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BARR

Technical Memorandum

To: Karl Beaster, Enbridge Energy
From: Ryan Erickson
Subject: Superior Terminal Tank 5 Basin Response - Historical Crude Oil Impacted Soil
Date: January 22, 2014
WDNR BRRTS: 02-16-558993
Barr Project: 49161092

This technical memorandum summarizes the field screening, analytical sampling and waste management assistance conducted by Barr Engineering (Barr) at the request of Enbridge Energy (Enbridge) in response to the discovery of historical, crude oil impacted soil during the construction of Tank 5 stairway footings at the Enbridge Superior Terminal in Superior, Wisconsin (Figure 1) in November of 2011.

Background and Response Activities

In November of 2011, Enbridge constructed a stairway on the east side of Tank 5 at the Superior Terminal (Figure 2). The purpose of the stairway was to improve access to Tank 5 infrastructure. As part of the stairway construction activities, soil was excavated in order to install buried footings.

On November 16, 2011, an Enbridge contractor discovered crude oil impacted soil on the east side of Tank 5 in the stairway footing excavation (Photo 1). Enbridge Environment was notified by the contractor when crude oil impacts were encountered at the site.

Excavation activities were conducted by Pipe Line Maintenance (PLM) personnel on November 16, 2011 to identify the source of the crude oil and remove the crude oil impacted soil. This initial response and remediation excavation was approximately 60-feet long by 30-feet wide and 8 to 10 feet deep (Photo 2; Figure 2). An abandoned 8-inch Murphy Oil pipeline located within the excavations was identified as the source of the discovered crude oil impacts. The abandoned pipeline was approximately 6-feet below ground surface (bgs) and was located between the Tank 5 and Tank 6 containment basins. It appeared that the abandoned pipeline had been cut, plugged with clay and buried without draining the product present within it. PLM personnel removed remaining oil from the pipeline using a vacuum truck and the pipeline was sealed with a welded cap. Crude oil impacted soil that was identified by the PLM, based on visual

and olfactory evidence, was excavated and stockpiled in a bermed and lined containment area in the Superior Terminal Soil Management Area (SMA) (Figure 2) until it could be characterized for disposal.

On November 17, 2011, Enbridge requested that Barr complete the following activities at the Tank 5 site:

- assess the environmental site conditions
- identify and segregate excavated crude oil impacted soil from unimpacted soil
- assist with the off-site disposal coordination and documentation of contaminated soil
- document the residual crude-oil impacts left in place, if applicable
- identify whether the impacts could be attributed to a reported historical release

During the week of November 28, 2011, the PLM directed additional excavation activities to remove the abandoned Murphy Oil pipeline. No additional crude oil impacts were encountered outside the initial remedial excavation footprint (Figure 2).

Barr checked the Wisconsin Department of Natural Resources (WDNR) Bureau for Remediation and Redevelopment Tracking System (BRRTS) database and no reported releases were identified near the Tank 5 excavation. Therefore, Enbridge submitted a Notification for Hazardous Substance Discharge to the WDNR on June 19, 2012 and BRRTS activity number 02-16-558993 was assigned (Attachment A).

Field Methods

Barr was onsite on November 17, 2011 to conduct site assessment and waste characterization activities. Barr field screened the PLM's remedial excavation extents for the presence of organic vapors using a photoionization detector (PID) and headspace procedures and documented other potential indicators of crude oil impacts such as odor, discoloration and sheen (Attachment B). Barr collected two soil samples from the base of the excavation (Tank 5-B-1 and Tank 5-B-2) and three soil samples from the excavation sidewalls (Tank 5-S-1, Tank 5-S-2, and Tank 5-S-3) to document residual soil impacts (Figure 2). The soil samples were submitted to Pace Analytical Services in Minneapolis, Minnesota for analysis of diesel range organics (DRO) and benzene, toluene, ethyl benzene and xylenes (BTEX). The Tank 5-S-3 sample was also analyzed for polycyclic aromatic hydrocarbons (PAH). The analytical results are summarized in Table 1 and the full laboratory analytical report is provided in Attachment C.

Geoprobe boring TK5-SB-1 was advanced on June 15, 2012 approximately twenty feet to the east of the remedial excavation footprint (Figure 2) to define the extents of the crude oil impacts identified in sidewall sample Tank 5-S-3. The boring was advanced to fifteen feet bgs and the soil recovered from the

boring was field screened and sampled for laboratory analysis of DRO and petroleum volatile organic compounds (Attachments B and C). Laboratory analytical results for soil sample are summarized in Table 1.

Results

Field screening and analytical sampling activities conducted at the remedial excavation and the Geoprobe boring are described below. Analytical results from each sample location were input into the WDNR Web Calculator to compare analyte detections to groundwater residual contaminant levels (RCL) and industrial direct contact RCL and determine whether the soil passes the Cumulative Hazard Index criteria described in WDNR guidance document PUB-RR-890 (Table 1).

Remedial Excavation

Field screening samples were collected from the excavation sidewalls and bottom. Headspace detections were between 0.2 parts per million (ppm) and 224 ppm in the eastern corner. No petroleum odor was detected and no visual staining was observed. Additional remedial excavation activity was limited due to the presence of Tank 5 infrastructure.

Analyte concentrations in base of excavation samples Tank 5-B-1 and Tank 5-B-2 and sidewall samples Tank 5-S-1and Tank 5-S-2 were below the WDNR groundwater RCL and the industrial direct contact RCL and passed the Cumulative Hazard Index. Sidewall sample Tank 5-S-3 had concentrations that exceeded the groundwater RCL for benzene (1.4 mg/kg), ethyl benzene (1.4 mg/kg) and xylenes (4.2 mg/kg) but were below the industrial direct contact RCL and passed the Cumulative Hazard Index criteria.

Geoprobe Boring

Soil recovered from Geoprobe boring TK 5-SB-1 was field screened for headspace, odor, sheen and discoloration. Headspace detections were between 0.5 ppm and 0.9 ppm and no other evidence of crude oil impacts was observed.

Analyte concentrations in sample TK5-SB-1, collected from 2 to 3 feet bgs, were below the groundwater RCL and the industrial direct contact RCL and passed the Cumulative Hazard Index.

Discussion

Analyte concentrations detected in the excavation and Geoprobe soil samples were below the groundwater RCL and the industrial direct contact RCL and passed the Cumulative Hazard Index criteria except for sidewall sample Tank 5-S-3, which exceeded the groundwater RCL for benzene, ethyl benzene and xylenes. Additional remedial excavation was limited by the presence of Tank 5 infrastructure. Crude oil impacted soil with free-product, a petroleum odor or petroleum staining was excavated by Enbridge, the abandoned pipeline was removed and the excavation was backfilled with clean fill.

Waste Disposal Coordination and Documentation

Barr collected two analytical waste characterization samples from the crude oil impacted soil stockpile (Tank 5-Stockpile-1 and Tank 5-Stockpile-2). The samples were submitted Pace Analytical Services for analysis of DRO and BTEX. A waste profile application with the laboratory results was submitted to Waste Management Voyageur Landfill near Canyon, Minnesota, and the soil was accepted under waste profile #102881MN. A total of 203.12 tons of crude oil impacted soil was hauled to the landfill in December of 2011. The waste disposal documentation is included in Attachment D.

Conclusions and Recommendations

Crude oil impacted soil from a buried abandoned pipeline was encountered during Tank 5 infrastructure construction. The crude oil impacted soil associated with the pipeline was excavated, to the extent possible, and disposed of at an approved landfill facility. The abandoned pipeline was removed and the excavation was backfilled with clean fill. Analyte concentrations in the excavation and Geoprobe soil samples were below the industrial direct contact RCL and passed the Cumulative Hazard Index criteria. The clean backfill 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 this historical release 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:

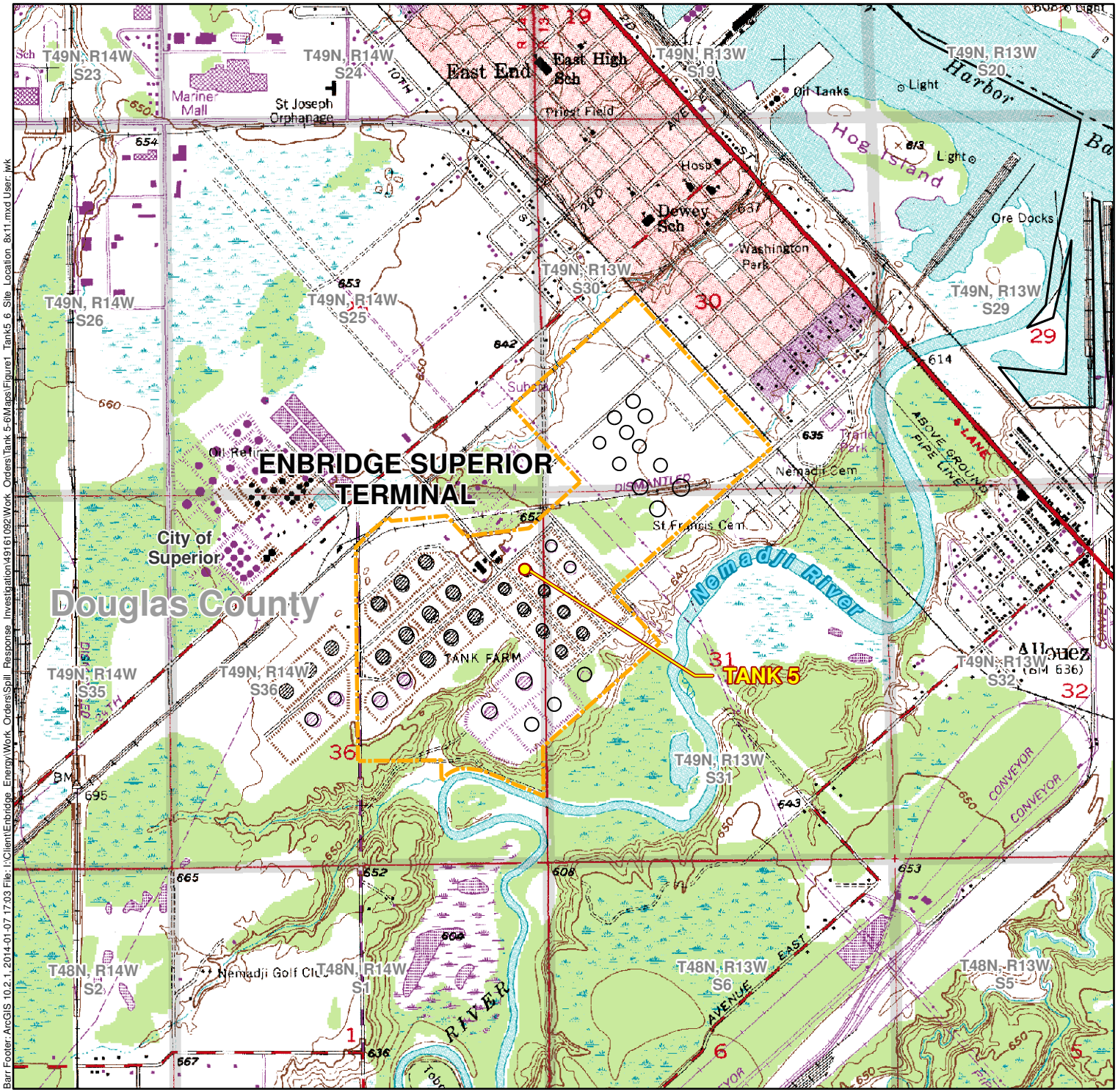
Photos	1 and 2
Figure 1	Site Location Map
Figure 2	Site Layout Map
Table 1	Soil Analytical Data Summary
Attachment A	WDNR Notification for Hazardous Substance Discharge and Communications
Attachment B	Enbridge Site Investigation Field Sampling and Screening Log and Geoprobe Boring Field Notes
Attachment C	Pace Analytical Laboratory Reports for Excavation Soil Samples
Attachment D	Waste Disposal Documents

Photos:

Photo 1

Photo 2

Photo 1: Tank 5 remedial excavation facing south. Tank 5 is on the right.
Photo 2: Final remedial excavation extent facing east. The abandoned Murphy Oil pipe is visible at bottom of excavation.



