

Sager, John E - DNR

From: Matthew Turner <Matthew.Turner@huskyenergy.com>
Sent: Tuesday, October 30, 2018 4:54 PM
To: Sager, John E - DNR
Subject: Tank 30 update memo
Attachments: Tk 30 Update Memo.pdf

Hey John,

I have attached a memo that provides an update regarding the ongoing efforts following the naphtha release inside the dike of Tank 30 (SERTS ID: 20180227NO16-1). On the second page of the memo you will find a table of the water sampling analytical results collected as of 10/11/18.

Let me know if you have any questions or would like any further information.

Thanks,

Matt Turner
Environmental Technologist
Superior Refining Company LLC
Office: 715-398-8434
Cell: 715-969-4873
2407 Stinson Ave, Superior, WI 54880
www.huskyenergy.com





Superior Refining Company LLC

2407 Stinson Avenue

Superior, WI 54880

Summary of Water Sampling Results for Tank 30 Naphtha Release 10/30/2018

The purpose of this memorandum is to provide an update of the ongoing efforts following the release of naphtha in the tank dike of Tank 30 on February 27th, 2018 (SERTS ID: 20180227NO16-1). Per our SPCC plan, the dike drain valve was closed at the time of the release. The plan to address contaminated water in the dike was similar to what was done when naphtha was released at the site in the past; keep the water contained in the dike, remove it with vacuum trucks and treat it via our waste water treatment plant until desirable contaminant levels are achieved through rain event flushing/dilution. The first batch of water samples was collected on March 14th, 2018 and elevated contaminant levels were indicated.

A second round of samples was collected on August 29th, 2018 and significantly better results (not completely non-detect) were achieved. The WDNR was consulted about potentially opening the dike drain valve and sending the water through the typical stormwater pathway, which ultimately exits the refinery at Outfall 003. Due to the Superior Refinery Fire and subsequent increased public scrutiny, it was decided that further efforts would be needed before the water could be discharged since there were still contaminants present.

A total of 3.23 inches of rain fell in Superior, WI between 10/1/18 and 10/11/18 causing a large volume of water to accumulate in the dike of Tank 30. The volume became unmanageable and the WDNR was consulted about discharging the water. It was agreed that the water could be discharged because it was likely greatly diluted due to the rain. A third sample was collected on October 11th, 2018 which yielded the best results to date.

Once the water level in the tank dike reaches a manageable level and the soil at the release site is exposed, soil samples will be taken. If desirable results are received (below direct contact limits), the site will be transferred to the facility wide ERP.

A summary of water sampling results is contained in Table 1.

Table 1: Sample Results

Analyte	Results (ug/L)		
	3/14/2018	8/29/2018	10/11/2018
Benzene	422	3.75	0.33
Toluene	878	3.81	0.48
Ethylbenzene	105	0.472	BDL
m&p-Xylene	484	11	BDL
o-Xylene	198	15.1	
Methyl tert-butyl ether	BDL	0.429	BDL
Naphthalene	5.59	0.228	BDL
1,3,5-Trimethylbenzene	39.9	8.15	BDL
1,2,4-Trimethylbenzene	148	1.4	BDL

BDL = Below Detection Limit



Superior Refining Company LLC

2407 Stinson Avenue

Superior, WI 54880

Tel: (715) 398-3533

January 18, 2019

John Sager
Emergency Response Coordinator / Hydrogeologist
Remediation and Redevelopment Program
Wisconsin Department of Natural Resources
1701 North 4th Street
Superior, WI 54880

Re: Naphtha Release SERTS ID 20180227NO16-1

Dear Mr. Sager,

Please find the attached report regarding the immediate action taken in response to the Naphtha release reported to the WDNR on February 27, 2018 as required under NR 708.09(1). Based on the conclusions and recommendations of this report, we are requesting no further action for this release.

If you have any additional questions, please feel free to contact me at (715) 398-8434.

Sincerely,

Matt Turner
Environmental Technologist

Enclosure



Superior Refining Company LLC

2407 Stinson Avenue

Superior, WI 54880

Tel: (715) 398-3533

Immediate Response Action Report

Naphtha Release SERTS ID 20180227NO16-1

Prepared By:
Matt Turner
Superior Refining Company LLC
2407 Stinson Avenue
Superior, WI 54880
(715) 398-8434

1/18/2019

TABLE OF CONTENTS

1.0	INTRODUCTION	1
2.0	TYPE OF HAZARDOUS SUBSTANCE DISCHARGED, TOXICITY, MOBILITY AND VOLUME- NR 708.09 (1)(a)..	1
3.0	DURATION OF DISCHARGE – NR 708.09(1)(b).....	1
4.0	TIME DISCHARGE WAS RESPONDED TO AND PROPERLY CONTAINED– NR 708.09(1)(c).....	1
5.0	MITIGATION EFFORTS THAT MAY HAVE ACCELERATED MIGRATION OF POLLUTION OR HAZARDOUS SUBSTANCES NR 708.09(1)(d).....	2
6.0	WEATHER CONDITIONS – NR 708.09 (1)(e)	2
7.0	MIGRATION POTENTIAL OF THE CONTAMINATION – NR 708.09 (1)(f).....	2
8.0	NATURE AND SCOPE OF IMMEDIATE ACTION CONDUCTED - NR 708.09 (1)(g)	2
9.0	SAMPLING RESULTS - NR 708.09 (1)(h).....	3
10.0	VISUAL AND OLFACATORY EVIDENCE OF CONTAMINATION - NR 708.09 (1)(i).....	3
11.0	ACTUAL OR POTENTIAL ENVIRONMENTAL IMPACTS - NR 708.09 (1)(j)	3
12.0	PROXIMITY OF CONTAMINATION TO RECEPTORS - NR 708.09 (1)(k).....	3
13.0	PRESENT AND ANTICIPATED FUTURE LAND USE - NR 708.09 (1)(l)	4
14.0	EVALUATE IF ROUTES OF EXPOSURE ARE PROTECTIVE AND ENVIRONMENT HAS BEEN RESTORED TO THE EXTENT PRACTICABLE – NR 708.09 (1)(m).....	4
15.0	OTHER RELEVANT INFORMATION – NR 708.09 (1)(n)	4
16.0	CONCLUSION and RECOMMENDATIONS	4

LIST OF TABLES

Table 1 Sample Results

LIST OF FIGURES

Figure 1 Site Vicinity Map

Figure 2 Site Map

LIST OF APPENDICES

Appendix A Photographs

Appendix B Historical Weather Information

Appendix C Laboratory Analytical Reports

1.0 INTRODUCTION

This report contains a summary of the immediate response actions at the Superior Refining Company LLC Superior, WI refinery in response to a Naphtha release on February 27th, 2018. The site location is shown in Figure 1. The response was initiated on February 27th, 2018 after the release was discovered. The following report was prepared in accordance with Wisconsin Administrative Code NR 708 final report criteria under NR 708.09 for no further response action.

2.0 TYPE OF HAZARDOUS SUBSTANCE DISCHARGED, TOXICITY, MOBILITY AND VOLUME- NR 708.09 (1)(a)

The naphtha spill occurred in an area located south of Tank 30. The total volume of the release was estimated to be 475 gallons, all of which stayed inside the tank dike containment. The 475 gallon estimate was determined by estimating the rate at which the naphtha was being released from a leaking valve (approximately 0.6 gallons per minute) and comparing that to the amount of time that had passed between when an operator was last at the tank and the release was discovered (approximately 13.5 hours). Please see Photo _ in Appendix A for a visual representation of flow rate of the release. The release had decreased mobility due to the cold air temperature at the time it occurred. Weather conditions are shown in appendix B. The spill did not reach any water bodies, and did not migrate from the impacted area. A site map is included in Figure 2. Spill site conditions are depicted in the photographs which are included in Appendix A. WTM coordinates of the spill are included in section 15.0.

3.0 DURATION OF DISCHARGE – NR 708.09(1)(b)

The duration of the discharge is unknown. Based on the last time an operator was at the tank conducting scheduled rounds to when the release was discovered, approximately 13.5 hours had passed.

4.0 TIME DISCHARGE WAS RESPONDED TO AND PROPERLY CONTAINED– NR 708.09(1)(c)

The released material made its way along the top of an ice layer beneath the snow present in the tank dike, allowing it to travel more horizontally than during non-winter months. Once the leaking valve was identified as the source of the release, the pipe it was on was blocked in to prevent any further material from entering that portion of the line. In the meantime, a plastic pool was placed under the leaking valve to collect further released material. This material was recovered using a vacuum truck. Please see Photo 1 in Appendix A for reference. On February 27th, 2018 cleanup of the spill was initiated by means of hand excavation and vacuum truck operations.

5.0 MITIGATION EFFORTS THAT MAY HAVE ACCELERATED MIGRATION OF POLLUTION OR HAZARDOUS SUBSTANCES NR 708.09(1)(d)

Due to the snow and ice cover in the release area, the material migrated horizontally along the top of the ice rather than pool directly beneath the release source. Steam hoses were deployed in multiple areas to melt the ice to allow the material to collect on the stormwater present in the containment dike. These melted pools became collection points for vacuum trucks to recover the material. The main collection area was the dike drain in the southeast corner of the area due to the designed drainage of the tank dike. This mitigation effort allowed for a more efficient recovery of the released material and prevented further horizontal migration.

6.0 WEATHER CONDITIONS – NR 708.09 (1)(e)

Weather conditions on the day of the release were typical for that time of year. The mean temperature was 38°F, there was little to no wind, and no measurable precipitation occurred. A summary of local weather conditions for February 2018 through December 2018 is located in Appendix B.

