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Immediate Action Report and NFA Request

To:Mathew Turner, Superior Refining Company, LLCFrom:Lynette Carney and Kaitlin MontzSubject:Tank 47 Crude Pipeline ReleaseDate:September 21, 2021Project:49161468.02 300 302WDNR SERTS:20210830NO16-1 – Crude Oil

This report summarizes the immediate environmental response activities performed by Barr Engineering Co. (Barr) and Insight Environmental (Insight) at the request of Superior Refining Company LLC (SRC) following the petroleum release from a crude oil pipeline in a maintenance excavation in the Tank 47 basin at the Superior Refinery in Superior, Wisconsin (Figure 1).

Background

On August 30, 2021, an SRC operator observed approximately 50 gallons of oil in a pipeline maintenance excavation in the southeast corner of the Tank 47 containment dike (Photo 1, Photo 2 and Figure 2). The source of the release was a leaking cold tap that had been installed on the pipeline during recent maintenance activities but began to leak sometime after it was installed. Immediately upon discovery, the cold tap leak was repaired, and free liquids were vacuumed up and a containment pan was put under the valve until the pipeline was de-inventoried later that same day.

The Wisconsin Department of Natural Resources (WDNR) was immediately notified of the release upon discovery by SRC on August 30, 2021. The WDNR assigned spill tracking number 20210830NO16-1 to the site. The associated WDNR *Notification For Hazardous Substance Discharge* reporting form and the Tank 47 release site and site contact information is provided in Attachment A.

In response to this release, and in accordance with NR 708.05 and the approved facility-wide *Site Investigation Report and Response Action Plan (SI/RAP)* (Gannett Fleming, 2014), SRC arranged for the hydrocarbon impacted soil to be excavated and temporarily stockpiled on site until arrangements for offsite disposal at a landfill could be made. SRC requested that Barr and Insight assist with the following activities:

- field screen soil during the excavation to guide remediation efforts;
- collect soil samples from the final excavation extents for laboratory analysis to document residual soil conditions, and
- prepare a report summarizing the response actions and site conditions upon the completion of remedial activities.

Documentation of the immediate actions are being provided in accordance with NR 708.05 (6) and to support a no further action request under NR 708.09.

Field Activity Summary and Sample Results

The soil excavation response action was initiated by SRC and their contractor on August 31, the day following discovery of the release. Barr and Insight were on site to document response cleanup activities and to collect soil field screening and analytical samples from the final excavation limits. Soil headspace screening samples were collected from the removed soil and from the final sidewalls and bottom of the excavation and tested for the presence of organic vapors using a 10.6 eV photoionization detector (PID). PID headspace readings and other evidence of hydrocarbon impacts, such as odor and soil discoloration, were recorded on the field sampling and screening logs provided in Attachment B.

The final excavation extent was approximately 24 feet (northwest to southeast) by 14 feet (northeast to southwest) and up to 11 feet below ground surface (bgs) (Photo 5, Photo 6 and Attachment B). Clay soil was observed in the excavation sidewalls and bottom.

Insight collected 17 field screening soil samples (Photo 3 and Photo 4) and nine analytical confirmation soil samples (*TK47-B-1, TK47-S-1, TK47-S-2, TK47-S-3, TK47-S-4, TK47-S-5, TK47-S-6, TK47-S-7, and TK47-S-8*). Two analytical confirmation soil samples were collected from each side wall, one shallow (< 4.0 ft. bgs) and one deep (>4.0 ft. bgs). The soil analytical samples were submitted to ALS Laboratory (ALS) in Holland, Michigan for analysis of petroleum volatile organic compounds (PVOCs) plus naphthalene. The analytical confirmation soil sample locations are shown on Figure 2. Field headspace screening soil sample location and results are shown on the field screening form in Attachment B.

The field headspace screening results for soil samples collected from the final excavation extents did not identify evidence of hydrocarbon impacts. Headspace readings from the final sidewall and bottom soil headspace screening samples resulted in concentrations between 0.7 parts per million and 2.8 ppm. Laboratory results from the correlating laboratory samples collected at the final excavation extents did not show PVOC or naphthalene compound detections in any of the nine samples. The analytical results are summarized in Table 1 and the ALS laboratory report is provided in Attachment C.

Receptor Survey

No direct contact risks were identified in shallow soil based on the field screening and analytical sampling results. No impacts to surface water were identified and there is little risk of future surface water impacts based on the site's location within the tank containment basin. No groundwater risks were identified based on the immediate nature of the response actions and as documented by the soil analytical sampling results, and no water supply wells were identified within 500 feet of the release site location.

In addition, the groundwater pathway at the Superior Refinery is addressed on a facility-wide basis through the established hydrogeologic performance standard approved by the WDNR. SRC samples the

Refinery monitoring well network (Figure 3) on a semi-annual basis and provides the data to the WDNR on an annual basis.

The nearest enclosed structures are slab-on-grade refinery buildings approximately 650 feet southeast. The risk of hazardous vapor accumulation in those structures is low due to the documented soil conditions, the distance to the structures, and the slab-on-grade construction.

Material Management

Free liquids were removed from the excavation the same day as the release with a vacuum truck and transported to the on-site API separator and treated at the on-site Wastewater Treatment Plant.

During the response excavation, soil with evidence of hydrocarbon contamination was transported to the on-site three-sided contaminated soil storage building for secure and temporary staging prior to arranging for offsite disposal (Photo 7). The contaminated soil was disposed of under the SRC's facility-specific waste profile at Shamrock Environmental, LLC landfill in Cloquet, Minnesota. A total of 45.30 tons of contaminated soil was hauled to the landfill on September 3, 2021. Waste profile documents and the landfill summary report are included in Attachment D.

Conclusions

Approximately 50 gallons of crude oil, diesel fuel, and LEL suppressant mix were found to have leaked from a crude pipeline into an open excavation in the southeast corner of the Tank 47 containment dike. Upon discovery, the leak was immediately fixed, the crude pipeline was de-inventoried, free liquids were removed, and contaminated soil was excavated. Excavated soil with evidence of hydrocarbon contamination was managed and disposed of at an offsite landfill.

Based on the field screening and confirmation analytical sample results from the final excavation limits, the release was successfully remediated with no residual soil contamination remaining above the WDNR Direct Contact or Groundwater Residual Contaminant Limits (RCLs) remains. Clean backfill will be used to replace the excavated material upon completion of the maintenance activities at this location.

Based on the information provided in this report, the spill was contained to the maintenance excavation and successfully and fully remediated within 24-36 hours of discovery. The land use at the facility is industrial and is not expected to change. The soil in the vicinity of the release were documented to be clay material with extremely low permeability (Gannett Fleming, 2014). No direct contact or vapor risks remain and no potential impacts to nearby surface water and groundwater receptors were identified.

This report provides the required documentation to demonstrate that the immediate response action is complete, and no further action is necessary to investigate or respond to this release. SRC is requesting a no further Action (NFA) determination from the WDNR for this release in accordance with NR708.09.

Reference

Gannett Fleming, 2014. *Site Investigation and Response Action Plan Calumet Superior LLC Refinery, Superior Wisconsin, WDNR BRRTS# 02-16-559511*. Prepared for Calumet Superior LLC, April 2014.

