02-16-576734

CORRESPONDENCE/MEMORANDUM — **State of Wisconsin**

DATE: November 17, 2015 FILE REF:

TO: File (Graymont Former Koch Marine Fueling)

FROM: John Sager

SUBJECT: November 17, 2015 telephone call with Ted Hubbes, Braun Intertech

Sager spoke with Ted Hubbes, Braun Intertech, the morning of 11/17/15. Hubbes told Sager that since the notification Braun installed several Geoprobes at the site of the petroleum seep and is going to be excavating soil on 11/18/2015. Sager told Hubbes that Sager would find out if this contamination can be combined with the existing site under the GIS Registry or if anew site had to be made depending on the results of the excavation on 11/18/2015.

Printed on Recycled Paper

02-16-576734

Sager, John E - DNR

From: Shafel, Kathleen S - DNR
Sent: Wednesday, August 05, 2015 10:59
To: Sager, John E - DNR
Subject: FW: Graymont (WI) LLC
Attachments: 1480_001.pdf

Follow Up Flag: Follow up
Flag Status: Flagged

Hi John – here is a Notification that has come in for the Graymont facility in Superior, Douglas Co. I did not assign this a Pending number due to the Location has two Closed sites BRRTS # 02-16-559001 & 03-16-562508. Is this an addition?

Let me know about developments.
Thanks,
We are committed to service excellence.
Visit our survey at http://dnr.wi.gov/customersurvey to evaluate how I did.

Kathleen Shafel
Phone: (715) 623-4190 x 3127
Kathleen.Shafel@wisconsin.gov

From: Hubbes, Ted [mailto:THubbes@braunintertec.com]
Sent: Wednesday, August 05, 2015 8:07 AM
To: DNR RR NOR
Subject: Graymont (WI) LLC

Please review the attached and contact me with questions. I have included some additional discussion below. Thank you. Ted

In July 2015, petroleum impacts (asphalt or heavy petroleum product) were observed during excavation/construction activities in the area of Pulverised Limestone 3 (PLS 3), as shown in Figure 1. The identified contaminated soil was separated visually and stockpiled on site pending off-site disposal. In addition, petroleum product (possibly Number 6 Fuel Oil) has been observed seeping through the pavement near a Hydrate Product Tank. The observed area is small (approximately three feet by six inches). It is anticipated that the source of these impacts are the previous aboveground storage tanks (ASTs) and/or historic property use as a Marine Fueling Terminal BRRTs #02-16-000446).

BRAUN INTERTEC
Ownership @ Work

Ted R. Hubbes, PG, CHMM
Senior Scientist/Associate Principal
Northern Minnesota - Duluth and Hibbing offices
218.263.8869 office | 218.969.5833 mobile
thubbes@braunintertec.com
braunintertec.com | Twitter: Braun Intertec | LinkedIn: Braun Intertec

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

Notification For Hazardous Substance Discharge (Non-Emergency Only)

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

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

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

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

- ☐ Underground Petroleum Storage Tank System (additional information may be required for Item 6 below)
- ☒ Aboveground Petroleum Storage Tank System
- ☐ Dry Cleaner Facility
- ☐ Other - Describe: _____

ATTN DNR: **R & R Program Associate** Date DNR Notified: 08/04/2015

1. Discharge Reported By

Name	Firm	Phone No. (include area code)
Ted R. Hubbes	Braun Intertec Corporation	(218) 263-8869

Mailing Address	Email Address
4511 West First Street, Duluth, MN 55807	thubbes@braunintertec.com

2. Site Information

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

Location: Include street address, not PO Box. If no street address, describe as precisely as possible, i.e., 1/4 mile NW of CTHs 60 & 123 on E side of CTH 60. 800 Hill Avenue, Superior, WI 54880

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

County:	Legal Description	WTM:
Douglas	W2 1/4 NW 1/4 Sec 13 Tn 49 Range 14 ○ E ● W	X _____ Y _____

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

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

☐ Reported in compliance with s. 292.11(2), Wis. Stats., by a local government exempt from liability under s. 292.11(9)(e), Wis. Stats. For more information see http://dnr.wi.gov/topic/Brownfields/Liability.html.

Contact Person Name (if different)	Phone Number	Email Address

Mailing Address	City	State	ZIP Code

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

Contact Person Name (if different)	Phone Number	Email Address
Phil Marquis	(715) 394-1711	pmarquis@graymont.com

Mailing Address	City	State	ZIP Code
800 Hill Avenue	Superior	WI	54880

(continued)

Notification For Hazardous Substance Discharge (Non-Emergency Only)

4. Hazardous Substance Information

Identify hazardous substance discharged (check all that apply):

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

5. Impacts to the Environment Information

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

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

Contamination was discovered as a result of:

☐ Tank closure assessment Date [ ]
☐ Site assessment Date [ ]
☒ Other - Describe: Construction Date [ ]

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

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

None

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

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

☐ Does not apply.

Source

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

Cause

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

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

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

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

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

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

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

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

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

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

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

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

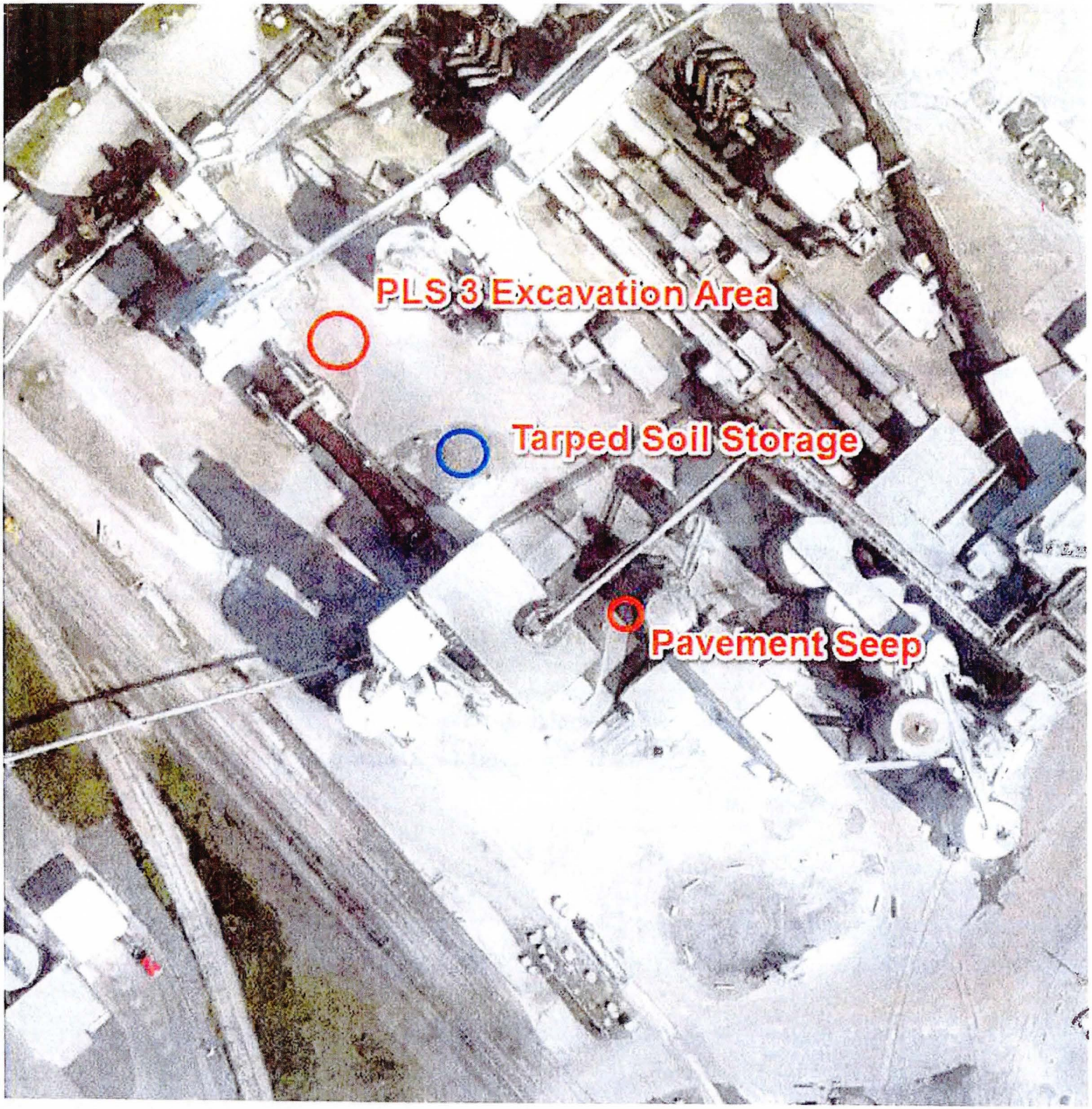


FIGURE 1

GIS REGISTRY INFORMATION

SITE NAME: Former Koch Marine Fueling

BRRTS #: 02-16-000446

CLOSURE DATE: Conditional: 6/3/99 Final:

STREET ADDRESS: #1 Hill Ave

CITY: Superior

SOURCE PROPERTY GPS COORDINATES (meters in WTM91 projection): X= _____ Y= _____

OFF-SOURCE CONTAMINATION (>ES): ☐ Yes ☒ No

IF YES, STREET ADDRESS 1: _____

GPS COORDINATES (meters in WTM91 projection): X= _____ Y= _____

IF YES, STREET ADDRESS 2: _____

GPS COORDINATES (meters in WTM91 projection): X= _____ Y= _____

IF YES, STREET ADDRESS 3: _____

GPS COORDINATES (meters in WTM91 projection): X= _____ Y= _____

IF YES, STREET ADDRESS 4: _____

GPS COORDINATES (meters in WTM91 projection): X= _____ Y= _____

IF YES, STREET ADDRESS 5: _____

GPS COORDINATES (meters in WTM91 projection): X= _____ Y= _____

CONTAMINATION IN RIGHT OF WAY: ☐ Yes ☒ No

DOCUMENTS NEEDED:

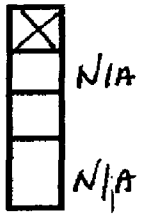
- **Closure Letter, and any conditional closure letter issued** ☒
- **Copy of most recent deed, including legal description, for all affected properties** ☒
- **Certified survey map or relevant portion of the recorded plat map (*If referenced in the legal description*) for all affected properties** ☒
- **County Parcel ID number, *if used for county*, for all affected properties** ☐
- **Location Map** which outlines all properties within contaminated site boundaries in sufficient detail to permit the parcels to be located easily (8.5x14" if paper copy) ☒
- **Detailed Site Map(s) for all affected properties,** showing buildings, roads, property boundaries, contaminant sources, utility lines, monitoring wells and potable wells. (8.5x14", if paper copy) ☒
- **Tables of Latest Groundwater Analytical Results (no shading or cross-hatching)** ☒
- **Isoconcentration map(s), *If available from site investigation (SI)* (**8.5x14" if paper copy). The isoconcentration map should have flow direction and extent of contamination defined. **If not available, include the following 2 types of maps:** ☐
  - **Latest groundwater flow/monitoring well location map** ☒
  - **Latest extent of contaminant plume map** ☐
- **Geologic cross-sections, *if available from SI.*** (8.5x14' if paper copy) ☒

RP certified statement that legal descriptions are complete and accurate

Copies of off-source notification letters (if applicable)

Letter informing ROW owner of residual contamination (if applicable)

Copy of (soil or land use) deed restriction(s) or deed notice *if any required as a condition of closure.*



WISCONSIN
DEPT. OF NATURAL RESOURCES

State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

Scott McCallum, Governor
Darrell Bazzell, Secretary
William H. Smith, Regional Director

Northern Region Headquarters
107 Sutliff Ave.
Rhinelander, Wisconsin 54501-0818
Telephone 715-365-8900
FAX 715-365-8932
TDD 715-365-8957

April 19, 2002

Mr. Greg Schrab
Koch Petroleum Group
PO Box 64596
St. Paul, MN 55164-0596

SUBJECT: Final Case Closure
Former Koch Marine Fueling Facility, #1 Hill Ave, Superior, WI
WDNR BRRTS #: 02-16-000446

Dear Mr. Schrab:

On June 3, 1999, your site as described above was reviewed for closure by the Northern Region Closure Committee. This committee reviews environmental remediation cases for compliance with state laws and standards to maintain consistency in the closure of these cases. On June 8, 1999, you were notified that the Closure Committee had granted conditional closure to this case.

On April 19, 2002, the Department received correspondence indicating that you have complied with the conditions of closure, specifically, submittal of materials and fees for placement on the GIS Registry. The Department considers this case closed and no further investigation, remediation or other action is required at this time.

Your site will be listed on the DNR Remediation and Redevelopment GIS Registry of Closed Remediation Sites. Information that was submitted with your closure request application will be included on the registry. To review the sites on the GIS Registry web page, visit http://gomapout.dnr.state.wi.us/org/at/et/geo/gwur/index.htm

Please be aware that this case may be reopened pursuant to s. NR 726.09, Wis. Adm. Code, if additional information regarding site conditions indicates that contamination on or from the site poses a threat to public health, safety or welfare, or the environment.

The Department appreciates your efforts to restore the environment at this site. If you have any questions regarding this letter, please contact me at 715-365-8990.

Sincerely,
NORTHERN REGION

Janet Kazda

Janet Kazda
Remediation and Redevelopment Program

cc: File

Mark Hagley
Barr Engineering Co
332 W Superior St
Duluth, MN 558002

Dana Stone
CLM Corporation
12th Ave West and Waterfront
Duluth, MN 55802

State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES

Tommy G. Thompson, Governor
George E. Meyer, Secretary
William H. Smith, Regional Director

Northern Region Headquarters
107 Sutliff Ave.
Rhinelander, Wisconsin 54501-0818
Telephone 715-365-8900
FAX 715-365-8932
TDD 715-365-8957

June 8, 1999

Koch Refining Co
PO Box 64596
St Paul, MN 55164

Subject: Marine Fueling Facility, #1 Hill Ave, Superior, WI BRRTS # 02-16-000446

To Whom It May Concern:

The Department of Natural Resources provided a notice to you that the degree and extent of fuel oil contamination at the above-named site was required to be investigated and remediated. We have since been informed that the required investigation and remediation has been accomplished.

On June 3, 1999, the above-named site was reviewed by the Northern Region Closeout Committee for a determination as to whether or not the case qualified for close out under ch. NR 726, Wis. Adm. Code.

Based on the investigative and remedial documentation provided to the Department, it appears that the fuel oil contamination at the above-named site has been remediated in compliance with the requirements of chs. NR 700 to 724, Wis. Adm. Code., and that no further action is necessary at this time. Therefore, the Department will consider the case "closed," if the responsible party sign and record a Groundwater Use Restriction for the property.

Enclosed is an example of a Groundwater Use Restriction. Please draft a specific Groundwater Use Restriction for this site and submit the draft to me. Department of Natural Resources attorneys will review the draft and return it to you with revisions. After you have made the revisions, you should sign and record the restriction with the County Register of Deeds. To document that this condition has been complied with, the responsible party must submit to the Department a copy of the recorded Groundwater Use Restriction, with the recording information stamped on it, within 15 days after the Register of Deeds returns the Groundwater Use Restriction to the responsible party. The Groundwater Use Restriction may be amended in the future with the approval of DNR is conditions change at the site and the residual contamination is remediated.

Please note that this case closure is contingent upon proper documentation of proper abandonment of the monitoring wells on site. If monitoring wells remain at this site, please provide the documentation that this action has been completed, or have your consultant do so. The documentation can be sent to my attention at the above address.

If you have any additional information which was not formerly provided to the Department, and which you feel would significantly impact this closure decision, you may submit that information for our re-evaluation of case closure.

If you have any questions, please call me at 715-365-8990.

Sincerely,
NORTHERN REGION

Janet Kazda

Janet Kazda
Case Closeout Committee

cc: File
Lori Huntoon, Dept of Commerce
Steve Karklins, DG/2
Chuck Fitzgerald, Rhinelander
Jim Hosch, Superior

Mark Hagley
Barr Engineering Co
332 W Superior St
Duluth, MN 55802

No. 381561 Deed Record, Vol. 211, Douglas County, Wisconsin

Henry LaLiberte et ux

TO

Cutler LaLiberte McDougall Corporation

Filed for record the 18 day of July A. D. 1946 at 9.40 o'clock A M.

Ralph Nelson Register of Deeds

By ..... Deputy

This Indenture, Made by Henry LaLiberte and Dorella M.LaLiberte, his wife

grantor s of St.Louis County, Minnesota Quitclaim to Cutler LaLiberte McDougall Corporation, a Wisconsin corporation, grantee of Douglas County, Wisconsin, for the sum of One Dollar($1.00) and other valuable consideration Dollars the following tract of land in Douglas County, State of Wisconsin:

Lots One(1) to Six(6) inclusive in Block Five Hundred Three(503); Lots One(1) to Seven(7) inclusive in Block Five Hundred Four(504); Lots One(1) to Eight(8) inclusive in Block Five Hundred Five(505) and Lots One(1) to Eleven(11) inclusive in Block Five Hundred Six(506), all in the Townsite of West Superior, Sweetser Division, according to the plat thereof on file and of record in the office of the Register of Deeds within and for said County;

Together with the rights acquired and subject to the easements granted under the terms of the instruments recorded in the office of the Register of Deeds in and for Douglas County, Wisconsin, in Book S of Agreements on page 40, and in Book 117 of Deeds on page 492;

And together with any and all rights of way, easements and riparian or other rights appurtenant to the demised premises or any part thereof;

Subject to the taxes on said premises for the year 1946 which the grantee assumes and agrees to pay, and to the rights of Superior Water, Light & Power Company under deed of easement to maintain a waterpipe, as recorded in the office of said Register of Deeds in Book 81 of Deeds on page 69;

As a part of the consideration for this conveyance, it is expressly agreed by the grantee that no part of the premises hereby conveyed shall be used for a period of twenty-five(25) years from April 23, 1945, for the handling of coal other than coal consumed by Cutler-Magner Company or its controlled subsidiaries in connection with operations at the Head of the Lakes.

1-$30.00 documentary US Revenue stamp cancelled

1-$5.00 documentary US Revenue stamp cancelled

1-$4.00 documentary US Revenue stamp cancelled

1-50¢ documentary US Revenue stamp cancelled

1-10¢ documentary US Revenue stamp cancelled

In Witness Whereof, the said grantor s hereunto set hand and seal this 10th day of May A. D. 1946.

Signed and Sealed in Presence of

Elda Zweigle

Elda Zweigle

Patricia Michaud

Patricia Michaud

Henry LaLiberte (Seal)

Henry LaLiberte (Seal)

Dorella M.LaLiberte (Seal)

Dorella M.LaLiberte (Seal)

STATE OF WISCONSIN Minnesota } ss.
County of St.Louis County }

Personally came before me this 10th day of May A. D. 1946, the above named Henry LaLiberte and Dorella M.LaLiberte his wife

to me known to be the person s who executed the foregoing instrument and acknowledged the same.

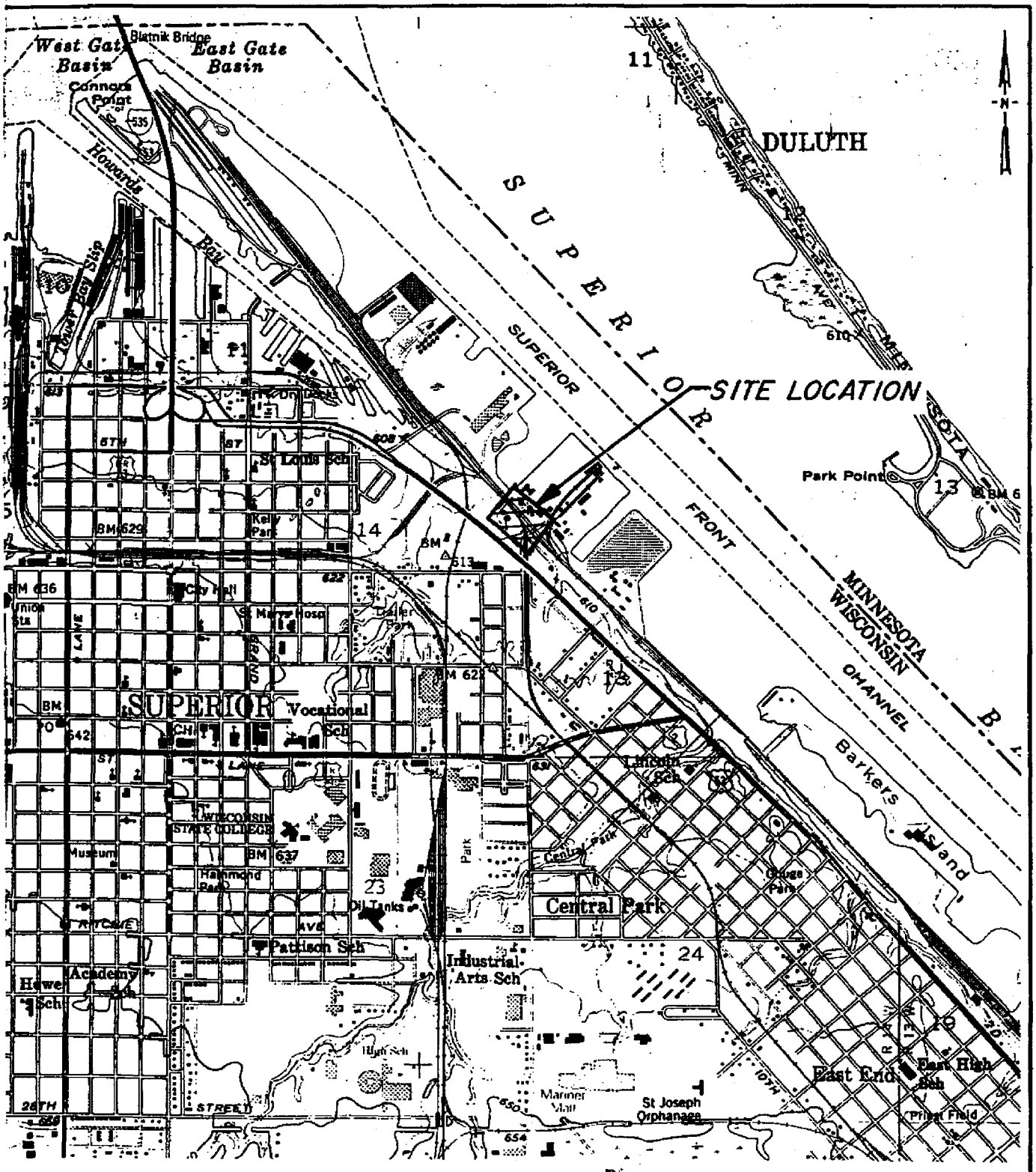
Ida Drannen Ida Drannen

Notary Public St.Louis County, Minn.

My commission expires Dec.10 A. D. 1950

Notarial Seal St.Louis County, Minn.

For Agreement see Vol. 32 of Agreements page 373.



MAP SOURCE: MODIFIED FROM SUPERIOR, WIS.–MINN. U.S.G.S. QUADRANGLE DATED 1993.

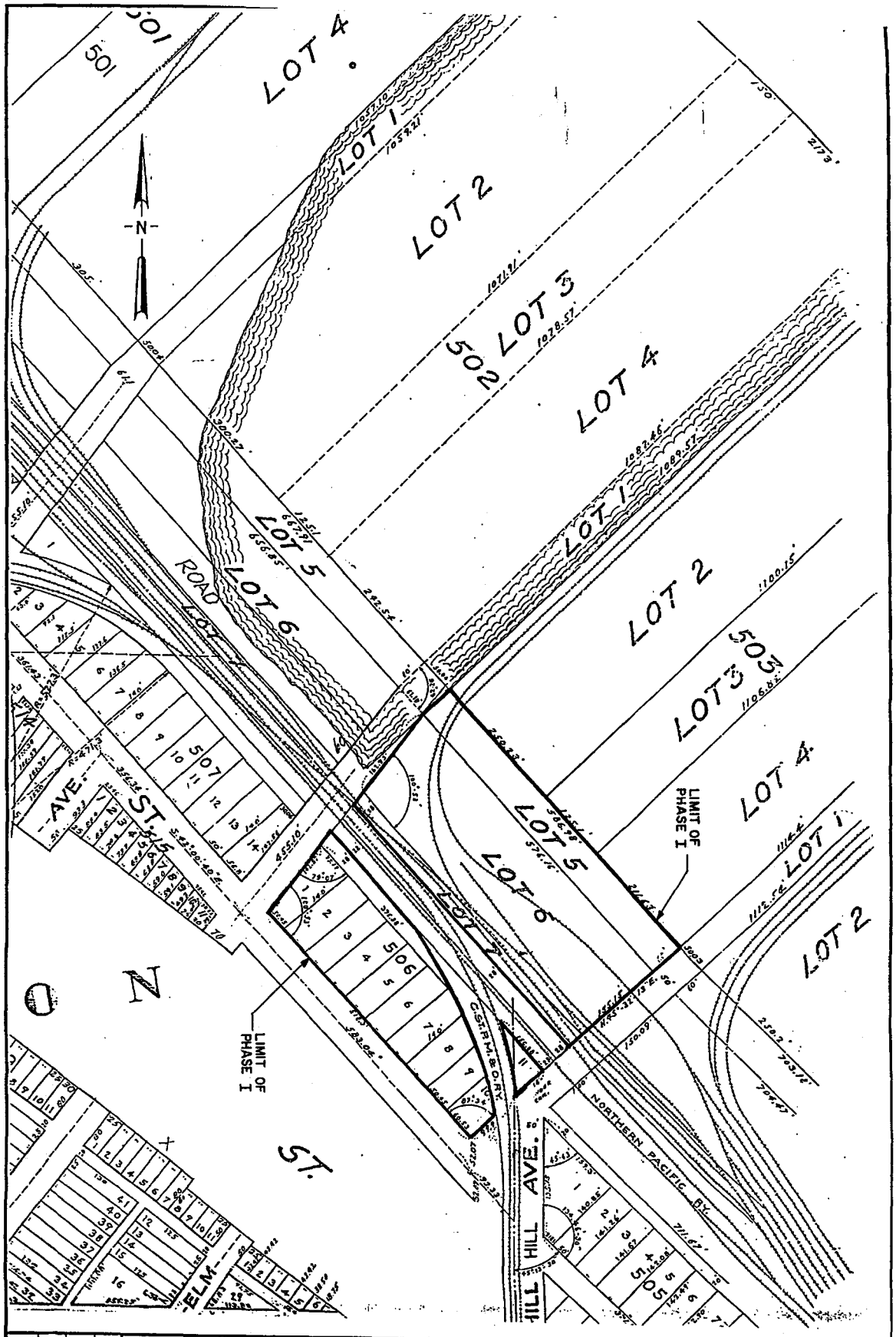
STS Consultants Ltd.
Consulting Engineers

PROJECT/CLIENT

SITE LOCATION DIAGRAM

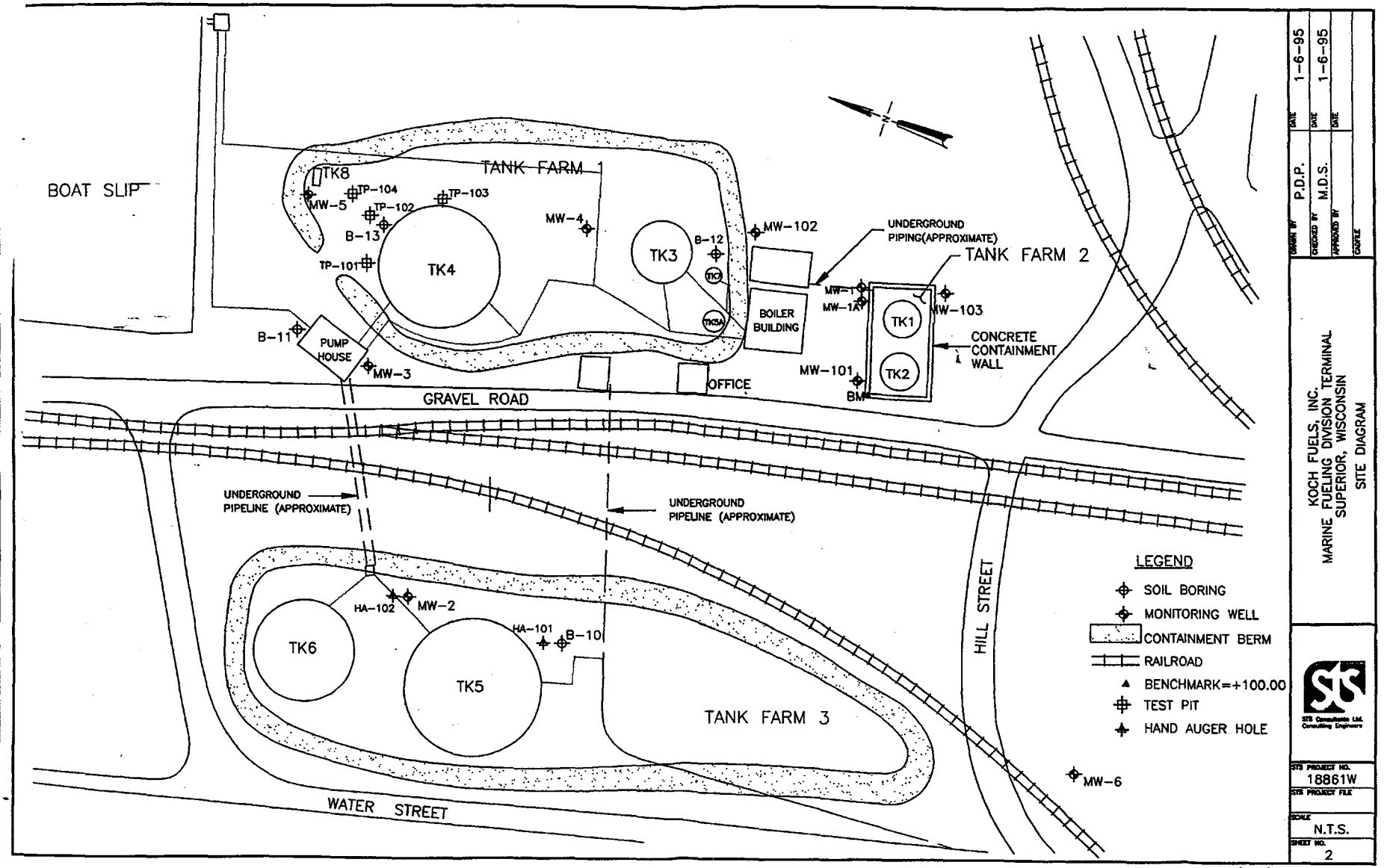
MARINE FUELING TERMINAL
SUPERIOR, WISCONSIN

DRAWN BY	P.D.P.	7-10-95
CHECKED BY		
APPROVED BY		
SCALE 1"=2000'	FIGURE NO. 1	
STS DRAWING NO.	18861W	



SHEET NO. 2	SITE PLAT DIAGRAM KOCH FUELS, INC. MARINE FUELING DIVISION TERMINAL SUPERIOR, WI	DRAWN BY RAB	DATE 9/9/91
SCALE		CHECKED BY	DATE
STS PROJECT FILE 1986IXP		APPROVED BY	DATE
STS PROJECT NO.		CADFILE	