7.0 MIGRATION POTENTIAL OF THE CONTAMINATION – NR 708.09 (1)(f)

Migration of the contamination beyond its original extent is unlikely due to the designed drainage of the containment dike as well as steam hoses deployed to create collection pools for the material. The tank dike drain valve was closed per our facility's SPCC plan. Typically, stormwater that flows through the area is inspected visually prior to being discharged to the adjacent tank dike and ultimately through stormwater outfall 003. Due to the naphtha release, the drain valve remained closed and the stormwater in the dike was collected via vacuum trucks and treated at our onsite waste water treatment plant.

8.0 NATURE AND SCOPE OF IMMEDIATE ACTION CONDUCTED - NR 708.09 (1)(g)

The release occurred due to a gasket seal failure in the bonnet of a gate valve. The valve was originally installed vertically, which allowed for the collection of water in the valve body. The entrained water then froze, damaging the gasket seal, and ultimately leaked when temperatures rose enough to thaw the ice. The valve has been replaced and installed in a horizontal fashion to prevent this from happening in the future. On February 27th, 2018 hand excavation of the contaminated snow and ice commenced. Soil excavation was delayed until stormwater levels in the tank dike were low enough to get access to the soil. The estimated volume of soil that was removed is 25 cubic feet. The excavated soil was brought to our "three-sided building" where it was stored before sending it to a proper TSD for disposal. Spill site conditions and remediation efforts are depicted in the photographs which are included in Appendix A. The area of the spill and excavation are depicted in Figure 2.

9.0 SAMPLING RESULTS - NR 708.09 (1)(h)

A total of five laboratory samples were taken from four different locations of the affected area. All soil samples were analyzed for petroleum volatile organic compounds (PVOCs) and naphthalene. Of the initial four samples sent off for analysis, all four tested above soil-to-groundwater residual contaminant labels (RCLs) established by the WDNR while three of the four were below industrial direct contact limits. The sample location where contaminant levels were above industrial direct contact limits (S3) was excavated an additional foot and another soil sample was taken (S5). This sample tested above soil-to-groundwater RCLs but below industrial direct contact limits. A summary of the analytical results is contained in Table 1. Full laboratory results are included in Appendix C.

10.0 VISUAL AND OLFACTORY EVIDENCE OF CONTAMINATION - NR 708.09 (1)(i)

Visual and olfactory evidence of the naphtha contamination was present upon arrival to the site and during the subsequent cleanup. As naphtha is clear in color, the most effective means to determine the extent of contamination is olfactory. As snow/ice was recovered, olfactory evidence diminished and was no longer noticeable after a few days. Petroleum sheen was visible in the various collection pools for a few days following the release but dissipated as material and water was recovered.

11.0 ACTUAL OR POTENTIAL ENVIRONMENTAL IMPACTS - NR 708.09 (1)(j)

The contaminated material collected during the excavation of the spill area was placed in the onsite contaminated soil storage building for bulk disposal. Disposal of the material kept in the contaminated soils building occurred at the SKB Shamrock Environmental Landfill located in Cloquet, MN. Potential environmental impacts are minimal. The spill was contained on-site and did not run off into other areas, and was restricted to the area of the excavation; therefore, the actual or potential environmental impacts are minimal.

12.0 PROXIMITY OF CONTAMINATION TO RECEPTORS - NR 708.09 (1)(k)

Exposure via the groundwater pathway is strongly a function of the soil permeability. Groundwater velocities in the clay are on the order of 0.013 ft/yr. Petroleum compounds will also be naturally attenuated by retardation and biodegradation processes, thus will have transport velocities less than groundwater velocities. The closest groundwater receptor is Newton Creek, which is several hundred feet downgradient from the impacted area. Using a contaminant transport velocity of 0.013 ft/yr (assumes no retardation), it would take thousands of years for groundwater from this area to reach Newton Creek. In reality, the small amount of residual petroleum contaminants will very likely naturally attenuate (biodegraded or sorbed onto the aquifer matrix) as they are being transported and it is highly unlikely that

any residual dissolved-phase compounds will ever reach Newton Creek. Based on the very low groundwater velocities and absence of any close proximity groundwater receptors, there is literally no groundwater exposure risk at the refinery.

13.0 PRESENT AND ANTICIPATED FUTURE LAND USE - NR 708.09 (1)(l)

The land where the release occurred is presently used as a tank perimeter dike for an oil refinery. The refinery was constructed in 1951 and has remained in the same use since that time. There is no anticipation the land will be used for another purpose in the future.

14.0 EVALUATE IF ROUTES OF EXPOSURE ARE PROTECTIVE AND ENVIRONMENT HAS BEEN RESTORED TO THE EXTENT PRACTICABLE – NR 708.09 (1)(m)

A good faith effort was undertaken to remove all newly contaminated material from the release site. No off-site receptors were impacted by the release. Given the amount of naphtha released, the site conditions at the time of the release, and the confirmation sampling results, there is little chance the naphtha migrated laterally beyond the tank containment dike. Confirmation samples indicate the spill has been remediated to below industrial direct contact limits.

15.0 OTHER RELEVANT INFORMATION – NR 708.09 (1)(n)

The site is located in the NW ¼ of the NW ¼ of Section 36, Township 49 North, Range 14 West, City of Superior, Douglas County, WI. The WTM coordinates for the spill site are 361513, 693123. A site vicinity map is included in Figure 1.

16.0 CONCLUSION AND RECOMMENDATIONS

Based on laboratory results indicating the presence of contaminants above the NR720 soil-to-groundwater RCL's, the spill has not been remediated to allow for clean closure. Therefore, it is recommended that site be transferred to the facility-wide SI-RAP and no further response action is necessary at the site.

TABLES

Table 1

Sample Results

Table 1 - Sample Results

Analyte	Results (mg/kg)				
	11/8/2018				12/6/2018
	S1	S2	S3	S4	S5
Benzene	0.568	3.8	13.0	1.86	3.09
Toluene	0.513	11.0	32.4	4.47	12.5
Ethylbenzene	0.803	2.4	16.2	1.05	2.88
m&p-Xylene	5.35	11.0	54.2	5.63	13
o-Xylene	1.29	3.74	24.7	2.38	4.87
Methyl tert-butyl ether	0.369	BDL	BDL	BDL	0.0961
Naphthalene	0.329	BDL	0.134	BDL	BDL
1,3,5-Trimethylbenzene	3.80	1.82	12.2	1.33	2.4
1,2,4-Trimethylbenzene	8.44	4.80	27.5	3.20	6.13

BDL = Below Detection Limit

Analyte	Amount Below Direct Contact Limit (mg/kg)				
	S1	S2	S3	S4	S5
Benzene	6.842	3.610	-5.590	5.550	4.320
Toluene	36.487	26.000	4.600	32.530	24.500
Ethylbenzene	817.197	815.600	801.800	816.950	815.120
m&p-Xylene	253.36	245.26	181.10	251.99	242.13
o-Xylene					
Methyl tert-butyl ether	292.631	293.000	293.000	293.000	292.904
Naphthalene	218.671	219.000	218.866	219.000	219.000
1,3,5-Trimethylbenzene	178.20	180.18	169.80	180.67	179.600
1,2,4-Trimethylbenzene	17.560	21.200	-1.500	22.800	19.870

Direct Contact Limits (mg/kg)	
Benzene	7.41
Ethylbenzene	37
Toluene	818
m&p-Xylene	260
o-Xylene	
Methyl tert-Butyl Ether	293
Trimethylbenzene, 1,2,4-	219
Trimethylbenzene, 1,3,5-	182
Naphthalene	26

FIGURES

Figure 1 Site Vicinity Map

Figure 2 Site Map



Figure 1 Tank 30 Naphtha Release



Legend

Notes

0.3 0 0.13 0.3 Miles

NAD_1983_HARN_Wisconsin_TM

© Latitude Geographics Group Ltd.

1: 7,920



DISCLAIMER: The information shown on these maps has been obtained from various sources, and are of varying age, reliability and resolution. These maps are not intended to be used for navigation, nor are these maps an authoritative source of information about legal land ownership or public access. No warranty, expressed or implied, is made regarding accuracy, applicability for a particular use, completeness, or legality of the information depicted on this map. For more information, see the DNR Legal Notices web page: <http://dnr.wi.gov/org/legal/>