Site Photographs

- Photo 1 Oil contamination in excavation. Photo taken by SRC on August 30, 2021.
- Photo 2 Oil contamination in excavation. Photo taken by SRC on August 30, 2021.
- Photo 3 Excavation area before second trench box removed. Photo taken facing northwest on August 31, 2021.
- Photo 4 Excavation activities. Photo taken facing northwest on August 31, 2021.
- Photo 5 Final excavation extent. Photo taken facing north on August 31, 2021.
- Photo 6 Final excavation extent. Photo taken facing west on August 31, 2021.
- Photo 7 Contaminated material in three-sided building. Photo taken by Insight on August 31, 2021.

Tables

Table 1 Analytical Data Summary

Figures

- Figure 1 Site Location
- Figure 2 Site Layout
- Figure 3 Receptor Survey

Attachments

- Attachment A WDNR Hazardous Substance Discharge Notification Form and Site Contact Information
- Attachment B Site Investigation Field Sampling and Screening Log
- Attachment C ALS Laboratory Report for Confirmation Soil Samples
- Attachment D Material Management Documentation

Site Photographs



Photo 1 Oil contamination in excavation. Photo taken by SRC on August 30, 2021.



Photo 2 Oil contamination in excavation. Photo taken by SRC on August 30, 2021.



Photo 3 Excavation area before second trench box removed. Photo taken facing northwest on August 31, 2021.



Photo 4 Excavation activities. Photo taken facing northwest on August 31, 2021.



Photo 5 Final excavation extent. Photo taken facing north on August 31, 2021.



Photo 6 Final excavation extent. Photo taken facing west on August 31, 2021.



Photo 7 Contaminated material in three-sided building. Photo taken by Insight on August 31, 2021.

Tables

Table 1 Analytical Data Summary Tank 47 Crude Pipeline

			Location Date	TK47-B-1 8/31/2021	TK47-S-1 8/31/2021	TK47-S-2 8/31/2021	TK47-S-3 8/31/2021	TK47-S-4 8/31/2021	TK47-S-5 8/31/2021	TK47-S-6 8/31/2021	TK47-S-7 8/31/2021	TK47-S-8 8/31/2021
Parameter	Units	Wisconsin Groundwater RCLs, DF=2	Wisconsin Not to Exceed Direct Contact Industrial RCLs	de renaing				ac renaing		de renaing		de rending
Effective Date		12/01/2018	12/01/2018									
Exceedance Key		No Exceedances	No Exceedances									
Volatile Organic Compounds												
1,2,4-Trimethylbenzene	mg/kg	1.3787 (1)	219	< 0.037 U	< 0.034 U	< 0.038 U	< 0.039 U	< 0.04 U	< 0.033 U	< 0.035 U	< 0.036 U	< 0.036 U
1,3,5-Trimethylbenzene	mg/kg	1.3787 (1)	182	< 0.059 U	< 0.054 U	< 0.061 U	< 0.061 U	< 0.063 U	< 0.053 U	< 0.056 U	< 0.057 U	< 0.057 U
Benzene	mg/kg	0.0051	7.07	< 0.024 U	< 0.022 U	< 0.025 U	< 0.025 U	< 0.026 U	< 0.022 U	< 0.023 U	< 0.024 U	< 0.024 U
Ethyl benzene	mg/kg	1.57	35.4	< 0.011 U	< 0.0097 U	< 0.011 U	< 0.011 U	< 0.011 U	< 0.0096 U	< 0.01 U	< 0.01 U	< 0.01 U
Methyl tertiary butyl ether (MTBE)	mg/kg	0.027	282	< 0.015 U	< 0.013 U	< 0.015 U	< 0.015 U	< 0.016 U	< 0.013 U	< 0.014 U	< 0.014 U	< 0.014 U
Naphthalene	mg/kg	0.6582	24.1	< 0.12 U	< 0.11 U	< 0.13 U	< 0.13 U	< 0.13 U	< 0.11 U	< 0.12 U	< 0.12 U	< 0.12 U
Toluene	mg/kg	1.1072	818	< 0.014 U	< 0.013 U	< 0.014 U	< 0.014 U	< 0.015 U	< 0.012 U	< 0.013 U	< 0.013 U	< 0.013 U
Xylene, total	mg/kg	3.96	260	< 0.067 U	< 0.061 U	< 0.07 U	< 0.07 U	< 0.072 U	< 0.06 U	< 0.064 U	< 0.065 U	< 0.065 U
Exceedance Count	no unit		0	0	0	0	0	0	0	0	0	0
Hazard Index	no unit		≤ 1.0	0.0013	0.0012	0.0013	0.0013	0.0014	0.0011	0.0012	0.0013	0.0013
Cumulative Cancer Risk	no unit		≤ 1E-0.5	0.00000038	0.00000035	0.000000041	0.000000041	0.000000041	0.00000035	0.00000038	0.00000038	0.00000038

U The analyte was analyzed for, but was not detected.(1) Representing the criteria for combined Trimethylbenzenes.

Figures







Barr Footer: ArcGIS 10.8.1, 2021-09-16 16:13 File: 1:\Client\Husky_Energy\Superior_Refinery\Maps\Spill_Response\Tank 47\Figure 3 Receptor Survey.mxd User: MAK3

Attachments

Attachment A

WDNR Hazardous Substance Discharge Notification Form

Hazardous Substance Discharge Notification Form - NR 706.05

Superior Refining Company LLC 2407 Stinson Ave., Superior, WI 54880 Phone: (715) 398-3533 Fax: (715) 398-8209

Refinery Map Coordinates: NW1/4, NW1/4, Sect. 36, T 49N, R 14N.

1) Reporting Information

<i>(</i> .	
Name:	Matt Turner
Date:	8/30/2021
Phone:	715-969-4873
Position:	Env. Technologist

2) Discharge Information

Date:	8/30/2021	
Time:	10:00	
Amount Re	leased:	~50 gallons
Duration:	Unknown	
Material/Pr	oduct:	Crude oil, diesel fuel and LEL suppressant mix
Response	Time:	Immediate
Specific Lo	cation:	In the pipeline excavation in the SE corner of the Tank 47 containment dike
How was s	pill detected:	Operator checked the excavation for water following recent rains and saw the oil
Cause:	A cold tap was	s installed on the pipeline but it began to leak sometime after it was installed.

If necessary, continue on back

3) Additional Information

Physical Characteristics:		Black liquid	ł			
Chemical Properties:						
Possible Hazards:						
Immediate Corrective Acti	ion/Clean-up):	Free liquid was vacuumed up; pan was put under the leaking valve until pipeline is deinventoried			
People/Companies Perfor	ming the Ac	tion:	Ops, shift f	oreman, pipeline projec	et management, WCS	
Speed and Movement of I	Discharge (if	any):	N/A			
Actual/Potential Impacts to	o Human He	ealth(if any):		N/A		
Actual/Potential Impacts to	o Environme	ent(if any):		Reportable spill immediately cleaned up		
Weather Conditions (i.e. precipitation, wind speed and direction):			Partly cloudy, 75 degrees F, wind NW12			
Agencies On-scene Durin	g Spill (if an	y):	None			
Further action needed (if a	any):	Cleanup af	fected soil of	once pipeline has been	emptied	
Amount reaching Navigab	le Waters:		None			
Total Oil Storage Capacity	y of Tanks/Li	ines Materia	al was Disch	arged From:	TBD	
Adequate Secondary Containment: Yes						
Steps taken to Reduce Po	Recurrence:		Review cold tap proce	dure		
Enforcement Actions (if any): N/A						
Effectiveness of Monitorin	g Equipmen	t (if any):		N/A		

