|---|
| | Document No.: **F-MN-L-213-rev.06** | Issuing Authority: Pace Minnesota Quality Office |

**Sample Condition Upon Receipt**

**Client Name:** BARR

**Project #:** WO# : 10233409

10233409

**Courier:** ☑ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other:_____

**Tracking Number:** 9470855 1500 1164

**Custody Seal on Cooler/Box Present?** ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No

**Optional:** Proj. Due Date: Proj. Name:

**Packing Material:** ☑ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other:_____ **Temp Blank?** ☑ Yes ☐ No

**Thermom. Used:** ☑ B88A912167504 ☐ 80512447 ☐ 72337080 **Type of Ice:** ☑ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

**Cooler Temp Read (°C):** 3.1 **Cooler Temp Corrected (°C):** 3.1 **Biological Tissue Frozen?** ☐ Yes ☐ No

Temp should be above freezing to 6°C **Correction Factor:** true **Date and Initials of Person Examining Contents:** AA 6/26/13

| | | | | **Comments:** |
|---|---|---|---|---|
| Chain of Custody Present? | ☑ Yes | ☐ No | ☐ N/A | 1. |
| Chain of Custody Filled Out? | ☑ Yes | ☐ No | ☐ N/A | 2. |
| Chain of Custody Relinquished? | ☑ Yes | ☐ No | ☐ N/A | 3. |
| Sampler Name and/or Signature on COC? | ☑ Yes | ☐ No | ☐ N/A | 4. |
| Samples Arrived within Hold Time? | ☑ Yes | ☐ No | ☐ N/A | 5. |
| **Short Hold Time Analysis (<72 hr)?** | ☐ Yes | ☑ No | ☐ N/A | 6. |
| **Rush Turn Around Time Requested?** | ☑ Yes | ☐ No | ☐ N/A | 7. |
| Sufficient Volume? | ☑ Yes | ☐ No | ☐ N/A | 8. |
| Correct Containers Used? | ☑ Yes | ☐ No | ☐ N/A | 9. |
| -Pace Containers Used? | ☐ Yes | ☐ No | ☐ N/A | |
| Containers Intact? | ☑ Yes | ☐ No | ☐ N/A | 10. |
| Filtered Volume Received for Dissolved Tests? | ☐ Yes | ☐ No | ☑ N/A | 11. |
| Sample Labels Match COC? | ☐ Yes | ☐ No | ☐ N/A | 12. |
| -Includes Date/Time/ID/Analysis Matrix: SL | | | | |
| All containers needing acid/base preservation have been checked? Noncompliances are noted in 13. | ☐ Yes | ☑ No | ☐ N/A | 13. ☐ $HNO_3$ ☐ $H_2SO_4$ ☐ NaOH ☐ HCl Sample # |
| All containers needing preservation are found to be in compliance with EPA recommendation? ($HNO_3$, $H_2SO_4$, HCl<2; NaOH>12) | ☐ Yes | ☐ No | ☑ N/A | |
| Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water) | ☐ Yes | ☑ No | | Initial when completed:_____ Lot # of added preservative:_____ |
| Headspace in VOA Vials ( >6mm)? | ☐ Yes | ☐ No | ☑ N/A | 14. |
| Trip Blank Present? | ☐ Yes | ☑ No | ☐ N/A | 15. |
| Trip Blank Custody Seals Present? | ☐ Yes | ☐ No | ☑ N/A | |
| Pace Trip Blank Lot # (if purchased):_____ | | | | |

**CLIENT NOTIFICATION/RESOLUTION** **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

**Project Manager Review:** _____ **Date:** 6/26/13

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e out of hold, incorrect preservative, out of temp, incorrect containers)

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

WLSSD

Western Lake Superior Sanitary District

August 12, 2013

Karl F. Beaster
Enbridge
1320 Grand Avenue
Superior, WI 54880

Re: WLSSD Discharge Approval (Enbridge Superior Terminal)

Dear Mr. Beaster:

Based on the analytical information provided on **8/9/2013**, the WLSSD approves the discharge of **approximately 8000 gallons of contaminated water from an Enbridge Superior Terminal excavation** provided there is no visual sign of the petroleum oil, grease or other petroleum related products. This contaminated water is to be disposed of at the WLSSD's main treatment facility, which is located at 2626 Courtland in Duluth.