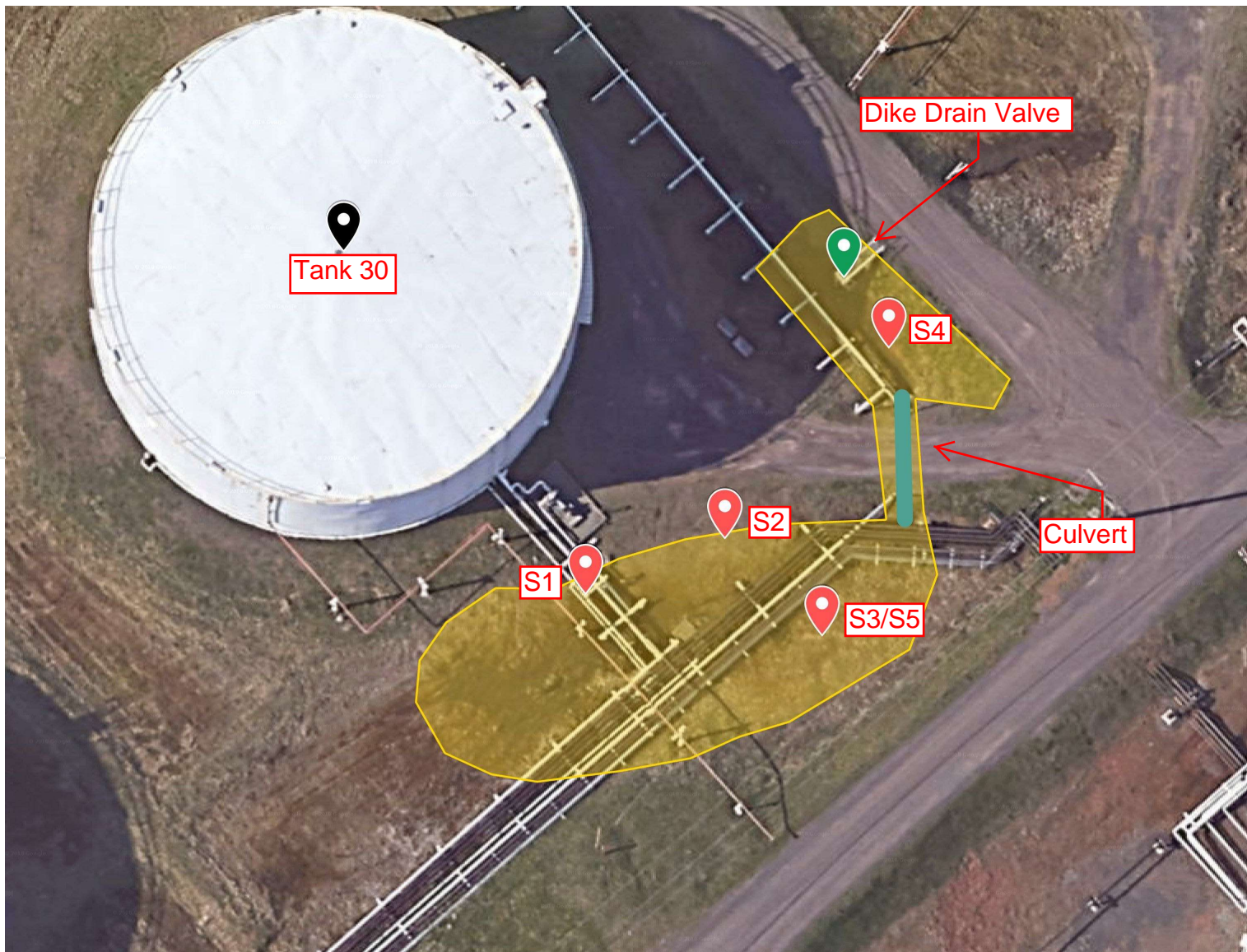
Note: Not all sites are mapped.

Figure 2 - Site Map

Superior Refinery

Tank 30 Naphtha Release

- Estimated Release Extent
- Tk 30 Dike Drain Valve
- Release Source
- Culvert
- S1
- S2
- S3/S5
- S4
- Tank 30



APPENDICES

Appendix A	Photographs
Appendix B	Weather Information
Appendix C	Laboratory Analytical Data

Appendix A - Photographs



Photo 1: Looking east at the leaking valve. Note the stream of naphtha coming from the gasket on the body of the valve. A plastic container was installed to catch the material and a vacuum truck hose can be seen recovering the material from the container.



Photo 2: Looking northwest at the release site. The leaking valve is pictured in the center of the image.



Photo 3: Looking east at the release site. Both the northern and western extent of the can be identified by the free liquid (mostly melt water) present in the grassy area.



Photo 4: Looking southeast at the release site.



Photo 5: Looking east at the tank dike drain valve pooling location. Steam coils were placed to melt the snow and ice to allow vacuum trucks to collect the recoverable product and contaminated water.



Photo 6: Looking northeast at the absorbent boom deployed along the eastern edge of the tank containment dike. This was placed upgradient (north) of the dike drain valve pooling location as a precaution.

Appendix B - Historical Weather Information

		February	March	April	May	June	July	August	September	October	November	December
Temperature	High	43	48	71	92	85	89	91	81	66	44	44
	Low	-17	4	2	29	40	49	48	30	26	-7	-9
	Average High	20.2	34.9	42.8	71.1	72.6	80	77.4	67.2	47.2	29.4	28.5
	Average Low	0.3	17.2	22.8	44.3	51	58.2	57.7	51	34.8	17	14.6
	Mean	10.3	26.1	32.8	57.7	61.8	69.1	67.5	59.1	41	23.2	21.6
Precipitation	Inches	1.73	0.44	1.39	2.21	4.64	3.28	3.86	3.51	4.67	1.67	1.77
Snowfall	Inches	24.2	5.1	18.5	0	0	0	0	0	1.5	12.2	19.8
Average Wind	MPH	11.1	10.3	10.2	9.1	9.3	8.1	7.3	9.9	10.8	9.3	9.7
Sky Cover	Days Fair	13	15	17	17	17	17	12	7	7	0	7
	Days PC	9	9	7	11	8	13	11	13	7	15	9
	Days Cloudy	6	7	6	3	5	1	8	10	17	15	15



ANALYTICAL REPORT

November 19, 2018

Superior Refinery Husky Energy

Sample Delivery Group: L1042936
Samples Received: 11/09/2018
Project Number:
Description: Tk 30 Soil Sampling

Report To: Matt Turner
2407 Stinson Avenue
Superior, WI 54880

Entire Report Reviewed By:

John Hawkins
Project Manager

Results relate only to the items tested or calibrated and are reported as rounded values. This test report shall not be reproduced, except in full, without written approval of the laboratory. Where applicable, sampling conducted by Pace National is performed per guidance provided in laboratory standard operating procedures: 060302, 060303, and 060304.



Cp: Cover Page	1	1 Cp
Tc: Table of Contents	2	
Ss: Sample Summary	3	2 Tc
Cn: Case Narrative	4	
Sr: Sample Results	5	3 Ss
S1 L1042936-01	5	
S2 L1042936-02	6	4 Cn
S3 L1042936-03	7	
S4 L1042936-04	8	5 Sr
Qc: Quality Control Summary	9	6 Qc
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	9	
Gl: Glossary of Terms	12	7 Gl
Al: Accreditations & Locations	13	8 Al
Sc: Sample Chain of Custody	14	9 Sc

SAMPLE SUMMARY



S1 L1042936-01 Solid

Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Collected by			Tim C.	Collected date/time	Received date/time
				11/08/18 12:55	11/09/18 08:45
Volatile Organic Compounds (GC) by Method 8021B	WG1197504	100	11/08/18 12:55	11/18/18 12:27	DWR
Volatile Organic Compounds (GC) by Method Wl(95) GRO	WG1197504	500	11/08/18 12:55	11/18/18 15:35	DWR

1 Cp

2 Tc

3 Ss

S2 L1042936-02 Solid

Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Collected by			Tim C.	Collected date/time	Received date/time
				11/08/18 13:07	11/09/18 08:45
Volatile Organic Compounds (GC) by Method 8021B/Wl(95) GRO	WG1195018	200	11/08/18 13:07	11/13/18 13:11	BMB

4 Cn

5 Sr

6 Qc

S3 L1042936-03 Solid

Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Collected by			Tim C.	Collected date/time	Received date/time
				11/08/18 13:15	11/09/18 08:45
Volatile Organic Compounds (GC) by Method 8021B	WG1195018	126	11/08/18 13:15	11/13/18 13:35	BMB
Volatile Organic Compounds (GC) by Method 8021B/Wl(95) GRO	WG1197504	1260	11/08/18 13:15	11/18/18 12:51	DWR

7 Gl

8 Al

9 Sc

S4 L1042936-04 Solid

Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Collected by			Tim C.	Collected date/time	Received date/time
				11/08/18 13:25	11/09/18 08:45
Volatile Organic Compounds (GC) by Method 8021B/Wl(95) GRO	WG1195018	100	11/08/18 13:25	11/13/18 13:59	BMB



All sample aliquots were received at the correct temperature, in the proper containers, with the appropriate preservatives, and within method specified holding times, unless qualified or notated within the report. Where applicable, all MDL (LOD) and RDL (LOQ) values reported for environmental samples have been corrected for the dilution factor used in the analysis. All Method and Batch Quality Control are within established criteria except where addressed in this case narrative, a non-conformance form or properly qualified within the sample results. By my digital signature below, I affirm to the best of my knowledge, all problems/anomalies observed by the laboratory as having the potential to affect the quality of the data have been identified by the laboratory, and no information or data have been knowingly withheld that would affect the quality of the data.

John Hawkins
Project Manager

- ¹ Cp
- ² Tc
- ³ Ss
- ⁴ Cn
- ⁵ Sr
- ⁶ Qc
- ⁷ Gl
- ⁸ Al
- ⁹ Sc



Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO

Analyte	Result	Qualifier	MDL	RDL	Dilution	Analysis	Batch
	mg/kg		mg/kg	mg/kg		date / time	
Benzene	0.568		0.00880	0.0293	100	11/18/2018 12:27	WG1197504
Toluene	0.513		0.0161	0.0537	100	11/18/2018 12:27	WG1197504
Ethylbenzene	0.803		0.00910	0.0303	100	11/18/2018 12:27	WG1197504
m&p-Xylene	5.35		0.0154	0.0513	100	11/18/2018 12:27	WG1197504
o-Xylene	1.29		0.00960	0.0320	100	11/18/2018 12:27	WG1197504
Methyl tert-butyl ether	0.369		0.0160	0.0533	100	11/18/2018 12:27	WG1197504
Naphthalene	0.329	<u>J J6</u>	0.104	0.347	100	11/18/2018 12:27	WG1197504
1,3,5-Trimethylbenzene	3.80	<u>J3 J6</u>	0.00820	0.0273	100	11/18/2018 12:27	WG1197504
1,2,4-Trimethylbenzene	8.44	<u>J6</u>	0.0107	0.0357	100	11/18/2018 12:27	WG1197504
TPH (GC/FID) Low Fraction	175		5.50	18.4	500	11/18/2018 15:35	WG1197504
<i>(S) a, a, a-Trifluorotoluene(PID)</i>	101			80.0-200		11/18/2018 12:27	WG1197504
<i>(S) a, a, a-Trifluorotoluene(PID)</i>	102			80.0-200		11/18/2018 15:35	WG1197504

Sample Narrative:

