

Technical Memorandum

To: Karl Beaster, Enbridge Energy
From: Ryan Erickson
Subject: Enbridge Superior Terminal Tank 10 Contractor Pump Release
Date: September 19, 2013
Project: 49161227

This memorandum summarizes the response actions conducted by Barr Engineering (Barr), at the request of Enbridge Energy (Enbridge), following two crude oil releases near Tank 10 at the Enbridge Superior Terminal in Superior, Wisconsin (Figure 1).

Background

In the spring of 2013, an Enbridge maintenance contractor had two separate pump hose failures which resulted in crude oil releases to the ground surface near the southeast side of Tank 10 (Figure 2). The pump was located on the gravel ring road surrounding the tank and was being used to transfer crude oil from Tank 11 to Tank 10 during routine tank maintenance activities.

The first release occurred on May 19, 2013 when a pump hose ruptured. According to Enbridge personnel, approximately 10 to 15 gallons of crude oil was released onto the ground surface. The second release occurred on June 11, 2013 when a pump hose was removed from the pump by the contractor. The contractor reported to Enbridge personnel that approximately one quart of crude oil sprayed onto the ground surface. Additional crude oil was released and captured in the pumps secondary containment.

Enbridge reported both releases to the Wisconsin Department of Natural Resources (WDNR) after they occurred. Enbridge requested that Barr assist with the initial site assessment, remedial excavation oversight and waste disposal coordination for both release events.

Release Response

Enbridge personnel and the maintenance contractors immediately responded to each release. Response efforts included using a vacuum truck and oil absorbent pads to remove free product from the ground surface and the secondary containment (Photo 6) and the use of hand tools to excavate crude impacted

gravel and soil (Photo 7). Crude oil recovered with the vacuum truck was injected back into the pipeline system. The crude oil impacted gravel that was excavated was stockpiled in the terminal soil management area (SMA) (Photo 4; Figure 2) until offsite disposal could be coordinated.

Barr was onsite on May 20 and June 11, 2013 to field screen the initial response remedial excavations to determine whether residual crude oil impacts were still present and to assist with the waste disposal process. Barr field screened gravel and soil from the excavations for the presence of organic vapors with a photoionization detector (PID) and evidence of crude oil impacts such as odor, visual discoloration and sheen were also noted (Attachment A). Barr observed that the initial Enbridge release response actions removed the majority of the crude oil impacted soil; however, gravel and soil with residual crude oil impacts remained (Attachment A). Additional remedial excavation activities would be required to remove the remaining impacts. The additional remedial excavation work could not be completed immediately after the releases due to weather conditions and excavation safety requirements. To prevent potential residual contaminant migration caused by rain water, Enbridge covered the impacted area with plastic and placed oil absorbent booms around the perimeter (Photo 1).

Remedial Excavations

The follow up remedial excavations for the two releases occurred on June 3, 2013 and on July 11, 2013, respectively. Barr field screened the extents of both excavations, and the gravel and soil with evidence of crude oil impacts such as headspace greater than ten parts per million (ppm), petroleum odor, visual discoloration and sheen were noted and removed. In both remedial excavations, the crude oil impacts were limited to the tank ring road material which consisted of approximately 1 to 1.5 feet of gravel roadbed fill overlying red clay soil/fill. The crude oil impacted gravel and soil that was excavated was stockpiled in the terminal SMA until offsite disposal could be coordinated (Photo 4; Figure 2). Analytical soil samples were also collected from each excavation. Additional details about each remedial excavation are summarized below.

June 3, 2013 Remedial Excavation

On June 3, 2013, gravel and soil that was impacted from the May 19 pump release were excavated by an Enbridge contractor. Based on Barr field screening results, an area approximately 24 feet long by 5-15 feet wide by 1.5 feet deep was excavated as shown in Photos 2 and 3, Figures 2 and 3 and Attachment A.

Field screening results from the final excavation extent identified no soil with a headspace greater than ten ppm, a petroleum odor or staining (Attachment A). A total of approximately 15 tons of crude oil impacted gravel and soil was excavated and temporarily stockpiled at the terminal SMA until it could be approved for offsite disposal (Photo 4).

Five analytical soil samples (TK10-Scrap-1 through TK10-Scrape-5) were collected from the excavation bottom (Figure 3; Attachment A) on June 3, 2013 and were submitted to Pace Analytical Services in Minneapolis, Minnesota for analysis of diesel range organics (DRO) and petroleum volatile organic compounds (PVOC). Only three low level DRO detections (<20 mg/kg) were reported above method detection limits. These results are summarized in Table 1 and the laboratory report is provided in Attachment B. No sidewall samples were collected due to the shallow nature of the remedial excavation/scrape (the maximum depth of the excavation was approximately 1.5 feet bgs).

The excavation was backfilled with clean material after the completion of field screening and sampling (Photo 5).

July 11, 2013 Remedial Excavation

On July 11, 2013, impacted gravel and soil from the June 11 pump release (Photo 8) was excavated by an Enbridge contractor. Based on Barr field screening results, an area approximately 5 feet long x 4 feet wide x 1.75 feet deep was excavated as shown in Photo 9, Figures 2 and 3 and Attachment A. Field screening results from the final excavation extent identified no soil with a headspace greater than ten ppm, a petroleum odor or staining (Attachment A). Approximately five tons of crude oil impacted gravel and soil was excavated and temporarily stockpiled at the terminal SMA until it could be approved for offsite disposal.

Analytical soil sample TK10-Scrape-6 was collected from the excavation bottom (Figure 3; Attachment A) on July 11, 2013 and was submitted to Pace Analytical Services for analysis of petroleum volatile organic compounds (PVOC). There were no PVOCs detected above method detection limits. The results are summarized on Table 1 and the laboratory report is provided in Attachment B.

The excavation was backfilled with clean material after the completion of field screening and sampling (Photo 10).

Waste Disposal Coordination

One representative soil sample was collected from the contaminated stockpile (Photo 4) by Barr on June 3, 2013 for waste characterization profiling. The sample was submitted to Legend Technical Services in St. Paul, Minnesota for laboratory analysis of diesel range organics (DRO) and benzene, toluene, ethylbenzene, and xylenes (BTEX). Upon receipt of the analytical results, the laboratory report was submitted to the Shamrock Landfill in Cloquet, Minnesota as part of the waste profile application (Attachment C) and the waste profile was accepted (profile #CL13-0025). Soil impacted in the June 11, 2013 release was included in the CL13-0025 profile based on generator knowledge. A total of 20.24 tons of contaminated soil was hauled to the landfill between June and August of 2013 (Attachment C).

Conclusions

The gravel and soil that had been impacted during the two separate Tank 10 pump release events was excavated and disposed of at an approved landfill facility. Residual soil impacts were not identified in the final excavation extents through field screening and the analytical soil samples did not result in PVOC concentrations above laboratory detection limits. Based on this information it appears that the response to the crude oil release sufficiently addressed the impacted soil and no further action is recommended.

Attachments:

Site Photos: 1-10

Figure 1: Tank 10 Site Location

Figure 2: Tank 10 Release Location Map

Figure 3: Tank 10 Site Layout Map

Table 1: Soil Analytical Data Summary - Tank 10 Excavation Samples

Attachment A: Enbridge Site Investigation Field Sampling and Screening Log

Attachment B: Pace Laboratory Report for the Excavation Extent Analytical Samples

Attachment C: Waste Disposal Documentation

SITE PHOTOS:

May 19, 2013 release response and remediation



Photo 1: Plastic sheeting and absorbent boom installed to prevent contaminant migration (May 20, 2013).



Photo 2

Photo 3

Photos 2 and 3: Remedial excavation activities at Tank 10 (June 3, 2013).



Photo 4

Photo 5

Photo 4: Approximately 5 cubic yards of Tank 10 crude oil impacted gravel and soil (June 3, 2013).

Photo 5: The backfilled Tank 10 release site (June 3, 2013).

June 11, 2013 release response and remediation



Photo 6



Photo 7

Photo 6: Contractor pump release response activities. The pump, oil absorbent pads and a vacuum truck are shown (June 11, 2013).

Photo 7: Hand tool excavation of crude oil impacted soil during the release response (June 11, 2013).



Photo 8



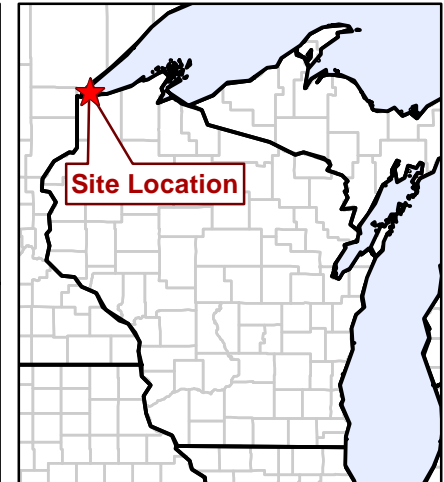
Photo 9

Photo 8: Crude oil stained ground prior to remedial excavation work (July 11, 2013).

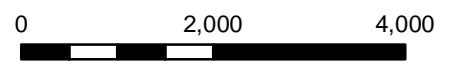
Photo 9: The Tank 10 remedial excavation (July 11, 2013).



Photo 10: The backfilled Tank 10 release site (July 11, 2013).



- Tank 10
- Terminal Property Boundary



Feet
1 Inch = 2,000 Feet

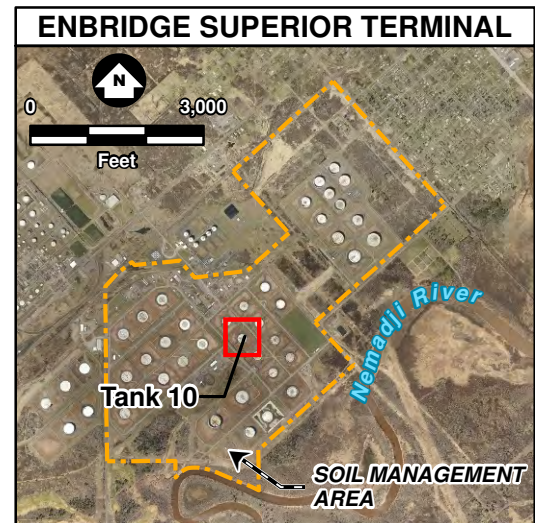
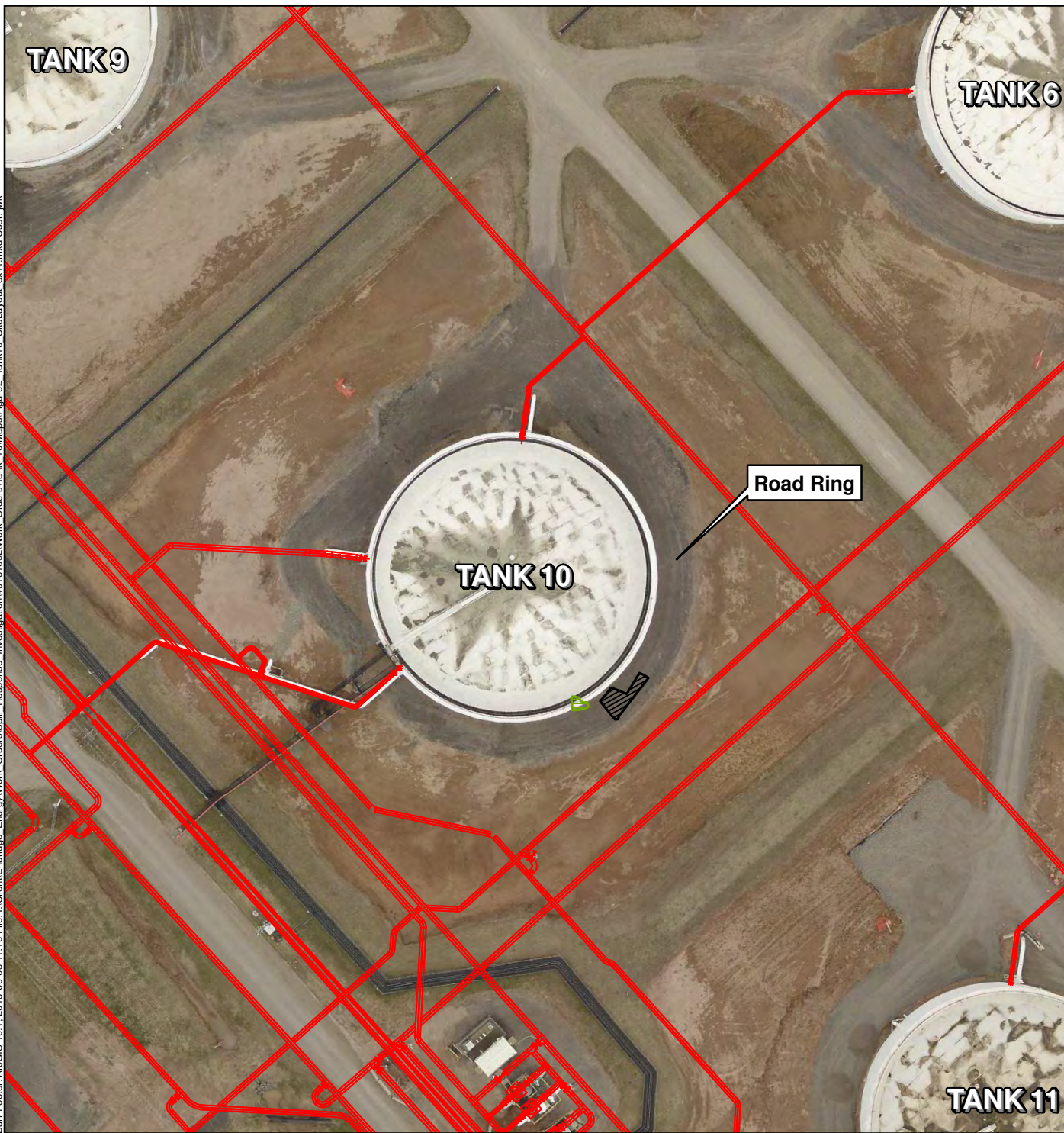
Figure 1