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

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

If there are any questions, please contact me at (218) 740-4815.

Sincerely,

Tim Tuominen
Chemist

LEGEND
Technical Services, Inc.
www.legend-group.com

88 Empire Drive
St Paul, MN 55103
Tel: 651-642-1150
Fax: 651-642-1239

August 02, 2013

Ms. Andrea Nord
Barr Engineering Co.
4700 W 77th St
Minneapolis, MN 55435

Work Order Number: 1303584
RE: 49161092

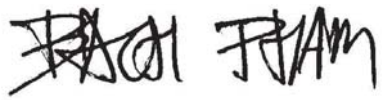
Enclosed are the results of analyses for samples received by the laboratory on 07/30/13. If you have any questions concerning this report, please feel free to contact me.

Results are not blank corrected unless noted within the report. Additionally, all QC results meet requirements unless noted.

All samples will be retained by Legend Technical Services, Inc., unless consumed in the analysis, at ambient conditions for 30 days from the date of this report and then discarded unless other arrangements are made. All samples were received in acceptable condition unless otherwise noted.

WI Accreditation #998022410

Prepared by,
LEGEND TECHNICAL SERVICES, INC



Bach Pham
Client Manager II
bpham@legend-group.com

Tyler Jones
Chemist I
tjones@legend-group.com

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Fax: 651-642-1239

| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 003 018 | Work Order #: 1303584 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/02/13 |

## ANALYTICAL REPORT FOR SAMPLES

| Sample ID | Laboratory ID | Matrix | Date Sampled | Date Received |
|---|---|---|---|---|
| Pipe Rack Water-1 | 1303584-01 | Water | 07/29/13 10:45 | 07/30/13 09:45 |

**Shipping Container Information**

**Default Cooler** Temperature (°C): 3.1

Received on ice: Yes
Temperature blank was present
Received on ice pack: No
Received on melt water: No
Ambient: No
Acceptable (IH/ISO only): No
Custody seals: No

**Case Narrative:**

The dry weight correction and dilution applies to the sample result, MDL, and RL.

Ethylbenzene was present in the method blank between the MDL and RL for the BTEX analysis.

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| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/02/13 |

## DRO/8015D
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **Pipe Rack Water-1 (1303584-01) Water** | **Sampled: 07/29/13 10:45** | | | **Received: 07/30/13 9:45** | | | | | | |
| **Diesel Range Organics** | **750** | 100 | 28 | ug/L | 1 | B3G3104 | 07/31/13 | 07/31/13 | WI(95) DRO | |
| *Surrogate: Triacontane (C-30)* | *86.6* | | | *70-130 %* | | " | " | " | " | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 003 018 | Work Order #: 1303584 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/02/13 |

## WI(95) GRO/8015D
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|---|---|---|---|---|---|---|---|---|---|---|
| **Pipe Rack Water-1 (1303584-01) Water** | **Sampled: 07/29/13 10:45** | | | **Received: 07/30/13 9:45** | | | | | | |
| Benzene | <0.13 | 1.0 | 0.13 | ug/L | 1 | B3G3106 | 07/31/13 | 07/31/13 | WI(95) GRO | |
| **Ethylbenzene** | **0.30** | 1.0 | 0.022 | ug/L | 1 | " | " | " | " | B-01, J |
| Gasoline range organics | <8.5 | 100 | 8.5 | ug/L | 1 | " | " | " | " | |
| Toluene | <0.15 | 1.0 | 0.15 | ug/L | 1 | " | " | " | " | |
| Xylenes (total) | <0.41 | 3.0 | 0.41 | ug/L | 1 | " | " | " | " | |
| *Surrogate: 4-Fluorochlorobenzene* | *104* | | | *80-150 %* | | " | " | " | " | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 003 018 | Work Order #: 1303584 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/02/13 |