L1042936-01 WG1197504: Peaks/Baseline rise detected outside GRO/DRO window

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc



Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO

Analyte	Result	Qualifier	MDL	RDL	Dilution	Analysis	Batch
	mg/kg		mg/kg	mg/kg		date / time	
Benzene	3.80		0.0176	0.0586	200	11/13/2018 13:11	WG1195018
Toluene	11.0		0.0322	0.107	200	11/13/2018 13:11	WG1195018
Ethylbenzene	2.40		0.0182	0.0606	200	11/13/2018 13:11	WG1195018
m&p-Xylene	11.0		0.0308	0.103	200	11/13/2018 13:11	WG1195018
o-Xylene	3.74		0.0192	0.0640	200	11/13/2018 13:11	WG1195018
Methyl tert-butyl ether	U		0.0320	0.107	200	11/13/2018 13:11	WG1195018
Naphthalene	U		0.208	0.694	200	11/13/2018 13:11	WG1195018
1,3,5-Trimethylbenzene	1.82		0.0164	0.0546	200	11/13/2018 13:11	WG1195018
1,2,4-Trimethylbenzene	4.80		0.0214	0.0714	200	11/13/2018 13:11	WG1195018
TPH (GC/FID) Low Fraction	261		2.20	7.34	200	11/13/2018 13:11	WG1195018
(S) a, a, a-Trifluorotoluene(PID)	95.6			80.0-200		11/13/2018 13:11	WG1195018

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc



Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO

Analyte	Result	Qualifier	MDL	RDL	Dilution	Analysis	Batch
	mg/kg		mg/kg	mg/kg		date / time	
Benzene	13.0		0.0111	0.0369	126	11/13/2018 13:35	WG1195018
Toluene	32.4		0.203	0.677	1260	11/18/2018 12:51	WG1197504
Ethylbenzene	16.2		0.0115	0.0382	126	11/13/2018 13:35	WG1195018
m&p-Xylene	54.2		0.0194	0.0646	126	11/13/2018 13:35	WG1195018
o-Xylene	24.7		0.0121	0.0403	126	11/13/2018 13:35	WG1195018
Methyl tert-butyl ether	U		0.0202	0.0672	126	11/13/2018 13:35	WG1195018
Naphthalene	0.134	J	0.131	0.437	126	11/13/2018 13:35	WG1195018
1,3,5-Trimethylbenzene	12.2		0.0103	0.0344	126	11/13/2018 13:35	WG1195018
1,2,4-Trimethylbenzene	27.5		0.0135	0.0450	126	11/13/2018 13:35	WG1195018
TPH (GC/FID) Low Fraction	341		13.9	46.2	1260	11/18/2018 12:51	WG1197504
(S) a,a,a-Trifluorotoluene(PID)	93.3			80.0-200		11/13/2018 13:35	WG1195018
(S) a,a,a-Trifluorotoluene(PID)	100			80.0-200		11/18/2018 12:51	WG1197504

- 1 Cp
- 2 Tc
- 3 Ss
- 4 Cn
- 5 Sr
- 6 Qc
- 7 Gl
- 8 Al
- 9 Sc

Sample Narrative:

L1042936-03 WG1197504: Peaks/Baseline rise detected outside GRO/DRO window



Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO

Analyte	Result mg/kg	Qualifier	MDL mg/kg	RDL mg/kg	Dilution	Analysis date / time	Batch
Benzene	1.86		0.00880	0.0293	100	11/13/2018 13:59	WG1195018
Toluene	4.47		0.0161	0.0537	100	11/13/2018 13:59	WG1195018
Ethylbenzene	1.05		0.00910	0.0303	100	11/13/2018 13:59	WG1195018
m&p-Xylene	5.63		0.0154	0.0513	100	11/13/2018 13:59	WG1195018
o-Xylene	2.38		0.00960	0.0320	100	11/13/2018 13:59	WG1195018
Methyl tert-butyl ether	U		0.0160	0.0533	100	11/13/2018 13:59	WG1195018
Naphthalene	U		0.104	0.347	100	11/13/2018 13:59	WG1195018
1,3,5-Trimethylbenzene	1.33		0.00820	0.0273	100	11/13/2018 13:59	WG1195018
1,2,4-Trimethylbenzene	3.20		0.0107	0.0357	100	11/13/2018 13:59	WG1195018
TPH (GC/FID) Low Fraction	168		1.10	3.67	100	11/13/2018 13:59	WG1195018
(S) a, a, a-Trifluorotoluene(PID)	93.6			80.0-200		11/13/2018 13:59	WG1195018

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc



Method Blank (MB)

(MB) R3360369-3 11/13/18 11:41

Analyte	MB Result mg/kg	MB Qualifier	MB MDL mg/kg	MB RDL mg/kg
Benzene	U		0.000880	0.000293
Toluene	U		0.000161	0.000537
Ethylbenzene	0.000102	↓	0.0000910	0.000303
m&p-Xylene	0.000169	↓	0.000154	0.000513
o-Xylene	U		0.0000960	0.000320
Methyl tert-butyl ether	U		0.000160	0.000533
Naphthalene	U		0.00104	0.00347
1,3,5-Trimethylbenzene	0.000126	↓	0.0000820	0.000273
1,2,4-Trimethylbenzene	0.000117	↓	0.000107	0.000357
TPH (GC/FID) Low Fraction	U		0.0110	0.0367
(S) a,a,a-Trifluorotoluene(PID)	99.8			80.0-200

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc

Laboratory Control Sample (LCS) • Laboratory Control Sample Duplicate (LCSD)

(LCS) R3360369-1 11/13/18 10:51 • (LCSD) R3360369-4 11/13/18 16:19

Analyte	Spike Amount mg/kg	LCS Result mg/kg	LCSD Result mg/kg	LCS Rec. %	LCSD Rec. %	Rec. Limits %	LCS Qualifier	LCSD Qualifier	RPD %	RPD Limits %
Benzene	0.0500	0.0498	0.0509	99.6	102	80.0-120			2.13	20
Toluene	0.0500	0.0513	0.0529	103	106	80.0-120			2.99	20
Ethylbenzene	0.0500	0.0508	0.0522	102	104	80.0-120			2.65	20
m&p-Xylene	0.100	0.102	0.105	102	105	80.0-120			3.60	20
o-Xylene	0.0500	0.0511	0.0527	102	105	80.0-120			3.21	20
Methyl tert-butyl ether	0.0500	0.0475	0.0479	95.1	95.9	80.0-120			0.870	20
Naphthalene	0.0500	0.0453	0.0515	90.7	103	80.0-120			12.7	20
1,3,5-Trimethylbenzene	0.0500	0.0513	0.0527	103	105	80.0-120			2.78	20
1,2,4-Trimethylbenzene	0.0500	0.0511	0.0531	102	106	80.0-120			3.86	20
(S) a,a,a-Trifluorotoluene(PID)				107	94.6	80.0-200				

Laboratory Control Sample (LCS) • Laboratory Control Sample Duplicate (LCSD)

(LCS) R3360369-2 11/13/18 10:51 • (LCSD) R3360369-5 11/13/18 16:19

Analyte	Spike Amount mg/kg	LCS Result mg/kg	LCSD Result mg/kg	LCS Rec. %	LCSD Rec. %	Rec. Limits %	LCS Qualifier	LCSD Qualifier	RPD %	RPD Limits %
TPH (GC/FID) Low Fraction	0.550	0.524	0.532	95.2	96.7	80.0-120			1.55	20
(S) a,a,a-Trifluorotoluene(PID)				107	94.6	80.0-200				



Method Blank (MB)

(MB) R3361011-3 11/18/18 11:03

Analyte	MB Result	MB Qualifier	MB MDL	MB RDL
	mg/kg		mg/kg	mg/kg
Benzene	U		0.0000880	0.000293
Toluene	U		0.000161	0.000537
Ethylbenzene	0.0000973	↓	0.0000910	0.000303
m&p-Xylene	0.000177	↓	0.000154	0.000513
o-Xylene	U		0.0000960	0.000320
Methyl tert-butyl ether	U		0.000160	0.000533
Naphthalene	U		0.00104	0.00347
1,3,5-Trimethylbenzene	0.000142	↓	0.0000820	0.000273
1,2,4-Trimethylbenzene	0.000136	↓	0.000107	0.000357
TPH (GC/FID) Low Fraction	U		0.0110	0.0367
^(S) a,a,a-Trifluorotoluene(PID)	103			80.0-200

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc

Laboratory Control Sample (LCS) • Laboratory Control Sample Duplicate (LCSD)

(LCS) R3361011-1 11/18/18 10:14 • (LCSD) R3361011-8 11/18/18 16:12

Analyte	Spike Amount	LCS Result	LCSD Result	LCS Rec.	LCSD Rec.	Rec. Limits	LCS Qualifier	LCSD Qualifier	RPD	RPD Limits
	mg/kg	mg/kg	mg/kg	%	%	%			%	%
Benzene	0.0500	0.0485	0.0488	96.9	97.6	80.0-120			0.697	20
Toluene	0.0500	0.0501	0.0504	100	101	80.0-120			0.586	20
Ethylbenzene	0.0500	0.0505	0.0505	101	101	80.0-120			0.00785	20
m&p-Xylene	0.100	0.100	0.101	100	101	80.0-120			0.476	20
o-Xylene	0.0500	0.0503	0.0504	101	101	80.0-120			0.148	20
Methyl tert-butyl ether	0.0500	0.0500	0.0498	100	99.6	80.0-120			0.362	20
Naphthalene	0.0500	0.0466	0.0419	93.2	83.8	80.0-120			10.6	20
1,3,5-Trimethylbenzene	0.0500	0.0512	0.0514	102	103	80.0-120			0.278	20
1,2,4-Trimethylbenzene	0.0500	0.0513	0.0515	103	103	80.0-120			0.482	20
^(S) a,a,a-Trifluorotoluene(PID)				106	102	80.0-200				

Laboratory Control Sample (LCS) • Laboratory Control Sample Duplicate (LCSD)

