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April 16, 2019
File #34265.003

Mr. John Sager
Wisconsin Department of Natural Resources
1701 North 4th Street
Superior, WI 54880

Re: Tank 30 Basin Water Analytical Results
Superior Refining Company LLC, Superior, WI
WDNR BRRTS#s 02-16-557992 and 02-16-583029
Facility ID: 816009590

Dear John:

On behalf of Superior Refining Company LLC (SRC), Gannett Fleming, Inc. (GF) is submitting a water analytical results report pertaining to the January 2011 (BRRTS# 02-16-557992) and February 2018 (BRRTS# 02-16-583029) Tank 30 basin release sites at the SRC refinery in Superior.

Periodic reporting of site remediation progress to the Wisconsin Department of Natural Resources (WDNR) is required pursuant to ss. NR 700.11(1) and 724.13(3), Wisconsin Administrative Code. A completed certification page for the report is also attached.

Pertinent Site Background

The Tank 30 basin is in the SW $\frac{1}{4}$ of the SW $\frac{1}{4}$ of Section 25, Township 49 North, Range 14 West, Superior Township of Douglas County. Tank 30 shares the basin with Tank 29. Approximately 6-foot-tall earthen berms surround Tanks 29 and 30, with an approximately 3-foot-tall earthen berm running between the two tanks.

The land surrounding the basin is also owned by SRC and is part of the refinery. The closest surface water is Newton Creek, located about 2,000 feet east. The Tank 30 basin is located on relatively level land in the northcentral area of the refinery. The basin's ground surface is unpaved and slopes slightly to the southeast. The basin is underlain by native clay.

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Depending on time of year and topography, the depth to “groundwater” in the basin is estimated to range from slightly above ground surface at the dike drain pool in the southeast corner to approximately 3 feet below ground surface (ft bgs). The general direction of shallow groundwater flow below the refinery is to the east.

The hydraulic conductivity of the native clay underlying the refinery is on the order of 10^{-7} centimeters per second. Assuming a horizontal hydraulic gradient of 0.003 and effective porosity of 0.06, the estimated horizontal groundwater flow velocity is approximately 0.01 foot per year (ft/yr). The red-brown lean clay till is relatively homogenous and extends to approximately 100 ft bgs beneath the site.

Drainage from the basin is managed according to the facility’s spill prevention, control, and countermeasure plan. Typically:

- The tank dike drain valve in the southeast corner of the basin (see Figure 1) is closed.
- Stormwater that flows through the area is inspected visually prior to being discharged into the adjacent tank dike system and ultimately through stormwater outfall 003.

On October 1, 2011, Calumet Superior LLC (Calumet) acquired the refinery from Murphy Oil (Murphy). In May 2014, the WDNR approved Calumet’s April 2014 *Site Investigation and Remedial Action Plan* (SI/RAP) for the refinery. Effective November 8, 2017, Husky Superior Refining Holding Corp (Husky Superior) purchased Calumet and changed its legal name to Superior Refining Company LLC. Effective April 4, 2018, the April 2014 SI/RAP became a component of the March 2018 Negotiated Agreement between SRC and the WDNR.

In conjunction with the SI/RAP, a network of 23 wells and 8 piezometers for monitoring overall groundwater quality was established, as shown on Figure 2. Twice a year, starting in 2015, all wells and piezometers in the network are gauged (to check for free product, track seasonal changes in water levels, and prepare groundwater contour maps), and the perimeter wells and all piezometers are purged and sampled.

Most samples are routinely analyzed for petroleum volatile organic compounds (PVOCs) and naphthalene. However, perimeter monitoring wells MW-1, MW-2, MW-3D, MW-8R, and MW-9B are also classified as “pond” wells. In the fall, samples from these wells are analyzed for volatile organic compounds (VOCs) and select inorganics, in conjunction with wastewater

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treatment Ponds 1 and 6 post-closure monitoring. As stipulated by the WDNR Groundwater and Environmental Monitoring System (GEMS) program, MW-8R serves as an upgradient monitoring well, located approximately 2,500 feet southwest of the ponds.

As part of the Negotiated Agreement, a single, new, refinery-wide Environmental Repair Program (ERP) site (BRRTS# 16-16-559511) was created at the refinery, and this site is referred to as a facility-wide ERP. Analytical results document that PVOC/naphthalene analytical concentrations were all non-detect in the ERP perimeter wells and all piezometers for 2015-2018, except the toluene concentration in the sample collected from MW-7 on October 9, 2018, was 1.9 micrograms per liter ($\mu\text{g}/\ell$). This is nearly two orders of magnitude below toluene's NR 140 preventative action limit (PAL) of 160 $\mu\text{g}/\ell$. In addition, all other analytical results were non-detect for PVOCs and naphthalene, and the detection limits for PVOCs and naphthalene were all below their respective PALs.

Note that there was an explosion and fire at the refinery in April 2018. During response activities, the 4-inch-diameter steel pipe that serves as a protective cover for MW-7 (constructed of 2-inch-diameter, Schedule 40 PVC) was bent. Consequently:

- Field staff used a peristaltic pump and disposable tubing to purge and sample MW-7 in 2018 instead of a disposable bailer.
- The well's PVC riser pipe could be damaged at or near the ground surface. Any crack could jeopardize the integrity of MW-7 and allow impacted surface water to seep into the well.
- As a precautionary measure, SRC plans to abandon the well and install MW-7R to replace MW-7 in 2019.

See GF's December 2018 *Facility-Wide ERP Groundwater Monitoring Report for 2018* on file with the WDNR for details.

January 2011 Release, Response, and Water Sample Results Summary

During routine inspections, refinery staff observed ponding of product on ice/frozen ground in the basin southeast of Tank 30, and the release was reported to the WDNR on January 13, 2011. Approximately 2,000 gallons of naphtha were released from aboveground piping located about 40 feet southeast of Tank 30, because ice accumulation caused a drain valve to break off.

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The degree and extent of soil contamination was limited because the release occurred in January on ice/frozen ground, which caused the product to pond on the surface in a topographically low area and allowed field personnel time to vacuum up most of the product immediately after the release occurred. Residual soil contamination extended from the source location to the contiguous low area in the southeast corner of the tank basin where naphtha-impacted melt water accumulated prior to being removed using vacuum trucks between February 18 and March 31, 2011.

Investigation work completed at the site includes surface water sampling in April 2011, soil sampling in May 2011, and soil and groundwater sampling in April/May 2012. Lab results confirm that the concentrations of VOCs in the soil within four of the ground surfaces were below generic NR 720 industrial direct contact residual concentration levels (RCLs) as summarized in Attachment A. See GF's February 2012 *Site Investigation Work Plan* and March 2014 *Remediation Progress Report* on file with the WDNR for details.

Table 1 includes a summary of the water analytical results. As presented in Table 1:

- On 04/11/11, a surface water sample was collected from the dike drain pool in the southeast corner of the tank basin and analyzed for gasoline range organics (GRO), total petroleum hydrocarbons (TPH), PVOCs, and naphthalene. Four of the PVOCs of common concern include benzene, toluene, ethylbenzene, and xylenes (BTEX). The other PVOCs include the two trimethylbenzenes (TMBs) and methyl tert butyl ether (MTBE).
- On 04/24/12, a groundwater sample was collected at approximately 3 ft bgs from the SS-8 Geoprobe borehole and analyzed for VOCs.

Figure 1 shows both water sample locations for reference. Copies of the laboratory reports and chain-of-custody records for all analyzed samples were submitted to the WDNR previously.

February 2018 Release and Response Summary

During routine inspections, refinery staff observed ponding of product on ice/frozen ground in the basin southeast of Tank 30, and the release was reported to the WDNR on February 27, 2018. Estimates indicate approximately 475 gallons of naphtha were released from aboveground piping located about 20 feet southeast of Tank 30 due to a gasket seal failure in the bonnet of a gate valve caused by freeze/thaw action.

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The degree and extent of soil contamination was limited because the release occurred in February on ice/frozen ground, which caused the product to pond on the surface in a topographically low area and allowed field personnel time to vacuum up most of the product immediately after the release occurred. Residual soil contamination extended from the source location to the contiguous low area in the southeast corner of the tank basin where naphtha-impacted melt water in the spring and stormwater in the summer and fall accumulated prior to being removed using vacuum trucks between February 27 and October 10, 2018. The fluids collected by the trucks were routed to an on-site API separator for recovery of any free product and then the remainder of the fluid was directed to the refinery's wastewater treatment plant. The normal discharge of stormwater resumed on October 11, 2018, as approved by the WDNR.

Investigation work completed at the site includes soil sampling in November/December 2018, when water levels in the tank dike were low enough for access. Approximately 1 cubic yard of benzene-impacted soil was excavated to address direct contact risk. Lab results confirm that the concentrations of PVOCs and naphthalene in the residual soil within four of the ground surfaces were below generic NR 720 industrial direct contact RCLs as summarized in Attachment B. See SRC's January 2019 *Naphtha Release SERTS ID 20180227NO16-1* report on file with the WDNR for details.

Dates and Descriptions of Tank 30 Basin Water Quality Sampling in 2018

Precipitation was above normal in 2018. Consequently, there was standing water or ice in the southeast corner of the Tank 30 basin all year. Following the completion of SRC's immediate response action to the February 2018 naphtha release outlined in the previous section, samples were periodically collected and analyzed to track the improvement in Tank 30 basin water quality over time. All surface water samples were collected from the dike drain pool in the southeast corner of the tank basin, as shown on Figure 1. As summarized in Table 1:

- On 03/14/18, a pair of samples was collected and analyzed for TPH, PVOCs, and naphthalene.
- On 08/29/18, one sample was collected and analyzed for TPH, PVOCs, and naphthalene.
- On 10/11/18, one sample was collected and analyzed for PVOCs and naphthalene.

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Field staff immediately transferred each sample collected into laboratory-supplied vials pre-filled with the appropriate volume of hydrochloric acid preservative. The sample vials were labelled, placed on ice stored in a cooler, and shipped overnight for analysis to ESC Lab Sciences/Pace Analytical of Mount Juliet, TN, (Wisconsin laboratory certification #9980939910) in March and August 2018 and Pace Analytical of Minneapolis, MN, (Wisconsin laboratory certification #999107970) in October 2018. Attachment C includes copies of the laboratory reports and chain of custody records for the water samples collected in 2018.

Tank 30 Basin Water Quality Findings and Proposed Path Forward

As presented above, analytical results from the samples collected in 2018 document the improvement in Tank 30 basin water quality over time. For example, Figure 3 presents trend analysis plots for BTEX concentrations. Best-fit exponential trend lines generated using Excel were included on the plots. As shown on Figure 3, dissolved-phase concentrations followed a downward trend.

Also, as summarized in Table 1, on 10/11/18:

- The benzene concentration was 0.33J micrograms per liter ($\mu\text{g}/\ell$), which is below its NR 140 preventative action limit (PAL) of 0.5 $\mu\text{g}\ell$.
- The toluene concentration was 0.48 $\mu\text{g}/\ell$, which is well below its NR 140 PAL of 160 $\mu\text{g}\ell$.
- All other analytical results were non-detect for PVOCs and naphthalene, and the detection limits for PVOCs and naphthalene were all below their respective PALs.

Based on the documented improvement in water quality and final concentrations, SRC plans to include both open sites in an upcoming ERP database submittal to the WDNR and request transfer of BRRTS #02-16-557992 and #02-16-583029 to BRRTS #16-16-559511.

At BRRTS #02-16-557992 and #02-16-583029:

- The soil and groundwater exposure pathways were adequately addressed because the immediate/interim action met the direct contact soil cleanup standards and the existing hydrogeology is being used as the interim action soil performance standard for the protection of groundwater. The vapor exposure pathway will be ruled out.

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- SRC will continue to utilize its network of 23 monitoring wells (MW-1, MW-1/T67, MW-2, MW-2/T66, MW-3/T50, MW-3D, MW-5/T40, MW-5/T70, MW-7/7R, MW-8R, MW-9B, and MW-11 through MW-22) and 8 piezometers (PZ-2/T66, PZ-3D, PZ-8R, PZ-11, PZ-13, PZ-16, PZ17, and PZ-21) for field verification of the performance standard for the protection of groundwater following transfer to BRRTS #16-16-559511.