Tank 5

Terminal Property Boundary

N

0 2,000 4,000

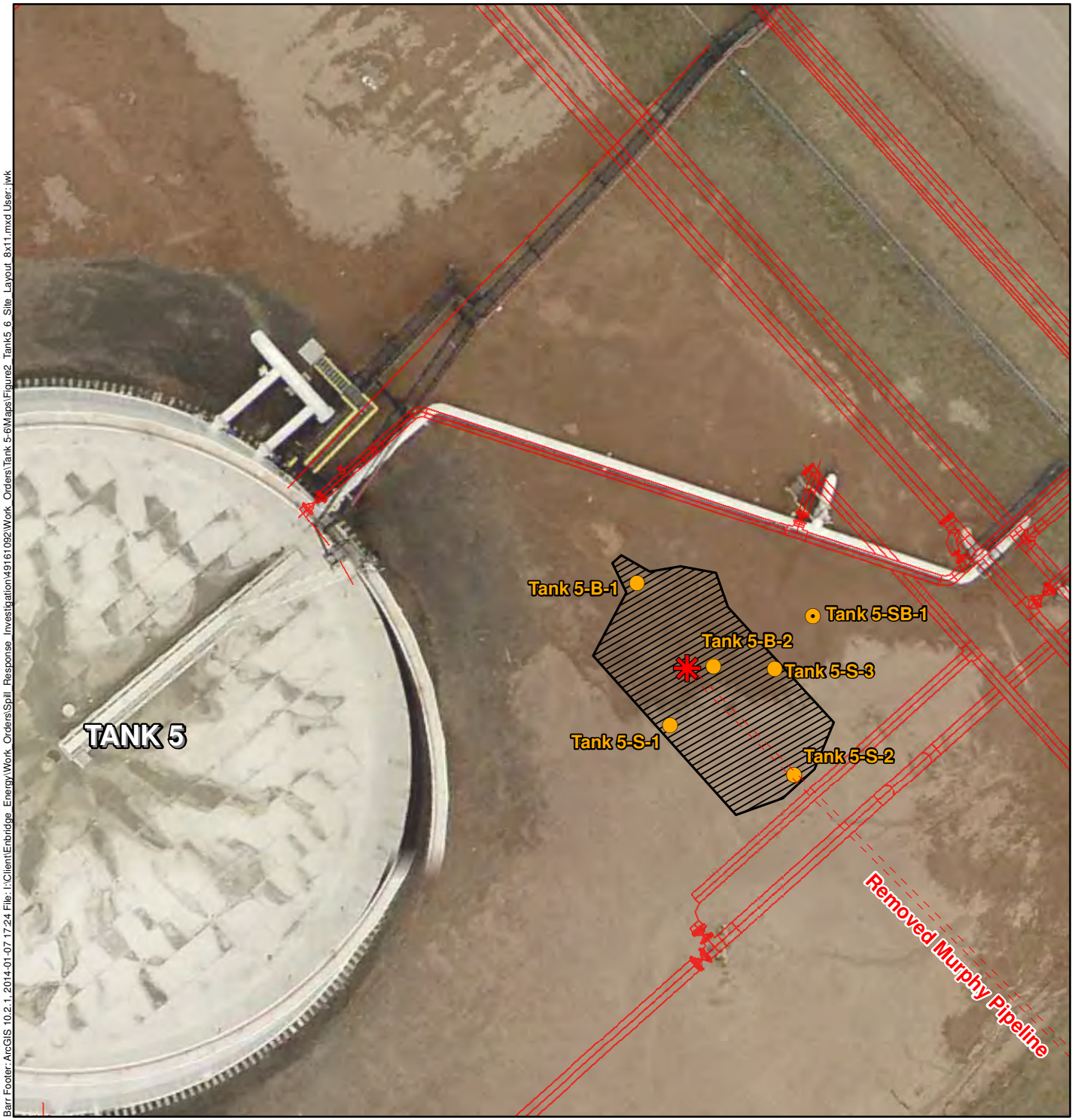
Feet

1 Inch = 2,000 Feet

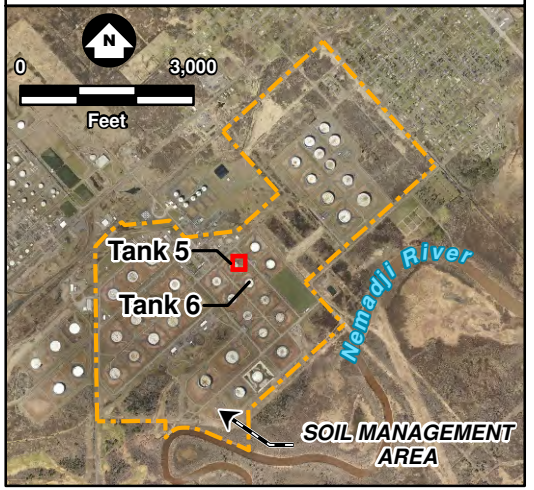
Figure 1

SITE LOCATION MAP
TANK 5 INVESTIGATION
SUPERIOR TERMINAL
Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE



ENBRIDGE SUPERIOR TERMINAL



- Approximate Release Location
- Sample Locations
- Geoprobe Boring Location
- Excavation Extent
- Pipeline Infrastructure
- Terminal Property Boundary

N

0 30 60

Feet

1 Inch = 30 Feet

Douglas County Imagery Circa May, 2013

Figure 2

SITE LAYOUT MAP
TANK 5 INVESTIGATION
SUPERIOR TERMINAL

Enbridge Energy, L.P.
Superior, Wisconsin

ENBRIDGE

Table 1
Soil Analytical Data Summary
Tank 5 Basin Historical Pipeline
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	Chrysene	Dibenz(a,h) anthracene
	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			0.0725	
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	211	0.211
Location	**Date**	**Depth (ft)**																		
TANK 5-B-1	11/17/2011	3	18.0 %	< 0.061	< 0.061	< 0.061	< 0.18	--	--	< 11.1	--	--	--	--	--	--	--	--	--	--
TANK 5-B-2	11/17/2011	10	23.8 %	< 0.071	< 0.071	< 0.071	< 0.21	--	--	< 11.5	--	--	--	--	--	--	--	--	--	--
TANK 5-S-1	11/17/2011	2	21.1 %	< 0.066	< 0.066	< 0.066	< 0.20	--	--	< 12.8	--	--	--	--	--	--	--	--	--	--
TANK 5-S-2	11/17/2011	3	19.8 %	< 0.064	< 0.064	< 0.064	< 0.19	--	--	19.6	--	--	--	--	--	--	--	--	--	--
TANK 5-S-3	11/17/2011	2	18.2 %	**1.4**	**1.4**	0.51	**4.2**	**--**	**--**	< 11.5	< 0.0121	< 0.0121	< 0.0121	< 0.0121	< 0.0121	< 0.0121	< 0.0121	< 0.0121	< 0.0121	< 0.0121
TK5-SB-1	6/15/2012	2-3	24.1 %	< 0.065	< 0.065	< 0.065	< 0.19	< 0.065	< 0.065	< 11.0	--	--	--	--	--	--	--	--	--	--

[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).

Table 1
Soil Analytical Data Summary
Tank 5 Basin Historical Pipeline
Enbridge Energy Terminal - Superior, Wisconsin
Units, mg/kg (unless otherwise noted)

		Parameter	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**	44.4089	7.4074		0.3294		27.2362				
Industrial Direct Contact RCL	05/01/2012	No Exceed	22000	22000	2.11	26	115	16500	0	1.0	0.00001	Pass
Location	**Date**	**Depth (ft)**										
TANK 5-B-1	11/17/2011	3	--	--	--	--	--	--	0	0.0001	9.9E-09	Pass
TANK 5-B-2	11/17/2011	10	--	--	--	--	--	--	0	0.0002	1.2E-08	Pass
TANK 5-S-1	11/17/2011	2	--	--	--	--	--	--	0	0.0002	1.1E-08	Pass
TANK 5-S-2	11/17/2011	3	--	--	--	--	--	--	0	0.0002	1.0E-08	Pass
TANK 5-S-3	11/17/2011	2	< 0.0121	< 0.0121	< 0.0121	< 0.0121	< 0.0121	< 0.0121	0	0.0034	3.6E-07	Pass
TK5-SB-1	6/15/2012	2-3	--	--	--	--	--	--	0	0.0003	1.1E-08	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).

Attachment A

WDNR Notification for Hazardous Substance Discharge and Communications

State of Wisconsin
Department of Natural Resources
dnr.wi.gov

Notification For Hazardous Substance Discharge (Non-Emergency Only)

Form 4400-225 (05/12) Page 1 of 2

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

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

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

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

- ☐ Underground Petroleum Storage Tank System (additional information may be required for Item 6 below)
- ☐ Aboveground Petroleum Storage Tank System
- ☐ Dry Cleaner Facility
- ☒ Other - Describe: Enbridge Superior Terminal - Historical Piping Contamination near Tank 5

ATTN DNR: **R & R Program Associate** Date DNR Notified: 05/31/2012

1. Discharge Reported By

Name	Firm	Phone No. (include area code)
Karl Beaster	Enbridge Energy	(715) 398-4754

Mailing Address	Email Address
1320 Grand Ave., Superior, WI 54880	karl.beaster@enbridge.com

2. Site Information

Name of site at which discharge occurred. Include local name of site/business, not responsible party name, unless a residence/vacant property. Enbridge Superior Terminal - Tank 5

Location: Include street address, not PO Box. If no street address, describe as precisely as possible, i.e., 1/4 mile NW of CTHs 60 & 123 on E side of CTH 60. 2800 East 21st Street, Superior, WI 54880

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

County:	Legal Description:	WTM:
Douglas	SW 1/4 NW 1/4 Sec 36 Tn 49N Range 13 ○ E ◉ W	X 362845 Y 692514

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

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

Enbridge Energy

☐ Reported in compliance with s. 292.11(2), Wis. Stats., by a local government exempt from liability under s. 292.11(9)(e), Wis. Stats. For more information see http://dnr.wi.gov/org/aw/rr/lgu/liability.htm.

Contact Person Name (if different)	Phone Number	Email Address
Karl Beaster	(715) 398-4757	karl.beaster@enbridge.com

Mailing Address	City	State	ZIP Code
1320 Grand Ave., Superior, WI 54880	Superior	WI	54880

Property owner if Different From RP: Business or owner name that is responsible for cleanup. If more than one, list all. Attach additional pages as necessary.

Contact Person Name (if different)	Phone Number	Email Address

Mailing Address	City	State	ZIP Code

(continued)

State of Wisconsin
Department of Natural Resources
dnr.wi.gov

Notification For Hazardous Substance Discharge (Non-Emergency Only)

4. Hazardous Substance Information

Identify hazardous substance discharged (check all that apply):

- [x] VOC's
- [ ] PAH's
- [ ] Metals (specify): _____
  - [ ] Arsenic
  - [ ] Chromium
  - [ ] Cyanide
  - [ ] Lead
  - [ ] PCB's
- [ ] Diesel
- [ ] Fuel Oil
- [ ] Gasoline
- [ ] Hydraulic Oil
- [ ] Jet Fuel
- [ ] Mineral Oil
- [ ] Waste Oil
- [ ] Petroleum-Unknown Type
- [ ] PERC (Dry Cleaners)
- [ ] RCRA Hazardous Waste
- [ ] Leachate
- [ ] Fertilizer
- [ ] Pesticide/Herbicide/Insecticide(s)
- [x] Other (specify): Crude oil
- [ ] Unknown

5. Impacts to the Environment Information

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

- ___ Air Contamination
- ___ Co-Contamination (Petroleum & Non-Petroleum)
- ___ Contamination Within 1 Meter of Bedrock
- ___ Contaminated Private Well
- ___ Contaminated Public Well
- ___ Contamination in Fractured Bedrock
- ___ Sanitary Sewer Contamination
- ___ Contamination in Right of Way
- ___ Fire Explosion Threat
- K Free Product
- P Groundwater Contamination
- ___ Off-Site Contamination
- ___ Other (specify): _____
- K Soil Contamination
- ___ Storm Sewer Contamination
- ___ Surface Water Contamination
- ___ Within 100 ft of Private Well
- ___ Within 1000 ft of Public Well

Contamination was discovered as a result of:

- [ ] Tank closure assessment — Date _____
- [ ] Site assessment — Date _____
- [x] Other - Describe: Tank maintenance construction — Date 11/16/2011

Lab results: [ ] Lab results will be faxed upon receipt [ ] Lab results are attached

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

Enbridge identified an abandoned 8-inch pipeline during construction activities. Hydrocarbons in the pipe were removed with a vacuum truck and the surrounding contaminated soil was excavated. The pipeline was cut, capped, and eventually removed.

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

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

- [ ] Does not apply.