Original: Refinery Manager CC: Operations Manager, Environmental Manager

Tank 47 Release Site and Facility Contact Information

Site Information:	WDNR SERTS Number: 20210830NO16-1
	Facility Identification Number: 816009590
	Superior Refining Company LLC
	2407 Stinson Avenue
	Superior, Wisconsin
	Douglas County, Wisconsin
	SW ¼, SW ¼ of Section 25, T49N, R14W
	Latitude / Longitude: 46.69276 / 92.07450
	WTM91 Coordinates: X: 361386, Y: 693110
Responsible Party:	Superior Refining Company LLC
	Attn: Matt Turner, Environmental Technologist
	2407 Stinson Avenue
	Superior, WI 54880
	Phone: (403) 298-6050
	Email: matthew.turner@cenovous.com
Environmental Consultant:	Barr Engineering Co.
	Attn: Lynette Carney, Project Manager
	325 South Lake Avenue, Suite 700
	Duluth, MN 55802
	Phone: (218) 529-7141
	Email: lcarney@barr.com

Attachment B

Site Investigation Field Sampling and Screening Log

			100							Page of
FIELD SAMPLING	AND SC	REENING	Data	8/31/2	21	٥	Equipment: Photoionization detector	with 10 eV bulb		
Client: <u>SKC</u>	- 1JT	TH UT	Sampler	Tim Cu	Garer	+ Ecolo	La ca	Calibration	Bump Test 1	Bump Test 2
Sample Nomenclati	ure (Loco	tion - san	nple type	- #):		- jest i	Time	1100		
$\mathbf{R} = \text{Removed } \mathbf{S} = \text{Side}$	ewall B =	Bottom S	tockpile =	Stockpile			Zero reading (ppm)	0.0		
							Span reading (ppm)	6.00		
			Soil	Colori	Odor/	Headspace	Background (ppm)	0.5	reas, sample locations ,bo	rings, wells, structures,
Commiss ID	Depth	Time	Туре	Discolor	Sheen	Reading	Site Sketch: north arrow, scale, excavation utilities, natural features	rextents & depths, impacted a		
Sample D	(FT) 	(mintory) 16:30	CL	Reddish brown	Petroleum/	275		ad	0 0	and the second second
Carl Ala		11:47	Sal	Brado	Rainbow	0.2	8/31/21:(11:28):2	box remose	I from crica	retion
Sand Ulean	8'011	11-11	110	0 al	Norie	742			$e^{-i\omega - i\omega}$	A State Val
<u>KI</u>	191	12:00	Gravel Sel	Brinne	none	07				1.1/
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) Z Remare	9	14.16	CH	Brown/Nue	Styn # The	75.0	1	2.9 15-2		
53	5'	14.18	ICH	Brown/None	None Mare	20	L' K	1 TKWI	N	/
54	5'	14:21	CH	Brown/Nove	Nore More	0.1				4 7 W
B B B 1	11'	14:23	CH	Brown/Nine	Nore Nore	0.7	051		+	
132	11'	14:25	CH	Brown Noe	None Inval	1.0	and the second	5957		
55	381	14:29	CH	Brann Norl	None / None	1.2				
B3	11'	16:20	CH	Brand Nove	Nare Nore	1.6		h	7 6-	4
56	9'	16:25	CH	Brown/Nac	Nove/Nare	0.7	3	8	12-1	5
57	2'	16:30	CH	Benn/Nore	None Was	1.1	52		N TE W7-S	5
58	91	16:33	CH	Grown None	None None	1.1	N 655		S TR	· · · · · · · · · · · · · · · · · · ·
59	9'	16-37	(H	Brown Inone	Nor pac	1.2	1 640		3	
510	2'	16:41	CH	Braven /None	None None	0.9	10 9411	a Gr	<i>L'</i>	\rightarrow
511	1'	16:44	CH	Brown Nere	None None	1.4	al -			of the second se
512	5	11:48	CH	Grann Mise	Abre Nere	1.2	X		~	X
513	12.	11:52	- CK	Brown Nor	e None Nord	1.2	0		0	
5()	- F	(10.0.								
	_						-	12 R1		
								Sold Sit		
							1 108	1 147-5-	to ul	X 7 7 1
							TK47-5-0-	Road 14	(serbix)	
	1	500							,	

Attachment C

ALS Laboratory Report for Confirmation Soil Samples



07-Sep-2021

Lynette Carney Barr Engineering Company 4300 Market Pointe Drive Suite 200 Minneapolis, MN 55435

Re: SRC TK47 (49161468.02 300 301)

Work Order: 21090215

Dear Lynette,

ALS Environmental received 9 samples on 02-Sep-2021 09:00 AM for the analyses presented in the following report.

The analytical data provided relates directly to the samples received by ALS Environmental - Holland and for only the analyses requested.

Sample results are compliant with industry accepted practices and Quality Control results achieved laboratory specifications. Any exceptions are noted in the Case Narrative, or noted with qualifiers in the report or QC batch information. Should this laboratory report need to be reproduced, it should be reproduced in full unless written approval has been obtained from ALS Environmental. Samples will be disposed in 30 days unless storage arrangements are made.

The total number of pages in this report is 18.

If you have any questions regarding this report, please feel free to contact me:

ADDRESS: 3352 128th Avenue, Holland, MI, USA PHONE: +1 (616) 399-6070 FAX: +1 (616) 399-6185

Sincerely,

Lodi Blouw

Electronically approved by: Jodi Blouw

Environmental 💭

Jodi Blouw

Report of Laboratory Analysis

Certificate No: WI: 399084510

ALS GROUP USA, CORP Part of the ALS Laboratory Group A Campbell Brothers Limited Company

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RIGHT SOLUTIONS RIGHT PARTNER

Date: 07-Sep-21

Client:	Barr Engineering Company
Project:	SRC TK47 (49161468.02 300 301)
Work Order:	21090215

Work Order Sample Summary

Lab Samp ID	<u>Client Sample ID</u>	<u>Matrix</u>	Tag Number	Collection Date	Date Received	<u>Hold</u>
21090215-01	TK47-B-1	Soil		8/31/2021 16:20	9/2/2021 09:00	
21090215-02	TK47-S-1	Soil		8/31/2021 16:25	9/2/2021 09:00	
21090215-03	TK47-S-2	Soil		8/31/2021 16:30	9/2/2021 09:00	
21090215-04	TK47-S-3	Soil		8/31/2021 16:33	9/2/2021 09:00	
21090215-05	TK47-S-4	Soil		8/31/2021 16:37	9/2/2021 09:00	
21090215-06	TK47-S-5	Soil		8/31/2021 16:41	9/2/2021 09:00	
21090215-07	TK47-S-6	Soil		8/31/2021 16:44	9/2/2021 09:00	
21090215-08	TK47-S-7	Soil		8/31/2021 16:48	9/2/2021 09:00	
21090215-09	TK47-S-8	Soil		8/31/2021 16:52	9/2/2021 09:00	

Client:	Barr Engineering Company	OUALIFIERS
Project:	SRC TK47 (49161468.02 300 301)	ACDONVMS UNITS
WorkOrder:	21090215	ACKON HVIS, UNITS

Qualifier	Description	
*	Value exceeds Regulatory Limit	
**	Estimated Value	

a Analyte is non-accreditedB Analyte detected in the associated Method Blank above the Reporting Limit

- E Value above quantitation range
- H Analyzed outside of Holding Time
 - Hr BOD/CBOD Sample was reset outside Hold Time, value should be considered estimated.
 - J Analyte is present at an estimated concentration between the MDL and Report Limit
 - ND Not Detected at the Reporting Limit
- O Sample amount is > 4 times amount spiked
- P Dual Column results percent difference > 40%
- R RPD above laboratory control limit
- S Spike Recovery outside laboratory control limits
- U Analyzed but not detected above the MDL
- X Analyte was detected in the Method Blank between the MDL and Reporting Limit, sample results may exhibit background or reagent contamination at the observed level.