Historical soil sampling results, etc. will be summarized in the ERP database submittal also, as you and I have discussed. Meanwhile, feel free to contact me or Matt Turner at Husky Superior if you have any questions or need additional information.

Sincerely,

GANNETT FLEMING, INC.




Clifford C. Wright, P.E., P.G.
Project Engineer

CCW/jec
Enc.

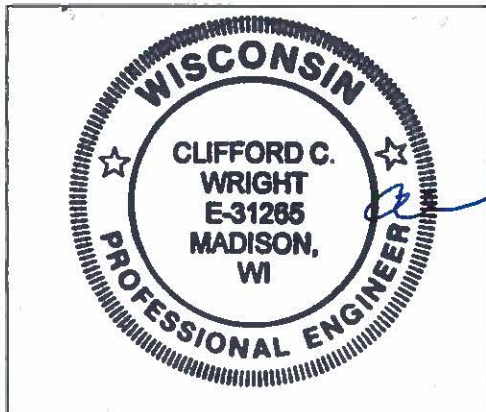
ecc: Matt Turner (Husky Superior)

ENGINEERING AND HYDROGEOLOGIST CERTIFICATIONS


I hereby certify that I am a registered professional engineer in the State of Wisconsin, registered in accordance with the requirements of ch. A-E 4, Wis. Adm. Code; that this document has been prepared in accordance with the rules of Professional Conduct in ch. A-E 8, Wis. Adm. Code; and that, to the best of my knowledge, all information contained in this document is correct and the document was prepared in compliance with all applicable requirements in chs. NR 700 to 726, Wis. Adm. Code.

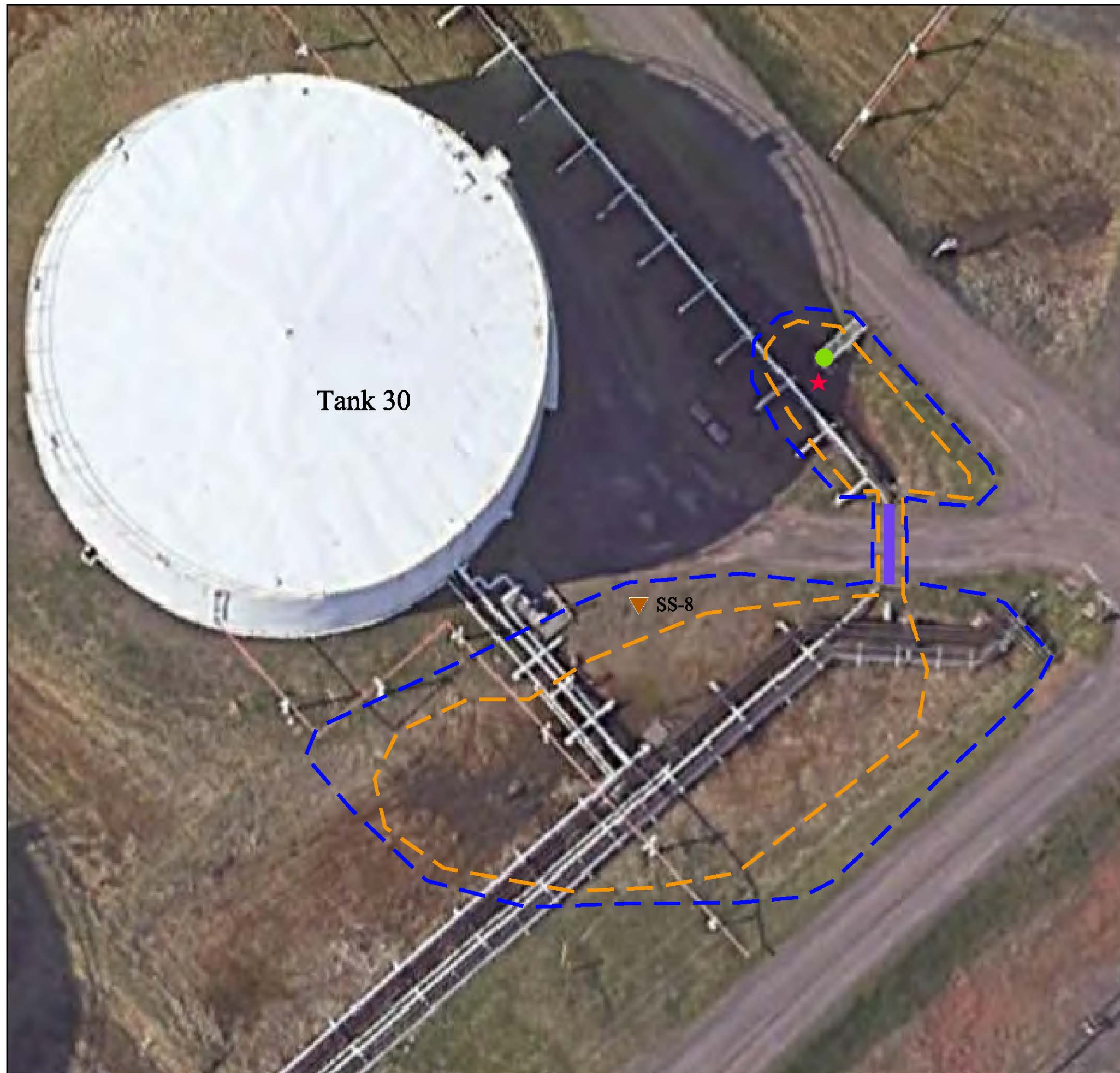
Print Name Clifford C. Wright	Title Project Engineer
Signature 	Date 4/16/2019

P.E. Seal for E-31265:



I hereby certify that I am a hydrogeologist as that term is defined in s. NR 712.03(1), Wis. Adm. Code, am registered in accordance with the requirements of ch. GHSS 2, Wis. Adm. Code, or licensed in accordance with the requirements of ch. GHSS 3, Wis. Adm. Code, and that, to the best of my knowledge, all information contained in this document is correct and the document was prepared in compliance with all applicable requirements in chs. NR 700 to 726, Wis. Adm. Code.

Print Name Clifford C. Wright	Title Project Geologist
Signature 	Date 4/16/2019

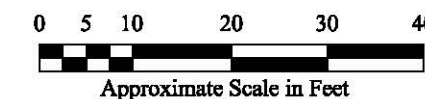


Legend

- ▼ Groundwater Sample Location (04/24/12)
- ★ Dike drain pool water samples (2011 & 2018)
- - Estimated extent of Jan 2011 release
- - Estimated extent of Feb 2018 release
- Dike drain valve
- Culvert

Notes:

- 1) BRRTS # 02-16-557992 Muphy Oil -Tank #30 site for the Jan 2011 release.
- 2) BRRTS # 02-16-583029 Superior Refining Spill 04-16-583035 site for the Feb 2018 release.
- 3) Parcel # 01-801-03339-0.



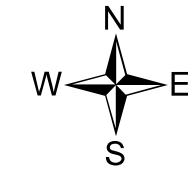
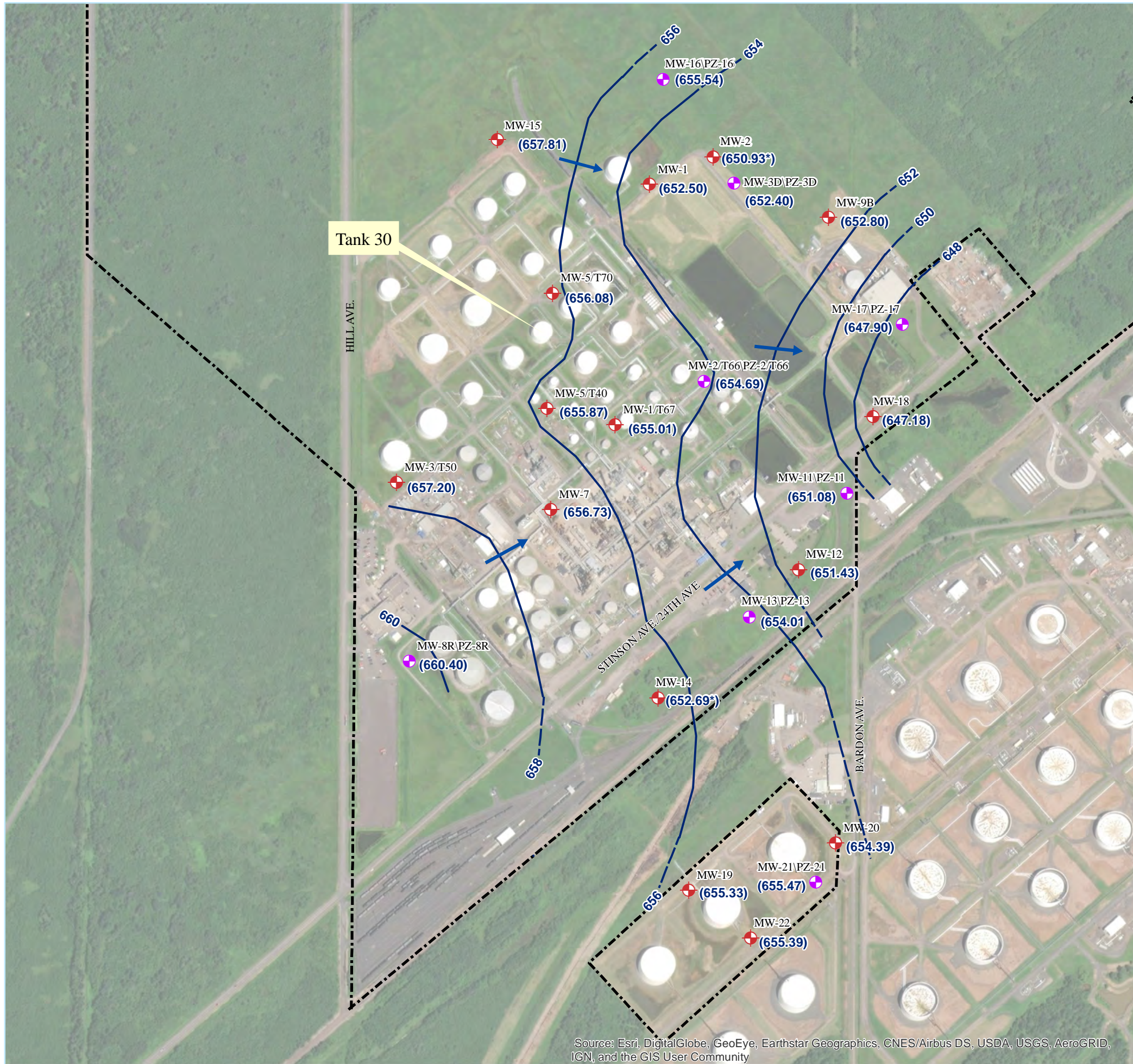
Tank 30 Basin Water Sample Map

SUPERIOR REFINING COMPANY LLC
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Project No.	34265.003	Date	03/18/19	Figure	1
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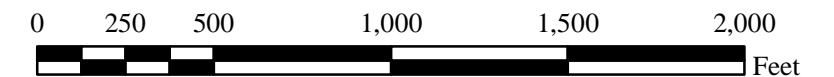


Legend

- 656** — Groundwater Contour (dashed where inferred)
- Groundwater Flow Direction
- (654.30)** Groundwater Elevation (ft MSL)
- ⊕ Monitoring Well
- ⊕ Monitoring Well\Piezometer Pair (groundwater elevation shown is for monitoring well)
- - - Approximate Facility Property Boundary

Notes:

1. The data from MW-2 and MW-14 were not used to develop the contour map.
2. Site datum = mean sea level (MSL).
3. Well\Piezometer locations based on 02/06/15 survey by TKDA using a Trimble GNSS RTK GPS R8 Model 3.