Source

- [ ] Tank
- [x] Piping
- [ ] Dispenser
- [ ] Submersible Turbine Pump
- [ ] Delivery Problem
- [ ] Other (specify): _____

Cause

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

Contact information to report non-emergency releases in DNR's five regions are as follows:

Northeast Region (FAX: 920-662-5197); Attention -- R&R Program Associate: DNRRRNER@wisconsin.gov

Brown, Calumet, Door, Fond du Lac **(except City of Waupun - see South Central Region)**, Green Lake, Kewaunee, Manitowoc, Marinette, Marquette, Menominee, Oconto, Outagamie, Shawano, Sheboygan, Waupaca, Waushara, Winnebago counties

Northern Region (FAX: 715-623-6773); Attention -- R&R Program Associate: DNRRRNOR@wisconsin.gov

Ashland, Barron, Bayfield, Burnett, Douglas, Forest, Florence, Iron, Langlade, Lincoln, Oneida, Polk, Price, Rusk, Sawyer, Taylor, Vilas, Washburn counties

South Central Region (FAX: 608-273-5610); Attention -- R&R Program Associate: DNRRRSCR@wisconsin.gov

Columbia, Dane, Dodge, Fond du Lac **(City of Waupun only)**, Grant, Green, Iowa, Jefferson, Lafayette, Richland, Rock, Sauk, Walworth counties

Southeast Region (FAX: 414-263-8550); Attention -- R&R Program Associate: DNRRRSER@wisconsin.gov

Kenosha, Milwaukee, Ozaukee, Racine, Washington, Waukesha counties

West Central Region (FAX: 715-839-6076); Attention -- R&R Program Associate: DNRRRWCR@wisconsin.gov

Adams, Buffalo, Chippewa, Clark, Crawford, Dunn, Eau Claire, Jackson, Juneau, LaCrosse, Marathon, Monroe, Pepin, Pierce, Portage, St. Croix, Trempealeau, Vernon, Wood counties

State of Wisconsin
DEPARTMENT OF NATURAL RESOURCES
Superior Service Center
1701 N. 4th Street
Superior WI 54880

COPY

Scott Walker, Governor
Cathy Stepp, Secretary
Telephone 608-266-2621
Toll Free 1-888-936-7463
TTY Access via relay - 711

WISCONSIN DEPT. OF NATURAL RESOURCES

December 11, 2012

Karl Beaster
Enbridge Energy
1320 Grand Ave
Superior WI 54880

Subject: Reported Contamination at Enbridge Energy – Tank 5, Superior, WI
WDNR BRRTS Activity # 02-16-558993
WDNR FID # 816010580

Dear Mr. Beaster:

On June 19, 2012, Enbridge Energy notified the Wisconsin Department of Natural Resources ("WDNR") that crude oil had been detected at the site described above.

Based on the information that has been submitted to the WDNR regarding this site, we believe you are responsible for investigating and restoring the environment at the above-described site under Section 292.11, Wisconsin Statutes, known as the hazardous substances spills law.

This letter describes the legal responsibilities of a person who is responsible under section 292.11, Wis. Stats., explains what you need to do to investigate and clean up the contamination.

Legal Responsibilities:
Your legal responsibilities are defined both in statute and in administrative codes. The hazardous substances spill law, Section 292.11 (3) Wisconsin Statutes, states:

- RESPONSIBILITY. A person who possesses or controls a hazardous substance which is discharged or who causes the discharge of a hazardous substance shall take the actions necessary to restore the environment to the extent practicable and minimize the harmful effects from the discharge to the air, lands, or waters of the state.

Wisconsin Administrative Code chapters NR 700 through NR 749 establish requirements for emergency and interim actions, public information, site investigations, design and operation of remedial action systems, and case closure. Wisconsin Administrative Code chapter NR 140 establishes groundwater standards for contaminants that reach groundwater.

Steps to Take:
The longer contamination is left in the environment, the farther it can spread and the more it may cost to clean up. Quick action may lessen damage to your property and neighboring properties and reduce your

costs in investigating and cleaning up the contamination. To ensure that your cleanup complies with Wisconsin's laws and administrative codes, you should hire a professional environmental consultant who understands what needs to be done. These are the first steps to take:

1. Within the next **30 days,** by January 15, 2013, you should submit written verification (such as a letter from the consultant) that you have hired an environmental consultant. If you do not take action within this time frame, the WDNR may initiate enforcement action against you.

2. Within the next **60 days,** by February 15, 2013, your consultant should submit a work plan and schedule for the investigation. The consultant must comply with the requirements in the NR 700 Wis. Adm. Code rule series and should adhere to current WDNR technical guidance documents.

In addition, within 30 days of completion of the site investigation, your consultant should submit a Site Investigation Report to the WDNR or other agency with administrative authority.

Sites where discharges to the environment have been reported are entered into the Bureau for Remediation and Redevelopment Tracking System ("BRRTS"), a version of which appears on the WDNR's internet site. You may view the information related to your site at any time (http://**dnr.wi.gov/botw/**SetUpBasicSearchForm.do) and use the feedback system to alert us to any errors in the data.

If you want a formal written response from the department on a specific submittal, please be aware that a review fee is required in accordance with ch. NR 749, Wis. Adm. Code. If a fee is not submitted with your reports, you should proceed under the advice of your consultant to complete the site investigation and cleanup to maintain your compliance with the spills law and chapters NR 700 through NR 749. **Do not delay the investigation of your site by waiting for an agency response.** We have provided detailed technical guidance to environmental consultants. Your consultant is expected to know our technical procedures and administrative rules and should be able to answer your questions on meeting cleanup requirements.

All correspondence regarding this site should be sent to me at the Superior office. Unless otherwise requested, please send only one copy of plans and reports. In addition to the paper copy, an electronic copy may also be submitted. To speed processing, correspondence should reference the BRRTS and FID numbers (if assigned) shown at the top of this letter.

Site Investigation and Vapor Pathway Analysis

As you develop the site investigation work plan, we want to remind you to include an assessment of the vapor intrusion pathway. Chapter NR 716, Wisconsin Administrative Code outlines the requirements for investigation of contamination in the environment. Specifically, s. NR 716.11(3)(a) requires that the field investigation determine the "nature, degree and extent, both areal and vertical, of the hazardous substances or environmental pollution in all affected media". In addition, section NR 716.11(5) specifies that the field investigation include an evaluation of the "pathways for migration of the contamination, including drainage improvements, utility corridors, bedrock and permeable material or soil along which vapors, free product or contaminated water may flow".

You will need to include documentation with the Site Investigation Report that explains how the assessment was done. If the pathway is being ruled out, then the report needs to provide the appropriate justification for reaching this conclusion. If the pathway cannot be ruled out, then investigation and, if appropriate, remedial action must be taken to address the risk presented prior to submitting the site for closure. The WDNR has developed guidance to help responsible parties and their consultants comply with the requirements described above. The guidance includes a detailed explanation of how to assess the vapor intrusion pathway and provides criteria which identify when an investigation is necessary. The guidance is available at: http://dnr.wi.gov/files/PDF/pubs/rr/RR800.pdf.

Additional Information for Site Owners:
We encourage you to visit our website at http://dnr.wi.gov/topic/Brownfields/, where you can find information on selecting a consultant, financial assistance and understanding the cleanup process. You will also find information there about liability clarification letters, post-cleanup liability and more.

If you have questions, contact me at 715-392-3126 or via email at erin.endsley@wisconsin.gov for more information or visit the RR web site at the address above.

Thank you for your cooperation.

Sincerely,

Erin Endsley

Erin Endsley
Hydrogeologist
Remediation & Redevelopment Program

cc: Hans Wronka, Barr Engineering

Attachment B

Enbridge Site Investigation Field Sampling and Screening Log

and Geoprobe Boring Field Notes

SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Date: 11/17/2011

Location: *Facility or Milepost* Superior Terminal Tank 5 Historical Line

Sampler: REE

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

Background Headspace: 0.0 ppm

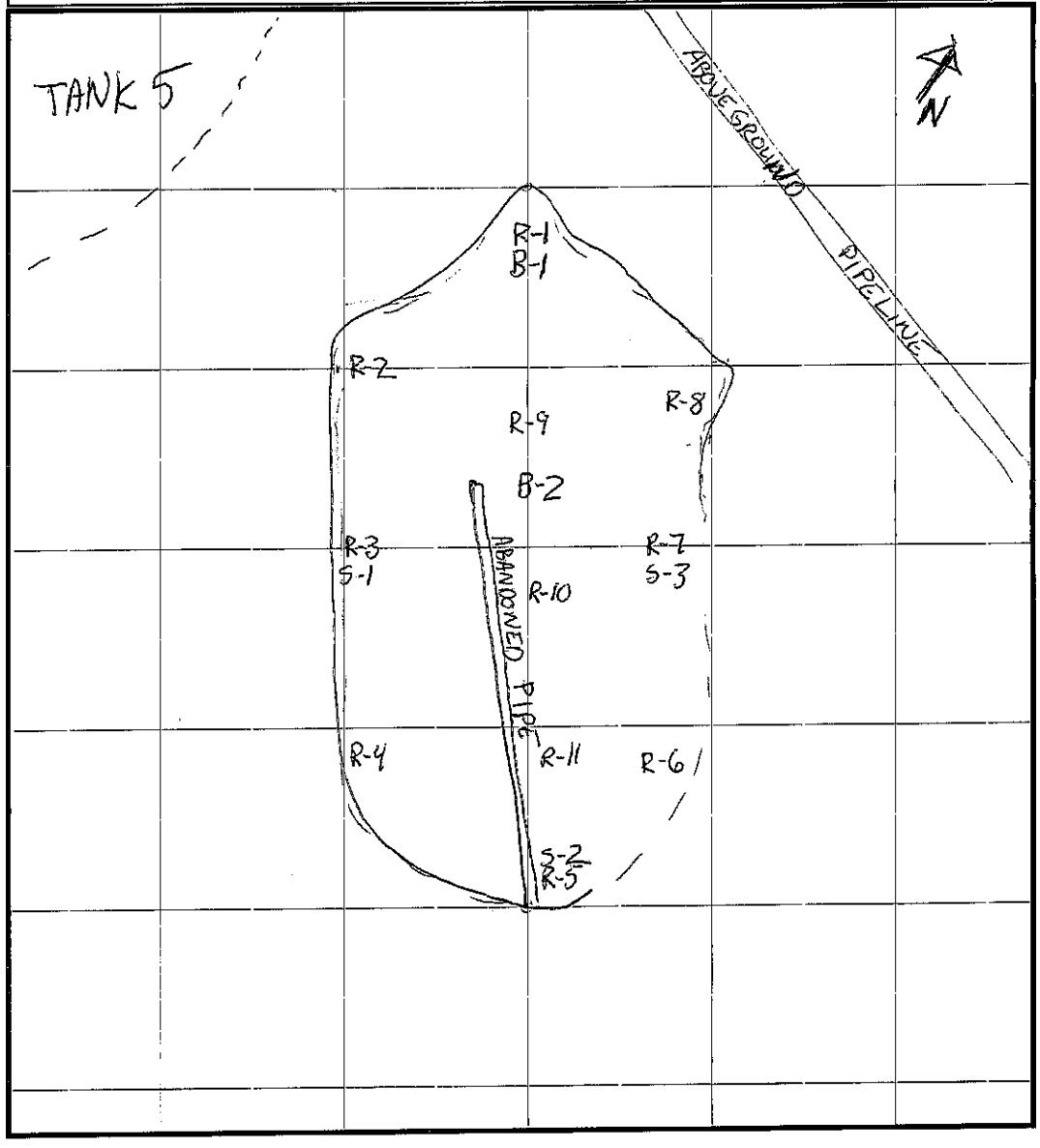
Calibration Time: 1430

Sample Nomenclature *(Location - sample type - #)*: Tank5 -

Soil Sample Types: R = Removed/*Screening 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	1430	CL	Reddish brown	N/-	0.7
R-2	2					-
R-3	2					12.0
R-4	2					-
R-5	3					0.2
R-6	2					-
R-7	2					224
R-8	2					-
R-9	8					-
R-10	8					69
R-11	8					-
ANALYTICAL SAMPLES						
B-1	3	1450	CL			-
B-2	10	1510				-
S-1	2	1455				-
S-2	3	1600				-
S-3	2	1505				-

SITE SKETCH: *north is top of page; excavation extent & depth, impacted area, sample locations, borings, wells, structures, utilities, natural features...* ***1 inch/grid = 15 FT***



TKS-SB-1

Run/Rec	Depth	% G/S/F	Od/st/sn	Color	ASTM
1/ 3'	0-5	0/0/100	N/N/N	4/4 10R	
2/ 5'	5-10	0/0/100	N/N/N	4/4 10R	
3/ 5'	10-15	0/0/100	〃	〃	

TKS-SB-1

Description	PID
0-5 - Red Clay, stiff	(0-5) = 0.7
5-10 - Same	(5-10) = 0.8
10-15 - Sand	(10-15) = 0.5

End Boring @ 15'