Acronym Description

DUP	Method Duplicate
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection (see MDL)
LOQ	Limit of Quantitation (see PQL)
MBLK	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
PQL	Practical Quantitation Limit
RPD	Relative Percent Difference
TDL	Target Detection Limit
TNTC	Too Numerous To Count
А	APHA Standard Methods
D	ASTM
Е	EPA
SW	SW-846 Update III
Units Reported	Description
% of sample	Percent of Sample
µg/Kg-dry	Micrograms per Kilogram Dry Weight

Date: 07-Sep-21

Client:	Barr Engineering Company	
Project:	SRC TK47 (49161468.02 300 301)	Case Narrative
Work Order:	21090215	

Samples for the above noted Work Order were received on 09/02/2021. The attached "Sample Receipt Checklist" documents the status of custody seals, container integrity, preservation, and temperature compliance.

Samples were analyzed according to the analytical methodology previously transmitted in the "Work Order Acknowledgement". Methodologies are also documented in the "Analytical Result" section for each sample. Quality control results are listed in the "QC Report" section. Sample association for the reported quality control is located at the end of each batch summary. If applicable, results are appropriately qualified in the Analytical Result and QC Report sections. The "Qualifiers" section documents the various qualifiers, units, and acronyms utilized in reporting. A copy of the laboratory's scope of accreditation is available upon request.

With the following exceptions, all sample analyses achieved analytical criteria.

Volatile Organics: No deviations or anomalies were noted.

Wet Chemistry: No deviations or anomalies were noted.

Client:	Barr Engineering Company	
Project:	SRC TK47 (49161468.02 300 301)	Work Order: 21090215
Sample ID:	TK47-B-1	Lab ID: 21090215-01
Collection Date:	8/31/2021 04:20 PM	Matrix: SOIL

Analyses	Result Q	Qual MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U	37	120	µg/Kg-dry	1	9/4/2021 07:51
1,3,5-Trimethylbenzene	U	59	200	µg/Kg-dry	1	9/4/2021 07:51
Benzene	U	24	81	µg/Kg-dry	1	9/4/2021 07:51
Ethylbenzene	U	11	36	µg/Kg-dry	1	9/4/2021 07:51
Methyl tert-butyl ether	U	15	49	µg/Kg-dry	1	9/4/2021 07:51
Naphthalene	U	120	400	µg/Kg-dry	1	9/4/2021 07:51
Toluene	U	14	46	µg/Kg-dry	1	9/4/2021 07:51
Xylenes, Total	U	67	220	µg/Kg-dry	1	9/4/2021 07:51
Surr: 1,2-Dichloroethane-d4	100		70-130	%REC	1	9/4/2021 07:51
Surr: 4-Bromofluorobenzene	102		70-130	%REC	1	9/4/2021 07:51
Surr: Dibromofluoromethane	97.4		70-130	%REC	1	9/4/2021 07:51
Surr: Toluene-d8	99.2		70-130	%REC	1	9/4/2021 07:51
MOISTURE		Method: SW3550C				Analyst: ALG
Moisture	31	0.10	0.10	% of sample	1	9/3/2021 11:33

Client:	Barr Engineering Company	
Project:	SRC TK47 (49161468.02 300 301)	Work
Sample ID:	TK47-S-1	I
Collection Date:	8/31/2021 04:25 PM	I

Work Order: 21090215 **Lab ID:** 21090215-02 **Matrix:** SOIL

Analyses	Result Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS	Me	ethod: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U	34	110	µg/Kg-dry	1	9/4/2021 08:10
1,3,5-Trimethylbenzene	U	54	180	µg/Kg-dry	1	9/4/2021 08:10
Benzene	U	22	74	µg/Kg-dry	1	9/4/2021 08:10
Ethylbenzene	U	9.7	32	µg/Kg-dry	1	9/4/2021 08:10
Methyl tert-butyl ether	U	13	44	µg/Kg-dry	1	9/4/2021 08:10
Naphthalene	U	110	370	µg/Kg-dry	1	9/4/2021 08:10
Toluene	U	13	42	µg/Kg-dry	1	9/4/2021 08:10
Xylenes, Total	U	61	200	µg/Kg-dry	1	9/4/2021 08:10
Surr: 1,2-Dichloroethane-d4	98.2		70-130	%REC	1	9/4/2021 08:10
Surr: 4-Bromofluorobenzene	102		70-130	%REC	1	9/4/2021 08:10
Surr: Dibromofluoromethane	97.0		70-130	%REC	1	9/4/2021 08:10
Surr: Toluene-d8	97.5		70-130	%REC	1	9/4/2021 08:10
MOISTURE	Me	ethod: SW3550C				Analyst: ALG
Moisture	26	0.10	0.10	% of sample	1	9/3/2021 11:33

Client:	Barr Engineering Company		
Project:	SRC TK47 (49161468.02 300 301)	Work Order:	21090215
Sample ID:	TK47-S-2	Lab ID:	21090215-03
Collection Date:	8/31/2021 04:30 PM	Matrix:	SOIL

Analyses	Result Qua	I MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS	Ν	/lethod: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U	38	130	µg/Kg-dry	1	9/4/2021 08:30
1,3,5-Trimethylbenzene	U	61	200	µg/Kg-dry	1	9/4/2021 08:30
Benzene	U	25	85	µg/Kg-dry	1	9/4/2021 08:30
Ethylbenzene	U	11	37	µg/Kg-dry	1	9/4/2021 08:30
Methyl tert-butyl ether	U	15	50	µg/Kg-dry	1	9/4/2021 08:30
Naphthalene	U	130	420	µg/Kg-dry	1	9/4/2021 08:30
Toluene	U	14	48	µg/Kg-dry	1	9/4/2021 08:30
Xylenes, Total	U	70	230	µg/Kg-dry	1	9/4/2021 08:30
Surr: 1,2-Dichloroethane-d4	101		70-130	%REC	1	9/4/2021 08:30
Surr: 4-Bromofluorobenzene	104		70-130	%REC	1	9/4/2021 08:30
Surr: Dibromofluoromethane	99.1		70-130	%REC	1	9/4/2021 08:30
Surr: Toluene-d8	101		70-130	%REC	1	9/4/2021 08:30
MOISTURE	Ν	lethod: SW3550C				Analyst: ALG
Moisture	26	0.10	0.10	% of sample	1	9/3/2021 11:33

Client:	Barr Engineering Company		
Project:	SRC TK47 (49161468.02 300 301)	Work Order:	21090215
Sample ID:	TK47-S-3	Lab ID:	21090215-04
Collection Date:	8/31/2021 04:33 PM	Matrix:	SOIL

Analyses	Result Q	ual MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U	39	130	µg/Kg-dry	1	9/4/2021 08:49
1,3,5-Trimethylbenzene	U	61	200	µg/Kg-dry	1	9/4/2021 08:49
Benzene	U	25	85	µg/Kg-dry	1	9/4/2021 08:49
Ethylbenzene	U	11	37	µg/Kg-dry	1	9/4/2021 08:49
Methyl tert-butyl ether	U	15	51	µg/Kg-dry	1	9/4/2021 08:49
Naphthalene	U	130	420	µg/Kg-dry	1	9/4/2021 08:49
Toluene	U	14	48	µg/Kg-dry	1	9/4/2021 08:49
Xylenes, Total	U	70	230	µg/Kg-dry	1	9/4/2021 08:49
Surr: 1,2-Dichloroethane-d4	99.3		70-130	%REC	1	9/4/2021 08:49
Surr: 4-Bromofluorobenzene	103		70-130	%REC	1	9/4/2021 08:49
Surr: Dibromofluoromethane	99.7		70-130	%REC	1	9/4/2021 08:49
Surr: Toluene-d8	99.2		70-130	%REC	1	9/4/2021 08:49
MOISTURE		Method: SW3550C				Analyst: ALG
Moisture	28	0.10	0.10	% of sample	1	9/3/2021 11:33