ERP Monitoring Well Network and Groundwater Flow Map (May 2018)

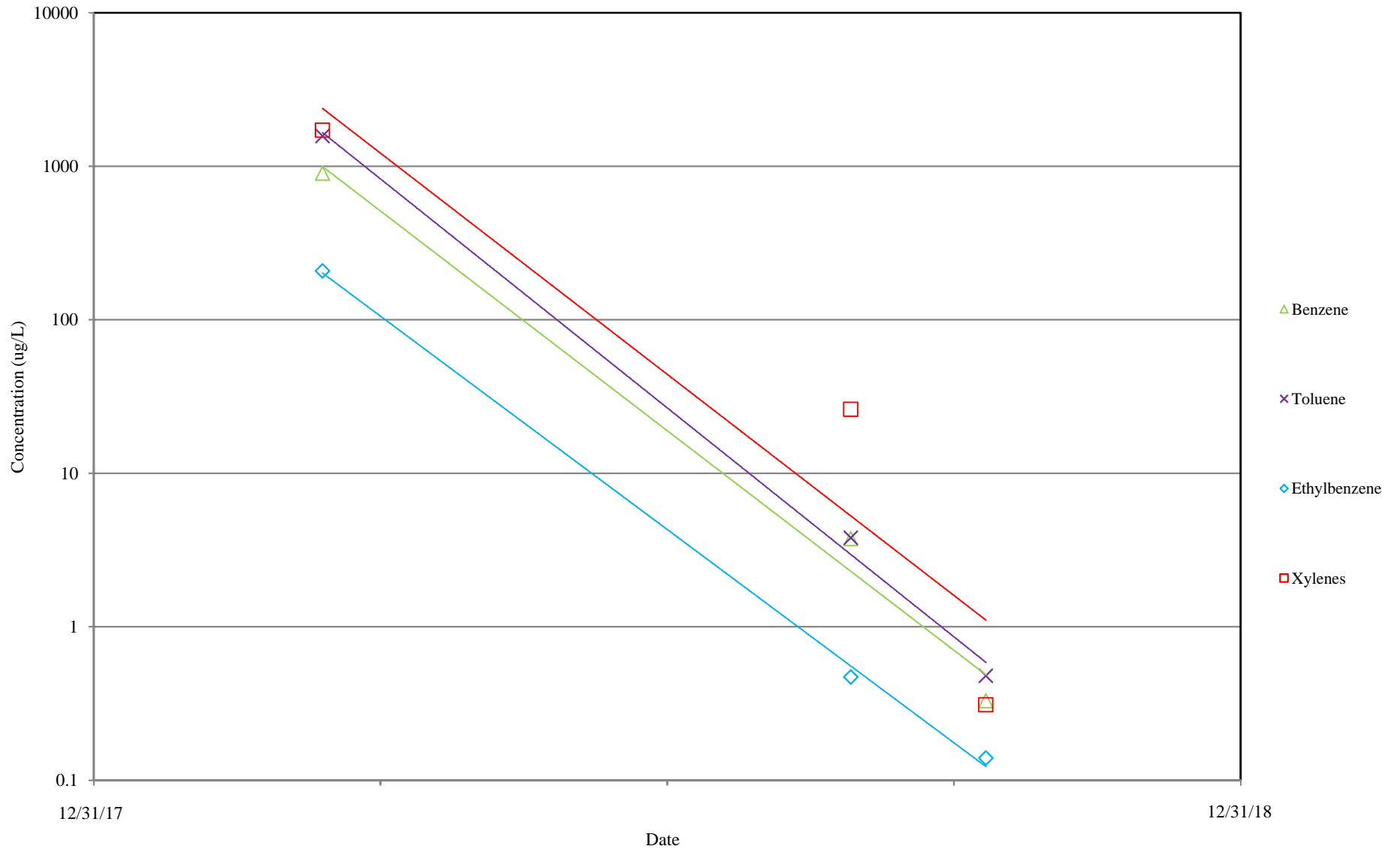
SUPERIOR REFINING COMPANY LLC REFINERY
SUPERIOR, WISCONSIN



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Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGRID, IGN, and the GIS User Community

FIGURE 3



Note: Best-fit exponential trend lines generated using Excel and non-detect concentrations plotted at detection limit.

BTEX WATER CONCENTRATIONS IN THE TANK 30 BASIN (2018)
SUPERIOR REFINING COMPANY LLC
SUPERIOR, WISCONSIN

SUPERIOR REFINING COMPANY LLC
SUPERIOR, WISCONSIN

TABLE 1

WATER ANALYTICAL RESULTS FOR GRO, TPH, DETECTED PVOCs/VOCs, AND NAPHTHALENE - TANK 30 RELEASE SITES

Sample		Substance and Concentration (µg/ℓ)													
Description	Date	GRO	TPH	Benzene RQ	Toluene RQ	Ethylbenzene RQ	Xylenes RQ	MTBE RQ	Naphthalene RQ	TMBs RQ	1,1,2,2- Tetrachloroethane	1,1,2-Trichloroethane	Bromomethane	Isopropylbenzene RQ	
NR 140 PAL		NS	NS	<i>0.5</i>	<i>160</i>	<i>140</i>	<i>400</i>	<i>12</i>	<i>10</i>	<i>96</i>	<i>0.02</i>	<i>0.5</i>	<i>1</i>	NS	
NR 140 ES		NS	NS	5	800	700	2,000	60	100	480	0.2	5	10	NS	
Surface water (SW)	04/11/11	350	650	<i>3.7</i>	5.3	0.57	10.8	<1.0	<5.0	11.6	na	na	na	na	
SS-8	04/24/12	na	na	5.73	2.20	0.87 J	5.67	<0.50	<1.00	2.03 J	<0.40	<0.40	<1.00	0.26 J	
SW and duplicate	03/14/18	na	11,355	901	1,574	<i>209</i>	<i>1,720</i>	25.8 U	<i>31.0</i> BJ	1,330	na	na	na	na	
SW	08/29/18	na	323	<i>3.75</i>	3.81	0.472	26.1	0.429 J	0.228 BJ	9.55 B	na	na	na	na	
SW	10/11/18	na	na	0.33 J	0.48	0.14 U	0.31 U	<0.16	<0.48	0.32 U	na	na	na	na	

NOTES:

- Results are in micrograms per liter (µg/ℓ), see Figure 1 for location of SS-8. All SW samples were collected at the dike drain pool in the southeast corner of the tank basin.
- Detected concentrations at or above an applicable NR 140 PAL are in red font and italicized; those at or above an NR 140 ES are in red font and bold.
- Duplicate sample results are averaged for statistical analysis/plotting, per December 2013 Interstate Technology & Regulatory Council guidance.
- The SS-8 sample was analyzed for VOCs; all other samples were analyzed for GRO/TPH/PVOCs and naphthalene, TPH/PVOCs and naphthalene, or PVOCs and naphthalene.
- B = The sample analyte was found in the associated blank.
- GRO = Gasoline range organics.
- J = Estimated concentration, concentration below the laboratory's level of quantitation.
- MTBE = Methyl tert butyl ether.
- na = Not analyzed.
- NR 140 ES = Wisconsin Administrative Code NR 140 Enforcement Standard.
- NR 140 PAL = Wisconsin Administrative Code NR 140 Preventive Action Limit.
- NS = No standard.
- RQ = Results qualifier.
- TMBs = Trimethylbenzenes.
- TPH = Total petroleum hydrocarbons.
- U = Compound not detected at or above the detection limit, which is the value shown for all parameters except xylenes and TMBs.

ATTACHMENT A

JANUARY 2011 RELEASE SITE SOIL SAMPLE RESULTS SUMMARY

SUPERIOR REFINING COMPANY LLC
SUPERIOR, WISCONSIN

TABLE A-1

SOIL ANALYTICAL RESULTS (TANK 30 BASIN, JANUARY 2011 RELEASE SITE)

Group/Substance/CCR-HI	Concentration (mg/kg)						NR 720 RCL (mg/kg)		
	Sample ID	SS-1	SS-2	SS-3	SS-4	SS-5	SS-6	Soil to Groundwater Pathway	Industrial Direct Contact
Sample Date	05/03/11	05/03/11	05/03/11	05/03/11	05/03/11	05/03/11	05/03/11		
Sample Depth (ft bgs)	0.5	0.5	0.5	0.5	0.5	0.5	0.5		
Saturated/Unsaturated ⁽¹⁾	Unsat.	Unsat.	Unsat.	Unsat.	Unsat.	Unsat.	Unsat.		
Gasoline range organics	28	47	20	32	7.6 J	23	NS	NS	
Diesel range organics	<0.77	1.4 J	<0.77	1.4 J	<0.77	<0.77	NS	NS	
Benzene	<i>0.37</i>	<i>0.15</i>	<i>0.13</i>	<i>1.2</i>	<i>0.054</i>	<i>0.070</i>	<i>0.0051</i>	7.07	
Ethylbenzene	0.16	0.24	0.22	0.74	0.12	0.19	<i>1.57</i>	35.4	
Toluene	0.12 J	0.20 J	0.076 J	<i>2.9</i>	0.078 J	0.12 J	<i>1.1072</i>	818	
Xylenes	0.71	1.28	0.73	3.0	0.35	0.66	<i>3.96</i>	260	
Methyl tert butyl ether	<0.0097	<0.0083	<0.011	<0.010	<0.0090	<0.0084	<i>0.027</i>	282	
1,2,4-TMB	0.42	1.0	0.33	0.54	0.11	0.38	NS	219	
1,3,5-TMB	0.20	0.55	0.19	0.26	0.06 J	0.21	NS	182	
TMBs (combined)	0.62	<i>1.55</i>	0.52	0.80	0.17 J	0.59	<i>1.3787</i>	NS	
Naphthalene	0.018 J	0.047 J	0.066 J	0.024 J	<0.012	0.026 J	<i>0.6582</i>	24.1	
Shallow Soil (Industrial) Multiple Contaminant Cumulative ⁽²⁾									
Cancer Risk (CCR)	5.8E-08	3.0E-08	2.7E-08	1.9E-07	1.1E-08	1.6E-08	NR 720 threshold = 1E-5		
Hazard Index (HI)	0.0011	0.0014	0.0007	0.0033	0.0003	0.0006	NR 720 threshold = 1		

NOTES:

Concentrations are in units of milligrams per kilogram (mg/kg) on a dry weight basis.
 No results exceed an applicable NR 720 industrial direct contact RCL, as shown in red font and bold.
 Detected concentrations at or above an applicable NR 720 soil to groundwater pathway RCL are in red font and italicized.
 NR 720 residual contaminant level (RCL) standards from WDNR's RR Program Soil RCL Excel workbook updated December 2018.
 Samples analyzed for gasoline range organics, diesel range organics, petroleum volatile organic compounds, and naphthalene.
 J = Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.
 NS = No standard.
 TMBs (combined) = Trimethylbenzenes (1,2,4- and 1,3,5- combined).

FOOTNOTES:

(1) Soil sample type based on the observed low water table in closest surveyed monitoring well, MW-5/T70 (2.34 ft bgs on 9/3/15).
 (2) Industrial multiple contaminant cumulative cancer risk (CCR) and hazard index (HI) levels, if applicable (for samples within 4 feet of ground surface and based on detected concentrations only). Thresholds are 1E-5 for CCR and 1 for HI per NR 720.12(1)(b). No CCR or HI levels at or above their respective threshold were calculated.