## DRO/8015D - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B3G3104 - EPA 3510C (Sep Funnel)** | | | | | | | | | | | |
| **Blank (B3G3104-BLK1)** | | | | | Prepared & Analyzed: 07/31/13 | | | | | | |
| Diesel Range Organics | < 28 | 100 | 28 | ug/L | | | | | | | |
| *Surrogate: Triacontane (C-30)* | *373* | | | *ug/L* | *400* | | *93.3* | *70-130* | | | |
| **LCS (B3G3104-BS1)** | | | | | Prepared & Analyzed: 07/31/13 | | | | | | |
| Diesel Range Organics | 1520 | 100 | 28 | ug/L | 1600 | | 95.2 | 75-115 | | | |
| *Surrogate: Triacontane (C-30)* | *373* | | | *ug/L* | *400* | | *93.2* | *70-130* | | | |
| **LCS Dup (B3G3104-BSD1)** | | | | | Prepared & Analyzed: 07/31/13 | | | | | | |
| Diesel Range Organics | 1540 | 100 | 28 | ug/L | 1600 | | 96.2 | 75-115 | 1.02 | 20 | |
| *Surrogate: Triacontane (C-30)* | *372* | | | *ug/L* | *400* | | *93.1* | *70-130* | | | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 003 018 | Work Order #: 1303584 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/02/13 |

## WI(95) GRO/8015D - Quality Control
## Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Batch B3G3106 - EPA 5030 Water (Purge and Trap)** | | | | | | | | | | | |
| **Blank (B3G3106-BLK1)** | | | | | Prepared & Analyzed: 07/31/13 | | | | | | |
| Benzene | < 0.13 | 1.0 | 0.13 | ug/L | | | | | | | |
| Ethylbenzene | 0.278 | 1.0 | 0.022 | ug/L | | | | | | | B-02, J |
| Gasoline range organics | < 8.5 | 100 | 8.5 | ug/L | | | | | | | |
| Toluene | < 0.15 | 1.0 | 0.15 | ug/L | | | | | | | |
| Xylenes (total) | < 0.41 | 3.0 | 0.41 | ug/L | | | | | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *26.1* | | | *ug/L* | *25.0* | | *105* | *80-150* | | | |
| **LCS (B3G3106-BS1)** | | | | | Prepared & Analyzed: 07/31/13 | | | | | | |
| Benzene | 97.5 | 1.0 | 0.13 | ug/L | 100 | | 97.5 | 80-120 | | | |
| Ethylbenzene | 97.0 | 1.0 | 0.022 | ug/L | 100 | | 97.0 | 80-120 | | | |
| Gasoline range organics | 962 | 100 | 8.5 | ug/L | 1000 | | 96.2 | 80-120 | | | |
| Toluene | 97.2 | 1.0 | 0.15 | ug/L | 100 | | 97.2 | 80-120 | | | |
| Xylenes (total) | 288 | 3.0 | 0.41 | ug/L | 300 | | 96.1 | 80-120 | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *25.0* | | | *ug/L* | *25.0* | | *99.9* | *80-150* | | | |
| **LCS Dup (B3G3106-BSD1)** | | | | | Prepared & Analyzed: 07/31/13 | | | | | | |
| Benzene | 100 | 1.0 | 0.13 | ug/L | 100 | | 100 | 80-120 | 2.71 | 20 | |
| Ethylbenzene | 101 | 1.0 | 0.022 | ug/L | 100 | | 101 | 80-120 | 3.79 | 20 | |
| Gasoline range organics | 973 | 100 | 8.5 | ug/L | 1000 | | 97.3 | 80-120 | 1.12 | 20 | |
| Toluene | 101 | 1.0 | 0.15 | ug/L | 100 | | 101 | 80-120 | 3.72 | 20 | |
| Xylenes (total) | 297 | 3.0 | 0.41 | ug/L | 300 | | 99.0 | 80-120 | 2.96 | 20 | |
| *Surrogate: 4-Fluorochlorobenzene* | *25.7* | | | *ug/L* | *25.0* | | *103* | *80-150* | | | |
| **Duplicate (B3G3106-DUP1)** | | **Source: 1303572-01** | | | Prepared & Analyzed: 07/31/13 | | | | | | |
| Gasoline range organics | 70.4 | 100 | 8.5 | ug/L | | <100 | | | NA | 20 | J |
| *Surrogate: 4-Fluorochlorobenzene* | *25.5* | | | *ug/L* | *25.0* | | *102* | *80-150* | | | |
| **Matrix Spike (B3G3106-MS1)** | | **Source: 1303584-01** | | | Prepared & Analyzed: 07/31/13 | | | | | | |
| Benzene | 101 | 1.0 | 0.13 | ug/L | 100 | <1.0 | 101 | 80-120 | | | |
| Ethylbenzene | 102 | 1.0 | 0.022 | ug/L | 100 | <1.0 | 101 | 80-120 | | | |
| Toluene | 102 | 1.0 | 0.15 | ug/L | 100 | <1.0 | 102 | 80-120 | | | |
| Xylenes (total) | 300 | 3.0 | 0.41 | ug/L | 300 | <3.0 | 100 | 80-120 | | | |
| *Surrogate: 4-Fluorochlorobenzene* | *25.8* | | | *ug/L* | *25.0* | | *103* | *80-150* | | | |