Client:	Barr Engineering Company		
Project:	SRC TK47 (49161468.02 300 301)	Work Order:	21090215
Sample ID:	TK47-S-4	Lab ID:	21090215-05
Collection Date:	8/31/2021 04:37 PM	Matrix:	SOIL

Analyses	Result Qua	al MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U	40	130	µg/Kg-dry	1	9/4/2021 09:09
1,3,5-Trimethylbenzene	U	63	210	µg/Kg-dry	1	9/4/2021 09:09
Benzene	U	26	88	µg/Kg-dry	1	9/4/2021 09:09
Ethylbenzene	U	11	38	µg/Kg-dry	1	9/4/2021 09:09
Methyl tert-butyl ether	U	16	52	µg/Kg-dry	1	9/4/2021 09:09
Naphthalene	U	130	430	µg/Kg-dry	1	9/4/2021 09:09
Toluene	U	15	49	µg/Kg-dry	1	9/4/2021 09:09
Xylenes, Total	U	72	240	µg/Kg-dry	1	9/4/2021 09:09
Surr: 1,2-Dichloroethane-d4	101		70-130	%REC	1	9/4/2021 09:09
Surr: 4-Bromofluorobenzene	105		70-130	%REC	1	9/4/2021 09:09
Surr: Dibromofluoromethane	97.7		70-130	%REC	1	9/4/2021 09:09
Surr: Toluene-d8	98.4		70-130	%REC	1	9/4/2021 09:09
MOISTURE		Method: SW3550C				Analyst: ALG
Moisture	30	0.10	0.10	% of sample	1	9/3/2021 11:33

Barr Engineering Company	
SRC TK47 (49161468.02 300 301)	Work Order
TK47-S-5	Lab ID
8/31/2021 04:41 PM	Matrix
	Barr Engineering Company SRC TK47 (49161468.02 300 301) TK47-S-5 8/31/2021 04:41 PM

Work Order: 21090215 **Lab ID:** 21090215-06 **Matrix:** SOIL

Analyses	Result Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS	Ме	thod: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U	33	110	µg/Kg-dry	1	9/4/2021 09:28
1,3,5-Trimethylbenzene	U	53	180	µg/Kg-dry	1	9/4/2021 09:28
Benzene	U	22	73	µg/Kg-dry	1	9/4/2021 09:28
Ethylbenzene	U	9.6	32	µg/Kg-dry	1	9/4/2021 09:28
Methyl tert-butyl ether	U	13	44	µg/Kg-dry	1	9/4/2021 09:28
Naphthalene	U	110	360	µg/Kg-dry	1	9/4/2021 09:28
Toluene	U	12	41	µg/Kg-dry	1	9/4/2021 09:28
Xylenes, Total	U	60	200	µg/Kg-dry	1	9/4/2021 09:28
Surr: 1,2-Dichloroethane-d4	101		70-130	%REC	1	9/4/2021 09:28
Surr: 4-Bromofluorobenzene	103		70-130	%REC	1	9/4/2021 09:28
Surr: Dibromofluoromethane	102		70-130	%REC	1	9/4/2021 09:28
Surr: Toluene-d8	98.8		70-130	%REC	1	9/4/2021 09:28
MOISTURE	Ме	thod: SW3550C				Analyst: ALG
Moisture	25	0.10	0.10	% of sample	1	9/3/2021 11:33

Client:	Barr Engineering Company	
Project:	SRC TK47 (49161468.02 300 301)	Work Order:
Sample ID:	TK47-S-6	Lab ID:
Collection Date:	8/31/2021 04:44 PM	Matrix:

21090215 21090215-07 SOIL

Analyses	Result Q	ual MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Method: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U	35	120	µg/Kg-dry	1	9/4/2021 09:48
1,3,5-Trimethylbenzene	U	56	190	µg/Kg-dry	1	9/4/2021 09:48
Benzene	U	23	78	µg/Kg-dry	1	9/4/2021 09:48
Ethylbenzene	U	10	34	µg/Kg-dry	1	9/4/2021 09:48
Methyl tert-butyl ether	U	14	46	µg/Kg-dry	1	9/4/2021 09:48
Naphthalene	U	120	390	µg/Kg-dry	1	9/4/2021 09:48
Toluene	U	13	44	µg/Kg-dry	1	9/4/2021 09:48
Xylenes, Total	U	64	210	µg/Kg-dry	1	9/4/2021 09:48
Surr: 1,2-Dichloroethane-d4	103		70-130	%REC	1	9/4/2021 09:48
Surr: 4-Bromofluorobenzene	103		70-130	%REC	1	9/4/2021 09:48
Surr: Dibromofluoromethane	101		70-130	%REC	1	9/4/2021 09:48
Surr: Toluene-d8	98.8		70-130	%REC	1	9/4/2021 09:48
MOISTURE		Method: SW3550C				Analyst: ALG
Moisture	24	0.10	0.10	% of sample	1	9/3/2021 11:33

Client:	Barr Engineering Company	
Project:	SRC TK47 (49161468.02 300 301)	Work O
Sample ID:	TK47-S-7	Lal
Collection Date:	8/31/2021 04:48 PM	Ma

Work Order: 21090215 **Lab ID:** 21090215-08 **Matrix:** SOIL

Analyses	Result Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS	Met	hod: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U	36	120	µg/Kg-dry	1	9/4/2021 10:08
1,3,5-Trimethylbenzene	U	57	190	µg/Kg-dry	1	9/4/2021 10:08
Benzene	U	24	79	µg/Kg-dry	1	9/4/2021 10:08
Ethylbenzene	U	10	35	µg/Kg-dry	1	9/4/2021 10:08
Methyl tert-butyl ether	U	14	47	µg/Kg-dry	1	9/4/2021 10:08
Naphthalene	U	120	390	µg/Kg-dry	1	9/4/2021 10:08
Toluene	U	13	45	µg/Kg-dry	1	9/4/2021 10:08
Xylenes, Total	U	65	220	µg/Kg-dry	1	9/4/2021 10:08
Surr: 1,2-Dichloroethane-d4	99.7		70-130	%REC	1	9/4/2021 10:08
Surr: 4-Bromofluorobenzene	105		70-130	%REC	1	9/4/2021 10:08
Surr: Dibromofluoromethane	100		70-130	%REC	1	9/4/2021 10:08
Surr: Toluene-d8	100		70-130	%REC	1	9/4/2021 10:08
MOISTURE	Met	hod: SW3550C				Analyst: ALG
Moisture	23	0.10	0.10	% of sample	1	9/3/2021 11:33

Client:	Barr Engineering Company		
Project:	SRC TK47 (49161468.02 300 301)	Work Order:	21090215
Sample ID:	TK47-S-8	Lab ID:	21090215-09
Collection Date:	8/31/2021 04:52 PM	Matrix:	SOIL