Attachment C

Pace Analytical Laboratory Reports for Excavation Soil Samples

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

December 05, 2011

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

RE: Project: 49161092 TANK 5
Pace Project No.: 10176549

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on November 22, 2011. 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.
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(612)607-1700

CERTIFICATIONS

Project: 49161092 TANK 5
Pace Project No.: 10176549

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
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Idaho Certification #: MN00064
Illinois Certification #: 200011
Iowa Certification #: 368
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 Mexico Certification #: Pace
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
North Dakota Certification #: R-036A
Ohio VAP Certification #: CL101
Oklahoma Certification #: D9921
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Washington Certification #: C754
Wisconsin Certification #: 999407970

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SAMPLE SUMMARY

Project: 49161092 TANK 5
Pace Project No.: 10176549

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10176549001	**TANK5-B-1_3-3**	Solid	11/17/11 14:50	11/22/11 09:55
10176549002	**TANK5-B-2_10-10**	Solid	11/17/11 15:10	11/22/11 09:55
10176549003	**TANK5-S-1_2-2**	Solid	11/17/11 14:55	11/22/11 09:55
10176549004	**TANK5-S-2_3-3**	Solid	11/17/11 15:00	11/22/11 09:55
10176549005	**TANK5-S-3_2-2**	Solid	11/17/11 15:05	11/22/11 09:55

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SAMPLE ANALYTE COUNT

Project: 49161092 TANK 5
Pace Project No.: 10176549

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10176549001	**TANK5-B-1_3-3**		AMO	2	
		WI MOD DRO	JRH	2	PASI-M
		WI MOD GRO	KT1	5	PASI-M
		% Moisture	JDL	1	PASI-M
10176549002	**TANK5-B-2_10-10**		AMO	2	
		WI MOD DRO	JRH	2	PASI-M
		WI MOD GRO	KT1	5	PASI-M
		% Moisture	JDL	1	PASI-M
10176549003	**TANK5-S-1_2-2**		AMO	2	
		WI MOD DRO	JRH	2	PASI-M
		WI MOD GRO	KT1	5	PASI-M
		% Moisture	JDL	1	PASI-M
10176549004	**TANK5-S-2_3-3**		AMO	2	
		WI MOD DRO	JRH	2	PASI-M
		WI MOD GRO	KT1	5	PASI-M
		% Moisture	JDL	1	PASI-M
10176549005	**TANK5-S-3_2-2**		AMO	2	
		WI MOD DRO	JRH	2	PASI-M
		WI MOD GRO	KT1	5	PASI-M
		% Moisture	JDL	1	PASI-M
		EPA 8270 by SIM	JLR	18	PASI-M

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ANALYTICAL RESULTS

Project: 49161092 TANK 5
Pace Project No.: 10176549

Sample: TANK5-B-1_3-3 **Lab ID: 10176549001** Collected: 11/17/11 14:50 Received: 11/22/11 09:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Field Data	Analytical Method:								
Start Depth	**3**	feet			1		11/22/11 17:17		
Stop Depth	**3**	feet			1		11/22/11 17:17		
WIDRO GCS Silica Gel	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO								
Diesel Range Organics	ND	mg/kg	11.1	5.6	1	11/23/11 07:23	11/26/11 16:50		
Surrogates									
n-Triacontane (S)	69	%	38-125		1	11/23/11 07:23	11/26/11 16:50		1M
WIGRO GCV	Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext.								
Benzene	ND	mg/kg	0.061	0.027	1	11/28/11 07:56	11/28/11 18:20	71-43-2	
Ethylbenzene	ND	mg/kg	0.061	0.023	1	11/28/11 07:56	11/28/11 18:20	100-41-4	
Toluene	ND	mg/kg	0.061	0.026	1	11/28/11 07:56	11/28/11 18:20	108-88-3	
Xylene (Total)	ND	mg/kg	0.18	0.061	1	11/28/11 07:56	11/28/11 18:20	1330-20-7	
Surrogates									
a,a,a-Trifluorotoluene (S)	95	%	80-125		1	11/28/11 07:56	11/28/11 18:20	98-08-8	
Dry Weight	Analytical Method: % Moisture								
Percent Moisture	**18.0**	%	0.10	0.10	1		12/01/11 00:00		

Sample: TANK5-B-2_10-10 **Lab ID: 10176549002** Collected: 11/17/11 15:10 Received: 11/22/11 09:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Field Data	Analytical Method:								
Start Depth	**10**	feet			1		11/22/11 17:18		
Stop Depth	**10**	feet			1		11/22/11 17:18		
WIDRO GCS Silica Gel	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO								
Diesel Range Organics	ND	mg/kg	11.5	5.7	1	11/23/11 07:23	11/26/11 16:57		
Surrogates									
n-Triacontane (S)	68	%	38-125		1	11/23/11 07:23	11/26/11 16:57		1M
WIGRO GCV	Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext.								
Benzene	ND	mg/kg	0.071	0.031	1	11/28/11 07:56	11/28/11 18:43	71-43-2	
Ethylbenzene	ND	mg/kg	0.071	0.027	1	11/28/11 07:56	11/28/11 18:43	100-41-4	
Toluene	ND	mg/kg	0.071	0.030	1	11/28/11 07:56	11/28/11 18:43	108-88-3	
Xylene (Total)	ND	mg/kg	0.21	0.071	1	11/28/11 07:56	11/28/11 18:43	1330-20-7	
Surrogates									
a,a,a-Trifluorotoluene (S)	97	%	80-125		1	11/28/11 07:56	11/28/11 18:43	98-08-8	
Dry Weight	Analytical Method: % Moisture								
Percent Moisture	**23.8**	%	0.10	0.10	1		12/01/11 00:00		

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ANALYTICAL RESULTS

Project: 49161092 TANK 5
Pace Project No.: 10176549

Sample: TANK5-S-1_2-2 **Lab ID: 10176549003** Collected: 11/17/11 14:55 Received: 11/22/11 09:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Field Data	Analytical Method:								
Start Depth	**2**	feet			1		11/22/11 17:18		
Stop Depth	**2**	feet			1		11/22/11 17:18		
WIDRO GCS Silica Gel	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO								
Diesel Range Organics	ND	mg/kg	12.8	6.4	1	11/23/11 07:23	11/26/11 16:37		
Surrogates									
n-Triacontane (S)	72	%	38-125		1	11/23/11 07:23	11/26/11 16:37		1M
WIGRO GCV	Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext.								
Benzene	ND	mg/kg	0.066	0.029	1	11/28/11 07:56	11/28/11 19:06	71-43-2	
Ethylbenzene	ND	mg/kg	0.066	0.025	1	11/28/11 07:56	11/28/11 19:06	100-41-4	
Toluene	ND	mg/kg	0.066	0.028	1	11/28/11 07:56	11/28/11 19:06	108-88-3	
Xylene (Total)	ND	mg/kg	0.20	0.066	1	11/28/11 07:56	11/28/11 19:06	1330-20-7	
Surrogates									
a,a,a-Trifluorotoluene (S)	96	%	80-125		1	11/28/11 07:56	11/28/11 19:06	98-08-8	
Dry Weight	Analytical Method: % Moisture								
Percent Moisture	**21.1**	%	0.10	0.10	1		12/01/11 00:00		

Sample: TANK5-S-2_3-3 **Lab ID: 10176549004** Collected: 11/17/11 15:00 Received: 11/22/11 09:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Field Data	Analytical Method:								
Start Depth	**3**	feet			1		11/22/11 17:19		
Stop Depth	**3**	feet			1		11/22/11 17:19		
WIDRO GCS Silica Gel	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO								
Diesel Range Organics	**19.6**	mg/kg	11.6	5.8	1	11/23/11 07:23	11/26/11 17:04		
Surrogates									
n-Triacontane (S)	98	%	38-125		1	11/23/11 07:23	11/26/11 17:04		1M
WIGRO GCV	Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext.								
Benzene	ND	mg/kg	0.064	0.028	1	11/28/11 07:56	11/28/11 19:29	71-43-2	
Ethylbenzene	ND	mg/kg	0.064	0.024	1	11/28/11 07:56	11/28/11 19:29	100-41-4	
Toluene	ND	mg/kg	0.064	0.027	1	11/28/11 07:56	11/28/11 19:29	108-88-3	
Xylene (Total)	ND	mg/kg	0.19	0.064	1	11/28/11 07:56	11/28/11 19:29	1330-20-7	
Surrogates									
a,a,a-Trifluorotoluene (S)	96	%	80-125		1	11/28/11 07:56	11/28/11 19:29	98-08-8	
Dry Weight	Analytical Method: % Moisture								
Percent Moisture	**19.8**	%	0.10	0.10	1		12/01/11 00:00		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: 49161092 TANK 5
Pace Project No.: 10176549

Sample: TANK5-S-3_2-2 **Lab ID: 10176549005** Collected: 11/17/11 15:05 Received: 11/22/11 09:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Field Data	Analytical Method:								
Start Depth	**2**	feet			1		11/22/11 17:19		
Stop Depth	**2**	feet			1		11/22/11 17:19		
WIDRO GCS Silica Gel	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO								
Diesel Range Organics	ND	mg/kg	11.5	5.7	1	11/23/11 07:23	11/26/11 16:43		
Surrogates									
n-Triacontane (S)	73	%	38-125		1	11/23/11 07:23	11/26/11 16:43		1M
WIGRO GCV	Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext.								
Benzene	**1.4**	mg/kg	0.30	0.13	5	11/28/11 07:56	11/29/11 17:19	71-43-2	
Ethylbenzene	**1.4**	mg/kg	0.30	0.11	5	11/28/11 07:56	11/29/11 17:19	100-41-4	
Toluene	**0.51**	mg/kg	0.30	0.13	5	11/28/11 07:56	11/29/11 17:19	108-88-3	
Xylene (Total)	**4.2**	mg/kg	0.89	0.30	5	11/28/11 07:56	11/29/11 17:19	1330-20-7	
Surrogates									
a,a,a-Trifluorotoluene (S)	86	%	80-125		5	11/28/11 07:56	11/29/11 17:19	98-08-8	D3
Dry Weight	Analytical Method: % Moisture								
Percent Moisture	**18.2**	%	0.10	0.10	1		12/01/11 00:00		
8270 MSSV PAH by SIM	Analytical Method: EPA 8270 by SIM Preparation Method: EPA 3550								
Acenaphthene	ND	ug/kg	12.1	0.36	1	11/23/11 10:53	11/29/11 17:09	83-32-9	
Acenaphthylene	ND	ug/kg	12.1	0.36	1	11/23/11 10:53	11/29/11 17:09	208-96-8	
Anthracene	ND	ug/kg	12.1	6.1	1	11/23/11 10:53	11/29/11 17:09	120-12-7	
Benzo(a)anthracene	ND	ug/kg	12.1	6.1	1	11/23/11 10:53	11/29/11 17:09	56-55-3	
Benzo(a)pyrene	ND	ug/kg	12.1	6.1	1	11/23/11 10:53	11/29/11 17:09	50-32-8	
Benzo(b)fluoranthene	ND	ug/kg	12.1	0.61	1	11/23/11 10:53	11/29/11 17:09	205-99-2	
Benzo(g,h,i)perylene	ND	ug/kg	12.1	0.61	1	11/23/11 10:53	11/29/11 17:09	191-24-2	
Benzo(k)fluoranthene	ND	ug/kg	12.1	0.73	1	11/23/11 10:53	11/29/11 17:09	207-08-9	
Chrysene	ND	ug/kg	12.1	0.61	1	11/23/11 10:53	11/29/11 17:09	218-01-9	
Dibenz(a,h)anthracene	ND	ug/kg	12.1	0.61	1	11/23/11 10:53	11/29/11 17:09	53-70-3	
Fluoranthene	ND	ug/kg	12.1	6.1	1	11/23/11 10:53	11/29/11 17:09	206-44-0	
Fluorene	ND	ug/kg	12.1	0.49	1	11/23/11 10:53	11/29/11 17:09	86-73-7	
Indeno(1,2,3-cd)pyrene	ND	ug/kg	12.1	0.61	1	11/23/11 10:53	11/29/11 17:09	193-39-5	
Naphthalene	ND	ug/kg	12.1	0.73	1	11/23/11 10:53	11/29/11 17:09	91-20-3	
Phenanthrene	ND	ug/kg	12.1	6.1	1	11/23/11 10:53	11/29/11 17:09	85-01-8	
Pyrene	ND	ug/kg	12.1	0.49	1	11/23/11 10:53	11/29/11 17:09	129-00-0	
Surrogates									
2-Fluorobiphenyl (S)	50	%	30-130		1	11/23/11 10:53	11/29/11 17:09	321-60-8	
Terphenyl-d14 (S)	56	%	30-150		1	11/23/11 10:53	11/29/11 17:09	1718-51-0	