TANK 10 SITE LOCATION
SUPERIOR TERMINAL
 Enbridge Energy, L.P.
 Superior, Wisconsin

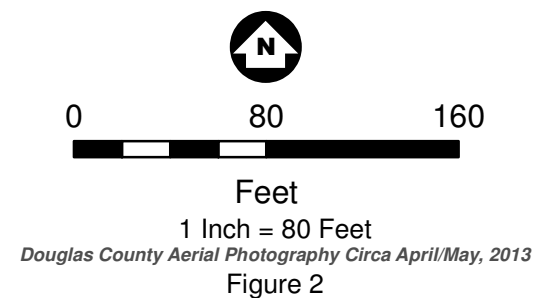


Barr Footer: ArcGIS 10.1, 2013-06-04 13:56 File: I:\Client\Enbridge_Energy\Work_Orders\Spill_Response_Investigation\49161092\Work_Orders\Tank_10\Maps\Figure1_Tank10_SiteLocation_8x11.mxd User: jrv

Barr Footer: ArcGIS 10.1, 2013-09-05 11:16 File: I:\Client\Enbridge_Energy\Work_Orders\Spill_Response_Investigation\49161092\Work_Orders\Tank_10\Map\Figure2_Tank10_SiteLayout_8x11.mxd User: jmk

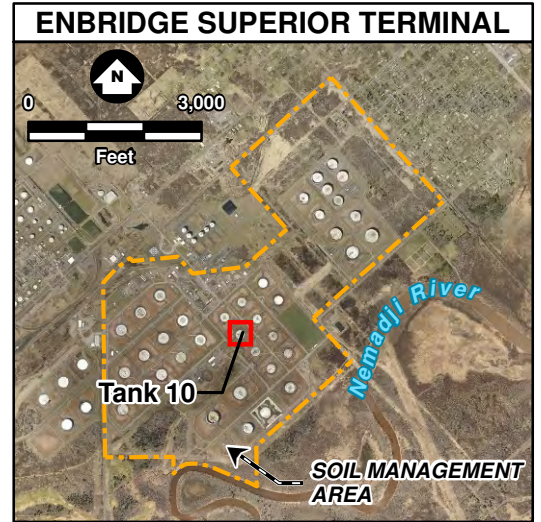
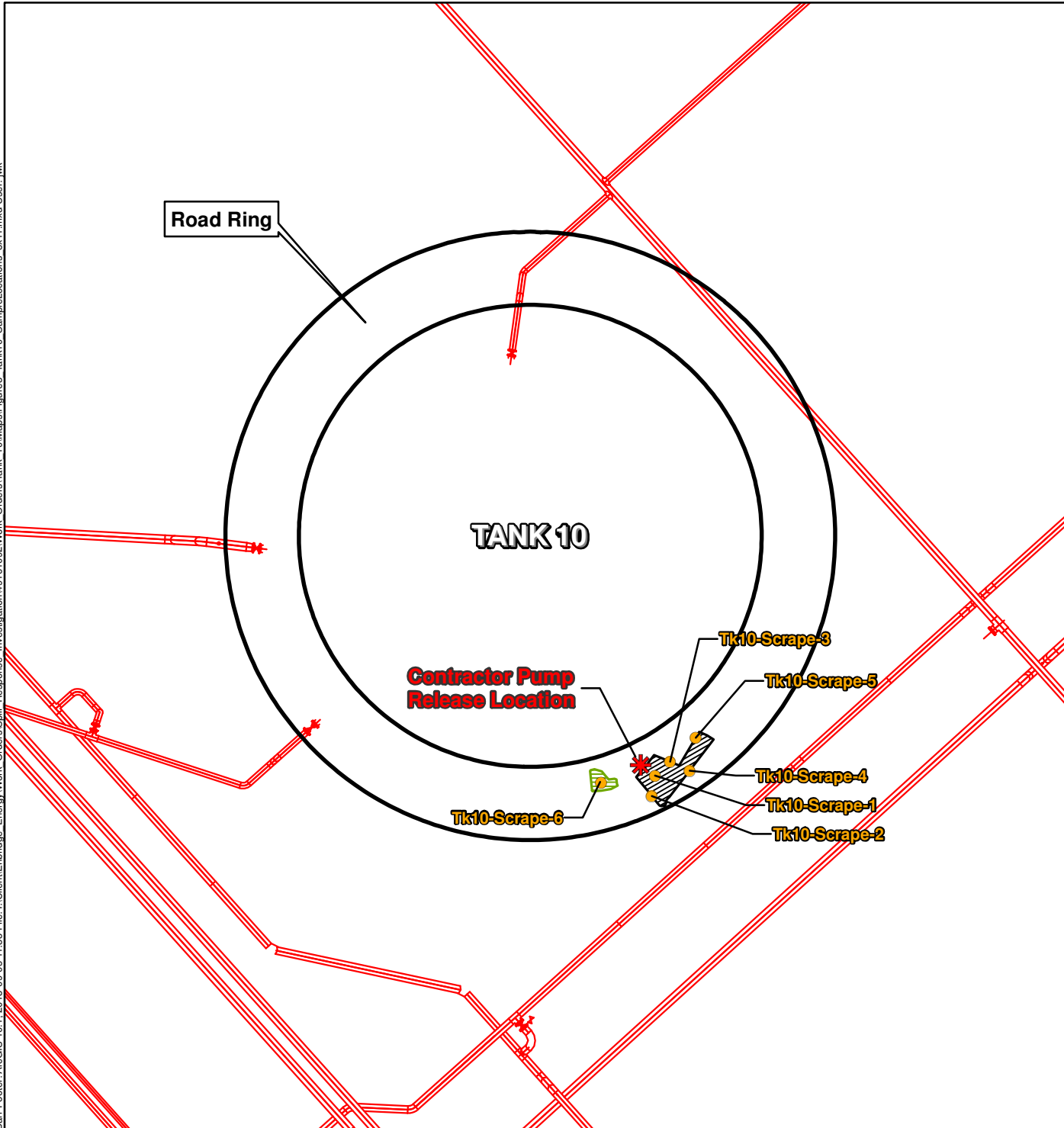


-  05/19/13 Release Remedial Excavation
-  06/11/13 Release Remedial Excavation
-  Pipeline Infrastructure
-  Terminal Property Boundary

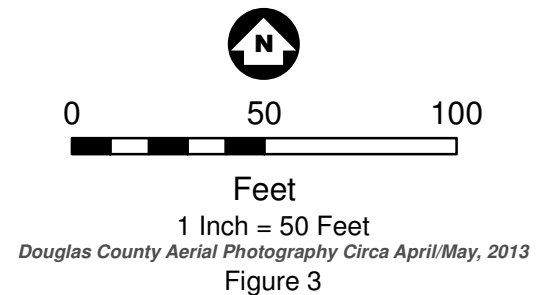


**TANK 10 SITE LAYOUT MAP
SUPERIOR TERMINAL**
Enbridge Energy, L.P.
Superior, Wisconsin





- Release Location
- Analytical Sample Locations
- 05/19/13 Release Remedial Excavation
- 06/11/13 Release Remedial Excavation
- Road Ring Boundary
- Pipeline Infrastructure
- Terminal Property Boundary



**TANK 10 SAMPLE LOCATIONS
SUPERIOR TERMINAL**
Enbridge Energy, L.P.
Superior, Wisconsin



Table 1
Soil Analytical Data Summary
Tank 10 Excavation Samples
Enbridge Superior Terminal, Superior, WI
Units, mg/kg (unless otherwise noted)

| Chemical Name | | | Moisture | 1,2,4-Trimethyl benzene | 1,3,5-Trimethyl benzene | Benzene | Ethyl benzene | Toluene | Xylene, total | Diesel Range Organics |
|--|----------------|-------------------|----------|-------------------------|-------------------------|---------|---------------|---------|---------------|-----------------------|
| Effective Date | Exceedance Key | | | | | | | | | |
| Wisconsin Direct Contact Levels NR 746.06 | 9/1/2007 | No Exceed | | | | 1.10 | | | | |
| Wisconsin Generic Residual Contaminant Levels NR 720.09 | 4/1/1997 | No Exceed | | | | 0.0055 | 2.9 | 1.5 | 4.1 | 250 (1) |
| Location | Date | Depth (ft) | | | | | | | | |
| TK10-Scrape-1 | 6/03/2013 | 1.5 | 15.0 % | < 0.068 | < 0.068 | < 0.068 | < 0.068 | < 0.068 | < 0.20 | < 12.4 |
| TK10-Scrape-2 | 6/03/2013 | 1.5 | 14.5 % | < 0.060 | < 0.060 | < 0.060 | < 0.060 | < 0.060 | < 0.18 | 12.7 |
| TK10-Scrape-3 | 6/03/2013 | 1.5 | 14.0 % | < 0.065 | < 0.065 | < 0.065 | < 0.065 | < 0.065 | < 0.20 | 17.3 |
| TK10-Scrape-4 | 6/03/2013 | 1.5 | 21.3 % | < 0.065 | < 0.065 | < 0.065 | < 0.065 | < 0.065 | < 0.20 | 14.2 |
| TK10-Scrape-5 | 6/03/2013 | 1.5 | 20.4 % | < 0.074 | < 0.074 | < 0.074 | < 0.074 | < 0.074 | < 0.22 | < 13.0 |
| TK10-Scrape-6 | 7/12/2013 | 1.75 | 8.6 % | < 0.055 | < 0.055 | < 0.055 | < 0.055 | < 0.055 | < 0.16 | -- |

-- Not analyzed/not available.

Attachment A

Enbridge Site Investigation Field Sampling and Screening Log

ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Date: 5/20/13

Location: Milepost or Facility Superior Terminal Tank 10

Sampler: CSG 2

Equipment used: Photo -ionization detector with 10.6 eV lamp ^{model RAE 3000} Background Headspace: 0.5 ppm
0.1

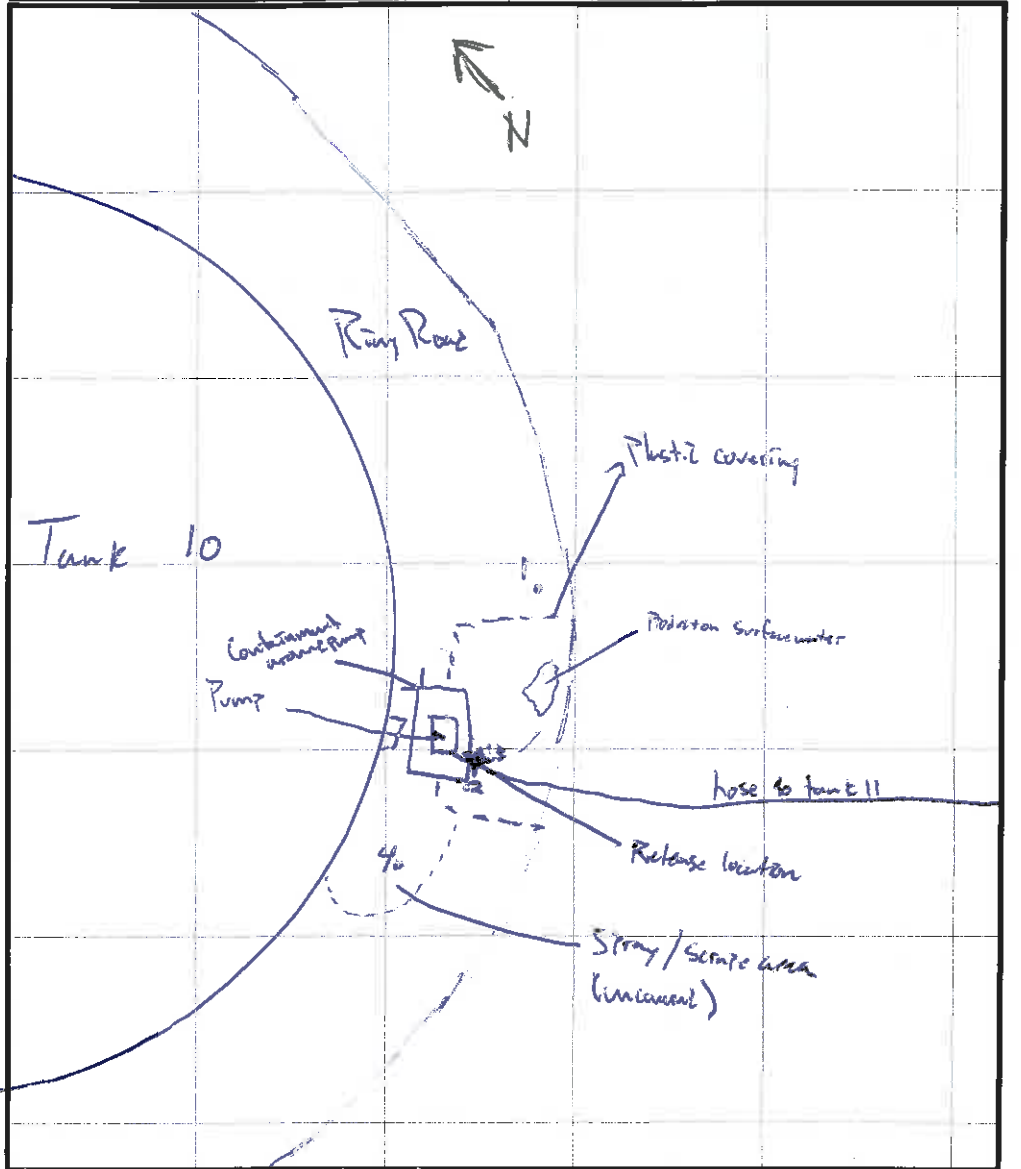
Calibration Time: 8:20

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

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

| Sample ID | Depth (ft) | Time (military) | Soil Type (USCS) | Color/ Discolor | Odor/ Sheen | Headspace Reading (ppm) |
|-------------|------------|-----------------|------------------|-----------------|-----------------------|-------------------------|
| Example R-1 | 4 | 16.30 | CL | Reddish brown | Petroleum/ Rainbow | 275 |
| 1 | 0.5 | 950 | GP/SM | Grey/black | no/no | 0.1 |
| 2 | 1.0 | 455 | GP | Grey | Yes/yes (faint) | 37.5 |
| 3 | 2.0 | 955 | SM | Grey | Yes/yes (faint) | 158.7 |
| 4 | 0.5 | 1000 | GP | Grey | no/slight sheen | 2.4 |
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SITE SKETCH: north is up; excavation extents and depths, sample locations, structures, utilities, boring locations, wells, natural features... **1 inch/grid = 30 FEET**



ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

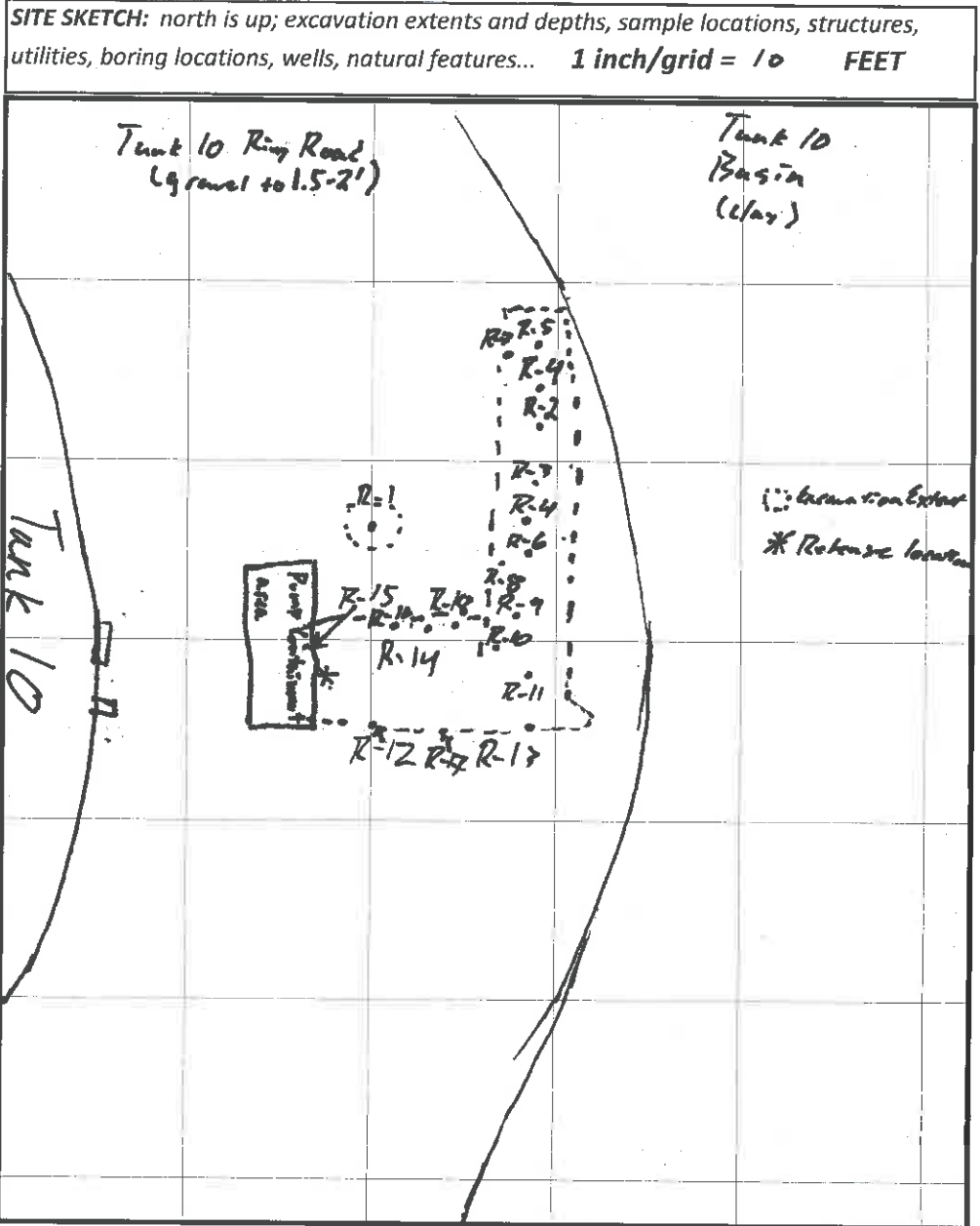
Location: Milepost or Facility Superior Terminal Tank 10 contractor Release
 Equipment used: Photo -ionization detector with 10.6 eV lamp Background Headspace: 0.0 ppm

Date: 6/3/13
 Sampler: CJOZ
 Calibration Time: 730

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

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

| Sample ID | Depth (ft) | Time (military) | Soil Type (USCS) | Color/Discolor | Odor/Sheen | Headspace Reading (ppm) |
|-------------|------------|-----------------|------------------|----------------|----------------------------|-------------------------|
| Example R-1 | 4 | 1630 | CL | Reddish brown | Petroleum/Rainbow | 275 |
| R-1 | 1.0 | 900 | SP | Red/brown | none/none | 0.2 |
| R-2 | 1.5 | 930 | GP | Gray | petroleum/white sheen | 86.7 |
| R-3 | 2.0 | 950 | LL | Red/brown | none/none | 0.0 |
| R-4 | 1.5 | 1000 | GP | Gray | petroleum/white sheen | 14.9 |
| R-5 | 1.5 | 1008 | GP | Gray | none/none | 0.9 |
| R-6 | 1.5 | 1010 | GP/CL | Red/brown | none/none | 0.6 |
| R-7 | 1.5 | 1015 | GP/LL | Gray | none/none | 1.5 |
| R-8 | 1.5 | 1030 | GP/LL | Red/brown | none/none | 0.8 |
| R-9 | 2.0 | 1130 | LL | Red/brown | none/none | 1.8 |
| R-10 | 1.5 | 1130 | LL | Red/brown | slight petrol/slight sheen | 22.8 |
| R-11 | 1.5 | 1200 | GP | Gray | none/slight | 2.4 |
| R-12 | 0.5 | 1200 | SP | Red/brown | none/none | 0.5 |
| R-13 | 1.0 | 1415 | SP | Red/brown | none/none | 0.4 |
| R-14 | 1.0 | 1415 | GP | Gray | none/none | 3.8 |
| R-15 | 1.5 | 1450 | SP/GP | Gray | none/none | 1.1 |
| R-16 | 1.5 | 1500 | GP | Gray | none/none | 4.7 |
| R-17 | 1.5 | 1500 | SP | Red/brown | none/none | 1.0 |
| R-18 | 1.5 | 1505 | GP | Gray | none/none | 3.7 |



ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Location: Milepost or Facility Tank 10 Contractor Release

Equipment used: Probe -ionization detector with 10.6 eV lamp

Background Headspace: 0.0 ppm

Date: 6/3/13

Sampler: ES62

Calibration Time: 7:0

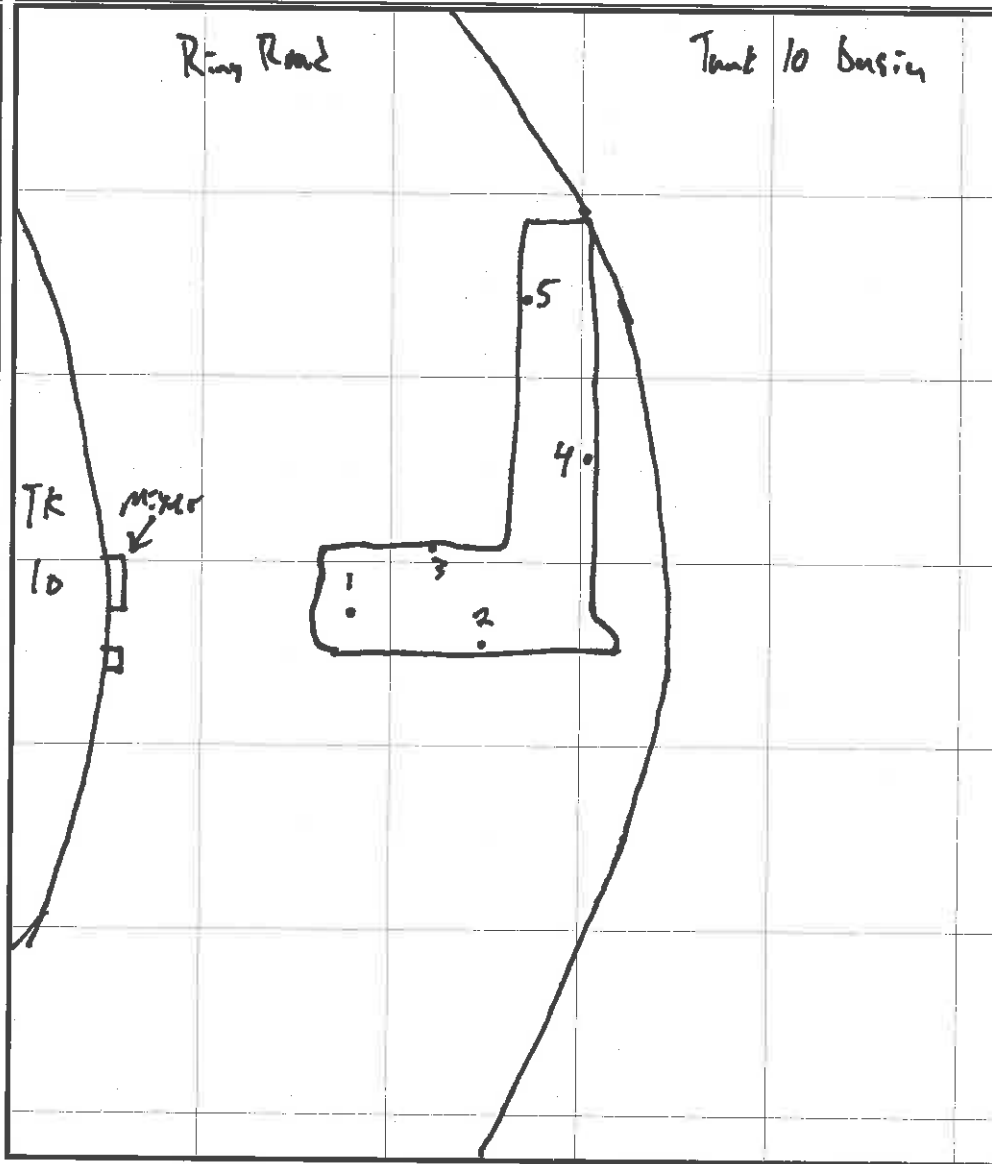
Sample Nomenclature (Location - sample type - #): Tank 10-Scrape-

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

| Sample ID | Depth (ft) | Time (military) | Soil Type (USCS) | Color/Discolor | Odor/Sheen | Headspace Reading (ppm) |
|-----------|------------|-----------------|------------------|----------------|------------|-------------------------|
|-----------|------------|-----------------|------------------|----------------|------------|-------------------------|

| Example R-1 | 4 | 16.30 | CL | Reddish brown | Petroleum/Rainbow | 275 |
|-------------|-----|-------|----|---------------|-------------------|-----|
| Scrape-1 | 1.5 | 1500 | CL | Red brown | none/none | 0.0 |
| Scrape-2 | 1.5 | 1505 | CL | Red brown | none/none | 0.1 |
| Scrape-3 | 1.5 | 1510 | CL | Red brown | none/none | 0.1 |
| Scrape-4 | 1.5 | 1515 | CL | Red brown | none/none | 0.0 |
| Scrape-5 | 1.5 | 1520 | CL | Red brown | none/none | 0.1 |

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



ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Location: Milepost or Facility Enbridge Superior Terminal, Tank 10
 Equipment used: photo -ionization detector with 10.6 eV lamp Background Headspace: 0.0 ppm