Analyses	Result	Qual	MDL	PQL	Units	Dilution Factor	Date Analyzed
VOLATILE ORGANIC COMPOUNDS		Meth	nod: SW8260C				Analyst: HJ
1,2,4-Trimethylbenzene	U		36	120	µg/Kg-dry	1	9/4/2021 10:27
1,3,5-Trimethylbenzene	U		57	190	µg/Kg-dry	1	9/4/2021 10:27
Benzene	U		24	79	µg/Kg-dry	1	9/4/2021 10:27
Ethylbenzene	U		10	34	µg/Kg-dry	1	9/4/2021 10:27
Methyl tert-butyl ether	U		14	47	µg/Kg-dry	1	9/4/2021 10:27
Naphthalene	U		120	390	µg/Kg-dry	1	9/4/2021 10:27
Toluene	U		13	44	µg/Kg-dry	1	9/4/2021 10:27
Xylenes, Total	U		65	220	µg/Kg-dry	1	9/4/2021 10:27
Surr: 1,2-Dichloroethane-d4	101			70-130	%REC	1	9/4/2021 10:27
Surr: 4-Bromofluorobenzene	103			70-130	%REC	1	9/4/2021 10:27
Surr: Dibromofluoromethane	99.9			70-130	%REC	1	9/4/2021 10:27
Surr: Toluene-d8	98.6			70-130	%REC	1	9/4/2021 10:27
MOISTURE		Meth	nod: SW3550C				Analyst: ALG
Moisture	23		0.10	0.10	% of sample	1	9/3/2021 11:33

Client:	Barr Engineering Company
Work Order:	21090215
Project:	SRC TK47 (49161468.02 300 301)

QC BATCH REPORT

Batch ID: 183138w

Instrument ID VMS8

Method: SW8260C

MBLK Sam	ple ID: MBLK-18313	8-183138w			Ur	nits: µg/K	(g-dry	Analysi	Analysis Date: 9/4/2021 07:31 AM			
Client ID:		Run ID: VMS	8_2109	03B	Seq	No: 7724	617	Prep Date: 9/3/2	2021	DF: 1		
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual	
1,2,4-Trimethylbenzene	U	22	73									
1,3,5-Trimethylbenzene	U	35	120									
Benzene	U	15	48									
Ethylbenzene	U	6.3	21									
Methyl tert-butyl ether	U	8.6	29									
Naphthalene	U	72	240									
Toluene	U	8.2	27									
Xylenes, Total	U	40	130									
Surr: 1,2-Dichloroethane	e-d4 1012	0	0	1000	0	101	70-130	0				
Surr: 4-Bromofluorobenz	tene 1020	0	0	1000	0	102	70-130	0				
Surr: Dibromofluorometh	nane 979.5	0	0	1000	0	98	70-130	0				
Surr: Toluene-d8	984.5	0	0	1000	0	98.4	70-130	0				

LCS Sample ID: LCS-183138-183138w				U	Units: µg/Kg-dry			Analysi	s Date:	9/4/2021 06	:33 AM	
Client ID:	Run ID: VMS8_210903B		03B	See	SeqNo: 7724615 Prep			ate: 9/3/2	2021	DF: 1		
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	R	PD Ref Value	%RPE	RPD Limit	Qual
1,2,4-Trimethylbenzene	e 811	22	73	1000	0	81.1	65-135		0			
1,3,5-Trimethylbenzene	862.5	35	120	1000	0	86.2	65-135		0			
Benzene	899.5	15	48	1000	C	90	75-125		0			
Ethylbenzene	855	6.3	21	1000	C	85.5	75-125		0			
Methyl tert-butyl ether	767.5	8.6	29	1000	C	76.8	75-125		0			
Naphthalene	826.5	72	240	1000	C	82.6	40-140		0			
Toluene	895	8.2	27	1000	C	89.5	70-125		0			
Xylenes, Total	2582	40	130	3000	C	86.1	75-125		0			
Surr: 1,2-Dichloroeth	ane-d4 987	0	0	1000	C	98.7	70-130		0			
Surr: 4-Bromofluorob	enzene 1016	0	0	1000	C	102	70-130		0			
Surr: Dibromofluoron	nethan 999	0_	0	1000	C	99.9	70-130		0			
Surr: Toluene-d8	1010	0	0	1000	C	101	70-130		0			

Batch ID: 183138w

Instrument ID VMS8

Method: SW8260C

MS	Sample ID: 21	1090215-01/	A MS			Ur	its: µg/K	g-dry	Analysis	s Date: 9	/4/2021 11	:06 AM
Client ID: TK47-B-1			Run ID: VMS8_210903B			Seq	No: 7724	628	Prep Date: 9/3/2	DF: 1		
Analyte		Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzen	е	1744	42	140	1899	0	91.9	65-135	0			
1,3,5-Trimethylbenzen	e	1821	66	220	1899	0	95.9	65-135	0			
Benzene		1937	28	92	1899	0	102	75-125	0			
Ethylbenzene		1794	12	40	1899	0	94.5	75-125	0			
Methyl tert-butyl ether		1902	16	55	1899	0	100	75-125	0			
Naphthalene		1679	140	460	1899	0	88.5	40-140	0			
Toluene		1831	16	52	1899	0	96.4	70-125	0			
Xylenes, Total		5382	76	250	5696	0	94.5	75-125	0			
Surr: 1,2-Dichloroet	hane-d4	1876	0	0	1899	0	98.8	70-130	0			
Surr: 4-Bromofluoro	benzene	1915	0	0	1899	0	101	70-130	0			
Surr: Dibromofluoro	methane	1893	0	0	1899	0	99.7	70-130	0			
Surr: Toluene-d8		1854	0	0	1899	0	97.6	70-130	0			

MSD S	1A MSD		Ur	nits: µg/K	g-dry	Analysi	Analysis Date: 9/4/2021 11				
Client ID: TK47-B-1		Run ID: VM	S8_2109	03B	Seq	No: 7724	629	Prep Date: 9/3/2	2021	DF: 1	
Analyte	Result	MDL	PQL	SPK Val	SPK Ref Value	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
1,2,4-Trimethylbenzene	1693	42	140	1899	0	89.2	65-135	1744	2.98	30	
1,3,5-Trimethylbenzene	1786	66	220	1899	0	94.1	65-135	1821	1.95	30	
Benzene	1872	28	92	1899	0	98.6	75-125	1937	3.39	30	
Ethylbenzene	1746	12	40	1899	0	92	75-125	1794	2.74	30	
Methyl tert-butyl ether	1800	16	55	1899	0	94.8	75-125	1902	5.54	30	
Naphthalene	1694	140	460	1899	0	89.3	40-140	1679	0.9	30	
Toluene	1776	16	52	1899	0	93.5	70-125	1831	3.05	30	
Xylenes, Total	5249	76	250	5696	0	92.2	75-125	5382	2.5	30	
Surr: 1,2-Dichloroetha	ane-d4 1872	0	0	1899	0	98.6	70-130	1876	0.203	30	
Surr: 4-Bromofluorob	enzen 1878	0	0	1899	0	98.9	70-130	1915	1.95	30	
Surr: Dibromofluorom	ethan 1893	0_	0	1899	0	99.7	70-130	1893	0	30	
Surr: Toluene-d8	1843	0	0	1899	0	97.1	70-130	1854	0.565	30	
The following samples	were analyzed in this	s batch:	210902 210902	215-01A 215-04A	210902 210902	215-02A 215-05A	21 21	090215-03A 090215-06A			