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QUALITY CONTROL DATA

Project: 49161092 TANK 5
Pace Project No.: 10176549

QC Batch: GCV/8704 Analysis Method: WI MOD GRO
QC Batch Method: TPH GRO/PVOC WI ext. Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10176549001, 10176549002, 10176549003, 10176549004, 10176549005

METHOD BLANK: 1105728 Matrix: Solid
Associated Lab Samples: 10176549001, 10176549002, 10176549003, 10176549004, 10176549005

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Benzene	mg/kg	ND	0.050	11/28/11 12:58	
Ethylbenzene	mg/kg	ND	0.050	11/28/11 12:58	
Toluene	mg/kg	ND	0.050	11/28/11 12:58	
Xylene (Total)	mg/kg	ND	0.15	11/28/11 12:58	
a,a,a-Trifluorotoluene (S)	%	95	80-125	11/28/11 12:58	

LABORATORY CONTROL SAMPLE & LCSD: 1105729 1105730

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
Benzene	mg/kg	5	5.3	5.9	106	119	80-120	11	20	
Ethylbenzene	mg/kg	5	5.3	5.8	107	115	80-120	8	20	
Toluene	mg/kg	5	5.4	5.9	108	118	80-120	9	20	
Xylene (Total)	mg/kg	15	16.3	17.5	109	117	80-120	7	20	
a,a,a-Trifluorotoluene (S)	%				95	95	80-125			

MATRIX SPIKE SAMPLE: 1105731

Parameter	Units	10176665001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Benzene	mg/kg	ND	5.2	5.2	102	80-120	
Ethylbenzene	mg/kg	ND	5.2	5.1	100	80-120	
Toluene	mg/kg	0.061	5.2	5.2	100	80-120	
Xylene (Total)	mg/kg	ND	15.5	15.7	101	80-120	
a,a,a-Trifluorotoluene (S)	%				96	80-125	

SAMPLE DUPLICATE: 1105732

Parameter	Units	10176665002 Result	Dup Result	RPD	Max RPD	Qualifiers
Benzene	mg/kg	ND	ND		20	
Ethylbenzene	mg/kg	ND	ND		20	
Toluene	mg/kg	ND	ND		20	
Xylene (Total)	mg/kg	ND	ND		20	
a,a,a-Trifluorotoluene (S)	%	97	95	5		

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QUALITY CONTROL DATA

Project: 49161092 TANK 5
Pace Project No.: 10176549

QC Batch: MPRP/30070 Analysis Method: % Moisture
QC Batch Method: % Moisture Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10176549001, 10176549002, 10176549003, 10176549004, 10176549005

SAMPLE DUPLICATE: 1108403

Parameter	Units	10176297022 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	16.2	17.0	5	30	

SAMPLE DUPLICATE: 1108404

Parameter	Units	10176631003 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	14.7	14.1	4	30	

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QUALITY CONTROL DATA

Project: 49161092 TANK 5
Pace Project No.: 10176549

QC Batch: OEXT/17333
QC Batch Method: EPA 3550
Analysis Method: EPA 8270 by SIM
Analysis Description: 8270 Solid PAH by SIM MSSV
Associated Lab Samples: 10176549005

METHOD BLANK: 1104889 Matrix: Solid
Associated Lab Samples: 10176549005

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Acenaphthene	ug/kg	ND	10.0	11/29/11 11:24	
Acenaphthylene	ug/kg	ND	10.0	11/29/11 11:24	
Anthracene	ug/kg	ND	10.0	11/29/11 11:24	
Benzo(a)anthracene	ug/kg	ND	10.0	11/29/11 11:24	
Benzo(a)pyrene	ug/kg	ND	10.0	11/29/11 11:24	
Benzo(b)fluoranthene	ug/kg	ND	10.0	11/29/11 11:24	
Benzo(g,h,i)perylene	ug/kg	ND	10.0	11/29/11 11:24	
Benzo(k)fluoranthene	ug/kg	ND	10.0	11/29/11 11:24	
Chrysene	ug/kg	ND	10.0	11/29/11 11:24	
Dibenz(a,h)anthracene	ug/kg	ND	10.0	11/29/11 11:24	
Fluoranthene	ug/kg	ND	10.0	11/29/11 11:24	
Fluorene	ug/kg	ND	10.0	11/29/11 11:24	
Indeno(1,2,3-cd)pyrene	ug/kg	ND	10.0	11/29/11 11:24	
Naphthalene	ug/kg	ND	10.0	11/29/11 11:24	
Phenanthrene	ug/kg	ND	10.0	11/29/11 11:24	
Pyrene	ug/kg	ND	10.0	11/29/11 11:24	
2-Fluorobiphenyl (S)	%	82	30-130	11/29/11 11:24	
Terphenyl-d14 (S)	%	86	30-150	11/29/11 11:24	

LABORATORY CONTROL SAMPLE: 1104890

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Acenaphthene	ug/kg	33.3	22.5	68	56-125	
Acenaphthylene	ug/kg	33.3	22.1	66	49-125	
Anthracene	ug/kg	33.3	22.4	67	49-125	
Benzo(a)anthracene	ug/kg	33.3	25.4	76	60-125	
Benzo(a)pyrene	ug/kg	33.3	26.0	78	58-125	
Benzo(b)fluoranthene	ug/kg	33.3	26.4	79	63-125	
Benzo(g,h,i)perylene	ug/kg	33.3	27.4	82	56-125	
Benzo(k)fluoranthene	ug/kg	33.3	28.7	86	56-127	
Chrysene	ug/kg	33.3	26.3	79	60-125	
Dibenz(a,h)anthracene	ug/kg	33.3	28.2	85	57-125	
Fluoranthene	ug/kg	33.3	26.3	79	58-125	
Fluorene	ug/kg	33.3	22.2	67	53-125	
Indeno(1,2,3-cd)pyrene	ug/kg	33.3	27.5	82	56-125	
Naphthalene	ug/kg	33.3	21.7	65	56-125	
Phenanthrene	ug/kg	33.3	23.6	71	53-125	
Pyrene	ug/kg	33.3	26.7	80	60-125	
2-Fluorobiphenyl (S)	%			84	30-130	
Terphenyl-d14 (S)	%			96	30-150	

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QUALITY CONTROL DATA

Project: 49161092 TANK 5
Pace Project No.: 10176549

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 1104891 1104892

Parameter	Units	10176570001 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	34.4	34.1	25.3	48.7	44	113	30-150	63	30	D6
Acenaphthylene	ug/kg	ND	34.4	34.1	23.1	28.8	67	84	30-150	22	30	
Anthracene	ug/kg	30.0	34.4	34.1	36.3	109	18	230	30-150	100	30	D6,M1
Benzo(a)anthracene	ug/kg	99.1	34.4	34.1	85.5	253	-39	452	30-150	99	30	D6,M1
Benzo(a)pyrene	ug/kg	98.7	34.4	34.1	88.4	253	-30	452	30-150	96	30	D6,M1
Benzo(b)fluoranthene	ug/kg	128	34.4	34.1	111	332	-50	596	30-150	100	30	D6,M1
Benzo(g,h,i)perylene	ug/kg	76.7	34.4	34.1	77.2	198	1	355	30-150	88	30	D6,M1
Benzo(k)fluoranthene	ug/kg	59.4	34.4	34.1	59.9	135	2	223	30-150	77	30	D6,M1
Chrysene	ug/kg	108	34.4	34.1	92.1	279	-45	502	30-150	101	30	D6,M1
Dibenz(a,h)anthracene	ug/kg	30.3	34.4	34.1	30.2	97.6	-.2	198	30-150	105	30	D6,M1
Fluoranthene	ug/kg	203	34.4	34.1	152	488	-150	834	30-150	105	30	D6,E, M1
Fluorene	ug/kg	ND	34.4	34.1	23.8	49.5	42	118	30-150	70	30	D6
Indeno(1,2,3-cd)pyrene	ug/kg	67.0	34.4	34.1	70.1	179	9	328	30-150	87	30	D6,M1
Naphthalene	ug/kg	ND	34.4	34.1	20.8	27.5	60	81	30-150	28	30	
Phenanthrene	ug/kg	118	34.4	34.1	81.4	336	-106	641	30-150	122	30	D6,M1
Pyrene	ug/kg	176	34.4	34.1	137	434	-116	756	30-150	104	30	D6,E, M1
2-Fluorobiphenyl (S)	%						74	83	30-130			
Terphenyl-d14 (S)	%						76	92	30-150			

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QUALITY CONTROL DATA

Project: 49161092 TANK 5
Pace Project No.: 10176549

QC Batch: OEXT/17328 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO Solid GCV
Associated Lab Samples: 10176549001, 10176549002, 10176549003, 10176549004, 10176549005

METHOD BLANK: 1104618 Matrix: Solid
Associated Lab Samples: 10176549001, 10176549002, 10176549003, 10176549004, 10176549005

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Diesel Range Organics	mg/kg	ND	10.0	11/26/11 16:23	
n-Triacontane (S)	%	78	38-125	11/26/11 16:23	

LABORATORY CONTROL SAMPLE & LCSD: 1104619 1104620

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
Diesel Range Organics	mg/kg	80	58.0	57.8	73	72	70-125	.5	20	
n-Triacontane (S)	%				78	75	38-125			

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QUALIFIERS

Project: 49161092 TANK 5
Pace Project No.: 10176549

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.

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.

LABORATORIES

PASI-M	Pace Analytical Services - Minneapolis

ANALYTE QUALIFIERS

1M	The sample was re-weighed into a new container because the original container was not the standard tared 4oz amber jar.
D3	Sample was diluted due to the presence of high levels of non-target analytes or other matrix interference.
D6	The relative percent difference (RPD) between the sample and sample duplicate exceeded laboratory control limits.
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.

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092 TANK 5
Pace Project No.: 10176549

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10176549001	**TANK5-B-1_3-3**		FLD/		
10176549002	**TANK5-B-2_10-10**		FLD/		
10176549003	**TANK5-S-1_2-2**		FLD/		
10176549004	**TANK5-S-2_3-3**		FLD/		
10176549005	**TANK5-S-3_2-2**		FLD/		
10176549001	**TANK5-B-1_3-3**	WI MOD DRO	OEXT/17328	WI MOD DRO	GCSV/8909
10176549002	**TANK5-B-2_10-10**	WI MOD DRO	OEXT/17328	WI MOD DRO	GCSV/8909
10176549003	**TANK5-S-1_2-2**	WI MOD DRO	OEXT/17328	WI MOD DRO	GCSV/8909
10176549004	**TANK5-S-2_3-3**	WI MOD DRO	OEXT/17328	WI MOD DRO	GCSV/8909
10176549005	**TANK5-S-3_2-2**	WI MOD DRO	OEXT/17328	WI MOD DRO	GCSV/8909
10176549001	**TANK5-B-1_3-3**	TPH GRO/PVOC WI ext.	GCV/8704	WI MOD GRO	GCV/8705
10176549002	**TANK5-B-2_10-10**	TPH GRO/PVOC WI ext.	GCV/8704	WI MOD GRO	GCV/8705
10176549003	**TANK5-S-1_2-2**	TPH GRO/PVOC WI ext.	GCV/8704	WI MOD GRO	GCV/8705
10176549004	**TANK5-S-2_3-3**	TPH GRO/PVOC WI ext.	GCV/8704	WI MOD GRO	GCV/8705
10176549005	**TANK5-S-3_2-2**	TPH GRO/PVOC WI ext.	GCV/8704	WI MOD GRO	GCV/8705
10176549001	**TANK5-B-1_3-3**	% Moisture	MPRP/30070		
10176549002	**TANK5-B-2_10-10**	% Moisture	MPRP/30070		
10176549003	**TANK5-S-1_2-2**	% Moisture	MPRP/30070		
10176549004	**TANK5-S-2_3-3**	% Moisture	MPRP/30070		
10176549005	**TANK5-S-3_2-2**	% Moisture	MPRP/30070		
10176549005	**TANK5-S-3_2-2**	EPA 3550	OEXT/17333	EPA 8270 by SIM	MSSV/7512

Chain of Custody

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

1137

10176549

COC 1 of 1

Project Number: 49161092

Project Name: Tank 5

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

COC Number: № 32220

Project Manager: FIAW

Project QC Contact: AAW

Sampled by: RBE

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	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.)	Soil: PAH	Total Number Of Containers
1. Tank5-B-1	3	3	FT	11/17/11	1450		X	X											1	1			1		3
2. Tank5-B-2	10	10	↓	↓	1510		↓	↓											↓	↓			↓		3
3. Tank5-5-1	2	2	↓	↓	1455		↓	↓											↓	↓			↓		3
4. Tank5-5-2	3	3	↓	↓	1500		↓	↓											↓	↓			↓		3
5. Tank5-5-3	2	2	↓	↓	1505		↓	↓											↓	↓			1		4
6.																									
7.																									
8.																									
9.																									
10.																									