Date: 6/11/13

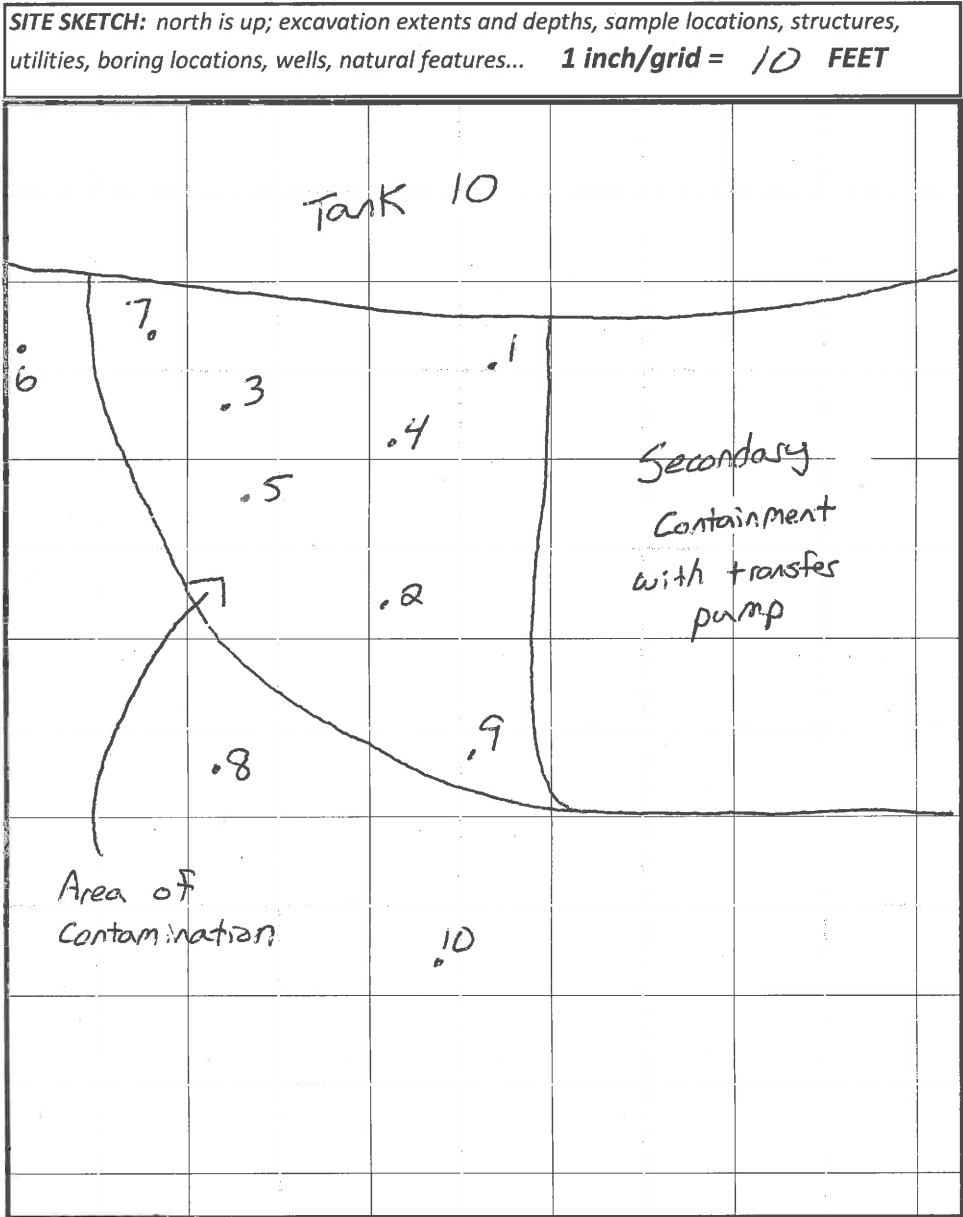
Sampler: BSL2

Calibration Time: —

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

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

| Sample ID | Depth (ft) | Time (military) | Soil Type (USCS) | Color/ Discolor | Odor/ Sheen | Headspace Reading (ppm) |
|--------------|------------|-----------------|------------------|-----------------|-----------------------|-------------------------|
| Example: R-1 | 4 | 16:30 | CL | Reddish brown | Petroleum/ Rainbow | 275 |
| 1 | Surface | 1245 | GP | Gray | Y/Y | 400+ |
| 2 | Surface | 1245 | GP | | Y/Y | 400+ |
| 3 | Surface | 1245 | GP | | Y/Y | 400+ |
| 4 | 1' | 1255 | GP | | Y/Y | 100+ |
| 5 | 1' | 1255 | GP | | Y/Y | 100+ |
| 6 | 0.5' | 1310 | GP | | N/N | 3.0 |
| 7 | Surface | 1315 | GP | | N/N | 14.6 |
| 8 | 0.25' | 1320 | GP | | N/N | 8.7 |
| 9 | 0.25' | 1325 | GP | | N/N | 135 |
| 10 | Surface | 1335 | GP | └ | N/N | 3.9 |
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ENBRIDGE SITE INVESTIGATION FIELD SAMPLING AND SCREENING LOG

Location: Milepost or Facility Tank 10 Contractor Release
 Equipment used: Photo -ionization detector with 10.6 eV lamp Background Headspace: 0.0 ppm

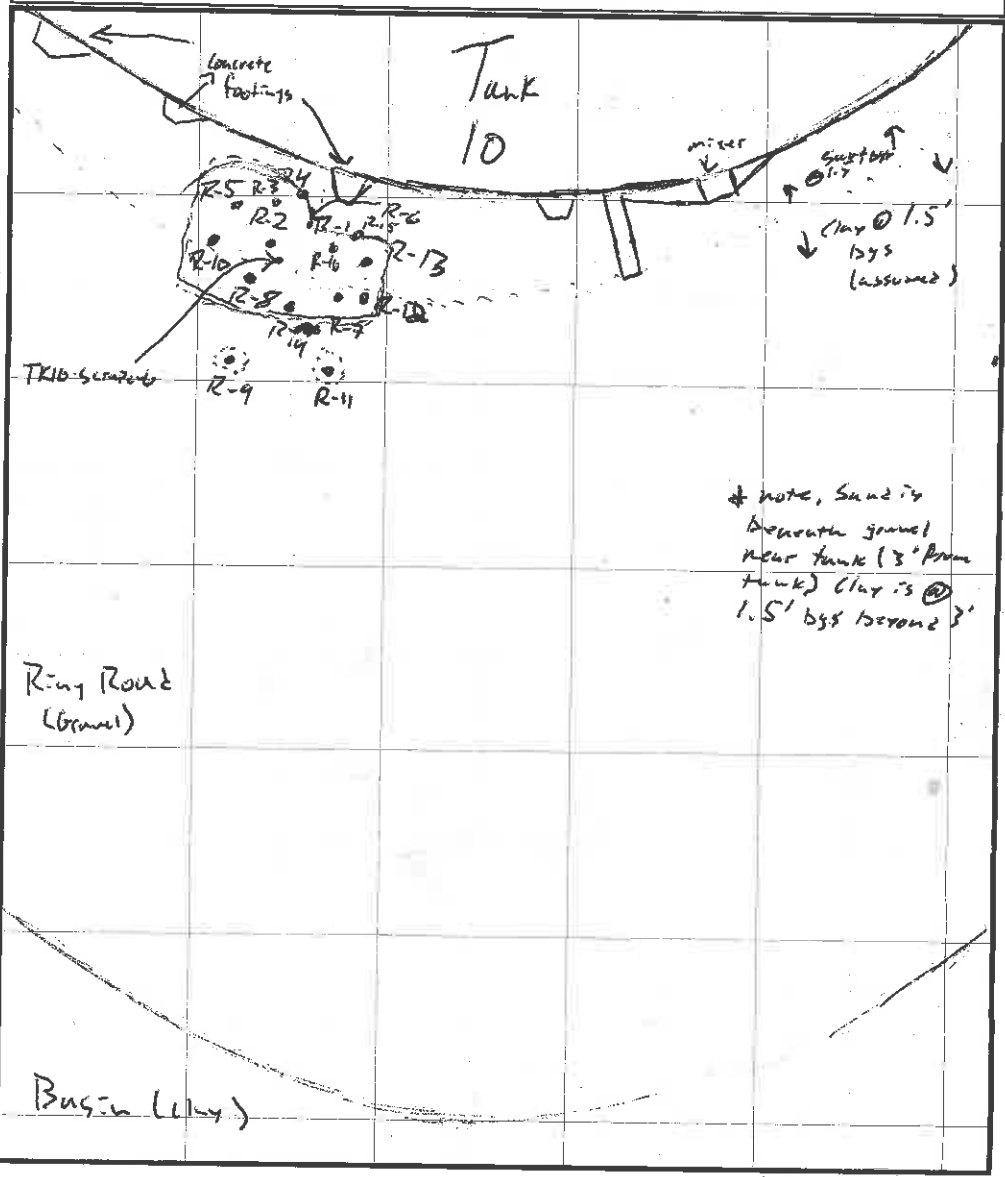
Date: 7/11/13
 Sampler: CT62
 Calibration Time: 1100

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

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

SITE SKETCH: north is up; excavation extents and depths, sample locations, structures, utilities, boring locations, wells, natural features... 1 inch/grid = 5 FEET

| 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 | 1.25 | 1200 | SP/LL | Red brown | none/none | 0.0 |
| R-2 | 1.25 | 1210 | SP/LL | Red brown | slight oil film sheen | 64.4 |
| R-3 | 1.25 | 1215 | SP/CL | Red brown | none/none | 1.8 |
| R-4 | 1.25 | 1215 | SP/CL | Red brown | none/none | 0.5 |
| R-5 | 1.5 | 1225 | SP/LL | Red brown | none/none | 0.0 |
| R-6 | 1.5 | 1225 | SP/LL | Red brown | none/none | 0.1 |
| R-7 | 1.75 | 1230 | CL | Red brown | none/none | 0.0 |
| R-8 | 1.5 | 1245 | CL | Red brown | none/none | 0.0 |
| R-9 | 1.5 | 1245 | CL | Red brown | none/none | 0.0 |
| R-10 | 1.5 | 1245 | CL/SP | Red brown | none/none | 0.0 |
| R-11 | 1.5 | 1305 | CL/SP | Red brown | none/none | 0.0 |
| R-12 | 1.5 | 1315 | LL | Red brown | none/none | 0.2 |
| R-13 | 1.5 | 1315 | CL/SP | Red brown | none/none | 0.0 |
| R-14 | 1.5 | 1315 | LL | Red brown | none/none | 0.5 |
| R-15 | 1.5 | 1325 | CL/SP | Red brown | none/slight | 0.2 |
| R-16 | 1.75 | 1335 | LL/SP | Red brown | none/slight | 0.3 |
| TK10-Slope-6 | 1.75 | 1405 | SP/LL | Red brown | none/none | 0.0 |



TK10-Slope-6
 ↓
 wcf (Mixer)

Attachment B

Pace Laboratory Report for the Excavation Extent Analytical Samples

July 02, 2013

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

RE: Project: 49/16-1227.00 Rev
Pace Project No.: 10230969

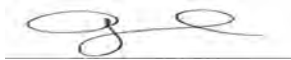
Dear Andrea Nord:

Enclosed are the analytical results for sample(s) received by the laboratory on June 05, 2013. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

This report was revised on July 2, 2013 to revise the project name.

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

Sincerely,



Andrea Opland

andrea.opland@pacelabs.com
Project Manager

Enclosures



REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,
without the written consent of Pace Analytical Services, Inc..

CERTIFICATIONS

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

Minnesota Certification IDs

1700 Elm Street SE Suite 200, Minneapolis, MN 55414

A2LA Certification #: 2926.01

Alaska Certification #: UST-078

Alaska Certification #MN00064

Arizona Certification #: AZ-0014

Arkansas Certification #: 88-0680

California Certification #: 01155CA

Colorado Certification #Pace

Connecticut Certification #: PH-0256

EPA Region 8 Certification #: Pace

Florida/NELAP Certification #: E87605

Georgia Certification #: 959

Hawaii Certification #Pace

Idaho Certification #: MN00064

Illinois Certification #: 200011

Kansas Certification #: E-10167

Louisiana Certification #: 03086

Louisiana Certification #: LA080009

Maine Certification #: 2007029

Maryland Certification #: 322

Michigan DEQ Certification #: 9909

Minnesota Certification #: 027-053-137

Mississippi Certification #: Pace

Montana Certification #: MT CERT0092

Nevada Certification #: MN_00064

Nebraska Certification #: Pace

New Jersey Certification #: MN-002

New York Certification #: 11647

North Carolina Certification #: 530

North Dakota Certification #: R-036

North Dakota Certification #: R-036A

Ohio VAP Certification #: CL101

Oklahoma Certification #: 9507

Oregon Certification #: MN200001

Oregon Certification #: MN300001

Pennsylvania Certification #: 68-00563

Puerto Rico Certification

Tennessee Certification #: 02818

Texas Certification #: T104704192

Utah Certification #: MN00064

Virginia/DCLS Certification #: 002521

Virginia/VELAP Certification #: 460163

Washington Certification #: C754

West Virginia Certification #: 382

Wisconsin Certification #: 999407970

REPORT OF LABORATORY ANALYSIS

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SAMPLE SUMMARY

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|-------------|---------------|--------|----------------|----------------|
| 10230969001 | TK10-Scrape-1 | Solid | 06/03/13 15:00 | 06/05/13 08:40 |
| 10230969002 | TK10-Scrape-2 | Solid | 06/03/13 15:05 | 06/05/13 08:40 |
| 10230969003 | TK10-Scrape-3 | Solid | 06/03/13 15:10 | 06/05/13 08:40 |
| 10230969004 | TK10-Scrape-4 | Solid | 06/03/13 15:15 | 06/05/13 08:40 |
| 10230969005 | TK10-Scrape-5 | Solid | 06/03/13 15:20 | 06/05/13 08:40 |

REPORT OF LABORATORY ANALYSIS

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SAMPLE ANALYTE COUNT

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|-------------|---------------|------------|----------|-------------------|------------|
| 10230969001 | TK10-Scrape-1 | WI MOD DRO | MT | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| 10230969002 | TK10-Scrape-2 | WI MOD DRO | MT | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| 10230969003 | TK10-Scrape-3 | WI MOD DRO | MT | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| 10230969004 | TK10-Scrape-4 | WI MOD DRO | MT | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |
| 10230969005 | TK10-Scrape-5 | WI MOD DRO | MT | 2 | PASI-M |
| | | WI MOD GRO | KT1 | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |

REPORT OF LABORATORY ANALYSIS

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PROJECT NARRATIVE

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

Method: WI MOD DRO

Description: WIDRO GCS

Client: Barr Engineering

Date: July 02, 2013

General Information:

5 samples were analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below.

Hold Time:

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

Sample Preparation:

The samples were prepared in accordance with WI MOD DRO with any exceptions noted below.

Initial Calibrations (including MS Tune as applicable):

All criteria were within method requirements with any exceptions noted below.

Continuing Calibration:

All criteria were within method requirements with any exceptions noted below.

Surrogates:

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

Method Blank:

All analytes were below the report limit in the method blank with any exceptions noted below.

Laboratory Control Spike:

All laboratory control spike compounds were within QC limits with any exceptions noted below.

QC Batch: OEXT/21915

R1: RPD value was outside control limits.