STS Consultants Ltd. Consulting Engineers



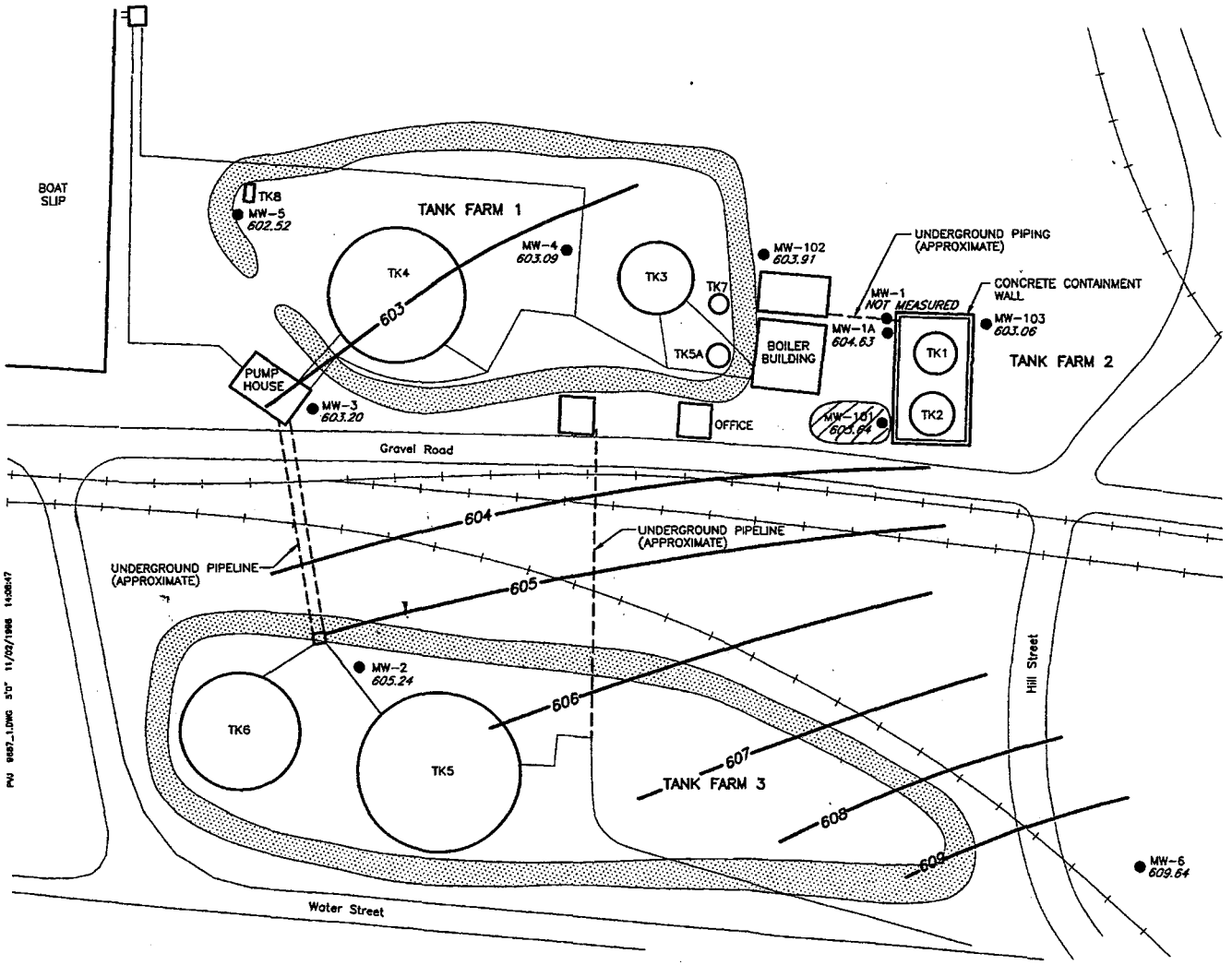
DRAWN BY P.D.P. DATE 1-6-95
CHECKED BY M.D.S. DATE 1-6-95
APPROVED BY DATE
CADFILE

KOCH FUELS, INC.
MARINE FUELING DIVISION TERMINAL
SUPERIOR, WISCONSIN
SITE DIAGRAM

STS Consultants Ltd.
Consulting Engineers

STS PROJECT NO. 18861W
STS PROJECT FILE
SCALE N.T.S.
SHEET NO. 2

W:\PDP\18861XP.DWG



0 60 120

APPROXIMATE SCALE IN FEET

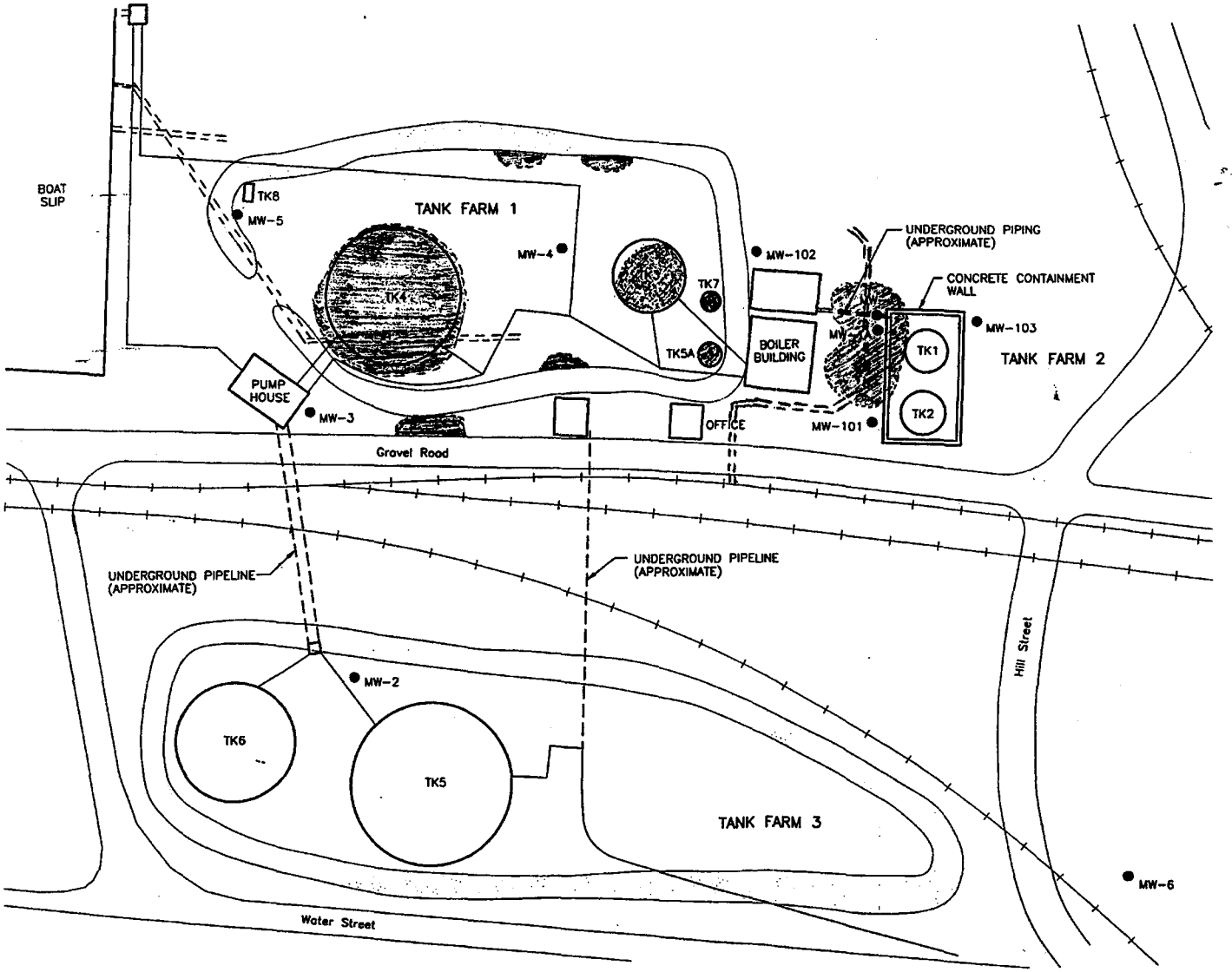
- Monitoring Well
- Containment Berm
- TK1 Aboveground Storage Tank
- 606 Water Table Elevation (Feet, MSL)

Well with PAL Exceedance during Most Recent Sampling

Figure 3

GENERALIZED GROUNDWATER CONTOURS, MAY 1998 (ELEVATION, FEET, MSL)

Marine Fueling
Superior, Wisconsin



0 60 120

APPROXIMATE SCALE IN FEET

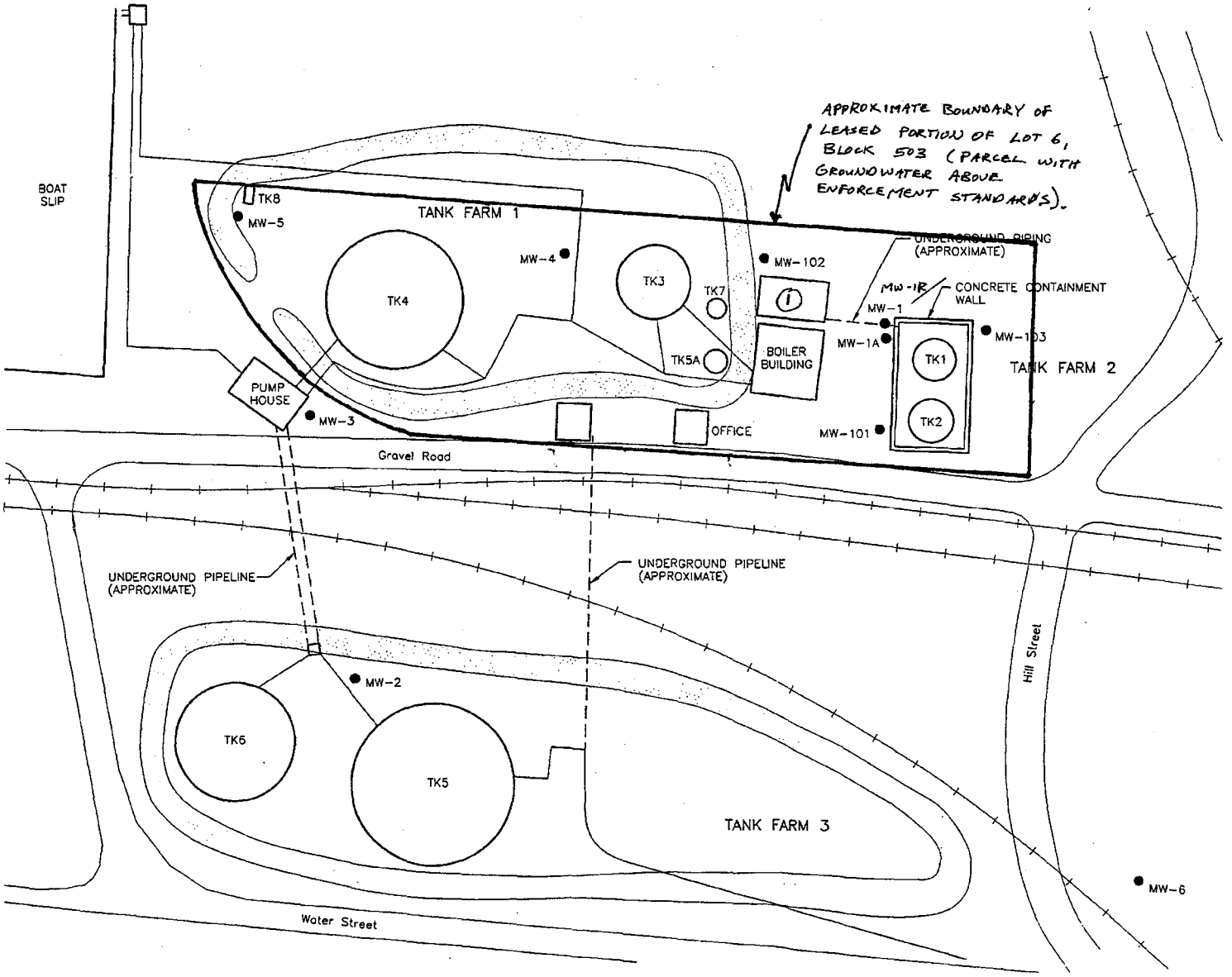
- Monitoring Well
- Containment Berm
- TK1 Aboveground Storage Tank
- Contaminated Soil Zones (Excavated)
- ==== Piping (Removed)

Figure 2

SITE MAP

Marine Fueling
Superior, Wisconsin

CAD M:\CAD\4916007\1850_1 60.00 10/06/1998 08:01:37



- Monitoring Well
- Containment Berm
- TK1 Aboveground Storage Tank

NOTE: ALL FACILITIES WITHIN LOT 6 HAVE BEEN REMOVED (I.E., TANKS, BUILDINGS, PIPING, WELLS), WITH THE EXCEPTION OF THE BUILDING LABELED AS (1) AND THE PUMP HOUSE BUILDING.

WELL MW-1R REPLACED WELL MW-1 (AT THE SAME LOCATION) IN 2000.

Figure 2

SITE MAP

Marine Fueling
Superior, Wisconsin

GAO M:\CAD\49\6007\4680_1 60.00 10/08/1996 08:01:37

Table 2

Groundwater Analytical Data Summary
Marine Fueling Terminal
Superior, Wisconsin

	Date	Benzene	Ethyl-Benzene	MTBE	Toluene	Total Xylene	Benzo(a) pyrene	Naph-thalene	DRO
MW-1*	08-03-91	18.9	67.3	NA	<5.0	206.6	<0.02	165	20,800
	10-21-94	<500	<1,000	<2,000	<2,000	<2,050	<6.0	1,410	1,360,000
	09-04-96	3.4	30	<1.0	<1.0	63	<0.08	48	18,000
	12-10-96	NS	NS	NS	NS	NS	NS	NS	NS
	03-26-97	NS	NS	NS	NS	NS	NS	NS	NS
MW-1A	08-03-91	NA	NA	NA	NA	NA	NA	NA	3,122
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	NA	NA	4,200
	09-04-96	0.7	<1.0	<1.0	<1.0	<2.0	<0.2	<5.0	8,900
	12-11-96	7.5	<1.0	<1.0	<1.0	<2.0	<0.2	3.2	2,100
	03-26-97	0.2 Q	<0.64	<0.64	0.2 Q	<1.6	NA	NA	3,100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	810
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	1,400
MW-2	08-03-91	<0.2	<1.0	NA	<0.5	<2.0	<0.02	<0.02	192
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	<50
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	400
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	120
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	<100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<110
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<100
MW-3	08-03-91	<0.2	<1.0	NA	<0.5	<2.0	<0.02	0.640	<100
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	118
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	450
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	<100
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	<100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<100
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	5,500
	ES	5	700	60	343	620	0.2	40	—
	PAL	0.5	140	12	68.6	124	0.02	8	—

NOTES: All units in micrograms per liter (µg/l)
8-3-91 DRO values reported above reflect TPH-Diesel concentrations
PAL = NR 140 Preventive Action Limit
ES = NR 140 Enforcement Standard
— = NR 140 Standard Not Established
NA = Not Analyzed
NS = Not Sampled
Q = Reported concentration is above the Limit of Detection but below the Limit of Quantification.
[ ] = Detected at or above NR 140 ES [ ] = Detected at or above NR 140 PAL

* Well MW-1 destroyed during site demolition activities. No samples after March 1997.
** Well MW-101 damaged during site demolition activities. No sample obtained in November 1997.

C:\OFFICE\WPWIN\WPDOCS\42211_1.WPD/MTH

Table 2 (continued)

Groundwater Analytical Data Summary
Marine Fueling Terminal
Superior, Wisconsin

	Date	Benzene	Ethyl-Benzene	MTBE	Toluene	Total Xylene	Benzo(a) pyrene	Naph-thalene	DRO
MW-4	08-03-91	<0.2	<1.0	NA	<0.5	<2.0	<0.02	<0.02	<100
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.12	<0.22	1,260
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	660
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	130
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	<100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<100
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	410
MW-5	08-03-91	0.2	<1.0	NA	<0.5	<2.0	<0.02	<0.02	1,460
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	1,760
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.08	<0.5	2,400
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	790
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	1,100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	600
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	240
MW-6	08-03-91	<0.2	<1.0	NA	<0.5	<2.0	5.54	2.70	1,320
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	5.69	<0.11	770
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	540
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	100
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	<110
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<120
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	240
MW-101	08-03-91	—Well Not Installed—			—	—	—	—	—
	10-21-94	0.573	<1.0	<2.0	<2.0	<3.38	<0.06	5.57	5,200
	09-04-96	1.2	<1.0	<1.0	<1.0	<2.0	<0.08	<2.0	16,000
	12-11-96	0.8	<1.0	<1.0	<1.0	4.0	<0.04	4.5	2,600
	03-26-97	0.5 Q	<0.64	<0.64	0.2Q	<1.0 Q	NA	NA	2,200
	11-15-97	NS **	NS **	NS **	NS **	NS **	NS **	NS **	NS **
	5-29-98	0.71	1.1 Q	<0.65	<0.58	<2.65	NA	NA	2,900
	ES	5	700	60	343	620	0.2	40	—
	PAL	0.5	140	12	68.6	124	0.02	8	—

NOTES: All units in micrograms per liter (µg/l)
8-3-91 DRO values reported above reflect TPH-Diesel concentrations
PAL = NR 140 Preventive Action Limit
ES = NR 140 Enforcement Standard
— = NR 140 Standard Not Established
NA = Not Analyzed
NS = Not Sampled
Q = Reported concentration is above the Limit of Detection but below the Limit of Quantification.
[ ] = Detected at or above NR 140 ES
[ ] = Detected at or above NR 140 PAL

* Well MW-1 destroyed during site demolition activities. No samples after March 1997.
** Well MW-101 damaged during site demolition activities. No sample obtained in November 1997.

C:\OFFICE\WPWIN\WPDOCS\42211_1.WPD/MTH

Table 2 (continued)

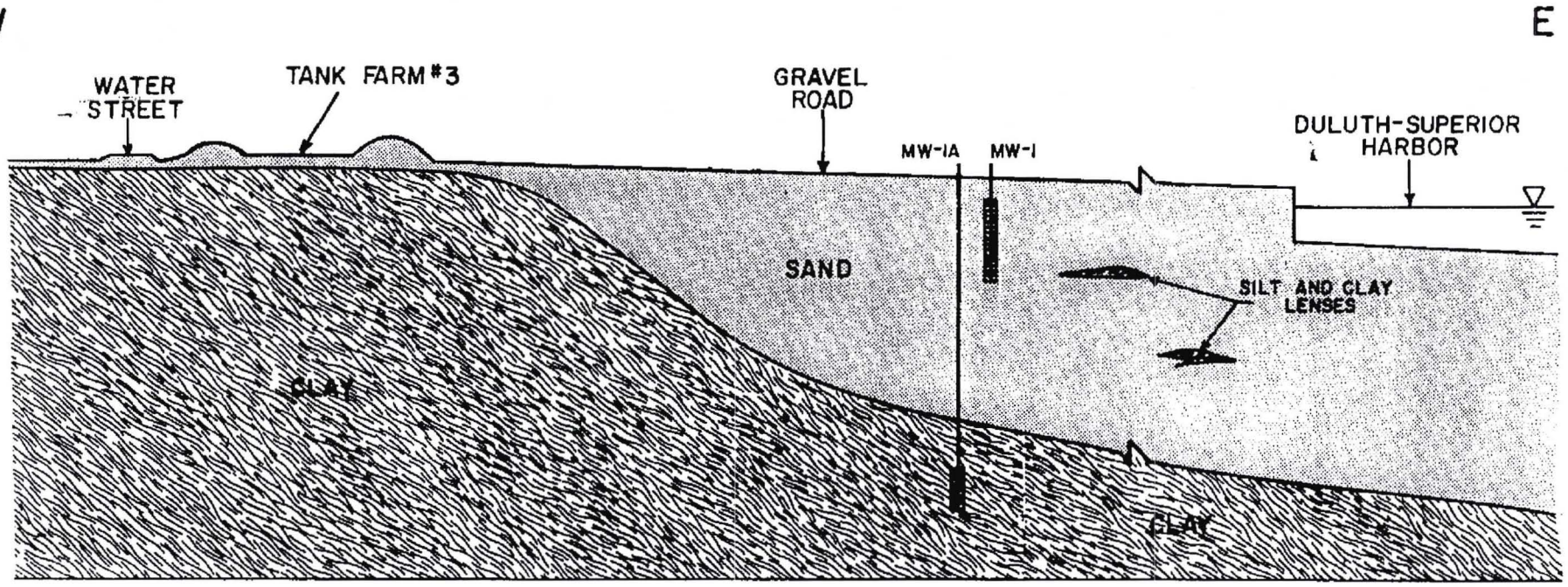
Groundwater Analytical Data Summary
Marine Fueling Terminal
Superior, Wisconsin

	Date	Benzene	Ethyl-Benzene	MTBE	Toluene	Total Xylene	Benzo(a) pyrene	Naph-thalene	DRO
MW-102	08-03-91	—Well Not Installed—			—	—	—	—	—
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	1,360
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	2,200
	12-11-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	310
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	490
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	150
	5-29-98	0.14 Q	<0.59	<0.65	<0.58	<1.85	NA	NA	440
MW-103	08-03-91	—Well Not Installed—			—	—	—	—	—
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	1,060
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	480
	12-11-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	430
	03-26-97	NS	NS	NS	NS	NS	NS	NS	NS
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	890
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	1,100
	ES	5	700	60	343	620	0.2	40	—
	PAL	0.5	140	12	68.6	124	0.02	8	—

NOTES: All units in micrograms per liter (µg/l)
8-3-91 DRO values reported above reflect TPH-Diesel concentrations
PAL = NR 140 Preventive Action Limit
ES = NR 140 Enforcement Standard
— = NR 140 Standard Not Established
NA = Not Analyzed
NS = Not Sampled
Q = Reported concentration is above the Limit of Detection but below the Limit of Quantification.
[ ] = Detected at or above NR 140 ES [ ] = Detected at or above NR 140 PAL

* Well MW-1 destroyed during site demolition activities. No samples after March 1997.
** Well MW-101 damaged during site demolition activities. No sample obtained in November 1997.

C:\OFFICE\WPWIN\WPDOCS\42211_1.WPD/MTH



BRUNING. 544370

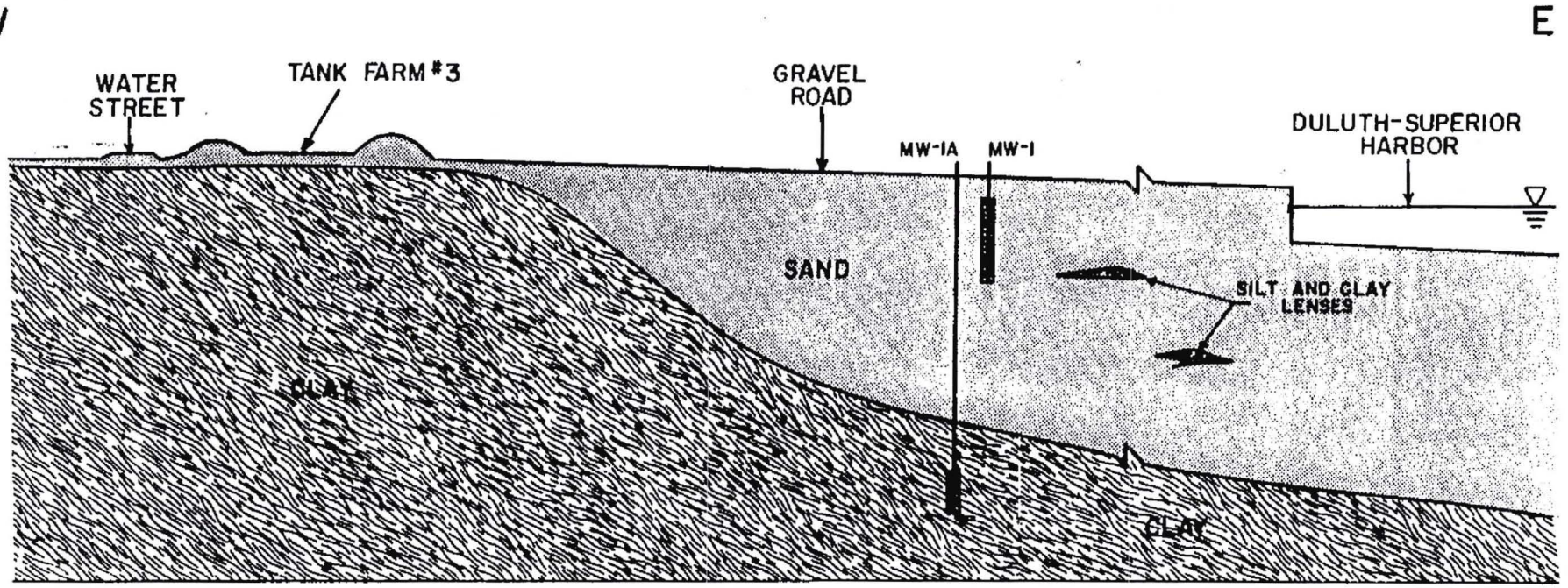
SHEET NO.	9
SCALE	N.T.S
STS PROJECT FILE	
STS PROJECT NO.	18861 XP

STS Consultants Ltd.
Consulting Engineers

KOCH FUELS, INCORPORATED
MARINE FUELING DIVISION TERMINAL
SUPERIOR, WISCONSIN

GENERALIZED GEOLOGIC CROSS SECTION

DRAWN BY	P.M.P	DATE	1-2-92
CHECKED BY		DATE	
APPROVED BY	MDS	DATE	1-3-92
CADFILE			



BRUNING. 544370

SHEET NO.	6
SCALE	N.T.S
STS PROJECT FILE	
STS PROJECT NO.	18861XP

STS Consultants Ltd.
Consulting Engineers

KOCH FUELS, INCORPORATED
MARINE FUELING DIVISION TERMINAL
SUPERIOR, WISCONSIN

GENERALIZED GEOLOGIC CROSS SECTION

DRAWN BY P.M.P	DATE 1-2-92
CHECKED BY	DATE
APPROVED BY MDS	DATE 1-3-92
CADFILE	

Table 1
Groundwater Concentrations in Well MW-1R
(Concentrations in ug/L)

Date	3/30/00	6/13/00	9/13/00	11/16/00
Diesel Range Organics	10,000	19,000	15,000	15,000
Benzene	7.5	10	11	13
Ethylbenzene	14	26	18	13
Toluene	0.32	0.49	0.6	0.51
1,3,5 Trimethylbenzene	3.6	1.4	1.5	0.81 b
1,2,4 Trimethylbenzene	18	43	38	31
Xylenes	7.61	10.5	10	6.4

b = potential false positive detect based on compound detection in the trip blank.

Table 2

Groundwater Analytical Data Summary
Marine Fueling Terminal
Superior, Wisconsin

	Date	Benzene	Ethyl-Benzene	MTBE	Toluene	Total Xylene	Benzo(a) pyrene	Naph-thalene	DRO
MW-1*	08-03-91	18.9	67.3	NA	<5.0	206.6	<0.02	165	20,800
	10-21-94	<500	<1,000	<2,000	<2,000	<2,050	<6.0	1,410	1,360,000
	09-04-96	3.4	30	<1.0	<1.0	63	<0.08	48	18,000
	12-10-96	NS	NS	NS	NS	NS	NS	NS	NS
	03-26-97	NS	NS	NS	NS	NS	NS	NS	NS
MW-1A	08-03-91	NA	NA	NA	NA	NA	NA	NA	3,122
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	NA	NA	4,200
	09-04-96	0.7	<1.0	<1.0	<1.0	<2.0	<0.2	<5.0	8,900
	12-11-96	7.5	<1.0	<1.0	<1.0	<2.0	<0.2	3.2	2,100
	03-26-97	0.2 Q	<0.64	<0.64	0.2 Q	<1.6	NA	NA	3,100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	810
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	1,400
MW-2	08-03-91	<0.2	<1.0	NA	<0.5	<2.0	<0.02	<0.02	192
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	<50
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	400
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	120
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	<100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<110
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<100
MW-3	08-03-91	<0.2	<1.0	NA	<0.5	<2.0	<0.02	0.640	<100
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	118
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	450
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	<100
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	<100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<100
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	5,500
	ES	5	700	60	343	620	0.2	40	—
	PAL	0.5	140	12	68.6	124	0.02	8	—

NOTES: All units in micrograms per liter (µg/l)
8-3-91 DRO values reported above reflect TPH-Diesel concentrations
PAL = NR 140 Preventive Action Limit
ES = NR 140 Enforcement Standard
— = NR 140 Standard Not Established
NA = Not Analyzed
NS = Not Sampled
Q = Reported concentration is above the Limit of Detection but below the Limit of Quantification.
[ ] = Detected at or above NR 140 ES [ ] = Detected at or above NR 140 PAL

* Well MW-1 destroyed during site demolition activities. No samples after March 1997.
** Well MW-101 damaged during site demolition activities. No sample obtained in November 1997.

C:\OFFICE\WPWIN\WPDOCS\42211_1.WPD/MTH

Table 2 (continued)

Groundwater Analytical Data Summary
Marine Fueling Terminal
Superior, Wisconsin

	Date	Benzene	Ethyl-Benzene	MTBE	Toluene	Total Xylene	Benzo(a) pyrene	Naph-thalene	DRO
MW-4	08-03-91	<0.2	<1.0	NA	<0.5	<2.0	<0.02	<0.02	<100
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.12	<0.22	1,260
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	660
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	130
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	<100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<100
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	410
MW-5	08-03-91	0.2	<1.0	NA	<0.5	<2.0	<0.02	<0.02	1,460
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	1,760
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.08	<0.5	2,400
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	790
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	1,100
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	600
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	240
MW-6	08-03-91	<0.2	<1.0	NA	<0.5	<2.0	5.54	2.70	1,320
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	5.69	<0.11	770
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	540
	12-10-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	100
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	<110
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	<120
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	240
MW-101	08-03-91	—Well Not Installed—			—	—	—	—	—
	10-21-94	0.573	<1.0	<2.0	<2.0	<3.38	<0.06	5.57	5,200
	09-04-96	1.2	<1.0	<1.0	<1.0	<2.0	<0.08	<2.0	16,000
	12-11-96	0.8	<1.0	<1.0	<1.0	4.0	<0.04	4.5	2,600
	03-26-97	0.5 Q	<0.64	<0.64	0.2Q	<1.0 Q	NA	NA	2,200
	11-15-97	NS **	NS **	NS **	NS **	NS **	NS **	NS **	NS **
	5-29-98	0.71	1.1 Q	<0.65	<0.58	<2.65	NA	NA	2,900
	ES	5	700	60	343	620	0.2	40	—
	PAL	0.5	140	12	68.6	124	0.02	8	—

NOTES: All units in micrograms per liter (µg/l)
8-3-91 DRO values reported above reflect TPH-Diesel concentrations
PAL = NR 140 Preventive Action Limit
ES = NR 140 Enforcement Standard
— = NR 140 Standard Not Established
NA = Not Analyzed
NS = Not Sampled
Q = Reported concentration is above the Limit of Detection but below the Limit of Quantification.

[ ] = Detected at or above NR 140 ES [ ] = Detected at or above NR 140 PAL

* Well MW-1 destroyed during site demolition activities. No samples after March 1997.
** Well MW-101 damaged during site demolition activities. No sample obtained in November 1997.