21090215-08A

21090215-09A

21090215-07A

QC BATCH REPORT

Batch ID: R326061 Instrument ID MOIST Method: SW3550C

MBLK	Sample ID: WBLKS-R326	6061			Un	its: % of	sample	Analysis	Date: §	9/3/2021 [·]	11:33 AM
Client ID:		Run ID: MO	IST_210903A		Seq	No: 7723	407	Prep Date:		DF: 1	l I
Analyte	Result	MDL	PQL SPK Val	SPK Val	Ref ue	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	U	0.1	0.10								
LCS	Sample ID: LCS-R326061				Un	its: % of	sample	Analysis	Date: 9	9/3/2021 [·]	11:33 AM
Client ID:		Run ID: MO	IST_210903A		Seq	No: 7723	406	Prep Date:		DF: 1	
Analyte	Result	MDL	PQL_SPK Val	SPK Val	Ref ue	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	99.97	0.1	0.10 100		0	100	98-102	0			
DUP	Sample ID: 21082361-01/	A DUP			Un	its: % of	sample	Analysis	Date: 9	9/3/2021 ⁻	11:33 AM
Client ID:		Run ID: MO	IST_210903A		Seq	No: 7723	385	Prep Date:		DF: 1	I
Analyte	Result	MDL	PQL_SPK Val	SPK Val	Ref ue	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	40.78	0.1	0.10 0		0	0	0-0	40.6	0.442	2 10	l
DUP	Sample ID: 21082612-01/	A DUP			Un	its: % of	sample	Analysis	Date: 9	9/3/2021 ⁻	11:33 AM
Client ID:		Run ID: MO	IST_210903A		Seq	No: 7723	388	Prep Date:		DF: 1	l
Analyte	Result	MDL	PQL SPK Val	SPK Val	Ref ue	%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture	17.25	0.1	0.10 0		0	0	0-0	16.99	1.52	2 10	1
The following s	samples were analyzed in this l	batch:	21090215-01B 21090215-04B 21090215-07B	2 ² 2 ² 2 ²	10902 ⁻ 10902 ⁻ 10902 ⁻	15-02B 15-05B 15-08B	21 21 21	090215-03B 090215-06B 090215-09B			

21090215

BARR Barr Engineering Co.	Cha	in o	F Cus	stody				-	<u> </u>								<u> </u>		~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~
Sample Origination State			r Cus	louy				I	╞	Ana	alysis Re	quested			COC Numb	er: Ng	589	644	
	□мо		🗆 ТХ	🗆 υτ	Xw	Other:				Water				1	coc	of	<u> </u>		
REPORT TO				IN	VOICE 1	0		1							Matrix (Code:	Preser	vative Co	de:
Company: Barr Engineering Co.		Comp	any:	Barr	·			1							GW = Grou	indwater	A =	None	
Address: 325 S. Late Ame		Addre	ess:	1					lers					ļ	SW = Surfa WW = Was	ace Water	B =	HCI HNO	
Address: Drinth, MN 55802	,	Addre	ess:					Z	tair				1		DW = Drin	king Water	D =	H₁₂SO₄	
Name: Lynethe Carney		Name	2					1≻	5				1	É	S = Soil/ SD = Sedi	Solid ment	E = F =	NaOH MeOH	
email: 1000000 Chave. com		email		V				۵	5					40	O = Othe	er	G =	NaHSO₄	,
Copy to: BarrDM@barr.com		P.O.	~					Ň	2				1	Ż			H =	Na ₂ S ₂ O Ascorbic	³ Acid
Project Name: SPC TK47		Barr	Project I	No: 491	614680	02 300 30	>1	MS	a m b					olids] =	Zn Acet	ate
	San	nple De	epth	Coll	ection	Collection	I	Ę	Ž				ć	S %			K =	Other	JN
Location	Start	Stop	Unit (m /ft	D	ate	Time	Matrix	rfoi	ta				Ŧ	A	Preservative	Code			
		ļ	or in.)	(mm/c	ld/yyyy)	(hh:mm)		Pe	P				^	JN	Field Filtered	I Y/N			
1. TK47-B-1	()	ι	f4	08/3	12021	1620	5	N	3				y	X	Δ	CAP	-	AT	
2 TK47-S-1	٩	9	4			1625	5	N	3				X	Ϋ́	7			j	
^{3.} TK47-5-2	2	2	f t			1630	5	N	3				X	¢χ					
4. TK47-5-3	٩	9	F+			1633	5	N	3)	(x					
5. TK 47-5-4	9	9	64			1637	5	N	3)	ſχ					
6. TK47-5-5	2	2	F4			1641	5	N	3				1	(x					
7. TK47-5-6	Í	١	F+			1644	5	W	3				γ	ίx,					
^{8.} TK47-5-7	9	9	F1			1648	5	N	3				X	Ύ					
^{9.} TK47-5-8	2	2	F4	J	/	1652	5	N	3				y	ίx		\neg	i		
10.																			
BARR USE ONLY	.	Relina	uished t	oy: N/m	d Al	A Qu	lce?	Date		Time	Receive	ed by:		ل ــــلـــ	L	I	Date	Time	e
Sampled by: KMJ3/GE				· µu	v~ / _//	1 0	N 9//	1/1	-4	1515			ŤÉ	Dt	57			<u> </u>	
Barr Proj. Manager: LMC		Kelinq	Relinquished by: FEDEL On Ice?					yate			Receive	by:					Date	Tim	e
Barr DQ Manager: JET		Samples Shipped VIA: Ground Courier					←r∡ \ir C	arrier		Air Bill	Number	/1_// r:			Reau	ested [L Due Date	:	
Lab Name: Also HADAJ3	1/1/1/3 🗌 Sampler 🗌 Other:								4.1%	1	F3		Standa		Around Ti	me			
Lab Location: Oracin Bury		Lab W	/0:			Temperature on	Receipt	(°C):	Custody	Seal li	ntact? 🗆	ΥŪ	⊒N	□ None	Rush	H)/f } (mm/dd/y	<u>'</u>] / [l i

Distribution - White-Original: Accompanies Shipment to Laboratory; Yellow Copy: Include in Field Documents; Scan and email: a copy to BarrDM@barr.com for tracking and filing procedures

Sample Receipt Checklist

Client Name: BARRENG-MN		Date/Time I	Received:	<u>02-Sep-2</u>	<u>1 09:00</u>
Work Order: 21090215		Received b	y:	<u>KRW</u>	
Checklist completed by Keith Wierenga (02-Sep-21 Date	Reviewed by:	<u>Locli Blou</u> eSignature	ue	02-Sep-21 Date
Matrices: <u>Soil</u> Carrier name: <u>FedEx</u>					I
Shipping container/cooler in good condition?	Yes 🗸	No 🗌	Not Pres	ent	
Custody seals intact on shipping container/cooler?	Yes	No 🗌	Not Pres	ent 🗹	
Custody seals intact on sample bottles?	Yes	No 🗌	Not Pres	ent 🗹	
Chain of custody present?	Yes 🗸	No 🗌			
Chain of custody signed when relinquished and received?	Yes 🗸	No 🗌			
Chain of custody agrees with sample labels?	Yes 🗸	No 🗌			
Samples in proper container/bottle?	Yes 🗸	No 🗌			
Sample containers intact?	Yes 🗸	No 🗌			
Sufficient sample volume for indicated test?	Yes 🗸	No 🗌			
All samples received within holding time?	Yes 🗸	No 🗌			
Container/Temp Blank temperature in compliance?	Yes 🗸	No 🗌			
Sample(s) received on ice? Temperature(s)/Thermometer(s):	Yes ✔ 4.1/5.1 C	No 🗌	IR	3]
Cooler(s)/Kit(s):					
Date/Time sample(s) sent to storage:	9/2/2021	1:44:13 PM			
Water - VOA vials have zero headspace?	Yes	No 🗌	No VOA vials	submitted	
Water - pH acceptable upon receipt?	Yes	No 🗌	N/A		
pH adjusted? pH adjusted by:	Yes 🗌	No 🗌	N/A 🗹		