TABLE A-1

SOIL ANALYTICAL RESULTS (TANK 30 BASIN, JANUARY 2011 RELEASE SITE)

Group/Substance/CCR-HI	Concentration (mg/kg)						NR 720 RCL (mg/kg)		
	Sample ID	SS-7	SS-7	SS-8	SS-8	SS-9	SS-9	Soil to	Industrial
Sample Date	04/24/12	04/24/12	04/24/12	04/24/12	04/24/12	04/24/12	04/24/12	Groundwater	Direct
Sample Depth (ft bgs)	0-2	4-6	0-2	6-8	0-2	6-8	Pathway	Contact	
Saturated/Unsaturated ⁽¹⁾	Unsat.	Saturated	Unsat.	Saturated	Unsat.	Saturated			
Total organic carbon	40,000	2,600	na	na	2,000	1,300	NS	NS	
Gasoline range organics	20.1	274	<5.00	154	19.7	272	NS	NS	
Diesel range organics	<5.09	214	6.08	60.9	33.3	38.3	NS	NS	
Benzene	<0.0255	<0.0260	<0.0252	<0.0254	<0.0248	<0.0240	<i>0.0051</i>	7.07	
Ethylbenzene	<0.0262	<0.0267	<0.0259	<0.0261	<0.0255	<i>3.850</i>	<i>1.57</i>	35.4	
Toluene	<0.0290	<0.0296	<0.0287	<0.0289	<0.0282	<0.0273	<i>1.1072</i>	818	
Xylenes	<0.0843	<0.0860	<0.0833	<0.0838	<0.0819	3.663	<i>3.96</i>	260	
Methyl tert butyl ether	<0.0595	<0.0607	<0.0587	<0.0592	<0.0579	<0.0559	<i>0.027</i>	282	
cis-1,2-Dichloroethylene	<0.0290	<0.0296	<0.0287	0.0400	<0.0282	<0.0273	<i>0.0412</i>	2340	
1,2,4-TMB	<0.0255	1.730	<0.0252	1.790	0.0372	3.430	NS	219	
1,3,5-TMB	<0.0255	0.214	<0.0252	<0.0254	<0.0248	0.747	NS	182	
TMBs (combined)	<0.0510	<i>1.944</i>	<0.0504	<1.815	<0.0620	<i>4.177</i>	<i>1.3787</i>	NS	
Naphthalene	<0.0305	0.241	<0.0301	0.469	<0.0296	0.0873	<i>0.6582</i>	24.1	
Bromobenzene	<0.0248	<0.0253	<0.0245	0.0391	<0.0241	<0.0233	NS	679	
Bromodichloromethane	<0.0269	<i>0.0276</i>	<0.0266	<0.0268	<0.0262	<i>0.0637</i>	<i>0.0003</i>	1.83	
Bromomethane	<i>0.158</i>	<0.145	<i>0.147</i>	<0.141	<0.138	<0.133	<i>0.0051</i>	43	
tert-Butylbenzene	<0.0262	<0.0267	<0.0259	0.0454	<0.0255	<0.0246	NS	183	
Isopropylbenzene (Cumene)	<0.0255	0.0474	<0.0252	0.0396	<0.0248	0.768	NS	268	
Dibromochloromethane	<0.0234	<0.0238	<0.0231	<0.0232	<0.0227	<i>0.0637</i>	<i>0.032</i>	38.9	
4-Isopropyltoluene	<0.0248	0.185	<0.0245	<0.0246	<0.0241	<0.0233	NS	162	
Propylbenzene	<0.0255	0.112	<0.0252	0.134	<0.0248	<0.0240	NS	264	
1,1,1,2-Tetrachloroethane	<0.0255	<0.0260	<0.0252	0.0409	<0.0248	<0.0240	<i>0.0534</i>	12.3	
1,1,2,2-Tetrachloroethane	<i>0.164</i>	<0.0260	<0.0252	<0.0254	<0.0248	<0.0240	<i>0.0002</i>	3.6	
1,1,2-Trichloroethane	<0.029	<0.0296	<0.0287	<0.0289	<i>0.0545</i>	<0.0273	<i>0.0032</i>	7.01	
Shallow Soil (Industrial) Multiple Contaminant Cumulative ⁽²⁾									
Cancer Risk (CCR)	4.6E-08	2.5E-08	--	2.3E-08	7.8E-09	1.5E-07	NR 720 threshold = 1E-5		
Hazard Index (HI)	0.0037	0.0011	--	0.0014	0.006	0.0031	NR 720 threshold = 1		

NOTES:

Concentrations are in units of milligrams per kilogram (mg/kg) on a dry weight basis.

No results exceed an applicable NR 720 industrial direct contact RCL, as shown in red font and bold.

Detected concentrations at or above an applicable NR 720 soil to groundwater pathway RCL are in red font and italicized.

NR 720 residual contaminant level (RCL) standards from WDNR's RR Program Soil RCL Excel workbook updated December 2018.

Samples analyzed for volatile organic compounds (EPA Method 8260B); only detected compounds are summarized in table.

na = Not analyzed.

NS = No standard.

TMBs (combined) = Trimethylbenzenes (1,2,4- and 1,3,5- combined).

FOOTNOTES:

(1) Soil sample type based on the observed low water table in closest surveyed monitoring well, MW-5/T70 (2.34 ft bgs on 9/3/15).

(2) Industrial multiple contaminant cumulative cancer risk (CCR) and hazard index (HI) levels, if applicable. Thresholds are 1E-5 for CCR and 1 for HI per NR 720.12(1)(b). No CCR or HI levels at or above their respective threshold were calculated.

TABLE A-1

SOIL ANALYTICAL RESULTS (TANK 30 BASIN, JANUARY 2011 RELEASE SITE)

Group/Substance/CCR-HI	Concentration (mg/kg)							NR 720 RCL (mg/kg)		
	Sample ID	SS-10	SS-10B	SS-11	SS-12	SS-12B	SS-13	SS-14	Soil to	Industrial
Sample Date	04/25/12	05/17/12	04/25/12	04/25/12	05/17/12	04/25/12	04/25/12		Groundwater	Direct
Sample Depth (ft bgs)	0-2	0-2	0-2	0-2	0-2	0-2	0-2		Pathway	Contact
Saturated/Unsaturated ⁽¹⁾	Unsat.	Unsat.	Unsat.	Unsat.	Unsat.	Unsat.	Unsat.			
Gasoline range organics	<5.22	na	<5.32	<5.00	na	26.0	<5.18		NS	NS
Diesel range organics	<5.06	na	<4.91	<5.09	na	7.61	<5.00		NS	NS
Benzene	<i>0.0356</i>	<0.0250	<0.0296	<i>0.0299</i>	<0.0250	<0.0267	<0.0239		<i>0.0051</i>	7.07
Ethylbenzene	<0.0243	<0.0250	<0.0304	<0.0273	0.0371 J	<0.0274	<0.0246		<i>1.57</i>	35.4
Toluene	0.0379	0.0518 J	0.0654	<0.0303	0.119	<0.0304	<0.0273		<i>1.1072</i>	818
Xylenes	<0.0782	<0.0750	0.1038	<0.0879	0.1922 J	0.0924	<0.0791		<i>3.96</i>	260
Methyl tert butyl ether	<0.0552	<0.0250	<0.0691	<0.0620	<0.0250	<0.0623	<0.0420		<i>0.027</i>	282
cis-1,2-Dichloroethylene	<0.0269	na	<0.0337	<0.0303	na	<0.0304	<0.0273		<i>0.0412</i>	2340
1,2,4-TMB	<0.0237	<0.0250	0.0475	<0.0266	0.128	0.0570	<0.0239		NS	219
1,3,5-TMB	<0.0237	<0.0250	0.0346	<0.0266	0.0971	0.0689	<0.0239		NS	182
TMBs (combined)	<0.0474	<0.0500	0.0821	<0.0532	0.2251	0.1259	<0.0478		<i>1.3787</i>	NS
Naphthalene	<0.0283	na	<0.0354	<0.0318	na	<0.0319	<0.0286		<i>0.6582</i>	24.1
Bromobenzene	<0.0230	na	<0.0288	<0.0258	na	<0.0260	<0.0233		NS	679
Bromodichloromethane	<0.0250	na	<0.0312	<0.0281	na	<0.0282	<0.0253		<i>0.0003</i>	1.83
Bromomethane	<i>0.217</i>	na	<0.164	<0.148	na	<0.148	<0.133		<i>0.0051</i>	43
tert-Butylbenzene	<0.0243	na	<0.0304	<0.0273	na	<0.0274	<0.0246		NS	183
Isopropylbenzene	<0.0237	na	<0.0296	<0.0266	na	<0.0267	<0.0239		NS	268
Dibromochloromethane	<0.0217	na	<0.0271	<0.0244	na	<0.0245	<0.0165		<i>0.032</i>	38.9
4-Isopropyltoluene	<0.0230	na	<0.0288	<0.0258	na	<0.0260	<0.0233		NS	162
Propylbenzene	<0.0237	na	<0.0296	<0.0266	na	<0.0267	<0.0239		NS	264
1,1,1,2-Tetrachloroethane	<0.0237	na	<0.0296	<0.0266	na	<0.0267	<0.0239		<i>0.0534</i>	12.3
1,1,2,2-Tetrachloroethane	<0.0237	na	<0.0296	<0.0266	na	<i>0.218</i>	<0.0239		<i>0.0002</i>	3.6
1,1,2-Trichloroethane	<0.0269	na	<0.0337	<0.0303	na	<0.0304	<0.0273		<i>0.0032</i>	7.01
Shallow Soil (Industrial) Multiple Contaminant Cumulative ⁽²⁾										
Cancer Risk (CCR)	5.0E-09	--	0.0E+00	--	1.0E-09	6.1E-08	--		NR 720 threshold = 1E-5	
Hazard Index (HI)	0.0051	--	0.0001	--	0.0002	0.0001	--		NR 720 threshold = 1	

NOTES:

Concentrations are in units of milligrams per kilogram (mg/kg) on a dry weight basis.

No results exceed an applicable NR 720 industrial direct contact RCL, as shown in red font and bold.

Detected concentrations at or above an applicable NR 720 soil to groundwater pathway RCL are in red font and italicized.

NR 720 residual contaminant level (RCL) standards from WDNR's RR Program Soil RCL Excel workbook updated December 2018.

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

na = Not analyzed.

NS = No standard.

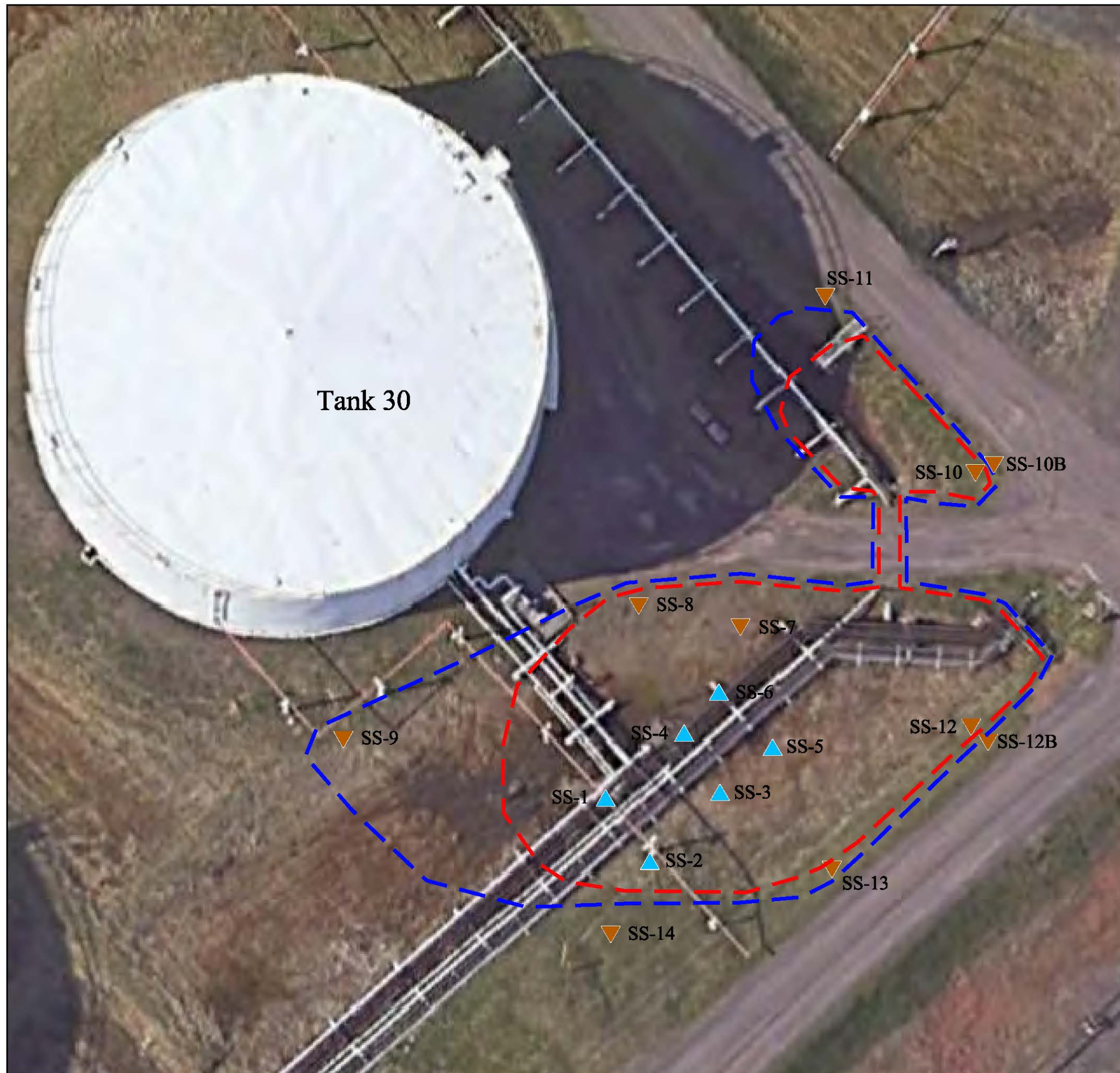
TMBs (combined) = Trimethylbenzenes (1,2,4- and 1,3,5- combined).