C:\OFFICE\WPWIN\WPDOCS\42211_1.WPD/MTH

Table 2 (continued)

Groundwater Analytical Data Summary
Marine Fueling Terminal
Superior, Wisconsin

	Date	Benzene	Ethyl-Benzene	MTBE	Toluene	Total Xylene	Benzo(a) pyrene	Naph-thalene	DRO
MW-102	08-03-91	—Well Not Installed—			—	—	—	—	—
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	1,360
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	2,200
	12-11-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	310
	03-26-97	<0.64	<0.64	<0.64	<0.64	<1.6	NA	NA	490
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	150
	5-29-98	0.14 Q	<0.59	<0.65	<0.58	<1.85	NA	NA	440
MW-103	08-03-91	—Well Not Installed—			—	—	—	—	—
	10-21-94	<0.5	<1.0	<2.0	<2.0	<2.0	<0.06	<0.11	1,060
	09-04-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	480
	12-11-96	<0.6	<1.0	<1.0	<1.0	<2.0	<0.02	<0.5	430
	03-26-97	NS	NS	NS	NS	NS	NS	NS	NS
	11-15-97	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	890
	5-29-98	<0.14	<0.59	<0.65	<0.58	<1.85	NA	NA	1,100
	ES	5	700	60	343	620	0.2	40	—
	PAL	0.5	140	12	68.6	124	0.02	8	—

NOTES: All units in micrograms per liter (µg/l)
8-3-91 DRO values reported above reflect TPH-Diesel concentrations
PAL = NR 140 Preventive Action Limit
ES = NR 140 Enforcement Standard
— = NR 140 Standard Not Established
NA = Not Analyzed
NS = Not Sampled
Q = Reported concentration is above the Limit of Detection but below the Limit of Quantification.
[ ] = Detected at or above NR 140 ES [ ] = Detected at or above NR 140 PAL

* Well MW-1 destroyed during site demolition activities. No samples after March 1997.
** Well MW-101 damaged during site demolition activities. No sample obtained in November 1997.

C:\OFFICE\WPWIN\WPDOCS\42211_1.WPD/MTH

The attached legal descriptions--in the form of the property deed, certified survey map, and the site location map and site map for the Former Koch-Marine Fueling Site (BRRTS #02-16-000446)--provide a complete and accurate legal description of the property, including the portion of the property to which the GIS registration applies.

Greg Schrab
Senior Environmental Scientist
Koch Pipeline Company, L.P.

State of Wisconsin

CORRESPONDENCE/MEMORANDUM

DATE: February 8, 2016 FILE REF:

TO: File (Graymont 02-16-576734)

FROM: John Sager

SUBJECT: Graymont NFA request

I presented the Graymont site (BRRTS ID 02-16-576734) to the Northern Region Closure Committee on 2/4/2016. Based on Braun's investigation and information contained in the 10/15/2015 Environmental Investigation Report and the 1/21/2016 Soil Remediation Letter Report, I concur with the recommendation by Braun that no further action regarding the petroleum seep area in necessary at this time. The site was given a No Further Action status in BRRTS based on concurrence from the closure committee.

Printed on Recycled Paper

Sager, John E - DNR

From: Sager, John E - DNR
Sent: Monday, February 08, 2016 10:25
To: 'Hubbes, Ted'
Cc: Phillip J. Marquis; Keith Miller
Subject: Graymont site BRRTS ID 02-16-576734
Attachments: 20160208101012782.pdf

Ted, Phillip, and Keith,

I presented the Graymont site (BRRTS ID 02-16-576734) to the Northern Region Closure Committee last week. Based on Braun's investigation and information contained in the 10/15/2015 Environmental Investigation Report and the 1/21/2016 Soil Remediation Letter Report, the Department is not requiring further action regarding the petroleum seep area at this time. The site was given a No Further Action status in BRRTS. Attached is the BRRTS Report. If Graymont wishes to receive a site specific letter documenting this decision then a fee is required as specified in NR749 Wis. Adm. Code. Thank you all for your efforts to investigate and clean up this release. Please contact me if you have any questions.

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

John Sager
Hydrogeologist – Remediation and Redevelopment Program
Wisconsin Department of Natural Resources
1701 N. 4th St.
Superior, WI 54880
Phone: (715) 392-7822
Fax: (715) 392-7993
john.sager@wisconsin.gov


dnr.wi.gov


From: Hubbes, Ted [mailto:THubbes@braunintertec.com]
Sent: Thursday, January 21, 2016 12:48
To: Sager, John E - DNR
Cc: Phillip J. Marquis; Keith Miller
Subject: BRRTS # 02-16-000446

John: We discussed this Site 1-2 months back. If you recall, the site is a former Koch Marine Terminal and was remediated and closed in the 1990s. The attached Phase II and Soil Remediation Reports describe actions conducted by Graymont LLC (current owner) in 2015 to address an area where a pavement seep was identified.

Please contact me or Phil Marquis at Graymont (715.394-1711) if you have questions or require further information. Can you also let me know if you would like us to mail paper copies of the report.

Thank you. Ted

BRAUN INTERTEC
Ownership @ Work

Ted R. Hubbes, PG, CHMM
Senior Scientist/Associate Principal
Northern Minnesota - Duluth and Hibbing offices
218.263.8869 office | 218.969.5833 mobile
thubbes@braunintertec.com
braunintertec.com | Twitter: Braun Intertec | LinkedIn: Braun Intertec

02-16-576734 GRAYMONT LLC

Location Name: GRAYMONT LLC
Activity Start Date: 2015-08-05
Activity End Date: 2016-02-04
Address: 800 HILL AVE
Activity Type: ERP
Muni: SUPERIOR
Zip: 54880
File Location:
Region: NO
Region of Management: NO
DSPS No:
County: Douglas
DATCP Case No:
DATCP Spill No:
FID: 816036430
EPA Cerclis ID:
Acres: 33 ☐ Right-of-Way ☐ Acres > 100
Activity Address:
PLSS: ? 1/4 of the ? 1/4 of Sec ?, T?N, R??
Latitude:
Longitude:
Location Comment: NOTIFIED OF NAME CHANGE BY STOEL RIVES ATTORNEYS LETTER OF 12/6/2007, FKA CLM CORP, MKP 01/25/2008; LOW HAZ EXEMPTION FOR 10,000-18,000 TONS LIME KILN DUST AS AGRICULTURAL LIMING AGENT IN NORTHWEST WIS/JIR/9/21/05/LQG FOR THE YEAR OF 2003 -COMPANY HAD REFRACTORY BRICK REMOVED FROM LIME KILN/JIR/03/16/04; STORED CEMENT/LIME FINES FOR BENEFICIAL REUSE, MAY HAVE SOLIDIFIED WHEN GOT WET, SITE IS BEING ARCHIVED FROM WASTE REGISTRY SINCE WASTE NO
Activity Comment:

☐ Transferred to DSPS ☐ Transferred to DATCP ☐ Transferred to WMM ☐ Tracked by DSPS ☐ Created by DSPS
☐ PECFA Eligible ☐ PECFA 80K ☐ PECFA 80K Failure ☐ Above Ground Storage Tank ☐ Co-Contamination
☐ VPLE at Location ☐ VPLE Inactive ☐ General Property at Location ☐ Drycleaner ☐ Superfund ☐ Superfund NPL
☐ Status Unclear (Mask BOTW) ☐ Geo-Located ☐ On GIS Registry

Other Activities at this Location

Activity Detail No	Type	Activity Detail Name	Start Date	End Date
02-16-559001	ERP	GRAYMONT SPECIALTY MINERALS	2012-06-29	2014-10-20
03-16-562508	LUST	GRAYMONT AST	2012-06-29	2014-10-20

Actions

Action Date	Code	Action Name / Comment	Audit
2015-08-05	1	**Notification**	Added 02/04/2016 by SHAFEK
2015-10-15	28	**Phase I Environmental Site Assessment Rpt Received** ENVIRONMENTAL INVESTIGATION RPT REC'D	Added 02/04/2016 by SHAFEK
2016-01-21	99	**Miscellaneous** SOIL REMEDIATION RPT REC'D	Added 02/04/2016 by SHAFEK
2016-02-04	83	**Close-out Under NR708.09** NFA - NO FEE NO LETTER	Added 02/04/2016 by SHAFEK
2016-02-04	11	**Activity Closed** NFA	Added 02/04/2016 by SHAFEK

Documents

Energy Act Details

Release Date: 2015-08-05
Source: Tank
Cause: Unknown

Impacts

Soil Contamination Added 02/04/2016 by SHAFEK

Priority

Risk

Not Applicable Assigned: 02/04/2016 Added 02/04/2016 by SHAFEK

Substances

Category: Petroleum
Fuel Oil Added 02/04/2016 by SHAFEK

02-16-576734 GRAYMONT LLC

Who

Responsible Party is GRAYMONT LLC

Title:
Address: 800 HILL AVE
SUPERIOR WI 54880

Phone: ( ) -
Fax: ( ) -
E-Mail:

Project Manager is JOHN SAGER

Title: HYDROGEOLOGIST
Address: 107 SUTLIFF AVE
RHINELANDER WI 54409

Phone: (715) 365-8959
Fax: (715) 365-8932
E-Mail: john.sager@wisconsin.gov

DNR File Contact is KATHLEEN SHAFEL

Title: ENVIRONMENTAL PROGRAM ASSOCIATE
Address: 223 E STEINFEST RD
ANTIGO WI 54409

Phone: (715) 623-4190 x3127
Fax: (715) 623-6773
E-Mail: kathleen.shafel@wisconsin.gov

RP Contact/Agent is PHIL MARQUIS

Title:
Address: 800 HILL AVE
SUPERIOR WI 54880

Phone: (715) 394-1711
Fax: ( ) -
E-Mail: pmarquis@graymont.com

Sager, John E - DNR

From: Hubbes, Ted <THubbes@braunintertec.com>
Sent: Thursday, January 21, 2016 12:48
To: Sager, John E - DNR
Cc: Phillip J. Marquis; Keith Miller
Subject: BRRTS # 02-16-000446
Attachments: B1507395 _GRAYMONT _ SOIL REMEDIATION REPORT.pdf; Graymont Phase II Report - FINAL.pdf

Follow Up Flag: Flag for follow up
Flag Status: Flagged

John: We discussed this Site 1-2 months back. If you recall, the site is a former Koch Marine Terminal and was remediated and closed in the 1990s. The attached Phase II and Soil Remediation Reports describe actions conducted by Graymont LLC (current owner) in 2015 to address an area where a pavement seep was identified.

Please contact me or Phil Marquis at Graymont (715.394-1711) if you have questions or require further information. Can you also let me know if you would like us to mail paper copies of the report.

Thank you. Ted

BRAUN INTERTEC
Ownership @ Work

Ted R. Hubbes, PG, CHMM
Senior Scientist/Associate Principal
Northern Minnesota - Duluth and Hibbing offices
218.263.8869 office | 218.969.5833 mobile
thubbes@braunintertec.com
braunintertec.com | Twitter: Braun Intertec | LinkedIn: Braun Intertec

Sager, John E - DNR

From: Shafel, Kathleen S - DNR
Sent: Wednesday, January 06, 2016 15:21
To: Sager, John E - DNR
Subject: FW: Graymont (WI) LLC
Attachments: 1480_001.pdf

Hi John – this Notification is still "Pending". See below. It was not assigned a BRRTS tracking number because of the association with two Closed sites at FID Location #816036430. Let me know whether this will be an add on to one of the exciting sites or if it needs its own unique BRRTS number.

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

Kathleen Shafel
Phone: (715) 623-4190 x 3127
Kathleen.Shafel@wisconsin.gov

From: Shafel, Kathleen S - DNR
Sent: Wednesday, August 05, 2015 10:59 AM
To: Sager, John E - DNR
Subject: FW: Graymont (WI) LLC

Hi John – here is a Notification that has come in for the Graymont facility in Superior, Douglas Co. I did not assign this a Pending number due to the Location has two Closed sites BRRTS # 02-16-559001 & 03-16-562508. Is this an addition?

Let me know about developments.
Thanks,
We are committed to service excellence.
Visit our survey at http://dnr.wi.gov/customersurvey to evaluate how I did.

Kathleen Shafel
Phone: (715) 623-4190 x 3127
Kathleen.Shafel@wisconsin.gov

From: Hubbes, Ted [mailto:THubbes@braunintertec.com]
Sent: Wednesday, August 05, 2015 8:07 AM
To: DNR RR NOR
Subject: Graymont (WI) LLC

Please review the attached and contact me with questions. I have included some additional discussion below. Thank you. Ted

In July 2015, petroleum impacts (asphalt or heavy petroleum product) were observed during excavation/construction activities in the area of Pulverised Limestone 3 (PLS 3), as shown in Figure 1. The identified contaminated soil was separated visually and stockpiled on site pending off-site disposal. In addition, petroleum product (possibly Number 6 Fuel Oil) has been observed seeping through the pavement near a Hydrate Product Tank. The observed area is small (approximately three feet by six inches). It is anticipated that the source of these impacts are the previous aboveground storage tanks (ASTs) and/or historic property use as a Marine Fueling Terminal BRRTs #02-16-000446).

BRAUN INTERTEC

Ownership @ Work

Ted R. Hubbes, PG, CHMM
Senior Scientist/Associate Principal
Northern Minnesota - Duluth and Hibbing offices
218.263.8869 office | 218.969.5833 mobile
thubbes@braunintertec.com
braunintertec.com | Twitter: Braun Intertec | LinkedIn: Braun Intertec

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

Notification For Hazardous Substance Discharge (Non-Emergency Only)

Form 4400-225 (09/13) Page 1 of 2

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

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

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

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

- ☐ Underground Petroleum Storage Tank System (additional information may be required for Item 6 below)
- ☒ Aboveground Petroleum Storage Tank System
- ☐ Dry Cleaner Facility
- ☐ Other - Describe: _____

ATTN DNR: **R & R Program Associate** Date DNR Notified: 08/04/2015

1. Discharge Reported By

Name	Firm	Phone No. (include area code)
Ted R. Hubbes	Braun Intertec Corporation	(218) 263-8869

Mailing Address	Email Address
4511 West First Street, Duluth, MN 55807	thubbes@braunintertec.com

2. Site Information

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

Location: Include street address, not PO Box. If no street address, describe as precisely as possible, i.e., 1/4 mile NW of CTHs 60 & 123 on E side of CTH 60. 800 Hill Avenue, Superior, WI 54880

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

County:	Legal Description:	WTM:
Douglas	W2 1/4 NW 1/4 Sec 13 Tn 49 Range 14 ○ E ◉ W	X _____ Y _____

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

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

☐ Reported in compliance with s. 292.11(2), Wis. Stats., by a local government exempt from liability under s. 292.11(9)(e), Wis. Stats. For more information see http://dnr.wi.gov/topic/Brownfields/Liability.html.

Contact Person Name (if different)	Phone Number	Email Address

Mailing Address	City	State	ZIP Code

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

Contact Person Name (if different)	Phone Number	Email Address
Phil Marquis	(715) 394-1711	pmarquis@graymont.com

Mailing Address	City	State	ZIP Code
800 Hill Avenue	Superior	WI	54880

(continued)

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

Notification For Hazardous Substance Discharge (Non-Emergency Only)

4. Hazardous Substance Information

Identify hazardous substance discharged (check all that apply):

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

5. Impacts to the Environment Information

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

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

Contamination was discovered as a result of:

☐ Tank closure assessment Date _____ ☐ Site assessment Date _____ ☒ Other - Describe: Construction Date _____

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

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

None

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

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

☐ Does not apply.

Source

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

Cause

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

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

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

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

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

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

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

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

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

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

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

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

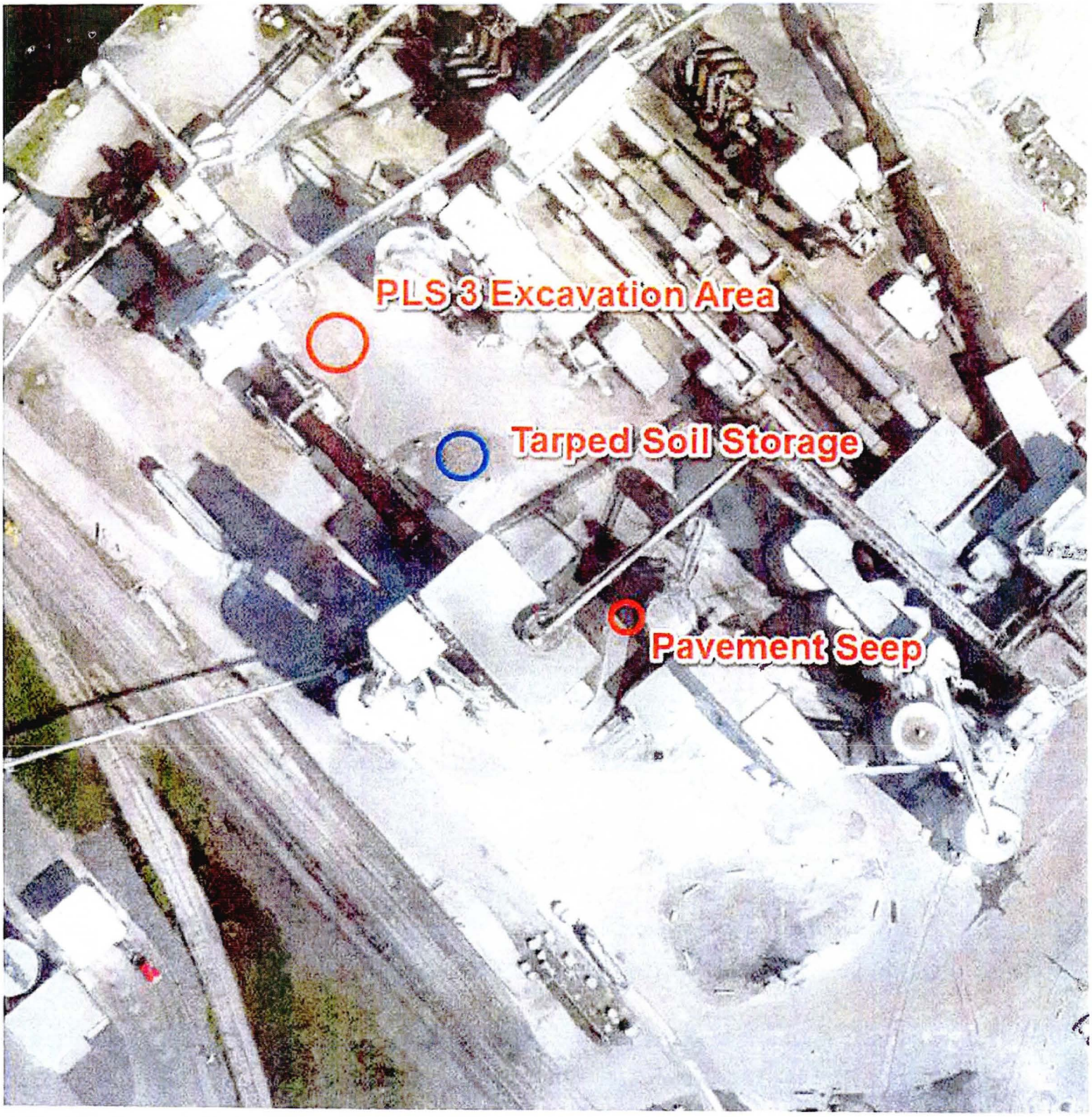


FIGURE 1

Recd 2/1/16

Soil Remediation Letter Report

Graymont (WI) LLC
800 Hill Avenue
Superior, WI

Prepared for

Graymont (WI) LLC

Project B1507395

Braun Intertec Corporation
January 21, 2016

BRAUN INTERTEC
The Science You Build On.

Braun Intertec Corporation
4511 West First Street, Suite 4
Duluth, MN 55807

Phone: 218.624.4967
Fax: 218.624.0196
Web: braunintertec.com

January 21, 2016

Project No. B1507395

Mr. Phil Marquis
Graymont (WI) LLC
800 Hill Avenue
Superior, WI 54880

Re: Soil Remediation Letter Report
Graymont (WI) LLC
800 Hill Avenue
Superior, WI 54880

Braun Intertec Corporation (Braun Intertec) is submitting this Soil Remediation Letter Report for the Graymont (WI) LLC. Facility site located at 800 Hill Avenue, Superior, Wisconsin. The location of the site is shown in the attached Figure 1. In August 2015, an Environmental Investigation was conducted to evaluate potential soil and groundwater petroleum impacts present at the Site, resulting from previous aboveground storage tanks (ASTs) and/or historic property use as a Marine Fueling Terminal. Background information regarding the recent site investigation as well as historical investigation and remediation activities is provided in our report entitled "Environmental Investigation Report, Graymont (WI) LLC", and dated October 15, 2015.

The attached report outlines the removal of impacted soil that was stockpiled during a recent construction project, as well as impacted soil from beneath the area of an observed pavement seep.

Remedial Excavation

Remedial action activities were completed to remove a small pile of impacted soils (1-2 cubic yards) that was excavated during a previous construction project, and soils near the ground surface in an area with a visible oil seep (both areas are shown in the attached Figure 2). Impacted soil was approved for disposal at the VONCO Landfill in Duluth, Minnesota.

On November 20, 2015, a focused remedial excavation was completed at the Site. After removing surface bituminous pavement, an abandoned pipe was encountered about 2 feet below grade near the observed pavement seep. The pipe was empty and surrounded by black, tarry soils. An approximately 225 square-foot area was excavated to three to four feet below grade to remove visually impacted soil in the direct-contact zone. This material was removed for off-site disposal to the extent possible. Excavation photographs are included in Attachment A. The location and area of the remedial excavation

AA/EOE

is illustrated in Figure 2. A total of 18.12 tons of impacted soil (including the previously excavated stockpile) was excavated and transported to the VONCO Landfill for disposal. Landfill disposal documentation is included in Attachment B.

Confirmatory Soil Sampling

A confirmation soil sample was collected at the base of the excavation at a depth of approximately 3 feet. The location of the sample collected is illustrated in Figure 2. Laboratory analytical results indicated that DRO concentrations at the base of the excavation were minimal [13.9 milligrams per kilogram (mg/kg)] and that concentrations of petroleum volatile organic compounds (PVOCs) were below laboratory detection limits. Concentrations of polynuclear aromatic hydrocarbons (PAHs) in the base sample were below the U.S. EPA generic soil screening level for groundwater protection [groundwater residual contaminant level (RCL)]. PAH compounds detected in the base sample were also below the U.S. EPA generic soil screening level for ingestion (direct-contact RCL). Confirmatory soil sampling results are included in the laboratory analytical report attached in Appendix C.

Recommendations

Prior to preparing this report, we conducted a brief review of the project reports provided by Graymont. Based on our review, it appears that the observed soil impacts are related to past use of the Site as a marine fueling terminal by Koch Materials Company. Previous investigation and remediation was conducted as part of WDNR BRRTS file # 02-16-000446.

In accordance with ch. 292 Wis. Stats., the detected soil impacts are reportable to the Wisconsin Department of Natural Resources. At the request of Graymont (WI) LLC., the observed seep was previously reported to WDNR in August 2015. We recommend submitting a copy of the Phase II ESA and this letter report to WDNR staff for review. Mr. John Sager, WDNR Project Manager, indicated WDNR staff would review site conditions and determine if additional actions are necessary.

Assessment Limitations

The analyses and conclusions submitted in this report are based on our field observations and the results of laboratory chemical analyses of soil samples and groundwater samples collected from the soil borings and limited excavation completed for this project.

In performing its services, Braun Intertec used that degree of care and skill ordinarily exercised under similar circumstances by reputable members of its profession currently practicing in the same locality. No warranty, express or implied, is made.

Please call Ted Hubbes at 218.263.8869 if you have questions concerning this project.

Sincerely,

BRAUN INTERTEC CORPORATION

Jennifer C Sparks for

Kevin D. Nestingen
Project Engineer

T. H

Ted R. Hubbes, PG, CHMM
Associate Principal

Attachments:

Figures 1-2
Attachment A - Photographs
Attachment B - Landfill Disposal Documentation
Attachment C - Laboratory Analytical Reports

W:\DRAFTS\B\2015\07395\GIS\B1507395_SiteLoc.mxd

Sheet: 1 of 1

Fig. 1

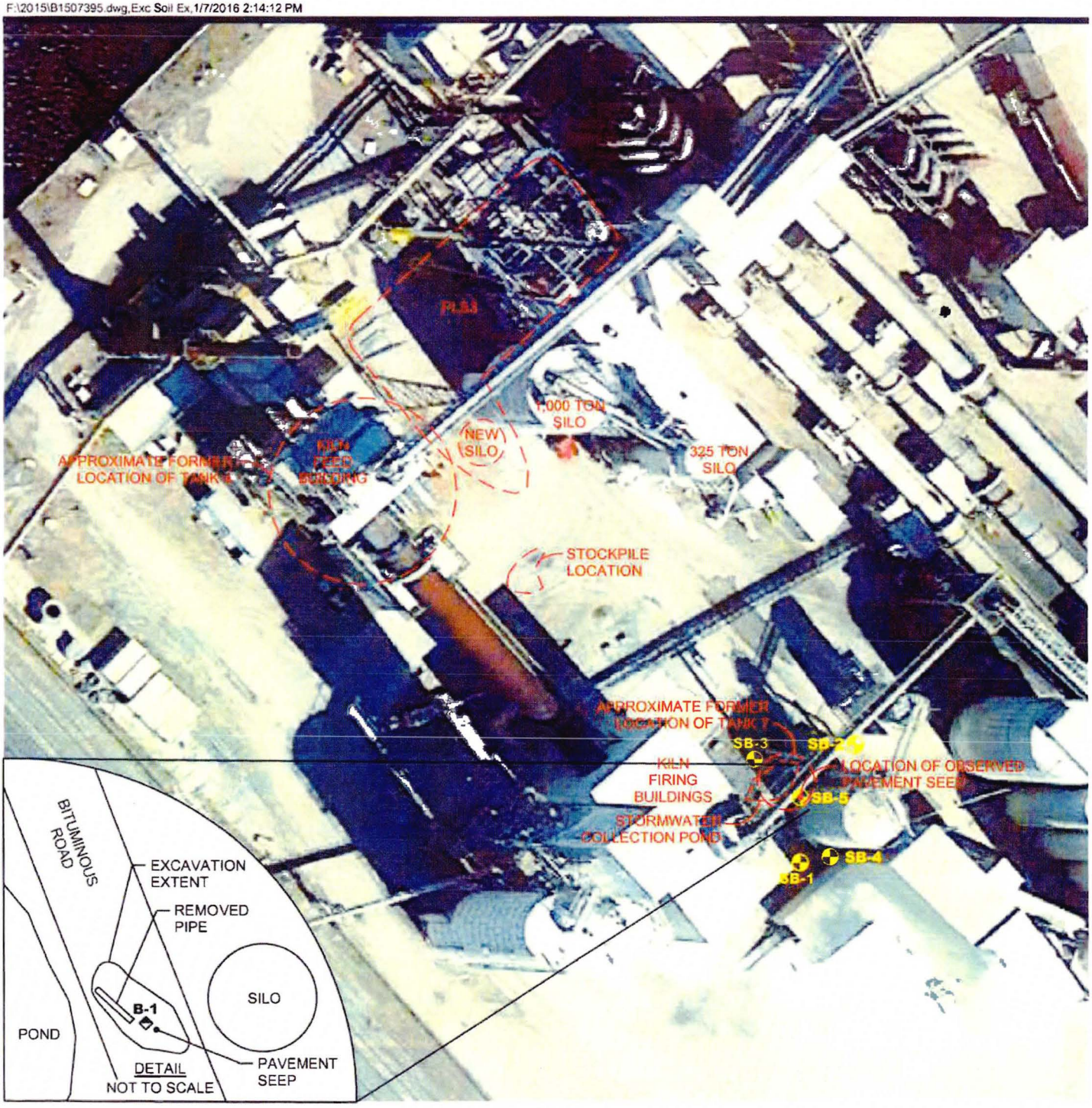
Project No:	B1507395
Drawing No.	B1507395_SiteLoc
Scale:	1 in = 2,000 ft
Drawn By:	FER
Date Drawn:	9/21/15
Checked By:	SB
Last Modified:	9/21/15

SITE LOCATION MAP

GRAYMONT
800 HILL AVENUE
SUPERIOR, WISCONSIN

BRAUN INTERTEC
11001 Hampshire Avenue So.
Minneapolis, MN 55438
PH. (952) 995-2000
FAX (952) 995-2020

F:\2015\B1507395.dwg,Exc Soil Ex,1/7/2016 2:14:12 PM



SOIL BORING LOCATION

EXCAVATION BASE SAMPLE

N

30' 0 60'

SCALE: 1"= 60'

Sheet: of
Fig. 2

Project No:	B1507395
Drawing No:	B1507395
Scale:	1"= 60'
Drawn By:	JAG
Date Drawn:	9/15/15
Checked By:	SB
Last Modified:	1/6/16

SOIL EXCAVATION SKETCH
SOIL INVESTIGATION
GRAYMONT
800 HILL AVENUE
SUPERIOR, WISCONSIN

BRAUN INTERTEC
The Science You Build On.
11001 Hampshire Avenue S
Minneapolis, MN 55438
PH. (952) 995-2000
FAX (952) 995-2020

Photograph #1	Graymont (WI) LLC	B1507395
Date:	November 20, 2015	BRAUN INTERTEC
Location:	800 Hill Avenue Superior, WI	
Subject:	Removed bituminous layer	

Photograph #2	Graymont (WI) LLC	B1507395
Date:	November 20, 2015	BRAUN INTERTEC
Location:	800 Hill Avenue Superior, WI	
Subject:	Pipe section removed	

Photograph #3	Graymont (WI) LLC	B1507395
Date:	November 20, 2015	BRAUN INTERTEC
Locations:	800 Hill Avenue Superior, WI	
Subject:	Excavation area	

Photograph #4	Graymont (WI) LLC	B1507395
Date:	November 20, 2015	BRAUN INTERTEC
Location:	800 Hill Avenue Superior, WI	
Subject:	Excavation limits	

Photograph #5	Graymont (WI) LLC	B1507395
Date:	November 20, 2015	BRAUN INTERTEC
Location:	800 Hill Avenue Superior, WI	
Subject:	Location of base sample	

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

15-149-I Graymont Superior WI

Date	Ticket	Customer	Truck	Material	Tons
11/20/2015	269181	001416 - Graymont	T95469W	Contaminated Soil Tons	18.12
				Total Tons	***18.12***
				Total Loads	***1***

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

December 08, 2015

Ted Hubbes
Braun Intertec
4511 W First St.
#4
Duluth, MN 55807

RE: Project: Graymont Superior
Pace Project No.: 10331311

Dear Ted Hubbes:
Enclosed are the analytical results for sample(s) received by the laboratory on November 24, 2015. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

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

Sincerely,

Zach Wagner
zach.wagner@pacelabs.com
Project Coordinator

Enclosures

cc: Mark Keefer, Braun Intertec

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

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

CERTIFICATIONS

Project: Graymont Superior
Pace Project No.: 10331311

Minnesota Certification IDs
1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Alabama Certification #40770
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: 8TMS-L
Florida/NELAP Certification #: E87605
Guam Certification #:14-008r
Georgia Certification #: 959
Georgia EPD #: Pace
Idaho Certification #: MN00064
Hawaii Certification #MN00064
Illinois Certification #: 200011
Indiana Certification#C-MN-01
Iowa Certification #: 368
Kansas Certification #: E-10167
Kentucky Dept of Envi. Protection - DW #90062
Kentucky Dept of Envi. Protection - WW #:90062
Louisiana DEQ Certification #: 3086
Louisiana DHH #: LA140001
Maine Certification #: 2013011
Maryland Certification #: 322
Michigan DEPH Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Carolina State Public Health #: 27700
North Dakota Certification #: R-036
Ohio EPA #: 4150
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Saipan (CNMI) #:MP0003
South Carolina #:74003001
Texas Certification #: T104704192
Tennessee Certification #: 02818
Utah Certification #: MN000642013-4
Virginia DGS Certification #: 251
Washington Certification #: C486
West Virginia Certification #: 382
West Virginia DHHR #:9952C
Wisconsin Certification #: 999407970

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

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

SAMPLE SUMMARY

Project: Graymont Superior
Pace Project No.: 10331311

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10331311001	B-1-3'	Solid	11/20/15 11:35	11/24/15 17:55

REPORT OF LABORATORY ANALYSIS

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

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

SAMPLE ANALYTE COUNT

Project: Graymont Superior
Pace Project No.: 10331311

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10331311001	**B-1-3'**	WI MOD DRO	JRH	2	PASI-M
		ASTM D2974	JDL	1	PASI-M
		EPA 8270D by SIM	JLR	19	PASI-M
		EPA 8260B	AH2	7	PASI-M

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

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

PROJECT NARRATIVE

Project: Graymont Superior
Pace Project No.: 10331311

Method: **WI MOD DRO**
Description: WIDRO GCS
Client: Braun-BLM
Date: December 08, 2015

General Information:
1 sample was analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

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, where applicable, with any exceptions noted below.