(LCS) R3361011-2 11/18/18 10:14 • (LCSD) R3361011-9 11/18/18 16:12

Analyte	Spike Amount	LCS Result	LCSD Result	LCS Rec.	LCSD Rec.	Rec. Limits	LCS Qualifier	LCSD Qualifier	RPD	RPD Limits
	mg/kg	mg/kg	mg/kg	%	%	%			%	%
TPH (GC/FID) Low Fraction	0.550	0.538	0.552	97.7	100	80.0-120			2.66	20
^(S) a,a,a-Trifluorotoluene(PID)				106	102	80.0-200				



L1042936-01 Original Sample (OS) • Matrix Spike (MS) • Matrix Spike Duplicate (MSD)

(OS) L1042936-01 11/18/18 12:27 • (MS) R3361011-4 11/18/18 13:16 • (MSD) R3361011-6 11/18/18 13:40

Analyte	Spike Amount mg/kg	Original Result mg/kg	MS Result mg/kg	MSD Result mg/kg	MS Rec. %	MSD Rec. %	Dilution	Rec. Limits %	MS Qualifier	MSD Qualifier	RPD %	RPD Limits %
Benzene	0.0500	0.568	3.84	3.82	65.4	65.0	100	32.0-137			0.462	39
Toluene	0.0500	0.513	4.09	4.11	71.6	71.8	100	20.0-142			0.304	42
Ethylbenzene	0.0500	0.803	4.72	4.76	78.3	79.1	100	10.0-150			0.760	44
m&p-Xylene	0.100	5.35	12.1	12.2	67.1	68.1	100	14.0-141			0.778	44
o-Xylene	0.0500	1.29	5.21	5.27	78.3	79.7	100	10.0-157			1.32	44
Methyl tert-butyl ether	0.0500	0.369	4.41	4.45	80.8	81.6	100	24.0-151			0.890	37
Naphthalene	0.0500	0.329	4.26	4.62	78.6	85.9	100	80.0-120	<u>J6</u>		8.18	20
1,3,5-Trimethylbenzene	0.0500	3.80	7.73	9.65	78.6	117	100	80.0-120	<u>J6</u>	<u>J3</u>	22.0	20
1,2,4-Trimethylbenzene	0.0500	8.44	10.7	12.5	45.2	80.4	100	80.0-120	<u>J6</u>		15.2	20
(S) a,a,a-Trifluorotoluene(PID)					99.1	96.6		80.0-200				

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc

L1042936-01 Original Sample (OS) • Matrix Spike (MS) • Matrix Spike Duplicate (MSD)

(OS) L1042936-01 11/18/18 12:27 • (MS) R3361011-5 11/18/18 13:16 • (MSD) R3361011-7 11/18/18 13:40

Analyte	Spike Amount mg/kg	Original Result mg/kg	MS Result mg/kg	MSD Result mg/kg	MS Rec. %	MSD Rec. %	Dilution	Rec. Limits %	MS Qualifier	MSD Qualifier	RPD %	RPD Limits %
TPH (GC/FID) Low Fraction	0.550		256	267	54.1	74.3	100	80.0-120	<u>E V</u>	<u>E V</u>	4.24	20
(S) a,a,a-Trifluorotoluene(PID)					99.1	96.6		80.0-200				



Guide to Reading and Understanding Your Laboratory Report

The information below is designed to better explain the various terms used in your report of analytical results from the Laboratory. This is not intended as a comprehensive explanation, and if you have additional questions please contact your project representative.

Abbreviations and Definitions

MDL	Method Detection Limit.
RDL	Reported Detection Limit.
Rec.	Recovery.
RPD	Relative Percent Difference.
SDG	Sample Delivery Group.
(S)	Surrogate (Surrogate Standard) - Analytes added to every blank, sample, Laboratory Control Sample/Duplicate and Matrix Spike/Duplicate; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in all environmental media.
U	Not detected at the Reporting Limit (or MDL where applicable).
Analyte	The name of the particular compound or analysis performed. Some Analyses and Methods will have multiple analytes reported.
Dilution	If the sample matrix contains an interfering material, the sample preparation volume or weight values differ from the standard, or if concentrations of analytes in the sample are higher than the highest limit of concentration that the laboratory can accurately report, the sample may be diluted for analysis. If a value different than 1 is used in this field, the result reported has already been corrected for this factor.
Limits	These are the target % recovery ranges or % difference value that the laboratory has historically determined as normal for the method and analyte being reported. Successful QC Sample analysis will target all analytes recovered or duplicated within these ranges.
Original Sample	The non-spiked sample in the prep batch used to determine the Relative Percent Difference (RPD) from a quality control sample. The Original Sample may not be included within the reported SDG.
Qualifier	This column provides a letter and/or number designation that corresponds to additional information concerning the result reported. If a Qualifier is present, a definition per Qualifier is provided within the Glossary and Definitions page and potentially a discussion of possible implications of the Qualifier in the Case Narrative if applicable.
Result	The actual analytical final result (corrected for any sample specific characteristics) reported for your sample. If there was no measurable result returned for a specific analyte, the result in this column may state "ND" (Not Detected) or "BDL" (Below Detectable Levels). The information in the results column should always be accompanied by either an MDL (Method Detection Limit) or RDL (Reporting Detection Limit) that defines the lowest value that the laboratory could detect or report for this analyte.
Case Narrative (Cn)	A brief discussion about the included sample results, including a discussion of any non-conformances to protocol observed either at sample receipt by the laboratory from the field or during the analytical process. If present, there will be a section in the Case Narrative to discuss the meaning of any data qualifiers used in the report.
Quality Control Summary (Qc)	This section of the report includes the results of the laboratory quality control analyses required by procedure or analytical methods to assist in evaluating the validity of the results reported for your samples. These analyses are not being performed on your samples typically, but on laboratory generated material.
Sample Chain of Custody (Sc)	This is the document created in the field when your samples were initially collected. This is used to verify the time and date of collection, the person collecting the samples, and the analyses that the laboratory is requested to perform. This chain of custody also documents all persons (excluding commercial shippers) that have had control or possession of the samples from the time of collection until delivery to the laboratory for analysis.
Sample Results (Sr)	This section of your report will provide the results of all testing performed on your samples. These results are provided by sample ID and are separated by the analyses performed on each sample. The header line of each analysis section for each sample will provide the name and method number for the analysis reported.
Sample Summary (Ss)	This section of the Analytical Report defines the specific analyses performed for each sample ID, including the dates and times of preparation and/or analysis.

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc

Qualifier Description

E	The analyte concentration exceeds the upper limit of the calibration range of the instrument established by the initial calibration (ICAL).
J	The identification of the analyte is acceptable; the reported value is an estimate.
J3	The associated batch QC was outside the established quality control range for precision.
J6	The sample matrix interfered with the ability to make any accurate determination; spike value is low.
V	The sample concentration is too high to evaluate accurate spike recoveries.



Pace National is the only environmental laboratory accredited/certified to support your work nationwide from one location. One phone call, one point of contact, one laboratory. No other lab is as accessible or prepared to handle your needs throughout the country. Our capacity and capability from our single location laboratory is comparable to the collective totals of the network laboratories in our industry. The most significant benefit to our one location design is the design of our laboratory campus. The model is conducive to accelerated productivity, decreasing turn-around time, and preventing cross contamination, thus protecting sample integrity. Our focus on premium quality and prompt service allows us to be YOUR LAB OF CHOICE.

* Not all certifications held by the laboratory are applicable to the results reported in the attached report.
 * Accreditation is only applicable to the test methods specified on each scope of accreditation held by Pace National.

State Accreditations

Alabama	40660	Nebraska	NE-OS-15-05
Alaska	17-026	Nevada	TN-03-2002-34
Arizona	AZ0612	New Hampshire	2975
Arkansas	88-0469	New Jersey-NELAP	TN002
California	2932	New Mexico ¹	n/a
Colorado	TN00003	New York	11742
Connecticut	PH-0197	North Carolina	Env375
Florida	E87487	North Carolina ¹	DW21704
Georgia	NELAP	North Carolina ³	41
Georgia ¹	923	North Dakota	R-140
Idaho	TN00003	Ohio-VAP	CL0069
Illinois	200008	Oklahoma	9915
Indiana	C-TN-01	Oregon	TN200002
Iowa	364	Pennsylvania	68-02979
Kansas	E-10277	Rhode Island	LA000356
Kentucky ^{1,6}	90010	South Carolina	84004
Kentucky ²	16	South Dakota	n/a
Louisiana	AI30792	Tennessee ^{1,4}	2006
Louisiana ¹	LA180010	Texas	T 104704245-17-14
Maine	TN0002	Texas ⁵	LAB0152
Maryland	324	Utah	TN00003
Massachusetts	M-TN003	Vermont	VT2006
Michigan	9958	Virginia	460132
Minnesota	047-999-395	Washington	C847
Mississippi	TN00003	West Virginia	233
Missouri	340	Wisconsin	9980939910
Montana	CERT0086	Wyoming	A2LA