-- = Not applicable.

FOOTNOTES:

(1) Soil sample type based on the observed low water table in closest surveyed monitoring well, MW-5/T70 (2.34 ft bgs on 9/3/15).

(2) Industrial multiple contaminant cumulative cancer risk (CCR) and hazard index (HI) levels, if applicable. Thresholds are 1E-5 for CCR and 1 for HI per NR 720.12(1)(b). No CCR or HI levels at or above their respective threshold were calculated.

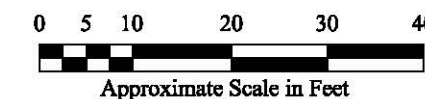


Legend

- - Estimated extent of unsaturated soil with one or more substances \geq a generic NR 720 soil to groundwater pathway RCL for PVOCs/Naphthalene
- - Estimated extent of unsaturated soil with one or more substances \geq a generic NR 720 soil to groundwater pathway residual contaminant level (RCL)
- ▲ Soil Sample Location (May 2011)
- ▼ Sample Location (April/May 2012)

Notes

- 1) No results \geq a generic NR720 industrial direct contact RCL.
- 2) WDNR BRRTS # 02-16-557992 Muphy Oil -Tank #30 site.
- 3) Parcel # 01-801-03339-0.



**Detailed Site Map and Soil Contamination
(April/May 2012)**

SUPERIOR REFINING COMPANY LLC
SUPERIOR, WISCONSIN



Gannett Fleming, Inc.
8025 Excelsior Drive
Madison WI 53717-1900
(608) 836-1500
www.gannettfleming.com

Project No.	34265.003	Date	03/18/19	Figure	A-1
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ATTACHMENT B

FEBRUARY 2018 RELEASE SITE SOIL SAMPLE RESULTS SUMMARY

SUPERIOR REFINING COMPANY LLC
SUPERIOR, WISCONSIN

TABLE B-1

SOIL ANALYTICAL RESULTS (TANK 30 BASIN, FEBRUARY 2018 RELEASE SITE)

Group/Substance/CCR-HI	Concentration (mg/kg)					NR 720 RCL (mg/kg)		
	Sample ID	S1	S2	S3	S4	S5	Soil to Groundwater Pathway	Industrial Direct Contact
Sample Date	11/08/18	11/08/18	11/08/18	11/08/18	11/08/18	12/06/18		
Saturated/Unsaturated ⁽¹⁾	Unsat.	Unsat.	Unsat.	Unsat.	Unsat.	Unsat.		
Excavated and landfilled ⁽²⁾	No	No	Yes	No	No	No		
Total petroleum hydrocarbons	175	261	341	168	383		NS	NS
Benzene	<i>0.568</i>	<i>3.80</i>	13.0	<i>1.86</i>	<i>3.09</i>		<i>0.0051</i>	7.07
Toluene	0.513	<i>11.0</i>	<i>32.4</i>	<i>4.47</i>	<i>12.5</i>		<i>1.1072</i>	818
Ethylbenzene	0.803	<i>2.40</i>	<i>16.2</i>	<i>1.05</i>	<i>2.88</i>		<i>1.57</i>	35.4
Xylenes	<i>6.64</i>	<i>14.74</i>	<i>78.9</i>	<i>8.01</i>	<i>17.87</i>		<i>3.96</i>	260
Methyl tert butyl ether	<i>0.369</i>	<0.0320	<0.0202	<0.0160	<i>0.0961</i>		<i>0.027</i>	282
Naphthalene	0.329 J J6	<0.208	0.134 J	<0.104	<0.0697 J3 J5		<i>0.6582</i>	24.1
1,2,4-TMB	8.44 J6	4.80	27.5	3.20	6.13 J5		NS	219
1,3,5-TMB	3.80 J3 J6	1.82	12.2	1.33	2.40		NS	182
TMBs (combined)	<i>12.24</i>	<i>6.62</i>	<i>39.7</i>	<i>4.53</i>	<i>8.53</i>		<i>1.3787</i>	NS
Shallow Soil (Industrial) Multiple Contaminant Cumulative ⁽³⁾								
Cancer Risk (CCR)	1.2E-07	6.1E-07	2.3E-06	2.9E-07	5.2E-07		NR 720 threshold = 1E-5	
Hazard Index (HI)	0.0086	0.0138	0.063	0.0075	0.0143		NR 720 threshold = 1	

NOTES:

Concentrations are in units of milligrams per kilogram (mg/kg) on a dry weight basis.

Detected concentrations at or above an applicable NR 720 industrial direct contact RCL are in red font and bold.

Detected concentrations at or above an applicable NR 720 soil to groundwater pathway RCL are in red font and italicized.

NR 720 residual contaminant level (RCL) standards from WDNR's RR Program Soil RCL Excel workbook updated December 2018.

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

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.

J6 = The sample matrix interfered with the ability to make any accurate determination; spike value is low.

NS = No standard.

TMBs (combined) = Trimethylbenzenes (1,2,4- and 1,3,5- combined).

FOOTNOTES:

(1) Soil sample type (unsat. = unsaturated) based on observed conditions when the sample was collected by hand.

(2) Excavated & Landfilled row indicates the one sample location that was excavated and disposed of at a local landfill.

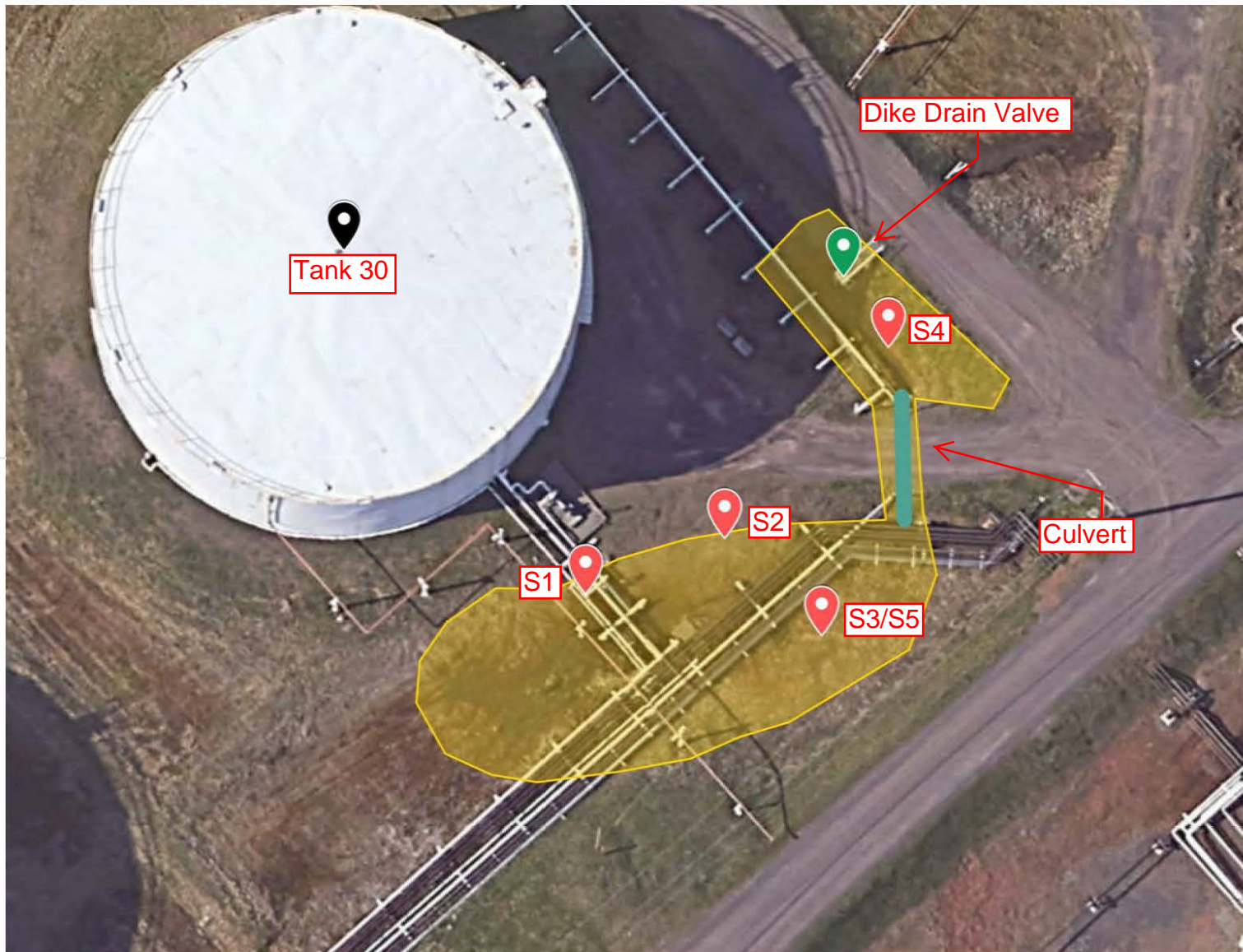
(3) Industrial multiple contaminant cumulative cancer risk (CCR) and hazard index (HI) levels, if applicable (for samples within 4 feet of ground surface and based on detected concentrations only). Thresholds are 1E-5 for CCR and 1 for HI per NR 720.12(1)(b). No CCR or HI levels at or above their respective threshold were calculated.

Figure B-1 - 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



ATTACHMENT C

LABORATORY REPORTS AND CHAIN-OF-CUSTODY RECORDS FOR
WATER SAMPLES COLLECTED IN 2018

Calumet Specialty Products

Project #34265.003

Superior Refining

Client Supplied

Sample Delivery Group: L977712
Samples Received: 03/15/2018
Project Number:
Description: Tk 30 Dike Water

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

Entire Report Reviewed By:



John Hawkins

Technical Service Representative

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 ESC is performed per guidance provided in laboratory standard operating procedures: 060302, 060303, and 060304.