BTEX, DRO, Moisture
QC1
QC2
QC3
QC4
+PAH QC5

Normal 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
Ryan Enoch	Y N	11/21/11	1200	K. Cox / Pace	11/22/11	9:55
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

T = 3.3

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

Pace Analytical®	Document Name: **Sample Condition Upon Receipt Form**	Revised Date: 02Jun2011 Page 1 of 1
	Document Number: **F-L-213 Rev.01**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Barr **Project #** 10176549

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

Tracking #: 797759635817

Optional: Proj. Due Date: Proj. Name:

Custody Seal on Cooler/Box Present: ☐ yes ☑ no Seals intact: ☐ yes ☑ no

Packing Material: ☐ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other _____ Temp Blank: Yes ✓ No _____

Thermometer Used 80344042 or 80512447 **Type of Ice:** (Wet) Blue None ☐ Samples on ice, cooling process has begun

Cooler Temperature 3.3 **Biological Tissue is Frozen:** Yes No

Temp should be above freezing to 6°C

Date and Initials of person examining contents: 11/22/11 LC

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 & Signature on COC:	☐Yes ☑No ☐N/A	4.
Samples Arrived within Hold Time:	☑Yes ☐No ☐N/A	5.
Short Hold Time Analysis (<72hr):	☐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. Noncompliance are noted in 13.	☐Yes ☐No ☑N/A	13. ☐ HNO3 ☐ H2SO4 ☐ NaOH ☐ HCl Samp #
All containers needing preservation are found to be in compliance with EPA recommendation.	☐Yes ☐No ☑N/A	
Exceptions: VOA,Coliform, TOC, Oil and Grease, WI-DRO (water)	☐Yes ☑No	Initial when completed / Lot # of added preservative
Samples checked for dechlorination:	☐Yes ☐No ☑N/A	14.
Headspace in VOA Vials (>6mm):	☐Yes ☐No ☑N/A	15.
Trip Blank Present:	☐Yes ☐No ☑N/A	16.
Trip Blank Custody Seals Present	☐Yes ☑No ☐N/A	
Pace Trip Blank Lot # (if purchased): _____		

Client Notification/ Resolution: Field Data Required? Y / N

Person Contacted: _____ Date/Time: _____

Comments/ Resolution: _____

Project Manager Review: _____ **Date:** 11/22/11

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

June 27, 2012

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

RE: Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on June 19, 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: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

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
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Idaho Certification #: MN00064
Illinois Certification #: 200011
Iowa Certification #: 368
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 Mexico Certification #: Pace
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
North Dakota Certification #: R-036A
Ohio VAP Certification #: CL101
Oklahoma Certification #: D9921
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Washington Certification #: C754
Wisconsin Certification #: 999407970

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SAMPLE SUMMARY

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10195972001	**TK5-SB-1_2-3'**	Solid	06/15/12 14:50	06/19/12 10:05

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SAMPLE ANALYTE COUNT

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10195972001	**TK5-SB-1_2-3'**	WI MOD DRO	JRH	2	PASI-M
		WI MOD GRO	KT1	7	PASI-M
		ASTM D2974	JDL	1	PASI-M

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PROJECT NARRATIVE

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

Method: **WI MOD DRO**
Description: WIDRO GCS
Client: Barr Engineering
Date: June 27, 2012

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.

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

Additional Comments:

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PROJECT NARRATIVE

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

Method: **WI MOD GRO**
Description: WIGRO GCV
Client: Barr Engineering
Date: June 27, 2012

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: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

Sample: TK5-SB-1_2-3' **Lab ID: 10195972001** Collected: 06/15/12 14:50 Received: 06/19/12 10:05 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	**<11.0**	mg/kg	11.0	1.2	1	06/20/12 12:29	06/22/12 20:33		
Surrogates									
n-Triacontane (S)	73	%	50-150		1	06/20/12 12:29	06/22/12 20:33		
WIGRO GCV	Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext.								
Benzene	**<0.065**	mg/kg	0.065	0.0078	1	06/21/12 08:13	06/22/12 03:16	71-43-2	
Ethylbenzene	**<0.065**	mg/kg	0.065	0.010	1	06/21/12 08:13	06/22/12 03:16	100-41-4	
Toluene	**<0.065**	mg/kg	0.065	0.0078	1	06/21/12 08:13	06/22/12 03:16	108-88-3	
1,2,4-Trimethylbenzene	**<0.065**	mg/kg	0.065	0.0091	1	06/21/12 08:13	06/22/12 03:16	95-63-6	
1,3,5-Trimethylbenzene	**<0.065**	mg/kg	0.065	0.014	1	06/21/12 08:13	06/22/12 03:16	108-67-8	
Xylene (Total)	**<0.19**	mg/kg	0.19	0.021	1	06/21/12 08:13	06/22/12 03:16	1330-20-7	
Surrogates									
a,a,a-Trifluorotoluene (S)	99	%	80-125		1	06/21/12 08:13	06/22/12 03:16	98-08-8	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**24.1**	%	0.10	0.10	1		06/20/12 00:00		

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QUALITY CONTROL DATA

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

QC Batch: GCV/9423 Analysis Method: WI MOD GRO
QC Batch Method: TPH GRO/PVOC WI ext. Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10195972001

METHOD BLANK: 1222948 Matrix: Solid
Associated Lab Samples: 10195972001

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
1,2,4-Trimethylbenzene	mg/kg	<0.050	0.050	06/21/12 20:09	
1,3,5-Trimethylbenzene	mg/kg	<0.050	0.050	06/21/12 20:09	
Benzene	mg/kg	<0.050	0.050	06/21/12 20:09	
Ethylbenzene	mg/kg	<0.050	0.050	06/21/12 20:09	
Toluene	mg/kg	<0.050	0.050	06/21/12 20:09	
Xylene (Total)	mg/kg	<0.15	0.15	06/21/12 20:09	
a,a,a-Trifluorotoluene (S)	%	99	80-125	06/21/12 20:09	

LABORATORY CONTROL SAMPLE & LCSD: 1222949 1222950

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	5.6	5.1	111	102	80-120	9	20	
1,3,5-Trimethylbenzene	mg/kg	5	5.6	5.2	112	103	80-120	8	20	
Benzene	mg/kg	5	5.5	5.2	109	104	80-120	5	20	
Ethylbenzene	mg/kg	5	5.6	5.2	112	105	80-120	7	20	
Toluene	mg/kg	5	5.5	5.2	110	104	80-120	6	20	
Xylene (Total)	mg/kg	15	16.7	15.5	111	103	80-120	7	20	
a,a,a-Trifluorotoluene (S)	%				97	99	80-125			

MATRIX SPIKE SAMPLE: 1222951

Parameter	Units	10195951001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
1,2,4-Trimethylbenzene	mg/kg	<0.065	6.3	6.0	96	80-120	
1,3,5-Trimethylbenzene	mg/kg	<0.065	6.3	6.1	97	80-120	
Benzene	mg/kg	<0.065	6.3	5.7	90	80-120	
Ethylbenzene	mg/kg	<0.065	6.3	6.0	95	80-120	
Toluene	mg/kg	<0.065	6.3	5.8	92	80-120	
Xylene (Total)	mg/kg	<0.20	18.9	18.0	95	80-120	
a,a,a-Trifluorotoluene (S)	%				98	80-125	

SAMPLE DUPLICATE: 1222952

Parameter	Units	10195951002 Result	Dup Result	RPD	Max RPD	Qualifiers
1,2,4-Trimethylbenzene	mg/kg	<0.070	<0.066		20	
1,3,5-Trimethylbenzene	mg/kg	<0.070	<0.066		20	
Benzene	mg/kg	<0.070	<0.066		20	
Ethylbenzene	mg/kg	<0.070	<0.066		20	
Toluene	mg/kg	<0.070	<0.066		20	

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Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

SAMPLE DUPLICATE: 1222952

Parameter	Units	10195951002 Result	Dup Result	RPD	Max RPD	Qualifiers
Xylene (Total)	mg/kg	<0.21	<0.20		20	
a,a,a-Trifluorotoluene (S)	%	97	100	3		

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QUALITY CONTROL DATA

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

QC Batch: MPRP/33113
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10195972001

SAMPLE DUPLICATE: 1222044

Parameter	Units	129022001 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	65.6	71.4	8	30	

SAMPLE DUPLICATE: 1222115

Parameter	Units	10195984006 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	20.1	20.4	2	30	

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QUALITY CONTROL DATA

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

QC Batch: OEXT/18913 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10195972001

METHOD BLANK: 1222197 Matrix: Solid
Associated Lab Samples: 10195972001

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Diesel Range Organics	mg/kg	<10.0	10.0	06/22/12 19:01	
n-Triacontane (S)	%	77	50-150	06/22/12 19:01	

LABORATORY CONTROL SAMPLE & LCSD: 1222198 1222199

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
Diesel Range Organics	mg/kg	80	66.0	77.9	82	97	70-120	17	20	
n-Triacontane (S)	%				86	100	50-150			

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QUALIFIERS

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

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

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

QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092.014 RESP 004 Enbridge
Pace Project No.: 10195972

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10195972001	**TK5-SB-1_2-3'**	WI MOD DRO	OEXT/18913	WI MOD DRO	GCSV/9725
10195972001	**TK5-SB-1_2-3'**	TPH GRO/PVOC WI ext.	GCV/9423	WI MOD GRO	GCV/9424
10195972001	**TK5-SB-1_2-3'**	ASTM D2974	MPRP/33113		

10195972

BARR

Chain of Custody

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

WO#: 10195972

10195972

Project Number: 49161092.01 RESP 004

Project Name: Enbridge GP Tank 5

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

COC Number: № 35255

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	Soil	Total Number Of Containers
												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	VOCs (tared MeOH) #1; GRO, BTEX (tared MeOH) #1; DRO (tared unpreserved); Metals (unpreserved); SVOCs (unpreserved) #2; % Solids (plastic vial, unpres.); PVOC - MTBE (tared MeOH)	
1. TKS-SB-1	2	3	ft	6/15/2012	14:50		X		X				2 1 2	5
2.														
3.														
4.														
5.														
6.														
7.														
8.														
9.														
10.														

COC 1 of 1

Project Manager: REE

Project QC Contact: AAN

Sampled by: ARP2 / AJM

Laboratory: PACE

10195972001

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	6/14/12	10:00		6/18/12	10:00
Relinquished By:	On Ice? Y N	Date	Time	Received by: Cassie Sicard FedEx	Date 6-19-12	Time 1605

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

Air Bill Number: 5-7 oc

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**	Revised Date: 15Feb2012 Page 1 of 1
	Document Number: **F-MN-L-213-rev.02**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Barr Project # 10195972

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

Tracking #: 793693089196

Optional: Proj. Due Date: Proj. Name:

Custody Seal on Cooler/Box Present: ☐ yes ☑ no Seals intact: ☐ yes ☑ no

Packing Material: ☐ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other _____ Temp Blank: Yes ✓ No _____

Thermometer Used 80344042 or 80512447 Type of Ice: Wet Blue None ☐ Samples on ice, cooling process has begun

Cooler Temperature 5.7 Biological Tissue is Frozen: Yes No

Temp should be above freezing to 6°C

Date and Initials of person examining contents: 05/6.19.12

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 & Signature on COC:	☑Yes ☐No ☐N/A	4.
Samples Arrived within Hold Time:	☑Yes ☐No ☐N/A	5.
Short Hold Time Analysis (<72hr):	☐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. Noncompliance are noted in 13.	☐Yes ☐No ☑N/A	13. ☐ HNO3 ☐ H2SO4 ☐ NaOH ☐ HCl Samp #
All containers needing preservation are found to be in compliance with EPA recommendation (HNO3, H2SO4, 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. AWO 06/19/12
Trip Blank Custody Seals Present	☐Yes ☐No ☑N/A	
Pace Trip Blank Lot # (if purchased): _____		

Client Notification/ Resolution: Field Data Required? Y / N

Person Contacted: _____ Date/Time: _____

Comments/ Resolution: _____

Project Manager Review: AWO **Date:** 06/19/12

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)

Attachment D

Waste Disposal Documents

Generator's Non-hazardous Waste Profile Sheet

Requested Disposal Facility: Voyaguer Landfill, Canyon, MN Profile Number: _____

☐ Renewal for Profile Number: _____ Waste Approval Expiration Date: _____

☐ Check here if there are multiple generating locations for this waste. Attach additional locations.