- LCSD (Lab ID: 1450698)
- Diesel Range Organics

Matrix Spikes:

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

Additional Comments:

Analyte Comments:

QC Batch: OEXT/21915

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

- TK10-Scrape-2 (Lab ID: 10230969002)
 - Diesel Range Organics
- TK10-Scrape-3 (Lab ID: 10230969003)
 - Diesel Range Organics
- TK10-Scrape-4 (Lab ID: 10230969004)
 - Diesel Range Organics

REPORT OF LABORATORY ANALYSIS

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PROJECT NARRATIVE

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

Method: WI MOD GRO

Description: WIGRO GCV

Client: Barr Engineering

Date: July 02, 2013

General Information:

5 samples were analyzed for WI MOD GRO. All samples were received in acceptable condition with any exceptions noted below.

Hold Time:

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

Sample Preparation:

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

Initial Calibrations (including MS Tune as applicable):

All criteria were within method requirements with any exceptions noted below.

Continuing Calibration:

All criteria were within method requirements with any exceptions noted below.

Internal Standards:

All internal standards were within QC limits with any exceptions noted below.

Surrogates:

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

Method Blank:

All analytes were below the report limit in the method blank with any exceptions noted below.

Laboratory Control Spike:

All laboratory control spike compounds were within QC limits with any exceptions noted below.

Matrix Spikes:

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

Duplicate Sample:

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

Additional Comments:

Analyte Comments:

QC Batch: GCV/10868

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

- DUP (Lab ID: 1449750)
- a,a,a-Trifluorotoluene (S)

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

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

Sample: TK10-Scrape-1 **Lab ID: 10230969001** Collected: 06/03/13 15:00 Received: 06/05/13 08:40 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 | <0.124 | mg/kg | 12.4 | 1.4 | 1 | 06/07/13 10:40 | 06/10/13 20:16 | | |
| Surrogates | | | | | | | | | |
| n-Triacontane (S) | 95 % | | 50-150 | | 1 | 06/07/13 10:40 | 06/10/13 20:16 | 638-68-6 | |
| WIGRO GCV Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | | |
| Benzene | <0.068 | mg/kg | 0.068 | 0.011 | 1 | 06/06/13 15:21 | 06/07/13 13:53 | 71-43-2 | |
| Ethylbenzene | <0.068 | mg/kg | 0.068 | 0.0095 | 1 | 06/06/13 15:21 | 06/07/13 13:53 | 100-41-4 | |
| Toluene | <0.068 | mg/kg | 0.068 | 0.0068 | 1 | 06/06/13 15:21 | 06/07/13 13:53 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | <0.068 | mg/kg | 0.068 | 0.011 | 1 | 06/06/13 15:21 | 06/07/13 13:53 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | <0.068 | mg/kg | 0.068 | 0.0095 | 1 | 06/06/13 15:21 | 06/07/13 13:53 | 108-67-8 | |
| Xylene (Total) | <0.20 | mg/kg | 0.20 | 0.023 | 1 | 06/06/13 15:21 | 06/07/13 13:53 | 1330-20-7 | |
| Surrogates | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 100 % | | 80-125 | | 1 | 06/06/13 15:21 | 06/07/13 13:53 | 98-08-8 | |
| Dry Weight Analytical Method: ASTM D2974 | | | | | | | | | |
| Percent Moisture | 15.0 % | | 0.10 | 0.10 | 1 | | 06/06/13 00:00 | | |

Sample: TK10-Scrape-2 **Lab ID: 10230969002** Collected: 06/03/13 15:05 Received: 06/05/13 08:40 Matrix: Solid

Results reported on a "dry-weight" basis

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|--|---------|-------|--------------|--------|----|----------------|----------------|-----------|------|
| WIDRO GCS Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | | | |
| Diesel Range Organics | 12.7 | mg/kg | 12.1 | 1.3 | 1 | 06/07/13 10:40 | 06/10/13 20:01 | | T6 |
| Surrogates | | | | | | | | | |
| n-Triacontane (S) | 94 % | | 50-150 | | 1 | 06/07/13 10:40 | 06/10/13 20:01 | 638-68-6 | |
| WIGRO GCV Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | | |
| Benzene | <0.060 | mg/kg | 0.060 | 0.0096 | 1 | 06/06/13 15:21 | 06/07/13 04:30 | 71-43-2 | |
| Ethylbenzene | <0.060 | mg/kg | 0.060 | 0.0084 | 1 | 06/06/13 15:21 | 06/07/13 04:30 | 100-41-4 | |
| Toluene | <0.060 | mg/kg | 0.060 | 0.0060 | 1 | 06/06/13 15:21 | 06/07/13 04:30 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | <0.060 | mg/kg | 0.060 | 0.0096 | 1 | 06/06/13 15:21 | 06/07/13 04:30 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | <0.060 | mg/kg | 0.060 | 0.0084 | 1 | 06/06/13 15:21 | 06/07/13 04:30 | 108-67-8 | |
| Xylene (Total) | <0.18 | mg/kg | 0.18 | 0.020 | 1 | 06/06/13 15:21 | 06/07/13 04:30 | 1330-20-7 | |
| Surrogates | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 103 % | | 80-125 | | 1 | 06/06/13 15:21 | 06/07/13 04:30 | 98-08-8 | |
| Dry Weight Analytical Method: ASTM D2974 | | | | | | | | | |
| Percent Moisture | 14.5 % | | 0.10 | 0.10 | 1 | | 06/06/13 00:00 | | |

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

Sample: TK10-Scrape-3 **Lab ID: 10230969003** Collected: 06/03/13 15:10 Received: 06/05/13 08:40 Matrix: Solid

Results reported on a "dry-weight" basis

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|--|---------|-------|--------------|--------|----|----------------|----------------|-----------|------|
| WIDRO GCS Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO | | | | | | | | | |
| Diesel Range Organics | 17.3 | mg/kg | 16.3 | 1.8 | 1 | 06/07/13 10:40 | 06/10/13 20:09 | | T6 |
| Surrogates | | | | | | | | | |
| n-Triacontane (S) | 96 | % | 50-150 | | 1 | 06/07/13 10:40 | 06/10/13 20:09 | 638-68-6 | |
| WIGRO GCV Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | | |
| Benzene | <0.065 | mg/kg | 0.065 | 0.010 | 1 | 06/06/13 15:21 | 06/07/13 04:49 | 71-43-2 | |
| Ethylbenzene | <0.065 | mg/kg | 0.065 | 0.0091 | 1 | 06/06/13 15:21 | 06/07/13 04:49 | 100-41-4 | |
| Toluene | <0.065 | mg/kg | 0.065 | 0.0065 | 1 | 06/06/13 15:21 | 06/07/13 04:49 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | <0.065 | mg/kg | 0.065 | 0.010 | 1 | 06/06/13 15:21 | 06/07/13 04:49 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | <0.065 | mg/kg | 0.065 | 0.0091 | 1 | 06/06/13 15:21 | 06/07/13 04:49 | 108-67-8 | |
| Xylene (Total) | <0.20 | mg/kg | 0.20 | 0.022 | 1 | 06/06/13 15:21 | 06/07/13 04:49 | 1330-20-7 | |
| Surrogates | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 103 | % | 80-125 | | 1 | 06/06/13 15:21 | 06/07/13 04:49 | 98-08-8 | |
| Dry Weight Analytical Method: ASTM D2974 | | | | | | | | | |
| Percent Moisture | 14.0 | % | 0.10 | 0.10 | 1 | | 06/06/13 00:00 | | |

Sample: TK10-Scrape-4 **Lab ID: 10230969004** Collected: 06/03/13 15:15 Received: 06/05/13 08:40 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 | 14.2 | mg/kg | 11.8 | 1.3 | 1 | 06/07/13 10:40 | 06/10/13 20:24 | | T6 |
| Surrogates | | | | | | | | | |
| n-Triacontane (S) | 106 | % | 50-150 | | 1 | 06/07/13 10:40 | 06/10/13 20:24 | 638-68-6 | |
| WIGRO GCV Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | | |
| Benzene | <0.065 | mg/kg | 0.065 | 0.010 | 1 | 06/06/13 15:21 | 06/07/13 05:09 | 71-43-2 | |
| Ethylbenzene | <0.065 | mg/kg | 0.065 | 0.0092 | 1 | 06/06/13 15:21 | 06/07/13 05:09 | 100-41-4 | |
| Toluene | <0.065 | mg/kg | 0.065 | 0.0065 | 1 | 06/06/13 15:21 | 06/07/13 05:09 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | <0.065 | mg/kg | 0.065 | 0.010 | 1 | 06/06/13 15:21 | 06/07/13 05:09 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | <0.065 | mg/kg | 0.065 | 0.0092 | 1 | 06/06/13 15:21 | 06/07/13 05:09 | 108-67-8 | |
| Xylene (Total) | <0.20 | mg/kg | 0.20 | 0.022 | 1 | 06/06/13 15:21 | 06/07/13 05:09 | 1330-20-7 | |
| Surrogates | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 101 | % | 80-125 | | 1 | 06/06/13 15:21 | 06/07/13 05:09 | 98-08-8 | |
| Dry Weight Analytical Method: ASTM D2974 | | | | | | | | | |
| Percent Moisture | 21.3 | % | 0.10 | 0.10 | 1 | | 06/06/13 00:00 | | |

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

Sample: TK10-Scrape-5 **Lab ID: 10230969005** Collected: 06/03/13 15:20 Received: 06/05/13 08:40 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 | <13.0 | mg/kg | 13.0 | 1.4 | 1 | 06/07/13 10:40 | 06/10/13 20:55 | | |
| Surrogates | | | | | | | | | |
| n-Triacontane (S) | 89 | % | 50-150 | | 1 | 06/07/13 10:40 | 06/10/13 20:55 | 638-68-6 | |
| WIGRO GCV | | | | | | | | | |
| Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | | |
| Benzene | <0.074 | mg/kg | 0.074 | 0.012 | 1 | 06/06/13 15:21 | 06/07/13 05:28 | 71-43-2 | |
| Ethylbenzene | <0.074 | mg/kg | 0.074 | 0.010 | 1 | 06/06/13 15:21 | 06/07/13 05:28 | 100-41-4 | |
| Toluene | <0.074 | mg/kg | 0.074 | 0.0074 | 1 | 06/06/13 15:21 | 06/07/13 05:28 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | <0.074 | mg/kg | 0.074 | 0.012 | 1 | 06/06/13 15:21 | 06/07/13 05:28 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | <0.074 | mg/kg | 0.074 | 0.010 | 1 | 06/06/13 15:21 | 06/07/13 05:28 | 108-67-8 | |
| Xylene (Total) | <0.22 | mg/kg | 0.22 | 0.025 | 1 | 06/06/13 15:21 | 06/07/13 05:28 | 1330-20-7 | |
| Surrogates | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 101 | % | 80-125 | | 1 | 06/06/13 15:21 | 06/07/13 05:28 | 98-08-8 | |
| Dry Weight | | | | | | | | | |
| Analytical Method: ASTM D2974 | | | | | | | | | |
| Percent Moisture | 20.4 | % | 0.10 | 0.10 | 1 | | 06/06/13 00:00 | | |

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

QC Batch: GCV/10868 Analysis Method: WI MOD GRO
 QC Batch Method: TPH GRO/PVOC WI ext. Analysis Description: WIGRO Solid GCV
 Associated Lab Samples: 10230969001, 10230969002, 10230969003, 10230969004, 10230969005

METHOD BLANK: 1449746 Matrix: Solid

Associated Lab Samples: 10230969001, 10230969002, 10230969003, 10230969004, 10230969005

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|----------------------------|-------|--------------|-----------------|----------------|------------|
| 1,2,4-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 06/07/13 01:15 | |
| 1,3,5-Trimethylbenzene | mg/kg | <0.050 | 0.050 | 06/07/13 01:15 | |
| Benzene | mg/kg | <0.050 | 0.050 | 06/07/13 01:15 | |
| Ethylbenzene | mg/kg | <0.050 | 0.050 | 06/07/13 01:15 | |
| Toluene | mg/kg | <0.050 | 0.050 | 06/07/13 01:15 | |
| Xylene (Total) | mg/kg | <0.15 | 0.15 | 06/07/13 01:15 | |
| a,a,a-Trifluorotoluene (S) | % | 102 | 80-125 | 06/07/13 01:15 | |

LABORATORY CONTROL SAMPLE & LCSD: 1449747 1449748

| 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 | 5.1 | 5.0 | 102 | 99 | 80-120 | 3 | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | 5 | 5.2 | 5.0 | 103 | 101 | 80-120 | 2 | 20 | |
| Benzene | mg/kg | 5 | 4.6 | 4.7 | 92 | 95 | 80-120 | 3 | 20 | |
| Ethylbenzene | mg/kg | 5 | 5.0 | 5.0 | 101 | 101 | 80-120 | .3 | 20 | |
| Toluene | mg/kg | 5 | 4.9 | 4.9 | 97 | 98 | 80-120 | .8 | 20 | |
| Xylene (Total) | mg/kg | 15 | 15.5 | 15.3 | 104 | 102 | 80-120 | 1 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 97 | 101 | 80-125 | | | |

MATRIX SPIKE SAMPLE: 1449749

| Parameter | Units | 10230979005 Result | Spike Conc. | MS Result | MS % Rec | % Rec Limits | Qualifiers |
|----------------------------|-------|--------------------|-------------|-----------|----------|--------------|------------|
| 1,2,4-Trimethylbenzene | mg/kg | 0.065 | 6.1 | 5.8 | 95 | 80-120 | |
| 1,3,5-Trimethylbenzene | mg/kg | ND | 6.1 | 5.9 | 96 | 80-120 | |
| Benzene | mg/kg | ND | 6.1 | 5.2 | 86 | 80-120 | |
| Ethylbenzene | mg/kg | ND | 6.1 | 5.7 | 94 | 80-120 | |
| Toluene | mg/kg | ND | 6.1 | 5.5 | 91 | 80-120 | |
| Xylene (Total) | mg/kg | ND | 18.2 | 17.6 | 97 | 80-120 | |
| a,a,a-Trifluorotoluene (S) | % | | | | 98 | 80-125 | |