Cp: Cover Page	1	¹Cp
Tc: Table of Contents	2	²Tc
Ss: Sample Summary	3	³Ss
Cn: Case Narrative	4	⁴Cn
Sr: Sample Results	5	⁵Sr
POOL L977712-01	5	
DRAIN VALVE L977712-02	6	
Qc: Quality Control Summary	7	⁶Qc
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	7	
Gl: Glossary of Terms	8	⁷Gl
Al: Accreditations & Locations	9	⁸Al
Sc: Sample Chain of Custody	10	⁹Sc

SAMPLE SUMMARY



POOL L977712-01 GW

Collected by Matt Turner	Collected date/time 03/14/18 10:02	Received date/time 03/15/18 08:45
-----------------------------	---------------------------------------	--------------------------------------

Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	WG1085469	5	03/16/18 12:34	03/16/18 12:34	JAH

1
Cp

2
Tc

3
Ss

DRAIN VALVE L977712-02 GW

Collected by Matt Turner	Collected date/time 03/14/18 10:07	Received date/time 03/15/18 08:45
-----------------------------	---------------------------------------	--------------------------------------

Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	WG1085469	200	03/16/18 11:57	03/16/18 11:57	JAH

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 radiochemical sample results for solids are reported on a dry weight basis with the exception of tritium, carbon-14 and radon, unless wet weight was requested by the client. 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
Technical Service Representative

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 date / time	Batch
Benzene	422		0.350	1.17	5	03/16/2018 12:34	WG1085469
Toluene	878		2.06	6.85	5	03/16/2018 12:34	WG1085469
Ethylbenzene	105		0.600	2.00	5	03/16/2018 12:34	WG1085469
m&p-Xylene	484		0.605	2.02	5	03/16/2018 12:34	WG1085469
o-Xylene	198		0.520	1.74	5	03/16/2018 12:34	WG1085469
Methyl tert-butyl ether	U		1.26	4.20	5	03/16/2018 12:34	WG1085469
Naphthalene	5.59	<u>B</u>	1.10	3.69	5	03/16/2018 12:34	WG1085469
1,3,5-Trimethylbenzene	39.9		0.395	1.32	5	03/16/2018 12:34	WG1085469
1,2,4-Trimethylbenzene	148		0.465	1.55	5	03/16/2018 12:34	WG1085469
TPH (GC/FID) Low Fraction	5010		75.0	250	5	03/16/2018 12:34	WG1085469
(S) <i>a,a,a</i> -Trifluorotoluene(PID)	101			80.0-200		03/16/2018 12:34	WG1085469

- 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 date / time	Batch
Benzene	1380		14.0	46.6	200	03/16/2018 11:57	WG1085469
Toluene	2270		82.4	274	200	03/16/2018 11:57	WG1085469
Ethylbenzene	312		24.0	80.0	200	03/16/2018 11:57	WG1085469
m&p-Xylene	1850		24.2	80.6	200	03/16/2018 11:57	WG1085469
o-Xylene	908		20.8	69.4	200	03/16/2018 11:57	WG1085469
Methyl tert-butyl ether	U		50.4	168	200	03/16/2018 11:57	WG1085469
Naphthalene	56.4	<u>BJ</u>	44.2	147	200	03/16/2018 11:57	WG1085469
1,3,5-Trimethylbenzene	432		15.8	52.6	200	03/16/2018 11:57	WG1085469
1,2,4-Trimethylbenzene	2040		18.6	62.0	200	03/16/2018 11:57	WG1085469
TPH (GC/FID) Low Fraction	17700		3000	10000	200	03/16/2018 11:57	WG1085469
(S) <i>a,a,a</i> -Trifluorotoluene(PID)	107			80.0-200		03/16/2018 11:57	WG1085469

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



Method Blank (MB)

(MB) R3295391-3 03/16/18 10:42

Analyte	MB Result	MB Qualifier	MB MDL	MB RDL
	ug/l		ug/l	ug/l
Benzene	U		0.0700	0.233
Toluene	U		0.412	1.37
Ethylbenzene	U		0.120	0.400
m&p-Xylene	U		0.121	0.403
o-Xylene	U		0.104	0.347
Methyl tert-butyl ether	U		0.252	0.840
Naphthalene	0.408	↓	0.221	0.737
1,3,5-Trimethylbenzene	U		0.0790	0.263
1,2,4-Trimethylbenzene	U		0.0930	0.310
TPH (GC/FID) Low Fraction	U		15.0	50.0
(S) a,a,a-Trifluorotoluene(PID)	109			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) R3295391-1 03/16/18 09:55 • (LCSD) R3295391-4 03/16/18 16:29

Analyte	Spike Amount	LCS Result	LCSD Result	LCS Rec.	LCSD Rec.	Rec. Limits	LCS Qualifier	LCSD Qualifier	RPD	RPD Limits
	ug/l	ug/l	ug/l	%	%	%			%	%
Benzene	50.0	53.0	52.2	106	104	80.0-120			1.56	20
Toluene	50.0	51.5	51.2	103	102	80.0-120			0.558	20
Ethylbenzene	50.0	50.1	49.9	100	99.8	80.0-120			0.482	20
m&p-Xylene	100	103	102	103	102	80.0-120			0.116	20
o-Xylene	50.0	51.2	51.6	102	103	80.0-120			0.832	20
Methyl tert-butyl ether	50.0	42.5	43.6	84.9	87.2	80.0-120			2.69	20
Naphthalene	50.0	47.2	45.1	94.4	90.2	80.0-120			4.60	20
1,3,5-Trimethylbenzene	50.0	49.6	49.4	99.2	98.8	80.0-120			0.457	20
1,2,4-Trimethylbenzene	50.0	50.9	50.6	102	101	80.0-120			0.617	20
(S) a,a,a-Trifluorotoluene(PID)				104	105	80.0-200				

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

(LCS) R3295391-2 03/16/18 09:55 • (LCSD) R3295391-5 03/16/18 16:29

Analyte	Spike Amount	LCS Result	LCSD Result	LCS Rec.	LCSD Rec.	Rec. Limits	LCS Qualifier	LCSD Qualifier	RPD	RPD Limits
	ug/l	ug/l	ug/l	%	%	%			%	%
TPH (GC/FID) Low Fraction	550	566	561	103	102	80.0-120			0.884	20
(S) a,a,a-Trifluorotoluene(PID)				104	105	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, 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.
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.
Qualifier	Description
B	The same analyte is found in the associated blank.
J	The identification of the analyte is acceptable; the reported value is an estimate.

1 Cp

2 Tc

3 Ss

4 Cn

5 Sr

6 Qc

7 Gl

8 Al

9 Sc



ESC Lab Sciences 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 ESC Lab Sciences.

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

1
Cp

2
Tc

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Ss

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Cn

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Sr

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Qc

7
Gl

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Al

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Sc

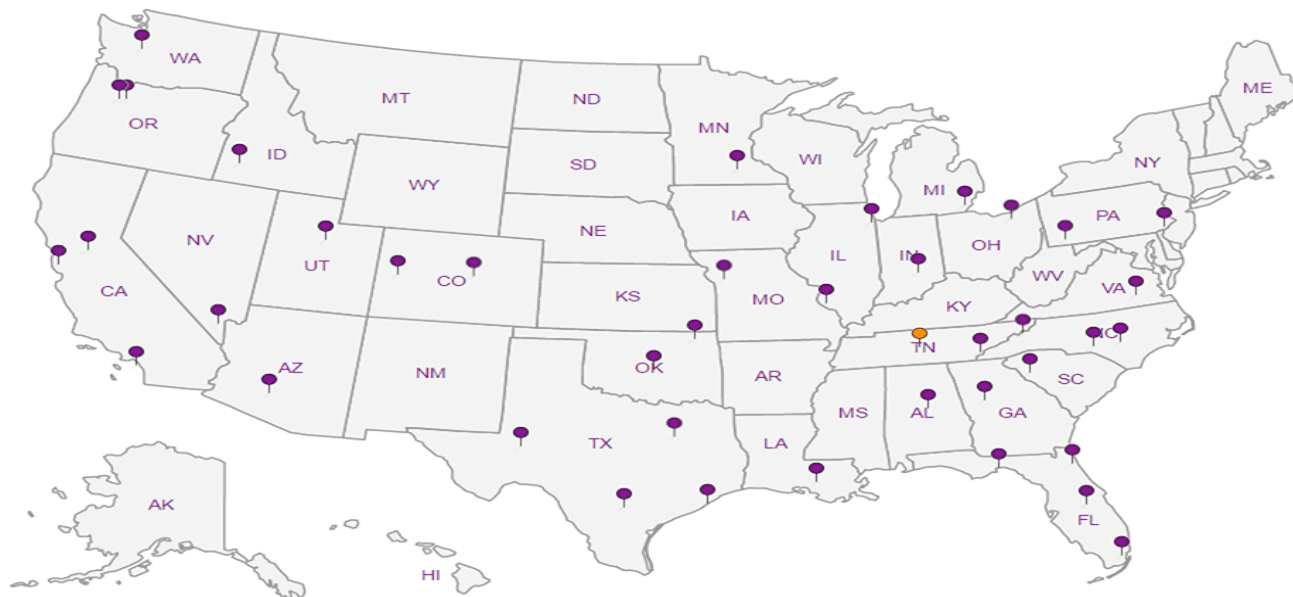
Third Party Federal Accreditations

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

ESC Lab Sciences 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. ESC Lab Sciences performs all testing at our central laboratory.



September 07, 2018

Superior Refinery Husky Energy

Sample Delivery Group: L1021912
Samples Received: 08/30/2018
Project Number:
Description: Tk 30

Project #34265.003
Superior Refining
Client supplied

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	
S-1 L1021912-01	5	
Qc: Quality Control Summary	6	
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	6	
Gl: Glossary of Terms	8	
Al: Accreditations & Locations	9	
Sc: Sample Chain of Custody	10	

SAMPLE SUMMARY



S-1 L1021912-01 GW

Collected by: Matt Turner
 Collected date/time: 08/29/18 13:04
 Received date/time: 08/30/18 08:45

Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	WG1161342	1	09/06/18 15:14	09/06/18 15:14	DWR

¹Cp

²Tc

³Ss

⁴Cn

⁵Sr

⁶Qc

⁷Gl

⁸Al

⁹Sc



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 ug/l	Qualifier	MDL ug/l	RDL ug/l	Dilution	Analysis date / time	Batch
Benzene	3.75		0.0700	0.233	1	09/06/2018 15:14	WG1161342
Toluene	3.81		0.412	1.37	1	09/06/2018 15:14	WG1161342
Ethylbenzene	0.472		0.120	0.400	1	09/06/2018 15:14	WG1161342
m&p-Xylene	11.0		0.121	0.403	1	09/06/2018 15:14	WG1161342
o-Xylene	15.1		0.104	0.347	1	09/06/2018 15:14	WG1161342
Methyl tert-butyl ether	0.429	<u>J</u>	0.252	0.840	1	09/06/2018 15:14	WG1161342
Naphthalene	0.228	<u>B J</u>	0.221	0.737	1	09/06/2018 15:14	WG1161342
1,3,5-Trimethylbenzene	8.15		0.0790	0.263	1	09/06/2018 15:14	WG1161342
1,2,4-Trimethylbenzene	1.40	<u>B</u>	0.0930	0.310	1	09/06/2018 15:14	WG1161342
TPH (GC/FID) Low Fraction	323		15.0	50.0	1	09/06/2018 15:14	WG1161342
(S) <i>a,a,a-Trifluorotoluene(PID)</i>	99.1			80.0-200		09/06/2018 15:14	WG1161342

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

Sample Narrative:

L1021912-01 WG1161342: Peaks/Baseline rise detected outside GRO/DRO window



Method Blank (MB)

(MB) R3339962-3 09/06/18 12:11

Analyte	MB Result	MB Qualifier	MB MDL	MB RDL
	ug/l		ug/l	ug/l
Benzene	0.0924	U	0.0700	0.233
Toluene	U		0.412	1.37
Ethylbenzene	U		0.120	0.400
m&p-Xylene	0.159	U	0.121	0.403
o-Xylene	U		0.104	0.347
Methyl tert-butyl ether	U		0.252	0.840
Naphthalene	0.341	U	0.221	0.737
1,3,5-Trimethylbenzene	0.208	U	0.0790	0.263
1,2,4-Trimethylbenzene	0.193	U	0.0930	0.310
TPH (GC/FID) Low Fraction	U		15.0	50.0
(S) a,a,a-Trifluorotoluene(PID)	99.3			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) R3339962-1 09/06/18 11:00 • (LCSD) R3339962-8 09/06/18 19:17