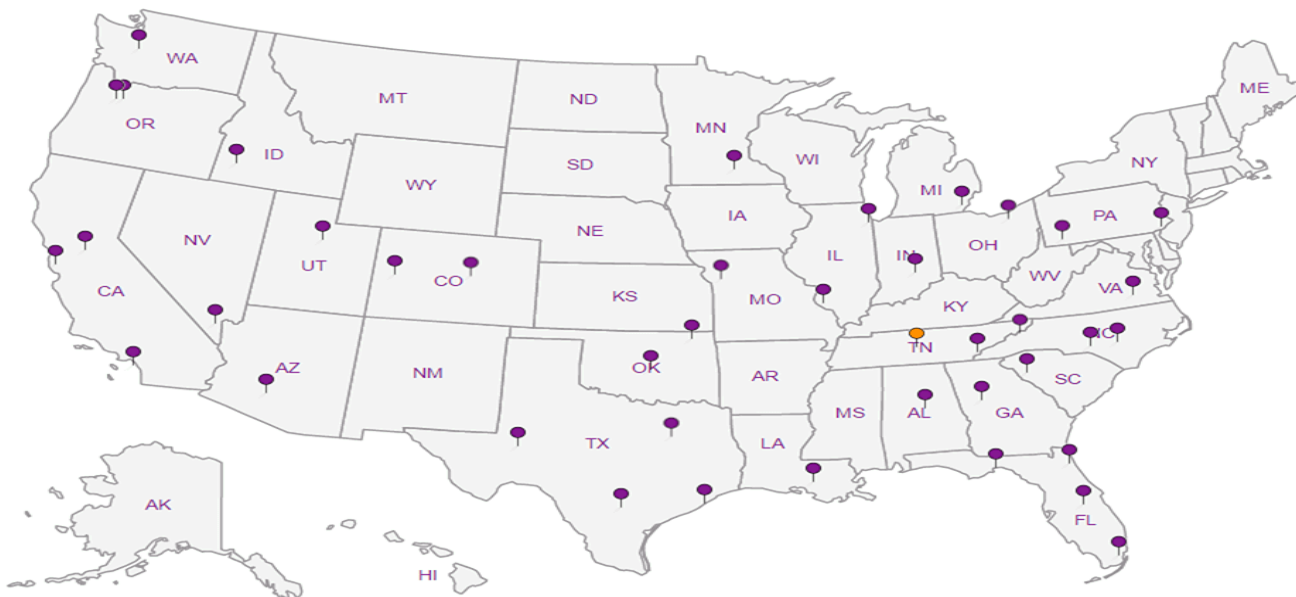
Third Party Federal Accreditations

A2LA – ISO 17025	1461.01	AIHA-LAP,LLC EMLAP	100789
A2LA – ISO 17025 ⁵	1461.02	DOD	1461.01
Canada	1461.01	USDA	P330-15-00234
EPA-Crypto	TN00003		

¹ Drinking Water ² Underground Storage Tanks ³ Aquatic Toxicity ⁴ Chemical/Microbiological ⁵ Mold ⁶ Wastewater n/a Accreditation not applicable

Our Locations

Pace National has sixty-four client support centers that provide sample pickup and/or the delivery of sampling supplies. If you would like assistance from one of our support offices, please contact our main office. Pace National performs all testing at our central laboratory.



1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc

Matt Turner
Environmental Engineer
Superior Refining Company LLC
2407 Stinson Avenue
Superior, Wisconsin 54800

Billing Information:
Please contact Matt Turner for billing information.

Report to:
Matt Turner

Email To:
matt.turner@huskyenergy.com

Project Description:
Tk 30 Soil Sampling

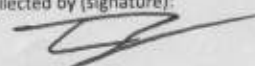
City/State Collected:
Superior, WI

Phone: **715-398-8434**
 Fax: **715-696-4873**

Client Project #
 Lab Project #

Collected by (print):
Tim Cyr

Site/Facility ID #
 P.O. #
8401302901

Collected by (signature):


Rush? (Lab MUST Be Notified)
 ___ Same Day ___ Five Day
 ___ Next Day ___ 5 Day (Rad Only)
 ___ Two Day ___ 10 Day (Rad Only)
 ___ Three Day

Date Results Needed

Immediately Packed on Ice: N ___ Y X

No. of Cntrs

Analysis / Container / Preservative

Chain of Custody Page ___ of ___

ESC
 L-A-B S-C-I-E-N-C-E-S
 YOUR LAB OF CHOICE

12065 Lebanon Rd
 Mount Juliet, TN 37122
 Phone: 615-758-5858
 Phone: 800-757-5859
 Fax: 615-758-5859

L# **L1042936**
E013

Acctnum: **MUROILSWI**
 Template:
 Prelogin:
 TSR: **John Hawkins (341)**
 PB:
 Shipped Via:

Sample ID	Comp/Grab	Matrix *	Depth	Date	Time	No. of Cntrs	Pres Chk	Analysis / Container / Preservative	Remarks	Sample # (lab only)
S1	Grab	SS	6"	11/8/18	12:55	1	X			-01
S2	Grab	SS	6"	11/8/18	13:07	1	X			02
S3	Grab	SS	6"	11/8/18	13:15	1	X			03
S4	Grab	SS	6"	11/8/18	13:25	1	X			04

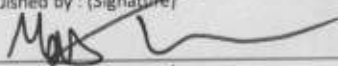
* Matrix:
 SS - Soil AIR - Air F - Filter
 GW - Groundwater B - Bioassay
 WW - WasteWater
 DW - Drinking Water
 OT - Other

Remarks: **RAD CORRECTED**

Samples returned via:
 ___ UPS ___ FedEx ___ Courier

Tracking # **7215 4522 1105**

pH ___ Temp ___
 Flow ___ Other ___

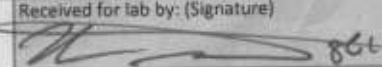
Relinquished by: (Signature)  Date: **11/8/18** Time: **13:48**

Received by: (Signature) Trip Blank Received: Yes No ___
 HCL / MeOH ___
 TBR ___

Relinquished by: (Signature) Date: ___ Time: ___

Received by: (Signature) Temp: **-0.1 °C** Bottles Received: **4**

Relinquished by: (Signature) Date: ___ Time: ___

Received for lab by: (Signature)  Date: **11/9/18** Time: **2:45**

Hold: ___ Condition: **NCR / OK**

Sample Receipt Checklist:
 COC Seal Present/Intact: Y ___ N ___
 COC Signed/Accurate: Y ___ N ___
 Bottles arrive intact: Y ___ N ___
 Correct bottles used: Y ___ N ___
 Sufficient volume sent: Y ___ N ___
 VOA Zero Headspace: ___ Y ___ N ___
 Preservation Correct/Checked: ___ Y ___ N ___

If preservation required by Login: Date/Time

Troy Dunlap



Login #: L1042936	Client: MUROI LSWI	Date: 11/9/18	Evaluated by: Jeremy
-------------------	--------------------	---------------	----------------------

Non-Conformance (check applicable items)

Sample Integrity	Chain of Custody Clarification	If Broken Container:
Parameter(s) past holding time <input checked="" type="checkbox"/>	Login Clarification Needed	
Improper temperature	Chain of custody is incomplete	Insufficient packing material around container
Improper container type	Please specify Metals requested.	Insufficient packing material inside cooler
Improper preservation	Please specify TCLP requested.	Improper handling by carrier (FedEx / UPS / Courier)
Insufficient sample volume.	Received additional samples not listed on coc.	Sample was frozen
Sample is biphasic.	Sample ids on containers do not match ids on coc	Container lid not intact
Vials received with headspace.	Trip Blank not received.	If no Chain of Custody:
Broken container	Client did not "X" analysis.	Received by:
Broken container:	Chain of Custody is missing	Date/Time:
Sufficient sample remains		Temp./Cont. Rec./pH:
		Carrier:
		Tracking#

Login Comments: Did not receive Containers for Total Solids. Wet-Weight ESC Key used.

Client informed by:	Call	Email	Voice Mail	Date:	Time:
TSR Initials: JGC	Client Contact:				

Login Instructions:

Report in wet weight.

December 14, 2018

Superior Refinery Husky Energy

Sample Delivery Group: L1051000

Samples Received: 12/07/2018

Project Number:

Description: TK30

Report To: Matt Turner

2407 Stinson Avenue

Superior, WI 54880

Entire Report Reviewed By:












John Hawkins

Project Manager

Results relate only to the items tested or calibrated and are reported as rounded values. This test report shall not be reproduced, except in full, without written approval of the laboratory. Where applicable, sampling conducted by Pace National is performed per guidance provided in laboratory standard operating procedures: 060302, 060303, and 060304.



Cp: Cover Page	1	
Tc: Table of Contents	2	
Ss: Sample Summary	3	
Cn: Case Narrative	4	
Sr: Sample Results	5	
S5 L1051000-01	5	
Qc: Quality Control Summary	6	
Volatile Organic Compounds (GC) by Method 8021B	6	
Volatile Organic Compounds (GC) by Method WI(95) GRO	8	
Gl: Glossary of Terms	9	
Al: Accreditations & Locations	10	
Sc: Sample Chain of Custody	11	

SAMPLE SUMMARY



S5 L1051000-01 Solid

Collected by: Justin Boucher
 Collected date/time: 12/06/18 13:20
 Received date/time: 12/07/18 09:00

Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Volatile Organic Compounds (GC) by Method 8021B	WG1209309	67	12/06/18 13:20	12/12/18 05:55	BMB
Volatile Organic Compounds (GC) by Method W(95) GRO	WG1210035	670	12/06/18 13:20	12/13/18 13:08	BMB

¹ Cp

² Tc

³ Ss

⁴ Cn

⁵ Sr

⁶ Qc

⁷ Gl

⁸ Al

⁹ Sc



All sample aliquots were received at the correct temperature, in the proper containers, with the appropriate preservatives, and within method specified holding times, unless qualified or notated within the report. Where applicable, all MDL (LOD) and RDL (LOQ) values reported for environmental samples have been corrected for the dilution factor used in the analysis. All Method and Batch Quality Control are within established criteria except where addressed in this case narrative, a non-conformance form or properly qualified within the sample results. By my digital signature below, I affirm to the best of my knowledge, all problems/anomalies observed by the laboratory as having the potential to affect the quality of the data have been identified by the laboratory, and no information or data have been knowingly withheld that would affect the quality of the data.