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

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

Additional Comments:

Analyte Comments:

QC Batch: OEXT/31800

D5: The sample was re-weighed into a new container because the sample weight in the original container exceeded the method specifications.

- B-1-3' (Lab ID: 10331311001)
  - n-Triacontane (S)

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

- B-1-3' (Lab ID: 10331311001)
  - WDRO C10-C28

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

Pace Analytical®
www.pacelabs.com

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

PROJECT NARRATIVE

Project: Graymont Superior
Pace Project No.: 10331311

Method: **EPA 8270D by SIM**
Description: 8270D MSSV PAH by SIM
Client: Braun-BLM
Date: December 08, 2015

General Information:
1 sample was analyzed for EPA 8270D by SIM. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

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

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

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

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

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

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

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

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

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

Additional Comments:

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

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

PROJECT NARRATIVE

Project: Graymont Superior
Pace Project No.: 10331311

Method: **EPA 8260B**
Description: 8260B MSV UST
Client: Braun-BLM
Date: December 08, 2015

General Information:
1 sample was analyzed for EPA 8260B. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

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

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

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

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

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

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

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

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

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

Additional Comments:

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

Melanie Ollila
Quality Assurance Officer

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

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

ANALYTICAL RESULTS

Project: Graymont Superior
Pace Project No.: 10331311

Sample: B-1-3' **Lab ID: 10331311001** Collected: 11/20/15 11:35 Received: 11/24/15 17:55 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	**13.9**	mg/kg	11.4	1	11/25/15 10:17	11/29/15 20:13		T6
Surrogates								
n-Triacontane (S)	69	%.	50-150	1	11/25/15 10:17	11/29/15 20:13	638-68-6	D5
Dry Weight	Analytical Method: ASTM D2974							
Percent Moisture	**13.3**	%	0.10	1		12/04/15 13:14		
8270D MSSV PAH by SIM	Analytical Method: EPA 8270D by SIM Preparation Method: EPA 3550							
Acenaphthene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	83-32-9	
Acenaphthylene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	208-96-8	
Anthracene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	120-12-7	
Benzo(a)anthracene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	56-55-3	
Benzo(a)pyrene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	50-32-8	
Benzo(b)fluoranthene	**0.014**	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	205-99-2	
Benzo(g,h,i)perylene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	191-24-2	
Benzo(k)fluoranthene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	207-08-9	
Chrysene	**0.015**	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	218-01-9	
Dibenz(a,h)anthracene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	53-70-3	
Fluoranthene	**0.037**	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	206-44-0	
Fluorene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	86-73-7	
Indeno(1,2,3-cd)pyrene	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	193-39-5	
Naphthalene	**0.018**	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	91-20-3	
Phenanthrene	**0.050**	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	85-01-8	
Pyrene	**0.018**	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52	129-00-0	
Total BaP Eq. MN 2006sh. ND=0	ND	mg/kg	0.012	1	11/25/15 09:20	12/07/15 22:52		
Surrogates								
2-Fluorobiphenyl (S)	68	%.	55-125	1	11/25/15 09:20	12/07/15 22:52	321-60-8	
p-Terphenyl-d14 (S)	79	%.	30-150	1	11/25/15 09:20	12/07/15 22:52	1718-51-0	
8260B MSV UST	Analytical Method: EPA 8260B Preparation Method: EPA 5035/5030B							
Benzene	ND	mg/kg	0.025	1	11/30/15 17:03	12/02/15 02:08	71-43-2	
Ethylbenzene	ND	mg/kg	0.061	1	11/30/15 17:03	12/02/15 02:08	100-41-4	
Toluene	ND	mg/kg	0.061	1	11/30/15 17:03	12/02/15 02:08	108-88-3	
Xylene (Total)	ND	mg/kg	0.18	1	11/30/15 17:03	12/02/15 02:08	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	104	%.	55-150	1	11/30/15 17:03	12/02/15 02:08	17060-07-0	
Toluene-d8 (S)	103	%.	61-125	1	11/30/15 17:03	12/02/15 02:08	2037-26-5	
4-Bromofluorobenzene (S)	102	%.	54-131	1	11/30/15 17:03	12/02/15 02:08	460-00-4	

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior
Pace Project No.: 10331311

QC Batch: MPRP/60117
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10331311001

SAMPLE DUPLICATE: 2149526

Parameter	Units	10332000005 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	1.5	1.5	6	30	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior
Pace Project No.: 10331311

QC Batch: MSV/33920
QC Batch Method: EPA 5035/5030B
Analysis Method: EPA 8260B
Analysis Description: 8260B MSV UST
Associated Lab Samples: 10331311001

METHOD BLANK: 2146029
Matrix: Solid
Associated Lab Samples: 10331311001

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Benzene	mg/kg	ND	0.020	12/01/15 22:19	
Ethylbenzene	mg/kg	ND	0.050	12/01/15 22:19	
Toluene	mg/kg	ND	0.050	12/01/15 22:19	
Xylene (Total)	mg/kg	ND	0.15	12/01/15 22:19	
1,2-Dichloroethane-d4 (S)	%.	98	55-150	12/01/15 22:19	
4-Bromofluorobenzene (S)	%.	102	54-131	12/01/15 22:19	
Toluene-d8 (S)	%.	101	61-125	12/01/15 22:19	

LABORATORY CONTROL SAMPLE: 2146030

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Benzene	mg/kg	1	0.92	92	69-125	
Ethylbenzene	mg/kg	1	0.90	90	72-125	
Toluene	mg/kg	1	0.87	87	72-125	
Xylene (Total)	mg/kg	3	2.8	92	74-125	
1,2-Dichloroethane-d4 (S)	%.			100	55-150	
4-Bromofluorobenzene (S)	%.			101	54-131	
Toluene-d8 (S)	%.			101	61-125	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 2146262 2146263

Parameter	Units	10331493001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Benzene	mg/kg	ND	1.1	1.1	1.1	1.1	95	97	63-126	2	30	
Ethylbenzene	mg/kg	ND	1.1	1.1	1.1	1.1	97	99	69-126	2	30	
Toluene	mg/kg	ND	1.1	1.1	1.1	1.1	93	94	66-128	2	30	
Xylene (Total)	mg/kg	ND	3.4	3.4	3.4	3.5	100	103	70-130	3	30	ES
1,2-Dichloroethane-d4 (S)	%.						96	101	55-150			
4-Bromofluorobenzene (S)	%.						100	99	54-131			
Toluene-d8 (S)	%.						101	101	61-125			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior
Pace Project No.: 10331311

QC Batch: OEXT/31797
QC Batch Method: EPA 3550
Analysis Method: EPA 8270D by SIM
Analysis Description: 8270D Solid PAH by SIM MSSV
Associated Lab Samples: 10331311001

METHOD BLANK: 2144408 Matrix: Solid
Associated Lab Samples: 10331311001

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Acenaphthene	mg/kg	ND	0.010	12/07/15 14:46	
Acenaphthylene	mg/kg	ND	0.010	12/07/15 14:46	
Anthracene	mg/kg	ND	0.010	12/07/15 14:46	
Benzo(a)anthracene	mg/kg	ND	0.010	12/07/15 14:46	
Benzo(a)pyrene	mg/kg	ND	0.010	12/07/15 14:46	
Benzo(b)fluoranthene	mg/kg	ND	0.010	12/07/15 14:46	
Benzo(g,h,i)perylene	mg/kg	ND	0.010	12/07/15 14:46	
Benzo(k)fluoranthene	mg/kg	ND	0.010	12/07/15 14:46	
Chrysene	mg/kg	ND	0.010	12/07/15 14:46	
Dibenz(a,h)anthracene	mg/kg	ND	0.010	12/07/15 14:46	
Fluoranthene	mg/kg	ND	0.010	12/07/15 14:46	
Fluorene	mg/kg	ND	0.010	12/07/15 14:46	
Indeno(1,2,3-cd)pyrene	mg/kg	ND	0.010	12/07/15 14:46	
Naphthalene	mg/kg	ND	0.010	12/07/15 14:46	
Phenanthrene	mg/kg	ND	0.010	12/07/15 14:46	
Pyrene	mg/kg	ND	0.010	12/07/15 14:46	
2-Fluorobiphenyl (S)	%.	64	55-125	12/07/15 14:46	
p-Terphenyl-d14 (S)	%.	74	30-150	12/07/15 14:46	

LABORATORY CONTROL SAMPLE: 2144409

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Acenaphthene	mg/kg	.033	0.021	64	53-125	
Acenaphthylene	mg/kg	.033	0.021	63	53-125	
Anthracene	mg/kg	.033	0.025	76	61-125	
Benzo(a)anthracene	mg/kg	.033	0.024	72	62-125	
Benzo(a)pyrene	mg/kg	.033	0.026	78	64-125	
Benzo(b)fluoranthene	mg/kg	.033	0.026	78	66-125	
Benzo(g,h,i)perylene	mg/kg	.033	0.028	85	59-125	
Benzo(k)fluoranthene	mg/kg	.033	0.029	86	61-125	
Chrysene	mg/kg	.033	0.025	75	63-125	
Dibenz(a,h)anthracene	mg/kg	.033	0.027	81	59-125	
Fluoranthene	mg/kg	.033	0.026	78	64-125	
Fluorene	mg/kg	.033	0.024	72	57-125	
Indeno(1,2,3-cd)pyrene	mg/kg	.033	0.028	84	58-125	
Naphthalene	mg/kg	.033	0.022	67	52-125	
Phenanthrene	mg/kg	.033	0.024	71	60-125	
Pyrene	mg/kg	.033	0.023	70	63-125	
2-Fluorobiphenyl (S)	%.			73	55-125	
p-Terphenyl-d14 (S)	%.			84	30-150	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior
Pace Project No.: 10331311

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 2144410 2144411

Parameter	Units	10331101010 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Acenaphthene	mg/kg	ND	.044	.044	0.026	0.025	58	57	39-125	2	30	
Acenaphthylene	mg/kg	ND	.044	.044	0.024	0.023	55	53	30-150	4	30	
Anthracene	mg/kg	ND	.044	.044	0.029	0.031	66	69	30-150	6	30	
Benzo(a)anthracene	mg/kg	ND	.044	.044	0.028	0.031	64	71	30-150	9	30	
Benzo(a)pyrene	mg/kg	ND	.044	.044	0.032	0.035	73	79	30-150	7	30	
Benzo(b)fluoranthene	mg/kg	ND	.044	.044	0.032	0.034	74	78	30-150	5	30	
Benzo(g,h,i)perylene	mg/kg	ND	.044	.044	0.035	0.037	80	84	30-150	5	30	
Benzo(k)fluoranthene	mg/kg	ND	.044	.044	0.034	0.037	77	85	30-150	9	30	
Chrysene	mg/kg	ND	.044	.044	0.029	0.032	66	72	30-150	9	30	
Dibenz(a,h)anthracene	mg/kg	ND	.044	.044	0.033	0.035	74	79	30-150	6	30	
Fluoranthene	mg/kg	ND	.044	.044	0.032	0.035	73	80	30-150	9	30	
Fluorene	mg/kg	ND	.044	.044	0.029	0.029	66	67	30-146	1	30	
Indeno(1,2,3-cd)pyrene	mg/kg	ND	.044	.044	0.033	0.036	76	81	30-150	6	30	
Naphthalene	mg/kg	ND	.044	.044	0.026	0.023	58	52	30-131	11	30	
Phenanthrene	mg/kg	ND	.044	.044	0.029	0.032	67	74	30-150	10	30	
Pyrene	mg/kg	ND	.044	.044	0.028	0.031	63	71	30-150	12	30	
2-Fluorobiphenyl (S)	%.						64	60	55-125			
p-Terphenyl-d14 (S)	%.						73	79	30-150			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

Pace Analytical®
www.pacelabs.com

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

QUALITY CONTROL DATA

Project: Graymont Superior
Pace Project No.: 10331311

QC Batch: OEXT/31800 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10331311001

METHOD BLANK: 2144503 Matrix: Solid
Associated Lab Samples: 10331311001

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
WDRO C10-C28	mg/kg	ND	10.0	11/29/15 18:57	
n-Triacontane (S)	%.	73	50-150	11/29/15 18:57	

LABORATORY CONTROL SAMPLE & LCSD: 2144504 2144505

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
WDRO C10-C28	mg/kg	80	57.3	62.3	72	78	70-120	8	20	
n-Triacontane (S)	%.				76	84	50-150			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALIFIERS

Project: Graymont Superior
Pace Project No.: 10331311

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

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.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

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

D5	The sample was re-weighed into a new container because the sample weight in the original container exceeded the method specifications.
ES	The reported result is estimated because one or more of the constituent results are qualified as such.
T6	High boiling point hydrocarbons are present in the sample.

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

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

QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: Graymont Superior
Pace Project No.: 10331311

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10331311001	**B-1-3'**	WI MOD DRO	OEXT/31800	WI MOD DRO	GCSV/17359
10331311001	**B-1-3'**	ASTM D2974	MPRP/60117		
10331311001	**B-1-3'**	EPA 3550	OEXT/31797	EPA 8270D by SIM	MSSV/13551
10331311001	**B-1-3'**	EPA 5035/5030B	MSV/33920	EPA 8260B	MSV/33929

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

Pace Analytical®
www.pacelabs.com

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

L0331311

Page: of

1744812

Section A Required Client Information:

Company: Braun Intertec
Address: Hibbing
Email To: THubbs@braunintertec.com
Phone: Fax:
Requested Due Date/TAT:

Section B Required Project Information:

Report To: Ted Hubbs
Copy To:
Purchase Order No.:
Project Name: Graymont Superior
Project Number:

Section C Invoice Information:

Attention:
Company Name:
Address:
Pace Quote Reference:
Pace Project Manager:
Pace Profile #:

REGULATORY AGENCY

☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER
☐ UST ☐ RCRA ☐ OTHER ______

Site Location STATE: ______

Section D Required Client Information

Matrix Codes MATRIX / CODE: Drinking Water DW, Water WT, Waste Water WW, Product P, Soil/Solid SL, Oil OL, Wipe WP, Air AR, Tissue TS, Other OT

SAMPLE ID (A-Z, 0-9 / ,-) Sample IDs MUST BE UNIQUE

Requested Analysis Filtered (Y/N)

ITEM #	SAMPLE ID	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test	WI-PVOC	DRO	PAH	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1	B-1-3'			11-20		11-20	11:35		7										X	X	X		001
2																							
3																							
4																							
5																							
6																							
7																							
8																							
9																							
10																							
11																							
12																							

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
	[signature]	11/23/15	16:00	[signature]	11/23/15	1600				
	[signature]	11/24/15	1500	[signature]	11/24/15	1500				
	[signature]	11/24/15		[signature] Pace	11/24/15	7.8	LS	Y	Y	Y

ORIGINAL

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Camron Vollbrecht

SIGNATURE of SAMPLER: [signature] DATE Signed (MM/DD/YY): 11-22-15

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

F-ALL-Q-020rev.07, 15-May-2007

Pace Analytical®	Document Name: Sample Condition Upon Receipt Form	Document Revised: 23Feb2015 Page 1 of 1
	Document No.: F-MN-L-213-rev.13	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt **Client Name:** Braun **Project #:** WO# : 10331311

10331311

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

Tracking Number: _____

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

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

Thermometer Used: ☐B88A9130516413 ☐B88A912167504 ☑B88A0143310098 **Type of Ice:** ☑Wet ☐Blue ☐None ☐Samples on ice, cooling process has begun

Cooler Temp Read (°C): 1.1 **Cooler Temp Corrected (°C):** 1.5 **Biological Tissue Frozen?** ☐Yes ☐No ☐N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.4 **Date and Initials of Person Examining Contents:** CLJ 11/24/15

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or WA (check maps)? ☐Yes ☐No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐Yes ☐No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☑Yes	☐No	☐N/A	1.
Chain of Custody Filled Out?	☑Yes	☐No	☐N/A	2.
Chain of Custody Relinquished?	☑Yes	☐No	☐N/A	3.
Sampler Name and/or Signature on COC?	☑Yes	☐No	☐N/A	4.
Samples Arrived within Hold Time?	☑Yes	☐No	☐N/A	5.
Short Hold Time Analysis (<72 hr)?	☐Yes	☑No	☐N/A	6.
Rush Turn Around Time Requested?	☐Yes	☑No	☐N/A	7.
Sufficient Volume?	☑Yes	☐No	☐N/A	8.
Correct Containers Used?	☑Yes	☐No	☐N/A	9.
-Pace Containers Used?	☑Yes	☐No	☐N/A	
Containers Intact?	☑Yes	☐No	☐N/A	10.
Filtered Volume Received for Dissolved Tests?	☐Yes	☐No	☑N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC?	☑Yes	☐No	☐N/A	12.
-Includes Date/Time/ID/Analysis Matrix: SL				
All containers needing acid/base preservation have been checked?	☐Yes	☐No	☑N/A	13. ☐HNO_3 ☐H_2SO_4 ☐NaOH ☐HCl Sample #
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐Yes	☐No	☑N/A	
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐Yes	☐No	☑N/A	Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐Yes	☐No	☑N/A	14.
Trip Blank Present?	☐Yes	☐No	☑N/A	15.
Trip Blank Custody Seals Present?	☐Yes	☐No	☐N/A	
Pace Trip Blank Lot # (if purchased):				

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

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

Project Manager Review: _____ Date: 11/25/2015

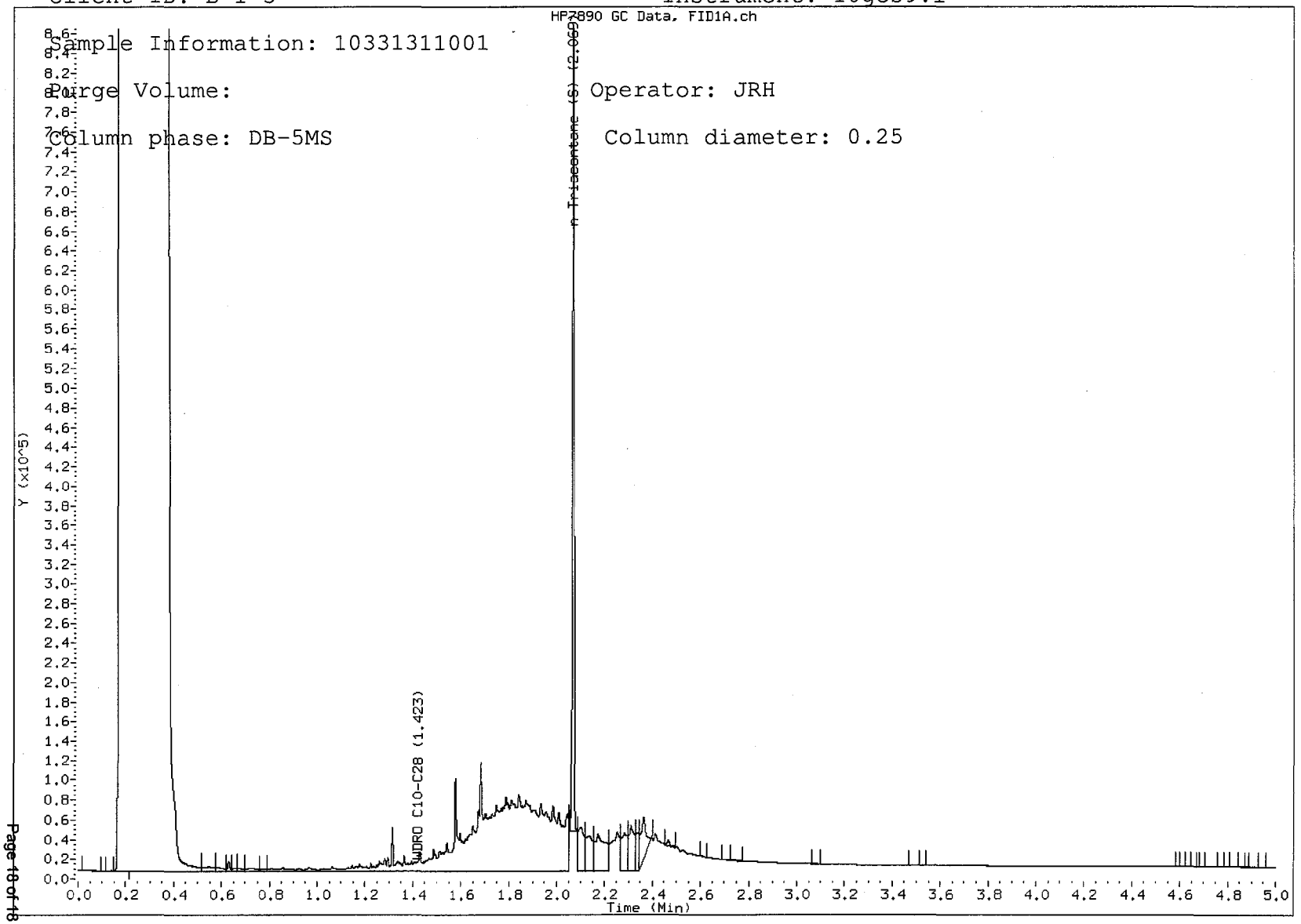
Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers).

Data File: \\192.168.10.12\chem\10gcs9.i\112915dro.b/112915000082.D

Report Date: 11/30/2015

Sample ID: 10331311001

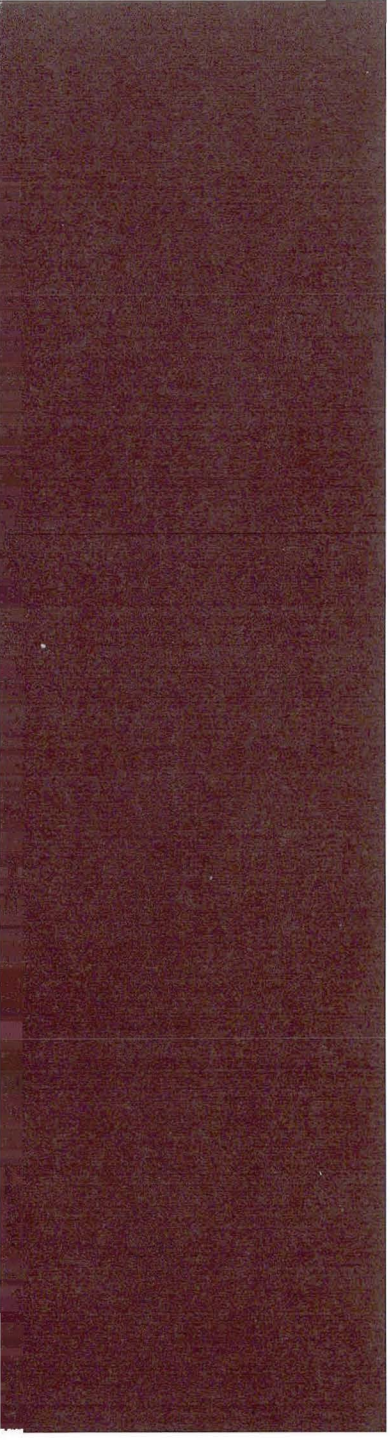
Client ID: B-1-3' Instrument: 10gcs9.i

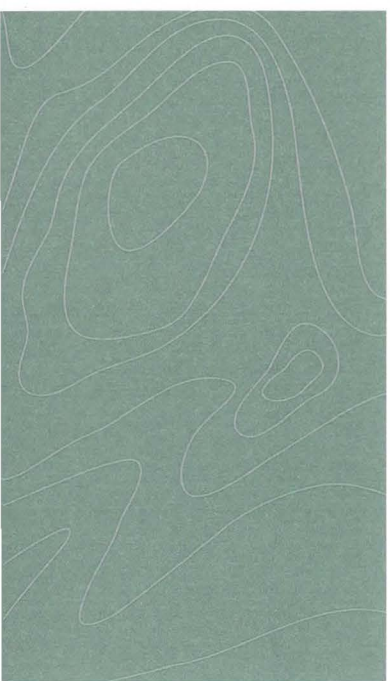


Sample Information: 10331311001

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25





BRAUN
INTERTEC

braunintertec.com

Rec'd 2/11/16

Environmental Investigation Report

Graymont (WI) LLC
800 Hill Avenue
Superior, WI

Prepared for

Graymont (WI) LLC

Project B1507395

Braun Intertec Corporation
October 15, 2015

BRAUN INTERTEC
The Science You Build On.

Braun Intertec Corporation
4511 West First Street, Suit 4
Duluth, MN 55807

Phone: 218.624.4967
Fax: 218.624.0196
Web: braunintertec.com

October 15, 2015

Project B1507395

Mr. Phil Marquis
Graymont (WI) LLC
800 Hill Avenue
Superior, WI 54880

Re: Environmental Investigation Report
Graymont (WI) LLC
800 Hill Avenue
Superior, WI

Dear Mr. Marquis:

On behalf of Graymont (WI) LLC, Braun Intertec Corporation conducted an Environmental Investigation of the referenced site (Site) in accordance with the authorized scope of services described in our proposal dated August 20, 2015. The Environmental Investigation was conducted to evaluate the potential environmental impacts to the soil and groundwater at the Site resulting from past storage and distribution of petroleum products during the time period when the property was managed by Koch Materials Company and operated as a Marine Fueling Terminal.

This Environmental Investigation Report was prepared on behalf of and for use by Graymont (WI) LLC. No other party has a right to rely on the contents of this Environmental Investigation without the written authorization of Braun Intertec.

For a complete discussion of our assessment, please refer to the attached Environmental Investigation Report.

We appreciate the opportunity to provide our professional services to you for this project. If you have any questions or comments regarding this report or the project in general, please call Ted Hubbes at 218.263.8869.

Sincerely,

BRAUN INTERTEC CORPORATION

Samantha Buhr
Environmental Technician

Ted Hubbes, PG, CHMM
Senior Scientist/Associate Principal

Attachment:
Environmental Investigation Report

AA/EOE

Table of Contents

A. Introduction

A.1. Authorization

Braun Intertec Corporation received authorization from Mr. Phil Marquis of Graymont (WI) LLC to conduct an Environmental Investigation of the Graymont located at 800 Hill Avenue in Superior, Wisconsin (Site) in accordance with the scope of services described in our proposal dated August 6, 2015. The Environmental Investigation was prepared to evaluate petroleum products observed seeping through a paved surface at the Site in the area of the Former Marine Fueling Terminal.

A.2. Project Objective

The objective of the Environmental Investigation was to evaluate potential soil and groundwater petroleum impacts present at the Site, resulting from previous aboveground storage tanks (ASTS) and/or historic property use as a Marine Fueling Terminal. Five soil borings were advanced in pre-determined locations throughout the Site on August 20, 2015, that were chosen by Braun Intertec and approved by Graymont (WI) LLC. The attached Site Sketch illustrates the general locations of the soil borings (Figure 2).

In addition, visually impacted soil (approximately 2-3 cubic yards) had been encountered and stockpiled during a recent construction project conducted by Graymont, in an area nearby a new silo.

B. Site Background

B.1. Site Location and Description

We accessed various documents and online sources to obtain Site location information. The following is a summary of our findings:

Address:	810 Hill Avenue
City:	Superior
County:	Douglas
Latitude:	46°43'44.11"N
Longitude:	92°04'39.20"W
Section, Township, Range:	W 1/2 of the NW 1/4 of Section 13 and the E 1/2 of the NE 1/4 of section 14 of Twp 49 North , Range 14W
Size:	33.38-acres/1,454,128 sq. ft.

The Site is currently developed as an industrial facility that takes delivery of limestone from quarries in Lower Michigan via self-unloading lake freighter, and processes the limestone into lime using kilns.

Historically, the Site was used as a bulk fuel terminal owned by Koch Materials Company from at least 1952 until about 1998.

The Site is bordered on the north by Superior Bay followed by Lake Superior beyond, on the south by commercial properties followed by Highway 53, on the east by Superior Bay followed by Lake Superior, and the west by vacant land followed by Highway 53. A Site Location Map is included as Figure 1.

B.2. Previous Investigation Data

Prior to preparing this report, we conducted a brief review of the following reports provided by Graymont:

- **Phase I Environmental Site Assessment** (Phase I ESA), Koch Materials Company, Marine Fueling Division Terminal, Superior, Wisconsin. Prepared by STS Consultants, Ltd., and dated December 6, 1991.
- **Phase II Contamination Assessment**, Koch Materials Company, Marine Fueling Terminal, Superior, Wisconsin. Prepared by STS Consultants, Ltd., and dated June 18, 1992.
- **Phase III Contamination Assessment**, Koch Materials Company, Marine Fueling Terminal, Superior, Wisconsin. Prepared by STS Consultants, Ltd., and dated March 1, 1996.
- **Site Closure Report**, Koch Refining Company, Former Marine Fueling Facility, Superior, Wisconsin. Prepared by Barr Engineering Company, and dated November 1998.
- **Submittal of Case Summary and Close Out Form**, Former Marine Fueling Facility, Superior, Wisconsin. Prepared by Barr Engineering Company, and dated December 7, 1998.

The Phase I report indicates that the observed impacts in the PLS3 area are in the general vicinity of "Tank 4" in the Former Marine Fueling Facility tank farm. The observed pavement seep is in the general vicinity of "Tank 7" in the Former Marine Fueling Facility tank farm. Previous soil borings conducted in these areas in the early 1990s indicated high concentrations of petroleum impacts in soil. Groundwater was present at 1.5 to 4.5 feet below ground surface (bgs) in these areas and free-floating petroleum product was documented.

The Site Closure Report indicated that tanks and other structures were removed from the site in 1997. At that time, site remediation was conducted and consisted of removal of 1,860 cubic yards of petroleum impacted soil. The report indicated that approximately 25% of the soil was impacted with number 2 fuel

oil and approximately 75% was impacted with number 6 fuel oil. The report also indicated that residual soil impacts and dissolved phase (groundwater) contamination was left in place to degrade naturally. The remediation was guided by visual observations and post excavation soil sampling was not conducted.

B.3. Physical Setting

B.3.a. Topography

According to the United States Geological Survey (USGS) 7.5-minute topographic map series, Superior, Wisconsin quadrangle, the Site is located at an elevation of approximately 610 feet above mean sea level.