Analyte	Spike Amount	LCS Result	LCSD Result	LCS Rec.	LCSD Rec.	Rec. Limits	LCS Qualifier	LCSD Qualifier	RPD	RPD Limits
	ug/l	ug/l	ug/l	%	%	%			%	%
TPH (GC/FID) Low Fraction	550	570	556	104	101	80.0-120			2.50	20
(S) a,a,a-Trifluorotoluene(PID)				95.8	98.0	80.0-200				

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

(LCS) R3339962-2 09/06/18 11:00 • (LCSD) R3339962-9 09/06/18 19:17

Analyte	Spike Amount	LCS Result	LCSD Result	LCS Rec.	LCSD Rec.	Rec. Limits	LCS Qualifier	LCSD Qualifier	RPD	RPD Limits
	ug/l	ug/l	ug/l	%	%	%			%	%
Benzene	50.0	54.3	54.9	109	110	80.0-120			1.08	20
Toluene	50.0	56.7	57.2	113	114	80.0-120			0.792	20
Ethylbenzene	50.0	55.7	55.9	111	112	80.0-120			0.324	20
m&p-Xylene	100	112	112	112	112	80.0-120			0.298	20
o-Xylene	50.0	53.9	54.1	108	108	80.0-120			0.460	20
Methyl tert-butyl ether	50.0	55.3	57.4	111	115	80.0-120			3.76	20
Naphthalene	50.0	45.9	43.3	91.7	86.6	80.0-120			5.72	20
1,3,5-Trimethylbenzene	50.0	54.3	54.0	109	108	80.0-120			0.557	20
1,2,4-Trimethylbenzene	50.0	53.7	53.5	107	107	80.0-120			0.364	20
(S) a,a,a-Trifluorotoluene(PID)				95.8	98.0	80.0-200				



L1021912-01 Original Sample (OS) • Matrix Spike (MS) • Matrix Spike Duplicate (MSD)

(OS) L1021912-01 09/06/18 15:14 • (MS) R3339962-4 09/06/18 12:49 • (MSD) R3339962-6 09/06/18 13:37

Analyte	Spike Amount ug/l	Original Result ug/l	MS Result ug/l	MSD Result ug/l	MS Rec. %	MSD Rec. %	Dilution	Rec. Limits %	MS Qualifier	MSD Qualifier	RPD %	RPD Limits %
TPH (GC/FID) Low Fraction	550	323	870	856	99.5	96.9	1	80.0-120			1.67	20
(S) a,a,a-Trifluorotoluene(PID)					96.3	96.6		80.0-200				

Sample Narrative:

OS: Peaks/Baseline rise detected outside GRO/DRO window

L1021912-01 Original Sample (OS) • Matrix Spike (MS) • Matrix Spike Duplicate (MSD)

(OS) L1021912-01 09/06/18 15:14 • (MS) R3339962-5 09/06/18 12:49 • (MSD) R3339962-7 09/06/18 13:37

Analyte	Spike Amount ug/l	Original Result ug/l	MS Result ug/l	MSD Result ug/l	MS Rec. %	MSD Rec. %	Dilution	Rec. Limits %	MS Qualifier	MSD Qualifier	RPD %	RPD Limits %
Benzene	50.0	3.75	51.7	56.3	96.0	105	1	35.0-147			8.51	20
Toluene	50.0	3.81	53.8	52.1	99.9	96.5	1	35.0-148			3.21	20
Ethylbenzene	50.0	0.472	52.1	50.7	103	101	1	39.0-141			2.72	20
m&p-Xylene	100	11.0	113	110	102	99.0	1	26.0-157			2.46	20
o-Xylene	50.0	15.1	64.6	63.1	99.0	96.0	1	40.0-145			2.33	20
Methyl tert-butyl ether	50.0	0.429	50.6	50.5	100	100	1	37.0-147			0.159	20
Naphthalene	50.0	0.228	46.5	45.7	92.5	90.9	1	80.0-120			1.79	20
1,3,5-Trimethylbenzene	50.0	8.15	59.1	57.8	102	99.2	1	80.0-120			2.28	20
1,2,4-Trimethylbenzene	50.0	1.40	52.5	51.2	102	99.7	1	80.0-120			2.44	20
(S) a,a,a-Trifluorotoluene(PID)					96.3	96.6		80.0-200				

Sample Narrative:

OS: 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



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.
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Qualifier	Description
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J	The identification of the analyte is acceptable; the reported value is an estimate.





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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

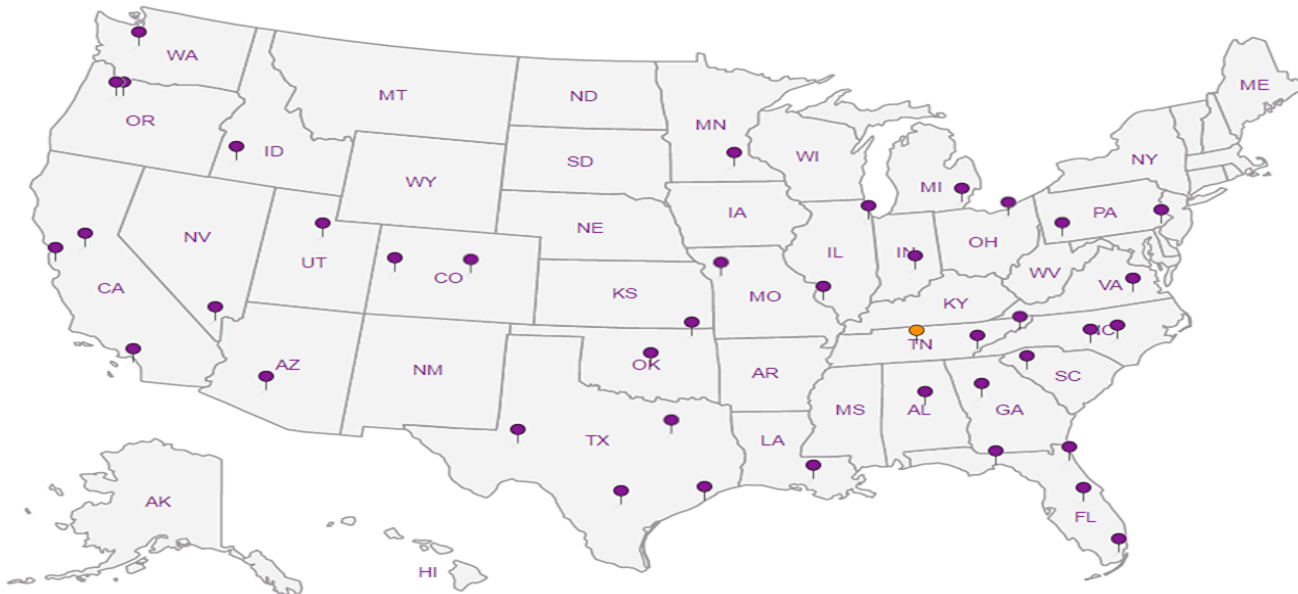
Third Party Federal Accreditations

A2LA – ISO 17025	1461.01	AIHA-LAP,LLC EMLAP	100789
A2LA – ISO 17025 ⁵	1461.02	DOD	1461.01
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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.

Pres
 Chk

HC

Analysis / Container / Preservative



12065 Lebanon Rd
 Mount Juliet, TN 37122
 Phone: 615-758-5858
 Phone: 800-767-5859
 Fax: 615-758-5859



L# 1021912
 G068

Table
 Accnum: MUROILSWI
 Template:
 Prelogin:
 TSR: John Hawkins (341)
 PB:

Shipped Via:
 Remarks Sample # (lab only)

61

Report to:
 Matt Turner

Email To:
 matt.turner@huskyenergy.com

Project Description:
 Tk 30

City/State Collected:
 Superior, WI

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

Client Project #

Lab Project #

Collected by (print):
 Matt Turner

Site/Facility ID #

P.O. #

Collected by (signature):
 [Signature]

Rush? (Lab MUST Be Notified)

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

Quote #

Date Results Needed

No. of
 Cntrs

Immediately Packed on Ice: N Y X

PVOCS + Naphthalene

Sample ID	Comp/Grab	Matrix *	Depth	Date	Time	No. of Cntrs
S-1	Grab	GW	-	8/29/18	13:04	2 X

RAD SOURCE: <0.5 mR/hr

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

Remarks:

Samples returned via:
 UPS FedEx Courier

Tracking # 72154522 1049

pH _____ Temp _____
 Flow _____ Other _____

Sample Receipt Checklist

COC Seal Present/Intact:	<input checked="" type="checkbox"/>	Y	N
COC Signed/Accurate:	<input checked="" type="checkbox"/>	Y	N
Bottles arrive intact:	<input checked="" type="checkbox"/>	Y	N
Correct bottles used:	<input checked="" type="checkbox"/>	Y	N
Sufficient volume sent:	<input checked="" type="checkbox"/>	Y	N
VOA Zero Headspace:	<input checked="" type="checkbox"/>	Y	N
Preservation Correct/Checked:	<input checked="" type="checkbox"/>	Y	N

Relinquished by: (Signature)
 [Signature]

Date: 8-29-18 Time: 13:46

Received by: (Signature)

Trip Blank Received: Yes / No
 HCL / MeOH
 TBR

if preservation required by Login: Date/Time

Relinquished by: (Signature)

Date: _____ Time: _____

Received by: (Signature)

Temp: 4.6°C Bottles Received: 2

Hold:

Condition: NCF [initials]

Relinquished by: (Signature)

Date: _____ Time: _____

Received for lab by: (Signature)
 [Signature]

Date: 8/30/18 Time: 0845

October 16, 2018

Project #34265.003
Superior Refining
Client supplied

Jim Taraldsen
Barr Engineering Company
325 S Lake Ave
Duluth, MN 55802

RE: Project: 49161427.06 Husky
Pace Project No.: 10451391

Dear Jim Taraldsen:

Enclosed are the analytical results for sample(s) received by the laboratory on October 11, 2018. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC 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,



Amanda Albrecht
amanda.albrecht@pacelabs.com
(612)607-6382
Project Manager

Enclosures

cc: BarrDM, Barr Engineering
Ryan Erickson, Barr Engineering
Timothy Harris, GHD
Brian Kwiatkoski, Barr Engineering
Dana Pasi, Barr Engineering
Mr. Ward Swanson, Barr Engineering Company



REPORT OF LABORATORY ANALYSIS

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

CERTIFICATIONS

Project: 49161427.06 Husky

Pace Project No.: 10451391

Minnesota Certification IDs

1700 Elm Street SE, Minneapolis, MN 55414-2485

A2LA Certification #: 2926.01

Alabama Certification #: 40770

Alaska Contaminated Sites Certification #: 17-009

Alaska DW Certification #: MN00064

Arizona Certification #: AZ0014

Arkansas DW Certification #: MN00064

Arkansas WW Certification #: 88-0680

California Certification #: 2929

CNMI Saipan Certification #: MP0003

Colorado Certification #: MN00064

Connecticut Certification #: PH-0256

EPA Region 8+Wyoming DW Certification #: via MN 027-053-137

Florida Certification #: E87605

Georgia Certification #: 959

Guam EPA Certification #: MN00064

Hawaii Certification #: MN00064

Idaho Certification #: MN00064

Illinois Certification #: 200011

Indiana Certification #: C-MN-01

Iowa Certification #: 368

Kansas Certification #: E-10167

Kentucky DW Certification #: 90062

Kentucky WW Certification #: 90062

Louisiana DEQ Certification #: 03086

Louisiana DW Certification #: MN00064

Maine Certification #: MN00064

Maryland Certification #: 322

Massachusetts Certification #: M-MN064

Michigan Certification #: 9909

Minnesota Certification #: 027-053-137

Minnesota Dept of Ag Certification #: via MN 027-053-137

Minnesota Petrofund Certification #: 1240

Mississippi Certification #: MN00064

Montana Certification #: CERT0092

Nebraska Certification #: NE-OS-18-06

Nevada Certification #: MN00064

New Hampshire Certification #: 2081

New Jersey Certification #: MN002

New York Certification #: 11647

North Carolina DW Certification #: 27700

North Carolina WW Certification #: 530

North Dakota Certification #: R-036

Ohio DW Certification #: 41244

Ohio VAP Certification #: CL101

Oklahoma Certification #: 9507

Oregon NwTPH Certification #: MN300001

Oregon Secondary Certification #: MN200001

Pennsylvania Certification #: 68-00563

Puerto Rico Certification #: MN00064

South Carolina Certification #:74003001

Tennessee Certification #: TN02818

Texas Certification #: T104704192

Utah Certification #: MN00064

Virginia Certification #: 460163

Washington Certification #: C486

West Virginia DW Certification #: 9952 C

West Virginia DEP Certification #: 382

Wisconsin Certification #: 999407970

Wyoming UST Certification #: via A2LA 2926.01

REPORT OF LABORATORY ANALYSIS

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SAMPLE SUMMARY

Project: 49161427.06 Husky

Pace Project No.: 10451391

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10451391001	SW-1_TK30	Water	10/11/18 12:50	10/11/18 19:30