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| | | | |
|---|---|---|---|
| Barr Engineering Co. | Project: | 49161092 | |
| 4700 W 77th St | Project Number: | 49161092 003 018 | Work Order #: 1303584 |
| Minneapolis, MN 55435 | Project Manager: | Ms. Andrea Nord | Date Reported: 08/02/13 |

## Notes and Definitions

| | |
|---|---|
| J | Parameter was present between the MDL and RL and should be considered an estimated value |
| B-02 | Target analyte was present in the method blank between the MDL and RL. |
| B-01 | Analyte was present in the method blank. Sample result is less than or equal to 10 times the blank concentration. |
| < | Less than value listed |
| dry | Sample results reported on a dry weight basis |
| NA | Not applicable. The %RPD is not calculated from values less than the reporting limit. |
| MDL | Method Detection Limit |
| RL | Reporting Limit |
| RPD | Relative Percent Difference |
| LCS | Laboratory Control Spike = Blank Spike (BS) = Laboratory Fortified Blank (LFB) |
| MS | Matrix Spike = Laboratory Fortified Matrix (LFM) |

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# Chain of Custody

BARR
4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

1303584

Project Number: 49/61092 003 018

Project Name: Enbridge Pipe Rack

Sample Origination State: WI (use two letter postal state abbreviation)

COC Number: № 40588

COC 1 of 1

Project Manager: REE

Project QC Contact: AAN

Sampled by: REE

Laboratory: Legend

| Location | Start Depth | Stop Depth | Depth Unit (m,ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix: Water | Matrix: Soil | Type: Grab | Type: Comp. | Type: QC | Number of Containers/Preservative: Water (VOCs (HCl) #1; SVOCs (unpreserved) #2; Dissolved Metals ($HNO_3$); Total Metals ($HNO_3$); General (unpreserved) #3; Diesel Range Organics (HCl); Nutrients ($H_2SO_4$) #4) | Number of Containers/Preservative: Soil (VOCs (tared MeOH) #1; GRO, BTEX (tared MeOH) #1; DRO (tared unpreserved); Metals (unpreserved); SVOCs (unpreserved) #2; % Solids (plastic vial, unpres.)) | Total Number Of Containers | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. Pipe Rack Water - 1 | — | — | | 7/29/13 | 1045 | X | | X | | | | | | DRO, BTEX, GRO |
| 2. | | | | | | | | | | | | | | TAT: By 8/2/13 |
| 3. | | | | | | | | | | | | | | |
| 4. | | | | | | | | | | | | | | |
| 5. | | | | | | | | | | | | | | |
| 6. | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | |

D/I
pH

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
#2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
#3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
#4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| | | | | | | |
|---|---|---|---|---|---|---|
| Relinquished By: | On Ice? Y / N | Date | Time | Received by: | Date | Time |
| Relinquished By: | On Ice? Y / N | Date | Time | Received by: | Date | Time |
| | | | | | 7/30/13 | 9:45 |

Samples Shipped VIA: ☐ Air Freight ☐ Federal Express ☐ Sampler ☐ Other: _____

Air Bill Number: 3/0

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

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