B.3.b. Geology

Surficial geology of the area consists of Lake-Modified Glacial Topography. Topography is subdued as the result of wave action or as the result of being deposited in a water-logged state during high stages of Lake Superior. Low relief, hummocky collapse topography is present in most places, but is drumlinized in some areas. Till is typically 1 to 20 meters thick (Clayton, Lee, 1984).

The depth to bedrock in the Site vicinity is approximately 50-100 feet below land surface (WI DNR, Ecological Landscapes of Wisconsin Handbook, 2011).

B.3.c. Hydrogeology

According to the USGS 7.5-minute topographic map series, Superior, Wisconsin quadrangle, the surface gradient in the vicinity of the Site was generally flat. The regional groundwater flow direction within the unconsolidated deposits in the vicinity of the Site may be to the north or northeast towards Superior Bay. The local direction of groundwater flow may be affected by nearby streams, lakes, wells and/or wetlands and may vary seasonally.

The Site-specific groundwater flow direction was not determined through direct measurement during this environmental investigation. Additional field investigation, beyond the Scope of Services of this field investigation, would be required to determine this information.

Groundwater was encountered in the soil borings advanced at the Site as part of the assessment at depths ranging from 3.5 to 5 feet bgs.

C. Scope of Services

The following tasks were conducted at the Site as part of this Environmental Investigation:

- Advanced five soil borings designated as SB-1 through SB-5 to evaluate soil and groundwater conditions.
- Screened soil samples collected from the soil borings for the presence of organic vapors using a photoionization detector (PID). Visual and olfactory observations regarding potential contamination also were made and recorded.
- Chemically analyzed all soil samples for the following parameters: BETX, polynuclear aromatic hydrocarbons (PAHs) and diesel-range organics (DRO).
- Chemically analyzed a soil sample from soils stockpiled during a recent construction project for landfill acceptance parameters: PAHs, DRO and total lead.
- Chemically analyzed groundwater samples for the presence of DROs and BETX. Groundwater was encountered during all but one (SB-5) soil borings.
- Evaluated data and prepared this report.

D. Investigation Methods and Procedures

The fieldwork relating to the investigation was conducted on August 20, 2015. The locations and ground surface elevations of the soil borings were approximated by measuring from the building walls. Investigation locations are shown on Figure 2.

Prior to beginning the field investigation, public utilities were cleared through Diggers Hotline.

Five direct push soil borings (designated SB-1 through SB-5) were advanced at the Site. In addition, one sample was collected from the stockpile. The following table identifies the investigation locations, rational for selection and analytical testing parameters.

Table 1. Soil Boring Summary

Investigation Location	Approx. Boring Depth (ft)	Rationale for Selection	Soil Testing	Groundwater Testing where encountered
SB-1	8	South of source area.	4' (1,2,3)	2,3
SB-2	8	Northeast of source area.	3.5' (1,2,3)	2,3
SB-3	8	Northwest of source area.	3' (1,2,3)	2,3
SB-4	8	Southeast of source area.	4' (1,2,3)	2,3
SB-5	8	Source area.	4' (1,2,3)	NA
Stockpile	NA	NA	1,2,4	NA

Notes:
S = Shallow soil sample
1 = BETX analysis
2 = DRO analysis
3 = PAH analysis
4 = Lead
NA = Not Applicable

The soil boring locations are shown on Figure 2.

Standard operating procedures used during the investigation are available upon request.

D.1. Soil Evaluation

D.1.a. Soil borings

We subcontracted Range Environmental Drilling, of Hibbing, Minnesota to advance five soil borings, designated as SB-1 through SB-5 at the Site to a depth of 8 feet below ground surface (bgs).

Soil borings SB-1 through SB-5 were advanced with a hydraulically-driven push-probe sampling rig. To collect the soil samples from the borings, a disposable thin-walled PVC liner was placed inside of a 5-foot long sampling tool. The borehole was then advanced using a dual-tube system, which allows for the inner sampling tool to be pushed through a larger outer-diameter rod to a total penetration depth of 5 feet. After advancing the tooling, the sample was removed from the borehole, but the outer rod remained, keeping the borehole open, and soil sample was retrieved from the PVC liner for field screening and classification. The process was then repeated to the termination depths of the borings.

Upon completion, soil borings were sealed in accordance with Wisconsin regulations.

D.1.b. Soil Classification and Monitoring

The soils encountered in the soil borings were visually and manually classified in the field by an environmental technician using ASTM D 2487 "Unified Soils Classification System" and ASTM D 2488 "Recommended Practice for Visual and Manual Description of Soils."

Soil samples retrieved were examined by an environmental technician, for unusual staining, odors, and other apparent signs of contamination. In addition, the soil samples were screened for the presence of organic vapors using a PID. The PID was equipped with a 10.6 electron-volt lamp and calibrated to an isobutylene standard. The PID was used to perform direct measurement and a headspace method of field-analyses.

D.1.c. Soil Chemical Analyses

Selected soil samples for chemical analyses were collected from the soil borings. If indications of contamination were observed in the field, samples for chemical analyses were collected from that interval. If no indications of contamination were observed, the soil samples were collected from the depth most likely to be impacted based on the potential contaminant source. Samples were analyzed for a combination of the following parameters:

- BETX using United States Environmental Protection Agency (EPA) Method 8260
- PAHs using EPA method 8270D
- DRO using the Wisconsin Department of Natural Resources Method (WDNR)
- Lead using US EPA Method 6010

The rationale that was used to select the sampling interval at each location along with the parameters in which samples were analyzed is shown below.

Table 2. Summary of Sampling Intervals and Chemical Analysis Parameters

Soil Boring	Sampling Depth (ft)	Rationale	Analytical Parameters
SB-1	4	North of source area.	BETX, PAHs and DRO
SB-2	3.5	South of source area.	BETX, PAHs and DRO
SB-3	4	West of source area.	BETX, PAHs and DRO
SB-4	4	East of source area.	BETX, PAHs and DRO
SB-5	3	Source area.	BETX, PAHs and DRO
Stock Pile	NA	NA	BETX, DRO, Lead

D.2. Groundwater Evaluation

To evaluate groundwater conditions at the Site, grab-groundwater samples were collected from all borings with the exception of boring SB-5, where no groundwater was encountered.

D.2.a. Groundwater Chemical Analyses

The groundwater samples collected were analyzed for the following parameters:

- BETX using EPA Method 8260
- PAHs using EPA Method 8270D
- DRO using the WDNR Method

E. Investigation Results

E.1. Geologic Conditions

Soil boring logs with descriptions of the various soil strata encountered during the soil boring operations and water-level information are contained in Appendix A. The depths shown as changes between the soil types are approximate. The actual changes may be transitional, and the transition depths are likely to be horizontally variable.

E.2. Field Screening

During the field investigation, soil samples were examined visually by an environmental technician for staining or other apparent signs of contamination. In addition, the soil samples were screened for the presence and concentration of organic vapors with a photoionization detector (PID) using a headspace procedure. The PID was equipped with a 10-electron-volt lamp and calibrated to an isobutylene standard to provide direct readings of relative organic vapor concentrations in parts per million (ppm).

The headspace procedure consisted of collection a soil sample to half fill a quart-size, self-sealing plastic bag. The bag was vigorously shaken for 15 seconds and allowed to set for at least 10 minutes. Following the 10-minute period, the sample bag was again vigorously shaken for 15 seconds. The bag was then opened slightly and the PID probe was inserted to one-half of the headspace depth. The highest reading observed on the PID was then recorded.

No obvious visual or olfactory indications of contamination were observed in the soil samples collected with the exception of the sample collected from SB-05 (3'), where a petroleum odor was detected..

A summary of the soil screening results for the soil samples is provided in Table 3.

E.3. Soil Analytical Testing Results

Chemical components detected in the soil samples are compared with their respective State of Wisconsin Non-industrial Not-to-Exceed Direct-Contact (D-C) Residual Contaminant Levels (RCL) and the Groundwater Pathway Residual Contaminant Level (GW RCL) based on the US EPA regional screening levels. The D-C RCLs and GW RCLs are expressed as a concentration in milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg).

Soil analytical results are discussed below. A detailed summary of the soil analytical results are provided in Table 4.

The results of the laboratory analysis indicated that:

- DRO was detected in Soil Samples SB-1 (4'), SB-3 (4'), SB-5 (3') and the Stockpile sample at concentrations of 23 mg/kg, 4,560 mg/kg, 24,100 mg/kg and 10,800 mg/kg, respectively. There is no established standard within the State of Wisconsin for DRO.

- The results of the laboratory analysis for lead indicated detections of lead in the stockpile sample at a concentration of 9.5 mg/kg which does not exceed the State of Wisconsin's Non-Industrial Not-to-Exceed D-C RCL and the GW RCL.

- The results of the laboratory analysis indicated detections of the following PAH compounds above their respective D-C RCLs:

    - Sample SB-3 (4') indicated detections of Benzo(a)pyrene 0.23 mg/kg and Chrysene of 0.65 mg/kg.
    - Sample SB-5 (3') indicated detections of Benzo(a)pyrene 5.4 mg/kg, Benzo(b)fluoranthene 2.9 mg/kg and Chrysene 12.7 mg/kg.

- No BETX compounds were detected at concentrations greater than or equal to the laboratory method reporting limits, with the exception of Toluene. Toluene was detected in all of the soil samples, but was at concentrations that did not exceed the State of Wisconsin's Non-Industrial Not-to-Exceed D-C RCL and the GW RCL.

The complete laboratory reports with chain of custody forms are attached in Appendix B.

E.4. Groundwater Analytical Testing Results

A summary of the analytical results for the groundwater samples is provided in Table 5.

The results of the laboratory analysis indicated that:

- DRO was detected in groundwater samples from SB-1, SB-3 and SB-4 at concentrations of 262 ug/l, 870 ug/l and 299 ug/l, respectively.
- No BETX was detected in any of the groundwater samples collected at concentrations greater than or equal to the laboratory method reporting limits.

The complete laboratory report with chain of custody forms is attached in Appendix B.

E.5. Quality Assurance/Quality Control

Samples were placed in clean, laboratory supplied containers, preserved, labeled, and transported to the Pace Analytical laboratory under refrigerated conditions using chain-of-custody procedures.

Analyses were performed using EPA or other recognized standard procedures. Data were reviewed prior to release, quality-control guidelines were generally met, and the data is considered usable. Specific information on standard operating procedures, detection limits, and quality-control measures is available upon request.

F. Conclusions

Based on the results of the Environmental Investigation, we conclude the following:

- Fill soil was identified at depths ranging from 0 feet to 8 feet bgs at the Site.
- DRO was detected in Soil Samples SB-1 (4'), SB-3 (4'), SB-5 (3') and the Stockpile sample at concentrations of 23 mg/kg, 4560 mg/kg, 24,100 mg/kg and 10,800 mg/kg, respectively. There is no established standard within the State of Wisconsin for DRO.
- The results of the laboratory analysis indicated detections in soil samples SB-3 and SB-5 of the following PAH compounds above their respective D-C RCLs,

  - Sample SB-3 (4') indicated detections of Benzo(a)pyrene 0.23 mg/kg and Chrysene of 0.65 mg/kg.
  - Sample SB-5 (3') indicated detections of Benzo(a)pyrene 5.4 mg/kg, Benzo(b)fluoranthene 2.9 mg/kg and Chrysene 12.7 mg/kg.
- No BETX compounds were detected in soil samples at concentrations greater than or equal to the laboratory method reporting limits, with the exception of Toluene. Toluene was detected in all of the soil samples, but was at concentrations that did not exceed the State of Wisconsin's Non-Industrial Not-to-Exceed D-C RCL and the GW RCL.
- DRO was detected in groundwater samples from SB-1, SB-3 and SB-4 at concentrations of 262 ug/l, 870 ug/l and 299 ug/l, respectively.
- No BETX was detected in any of the groundwater samples collected at concentrations greater than or equal to the laboratory method reporting limits.

G. Recommendations

In accordance with ch. 292 Wis. Stats. the detected soil impacts are reportable to the Wisconsin Department of Natural Resources. Spills can be reported online using WDNRs form "Notification for Hazardous Substance Discharge Non-Emergence Only" http://dnr.wi.gov/files/pdf/forms/4400/4400-225.pdf . At the request of Graymont (WI) LLC. the observed seep was previously reported to WDNR. We are not aware of any correspondence or actions taken by WDNR related to the reported release.

It appears likely that the excavated soil stockpile would be acceptable for disposal at a licensed landfill. We recommend obtaining proper approvals and manifests, and disposing of the soil at a licensed facility.

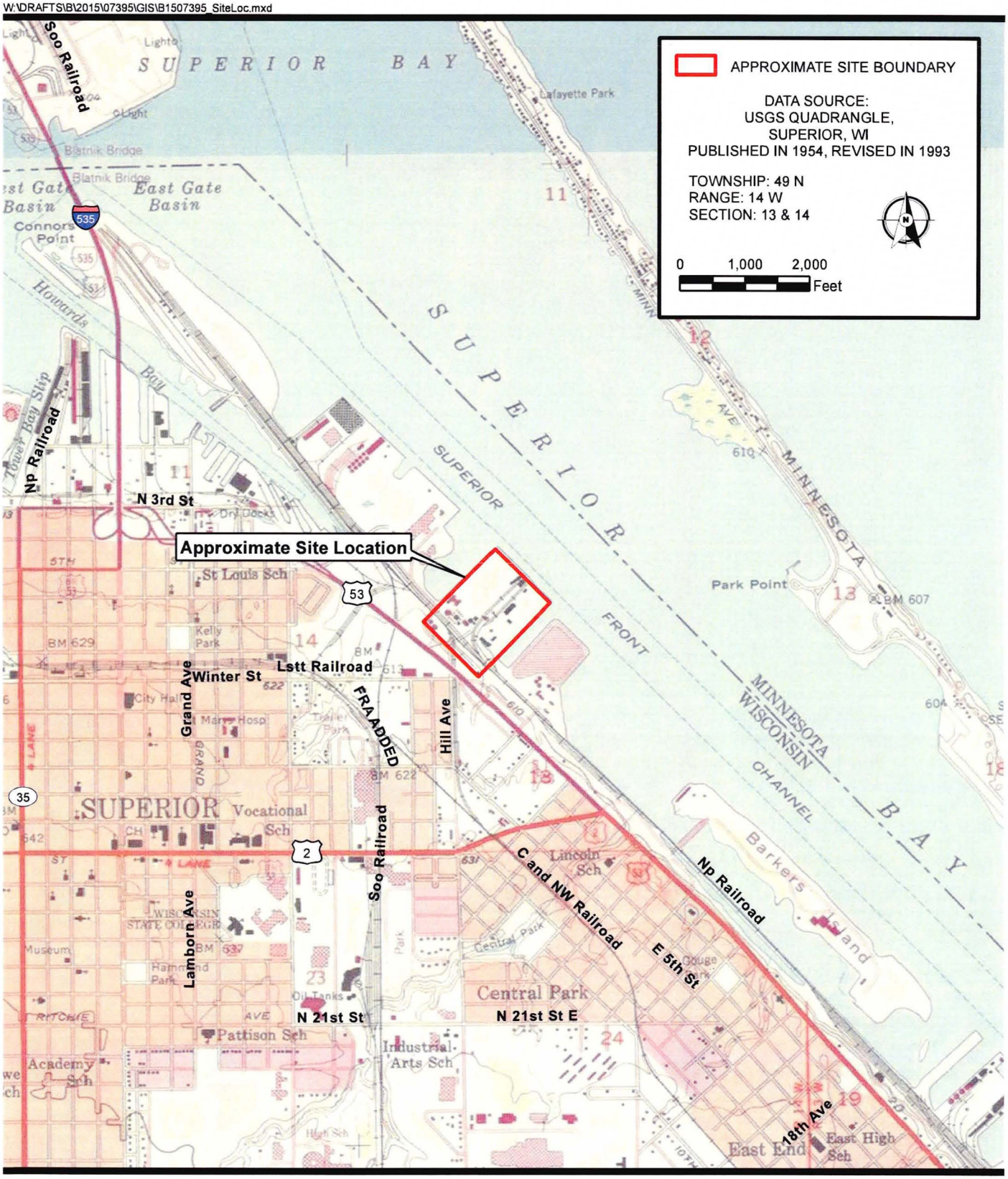
H. Assessment Limitations

The analyses and conclusions submitted in this report are based on our field observations and the results of laboratory chemical analyses of soil samples and groundwater samples collected from the soil borings completed for this project.

In performing its services, Braun Intertec used that degree of care and skill ordinarily exercised under similar circumstances by reputable members of its profession currently practicing in the same locality. No warranty, express or implied, is made.

Figures

W:\DRAFTS\B\2015\07395\GIS\B1507395_SiteLoc.mxd



Project No:	B1507395
Drawing No.	B1507395_SiteLoc
Scale:	1 in = 2,000 ft
Drawn By:	FER
Date Drawn:	9/21/15
Checked By:	SB
Last Modified:	9/21/15

Sheet: 1 of 1

Fig: 1

SITE LOCATION MAP
GRAYMONT
800 HILL AVENUE
SUPERIOR, WISCONSIN

BRAUN INTERTEC
11001 Hampshire Avenue So.
Minneapolis, MN 55438
PH. (952) 995-2000
FAX (952) 995-2020

F:\2015\B1507395.dwg,Soil Inv,10/14/2015 7:04:56 AM

SOIL BORING LOCATION

N

30' 0 60'

SCALE: 1"= 60'

Sheet: of

Fig:

Project No:	B1507395
Drawing No:	B1507395
Scale:	1"= 60'
Drawn By:	JAG
Date Drawn:	9/15/15
Checked By:	SB
Last Modified:	10/14/15

SOIL BORING LOCATION SKETCH
SOIL INVESTIGATION
GRAYMONT P.O. NO. 265591 OP
800 HILL AVENUE
SUPERIOR, WISCONSIN

BRAUN INTERTEC
The Science You Build On.
11001 Hampshire Avenue S
Minneapolis, MN 55438
PH. (952) 995-2000
FAX (952) 995-2020

Tables

Table 3
Summary of Soil Screening Results
Graymont
Superior, WI
Project: B1507395

Depth (ft)	SB-1	SB-2	SB-3	SB-4	SB-5
0-2'	ND	ND	ND	ND	ND
2-4'	ND	ND	ND	ND	3.0
4-6'	ND	ND	2.0	ND	ND
6-8'	ND	ND	ND	ND	ND

Note:
Concentrations in parts per million (ppm)
- = No sample recovery
ND = Non Detected

Table 4

Soil Analytical Results

Graymont

800 Hill Avenue Superior, WI

B1507395

Compound/Parameter	Sample SB-4 (4') 8/20/15 1:50	Sample SB-3 (4') 8/20/15 12:40	Sample SB-5 (3') 8/20/15 3:05	Sample SB-2 (3.5') 8/20/15 11:15	Sample SB-1 (4') 8/20/15 10:00	Sample STOCK PILE 8/20/15 3:30	Not to Exceed D-C RCL[2] (mg/kg)	GW RCL[3] (mg/kg)
Volatile Organic Compounds (mg/kg)								
Benzene	<0.025	<0.023	<0.027	<0.024	<0.025	<0.021	7.41	0.0051
Ethylbenzene	<0.062	<0.056	<0.066	<0.060	<0.063	<0.054	37	1.57
Toluene	**0.15**	**0.12**	**0.12**	**0.14**	**0.16**	**0.13**	818	1.1072
Xylene (Total)	<0.19	<0.17	<0.20	<0.18	<0.19	<0.16	258	3.94
Semivolatile Organic Compounds (mg/kg)								
Acenaphthene	<0.012	**0.14**	**9.4**	<0.012	<0.012	---	33,000	NE
Acenaphthylene	<0.012	**0.057**	<2.6	<0.012	<0.012	---	NE	NE
Anthracene	<0.012	**0.53**	<2.6	<0.012	<0.012	---	100,000	197.7273
Benzo(a)anthracene	**0.028**	**0.41**	**6.0**	<0.012	<0.012	---	NE	NE
Benzo(a)pyrene	**0.031**	**0.23**	**5.4**	<0.012	<0.012	---	0.211	0.47
Benzo(b)fluoranthene	**0.019**	**0.12**	**2.9**	<0.012	<0.012	---	2.11	0.4793
Benzo(g,h,i)perylene	**0.020**	**0.097**	<2.6	<0.012	<0.012	---	NE	NE
Benzo(k)fluoranthene	<0.012	<0.056	<2.6	<0.012	<0.012	---	21.1	NE
Chrysene	**0.045**	**0.65**	**12.7**	<0.012	<0.012	---	211	0.1446
Dibenz(a,h)anthracene	<0.012	<0.056	<2.6	<0.012	<0.012	---	0.211	NE
Fluoranthene	**0.026**	**0.27**	**3.3**	<0.012	<0.012	---	22,000	88.878
Fluorene	<0.012	**0.41**	**11.2**	<0.012	<0.012	---	22,000	14.8027
Indeno(1,2,3-cd)pyrene	<0.012	<0.056	<2.6	<0.012	<0.012	---	2.11	NE
Naphthalene	**0.026**	<0.056	<2.6	<0.012	<0.012	---	26	0.6582
Phenanthrene	**0.057**	**0.68**	**16.5**	<0.012	<0.012	---	NE	NE
Pyrene	**0.077**	**1.7**	**12.6**	<0.012	<0.012	---	16,500	54.1322
Total Petroleum Hydrocarbons (mg/kg)								
WDRO C10-C28	<11.1	**4560**	**24100**	<11.3	**23**	**10800**	NE	NE
Metals (mg/kg)								
Lead	---	---	---	---	---	**9.5**	400	27

Notes:

mg/kg = Milligrams per kilogram.

< = Less than the reporting limit

---- = Not analyzed or calculated for this

NE = No Wisconsin Soil Standards have been

[2]- Not-to-Exceed Direct - Contact (D-C) Redidual Contaminant Level (RCL) based on U.S. EPA reginal screening level web calculator (non industrial)

[3]- Groundwater Pathway RCL based on the U.S. EPA regional screening level web calculator

Exceeds D-C RCL[2] and/or GW RCL[3]

Table 5

Groundwater Analytical Results

Graymont

800 Hill Avenue Superior, WI

B1507395

Compound/Parameter	Sample	Sample	Sample	Sample	WI DNR 140 ES
	SB-4	**SB-3**	**SB-2**	**SB-1**	
	8/20/15 1:59	**8/20/15 12:52**	**8/20/15 11:30**	**8/20/15 10:11**	
Volatile Organic Compounds (ug/L)					
Benzene	<1.0	<1.0	<1.0	<1.0	5
Ethylbenzene	<1.0	<1.0	<1.0	<1.0	700
Toluene	<1.0	<1.0	<1.0	<1.0	800
Xylene (Total)	<3.0	<3.0	<3.0	<3.0	2000
Total Petroleum Hydrocarbons (ug/L)					
Diesel Range Organics	299	870	<116	262	NE

Notes:

ug/L = Micrograms per liter.

< = Less than the reporting limit

NE = Not Established

WI DEPARTMENT OF NATURAL RESOURCES, REGISTER, NO.578, SUBCHAPTER II- GROUNDWATER QUALITY STANDARDS NR140.10 EFFECTIVE FEBRUARY, 2005

NR 140 ES = Enforcement Standards

Appendix A

Soil Borings Logs

BRAUN INTERTEC

LOG OF BORING

Braun Project B1507395
Soil Investigation
Graymont
800 Hill Avenue
Superior, Wisconsin

BORING: **SB-01**

LOCATION: See attached sketch.

DRILLER: Range Environmental | METHOD: Geoprobe | DATE: **8/20/15** | SCALE: **1" = 4'**

Depth feet	Symbol	Description of Materials (Soil-ASTM D2488 or D2487, Rock-USACE EM1110-1-2908)	BPF	WL	PID ppm	Tests or Notes
0.0	FILL	FILL: Poorly Graded Sand with Silt.			ND	
					ND	
				▽		An open triangle in the water level (WL) column indicates the depth at which groundwater was observed while drilling. Groundwater levels fluctuate.
					ND	
8.0					ND	
		END OF BORING.				
		Water observed at 4 feet while drilling.				

(See Descriptive Terminology sheet for explanation of abbreviations)

LOG OF BORING N:\GINT\PROJECTS\AX PROJECTS\2015\07395.GPJ BRAUN_V8_CURRENT.GDT 9/23/15 14:37

BRAUN INTERTEC

LOG OF BORING

Braun Project B1507395
Soil Investigation
Graymont
800 Hill Avenue
Superior, Wisconsin

BORING: **SB-02**

LOCATION: See attached sketch.

DRILLER: Range Environmental | METHOD: Geoprobe | DATE: **8/20/15** | SCALE: **1" = 4'**

(See Descriptive Terminology sheet for explanation of abbreviations)

Depth feet	Symbol	Description of Materials (Soil-ASTM D2488 or D2487, Rock-USACE EM1110-1-2908)	BPF	WL	PID ppm	Tests or Notes
0.0						
0.3	BIT	4 inches of bituminous surfacing.				
	FILL	FILL: Poorly Graded Sand with Silt.			ND	
				▽	ND	
					ND	
					ND	
8.0		END OF BORING.				
		Water observed at 3 1/2 feet while drilling.				

LOG OF BORING N:\GINT\PROJECTS\AX PROJECTS\2015\07395.GPJ BRAUN_V8_CURRENT.GDT 9/23/15 14:37

BRAUN INTERTEC

LOG OF BORING

Braun Project B1507395
Soil Investigation
Graymont
800 Hill Avenue
Superior, Wisconsin

BORING: **SB-03**

LOCATION: See attached sketch.

DRILLER: Range Environmental | METHOD: Geoprobe | DATE: **8/20/15** | SCALE: **1" = 4'**

(See Descriptive Terminology sheet for explanation of abbreviations)

Depth feet	Symbol	Description of Materials (Soil-ASTM D2488 or D2487, Rock-USACE EM1110-1-2908)	BPF	WL	PID ppm	Tests or Notes
0.0	FILL	FILL: Poorly Graded Sand with Silt.			ND	
					ND	
5.0				▽		
5.5	FILL	FILL: Poorly Graded Sand with Silt, black.			2.0	
	FILL	FILL: Poorly Graded Sand with Silt.			ND	
8.0		END OF BORING.				
		Water observed at 5 feet while drilling.				

LOG OF BORING N:\GINT\PROJECTS\AX PROJECTS\2015\07395.GPJ BRAUN_V8_CURRENT.GDT 9/23/15 14:37

BRAUN INTERTEC

LOG OF BORING

Braun Project B1507395
Soil Investigation
Graymont
800 Hill Avenue
Superior, Wisconsin

BORING: **SB-04**

LOCATION: See attached sketch.

DRILLER: Range Environmental | METHOD: Geoprobe | DATE: **8/20/15** | SCALE: **1" = 4'**

Depth feet	Symbol	Description of Materials (Soil-ASTM D2488 or D2487, Rock-USACE EM1110-1-2908)	BPF	WL	PID ppm	Tests or Notes
0.0	FILL	FILL: Poorly Graded Sand with Silt.			ND	
				▽	ND	
					ND	
					ND	
8.0		END OF BORING.				
		Water observed at 4 feet while drilling.				

(See Descriptive Terminology sheet for explanation of abbreviations)

LOG OF BORING N:\GINT\PROJECTS\AX PROJECTS\2015\07395.GPJ BRAUN_V8_CURRENT.GDT 9/23/15 14:37

BRAUN INTERTEC

LOG OF BORING

Braun Project B1507395
Soil Investigation
Graymont
800 Hill Avenue
Superior, Wisconsin

BORING: **SB-05**

LOCATION: See attached sketch.

DRILLER: Range Environmental | METHOD: Geoprobe | DATE: **8/20/15** | SCALE: **1" = 4'**

Depth feet	Symbol	Description of Materials (Soil-ASTM D2488 or D2487, Rock-USACE EM1110-1-2908)	BPF	WL	PID ppm	Tests or Notes
0.0						
0.3	BIT	4 inches of bituminous surfacing.				
	FILL	FILL: Poorly Graded Sand with Silt. (Limestone)			ND	
2.0	CL-ML	SILTY CLAY, with rocks.			3.0*	*Petroleum odor.
					ND	
8.0					ND	
		END OF BORING.				

(See Descriptive Terminology sheet for explanation of abbreviations)

LOG OF BORING N:\GINT\PROJECTS\AX PROJECTS\2015\07395.GPJ BRAUN_V8_CURRENT.GDT 9/23/15 14:37

Appendix B

Laboratory Reports

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

September 02, 2015

Ted Hubbes
Braun Intertec
4511 W First St.
#4
Duluth, MN 55807

RE: Project: Graymont Superior, WI
Pace Project No.: 10319784

Dear Ted Hubbes:
Enclosed are the analytical results for sample(s) received by the laboratory on August 25, 2015. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

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

Sincerely,

Zach Wagner
zach.wagner@pacelabs.com
Project Coordinator

Enclosures

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

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

CERTIFICATIONS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Minnesota Certification IDs
1700 Elm Street SE Suite 200, Minneapolis, MN 55414
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Alabama Certification #40770
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: 8TMS-L
Florida/NELAP Certification #: E87605
Guam Certification #:14-008r
Georgia Certification #: 959
Georgia EPD #: Pace
Idaho Certification #: MN00064
Hawaii Certification #MN00064
Illinois Certification #: 200011
Indiana Certification#C-MN-01
Iowa Certification #: 368
Kansas Certification #: E-10167
Kentucky Dept of Envi. Protection - DW #90062
Kentucky Dept of Envi. Protection - WW #:90062
Louisiana DEQ Certification #: 3086
Louisiana DHH #: LA140001
Maine Certification #: 2013011
Maryland Certification #: 322
Michigan DEPH Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Carolina State Public Health #: 27700
North Dakota Certification #: R-036
Ohio EPA #: 4150
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Saipan (CNMI) #:MP0003
South Carolina #:74003001
Texas Certification #: T104704192
Tennessee Certification #: 02818
Utah Certification #: MN000642013-4
Virginia DGS Certification #: 251
Virginia/VELAP Certification #: Pace
Washington Certification #: C486
West Virginia Certification #: 382
West Virginia DHHR #:9952C
Wisconsin Certification #: 999407970

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

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

SAMPLE SUMMARY

Project: Graymont Superior, WI
Pace Project No.: 10319784

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10319784001	SB-4	Water	08/20/15 01:59	08/25/15 09:10
10319784002	SB-4 (4')	Solid	08/20/15 01:50	08/25/15 09:10
10319784003	SB-3	Water	08/20/15 12:52	08/25/15 09:10
10319784004	SB-3 (4')	Solid	08/20/15 12:40	08/25/15 09:10
10319784005	SB-5 (3')	Solid	08/20/15 03:05	08/25/15 09:10
10319784006	SB-2	Water	08/20/15 11:30	08/25/15 09:10
10319784007	SB-2 (3.5')	Solid	08/20/15 11:15	08/25/15 09:10
10319784008	SB-1	Water	08/20/15 10:11	08/25/15 09:10
10319784009	SB-1 (4')	Solid	08/20/15 10:00	08/25/15 09:10
10319784010	STOCK PILE	Solid	08/20/15 03:30	08/25/15 09:10

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

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

SAMPLE ANALYTE COUNT

Project: Graymont Superior, WI
Pace Project No.: 10319784

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10319784001	SB-4	WI MOD DRO	JRH	2	PASI-M
		EPA 8260B	DJB	7	PASI-M
10319784002	SB-4 (4')	WI MOD DRO	JRH	2	PASI-M
		ASTM D2974	AH3	1	PASI-M
		EPA 8270D by SIM	AS1	19	PASI-M
		EPA 8260B	AMT	7	PASI-M
10319784003	SB-3	WI MOD DRO	JRH	2	PASI-M
		EPA 8260B	DJB	7	PASI-M
10319784004	SB-3 (4')	WI MOD DRO	JRH	2	PASI-M
		ASTM D2974	AH3	1	PASI-M
		EPA 8270D by SIM	AS1	19	PASI-M
		EPA 8260B	AMT	7	PASI-M
10319784005	SB-5 (3')	WI MOD DRO	JRH	2	PASI-M
		ASTM D2974	AH3	1	PASI-M
		EPA 8270D by SIM	AS1	19	PASI-M
		EPA 8260B	AMT	7	PASI-M
10319784006	SB-2	WI MOD DRO	JRH	2	PASI-M
		EPA 8260B	DJB	7	PASI-M
10319784007	SB-2 (3.5')	WI MOD DRO	JRH	2	PASI-M
		ASTM D2974	AH3	1	PASI-M
		EPA 8270D by SIM	AS1	19	PASI-M
		EPA 8260B	AH2	7	PASI-M
10319784008	SB-1	WI MOD DRO	JRH	2	PASI-M
		EPA 8260B	DJB	7	PASI-M
10319784009	SB-1 (4')	WI MOD DRO	JRH	2	PASI-M
		ASTM D2974	AH3	1	PASI-M
		EPA 8270D by SIM	AS1	19	PASI-M
		EPA 8260B	AMT	7	PASI-M
10319784010	STOCK PILE	WI MOD DRO	JRH	2	PASI-M
		EPA 6010C	BD1	1	PASI-M
		ASTM D2974	AH3	1	PASI-M
		EPA 8260B	AMT	7	PASI-M

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

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **WI MOD DRO**
Description: WIDRO GCS
Client: Braun-BLM
Date: September 02, 2015

General Information:
6 samples were analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

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.