REPORT OF LABORATORY ANALYSIS

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SAMPLE ANALYTE COUNT

Project: 49161427.06 Husky

Pace Project No.: 10451391

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10451391001	SW-1_TK30	EPA 8260B	MJD	11	PASI-M

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: 49161427.06 Husky

Pace Project No.: 10451391

Sample: SW-1_TK30 **Lab ID: 10451391001** Collected: 10/11/18 12:50 Received: 10/11/18 19:30 Matrix: Water

Parameters	Results	Units	LOQ	LOD	DF	Prepared	Analyzed	CAS No.	Qual
8260B MSV UST		Analytical Method: EPA 8260B							
Benzene	0.33J	ug/L	0.34	0.10	1		10/13/18 15:41	71-43-2	
Ethylbenzene	<0.14	ug/L	0.46	0.14	1		10/13/18 15:41	100-41-4	
Methyl-tert-butyl ether	<0.16	ug/L	0.54	0.16	1		10/13/18 15:41	1634-04-4	
Naphthalene	<0.48	ug/L	1.6	0.48	1		10/13/18 15:41	91-20-3	
Toluene	0.48	ug/L	0.28	0.083	1		10/13/18 15:41	108-88-3	
1,2,4-Trimethylbenzene	<0.20	ug/L	0.65	0.20	1		10/13/18 15:41	95-63-6	
1,3,5-Trimethylbenzene	<0.12	ug/L	0.41	0.12	1		10/13/18 15:41	108-67-8	
Xylene (Total)	<0.31	ug/L	1.0	0.31	1		10/13/18 15:41	1330-20-7	
Surrogates									
1,2-Dichloroethane-d4 (S)	96	%	75-125		1		10/13/18 15:41	17060-07-0	
Toluene-d8 (S)	98	%	75-125		1		10/13/18 15:41	2037-26-5	
4-Bromofluorobenzene (S)	98	%	75-125		1		10/13/18 15:41	460-00-4	

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: 49161427.06 Husky
Pace Project No.: 10451391

QC Batch: 569014 Analysis Method: EPA 8260B
QC Batch Method: EPA 8260B Analysis Description: 8260B MSV UST-WATER
Associated Lab Samples: 10451391001

METHOD BLANK: 3088026 Matrix: Water
Associated Lab Samples: 10451391001

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
1,2,4-Trimethylbenzene	ug/L	<0.20	0.65	10/13/18 12:35	
1,3,5-Trimethylbenzene	ug/L	<0.12	0.41	10/13/18 12:35	
Benzene	ug/L	<0.10	0.34	10/13/18 12:35	
Ethylbenzene	ug/L	<0.14	0.46	10/13/18 12:35	
Methyl-tert-butyl ether	ug/L	<0.16	0.54	10/13/18 12:35	
Naphthalene	ug/L	<0.48	1.6	10/13/18 12:35	
Toluene	ug/L	<0.083	0.28	10/13/18 12:35	
Xylene (Total)	ug/L	<0.31	1.0	10/13/18 12:35	
1,2-Dichloroethane-d4 (S)	%	96	75-125	10/13/18 12:35	
4-Bromofluorobenzene (S)	%	99	75-125	10/13/18 12:35	
Toluene-d8 (S)	%	99	75-125	10/13/18 12:35	

LABORATORY CONTROL SAMPLE: 3088027

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1,2,4-Trimethylbenzene	ug/L	20	19.2	96	75-125	
1,3,5-Trimethylbenzene	ug/L	20	19.6	98	75-125	
Benzene	ug/L	20	21.3	106	75-126	
Ethylbenzene	ug/L	20	19.7	98	75-125	
Methyl-tert-butyl ether	ug/L	20	16.4	82	73-129	
Naphthalene	ug/L	20	18.9	95	65-126	
Toluene	ug/L	20	19.9	99	74-125	
Xylene (Total)	ug/L	60	60.4	101	75-125	
1,2-Dichloroethane-d4 (S)	%			96	75-125	
4-Bromofluorobenzene (S)	%			96	75-125	
Toluene-d8 (S)	%			99	75-125	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3088028 3088029

Parameter	Units	MS		MSD		MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	Max RPD	Qual
		10450927004 Result	Spike Conc.	Spike Conc.	Conc.							
1,2,4-Trimethylbenzene	ug/L	ND	20	20	19.9	17.0	100	85	67-130	16	30	
1,3,5-Trimethylbenzene	ug/L	ND	20	20	20.7	17.4	103	87	63-139	17	30	
Benzene	ug/L	1.2	20	20	25.4	22.3	121	105	62-140	13	30	
Ethylbenzene	ug/L	ND	20	20	21.0	17.6	105	88	75-131	18	30	
Methyl-tert-butyl ether	ug/L	ND	20	20	25.2	21.9	126	110	65-130	14	30	
Naphthalene	ug/L	ND	20	20	19.8	17.3	99	86	48-134	14	30	
Toluene	ug/L	ND	20	20	21.8	18.4	109	92	68-132	17	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

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QUALITY CONTROL DATA

Project: 49161427.06 Husky

Pace Project No.: 10451391

Parameter	Units	3088028		3088029		MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	Max		Qual
		10450927004 Result	MS Spike Conc.	MSD Spike Conc.	MS Result						MSD Result	RPD	
Xylene (Total)	ug/L	ND	60	60	64.0	54.3	107	91	91	69-135	16	30	
1,2-Dichloroethane-d4 (S)	%.							93	95	75-125			
4-Bromofluorobenzene (S)	%.							98	98	75-125			
Toluene-d8 (S)	%.							98	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

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QUALIFIERS

Project: 49161427.06 Husky

Pace Project No.: 10451391

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 LOD.

J - Estimated concentration at or above the LOD and below the LOQ.

LOD - Limit of Detection adjusted for dilution factor and percent moisture.

LOQ - Limit of Quantitation adjusted for dilution factor and percent moisture.

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 at or above the adjusted LOD.

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

WORKORDER QUALIFIERS

WO: 10451391

[1] The samples were received outside of required temperature range. Analysis was completed upon client approval.

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: 49161427.06 Husky
Pace Project No.: 10451391

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10451391001	SW-1_TK30	EPA 8260B	569014		

REPORT OF LABORATORY ANALYSIS

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Barr Engineering Co. Chain of Custody

Sample Origination State:

- Ann Arbor Duluth Hibbing Minneapolis Bismarck Grand Rapids Jefferson City Salt Lake City

- KS MO UT MI ND WI MN SD Other: _____

Analysis Requested

Water Soil

COC Number: **57992**

COC 1 of 1

Matrix Code:

Preservative Code:

WO#: 10451391



10451391

- A = None
- B = HCl
- C = HNO₃
- D = H₂SO₄
- E = NaOH
- F = MeOH
- G = NaHSO₄
- H = Na₂S₂O₃
- I = Ascorbic Acid
- J = NH₄Cl
- K = Zn Acetate
- O = Other

REPORT TO	INVOICE TO
Company: <u>Barr Engineering</u>	Company: <u>Barr</u>
Address: <u>325 S Lake Ave Duluth, MN</u>	Address: <u>↓</u>
Name: <u>Ryan Erickson</u>	Name: <u>↓</u>
email: <u>rerickson@barr.com</u>	email: <u>↓</u>
Copy to: <u>datamgt@barr.com</u>	PO: <u>↓</u>
Project Name: <u>Husky</u>	Barr Project No: <u>49161427.06</u>

Location	Sample Depth			Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix Code	Perform MS/MSD Y / N	Total Number Of Containers	Water	Soil	% Solids
	Start	Stop	Unit (m./ft. or in.)								
1. <u>SW - 1 - TK30</u>	<u>---</u>	<u>---</u>	<u>---</u>	<u>10/11/2018</u>	<u>12:50</u>	<u>SW</u>	<u>N</u>	<u>3</u>	<u>3</u>		
2.											
3.											
4.											
5.											
6.											
7.											
8.											
9.											
10.											

Preservative Code
Field Filtered Y/N
PVOCs + Naphthalene

BARR USE ONLY

Sampled by: SGJ

Barr Proj. Manager: REE

Barr DQ Manager: JET

Lab Name: Pace

Lab Location: _____

Relinquished by: [Signature]

On Ice? N Y

Date: 10/11/18

Time: 13:45

Relinquished by: [Signature]

On Ice? N Y

Date: 10-11-18

Time: 1930

Samples Shipped VIA: Courier Federal Express Sampler Other: _____

Lab WO: _____

Received by: [Signature]

Date: 10/11/18

Time: 1345

Received by: [Signature]

Date: 10/11/18

Time: 1930

Air Bill Number: _____

Requested Due Date: Standard Turn Around Time Rush _____ (mm/dd/yyyy)

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Pink Copy: Send to Data Management Administrators.

T=0.4

Sample Condition Upon Receipt

Client Name: Barr Engineering Project #: WO# : 10451391

WO# : 10451391
 PM: AA1 Due Date: 10/12/18
 CLIENT: BARR

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: G87A9170600254 G87A9155100842
 Type of Ice: Wet Blue None Dry Melted

Cooler Temp Read (°C): -0.7 Cooler Temp Corrected (°C): -0.4 Biological Tissue Frozen? Yes No N/A
 Temp should be above freezing to 6°C Correction Factor: +0.3 Date and initials of Person Examining Contents: AS 10/11/18

USDA Regulated Soil (N/A, water sample)
 Did samples originate in a quarantine zone within the United States: AL, AR, CA, FL, GA, ID, LA, MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (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?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	1.
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	2.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	3.
Sampler Name and/or Signature on COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	4.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	5.
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	6.
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	7.
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	8.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	9.
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	10.
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11. Note if sediment is visible in the dissolved container
Is sufficient information available to reconcile the samples to the COC? Matrix: <u>WT</u>	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	12.
All containers needing acid/base preservation have been checked?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	13. <input type="checkbox"/> HNO ₃ <input type="checkbox"/> H ₂ SO ₄ <input type="checkbox"/> NaOH Positive for Res. Chlorine? Y N
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO ₃ , H ₂ SO ₄ , >2pH, NaOH >9 Sulfide, NaOH >12 Cyanide) Exceptions: <u>CO</u> Conform, TOC/DOC Oil and Grease, DRO/8015 (water) and Dioxin/PFAS	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Sample #
Headspace in VOA Vials (>6mm)?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	Initial when completed: _____ Lot # of added preservative: _____
Trip Blank Present?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	14. <u>No headspace</u>
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	15.
Pace Trip Blank Lot # (if purchased): <u>N/A</u>		<u>No TB</u>

CLIENT NOTIFICATION/RESOLUTION

Person Contacted: J. Taraldsen Date/Time: 10/12/18 Field Data Required? Yes No
 Comments/Resolution: Notified of temperature.

Project Manager Review: Amanda J. Albrecht Date: 10/12/18

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).