SAMPLE DUPLICATE: 1449750

| Parameter | Units | 10230979006 Result | Dup Result | RPD | Max RPD | Qualifiers |
|------------------------|-------|--------------------|------------|-----|---------|------------|
| 1,2,4-Trimethylbenzene | mg/kg | 6.6 | 7.7 | 15 | 20 | |
| 1,3,5-Trimethylbenzene | mg/kg | 2.4 | 2.9 | 17 | 20 | |
| Benzene | mg/kg | ND | <0.061 | | 20 | |
| Ethylbenzene | mg/kg | 0.48 | 0.46 | 4 | 20 | |
| Toluene | mg/kg | 0.13 | 0.13 | 5 | 20 | |

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

SAMPLE DUPLICATE: 1449750

| Parameter | Units | 10230979006 Result | Dup Result | RPD | Max RPD | Qualifiers |
|----------------------------|-------|-----------------------|---------------|-----|------------|------------|
| Xylene (Total) | mg/kg | 3.1 | 3.1 | 3 | 20 | |
| a,a,a-Trifluorotoluene (S) | % | 54 | 60 | 15 | | 1M |

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

QC Batch: MPRP/39672

Analysis Method: ASTM D2974

QC Batch Method: ASTM D2974

Analysis Description: Dry Weight/Percent Moisture

Associated Lab Samples: 10230969001, 10230969002, 10230969003

SAMPLE DUPLICATE: 1449117

| Parameter | Units | 10230776001 Result | Dup Result | RPD | Max RPD | Qualifiers |
|------------------|-------|-----------------------|---------------|-----|------------|------------|
| Percent Moisture | % | 13.6 | 15.1 | 11 | 30 | |

SAMPLE DUPLICATE: 1449118

| Parameter | Units | 10230969003 Result | Dup Result | RPD | Max RPD | Qualifiers |
|------------------|-------|-----------------------|---------------|-----|------------|------------|
| Percent Moisture | % | 14.0 | 11.4 | 21 | 30 | |

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

QC Batch: MPRP/39673

Analysis Method: ASTM D2974

QC Batch Method: ASTM D2974

Analysis Description: Dry Weight/Percent Moisture

Associated Lab Samples: 10230969004, 10230969005

SAMPLE DUPLICATE: 1449127

| Parameter | Units | 10230969004 Result | Dup Result | RPD | Max RPD | Qualifiers |
|------------------|-------|-----------------------|---------------|-----|------------|------------|
| Percent Moisture | % | 21.3 | 20.4 | 4 | 30 | |

SAMPLE DUPLICATE: 1449128

| Parameter | Units | 10231015003 Result | Dup Result | RPD | Max RPD | Qualifiers |
|------------------|-------|-----------------------|---------------|-----|------------|------------|
| Percent Moisture | % | 12.4 | 12.9 | 4 | 30 | |

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

QC Batch: OEXT/21915 Analysis Method: WI MOD DRO

QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS

Associated Lab Samples: 10230969001, 10230969002, 10230969003, 10230969004, 10230969005

METHOD BLANK: 1450696 Matrix: Solid

Associated Lab Samples: 10230969001, 10230969002, 10230969003, 10230969004, 10230969005

| Parameter | Units | Blank Result | Reporting Limit | Analyzed | Qualifiers |
|-----------------------|-------|--------------|-----------------|----------------|------------|
| Diesel Range Organics | mg/kg | <10.0 | 10.0 | 06/10/13 19:30 | |
| n-Triacontane (S) | % | 79 | 50-150 | 06/10/13 19:30 | |

LABORATORY CONTROL SAMPLE & LCSD: 1450697 1450698

| Parameter | Units | Spike Conc. | LCS Result | LCSD Result | LCS % Rec | LCSD % Rec | % Rec Limits | RPD | Max RPD | Qualifiers |
|-----------------------|-------|-------------|------------|-------------|-----------|------------|--------------|-----|---------|------------|
| Diesel Range Organics | mg/kg | 80 | 62.9 | 78.8 | 79 | 98 | 70-120 | 22 | 20 | R1 |
| n-Triacontane (S) | % | | | | 84 | 93 | 50-150 | | | |

REPORT OF LABORATORY ANALYSIS

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QUALIFIERS

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

DEFINITIONS

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

ND - Not Detected at or above adjusted reporting limit.

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

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

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

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

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

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

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

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

TNI - The NELAC Institute.

LABORATORIES

PASI-M Pace Analytical Services - Minneapolis

ANALYTE QUALIFIERS

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

R1 RPD value was outside control limits.

T6 High boiling point hydrocarbons are present in the sample.

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49/16-1227.00 Rev

Pace Project No.: 10230969

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|-------------|---------------|----------------------|------------|-------------------|------------------|
| 10230969001 | TK10-Scrape-1 | WI MOD DRO | OEXT/21915 | WI MOD DRO | GCSV/11470 |
| 10230969002 | TK10-Scrape-2 | WI MOD DRO | OEXT/21915 | WI MOD DRO | GCSV/11470 |
| 10230969003 | TK10-Scrape-3 | WI MOD DRO | OEXT/21915 | WI MOD DRO | GCSV/11470 |
| 10230969004 | TK10-Scrape-4 | WI MOD DRO | OEXT/21915 | WI MOD DRO | GCSV/11470 |
| 10230969005 | TK10-Scrape-5 | WI MOD DRO | OEXT/21915 | WI MOD DRO | GCSV/11470 |
| 10230969001 | TK10-Scrape-1 | TPH GRO/PVOC WI ext. | GCV/10868 | WI MOD GRO | GCV/10869 |
| 10230969002 | TK10-Scrape-2 | TPH GRO/PVOC WI ext. | GCV/10868 | WI MOD GRO | GCV/10869 |
| 10230969003 | TK10-Scrape-3 | TPH GRO/PVOC WI ext. | GCV/10868 | WI MOD GRO | GCV/10869 |
| 10230969004 | TK10-Scrape-4 | TPH GRO/PVOC WI ext. | GCV/10868 | WI MOD GRO | GCV/10869 |
| 10230969005 | TK10-Scrape-5 | TPH GRO/PVOC WI ext. | GCV/10868 | WI MOD GRO | GCV/10869 |
| 10230969001 | TK10-Scrape-1 | ASTM D2974 | MPRP/39672 | | |
| 10230969002 | TK10-Scrape-2 | ASTM D2974 | MPRP/39672 | | |
| 10230969003 | TK10-Scrape-3 | ASTM D2974 | MPRP/39672 | | |
| 10230969004 | TK10-Scrape-4 | ASTM D2974 | MPRP/39673 | | |
| 10230969005 | TK10-Scrape-5 | ASTM D2974 | MPRP/39673 | | |

REPORT OF LABORATORY ANALYSIS

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

1127 10228669

Project Number: 4916123.00 100 002
Project Name: Tank 10 Contractor Release
Sample Origination State W (use two letter postal state abbreviation)

COC Number: **No 40533**

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix | | Collection Time (hh:mm) | On Ice? Y N | Date | Time | Number of Containers/Preservative | | | | | | | | | | | | | | | |
|------------------|-------------|------------|----------------------------|------------------------------|-------------------------|--------|------|-------------------------|-------------|--------|------|-----------------------------------|--------------------------------------|----------------------------------|--------------------------|-----------------------------|--|----------------------|---------------------------|-------------------------|----------------------|------------------------|----------------------------------|----------------------------|---|---|--|
| | | | | | | Water | Soil | | | | | Water | Soil | OC | Water | | | | Soil | | | | | | | | |
| | | | | | | | | | | | | VOCs (unpreserved) #2 | Dissolved Metals (HNO ₃) | Total Metals (HNO ₃) | General (unpreserved) #3 | Diesel Range Organics (HCl) | Nutrients (H ₂ SO ₄) #4 | VOCs (tared MeOH) #1 | GRO, BTEX (tared MeOH) #1 | DRO (tared unpreserved) | Metals (unpreserved) | SVOCS (unpreserved) #2 | % Solids (plastic vial, unpres.) | Total Number Of Containers | | | |
| 1. TK10-Scrape-1 | 1.5 | 1.5 | F+ | 6/3/13 | 1500 | X | X | X | Y | 6/4/13 | 1040 | | | | | | | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 3 | |
| 2. TK10-Scrape-2 | 1.5 | 1.5 | F+ | | 1505 | | | | N | | | | | | | | | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 3 | |
| 3. TK10-Scrape-3 | 1.5 | 1.5 | F+ | | 1510 | | | | | | | | | | | | | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 3 | |
| 4. TK10-Scrape-4 | 1.5 | 1.5 | F+ | | 1515 | | | | | | | | | | | | | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 3 | |
| 5. TK10-Scrape-5 | 1.5 | 1.5 | F+ | ↓ | 1520 | ↓ | ↓ | ↓ | | | | | | | | | | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 3 | |
| 6. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 7. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 8. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 9. | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 10. | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Relinquished By: [Signature] Date: 6/4/13 Time: 1040
 Relinquished By: [Signature] Date: 6/4/13 Time: 1040
 Received by: KO/Pace Date: 6-5-13 Time: 8:40
 Received by: Pace Date: 6-5-13 Time: 8:40
 Samples Shipped VIA: Air Freight Federal Express Sampler
 Other: Dropped off @ Pace Delta Air Bill Number:
 Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

Sample Condition Upon Receipt

Client Name: Barr

Project #: _____

WO# : 10230969



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

Tracking Number: 9470855 15000891

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

Optional: Proj. Due Date: _____ Proj. Name: _____

Packing Material: Bubble Wrap Bubble Bags None Other: _____

Temp Blank? Yes No

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

Cooler Temp Read (°C): 3.3 Cooler Temp Corrected (°C): 3.2

Biological Tissue Frozen? Yes No

Temp should be above freezing to 6°C

Correction Factor: -1

Date and Initials of Person Examining Contents: RO 6-5-13

Comments:

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

CLIENT NOTIFICATION/RESOLUTION

Field Data Required? Yes No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

Project Manager Review: [Signature]

Date: 6/5/13

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

July 26, 2013

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

RE: Project: 49/16-1227 Rev
Pace Project No.: 10235238

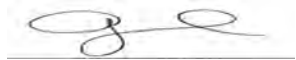
Dear Andrea Nord:

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

This report was revised on July 26, 2013 to correct the project name.

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

Sincerely,



Andrea Opland

andrea.opland@pacelabs.com
Project Manager

Enclosures



REPORT OF LABORATORY ANALYSIS

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CERTIFICATIONS

Project: 49/16-1227 Rev

Pace Project No.: 10235238

Minnesota Certification IDs

1700 Elm Street SE Suite 200, Minneapolis, MN 55414

A2LA Certification #: 2926.01

Alaska Certification #: UST-078

Alaska Certification #MN00064

Arizona Certification #: AZ-0014

Arkansas Certification #: 88-0680

California Certification #: 01155CA

Colorado Certification #Pace

Connecticut Certification #: PH-0256

EPA Region 8 Certification #: Pace

Florida/NELAP Certification #: E87605

Georgia Certification #: 959

Hawaii Certification #Pace

Idaho Certification #: MN00064

Illinois Certification #: 200011

Kansas Certification #: E-10167

Louisiana Certification #: 03086

Louisiana Certification #: LA080009

Maine Certification #: 2007029

Maryland Certification #: 322

Michigan DEQ Certification #: 9909

Minnesota Certification #: 027-053-137

Mississippi Certification #: Pace

Montana Certification #: MT CERT0092

Nebraska Certification #: Pace

Nevada Certification #: MN_00064

New Jersey Certification #: MN-002

New York Certification #: 11647

North Carolina Certification #: 530

North Dakota Certification #: R-036

Ohio VAP Certification #: CL101

Oklahoma Certification #: 9507

Oregon Certification #: MN200001

Oregon Certification #: MN300001

Pennsylvania Certification #: 68-00563

Puerto Rico Certification

Tennessee Certification #: 02818

Texas Certification #: T104704192

Utah Certification #: MN00064

Virginia/DCLS Certification #: 002521

Virginia/VELAP Certification #: 460163

Washington Certification #: C754

West Virginia Certification #: 382

Wisconsin Certification #: 999407970

REPORT OF LABORATORY ANALYSIS

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SAMPLE SUMMARY

Project: 49/16-1227 Rev

Pace Project No.: 10235238

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|-------------|---------------|--------|----------------|----------------|
| 10235238001 | TK10-SCRAPE-6 | Solid | 07/12/13 14:05 | 07/13/13 08:15 |

REPORT OF LABORATORY ANALYSIS

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SAMPLE ANALYTE COUNT

Project: 49/16-1227 Rev

Pace Project No.: 10235238

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|-------------|---------------|------------|----------|-------------------|------------|
| 10235238001 | TK10-SCRAPE-6 | WI MOD GRO | LLC | 7 | PASI-M |
| | | ASTM D2974 | JDL | 1 | PASI-M |

REPORT OF LABORATORY ANALYSIS

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PROJECT NARRATIVE

Project: 49/16-1227 Rev

Pace Project No.: 10235238

Method: WI MOD GRO

Description: WIGRO GCV

Client: Barr Engineering

Date: July 26, 2013

General Information:

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

Hold Time:

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

Sample Preparation:

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

Initial Calibrations (including MS Tune as applicable):

All criteria were within method requirements with any exceptions noted below.

Continuing Calibration:

All criteria were within method requirements with any exceptions noted below.

Internal Standards:

All internal standards were within QC limits with any exceptions noted below.

Surrogates:

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

Method Blank:

All analytes were below the report limit in the method blank with any exceptions noted below.

Laboratory Control Spike:

All laboratory control spike compounds were within QC limits with any exceptions noted below.