QC Batch: OEXT/30569

S4: Surrogate recovery not evaluated against control limits due to sample dilution.
- SB-3 (4') (Lab ID: 10319784004)
  - n-Triacontane (S)
- SB-5 (3') (Lab ID: 10319784005)
  - n-Triacontane (S)
- STOCK PILE (Lab ID: 10319784010)
  - n-Triacontane (S)

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

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

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

Additional Comments:

Analyte Comments:

QC Batch: OEXT/30569

T6: High boiling point hydrocarbons are present in the sample.
- SB-1 (4') (Lab ID: 10319784009)
  - WDRO C10-C28

REPORT OF LABORATORY ANALYSIS

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

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **WI MOD DRO**
Description: WIDRO GCS
Client: Braun-BLM
Date: September 02, 2015

Analyte Comments:

QC Batch: OEXT/30569

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

- SB-3 (4') (Lab ID: 10319784004)
  - WDRO C10-C28
- SB-5 (3') (Lab ID: 10319784005)
  - WDRO C10-C28
- STOCK PILE (Lab ID: 10319784010)
  - WDRO C10-C28

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

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **WI MOD DRO**
Description: WIDRO GCS
Client: Braun-BLM
Date: September 02, 2015

General Information:
4 samples were analyzed for WI MOD DRO. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

P4: Sample field preservation does not meet EPA or method recommendations for this analysis.
- SB-1 (Lab ID: 10319784008)
- SB-2 (Lab ID: 10319784006)
- SB-3 (Lab ID: 10319784003)
- SB-4 (Lab ID: 10319784001)

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, where applicable, with any exceptions noted below.

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

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

Additional Comments:

Analyte Comments:

QC Batch: OEXT/30558

T6: High boiling point hydrocarbons are present in the sample.
- SB-3 (Lab ID: 10319784003)
  - WDRO C10-C28

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

Pace Analytical®
www.pacelabs.com

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **EPA 6010C**
Description: 6010C MET ICP
Client: Braun-BLM
Date: September 02, 2015

General Information:
1 sample was analyzed for EPA 6010C. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

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

Sample Preparation:
The samples were prepared in accordance with EPA 3050 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.

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

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

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

Additional Comments:

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

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **EPA 8270D by SIM**
Description: 8270D MSSV PAH by SIM
Client: Braun-BLM
Date: September 02, 2015

General Information:
5 samples were analyzed for EPA 8270D by SIM. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

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

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

QC Batch: OEXT/30583

P3: Sample extract could not be concentrated to the routine final volume, resulting in elevated reporting limits.
- SB-5 (3') (Lab ID: 10319784005)

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

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

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

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

QC Batch: OEXT/30583

S4: Surrogate recovery not evaluated against control limits due to sample dilution.
- SB-5 (3') (Lab ID: 10319784005)
  - 2-Fluorobiphenyl (S)
  - p-Terphenyl-d14 (S)

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

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

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

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

Pace Analytical®
www.pacelabs.com

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **EPA 8270D by SIM**
Description: 8270D MSSV PAH by SIM
Client: Braun-BLM
Date: September 02, 2015

QC Batch: OEXT/30583

A matrix spike and/or matrix spike duplicate (MS/MSD) were performed on the following sample(s): 10319794002

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

- MSD (Lab ID: 2063448)
  - Acenaphthene
  - Anthracene
  - Benzo(a)anthracene
  - Benzo(a)pyrene
  - Benzo(b)fluoranthene
  - Benzo(g,h,i)perylene
  - Benzo(k)fluoranthene
  - Chrysene
  - Dibenz(a,h)anthracene
  - Fluoranthene
  - Fluorene
  - Indeno(1,2,3-cd)pyrene
  - Naphthalene
  - Phenanthrene
  - Pyrene

R1: RPD value was outside control limits.

- MSD (Lab ID: 2063448)
  - Acenaphthene
  - Anthracene
  - Benzo(a)anthracene
  - Benzo(a)pyrene
  - Benzo(b)fluoranthene
  - Benzo(g,h,i)perylene
  - Benzo(k)fluoranthene
  - Chrysene
  - Dibenz(a,h)anthracene
  - Fluoranthene
  - Fluorene
  - Indeno(1,2,3-cd)pyrene
  - Naphthalene
  - Phenanthrene
  - Pyrene

Additional Comments:

Analyte Comments:

QC Batch: OEXT/30583

D4: Sample was diluted due to the presence of high levels of target analytes.

- SB-3 (4') (Lab ID: 10319784004)
  - 2-Fluorobiphenyl (S)

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

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **EPA 8270D by SIM**
Description: 8270D MSSV PAH by SIM
Client: Braun-BLM
Date: September 02, 2015

Analyte Comments:

QC Batch: OEXT/30583

D4: Sample was diluted due to the presence of high levels of target analytes.
- SB-5 (3') (Lab ID: 10319784005)
  - 2-Fluorobiphenyl (S)

E: Analyte concentration exceeded the calibration range. The reported result is estimated.
- MSD (Lab ID: 2063448)
  - Anthracene
  - Benzo(k)fluoranthene
  - Benzo(g,h,i)perylene
  - Benzo(a)anthracene
  - Benzo(b)fluoranthene
  - Benzo(a)pyrene
  - Chrysene
  - Fluoranthene
  - Indeno(1,2,3-cd)pyrene
  - Phenanthrene
  - Pyrene

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

Pace Analytical®
www.pacelabs.com

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **EPA 8260B**
Description: 8260B MSV UST
Client: Braun-BLM
Date: September 02, 2015

General Information:
10 samples were analyzed for EPA 8260B. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

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

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

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

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

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

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

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

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

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

Additional Comments:

Analyte Comments:

QC Batch: MSV/32969

1M: The sample containers provided did not have a tare weight for the methanol and sample container. The lab is assuming 10 mLs of methanol and 10 grams of sample. The results should be considered an estimation.

- SB-1 (4') (Lab ID: 10319784009)
  - 1,2-Dichloroethane-d4 (S)
- SB-2 (3.5') (Lab ID: 10319784007)
  - 1,2-Dichloroethane-d4 (S)
- SB-3 (4') (Lab ID: 10319784004)
  - 1,2-Dichloroethane-d4 (S)

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

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

PROJECT NARRATIVE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Method: **EPA 8260B**
Description: 8260B MSV UST
Client: Braun-BLM
Date: September 02, 2015

Analyte Comments:

QC Batch: MSV/32969

1M: The sample containers provided did not have a tare weight for the methanol and sample container. The lab is assuming 10 mLs of methanol and 10 grams of sample. The results should be considered an estimation.

- SB-4 (4') (Lab ID: 10319784002)
  - 1,2-Dichloroethane-d4 (S)
- SB-5 (3') (Lab ID: 10319784005)
  - 1,2-Dichloroethane-d4 (S)
- STOCK PILE (Lab ID: 10319784010)
  - 1,2-Dichloroethane-d4 (S)

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

Melanie Ollila
Quality Assurance Officer

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

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-4 **Lab ID: 10319784001** Collected: 08/20/15 01:59 Received: 08/25/15 09:10 Matrix: Water

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	**299**	ug/L	113	1	08/27/15 08:39	08/28/15 15:07		
Surrogates								
n-Triacontane (S)	92	%.	50-150	1	08/27/15 08:39	08/28/15 15:07	638-68-6	P4
8260B MSV UST	Analytical Method: EPA 8260B							
Benzene	ND	ug/L	1.0	1		08/27/15 19:49	71-43-2	
Ethylbenzene	ND	ug/L	1.0	1		08/27/15 19:49	100-41-4	
Toluene	ND	ug/L	1.0	1		08/27/15 19:49	108-88-3	
Xylene (Total)	ND	ug/L	3.0	1		08/27/15 19:49	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	103	%.	75-125	1		08/27/15 19:49	17060-07-0	
Toluene-d8 (S)	98	%.	75-125	1		08/27/15 19:49	2037-26-5	
4-Bromofluorobenzene (S)	101	%.	75-125	1		08/27/15 19:49	460-00-4	

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

Pace Analytical®
www.pacelabs.com

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-4 (4') **Lab ID: 10319784002** Collected: 08/20/15 01:50 Received: 08/25/15 09:10 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	ND	mg/kg	11.1	1	08/27/15 12:32	08/28/15 10:25		
Surrogates								
n-Triacontane (S)	72	%.	50-150	1	08/27/15 12:32	08/28/15 10:25	638-68-6	
Dry Weight	Analytical Method: ASTM D2974							
Percent Moisture	**19.0**	%	0.10	1		08/31/15 19:15		
8270D MSSV PAH by SIM	Analytical Method: EPA 8270D by SIM Preparation Method: EPA 3550							
Acenaphthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	83-32-9	
Acenaphthylene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	208-96-8	
Anthracene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	120-12-7	
Benzo(a)anthracene	**0.028**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	56-55-3	
Benzo(a)pyrene	**0.031**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	50-32-8	
Benzo(b)fluoranthene	**0.019**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	205-99-2	
Benzo(g,h,i)perylene	**0.020**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	191-24-2	
Benzo(k)fluoranthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	207-08-9	
Chrysene	**0.045**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	218-01-9	
Dibenz(a,h)anthracene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	53-70-3	
Fluoranthene	**0.026**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	206-44-0	
Fluorene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	86-73-7	
Indeno(1,2,3-cd)pyrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	193-39-5	
Naphthalene	**0.026**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	91-20-3	
Phenanthrene	**0.057**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	85-01-8	
Pyrene	**0.077**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49	129-00-0	
Total BaP Eq. MN 2006sh. ND=0	**0.037**	mg/kg	0.012	1	08/28/15 09:36	08/30/15 18:49		
Surrogates								
2-Fluorobiphenyl (S)	74	%.	55-125	1	08/28/15 09:36	08/30/15 18:49	321-60-8	
p-Terphenyl-d14 (S)	79	%.	30-150	1	08/28/15 09:36	08/30/15 18:49	1718-51-0	
8260B MSV UST	Analytical Method: EPA 8260B Preparation Method: EPA 5035/5030B							
Benzene	ND	mg/kg	0.025	1	08/30/15 11:56	08/30/15 20:47	71-43-2	
Ethylbenzene	ND	mg/kg	0.062	1	08/30/15 11:56	08/30/15 20:47	100-41-4	
Toluene	**0.15**	mg/kg	0.062	1	08/30/15 11:56	08/30/15 20:47	108-88-3	
Xylene (Total)	ND	mg/kg	0.19	1	08/30/15 11:56	08/30/15 20:47	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	124	%.	55-150	1	08/30/15 11:56	08/30/15 20:47	17060-07-0	1M
Toluene-d8 (S)	103	%.	61-125	1	08/30/15 11:56	08/30/15 20:47	2037-26-5	
4-Bromofluorobenzene (S)	105	%.	54-131	1	08/30/15 11:56	08/30/15 20:47	460-00-4	

REPORT OF LABORATORY ANALYSIS

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

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-3 **Lab ID: 10319784003** Collected: 08/20/15 12:52 Received: 08/25/15 09:10 Matrix: Water

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	**870**	ug/L	114	1	08/27/15 08:39	08/28/15 11:19		T6
Surrogates								
n-Triacontane (S)	104	%.	50-150	1	08/27/15 08:39	08/28/15 11:19	638-68-6	P4
8260B MSV UST	Analytical Method: EPA 8260B							
Benzene	ND	ug/L	1.0	1		08/27/15 20:06	71-43-2	
Ethylbenzene	ND	ug/L	1.0	1		08/27/15 20:06	100-41-4	
Toluene	ND	ug/L	1.0	1		08/27/15 20:06	108-88-3	
Xylene (Total)	ND	ug/L	3.0	1		08/27/15 20:06	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	102	%.	75-125	1		08/27/15 20:06	17060-07-0	
Toluene-d8 (S)	97	%.	75-125	1		08/27/15 20:06	2037-26-5	
4-Bromofluorobenzene (S)	102	%.	75-125	1		08/27/15 20:06	460-00-4	

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

Pace Analytical®
www.pacelabs.com

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-3 (4') **Lab ID: 10319784004** Collected: 08/20/15 12:40 Received: 08/25/15 09:10 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	**4560**	mg/kg	523	50	08/27/15 12:32	08/28/15 13:00		T6
Surrogates								
n-Triacontane (S)	0	%.	50-150	50	08/27/15 12:32	08/28/15 13:00	638-68-6	S4
Dry Weight	Analytical Method: ASTM D2974							
Percent Moisture	**11.2**	%	0.10	1		08/31/15 19:17		
8270D MSSV PAH by SIM	Analytical Method: EPA 8270D by SIM Preparation Method: EPA 3550							
Acenaphthene	**0.14**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	83-32-9	
Acenaphthylene	**0.057**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	208-96-8	
Anthracene	**0.53**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	120-12-7	
Benzo(a)anthracene	**0.41**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	56-55-3	
Benzo(a)pyrene	**0.23**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	50-32-8	
Benzo(b)fluoranthene	**0.12**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	205-99-2	
Benzo(g,h,i)perylene	**0.097**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	191-24-2	
Benzo(k)fluoranthene	ND	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	207-08-9	
Chrysene	**0.65**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	218-01-9	
Dibenz(a,h)anthracene	ND	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	53-70-3	
Fluoranthene	**0.27**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	206-44-0	
Fluorene	**0.41**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	86-73-7	
Indeno(1,2,3-cd)pyrene	ND	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	193-39-5	
Naphthalene	ND	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	91-20-3	
Phenanthrene	**0.68**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	85-01-8	
Pyrene	**1.7**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54	129-00-0	
Total BaP Eq. MN 2006sh. ND=0	**0.29**	mg/kg	0.056	5	08/28/15 09:36	08/30/15 19:54		
Surrogates								
2-Fluorobiphenyl (S)	84	%.	55-125	5	08/28/15 09:36	08/30/15 19:54	321-60-8	D4
p-Terphenyl-d14 (S)	88	%.	30-150	5	08/28/15 09:36	08/30/15 19:54	1718-51-0	
8260B MSV UST	Analytical Method: EPA 8260B Preparation Method: EPA 5035/5030B							
Benzene	ND	mg/kg	0.023	1	08/30/15 11:56	08/31/15 00:30	71-43-2	
Ethylbenzene	ND	mg/kg	0.056	1	08/30/15 11:56	08/31/15 00:30	100-41-4	
Toluene	**0.12**	mg/kg	0.056	1	08/30/15 11:56	08/31/15 00:30	108-88-3	
Xylene (Total)	ND	mg/kg	0.17	1	08/30/15 11:56	08/31/15 00:30	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	109	%.	55-150	1	08/30/15 11:56	08/31/15 00:30	17060-07-0	1M
Toluene-d8 (S)	99	%.	61-125	1	08/30/15 11:56	08/31/15 00:30	2037-26-5	
4-Bromofluorobenzene (S)	105	%.	54-131	1	08/30/15 11:56	08/31/15 00:30	460-00-4	

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

Pace Analytical®
www.pacelabs.com

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-5 (3') **Lab ID: 10319784005** Collected: 08/20/15 03:05 Received: 08/25/15 09:10 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	**24100**	mg/kg	1220	10	08/27/15 12:32	08/28/15 09:54		T6
Surrogates								
n-Triacontane (S)	0	%.	50-150	10	08/27/15 12:32	08/28/15 09:54	638-68-6	S4
Dry Weight	Analytical Method: ASTM D2974							
Percent Moisture	**24.7**	%	0.10	1		08/31/15 19:18		
8270D MSSV PAH by SIM	Analytical Method: EPA 8270D by SIM Preparation Method: EPA 3550							
Acenaphthene	**9.4**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	83-32-9	
Acenaphthylene	ND	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	208-96-8	
Anthracene	ND	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	120-12-7	
Benzo(a)anthracene	**6.0**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	56-55-3	
Benzo(a)pyrene	**5.4**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	50-32-8	
Benzo(b)fluoranthene	**2.9**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	205-99-2	
Benzo(g,h,i)perylene	ND	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	191-24-2	
Benzo(k)fluoranthene	ND	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	207-08-9	
Chrysene	**12.7**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	218-01-9	
Dibenz(a,h)anthracene	ND	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	53-70-3	
Fluoranthene	**3.3**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	206-44-0	
Fluorene	**11.2**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	86-73-7	
Indeno(1,2,3-cd)pyrene	ND	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	193-39-5	
Naphthalene	ND	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	91-20-3	
Phenanthrene	**16.5**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	85-01-8	
Pyrene	**12.6**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15	129-00-0	
Total BaP Eq. MN 2006sh. ND=0	**6.4**	mg/kg	2.6	20	08/28/15 09:36	08/30/15 20:15		
Surrogates								
2-Fluorobiphenyl (S)	0	%.	55-125	20	08/28/15 09:36	08/30/15 20:15	321-60-8	D4,P3, S4
p-Terphenyl-d14 (S)	0	%.	30-150	20	08/28/15 09:36	08/30/15 20:15	1718-51-0	S4
8260B MSV UST	Analytical Method: EPA 8260B Preparation Method: EPA 5035/5030B							
Benzene	ND	mg/kg	0.027	1	08/30/15 11:56	08/31/15 00:48	71-43-2	
Ethylbenzene	ND	mg/kg	0.066	1	08/30/15 11:56	08/31/15 00:48	100-41-4	
Toluene	**0.12**	mg/kg	0.066	1	08/30/15 11:56	08/31/15 00:48	108-88-3	
Xylene (Total)	ND	mg/kg	0.20	1	08/30/15 11:56	08/31/15 00:48	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	108	%.	55-150	1	08/30/15 11:56	08/31/15 00:48	17060-07-0	1M
Toluene-d8 (S)	101	%.	61-125	1	08/30/15 11:56	08/31/15 00:48	2037-26-5	
4-Bromofluorobenzene (S)	102	%.	54-131	1	08/30/15 11:56	08/31/15 00:48	460-00-4	

REPORT OF LABORATORY ANALYSIS

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

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-2 **Lab ID: 10319784006** Collected: 08/20/15 11:30 Received: 08/25/15 09:10 Matrix: Water

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	ND	ug/L	116	1	08/27/15 08:39	08/28/15 12:27		
Surrogates								
n-Triacontane (S)	81	%.	50-150	1	08/27/15 08:39	08/28/15 12:27	638-68-6	P4
8260B MSV UST	Analytical Method: EPA 8260B							
Benzene	ND	ug/L	1.0	1		08/27/15 20:22	71-43-2	
Ethylbenzene	ND	ug/L	1.0	1		08/27/15 20:22	100-41-4	
Toluene	ND	ug/L	1.0	1		08/27/15 20:22	108-88-3	
Xylene (Total)	ND	ug/L	3.0	1		08/27/15 20:22	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	102	%.	75-125	1		08/27/15 20:22	17060-07-0	
Toluene-d8 (S)	97	%.	75-125	1		08/27/15 20:22	2037-26-5	
4-Bromofluorobenzene (S)	101	%.	75-125	1		08/27/15 20:22	460-00-4	

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

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-2 (3.5') **Lab ID: 10319784007** Collected: 08/20/15 11:15 Received: 08/25/15 09:10 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	ND	mg/kg	11.3	1	08/27/15 12:32	08/28/15 10:48		
Surrogates								
n-Triacontane (S)	84	%.	50-150	1	08/27/15 12:32	08/28/15 10:48	638-68-6	
Dry Weight	Analytical Method: ASTM D2974							
Percent Moisture	**16.6**	%	0.10	1		08/31/15 19:20		
8270D MSSV PAH by SIM	Analytical Method: EPA 8270D by SIM Preparation Method: EPA 3550							
Acenaphthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	83-32-9	
Acenaphthylene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	208-96-8	
Anthracene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	120-12-7	
Benzo(a)anthracene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	56-55-3	
Benzo(a)pyrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	50-32-8	
Benzo(b)fluoranthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	205-99-2	
Benzo(g,h,i)perylene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	191-24-2	
Benzo(k)fluoranthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	207-08-9	
Chrysene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	218-01-9	
Dibenz(a,h)anthracene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	53-70-3	
Fluoranthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	206-44-0	
Fluorene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	86-73-7	
Indeno(1,2,3-cd)pyrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	193-39-5	
Naphthalene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	91-20-3	
Phenanthrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	85-01-8	
Pyrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58	129-00-0	
Total BaP Eq. MN 2006sh. ND=0	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 15:58		
Surrogates								
2-Fluorobiphenyl (S)	66	%.	55-125	1	08/28/15 09:36	08/30/15 15:58	321-60-8	
p-Terphenyl-d14 (S)	73	%.	30-150	1	08/28/15 09:36	08/30/15 15:58	1718-51-0	
8260B MSV UST	Analytical Method: EPA 8260B Preparation Method: EPA 5035/5030B							
Benzene	ND	mg/kg	0.024	1	08/30/15 11:56	08/31/15 18:55	71-43-2	
Ethylbenzene	ND	mg/kg	0.060	1	08/30/15 11:56	08/31/15 18:55	100-41-4	
Toluene	**0.14**	mg/kg	0.060	1	08/30/15 11:56	08/31/15 18:55	108-88-3	
Xylene (Total)	ND	mg/kg	0.18	1	08/30/15 11:56	08/31/15 18:55	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	94	%.	55-150	1	08/30/15 11:56	08/31/15 18:55	17060-07-0	1M
Toluene-d8 (S)	101	%.	61-125	1	08/30/15 11:56	08/31/15 18:55	2037-26-5	
4-Bromofluorobenzene (S)	106	%.	54-131	1	08/30/15 11:56	08/31/15 18:55	460-00-4	

REPORT OF LABORATORY ANALYSIS

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

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-1 **Lab ID: 10319784008** Collected: 08/20/15 10:11 Received: 08/25/15 09:10 Matrix: Water

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	**262**	ug/L	129	1	08/27/15 08:39	08/28/15 12:34		
Surrogates								
n-Triacontane (S)	84	%.	50-150	1	08/27/15 08:39	08/28/15 12:34	638-68-6	P4
8260B MSV UST	Analytical Method: EPA 8260B							
Benzene	ND	ug/L	1.0	1		08/27/15 20:38	71-43-2	
Ethylbenzene	ND	ug/L	1.0	1		08/27/15 20:38	100-41-4	
Toluene	ND	ug/L	1.0	1		08/27/15 20:38	108-88-3	
Xylene (Total)	ND	ug/L	3.0	1		08/27/15 20:38	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	102	%.	75-125	1		08/27/15 20:38	17060-07-0	
Toluene-d8 (S)	98	%.	75-125	1		08/27/15 20:38	2037-26-5	
4-Bromofluorobenzene (S)	101	%.	75-125	1		08/27/15 20:38	460-00-4	

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

Pace Analytical®
www.pacelabs.com

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: SB-1 (4') **Lab ID: 10319784009** Collected: 08/20/15 10:00 Received: 08/25/15 09:10 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	**23.0**	mg/kg	9.7	1	08/27/15 12:32	08/28/15 10:32		T6
Surrogates								
n-Triacontane (S)	78	%.	50-150	1	08/27/15 12:32	08/28/15 10:32	638-68-6	
Dry Weight	Analytical Method: ASTM D2974							
Percent Moisture	**20.1**	%	0.10	1		08/31/15 19:21		
8270D MSSV PAH by SIM	Analytical Method: EPA 8270D by SIM Preparation Method: EPA 3550							
Acenaphthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	83-32-9	
Acenaphthylene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	208-96-8	
Anthracene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	120-12-7	
Benzo(a)anthracene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	56-55-3	
Benzo(a)pyrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	50-32-8	
Benzo(b)fluoranthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	205-99-2	
Benzo(g,h,i)perylene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	191-24-2	
Benzo(k)fluoranthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	207-08-9	
Chrysene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	218-01-9	
Dibenz(a,h)anthracene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	53-70-3	
Fluoranthene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	206-44-0	
Fluorene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	86-73-7	
Indeno(1,2,3-cd)pyrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	193-39-5	
Naphthalene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	91-20-3	
Phenanthrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	85-01-8	
Pyrene	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19	129-00-0	
Total BaP Eq. MN 2006sh. ND=0	ND	mg/kg	0.012	1	08/28/15 09:36	08/30/15 16:19		
Surrogates								
2-Fluorobiphenyl (S)	66	%.	55-125	1	08/28/15 09:36	08/30/15 16:19	321-60-8	
p-Terphenyl-d14 (S)	67	%.	30-150	1	08/28/15 09:36	08/30/15 16:19	1718-51-0	
8260B MSV UST	Analytical Method: EPA 8260B Preparation Method: EPA 5035/5030B							
Benzene	ND	mg/kg	0.025	1	08/30/15 11:56	08/30/15 23:53	71-43-2	
Ethylbenzene	ND	mg/kg	0.063	1	08/30/15 11:56	08/30/15 23:53	100-41-4	
Toluene	**0.16**	mg/kg	0.063	1	08/30/15 11:56	08/30/15 23:53	108-88-3	
Xylene (Total)	ND	mg/kg	0.19	1	08/30/15 11:56	08/30/15 23:53	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	107	%.	55-150	1	08/30/15 11:56	08/30/15 23:53	17060-07-0	1M
Toluene-d8 (S)	101	%.	61-125	1	08/30/15 11:56	08/30/15 23:53	2037-26-5	
4-Bromofluorobenzene (S)	105	%.	54-131	1	08/30/15 11:56	08/30/15 23:53	460-00-4	

REPORT OF LABORATORY ANALYSIS

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

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

ANALYTICAL RESULTS

Project: Graymont Superior, WI
Pace Project No.: 10319784

Sample: STOCK PILE **Lab ID: 10319784010** Collected: 08/20/15 03:30 Received: 08/25/15 09:10 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
WIDRO GCS	Analytical Method: WI MOD DRO Preparation Method: WI MOD DRO							
WDRO C10-C28	**10800**	mg/kg	915	10	08/27/15 12:32	08/28/15 10:02		T6
Surrogates								
n-Triacontane (S)	0	%.	50-150	10	08/27/15 12:32	08/28/15 10:02	638-68-6	S4
6010C MET ICP	Analytical Method: EPA 6010C Preparation Method: EPA 3050							
Lead	**9.5**	mg/kg	0.50	1	08/30/15 16:52	08/31/15 01:12	7439-92-1	
Dry Weight	Analytical Method: ASTM D2974							
Percent Moisture	**6.7**	%	0.10	1		08/31/15 19:22		
8260B MSV UST	Analytical Method: EPA 8260B Preparation Method: EPA 5035/5030B							
Benzene	ND	mg/kg	0.021	1	08/30/15 11:56	08/31/15 00:12	71-43-2	
Ethylbenzene	ND	mg/kg	0.054	1	08/30/15 11:56	08/31/15 00:12	100-41-4	
Toluene	**0.13**	mg/kg	0.054	1	08/30/15 11:56	08/31/15 00:12	108-88-3	
Xylene (Total)	ND	mg/kg	0.16	1	08/30/15 11:56	08/31/15 00:12	1330-20-7	
Surrogates								
1,2-Dichloroethane-d4 (S)	112	%.	55-150	1	08/30/15 11:56	08/31/15 00:12	17060-07-0	1M
Toluene-d8 (S)	101	%.	61-125	1	08/30/15 11:56	08/31/15 00:12	2037-26-5	
4-Bromofluorobenzene (S)	100	%.	54-131	1	08/30/15 11:56	08/31/15 00:12	460-00-4	

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior, WI
Pace Project No.: 10319784

QC Batch: MPRP/57277
QC Batch Method: EPA 3050
Analysis Method: EPA 6010C
Analysis Description: 6010C Solids
Associated Lab Samples: 10319784010

METHOD BLANK: 2061730 Matrix: Solid
Associated Lab Samples: 10319784010

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Lead	mg/kg	ND	0.46	08/31/15 00:25	

LABORATORY CONTROL SAMPLE: 2061731

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Lead	mg/kg	49	46.9	96	80-120	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 2061732 2061733

Parameter	Units	10319553001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Lead	mg/kg	5.2	54.9	57.5	46.7	49.6	75	77	75-125	6	20	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior, WI
Pace Project No.: 10319784

QC Batch: MPRP/57391 Analysis Method: ASTM D2974
QC Batch Method: ASTM D2974 Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10319784002, 10319784004, 10319784005, 10319784007, 10319784009, 10319784010

SAMPLE DUPLICATE: 2065435

Parameter	Units	10319595005 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	3.4	3.4	2	30	

SAMPLE DUPLICATE: 2065436

Parameter	Units	10320162012 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	21.3	18.2	16	30	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior, WI
Pace Project No.: 10319784

QC Batch: MSV/32969 Analysis Method: EPA 8260B
QC Batch Method: EPA 5035/5030B Analysis Description: 8260B MSV UST
Associated Lab Samples: 10319784002, 10319784004, 10319784005, 10319784007, 10319784009, 10319784010

METHOD BLANK: 2063502 Matrix: Solid
Associated Lab Samples: 10319784002, 10319784004, 10319784005, 10319784007, 10319784009, 10319784010

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Benzene	mg/kg	ND	0.020	08/30/15 16:26	
Ethylbenzene	mg/kg	ND	0.050	08/30/15 16:26	
Toluene	mg/kg	ND	0.050	08/30/15 16:26	
Xylene (Total)	mg/kg	ND	0.15	08/30/15 16:26	
1,2-Dichloroethane-d4 (S)	%.	119	55-150	08/30/15 16:26	
4-Bromofluorobenzene (S)	%.	103	54-131	08/30/15 16:26	
Toluene-d8 (S)	%.	104	61-125	08/30/15 16:26	