A. Waste Generator Facility Information (must reflect location of waste generation/origin)

1. Generator Name: Enbridge Pipelines, Limited Partnership, LLC
2. Site Address: Tank 5-6
3. City/ZIP: Superior, 55880
4. State: Wisconsin
5. County: Douglas
6. Contact Name/Title: Tom Peterson, Operation Team Lead
7. Email Address: tom.peterson@enbridge.com
8. Phone: 715-398-8327 9. FAX: (715) 398-3223
10. NAICS Code: 486110
11. Generator USEPA ID #: 486110
12. State ID# (if applicable): NA

B. Customer Information ☐ same as above

P. O. Number: Tank 5 - 11/2011 - 49161092

1. Customer Name: Enbridge Pipelines
2. Billing Address: 1100 Louisiana Ave, STE. 3300
3. City, State and ZIP: Houston, TX 77002
4. Contact Name: Tom Peterson, Hans Wronka (Barr Eng.)
5. Contact Email: tom.peterson@enbridge.com, haw@barr.com
6. Phone: 715-398-8327 FAX: (715) 398-3223
7. Transporter Name: various transporters
8. Transporter ID # (if appl.): _____
9. Transporter Address: _____
10. City, State and ZIP: _____

C. Waste Stream Information

1. DESCRIPTION

a. Common Waste Name: Crude oil contaminated soil

State Waste Code(s): _____

b. Describe Process Generating Waste or Source of Contamination:

Historically hydrocarbon impacted soil

c. Typical Color(s): brown

d. Strong Odor? ☐ Yes ☑ No Describe: varies from little to moderate petroleum odor

e. Physical State at 70°F: ☑ Solid ☐ Liquid ☐ Powder ☐ Semi-Solid or Sludge ☐ Other: _____

f. Layers? ☑ Single layer ☐ Multi- layer ☐ NA

g. Water Reactive? ☐ Yes ☑ No If Yes, Describe: _____

h. Free Liquid Range (%): _____ to _____ ☑ NA(solid)

i. pH Range: _____ to _____ ☑ NA(solid)

j. Liquid Flash Point: ☐ < 140°F ☐ 140°- 199°F ☐ ≥ 200°F ☑ NA(solid)

k. Flammable Solid: ☐ Yes ☑ No

l. Physical Constituents: List all constituents of waste stream - (e.g. Soil 0-80%, Wood 0-20%): ☐ (See Attached)

Constituents (Total Composition Must be ≥ 100%)	Lower Range	Unit of Measure	Upper Range	Unit of Measure
1. soil	100%			
2.				
3.				
4.				
5.				
6.				

2. ESTIMATED QUANTITY OF WASTE AND SHIPPING INFORMATION

a. ☑ One Time Event ☐ Base ☐ Repeat Event

b. Estimated Annual Quantity: 250 ☐ Tons ☑ Cubic Yards ☐ Drums ☐ Gallons ☐ Other (specify): _____

c. Shipping Frequency: _____ Units per ☐ Month ☐ Quarter ☐ Year ☑ One Time ☐ Other

d. Is this a U.S. Department of Transportation (USDOT) Hazardous Material? (If yes, answer e.) ☐ Yes ☑ No

e. USDOT Shipping Description (if applicable): _____

3. SAFETY REQUIREMENTS (Handling, PPE, etc.): _____

WASTE MANAGEMENT

Generator's Non-hazardous Waste Profile Sheet

D. Regulatory Status (Please check appropriate responses)

1. Waste Identification:
   a. Does the waste meet the definition of a USEPA listed or characteristic hazardous waste as defined by 40 CFR Part 261? ☐ Yes ☑ No
      1. If yes, please complete a hazardous waste profile.
   b. Does the waste meet the definition of a state hazardous waste other than identified in D.1.a? ☐ Yes ☑ No
      1. If yes, please complete a hazardous waste profile.
2. Is this waste included in one or more of categories below (Check all that apply)? If yes, attach supporting documentation. ☐ Yes ☑ No
   ☐ Delisted Hazardous Waste ☐ Excluded Wastes Under 40CFR 261.4
   ☐ Treated Hazardous Waste Debris ☐ Treated Characteristic Hazardous Waste
3. Is the waste from a Federal (40 CFR 300, Appendix B) or state mandated clean-up? If yes, see instructions. ☐ Yes ☑ No
4. Does the waste represented by this waste profile sheet contain radioactive material? ☐ Yes ☑ No
   a. If yes, is disposal regulated by the Nuclear Regulatory Commission? ☐ Yes ☑ No
   b. If yes, is disposal regulated by a State Agency for radioactive waste/NORM? ☐ Yes ☑ No
5. Does the waste represented by this waste profile sheet contain Polychlorinated Biphenyls (PCBs)? ☐ Yes ☑ No
   (If yes, list in Chemical Composition - C.1.l)
   a. If yes, are the PCBs regulated by 40 CFR 761? ☐ Yes ☑ No
   b. If yes, is it remediation waste from a project being performed under the Self-Implementing option provided in 40 CFR 761.61(a)? ☐ Yes ☑ No
   c. If yes, were the PCBs imported into the US? ☐ Yes ☑ No
6. Does the waste contain untreated, regulated medical or infectious waste? ☐ Yes ☑ No
7. Does the waste contain asbestos? ☐ Yes ☑ No
   a. If Yes, ☐ Friable ☐ Non Friable
8. Is this profile for remediation waste from a facility that is a major source of Hazardous Air Pollutants (Site Remediation NESHAP, 40 CFR 63 subpart GGGGG)? ☐ Yes ☑ No
   a. If yes, does the waste contain <500 ppmw VOHAPs at the point of determination? ☐ Yes ☑ No

E. Generator Certifcation (Please read and certify by signature below)

By signing this Generator's Waste Profile Sheet, I hereby certify that all:

1. Information submitted in this profile and all attached documents contain true and accurate descriptions of the waste material;
2. Relevant information within the possession of the Generator regarding known or suspected hazards pertaining to this waste has been disclosed to WM/the Contractor;
3. Analytical data attached pertaining to the profiled waste was derived from testing a representative sample in accordance with 40 CFR 261.20(c) or equivalent rules; and
4. Changes that occur in the character of the waste (i.e. changes in the process or new analytical) will be identified by the Generator and disclosed to WM (and the Contractor if applicable) prior to providing the waste to WM (and the contractor if applicable).
5. Check all that apply:
   ☑ a. Attached analytical pertains to the waste. Identify laboratory & sample ID #'s and parameters tested: Pace Analytical, Tank 5-Stockpile-1 & 2, BTEX and DRO # Pages: 12
   ☐ b. Only the analysis identified on the attachment pertain to the waste (identify by laboratory & sample ID #'s and parameters tested). Attachment #: _____
   ☐ c. Additional information necessary to characterize the profiled waste has been attached (other then analytical, such as MSDS). Indicate the number of attached pages: _____
   ☐ d. I am an agent signing on behalf of the Generator and the delegation of authority to me from the Generator for this signature is available upon request.

Certification: _____ Title: Operation Team Lead

Company Name: Enbridge Pipelines, Limited Partnership, LLC Name (Print): Tom Peterson

Date: 11/29/2011

GENERATOR ANALYTICAL CERTIFICATION FORM
Appendix B

In completing this form, the Generator certifies that, unless otherwise indicated on the attached analytical, to the best of his/her knowledge:

- ☑ This waste does not contain regulated concentrations of the following metals: arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver.
- ☑ This waste does not contain regulated concentrations of the following pesticides and herbicides: Chlordane, Endrin, Heptachlor (and its epoxide), Lindane, Methoxychlor, Toxaphene, 2, 4-D, or 2, 4, 5-TP (Silvex).
- ☑ This waste does not contain regulated concentrations of the following organics: benzene, carbon tetrachloride, chlorobenzene, chloroform, o-cresol, m-cresol, p-cresol, cresol (total), 1, 4-dichlorobenzene, 1, 2-dichloroethane, 1, 1-dichloroethylene, 2, 4-dinitrotoluene, hexachlorobenzene, hexachlorobutadiene, hexachloroethane, methyl ethyl ketone, nitrobenzene, pentachlorophenol, pyridine, tetrachloroethylene, trichloroethylene (TCE), 2, 4, 5-trichlorophenol, 2, 4, 6-trichlorophenol, or vinyl chloride.
- ☑ This waste does not exhibit the characteristic of ignitability.
- ☑ This waste does not exhibit the characteristic of reactivity.
- ☑ This waste does not exhibit the characteristic of corrosivity.
- ☑ This waste does not contain regulated concentrations of PCBs (Polychlorinated Biphenyls).
- ☑ This waste does not contain regulated concentrations of TPH (oil and grease).
- ☑ This waste does not contain infectious wastes as defined by Minnesota Rules, Chapter 7035.

Generator Name: Enbridge Energy, Limited Partnership, LLC

Contractor/Generator Signature: [signature]

Title: Operation Team Lead Date: 11/29/2011

Common Name of Waste: Crude impacted soil

Attachment A

Master Enbridge Energy Soil Disposal Profile

Enbridge Energy, Limited Partnership

Generating site Address or Site ID:
Tank 5 - Enbridge Superior Terminal

Estimated Quantity: 250 cubic yards

Representative Sample ID Numbers: Tank 5-Stockpile-1 & 2

Requested Disposal Facility: Canyon Landfill

By completing this form the generator certifies that the waste is identical to that described on the Master Enbridge Energy Soil Disposal Profile signed on 11/29/2011, is the result of spills and/or leaks in uncontaminated soil and is not a hazardous waste, does not contain regulated radioactive material or regulated concentrations of PCBs. Any spills and/or leaks in any area with potential for additional contamination will be identified and profiled separately.

Project Reference Number (to appear on invoice): Tank 5 - 11/2011 - 49161092

*project number and site ID must appear on the invoice

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

November 29, 2011

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

RE: Project: 49161092 TANK 5
Pace Project No.: 10176510

Dear Andrea Nord:
Enclosed are the analytical results for sample(s) received by the laboratory on November 22, 2011. 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 TANK 5
Pace Project No.: 10176510

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
EPA Region 8 Certification #: Pace
Florida/NELAP Certification #: E87605
Georgia Certification #: 959
Idaho Certification #: MN00064
Illinois Certification #: 200011
Iowa Certification #: 368
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 Mexico Certification #: Pace
New York Certification #: 11647
North Carolina Certification #: 530
North Dakota Certification #: R-036
North Dakota Certification #: R-036A
Ohio VAP Certification #: CL101
Oklahoma Certification #: D9921
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Tennessee Certification #: 02818
Texas Certification #: T104704192
Washington Certification #: C754
Wisconsin Certification #: 999407970

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

SAMPLE SUMMARY

Project: 49161092 TANK 5
Pace Project No.: 10176510

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10176510001	**TANK 5-STOCKPILE-1**	Solid	11/17/11 15:20	11/22/11 09:55
10176510002	**TANK 5-STOCKPILE-2**	Solid	11/17/11 15:25	11/22/11 09:55

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

SAMPLE ANALYTE COUNT

Project: 49161092 TANK 5
Pace Project No.: 10176510

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10176510001	**TANK 5-STOCKPILE-1**	WI MOD DRO	JRH	2	PASI-M
		WI MOD GRO	MJH	5	PASI-M
		% Moisture	JDL	1	PASI-M
10176510002	**TANK 5-STOCKPILE-2**	WI MOD DRO	JRH	2	PASI-M
		WI MOD GRO	MJH	5	PASI-M
		% Moisture	JDL	1	PASI-M

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

ANALYTICAL RESULTS

Project: 49161092 TANK 5
Pace Project No.: 10176510

Sample: TANK 5-STOCKPILE-1 **Lab ID: 10176510001** Collected: 11/17/11 15:20 Received: 11/22/11 09:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS Silica Gel	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO								
Diesel Range Organics	**1190**	mg/kg	133	66.7	10	11/23/11 07:23	11/27/11 13:57		T6
Surrogates									
n-Triacontane (S)	69	%	38-125		10	11/23/11 07:23	11/27/11 13:57		2M
WIGRO GCV	Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext.								
Benzene	**0.10**	mg/kg	0.066	0.029	1	11/22/11 15:54	11/23/11 18:17	71-43-2	
Ethylbenzene	**0.52**	mg/kg	0.066	0.025	1	11/22/11 15:54	11/23/11 18:17	100-41-4	
Toluene	**0.23**	mg/kg	0.066	0.028	1	11/22/11 15:54	11/23/11 18:17	108-88-3	
Xylene (Total)	**2.0**	mg/kg	0.20	0.066	1	11/22/11 15:54	11/23/11 18:17	1330-20-7	
Surrogates									
a,a,a-Trifluorotoluene (S)	66	%	80-125		1	11/22/11 15:54	11/23/11 18:17	98-08-8	1M
Dry Weight	Analytical Method: % Moisture								
Percent Moisture	**24.2**	%	0.10	0.10	1		11/28/11 00:00		