Matrix Spikes:

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

Duplicate Sample:

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

Additional Comments:

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

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: 49/16-1227 Rev

Pace Project No.: 10235238

Sample: TK10-SCRAPE-6 **Lab ID: 10235238001** Collected: 07/12/13 14:05 Received: 07/13/13 08:15 Matrix: Solid

Results reported on a "dry-weight" basis

| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
|---|---------|-------|--------------|--------|----|----------------|----------------|-----------|------|
| WIGRO GCV | | | | | | | | | |
| Analytical Method: WI MOD GRO Preparation Method: TPH GRO/PVOC WI ext. | | | | | | | | | |
| Benzene | <0.055 | mg/kg | 0.055 | 0.0087 | 1 | 07/15/13 10:17 | 07/16/13 07:57 | 71-43-2 | |
| Ethylbenzene | <0.055 | mg/kg | 0.055 | 0.0076 | 1 | 07/15/13 10:17 | 07/16/13 07:57 | 100-41-4 | |
| Toluene | <0.055 | mg/kg | 0.055 | 0.0055 | 1 | 07/15/13 10:17 | 07/16/13 07:57 | 108-88-3 | |
| 1,2,4-Trimethylbenzene | <0.055 | mg/kg | 0.055 | 0.0087 | 1 | 07/15/13 10:17 | 07/16/13 07:57 | 95-63-6 | |
| 1,3,5-Trimethylbenzene | <0.055 | mg/kg | 0.055 | 0.0076 | 1 | 07/15/13 10:17 | 07/16/13 07:57 | 108-67-8 | |
| Xylene (Total) | <0.16 | mg/kg | 0.16 | 0.019 | 1 | 07/15/13 10:17 | 07/16/13 07:57 | 1330-20-7 | |
| Surrogates | | | | | | | | | |
| a,a,a-Trifluorotoluene (S) | 100 | % | 80-125 | | 1 | 07/15/13 10:17 | 07/16/13 07:57 | 98-08-8 | |
| Dry Weight | | | | | | | | | |
| Analytical Method: ASTM D2974 | | | | | | | | | |
| Percent Moisture | 8.6 | % | 0.10 | 0.10 | 1 | | 07/17/13 00:00 | | |

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49/16-1227 Rev

Pace Project No.: 10235238

| | | | |
|-------------------------|----------------------|-----------------------|-----------------|
| QC Batch: | GCV/11048 | Analysis Method: | WI MOD GRO |
| QC Batch Method: | TPH GRO/PVOC WI ext. | Analysis Description: | WIGRO Solid GCV |
| Associated Lab Samples: | 10235238001 | | |

METHOD BLANK: 1478093 Matrix: Solid

Associated Lab Samples: 10235238001

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

LABORATORY CONTROL SAMPLE & LCSD: 1478094 1478095

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

MATRIX SPIKE SAMPLE: 1478096

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

SAMPLE DUPLICATE: 1478097

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

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49/16-1227 Rev

Pace Project No.: 10235238

SAMPLE DUPLICATE: 1478097

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

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49/16-1227 Rev

Pace Project No.: 10235238

QC Batch: MPRP/40608

Analysis Method: ASTM D2974

QC Batch Method: ASTM D2974

Analysis Description: Dry Weight/Percent Moisture

Associated Lab Samples: 10235238001

SAMPLE DUPLICATE: 1480064

| Parameter | Units | 10234875007 Result | Dup Result | RPD | Max RPD | Qualifiers |
|------------------|-------|-----------------------|---------------|-----|------------|------------|
| Percent Moisture | % | 13.0 | 13.2 | 2 | 30 | |

SAMPLE DUPLICATE: 1480065

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

REPORT OF LABORATORY ANALYSIS

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QUALIFIERS

Project: 49/16-1227 Rev

Pace Project No.: 10235238

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

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49/16-1227 Rev

Pace Project No.: 10235238

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|---------------|------------------|------------------------|-----------------|--------------------------|-------------------------|
| 10235238001 | TK10-SCRAPE-6 | TPH GRO/PVOC WI ext. | GCV/11048 | WI MOD GRO | GCV/11050 |
| 10235238001 | TK10-SCRAPE-6 | ASTM D2974 | MPRP/40608 | | |

REPORT OF LABORATORY ANALYSIS

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without the written consent of Pace Analytical Services, Inc..



Chain of Custody

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

10235238

1128

Project Number: 49161227.00 100 002

Project Name: Entridge Tank 10 Contractor Release

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

COC Number: No 40528

| Number of Containers/Preservative | | Total Number Of Containers |
|--|----------------------------------|----------------------------|
| Water | Soil | |
| VOCs (HCl) #1 | VOCs (tared MeOH) #1 | 2 |
| SVOCs (unpreserved) #2 | GRO, BTEX (tared MeOH) #1 | |
| Dissolved Metals (HNO ₃) | DRO (tared unpreserved) | |
| Total Metals (HNO ₃) | Metals (unpreserved) | |
| General (unpreserved) #3 | SVOCs (unpreserved) #2 | |
| Diesel Range Organics (HCl) | % Solids (plastic vial, unpres.) | |
| Diesel Range Organics (HCl) | Pvol (-MTBE) | |
| Nutrients (H ₂ SO ₄) #4 | | |
| | | |
| | | |

COC 1 of 1

Project Manager: REE

Project QC Contact: AAN

Sampled by: LSG2

Laboratory: Pace

| Location | Start Depth | Stop Depth | Depth Unit (m./ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix | | Type | | |
|------------------------|-------------|-------------|----------------------------|------------------------------|-------------------------|----------|----------|------|-------|----|
| | | | | | | Water | Soil | Grab | Comp. | QC |
| <u>1 Tk10-Scrape-6</u> | <u>1.75</u> | <u>1.75</u> | <u>R+</u> | <u>7/11/13</u> | <u>1405</u> | <u>X</u> | <u>X</u> | | | |
| 2. | | | | | | | | | | |
| 3. | | | | | | | | | | |
| 4. | | | | | | | | | | |
| 5. | | | | | | | | | | |
| 6. | | | | | | | | | | |
| 7. | | | | | | | | | | |
| 8. | | | | | | | | | | |
| 9. | | | | | | | | | | |
| 10. | | | | | | | | | | |


Pvol(-MTBE), % Solids,

Normal Turn around Time

- Common Parameter/Container - Preservation Key**
- #1 7 Volatile Organics = BTEX, GRO, TPH, 8260 Full List
 - #2 8 Semivolatile Organics = PAHs, PCR, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs
 - #3 3 General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate
 - #4 4 Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

| | | | | | | |
|---|---|---------------------|------------------|-------------------------------|---------------------|-----------------|
| Relinquished By: | On Ice? <input checked="" type="checkbox"/> N | Date <u>7/13/13</u> | Time <u>1345</u> | Received by: <u>PAVE T-24</u> | Date <u>7/13/13</u> | Time <u>815</u> |
| Relinquished By: | On Ice? <input type="checkbox"/> Y <input type="checkbox"/> N | Date | Time | Received by: | Date | Time |
| Samples Shipped VIA: <input type="checkbox"/> Air Freight <input type="checkbox"/> Federal Express <input type="checkbox"/> Sampler <input checked="" type="checkbox"/> Other: <u>Dropphoff @ Pace Dakota</u> | | | | Air Bill Number: | | |

HLR\GUSTDFORMS\Chain Of Custody Form 2009 RLG Rev. 09/01/09

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

Sample Condition Upon Receipt

Client Name: BARZ Project #: _____

WO# : 10235238



10235238

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

Tracking Number: 7962 7031 8584

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

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

Thermom. Used: 888A912167504 80512447 72337080 Type of Ice: Wet Blue None Samples on ice, cooling process has begun

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

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

Comments: _____

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

HNO₃ H₂SO₄ NaOH HCl

Sample # _____

Initial when completed: _____ Lot # of added preservative: _____

CLIENT NOTIFICATION/RESOLUTION

Field Data Required? Yes No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

Project Manager Review: AW

Date: 7/15/13

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

Attachment C

Waste Disposal Documentation

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

I. Generator Information

| | | | |
|---|-----------------|--|-----------------------------|
| Generator Name: Enbridge Pipelines Limited Partnership, LLC | | Generator EPA ID Number | SIC Code |
| Generator Location: Enbridge Superior Terminal -Tank 10 | County: Douglas | Generator Contact: Alex Smith | |
| | | Phone: 715-398-4795 | Fax: 832-325-5511 |
| Generator Mailing Address (if different): 1320 Grand Ave, Superior, WI 54880 | | Generator Email Address: alex.smith@enbridge.com | |
| Bill To Name & Address: Enbridge Energy, 1100 Louisiana Ave, STE. 3300, Houston, TX 77002 | | Bill To #: | Billing Contact: Alex Smith |
| | | Phone: 715-398-4795 | Fax: 832-325-5511 |
| | | Billing Email Address: alex.smith@enbridge.com | |
| Invoice Contact: | | | |

II. Waste Generation Information

| | | |
|--|---|---|
| Waste Name: Crude contaminated soil - Tank 10 | Estimated rate of waste generation: <u>10</u> | <input checked="" type="checkbox"/> one time |
| | | <input type="checkbox"/> Lbs. <input type="checkbox"/> tons <input checked="" type="checkbox"/> cy <input type="checkbox"/> drums |
| Generator Facility Operations and/or Site History: Enbridge Pipeline Terminal | | |
| Describe the generating process or source of contaminated soil/debris and/or waste: Pipeline Terminal Activities | | |

III. Waste Composition and Constituents (list all known)

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

IV. Waste Properties

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

V. Waste Classification

| | | |
|--|--|---|
| Waste stream properties (answer ALL questions) | Does this waste contain absorbents? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Does this waste stream contain any D, F, K, U or P listed as hazardous waste, either in pure form, as a mixture, or treatment residue? | Is this waste lethal (by Minn. Rules 7045.0131 Subp. 6)? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Does this waste stream contain PCB material | Is this waste recyclable? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| If yes, concentration: _____ppm | Is this waste explosive? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Does this waste stream contain fuming acids? | Is this waste infectious? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Does this waste contain asbestos? | Is this putrescible waste? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Does this waste contain oxidizers? | Is this waste demolition debris? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Does this waste contain radioactive material? | Is this waste sewer sludge? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Please attach any available information or analytical test results that have previously been performed on this waste that substantiates these determinations. Include MSDS's and any information from other agencies (i.e., MPCA, USEPA) | | |

VI. Shipping Information

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

VII. Certification of Non Hazardous Waste & Approval Conditions

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

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

| | | | |
|--|--------------|-----------------------|--------------|
|  | Alex Smith | Environmental Analyst | 10 June 2013 |
| Signature | Printed Name | Title | Date |



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

June 06, 2013

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

Work Order Number: 1302482
RE: 49161227

Enclosed are the results of analyses for samples received by the laboratory on 06/04/13. If you have any questions concerning this report, please feel free to contact me.

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

WI Certification #998022410

Prepared by,
LEGEND TECHNICAL SERVICES, INC

A handwritten signature in black ink that reads "Bach Pham". The signature is stylized and written over a horizontal line.

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

A handwritten signature in black ink that reads "Tyler Jones". The signature is written in a cursive style over a horizontal line.

Tyler Jones
Chemist I
tjones@legend-group.com

| | | |
|---|--|--|
| Barr Engineering Co. 4700 W 77th St Minneapolis, MN 55435 | Project: 49161227 Project Number: 49161227 TK10 Project Manager: Ms. Andrea Nord | Work Order #: 1302482 Date Reported: 06/06/13 |
|---|--|--|

ANALYTICAL REPORT FOR SAMPLES

| Sample ID | Laboratory ID | Matrix | Date Sampled | Date Received |
|---------------------|---------------|--------|----------------|----------------|
| Tank 10-stockpile-1 | 1302482-01 | Soil | 06/03/13 13:50 | 06/04/13 09:30 |

Shipping Container Information

Default Cooler Temperature (°C): 5.9

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

Case Narrative:

| | | |
|---|--|--|
| Barr Engineering Co. 4700 W 77th St Minneapolis, MN 55435 | Project: 49161227 Project Number: 49161227 TK10 Project Manager: Ms. Andrea Nord | Work Order #: 1302482 Date Reported: 06/06/13 |
|---|--|--|

DRO/8015D
Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|--|------------|----|-----|-----------|----------|---------|----------|----------|------------|-------|
| Tank 10-stockpile-1 (1302482-01) Soil Sampled: 06/03/13 13:50 Received: 06/04/13 9:30 | | | | | | | | | | |
| Diesel Range Organics | 670 | 48 | 5.5 | mg/kg dry | 5 | B3F0503 | 06/05/13 | 06/06/13 | WI(95) DRO | |
| Surrogate: <i>Triacontane (C-30)</i> | 109 | | | 70-130 % | | " | " | " | " | |

| | | |
|---|--|--|
| Barr Engineering Co. 4700 W 77th St Minneapolis, MN 55435 | Project: 49161227 Project Number: 49161227 TK10 Project Manager: Ms. Andrea Nord | Work Order #: 1302482 Date Reported: 06/06/13 |
|---|--|--|

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

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes | |
|--|--------------------------------|-------|--------------------------------|-----------------|----------|---------|----------|----------|------------|-------|--|
| Tank 10-stockpile-1 (1302482-01) Soil | | | | | | | | | | W-03 | |
| | Sampled: 06/03/13 13:50 | | Received: 06/04/13 9:30 | | | | | | | | |
| Benzene | <0.038 | 0.038 | 0.0048 | mg/kg dry | 1 | B3F0412 | 06/04/13 | 06/04/13 | WI(95) GRO | | |
| Ethylbenzene | <0.038 | 0.038 | 0.0033 | mg/kg dry | 1 | " | " | " | " | | |
| Toluene | <0.038 | 0.038 | 0.0042 | mg/kg dry | 1 | " | " | " | " | | |
| Xylenes (total) | 0.47 | 0.12 | 0.012 | mg/kg dry | 1 | " | " | " | " | | |
| <i>Surrogate: 4-Fluorochlorobenzene</i> | <i>106</i> | | | <i>80-150 %</i> | | " | " | " | " | | |

| | | |
|---|--|--|
| Barr Engineering Co. 4700 W 77th St Minneapolis, MN 55435 | Project: 49161227 Project Number: 49161227 TK10 Project Manager: Ms. Andrea Nord | Work Order #: 1302482 Date Reported: 06/06/13 |
|---|--|--|