LABORATORY CONTROL SAMPLE & LCSD: 2063503 2064419

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
Benzene	mg/kg	1	1.0	1.0	103	103	69-125	0	20	
Ethylbenzene	mg/kg	1	1.1	1.1	107	109	72-125	2	20	
Toluene	mg/kg	1	1.1	1.1	108	108	72-125	0	20	
Xylene (Total)	mg/kg	3	3.2	3.2	108	107	74-125	1	20	
1,2-Dichloroethane-d4 (S)	%.				97	94	55-150			
4-Bromofluorobenzene (S)	%.				99	109	54-131			
Toluene-d8 (S)	%.				104	102	61-125			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior, WI
Pace Project No.: 10319784

QC Batch: MSV/32953 Analysis Method: EPA 8260B
QC Batch Method: EPA 8260B Analysis Description: 8260B MSV UST-WATER
Associated Lab Samples: 10319784001, 10319784003, 10319784006, 10319784008

METHOD BLANK: 2062223 Matrix: Water
Associated Lab Samples: 10319784001, 10319784003, 10319784006, 10319784008

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Benzene	ug/L	ND	1.0	08/27/15 14:21	
Ethylbenzene	ug/L	ND	1.0	08/27/15 14:21	
Toluene	ug/L	ND	1.0	08/27/15 14:21	
Xylene (Total)	ug/L	ND	3.0	08/27/15 14:21	
1,2-Dichloroethane-d4 (S)	%.	100	75-125	08/27/15 14:21	
4-Bromofluorobenzene (S)	%.	101	75-125	08/27/15 14:21	
Toluene-d8 (S)	%.	98	75-125	08/27/15 14:21	

LABORATORY CONTROL SAMPLE: 2062224

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Benzene	ug/L	20	17.2	86	71-125	
Ethylbenzene	ug/L	20	17.7	89	75-125	
Toluene	ug/L	20	17.6	88	74-125	
Xylene (Total)	ug/L	60	54.1	90	75-125	
1,2-Dichloroethane-d4 (S)	%.			102	75-125	
4-Bromofluorobenzene (S)	%.			100	75-125	
Toluene-d8 (S)	%.			99	75-125	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 2062474 2062475

Parameter	Units	10319581009 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Benzene	ug/L	469	1000	1000	1620	1590	115	112	53-139	2	30	
Ethylbenzene	ug/L	2580	1000	1000	3720	3620	114	104	55-139	3	30	
Toluene	ug/L	2450	1000	1000	3610	3500	116	106	52-148	3	30	
Xylene (Total)	ug/L	8740	3000	3000	12200	12000	114	108	54-144	2	30	
1,2-Dichloroethane-d4 (S)	%.						104	104	75-125			
4-Bromofluorobenzene (S)	%.						101	101	75-125			
Toluene-d8 (S)	%.						99	99	75-125			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior, WI
Pace Project No.: 10319784

QC Batch: OEXT/30583 Analysis Method: EPA 8270D by SIM
QC Batch Method: EPA 3550 Analysis Description: 8270D Solid PAH by SIM MSSV
Associated Lab Samples: 10319784002, 10319784004, 10319784005, 10319784007, 10319784009

METHOD BLANK: 2063445 Matrix: Solid
Associated Lab Samples: 10319784002, 10319784004, 10319784005, 10319784007, 10319784009

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Acenaphthene	mg/kg	ND	0.010	08/30/15 11:19	
Acenaphthylene	mg/kg	ND	0.010	08/30/15 11:19	
Anthracene	mg/kg	ND	0.010	08/30/15 11:19	
Benzo(a)anthracene	mg/kg	ND	0.010	08/30/15 11:19	
Benzo(a)pyrene	mg/kg	ND	0.010	08/30/15 11:19	
Benzo(b)fluoranthene	mg/kg	ND	0.010	08/30/15 11:19	
Benzo(g,h,i)perylene	mg/kg	ND	0.010	08/30/15 11:19	
Benzo(k)fluoranthene	mg/kg	ND	0.010	08/30/15 11:19	
Chrysene	mg/kg	ND	0.010	08/30/15 11:19	
Dibenz(a,h)anthracene	mg/kg	ND	0.010	08/30/15 11:19	
Fluoranthene	mg/kg	ND	0.010	08/30/15 11:19	
Fluorene	mg/kg	ND	0.010	08/30/15 11:19	
Indeno(1,2,3-cd)pyrene	mg/kg	ND	0.010	08/30/15 11:19	
Naphthalene	mg/kg	ND	0.010	08/30/15 11:19	
Phenanthrene	mg/kg	ND	0.010	08/30/15 11:19	
Pyrene	mg/kg	ND	0.010	08/30/15 11:19	
2-Fluorobiphenyl (S)	%.	80	55-125	08/30/15 11:19	
p-Terphenyl-d14 (S)	%.	86	30-150	08/30/15 11:19	

LABORATORY CONTROL SAMPLE: 2063446

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Acenaphthene	mg/kg	.033	0.025	75	53-125	
Acenaphthylene	mg/kg	.033	0.027	80	53-125	
Anthracene	mg/kg	.033	0.030	90	61-125	
Benzo(a)anthracene	mg/kg	.033	0.031	93	62-125	
Benzo(a)pyrene	mg/kg	.033	0.032	95	64-125	
Benzo(b)fluoranthene	mg/kg	.033	0.033	99	66-125	
Benzo(g,h,i)perylene	mg/kg	.033	0.031	92	59-125	
Benzo(k)fluoranthene	mg/kg	.033	0.031	93	61-125	
Chrysene	mg/kg	.033	0.029	88	63-125	
Dibenz(a,h)anthracene	mg/kg	.033	0.029	87	59-125	
Fluoranthene	mg/kg	.033	0.031	93	64-125	
Fluorene	mg/kg	.033	0.028	83	57-125	
Indeno(1,2,3-cd)pyrene	mg/kg	.033	0.029	87	58-125	
Naphthalene	mg/kg	.033	0.024	73	52-125	
Phenanthrene	mg/kg	.033	0.026	77	60-125	
Pyrene	mg/kg	.033	0.032	95	63-125	
2-Fluorobiphenyl (S)	%.			79	55-125	
p-Terphenyl-d14 (S)	%.			90	30-150	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior, WI
Pace Project No.: 10319784

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 2063447 2063448

Parameter	Units	10319794002 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Acenaphthene	mg/kg	ND	.038	.038	0.039	0.34	89	889	39-125	159	30	M1,R1
Acenaphthylene	mg/kg	ND	.038	.038	0.034	0.038	89	100	30-150	12	30	
Anthracene	mg/kg	0.016	.038	.038	0.061	0.59	118	1520	30-150	163	30	E,M1, R1
Benzo(a)anthracene	mg/kg	0.056	.038	.038	0.093	1.1	95	2760	30-150	169	30	E,M1, R1
Benzo(a)pyrene	mg/kg	0.059	.038	.038	0.094	0.89	91	2200	30-150	162	30	E,M1, R1
Benzo(b)fluoranthene	mg/kg	0.077	.038	.038	0.097	1.1	52	2680	30-150	167	30	E,M1, R1
Benzo(g,h,i)perylene	mg/kg	0.044	.038	.038	0.074	0.48	78	1150	30-150	146	30	E,M1, R1
Benzo(k)fluoranthene	mg/kg	0.027	.038	.038	0.070	0.56	113	1400	30-150	156	30	E,M1, R1
Chrysene	mg/kg	0.064	.038	.038	0.10	1.1	97	2750	30-150	167	30	E,M1, R1
Dibenz(a,h)anthracene	mg/kg	0.011	.038	.038	0.042	0.22	82	548	30-150	135	30	M1,R1
Fluoranthene	mg/kg	0.10	.038	.038	0.14	1.9	97	4720	30-150	173	30	E,M1, R1
Fluorene	mg/kg	ND	.038	.038	0.043	0.34	96	875	30-146	155	30	M1,R1
Indeno(1,2,3-cd)pyrene	mg/kg	0.033	.038	.038	0.061	0.46	74	1120	30-150	153	30	E,M1, R1
Naphthalene	mg/kg	ND	.038	.038	0.030	0.11	80	293	30-131	114	30	M1,R1
Phenanthrene	mg/kg	0.053	.038	.038	0.10	1.6	134	4100	30-150	176	30	E,M1, R1
Pyrene	mg/kg	0.096	.038	.038	0.14	1.5	110	3770	30-150	167	30	E,M1, R1
2-Fluorobiphenyl (S)	%.						76	86	55-125			
p-Terphenyl-d14 (S)	%.						82	86	30-150			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

Pace Analytical®
www.pacelabs.com

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

QUALITY CONTROL DATA

Project: Graymont Superior, WI
Pace Project No.: 10319784

QC Batch: OEXT/30569 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10319784002, 10319784004, 10319784005, 10319784007, 10319784009, 10319784010

METHOD BLANK: 2062159 Matrix: Solid
Associated Lab Samples: 10319784002, 10319784004, 10319784005, 10319784007, 10319784009, 10319784010

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
WDRO C10-C28	mg/kg	ND	10.0	08/28/15 09:30	
n-Triacontane (S)	%.	85	50-150	08/28/15 09:30	

LABORATORY CONTROL SAMPLE & LCSD: 2062160 2062161

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
WDRO C10-C28	mg/kg	80	60.2	59.2	75	74	70-120	2	20	
n-Triacontane (S)	%.				97	92	50-150			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALITY CONTROL DATA

Project: Graymont Superior, WI
Pace Project No.: 10319784

QC Batch: OEXT/30558 Analysis Method: WI MOD DRO
QC Batch Method: WI MOD DRO Analysis Description: WIDRO GCS
Associated Lab Samples: 10319784001, 10319784003, 10319784006, 10319784008

METHOD BLANK: 2061831 Matrix: Water
Associated Lab Samples: 10319784001, 10319784003, 10319784006, 10319784008

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
WDRO C10-C28	ug/L	ND	100	08/28/15 11:05	
n-Triacontane (S)	%.	101	50-150	08/28/15 11:05	

LABORATORY CONTROL SAMPLE & LCSD: 2061832 2061833

Parameter	Units	Spike Conc.	LCS Result	LCSD Result	LCS % Rec	LCSD % Rec	% Rec Limits	RPD	Max RPD	Qualifiers
WDRO C10-C28	ug/L	2000	1810	1980	91	99	75-115	9	20	
n-Triacontane (S)	%.				90	95	50-150			

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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

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

QUALIFIERS

Project: Graymont Superior, WI
Pace Project No.: 10319784

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

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.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

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	The sample containers provided did not have a tare weight for the methanol and sample container. The lab is assuming 10 mLs of methanol and 10 grams of sample. The results should be considered an estimation.
D4	Sample was diluted due to the presence of high levels of target analytes.
E	Analyte concentration exceeded the calibration range. The reported result is estimated.
M1	Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.
P3	Sample extract could not be concentrated to the routine final volume, resulting in elevated reporting limits.
P4	Sample field preservation does not meet EPA or method recommendations for this analysis.
R1	RPD value was outside control limits.
S4	Surrogate recovery not evaluated against control limits due to sample dilution.
T6	High boiling point hydrocarbons are present in the sample.

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

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

QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: Graymont Superior, WI
Pace Project No.: 10319784

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10319784002	SB-4 (4')	WI MOD DRO	OEXT/30569	WI MOD DRO	GCSV/16561
10319784004	SB-3 (4')	WI MOD DRO	OEXT/30569	WI MOD DRO	GCSV/16561
10319784005	SB-5 (3')	WI MOD DRO	OEXT/30569	WI MOD DRO	GCSV/16561
10319784007	SB-2 (3.5')	WI MOD DRO	OEXT/30569	WI MOD DRO	GCSV/16561
10319784009	SB-1 (4')	WI MOD DRO	OEXT/30569	WI MOD DRO	GCSV/16561
10319784010	STOCK PILE	WI MOD DRO	OEXT/30569	WI MOD DRO	GCSV/16561
10319784001	SB-4	WI MOD DRO	OEXT/30558	WI MOD DRO	GCSV/16563
10319784003	SB-3	WI MOD DRO	OEXT/30558	WI MOD DRO	GCSV/16563
10319784006	SB-2	WI MOD DRO	OEXT/30558	WI MOD DRO	GCSV/16563
10319784008	SB-1	WI MOD DRO	OEXT/30558	WI MOD DRO	GCSV/16563
10319784010	STOCK PILE	EPA 3050	MPRP/57277	EPA 6010C	ICP/25099
10319784002	SB-4 (4')	ASTM D2974	MPRP/57391		
10319784004	SB-3 (4')	ASTM D2974	MPRP/57391		
10319784005	SB-5 (3')	ASTM D2974	MPRP/57391		
10319784007	SB-2 (3.5')	ASTM D2974	MPRP/57391		
10319784009	SB-1 (4')	ASTM D2974	MPRP/57391		
10319784010	STOCK PILE	ASTM D2974	MPRP/57391		
10319784002	SB-4 (4')	EPA 3550	OEXT/30583	EPA 8270D by SIM	MSSV/13001
10319784004	SB-3 (4')	EPA 3550	OEXT/30583	EPA 8270D by SIM	MSSV/13001
10319784005	SB-5 (3')	EPA 3550	OEXT/30583	EPA 8270D by SIM	MSSV/13001
10319784007	SB-2 (3.5')	EPA 3550	OEXT/30583	EPA 8270D by SIM	MSSV/13001
10319784009	SB-1 (4')	EPA 3550	OEXT/30583	EPA 8270D by SIM	MSSV/13001
10319784002	SB-4 (4')	EPA 5035/5030B	MSV/32969	EPA 8260B	MSV/32981
10319784004	SB-3 (4')	EPA 5035/5030B	MSV/32969	EPA 8260B	MSV/32981
10319784005	SB-5 (3')	EPA 5035/5030B	MSV/32969	EPA 8260B	MSV/32981
10319784007	SB-2 (3.5')	EPA 5035/5030B	MSV/32969	EPA 8260B	MSV/32981
10319784009	SB-1 (4')	EPA 5035/5030B	MSV/32969	EPA 8260B	MSV/32981
10319784010	STOCK PILE	EPA 5035/5030B	MSV/32969	EPA 8260B	MSV/32981
10319784001	SB-4	EPA 8260B	MSV/32953		
10319784003	SB-3	EPA 8260B	MSV/32953		
10319784006	SB-2	EPA 8260B	MSV/32953		
10319784008	SB-1	EPA 8260B	MSV/32953		

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

For Braun Intertec Use Only
Laboratory Work Order No.

BRAUN INTERTEC
Braun Intertec Corporation
11001 Hampshire Ave. S
Minneapolis, MN 55438

REQUEST FOR LABORATORY ANALYTICAL SERVICES

Bottle orders and sampling inquires:
labservices@braunintertec.com
Phone: 952-995-2600 Fax: 952-995-2601

IMPORTANT
Date Results Requested: _____
Time _____
Rush Charges Authorized? ____ Yes ____ No
Rush / Quote # _____

010575
10319784

REPORT RESULTS TO
Contact Name Ted Hubbes | Project ID/Name Graymont Superior, WI | P.O. #/Project #
Company Braun Intertec Hibbing
Mailing Address
City, State, Zip
Telephone # | Fax #
E-mail

SEND INVOICE TO
Contact Name | Company
Address
City, State, Zip
Telephone # | Fax #

Special Instructions and/or Specific Regulatory Requirements:
(method, limit of detection, petrofund, reporting units)
email to THubbes@braunintertec.com

ANALYSIS REQUESTED
(Enter an 'X' in the box below to indicate request)

LAB ID#	CLIENT SAMPLE IDENTIFICATION (IDs must be unique)	DATE SAMPLED	TIME SAMPLED	MATRIX/ MEDIA	VOLUME/AREA (specify units)	Number of Containers	Metals Field Filtered Y/N	Site Location (State)	DRO	PAH	PVOC	Lead	FOR LAB USE ONLY
	SB-4	8-20	1:59	H_2O		4			X	X	X		001
	SB-4 (4')	8-20	1:50	Soil		6			↓	↓	↓		002
	SB-3	8-20	12:52	H_2O		4							003
	SB-3 (4')	8-20	12:40	Soil		6							004
	SB-5 (3')	8-20	3:05	Soil		6							005
	SB-2	8-20	11:30	H_2O		4							006
	SB-2 (3.5')	8-20	11:15	Soil		6							007
	SB-1	8-20	10:11	H_2O		4							008
	SB-1 (4')	8-20	10:00	Soil		6				↓			009
	Stock pile	8-20	3:30	Soil		6			↓		↓	X	010

CHAIN OF CUSTODY
Collected by: (Print) Cameron Vollbrecht | Collector's Signature:
Relinquished by: | Date/Time
Relinquished by: | Date/Time
Received by: BK Mn Race | Date/Time 8/25/15 9:10
Received Contents Not Verified: | Date/Time
Received Contents Verified: | Date/Time

Custody Seal Intact ☐ Yes ☐ No ☒ N/A ☐ Hand Delivered by Client
On Ice ☒ Yes ☐ No
Temp Blank ☐ Yes ☒ No
Temp: 2.8 °C

Comments:

Form # CS02.03 F:\Groups\QA-QC\Forms\clientservices\COC-CS02 Effective Date: 10/10/07

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 23Feb2015 Page 1 of 1
	Document No.: **F-MN-L-213-rev.13**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt **Client Name:** Braun Intertec **Project #:** WO# : 10319784

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

Tracking Number: 1Z 22W 847 03 6725 4553 9

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

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

Thermometer Used: ☐B88A9130516413 ☒B88A912167504 ☐B88A0143310098 **Type of Ice:** ☒Wet ☐Blue ☐None ☐Samples on ice, cooling process has begun

Cooler Temp Read (°C): 3.0 **Cooler Temp Corrected (°C):** 2.8 **Biological Tissue Frozen?** ☐Yes ☐No ☒N/A

Temp should be above freezing to 6°C **Correction Factor:** -0.2 **Date and Initials of Person Examining Contents:** BM 8/25/15

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or WA (check maps)? ☐Yes ☒No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐Yes ☒No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☒Yes	☐No	☐N/A	1.
Chain of Custody Filled Out?	☒Yes	☐No	☐N/A	2.
Chain of Custody Relinquished?	☒Yes	☒No	☐N/A	3. BM 8/25/15
Sampler Name and/or Signature on COC?	☒Yes	☐No	☐N/A	4.
Samples Arrived within Hold Time?	☒Yes	☐No	☐N/A	5.
Short Hold Time Analysis (<72 hr)?	☐Yes	☒No	☐N/A	6.
Rush Turn Around Time Requested?	☐Yes	☒No	☐N/A	7.
Sufficient Volume?	☒Yes	☐No	☐N/A	8.
Correct Containers Used?	☒Yes	☐No	☐N/A	9.
-Pace Containers Used?	☒Yes	☐No	☐N/A	
Containers Intact?	☒Yes	☐No	☐N/A	10.
Filtered Volume Received for Dissolved Tests?	☐Yes	☐No	☒N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL/WT	☒Yes	☐No	☐N/A	12. one extra container for sample 010
All containers needing acid/base preservation have been checked?	☐Yes	☐No	☒N/A	13. ☐HNO_3 ☐H_2SO_4 ☐NaOH ☐HCl
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐Yes	☐No	☒N/A	Sample #
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☒Yes	☐No	☐N/A	Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐Yes	☒No	☐N/A	14.
Trip Blank Present?	☐Yes	☐No	☒N/A	15.
Trip Blank Custody Seals Present?	☐Yes	☐No	☒N/A	
Pace Trip Blank Lot # (if purchased):				

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

Person Contacted: Date/Time:

Comments/Resolution:

Project Manager Review: TO for ZW **Date:** Aug 27, 2015

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).