Sample: TANK 5-STOCKPILE-2 **Lab ID: 10176510002** Collected: 11/17/11 15:25 Received: 11/22/11 09:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS Silica Gel	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO								
Diesel Range Organics	**204**	mg/kg	12.4	6.2	1	11/23/11 07:23	11/26/11 17:11		T6
Surrogates									
n-Triacontane (S)	72	%	38-125		1	11/23/11 07:23	11/26/11 17:11		2M
WIGRO GCV	Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext.								
Benzene	**0.20**	mg/kg	0.070	0.031	1	11/22/11 15:54	11/23/11 18:40	71-43-2	
Ethylbenzene	**0.27**	mg/kg	0.070	0.027	1	11/22/11 15:54	11/23/11 18:40	100-41-4	
Toluene	**0.10**	mg/kg	0.070	0.029	1	11/22/11 15:54	11/23/11 18:40	108-88-3	
Xylene (Total)	**1.1**	mg/kg	0.21	0.070	1	11/22/11 15:54	11/23/11 18:40	1330-20-7	
Surrogates									
a,a,a-Trifluorotoluene (S)	82	%	80-125		1	11/22/11 15:54	11/23/11 18:40	98-08-8	
Dry Weight	Analytical Method: % Moisture								
Percent Moisture	**24.6**	%	0.10	0.10	1		11/28/11 00:00		

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Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA

Project: 49161092 TANK 5
Pace Project No.: 10176510

QC Batch: GCV/8690 Analysis Method: WI MOD GRO
QC Batch Method: TPH GRO/PVOC WI ext. Analysis Description: WIGRO Solid GCV
Associated Lab Samples: 10176510001, 10176510002

METHOD BLANK: 1104285 Matrix: Solid
Associated Lab Samples: 10176510001, 10176510002

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Benzene	mg/kg	ND	0.050	11/23/11 16:00	
Ethylbenzene	mg/kg	ND	0.050	11/23/11 16:00	
Toluene	mg/kg	ND	0.050	11/23/11 16:00	
Xylene (Total)	mg/kg	ND	0.15	11/23/11 16:00	
a,a,a-Trifluorotoluene (S)	%	97	80-125	11/23/11 16:00	

LABORATORY CONTROL SAMPLE & LCSD: 1104286 1104287

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
Benzene	mg/kg	5	5.4	5.1	108	101	80-120	6	20	
Ethylbenzene	mg/kg	5	5.2	5.0	103	99	80-120	4	20	
Toluene	mg/kg	5	5.3	5.1	106	102	80-120	4	20	
Xylene (Total)	mg/kg	15	15.7	15.2	105	101	80-120	3	20	
a,a,a-Trifluorotoluene (S)	%				97	96	80-125			

MATRIX SPIKE SAMPLE: 1104288

Parameter	Units	10176383001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Benzene	mg/kg	ND	5	6.5	129	80-120	M1
Ethylbenzene	mg/kg	ND	5	6.3	125	80-120	M1
Toluene	mg/kg	ND	5	6.4	128	80-120	M1
Xylene (Total)	mg/kg	ND	15	19.0	126	80-120	ES
a,a,a-Trifluorotoluene (S)	%				98	80-125	

SAMPLE DUPLICATE: 1104289

Parameter	Units	10176383002 Result	Dup Result	RPD	Max RPD	Qualifiers
Benzene	mg/kg	ND	ND		20	
Ethylbenzene	mg/kg	ND	ND		20	
Toluene	mg/kg	ND	ND		20	
Xylene (Total)	mg/kg	ND	ND		20	
a,a,a-Trifluorotoluene (S)	%	96	96	21		

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QUALITY CONTROL DATA

Project: 49161092 TANK 5
Pace Project No.: 10176510

QC Batch: MPRP/29997
QC Batch Method: % Moisture
Analysis Method: % Moisture
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10176510001, 10176510002

SAMPLE DUPLICATE: 1105888

Parameter	Units	10175623001 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	18.1	18.0	.6	30	

SAMPLE DUPLICATE: 1105889

Parameter	Units	10176510002 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	24.6	24.5	.4	30	

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QUALITY CONTROL DATA

Project: 49161092 TANK 5
Pace Project No.: 10176510

QC Batch: OEXT/17328 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO Solid GCV
Associated Lab Samples: 10176510001, 10176510002

METHOD BLANK: 1104618 Matrix: Solid
Associated Lab Samples: 10176510001, 10176510002

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Diesel Range Organics	mg/kg	ND	10.0	11/26/11 16:23	
n-Triacontane (S)	%	78	38-125	11/26/11 16:23	

LABORATORY CONTROL SAMPLE & LCSD: 1104619 1104620

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
Diesel Range Organics	mg/kg	80	58.0	57.8	73	72	70-125	.5	20	
n-Triacontane (S)	%				78	75	38-125			

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QUALIFIERS

Project: 49161092 TANK 5
Pace Project No.: 10176510

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.

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.

LABORATORIES

PASI-M	Pace Analytical Services - Minneapolis

ANALYTE QUALIFIERS

1M	Surrogate recovery outside laboratory control limits due to matrix interferences.
2M	The sample was re-weighed into a new container because the original container was not the standard tared 4oz amber jar.
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.
T6	High boiling point hydrocarbons are present in the sample.

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161092 TANK 5
Pace Project No.: 10176510

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10176510001	**TANK 5-STOCKPILE-1**	WI MOD DRO	OEXT/17328	WI MOD DRO	GCSV/8909
10176510002	**TANK 5-STOCKPILE-2**	WI MOD DRO	OEXT/17328	WI MOD DRO	GCSV/8909
10176510001	**TANK 5-STOCKPILE-1**	TPH GRO/PVOC WI ext.	GCV/8690	WI MOD GRO	GCV/8691
10176510002	**TANK 5-STOCKPILE-2**	TPH GRO/PVOC WI ext.	GCV/8690	WI MOD GRO	GCV/8691
10176510001	**TANK 5-STOCKPILE-1**	% Moisture	MPRP/29997		
10176510002	**TANK 5-STOCKPILE-2**	% Moisture	MPRP/29997		

Chain of Custody

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

RUSH! 11/22/11 CC

1076510

COC 1 of 1

Project Number: 49161092

Project Name: Tank 5

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

COC Number: № 32221

Project Manager: HAW

Project QC Contact: AAN

Sampled by: RLE

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	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; 3 blank)	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.); 2 blank)	Total Number Of Containers	
1. Tank5-stockpile-1	—	—	—	11/17/11	1520		X		X				XX X	3	DRO, BTEX, Moisture co1
2. Tank5-stockpile-2	—	—	—	↓	1525		X		X				XX X	3	↓ co2
3.															
4.															
5.															
6.															ASAP TAT
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
[signature]	(Y) N	11/21/11	1200	L. Carlson/Pace	11/22/11	955
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

Pace Analytical®	Document Name: **Sample Condition Upon Receipt Form**	Revised Date: 02Jun2011 Page 1 of 1
	Document Number: **F-L-213 Rev.01**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Barr Project # 10176510

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

Tracking #: 797759635817

Optional: Proj. Due Date: Proj. Name:

Custody Seal on Cooler/Box Present: ☐ yes ☑ no Seals intact: ☐ yes ☑ no

Packing Material: ☑ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other _____ Temp Blank: Yes ✓ No _____

Thermometer Used 80344042 or 80512447 **Type of Ice:** (Wet) Blue None ☐ Samples on ice, cooling process has begun

Cooler Temperature 3.3 **Biological Tissue is Frozen:** Yes No

Temp should be above freezing to 6°C

Date and Initials of person examining contents: 11/22/11 LC

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 & Signature on COC:	☐Yes ☑No ☐N/A	4.
Samples Arrived within Hold Time:	☑Yes ☐No ☐N/A	5.
Short Hold Time Analysis (<72hr):	☐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. Noncompliance are noted in 13.	☐Yes ☐No ☑N/A	13. ☐ HNO3 ☐ H2SO4 ☐ NaOH ☐ HCl Samp #
All containers needing preservation are found to be in compliance with EPA recommendation.	☐Yes ☐No ☑N/A	
Exceptions: VOA,Coliform, TOC, Oil and Grease, WI-DRO (water)	☐Yes ☑No	Initial when completed / Lot # of added preservative
Samples checked for dechlorination:	☐Yes ☐No ☑N/A	14.
Headspace in VOA Vials (>6mm):	☐Yes ☐No ☑N/A	15.
Trip Blank Present:	☐Yes ☑No ☐N/A	16.
Trip Blank Custody Seals Present	☐Yes ☑No ☐N/A	
Pace Trip Blank Lot # (if purchased): _____		

Client Notification/ Resolution: Field Data Required? Y / N

Person Contacted: _____ Date/Time: _____

Comments/ Resolution: _____

Project Manager Review: _____ **Date:** 11/22/11

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)

Customer Summary Report
Criteria: 11/02/2011 12:00 AM to 01/22/2014 11:59 PM
Business Unit Name: WM of Northern MN - Canyon - S05314 (USA)
User: rbaumann
Date: Jan 22 2014, 11:30:22 AM - Central Standard Time
Operation Type: All
Customer Name: All
Ticket Type: All
Customer Type: All
PMT Category: All
Profile: 102881MN

Ticket Date	Ticket ID	Cust Code	MAS Unique ID	Generator	Manifest	Profile	Truck
12/1/2011	200148	0064205	53721303004	148-ENBRIDGEPIPELINE		102881MN	.
Material Total	1						
12/1/2011	200150	0064205	53721303004	148-ENBRIDGEPIPELINE	89527	102881MN	2510
12/1/2011	200151	0064205	53721303004	148-ENBRIDGEPIPELINE	89525	102881MN	2520
12/1/2011	200152	0064205	53721303004	148-ENBRIDGEPIPELINE	89526	102881MN	2506
12/1/2011	200153	0064205	53721303004	148-ENBRIDGEPIPELINE	89528	102881MN	2513
12/1/2011	200169	0064205	53721303004	148-ENBRIDGEPIPELINE	89529	102881MN	2520
12/1/2011	200170	0064205	53721303004	148-ENBRIDGEPIPELINE	89530	102881MN	2506
12/1/2011	200175	0064205	53721303004	148-ENBRIDGEPIPELINE	89531	102881MN	2510
12/1/2011	200176	0064205	53721303004	148-ENBRIDGEPIPELINE	89532	102881MN	2513
12/1/2011	200182	0064205	53721303004	148-ENBRIDGEPIPELINE	89533	102881MN	2520
12/1/2011	200185	0064205	53721303004	148-ENBRIDGEPIPELINE	89534	102881MN	2506
12/1/2011	200187	0064205	53721303004	148-ENBRIDGEPIPELINE	89535	102881MN	2510
12/1/2011	200190	0064205	53721303004	148-ENBRIDGEPIPELINE	89536	102881MN	2513
Material Total	12						
Customer Total	13						
Ticket Totals	13						

Customer Summary Report
Criteria: 11/02/2011 12:00 AM to 01/22/2014 11:59 PM
Business Unit Name: WM of Northern MN - Canyon - S05314 (USA)
User: rbaumann
Date: Jan 22 2014, 11:30:22 AM - Central Standard Time
Operation Type: All
Customer Name: All
Ticket Type: All
Customer Type: All
PMT Category: All
Profile: 102881MN

Internal Customer	Loads	Yards	Tons

Customer Summary Report
Criteria: 11/02/2011 12:00 AM to 01/22/2014 11:59 PM

Business Unit Name: WM of Northern MN - Canyon - S05314 (USA)
User: rbaumann
Date: Jan 22 2014, 11:30:22 AM - Central Standard Time
Operation Type: All
Customer Name: All
Ticket Type: All
Customer Type: All
PMT Category: All
Profile: 102881MN

External Customer	Loads	Yards	Tons
ENBRIDGE PIPELINES	13	0	203.12

Material	Rate Unit	Tons
APV	EA	0
		0
C&D INDUSTRIAL-Tons	TON	19.7
C&D INDUSTRIAL-Tons	TON	18.45
C&D INDUSTRIAL-Tons	TON	18.6
C&D INDUSTRIAL-Tons	TON	18.24
C&D INDUSTRIAL-Tons	TON	18.3
C&D INDUSTRIAL-Tons	TON	18.19
C&D INDUSTRIAL-Tons	TON	17.42
C&D INDUSTRIAL-Tons	TON	17.6
C&D INDUSTRIAL-Tons	TON	17.87
C&D INDUSTRIAL-Tons	TON	15.34
C&D INDUSTRIAL-Tons	TON	11.58
C&D INDUSTRIAL-Tons	TON	11.83
		203.12
		203.12
		203.12