PERCENT SOLIDS
Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Dilution | Batch | Prepared | Analyzed | Method | Notes |
|--|-----------|----|-----|-------|----------|---------|----------|----------|---------------|-------|
| Tank 10-stockpile-1 (1302482-01) Soil Sampled: 06/03/13 13:50 Received: 06/04/13 9:30 | | | | | | | | | | |
| % Solids | 92 | | | % | 1 | B3F0609 | 06/06/13 | 06/06/13 | % calculation | |

| | | |
|---|--|--|
| Barr Engineering Co. 4700 W 77th St Minneapolis, MN 55435 | Project: 49161227 Project Number: 49161227 TK10 Project Manager: Ms. Andrea Nord | Work Order #: 1302482 Date Reported: 06/06/13 |
|---|--|--|

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

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|--|--------|-----|------|-----------|-------------|---------------------------------------|------|-------------|------|------------|-------|
| Batch B3F0503 - Sonication (Wisc DRO) | | | | | | | | | | | |
| Blank (B3F0503-BLK1) | | | | | | | | | | | |
| | | | | | | Prepared & Analyzed: 06/05/13 | | | | | |
| Diesel Range Organics | < 6.7 | 6.7 | 0.78 | mg/kg wet | | | | | | | |
| Surrogate: <i>Triacontane (C-30)</i> | 11.3 | | | mg/kg wet | 13.3 | | 84.5 | 70-130 | | | |
| LCS (B3F0503-BS1) | | | | | | | | | | | |
| | | | | | | Prepared & Analyzed: 06/05/13 | | | | | |
| Diesel Range Organics | 60.1 | 8.0 | 0.93 | mg/kg wet | 64.0 | | 93.9 | 70-120 | | | |
| Surrogate: <i>Triacontane (C-30)</i> | 14.5 | | | mg/kg wet | 16.0 | | 90.9 | 70-130 | | | |
| LCS Dup (B3F0503-BSD1) | | | | | | | | | | | |
| | | | | | | Prepared: 06/05/13 Analyzed: 06/06/13 | | | | | |
| Diesel Range Organics | 61.4 | 8.0 | 0.93 | mg/kg wet | 64.0 | | 95.9 | 70-120 | 2.03 | 20 | |
| Surrogate: <i>Triacontane (C-30)</i> | 15.2 | | | mg/kg wet | 16.0 | | 95.0 | 70-130 | | | |

| | | |
|---|--|--|
| Barr Engineering Co. 4700 W 77th St Minneapolis, MN 55435 | Project: 49161227 Project Number: 49161227 TK10 Project Manager: Ms. Andrea Nord | Work Order #: 1302482 Date Reported: 06/06/13 |
|---|--|--|

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

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|---|---------|-------|--------|-----------|-------------|--|------|-------------|--------|------------|-------|
| Batch B3F0412 - EPA 5035 Soil (Purge and Trap) | | | | | | | | | | | |
| Blank (B3F0412-BLK1) | | | | | | Prepared & Analyzed: 06/04/13 | | | | | |
| Benzene | < 0.025 | 0.025 | 0.0031 | mg/kg wet | | | | | | | |
| Ethylbenzene | < 0.025 | 0.025 | 0.0022 | mg/kg wet | | | | | | | |
| Toluene | < 0.025 | 0.025 | 0.0027 | mg/kg wet | | | | | | | |
| Xylenes (total) | < 0.075 | 0.075 | 0.0080 | mg/kg wet | | | | | | | |
| Surrogate: 4-Fluorochlorobenzene | 24.9 | | | ug/L | 25.0 | | 99.6 | 80-150 | | | |
| LCS (B3F0412-BS1) | | | | | | Prepared & Analyzed: 06/04/13 | | | | | |
| Benzene | 99.8 | | | ug/L | 100 | | 99.8 | 80-120 | | | |
| Ethylbenzene | 103 | | | ug/L | 100 | | 103 | 80-120 | | | |
| Toluene | 103 | | | ug/L | 100 | | 103 | 80-120 | | | |
| Xylenes (total) | 313 | | | ug/L | 300 | | 104 | 80-120 | | | |
| Surrogate: 4-Fluorochlorobenzene | 25.2 | | | ug/L | 25.0 | | 101 | 80-150 | | | |
| LCS Dup (B3F0412-BSD1) | | | | | | Prepared & Analyzed: 06/04/13 | | | | | |
| Benzene | 100 | | | ug/L | 100 | | 100 | 80-120 | 0.669 | 20 | |
| Ethylbenzene | 101 | | | ug/L | 100 | | 101 | 80-120 | 1.32 | 20 | |
| Toluene | 102 | | | ug/L | 100 | | 102 | 80-120 | 0.516 | 20 | |
| Xylenes (total) | 313 | | | ug/L | 300 | | 104 | 80-120 | 0.0386 | 20 | |
| Surrogate: 4-Fluorochlorobenzene | 26.0 | | | ug/L | 25.0 | | 104 | 80-150 | | | |
| Matrix Spike (B3F0412-MS1) | | | | | | Source: 1302479-01 Prepared & Analyzed: 06/04/13 | | | | | |
| Benzene | 101 | | | ug/L | 100 | < | 101 | 80-120 | | | |
| Ethylbenzene | 104 | | | ug/L | 100 | 0.274 | 104 | 80-120 | | | |
| Toluene | 103 | | | ug/L | 100 | 0.116 | 103 | 80-120 | | | |
| Xylenes (total) | 323 | | | ug/L | 300 | 0.195 | 108 | 80-120 | | | |
| Surrogate: 4-Fluorochlorobenzene | 26.6 | | | ug/L | 25.0 | | 106 | 80-150 | | | |

| | | |
|---|--|--|
| Barr Engineering Co. 4700 W 77th St Minneapolis, MN 55435 | Project: 49161227 Project Number: 49161227 TK10 Project Manager: Ms. Andrea Nord | Work Order #: 1302482 Date Reported: 06/06/13 |
|---|--|--|

PERCENT SOLIDS - Quality Control
Legend Technical Services, Inc.

| Analyte | Result | RL | MDL | Units | Spike Level | Source Result | %REC | %REC Limits | %RPD | %RPD Limit | Notes |
|--|--------|----|-----|-------|-------------|---------------|------|-------------|------|------------|-------|
| Batch B3F0609 - General Preparation | | | | | | | | | | | |
| Duplicate (B3F0609-DUP1) | | | | | | | | | | | |
| % Solids | 93.0 | | | % | | 92.0 | | | 1.08 | 20 | |
| | | | | | | | | | | | |
| Duplicate (B3F0609-DUP2) | | | | | | | | | | | |
| % Solids | 89.0 | | | % | | 89.0 | | | 0.00 | 20 | |
| | | | | | | | | | | | |
| Duplicate (B3F0609-DUP3) | | | | | | | | | | | |
| % Solids | 92.0 | | | % | | 92.0 | | | 0.00 | 20 | |

| | | |
|---|--|--|
| Barr Engineering Co. 4700 W 77th St Minneapolis, MN 55435 | Project: 49161227 Project Number: 49161227 TK10 Project Manager: Ms. Andrea Nord | Work Order #: 1302482 Date Reported: 06/06/13 |
|---|--|--|

Notes and Definitions

| | |
|------|---|
| W-03 | The initial sample weight was less than 8.0 grams. |
| < | Less than value listed |
| dry | Sample results reported on a dry weight basis |
| NA | Not applicable. The %RPD is not calculated from values less than the reporting limit. |
| MDL | Method Detection Limit |
| RL | Reporting Limit |
| RPD | Relative Percent Difference |
| LCS | Laboratory Control Spike = Blank Spike (BS) = Laboratory Fortified Blank (LFB) |
| MS | Matrix Spike = Laboratory Fortified Matrix (LFM) |

Chain of Custody

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

1302482

Project Number: 49161227
 Project Name: Tank 10 Contractor Release Enbridge
 Sample Origination State: Wf (use two letter postal state abbreviation)
 COC Number: NO 37900

| Number of Containers/Preservative | | COC <u>1</u> of <u>1</u> |
|--|----------------------------------|--|
| Water | Soil | |
| VOCs (HCl) #1 | VOCs (Unred MeOH) #1 | Total Number Of Containers <u>6</u> |
| Dissolved Metals (HNO ₃) | GRO (TEX) (Red MeOH) #1 | |
| Total Metals (HNO ₃) | DRO (Unred unpreserved) | |
| General (unpreserved) #1 | Metals (unpreserved) | |
| Diesel Range Organics (HCl) | SVOCs (unpreserved) #2 | |
| Nutrients (H ₂ SO ₄) #4 | % Solids (plastic vial, unpres.) | |
| | | |
| | | |
| | | |
| | | |

Project Manager: REE
 Project OC Contact: AAN
 Sampled by: CJG/R
 Laboratory: Legend

| Location | Start Depth | Stop Depth | Depth Unit (m, ft. or in.) | Collection Date (mm/dd/yyyy) | Collection Time (hh:mm) | Matrix | | Type | | Total Number Of Containers |
|---------------------------------------|-------------|------------|----------------------------|------------------------------|-------------------------|----------|----------|------|----------|----------------------------|
| | | | | | | Water | Soil | Grab | Comp. OC | |
| 1. <u>Tank 10 contractor - street</u> | - | - | - | <u>6/3/13</u> | <u>1350</u> | <u>X</u> | <u>X</u> | | | <u>6</u> |
| 2. <u>Temp blank - 1</u> | | | | | | <u>1</u> | | | | |
| 3. | | | | | | | | | | |
| 4. | | | | | | | | | | |
| 5. | | | | | | | | | | |
| 6. | | | | | | | | | | |
| 7. | | | | | | | | | | |
| 8. | | | | | | | | | | |
| 9. | | | | | | | | | | |
| 10. | | | | | | | | | | |

OK

ASAP TAT

Common Parameter/Container - Preservation Key

#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List
 #2 - Semivolatile Organics = PAHs, PCB, 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: [Signature] On Ice? Date: 6/3/13 Time: 7:00
 Relinquished By: [Signature] On Ice? Date: 6/3/13 Time: 9:13
 Samples Shipped VIA: Air Freight Federal Express Sampler Air Bill Number: 592
 Other: _____

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

H:\RUGS\TDF\ORMS\Chain Of Custody Form 2009 - RLG - Rev. 09/01/09

June 12, 2013

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

RE: CL13-0025 Crude Contaminated Soil - Tank 10

Dear Smith,

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

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

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

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

Shamrock Landfill


Steve Opstad

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

DATE: 13 June 2013

WASTE APPROVAL Period: 6/12/2013 to 6/3/2015

Bill To Customer

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

Service For Generator

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

Disposal

Waste Description: Crude Contaminated Soil - Tank 10

Estimated Volume: 10 YARDS / ONE TIME ONLY

Disposal Method: Secure Non-Hazardous Landfill

Treatment Method: None Expected For Conforming Waste

Pricing

| | | | |
|----------|---------|---------|-----------------------------------|
| Disposal | \$16.00 | Per Ton | Crude Contaminated Soil - Tank 10 |
|----------|---------|---------|-----------------------------------|

Notification of Waste Acceptance

PAGE 1 of 2
6/12/2013

CUSTOMER INFORMATION

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

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

INVOICE INFORMATION

Bill #: 2133
Enbridge Pipelines Limited Partnership,
Accounts Payable

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

Profile Sheet #:
Waste Stream #: CL13-0025
Waste Name: Crude Contaminated Soil - Tank 10

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

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

ACCEPTANCE INFORMATION

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

This waste is acceptable for delivery beginning on 6/12/2013 thru 6/3/2015 at which time the material will need to be reanalyzed and recertified.

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

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

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

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

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

WASTE STREAM ANALYSIS INFORMATION

Waste Name: Crude Contaminated Soil - Tank 10
Physical State: Solid
Process Producing Waste: pipeline terminal activities

PRE-ACCEPTANCE SAMPLE RESULTS

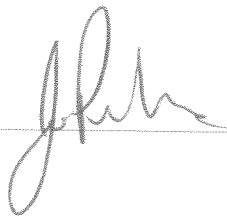
| | | | |
|--------------------|---|-------------------|---|
| Color: | | Physical State: | |
| Dust Present: | 0 | Free Liquids: | 0 |
| Paint Filter Test: | 0 | Odor: | |
| Flash Point Range: | | Density: | |
| Radioactive?: | 0 | Water Reactivity: | 0 |
| pH Range: | | React to Acid: | 0 |
| React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

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

COMMENTS

AUTHORIZATION

Approval: _____



Date: _____

6/12/13



REPORT NAME: Tons Each Load By WSID
DESCRIPTION: Tonnage for EACH LOAD, grouped by customer
DATE RANGE: 01/01/2013 to 08/26/2013
PRINTED ON (DATE): Monday, August 26, 2013

ENBSI

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

| LOAD # | MANIFEST | ARRIVED | WASTE STREAM | WASTE NAME | CELL | SPOT | LIFT | TONS |
|-----------------------------|----------|-----------|--------------|-----------------------------------|------|------|--------------------|--------------|
| 10824 (A) | 10422 | 6/26/2013 | CL13-0025 | Crude Contaminated Soil - Tank 10 | 2A | Q45 | 1160 | 14.56 |
| 11896 (A) | 16400 | 8/5/2013 | CL13-0025 | Crude Contaminated Soil - Tank 10 | 2A | T45 | 1175 | 5.68 |
| Total # of Loads: 2 | | | | | | | Total Tons: | 20.24 |
| Grand Total (Tons): | | | | | | | | 20.24 |
| Grand Total (Loads): | | | | | | | | 2 |