Data File: \\192.168.10.12\chem\10gcs4.i\082815dro.b/08280014.D

Report Date: 08/28/2015

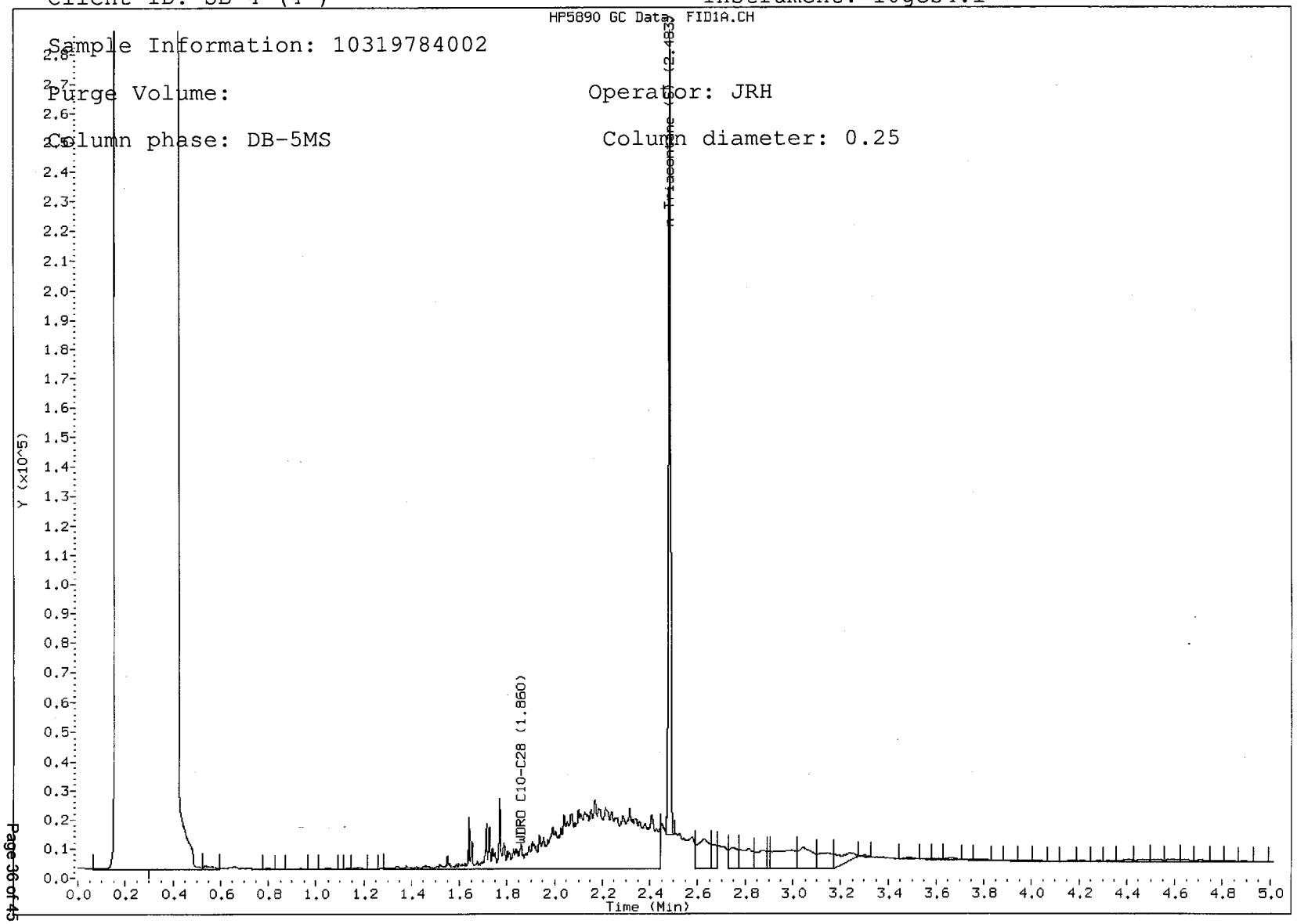
Sample ID: 10319784002

Client ID: SB-4 (4') Instrument: 10gcs4.i

Sample Information: 10319784002

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25



Data File: \\192.168.10.12\chem\10gcs4.i\082815dro.b/08280034.D

Report Date: 08/28/2015

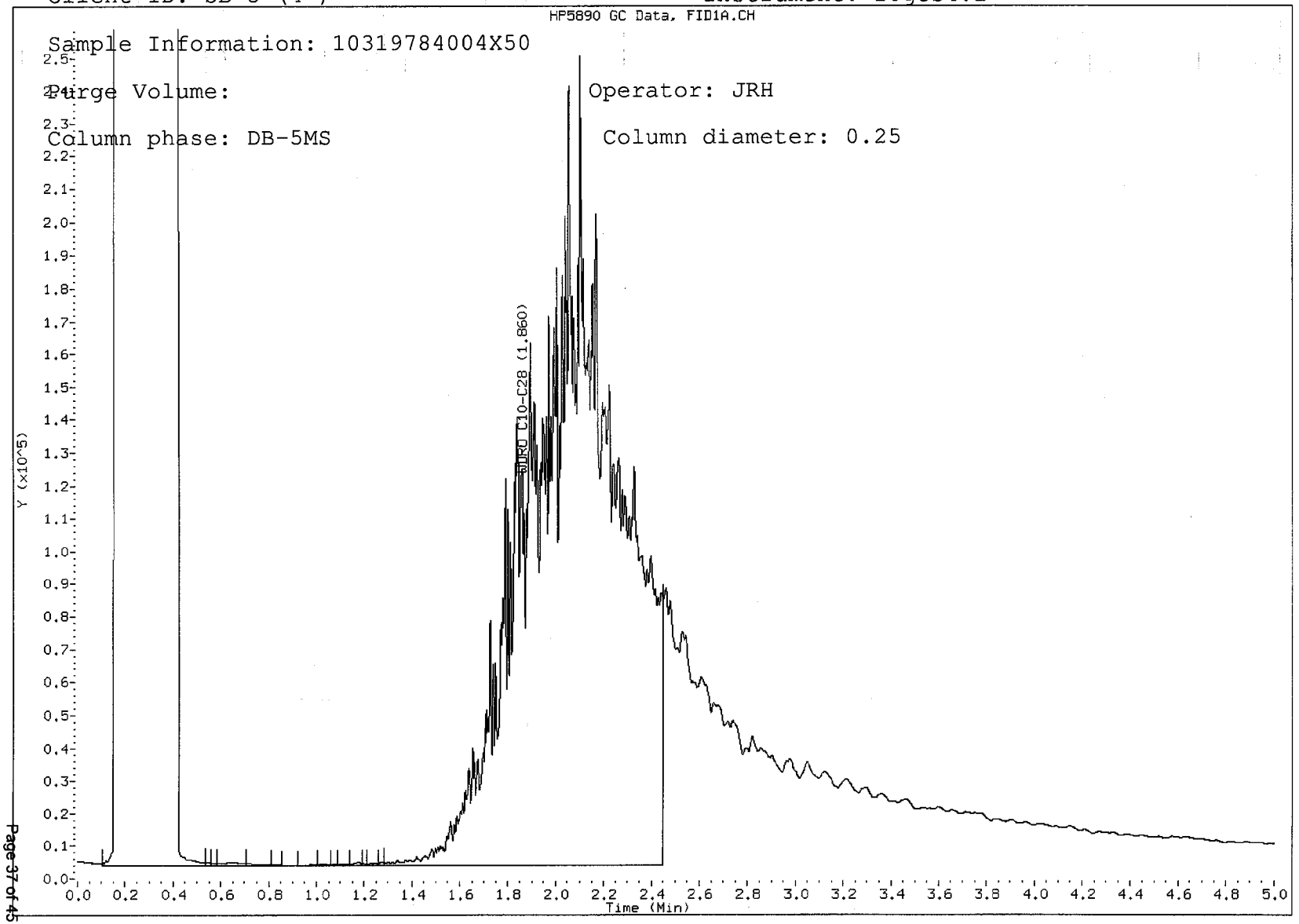
Sample ID: 10319784004

Client ID: SB-3 (4') Instrument: 10gcs4.i

Sample Information: 10319784004X50

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25



Data File: \\192.168.10.12\chem\10gcs4.i\082815dro.b/08280010.D

Report Date: 08/28/2015

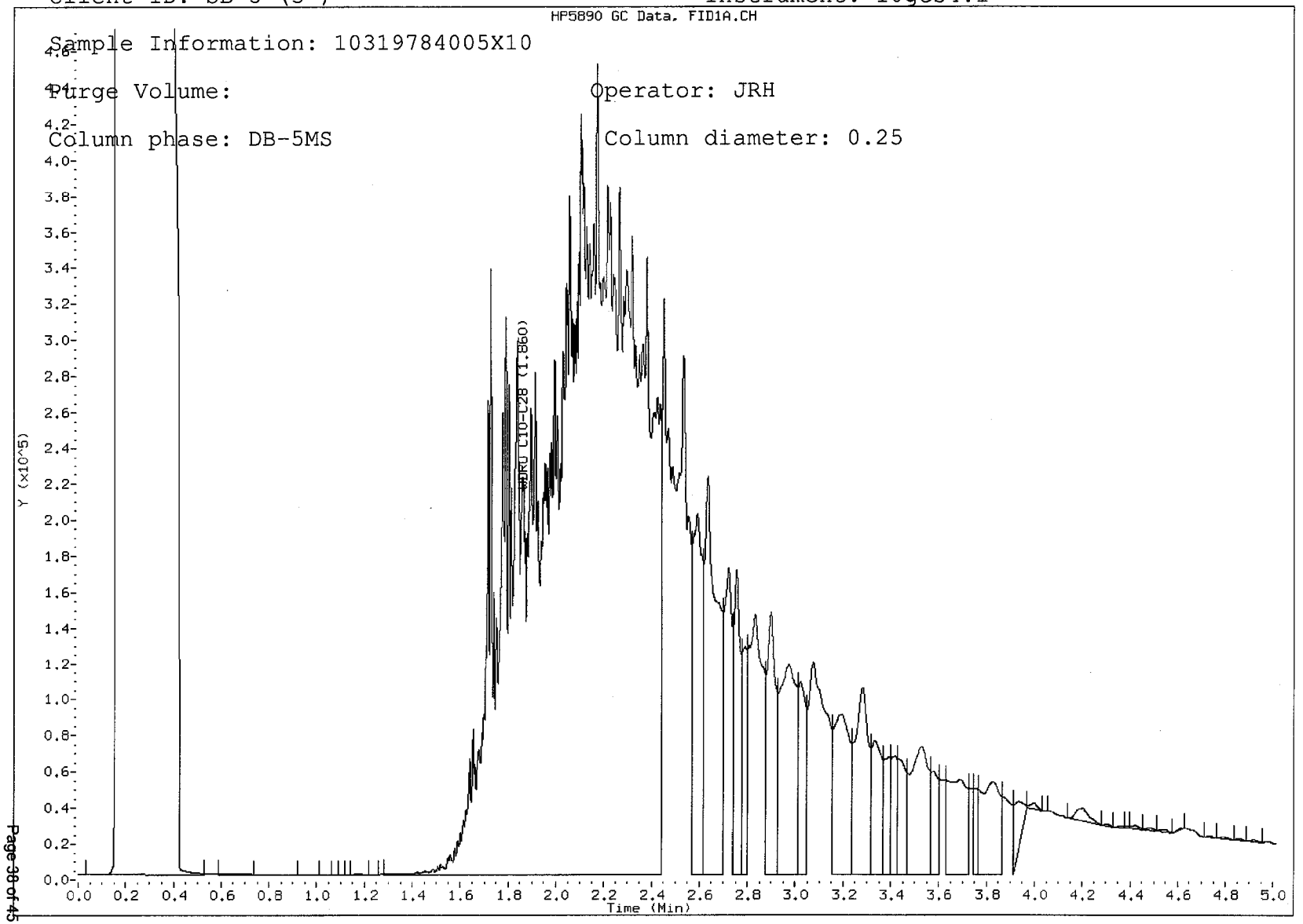
Sample ID: 10319784005

Client ID: SB-5 (3') Instrument: 10gcs4.i

Sample Information: 10319784005X10

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25



Data File: \\192.168.10.12\chem\10gcs4.i\082815dro.b/08280017.D

Report Date: 08/28/2015

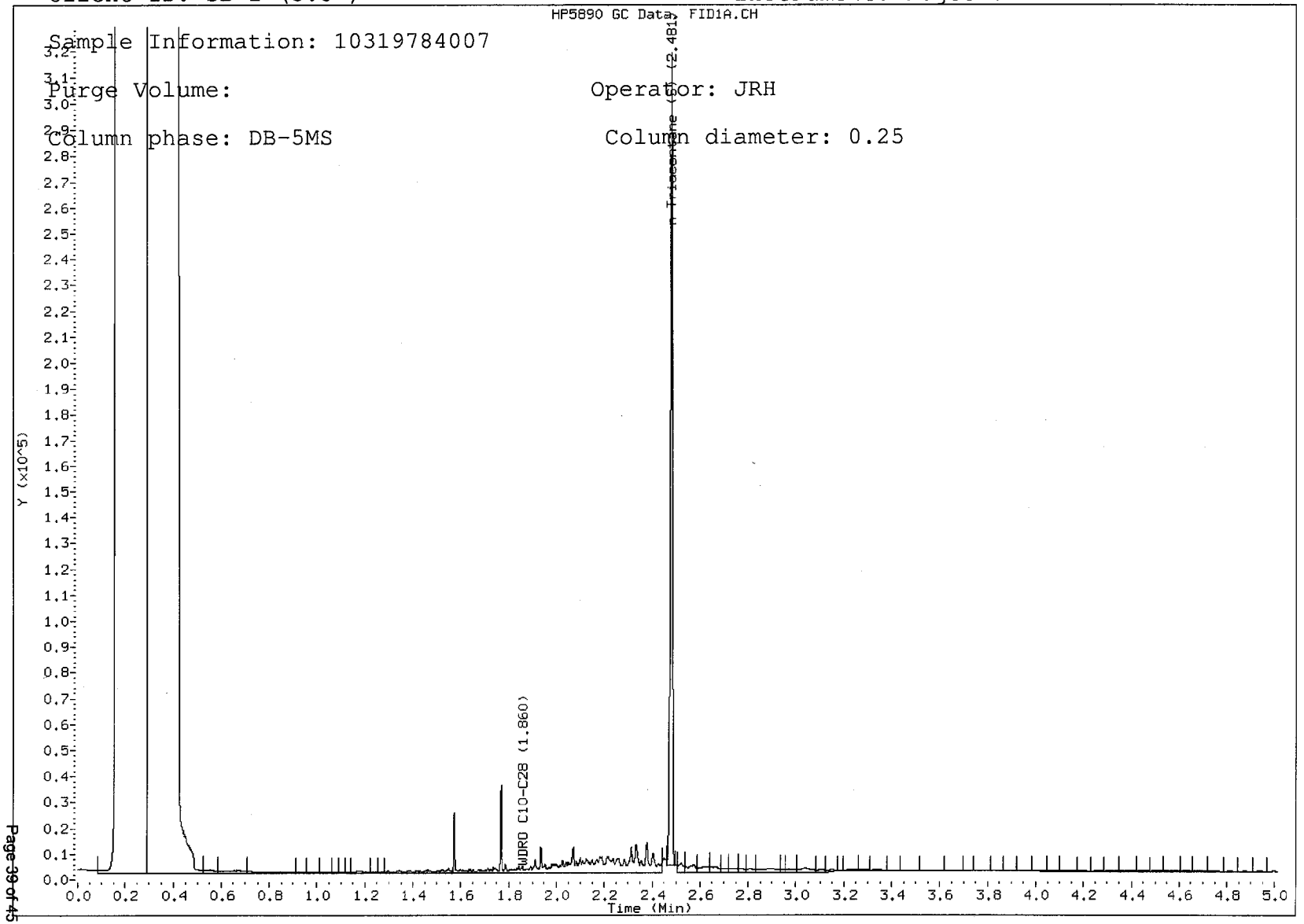
Sample ID: 10319784007

Client ID: SB-2 (3.5') Instrument: 10gcs4.i

Sample Information: 10319784007

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25



Data File: \\192.168.10.12\chem\10gcs4.i\082815dro.b/08280015.D

Report Date: 08/28/2015

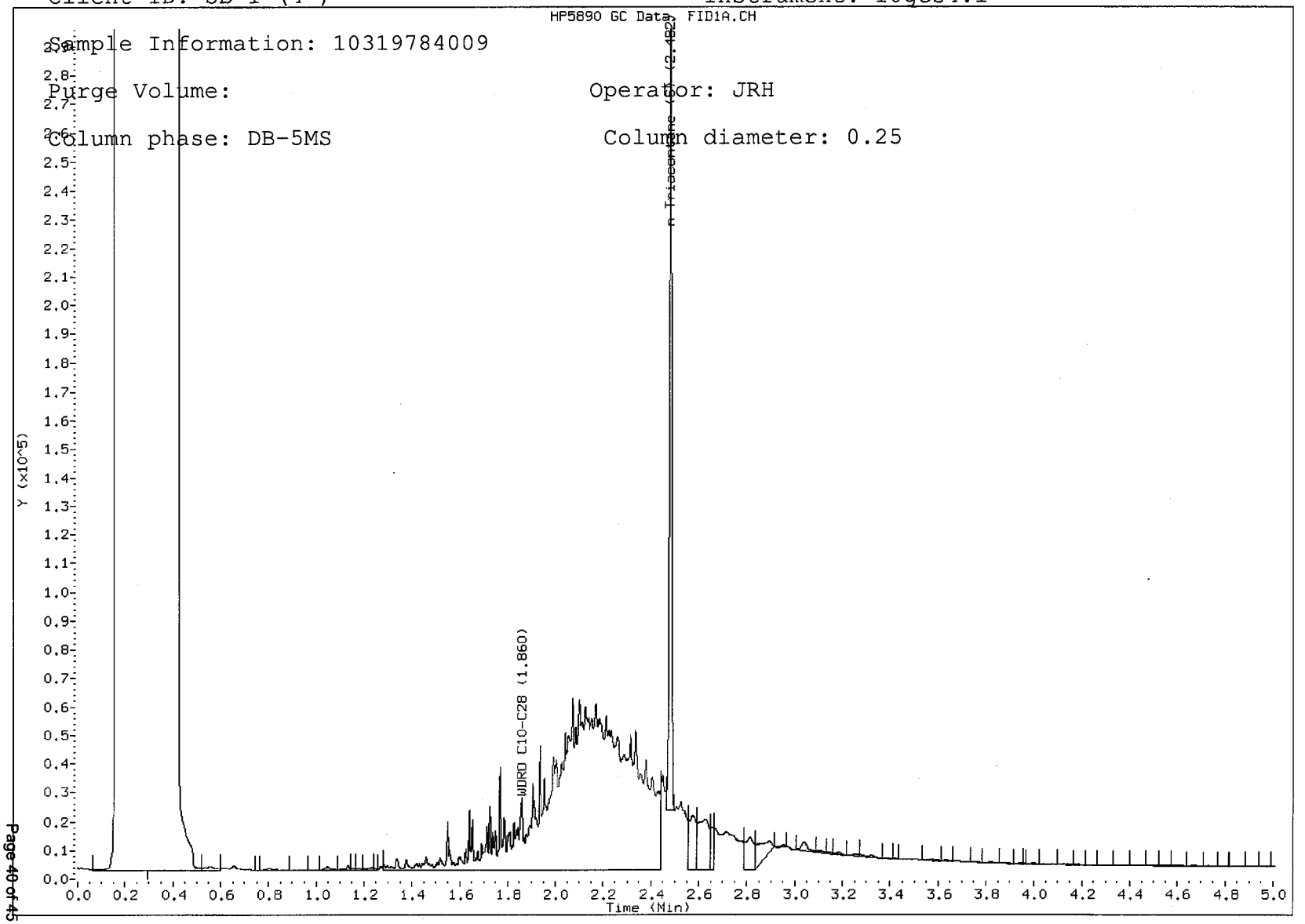
Sample ID: 10319784009

Client ID: SB-1 (4') Instrument: 10gcs4.i

Sample Information: 10319784009

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25



Data File: \\192.168.10.12\chem\10gcs4.i\082815dro.b/08280011.D

Report Date: 08/28/2015

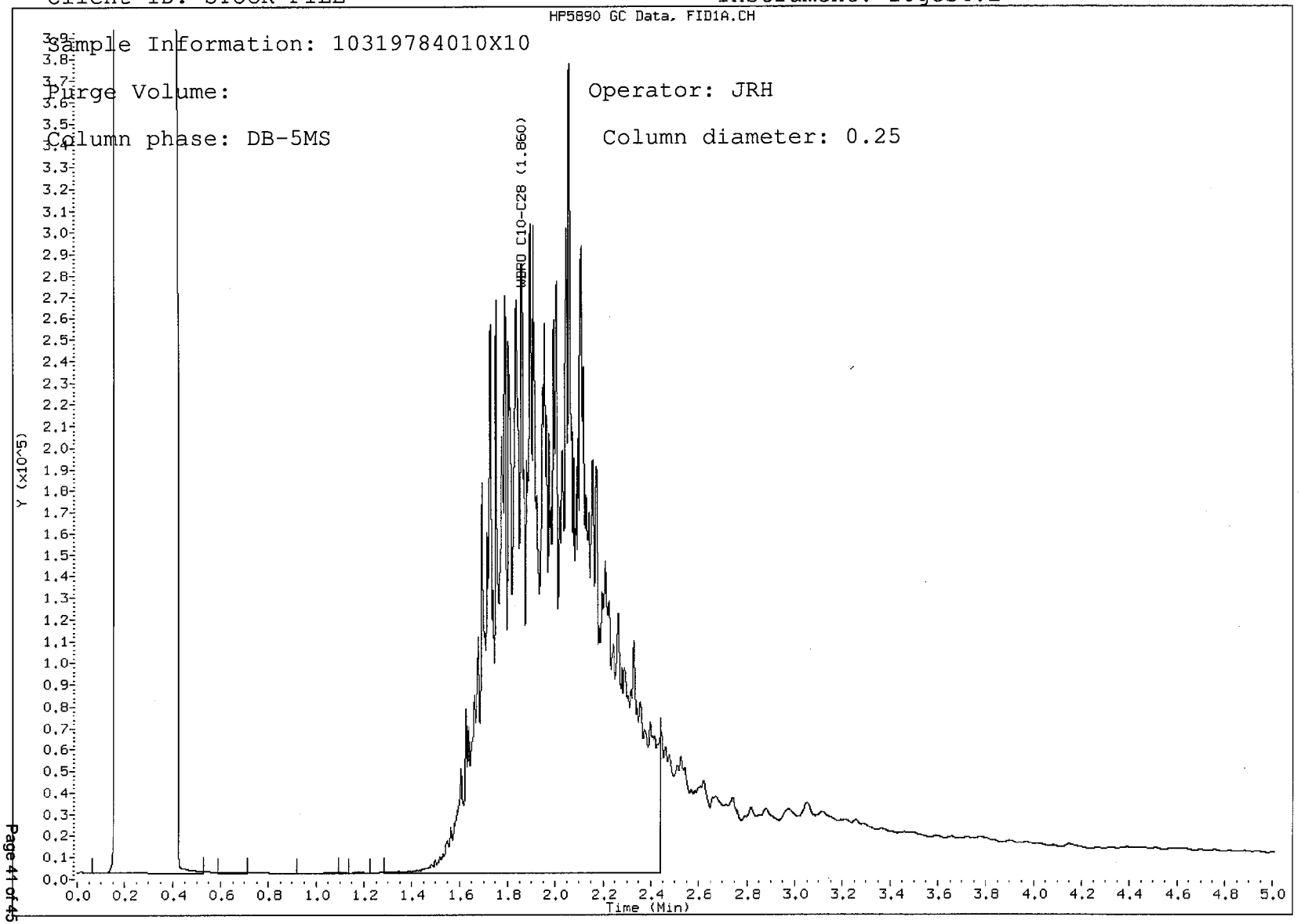
Sample ID: 10319784010

Client ID: STOCK PILE Instrument: 10gcs4.i

Sample Information: 10319784010X10

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25



Data File: \\192.168.10.12\chem\10gcs9.i\082815dro.b/082815000039.D

Report Date: 08/28/2015

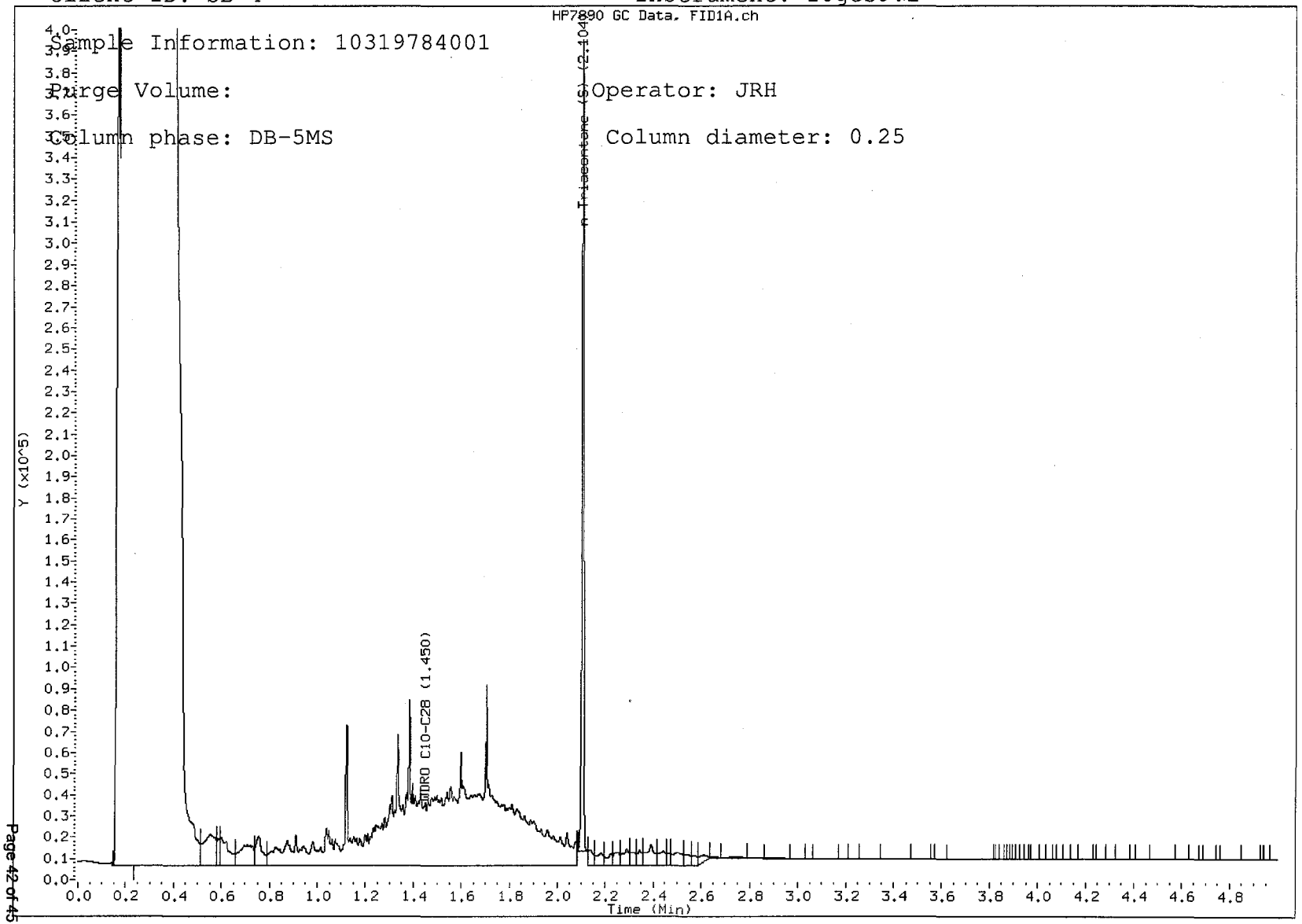
Sample ID: 10319784001

Client ID: SB-4 Instrument: 10gcs9.i

Sample Information: 10319784001

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25



Data File: \\192.168.10.12\chem\10gcs9.i\082815dro.b/082815000015.D

Report Date: 08/28/2015

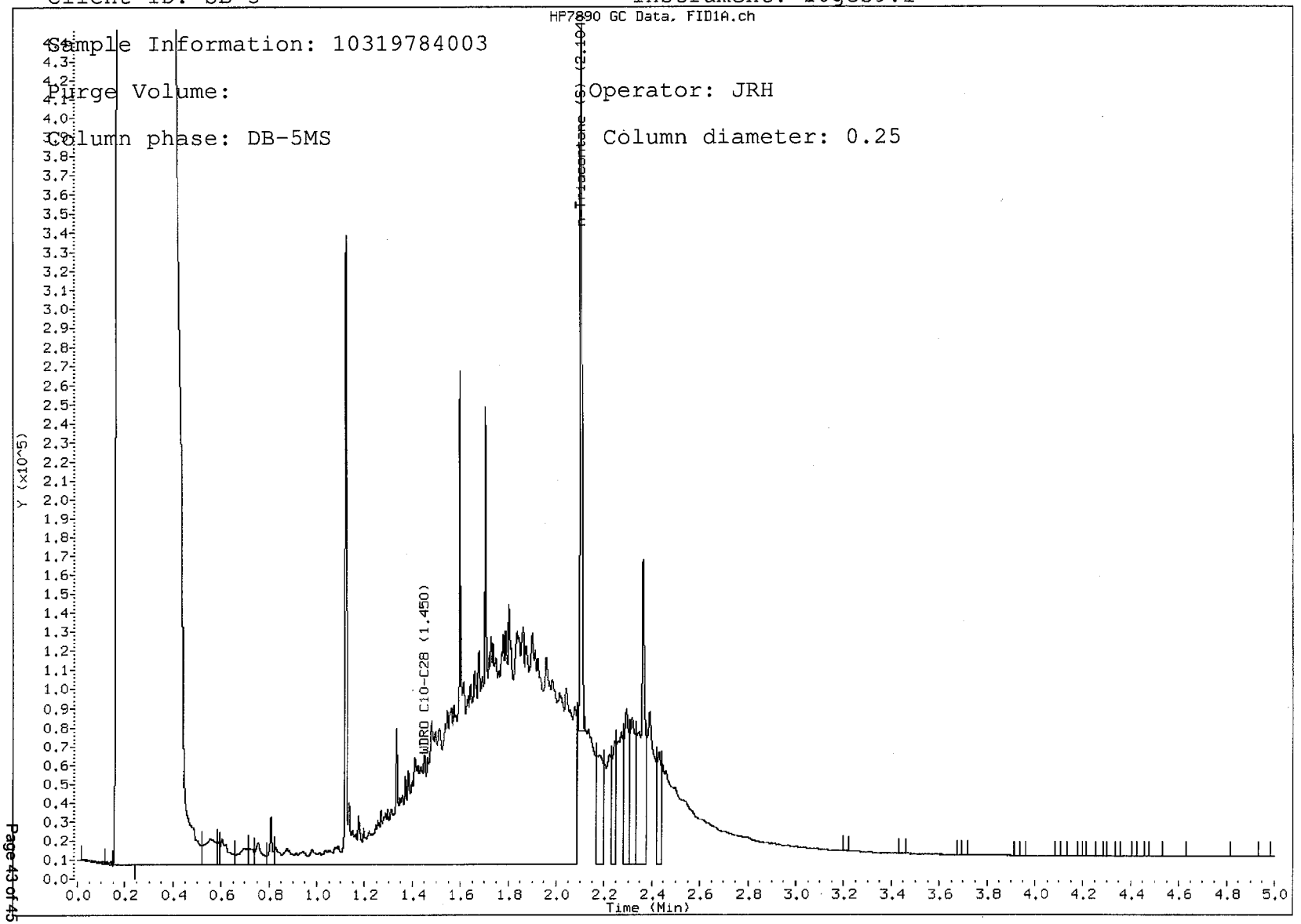
Sample ID: 10319784003

Client ID: SB-3 Instrument: 10gcs9.i

Sample Information: 10319784003

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25

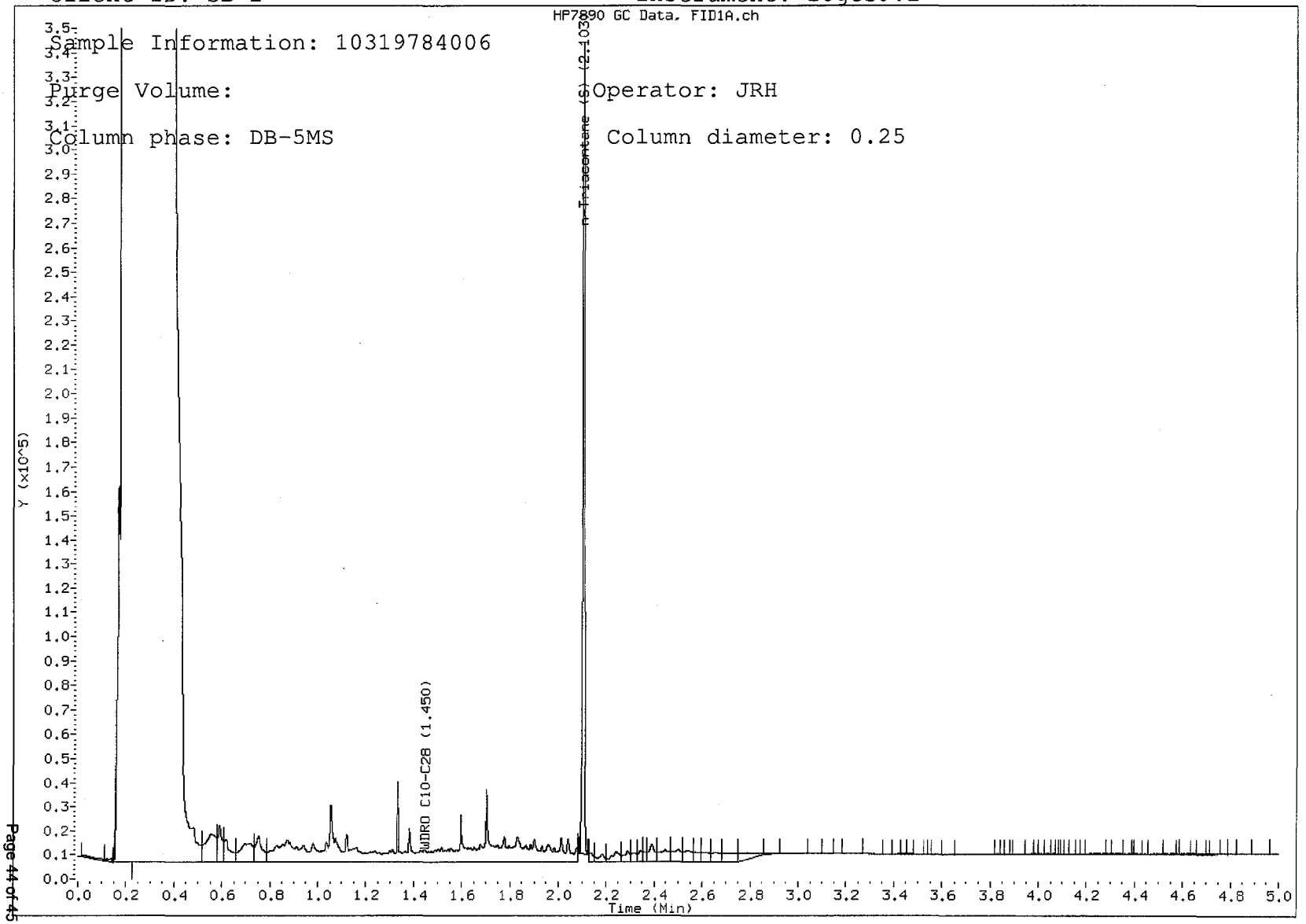


Data File: \\192.168.10.12\chem\10gcs9.i\082815dro.b/082815000025.D

Report Date: 08/28/2015

Sample ID: 10319784006

Client ID: SB-2 Instrument: 10gcs9.i



Data File: \\192.168.10.12\chem\10gcs9.i\082815dro.b/082815000026.D

Report Date: 08/28/2015

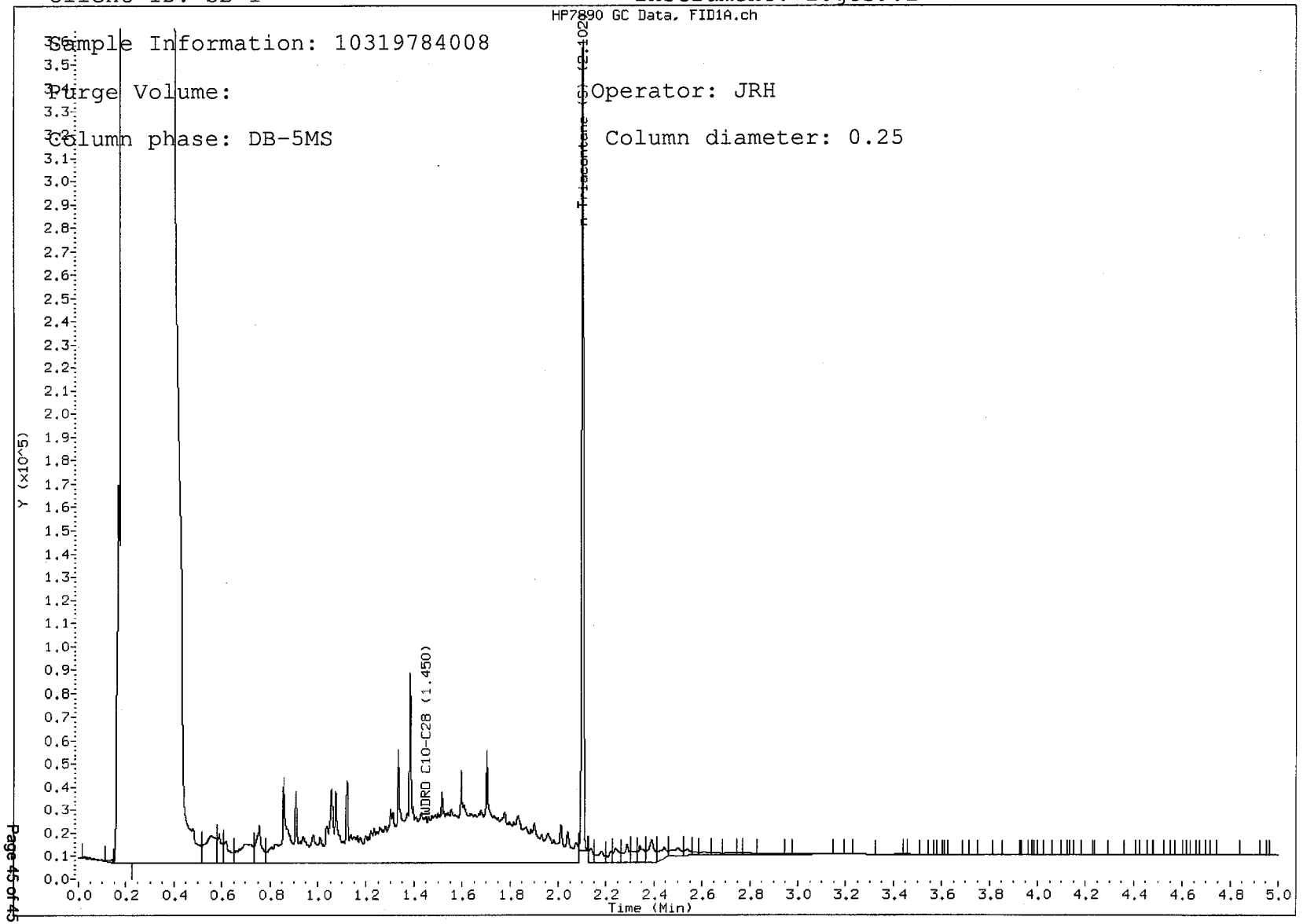
Sample ID: 10319784008

Client ID: SB-1 Instrument: 10gcs9.i

Sample Information: 10319784008

Purge Volume: Operator: JRH

Column phase: DB-5MS Column diameter: 0.25

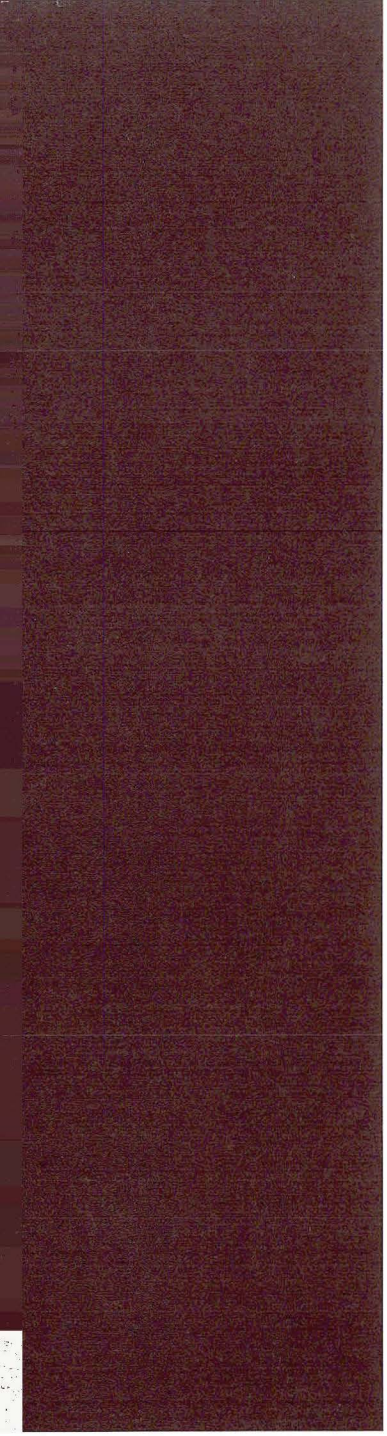


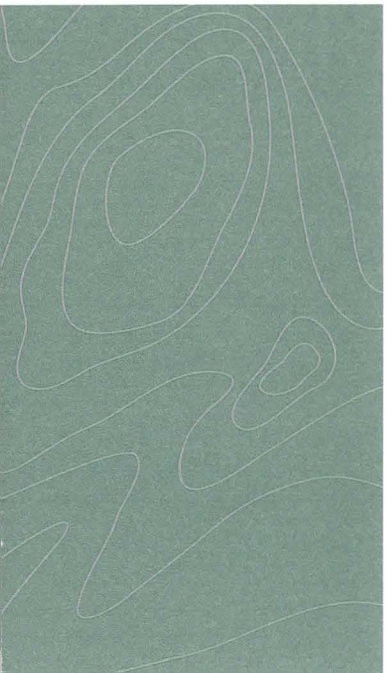
Appendix C

References

Clayton, Lee, 1984, Pleistocene Geology of the Superior Region, Wisconsin: Wisconsin Geological and Natural History Survey, Information Circular 46, scale 1:250,000. http://ngmdb.usgs.gov.

Ecological Landscapes of Wisconsin Handbook – 1805.1 WIDNR 2011. Depth to Bedrock, Scale 1:2,750,000. Wisconsin Transverse Mercator NAD83(91). Map S14-ams. http://dnr.wi.gov/topic/landscapes/documents/StateMaps/Map_S14_Bedrock_Depth.pdf.





BRAUN
INTERTEC

braunintertec.com