John Hawkins
Project Manager

- 1 Cp
- 2 Tc
- 3 Ss
- 4 Cn
- 5 Sr
- 6 Qc
- 7 Gl
- 8 Al
- 9 Sc



Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO

Analyte	Result	Qualifier	MDL	RDL	Dilution	Analysis	Batch
	mg/kg		mg/kg	mg/kg		date / time	
Benzene	3.09		0.00590	0.0196	67	12/12/2018 05:55	WG1209309
Toluene	12.5		0.0108	0.0360	67	12/12/2018 05:55	WG1209309
Ethylbenzene	2.88		0.00610	0.0203	67	12/12/2018 05:55	WG1209309
m&p-Xylene	13.0		0.0103	0.0344	67	12/12/2018 05:55	WG1209309
o-Xylene	4.87		0.00643	0.0214	67	12/12/2018 05:55	WG1209309
Methyl tert-butyl ether	0.0961		0.0107	0.0357	67	12/12/2018 05:55	WG1209309
Naphthalene	U	<u>J3 J5</u>	0.0697	0.232	67	12/12/2018 05:55	WG1209309
1,3,5-Trimethylbenzene	2.40		0.00549	0.0183	67	12/12/2018 05:55	WG1209309
1,2,4-Trimethylbenzene	6.13	<u>J5</u>	0.00717	0.0239	67	12/12/2018 05:55	WG1209309
TPH (GC/FID) Low Fraction	383		7.37	24.6	670	12/13/2018 13:08	WG1210035
<i>(S) a,a,a-Trifluorotoluene(PID)</i>	95.4			80.0-200		12/12/2018 05:55	WG1209309
<i>(S) a,a,a-Trifluorotoluene(PID)</i>	97.0			80.0-200		12/13/2018 13:08	WG1210035

Sample Narrative:

L1051000-01 WG1209309: Peaks/Baseline rise detected outside GRO window

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc

Method Blank (MB)

(MB) R3367703-3 12/11/18 11:46

Analyte	MB Result mg/kg	MB Qualifier	MB MDL mg/kg	MB RDL mg/kg
Benzene	0.000192	↓	0.0000880	0.000293
Toluene	U		0.000161	0.000537
Ethylbenzene	0.000111	↓	0.0000910	0.000303
m&p-Xylene	0.000154	↓	0.000154	0.000513
o-Xylene	U		0.0000960	0.000320
Methyl tert-butyl ether	U		0.000160	0.000533
Naphthalene	U		0.00104	0.00347
1,3,5-Trimethylbenzene	0.0000878	↓	0.0000820	0.000273
1,2,4-Trimethylbenzene	U		0.000107	0.000357
^(S) a,a,a-Trifluorotoluene(PID)	99.6		80.0-200	

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 GI

8 AI

9 SC

Laboratory Control Sample (LCS) • Laboratory Control Sample Duplicate (LCSD)

(LCS) R3367703-1 12/11/18 10:13 • (LCSD) R3367703-8 12/12/18 07:34

Analyte	Spike Amount mg/kg	LCS Result mg/kg	LCSD Result mg/kg	LCS Rec. %	LCSD Rec. %	Rec. Limits %	LCS Qualifier	LCSD Qualifier	RPD %	RPD Limits %
Benzene	0.0500	0.0487	0.0483	97.5	96.6	80.0-120			0.852	20
Toluene	0.0500	0.0521	0.0521	104	104	80.0-120			0.0482	20
Ethylbenzene	0.0500	0.0506	0.0505	101	101	80.0-120			0.161	20
m&p-Xylene	0.100	0.104	0.105	104	105	80.0-120			0.655	20
o-Xylene	0.0500	0.0506	0.0510	101	102	80.0-120			0.792	20
Methyl tert-butyl ether	0.0500	0.0471	0.0473	94.1	94.5	80.0-120			0.444	20
Naphthalene	0.0500	0.0507	0.0487	101	97.3	80.0-120			4.06	20
1,3,5-Trimethylbenzene	0.0500	0.0501	0.0490	100	98.1	80.0-120			2.07	20
1,2,4-Trimethylbenzene	0.0500	0.0506	0.0494	101	98.7	80.0-120			2.46	20
^(S) a,a,a-Trifluorotoluene(PID)				99.8	96.9	80.0-200				

L1051000-01 Original Sample (OS) • Matrix Spike (MS) • Matrix Spike Duplicate (MSD)

(OS) L1051000-01 12/12/18 05:55 • (MS) R3367703-4 12/12/18 06:19 • (MSD) R3367703-6 12/12/18 06:44

Analyte	Spike Amount mg/kg	Original Result mg/kg	MS Result mg/kg	MSD Result mg/kg	Dilution	Rec. Limits %	MSD Rec. %	MSD Rec. %	Dilution	Rec. Limits %	MS Qualifier	MSD Qualifier	RPD %	RPD Limits %
Benzene	0.0500	3.09	5.30	6.16	67	32.0-137	91.7	66.0	67	32.0-137	15.0	15.0	39	39
Toluene	0.0500	12.5	14.7	16.5	67	20.0-142	119	67.3	67	20.0-142	11.2	11.2	42	42
Ethylbenzene	0.0500	2.88	5.71	6.75	67	10.0-150	115	84.4	67	10.0-150	16.6	16.6	44	44
m&p-Xylene	0.100	13.0	17.9	20.3	67	14.0-141	110	73.7	67	14.0-141	12.8	12.8	44	44
o-Xylene	0.0500	4.87	7.66	8.88	67	10.0-157	120	83.4	67	10.0-157	14.7	14.7	44	44
Methyl tert-butyl ether	0.0500	0.0961	2.35	3.05	67	24.0-151	88.2	67.4	67	24.0-151	25.7	25.7	37	37



L1051000-01 Original Sample (OS) • Matrix Spike (MS) • Matrix Spike Duplicate (MSD)

(OS) L1051000-01 12/12/18 05:55 • (MS) R3367703-4 12/12/18 06:19 • (MSD) R3367703-6 12/12/18 06:44

Analyte	Spike Amount mg/kg	Original Result mg/kg	MS Result mg/kg	MSD Result mg/kg	MS Rec. %	MSD Rec. %	Dilution	Rec. Limits %	MS Qualifier	MSD Qualifier	RPD %	RPD Limits %
Naphthalene	0.0500	U	3.19	4.16	95.2	124	67	80.0-120		J3 J5	26.5	20
1,3,5-Trimethylbenzene	0.0500	2.40	5.41	6.40	89.8	119	67	80.0-120			16.8	20
1,2,4-Trimethylbenzene	0.0500	6.13	9.01	10.3	85.8	124	67	80.0-120		J5	13.4	20
^(S) a,a,a-Trifluorotoluene(PID)					94.5	93.8		80.0-200				

Sample Narrative:

OS: Peaks/Baseline rise detected outside GRO window

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 GI

8 AI

9 Sc



Method Blank (MB)

(MB) R3368098-3 12/13/18 11:57

Analyte	MB Result mg/kg	MB Qualifier	MB MDL mg/kg	MB RDL mg/kg
TPH (GC/FID) Low Fraction	U	0.0110	0.0110	0.0367
^(S) <i>a,a,o</i> -Trifluorotoluene(PID)	98.5			80.0-200

Laboratory Control Sample (LCS) • Laboratory Control Sample Duplicate (LCSD)

(LCS) R3368098-2 12/13/18 11:08 • (LCSD) R3368098-5 12/13/18 18:39

Analyte	Spike Amount mg/kg	LCS Result mg/kg	LCSD Result mg/kg	LCS Rec. %	LCSD Rec. %	Rec. Limits %	LCS Qualifier	LCSD Qualifier	RPD %	RPD Limits %
TPH (GC/FID) Low Fraction	0.550	0.525	0.548	95.4	99.6	80.0-120			4.27	20
^(S) <i>a,a,o</i> -Trifluorotoluene(PID)				98.9	97.6	80.0-200				

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 GI

8 AI

9 Sc



Guide to Reading and Understanding Your Laboratory Report

The information below is designed to better explain the various terms used in your report of analytical results from the Laboratory. This is not intended as a comprehensive explanation, and if you have additional questions please contact your project representative.

Abbreviations and Definitions

MDL	Method Detection Limit.
RDL	Reported Detection Limit.
Rec.	Recovery.
RPD	Relative Percent Difference.
SDG	Sample Delivery Group.
(S)	Surrogate (Surrogate Standard) - Analytes added to every blank, sample, Laboratory Control Sample/Duplicate and Matrix Spike/Duplicate; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in all environmental media.
U	Not detected at the Reporting Limit (or MDL where applicable).
Analyte	The name of the particular compound or analysis performed. Some Analyses and Methods will have multiple analytes reported.
Dilution	If the sample matrix contains an interfering material, the sample preparation volume or weight values differ from the standard, or if concentrations of analytes in the sample are higher than the highest limit of concentration that the laboratory can accurately report, the sample may be diluted for analysis. If a value different than 1 is used in this field, the result reported has already been corrected for this factor.
Limits	These are the target % recovery ranges or % difference value that the laboratory has historically determined as normal for the method and analyte being reported. Successful QC Sample analysis will target all analytes recovered or duplicated within these ranges.
Original Sample	The non-spiked sample in the prep batch used to determine the Relative Percent Difference (RPD) from a quality control sample. The Original Sample may not be included within the reported SDG.
Qualifier	This column provides a letter and/or number designation that corresponds to additional information concerning the result reported. If a Qualifier is present, a definition per Qualifier is provided within the Glossary and Definitions page and potentially a discussion of possible implications of the Qualifier in the Case Narrative if applicable.
Result	The actual analytical final result (corrected for any sample specific characteristics) reported for your sample. If there was no measurable result returned for a specific analyte, the result in this column may state "ND" (Not Detected) or "BDL" (Below Detectable Levels). The information in the results column should always be accompanied by either an MDL (Method Detection Limit) or RDL (Reporting Detection Limit) that defines the lowest value that the laboratory could detect or report for this analyte.
Uncertainty (Radiochemistry)	Confidence level of 2 sigma.
Case Narrative (Cn)	A brief discussion about the included sample results, including a discussion of any non-conformances to protocol observed either at sample receipt by the laboratory from the field or during the analytical process. If present, there will be a section in the Case Narrative to discuss the meaning of any data qualifiers used in the report.
Quality Control Summary (Qc)	This section of the report includes the results of the laboratory quality control analyses required by procedure or analytical methods to assist in evaluating the validity of the results reported for your samples. These analyses are not being performed on your samples typically, but on laboratory generated material.
Sample Chain of Custody (Sc)	This is the document created in the field when your samples were initially collected. This is used to verify the time and date of collection, the person collecting the samples, and the analyses that the laboratory is requested to perform. This chain of custody also documents all persons (excluding commercial shippers) that have had control or possession of the samples from the time of collection until delivery to the laboratory for analysis.
Sample Results (Sr)	This section of your report will provide the results of all testing performed on your samples. These results are provided by sample ID and are separated by the analyses performed on each sample. The header line of each analysis section for each sample will provide the name and method number for the analysis reported.
Sample Summary (Ss)	This section of the Analytical Report defines the specific analyses performed for each sample ID, including the dates and times of preparation and/or analysis.

Qualifier Description

J	The identification of the analyte is acceptable; the reported value is an estimate.
J3	The associated batch QC was outside the established quality control range for precision.
J5	The sample matrix interfered with the ability to make any accurate determination; spike value is high.

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc



Pace National is the only environmental laboratory accredited/certified to support your work nationwide from one location. One phone call, one point of contact, one laboratory. No other lab is as accessible or prepared to handle your needs throughout the country. Our capacity and capability from our single location laboratory is comparable to the collective totals of the network laboratories in our industry. The most significant benefit to our one location design is the design of our laboratory campus. The model is conducive to accelerated productivity, decreasing turn-around time, and preventing cross contamination, thus protecting sample integrity. Our focus on premium quality and prompt service allows us to be YOUR LAB OF CHOICE.

* Not all certifications held by the laboratory are applicable to the results reported in the attached report.

* Accreditation is only applicable to the test methods specified on each scope of accreditation held by Pace National.

State Accreditations

Alabama	40660	Nebraska	NE-OS-15-05
Alaska	17-026	Nevada	TN-03-2002-34
Arizona	AZ0612	New Hampshire	2975
Arkansas	88-0469	New Jersey-NELAP	TN002
California	2932	New Mexico ¹	n/a
Colorado	TN00003	New York	11742
Connecticut	PH-0197	North Carolina	Env375
Florida	E87487	North Carolina ¹	DW21704
Georgia	NELAP	North Carolina ³	41
Georgia ¹	923	North Dakota	R-140
Idaho	TN00003	Ohio-VAP	CL0069
Illinois	200008	Oklahoma	9915
Indiana	C-TN-01	Oregon	TN200002
Iowa	364	Pennsylvania	68-02979
Kansas	E-10277	Rhode Island	LA000356
Kentucky ^{1,6}	90010	South Carolina	84004
Kentucky ²	16	South Dakota	n/a
Louisiana	AI30792	Tennessee ^{1,4}	2006
Louisiana ¹	LA180010	Texas	T 104704245-17-14
Maine	TN0002	Texas ⁵	LAB0152
Maryland	324	Utah	TN00003
Massachusetts	M-TN003	Vermont	VT2006
Michigan	9958	Virginia	460132
Minnesota	047-999-395	Washington	C847
Mississippi	TN00003	West Virginia	233
Missouri	340	Wisconsin	9980939910
Montana	CERT0086	Wyoming	A2LA

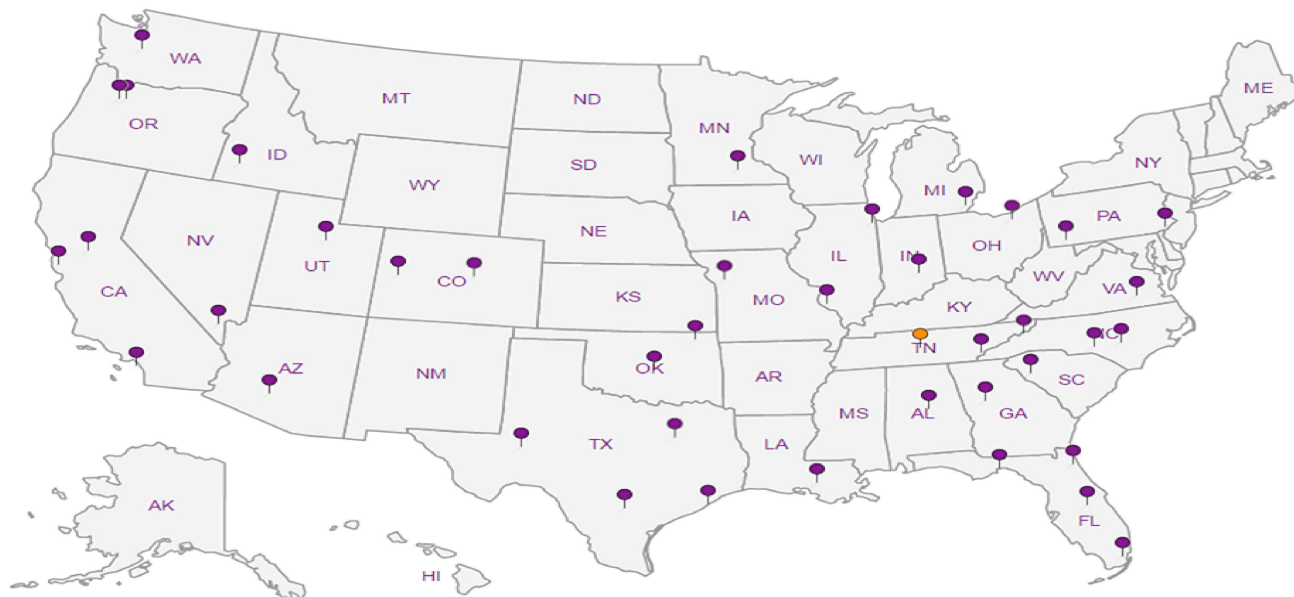
Third Party Federal Accreditations

A2LA – ISO 17025	1461.01	AIHA-LAP,LLC EMLAP	100789
A2LA – ISO 17025 ⁵	1461.02	DOD	1461.01
Canada	1461.01	USDA	P330-15-00234
EPA-Crypto	TN00003		

¹ Drinking Water ² Underground Storage Tanks ³ Aquatic Toxicity ⁴ Chemical/Microbiological ⁵ Mold ⁶ Wastewater n/a Accreditation not applicable

Our Locations

Pace National has sixty-four client support centers that provide sample pickup and/or the delivery of sampling supplies. If you would like assistance from one of our support offices, please contact our main office. Pace National performs all testing at our central laboratory.



Matt Turner

**Environmental Engineer
Superior Refining Company LLC
2407 Stinson Avenue
Superior, Wisconsin 54800**

Report to:

Matt Turner

Project:

Tk-25-Historical-Contamination Tk 30

Description:

City/State Collected: **Superior, WI**

Phone: **715-398-8434**

Client Project #

Lab Project #

Fax: **715-696-4873**

Collected by (print): **Justin Boucher**

P.O. #

Collected by (signature): *J. Boucher*

Quote #

Rush? (Lab MUST Be Notified)
 Same Day Five Day
 Next Day 5 Day (Rad Only)
 Two Day 10 Day (Rad Only)
 Three Day

Immediately Packed on Ice N ___ Y X

Sample ID

SS

Comp/Grab

GRAB SS

Depth

12"

Date

12-6-18

Time

1320

No. of Cntrs

1

Analysis / Container / Preservative

X PVC + Nephthale

Account: **MUROILSWI**

Template:

Prelogin:

TSR: **John Hawkins (341)**

PB:

Shipped Via:

Remarks:

Sample # (lab only) *101*

Chain of Custody Page *1* of *1*



12065 Lebanon Rd
 Mount Juliet, TN 37122
 Phone: 615-758-5058
 Phone: 800-767-5859
 Fax: 615-758-5859



Account: **MUROILSWI**

Template:

Prelogin:

TSR: **John Hawkins (341)**

PB:

Shipped Via:

Remarks:

Sample # (lab only) *101*

Sample Receipt Checklist
 CQC Seal Present/Intact: ___ M ___ P ___
 CQC Signed/Accurate: ___ Y ___ N ___
 Bottles arrive intact: ___ Y ___ N ___
 Correct bottles used: ___ Y ___ N ___
 Sufficient volume sent: ___ Y ___ N ___
 VOA Zero Headspace: ___ Y ___ N ___
 Preservation Correct/Checked: ___ Y ___ N ___
RAD SCREEN: <0.5 mR/hr

If preservation required by LogIn: Date/Time

Hold: Condition: **NCF / OK**

Billing Information:

Please contact **Matt Turner** for billing information.

Email To:

matt.turner@huskyenergy.com

City/State Collected: **Superior, WI**

Lab Project #

Quote #

Rush? (Lab MUST Be Notified)

Same Day ___ Five Day ___
 Next Day ___ 5 Day (Rad Only) ___
 Two Day ___ 10 Day (Rad Only) ___
 Three Day ___

Sample ID

SS

Comp/Grab

GRAB SS

Depth

12"

Date

12-6-18

Time

1320

No. of Cntrs

1

Remarks:

- * Matrix: **SS** - Soil **AIR** - Air **F** - Filter
- GW** - Groundwater **B** - Bioassay
- WW** - Wastewater
- DW** - Drinking Water
- OT** - Other

Samples returned via:

FedEx ___ Courier

Relinquished by: (Signature) *[Signature]*

Date: **12/6/18** Time: **15:24**

Relinquished by: (Signature) *[Signature]*

Date: ___ Time: ___

Relinquished by: (Signature)

Date: ___ Time: ___

Tracking # **7215 4522 1114**

Received by: (Signature)

Received by: (Signature)

Received for lab by: (Signature) *dam*

pH ___ Temp ___
 Flow ___ Other ___

Trip Blank Received: Yes / No HCL / MeOH TB#

Bottles Received: *Temp: -0.4 °C 0.50 / hr*

Date: **12/1/18** Time: **900**

If preservation required by LogIn: Date/Time