Login Notes:

Client Contacted:	Date Contacted:	Person Contacted:	
Contacted By:	Regarding:		
Comments:			
CorrectiveAction:			
			SR

Attachment D

Material Management Documentation

Start Date: 9/3/2021

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BILL TO ACCOUNT

Shamrock Landfill Tonnage Report JCTORS 9/3/21 19-0020-02 L

2708 LAKEHEAD CONSTRUCTORS HUSS1

Husky Energy/Superior Refining 2407 Stinson Ave V Superior. WI 54880

TICKET	Manifest	DATE	Waste Stream	Waste Name	TONS
78821	1655	9/3/21	19-0020-02	Petroleum Impacted Soil	14.87
78851	1656	9/3/21	19-0020-02	Petroleum Impacted Soil	15.72
78870	1657	9/3/21	19-0020-02	Petroleum Impacted Soil	14.71
/00/9	# of Loads: 3		SUBTO	OTAL FORWaste Stream	45.30
	G	RAND T	OTALS		45.30

			SITE	<u>г</u>	ICKET	GF	ID	WEIGHMASTER		
Shamrock Landfill			01	00	078879	LANDFIL	L	ALEXUS P		
761 Minnesota 45 Cloquet, MN 55720		INBOUND CHARGE	DAT	F IN	DATE OU	T TIRAE IN	TIME OU		BOLLOFF	
			09/03	3/21	09/03/21	14:02	14:02	K1	19-0020-02	
002708 LAKEHEAD (CONSTRUC	TORS INC							19 0020 02	
3801 WINT SUPERIOR 1	ER ST NT 54880	-5560			RENCE		(TN: 000	ORIGIN		
		5500		19-00	20-02		VIN: KB3	1867-WI-5AXL	.ES	
Ścale 1 Gross Wt.		57720 LB							~~~~	
Net Weight		28300 LB 29420 LB								
QTY.	UNIT	DESCRIPTION			DATE		TENOION	line for som		
14.71	TON	Industrial/ton			NAIC	E/	TENSION		TOTAL	
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				Weiling and the second second						
<u></u>		L		Ì					NET AMOUNT	
									TENDERED	
Generator: HUS	KY ENERG	GY/SUPERIOR REFINING							CHANGE	
Address: 240	7 STINSC	DN AVE								
Manifest: 165	скток, и 7	1 54880							CHECK NO.	
SKB-WCI 4410		SIGNA								
·····										
Shamrock Landfill				000	CKET		D	WEIG	HMASTER	
761 Minnesota 45		INBOUND CHARGE	01	000	100/9			ALEXUS P		
Cloquet, MN 55720			DATE	IN	DATE OUT	TIME IN	TIME OUT	VEHICLE	ROLL OFF	
002708 LAKEHEAD CO	ONSTRUCT		09/03/	/21	09/03/21	14:02	14:02	К1	19-0020-02	
3801 WINTER	R ST			REFER	ENCE			OBIGIN		
SUPERIOR W	I 54880-	5560	19	9-002	0-02	v	IN: RB31	.867-WI-5AXLE	S	
Scale 1 Gross Wt		5770 J.D.						·····		
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Net Weight		29420 LB								
	UNIT	DESCRIPTION			RATE	EXT	ENSION	FEE		
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ddress: 2407	Y ENERGY	//SUPERIOR REFINING							CHANGE	
ity/ST: SUPE	RIOR. WT	54880								
1anifest: 1657	***								CHECK NO.	

SKB-WCI 4410

Shipping Manifest	1. Generator's	s US EPA ID No	. (if a	ny)			-		2. Pa	ige 1 of	page(s)
3. Generator's Name and Facility Address Husky Energy/Superior Refining Co 2407 Stinson Ave Superior, WI 54880 4. Generator's Phone: (715) 817-6621 5. Transporter 1 Company Name	F. K)		F	/ailing A ⊢ 2 S	ddre lusky 407 Super	ss / Ene Stins ior, W	rgy/S on Av /I 548	uper 'e 380	ior Refinin	g Co
 Fransporter 2 Company Name 7. Designated Facility Name and Site Address 	Shamrocl	k Environm	enta	F 1, 1	Phone:						
	Cloquet.	Highway 4: MN 55720)			F	Phor	ie: 2	218-	-878-01	12
8. U.S. DOT Description (including Proper Shippin	g Name)		9. No	Cor	itainers Type	_	1 Te Qua	0. otal		11. Unit Wt/Vol	12. Waste Profile Sheet#
a) Non Hazardous Industrial Waste (PETROLEUM IMPACTED SOIL)											
).											
,									 		
i.											
 Additional Descriptions for Materials Listed Above (Inc. a. CL CL19-0020-02 PETROLEUM IMPACTED S D. CL CL J. CL 15. Special Handling Instructions and Additional Inf Emergency Contact: Eric Gruber 507 	licate waste stream A OIL ormation -351-4116	Approval # below)	14.	Spe	ecial Hand	dling F	Procee	lures	for W	fice Use C ad #	i Above
6. GENERATOR'S CERTIFICATION: I hereby dea proper shipping name and are classified, packe according to applicable international and nation	clare that the co d, marked, and al government r	ntents of this c labeled, and ar regulations.	onsig e in a	nme II re:	ent are fu spects in	illy ar prop	nd ac per co	curat onditi	ely d on fo	lescribed a or transpor	above by t by highway
Transporter 1 Acknowledged of Receipt of Mate	rials		Da A	vr	:17.	17.	ry A	al	1	Mon 	th Day Ye
B. Transporter 2 Acknowledgement of Receipt of N	1aterials	Signature	45	4	12	2.//	C			Mon 	th Day Ye 2 3 2
Printed/Typed Name 9. Discrepancy Indication Space	S	Signature								Mon	th Day Ye
0. Facility Owner or Operator: Certification of rece	ipt of non-hazar	rdous materials	cové	ered	by this N	/lanif	est e	cept	as r	noted in ite	əm 19.
	and the second	- A	-								

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bi minesci 4	Shamrock Landfill			01	96	078851	LANDFILL	-	ALEXUS P	
Columber, M. 19 5720 09/03/21 13:34 11:3	61 Minnesota 45		INBOUND CHARGE	DAT	E IN	DATE OUT	TIME IN	TIME OUT	VEHICLE	ROLL OFF
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Shipping Manifest	1. Generato	or's US EPA ID No	o. (if any)			2. Pa	age 1 of	page(s)
. Generator's Name and Facility Address			<u> </u>	Mailing Add	Iress			
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Transporter 1 Company Name		3		ax.				
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Transporter 2 Company Name								
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. Designated Facility Name and Site Address	Shamro	ck Environn	nental, I	LLC				
	761 MN	V Highway 4	5					
	Cloquet	, MN 55720		·····	Phone:	218-	-878-01	12
6. U.S. DOT Description (including Proper Shipp	ing Name)		9. Cor	ntainers	10. Tota		11. Unit	12. Waste Profile
)			No.	Туре	Quant	ty	Wt/Vol	Sheet#
Von Hazardous Industrial Waste						I		
(PETROLEUM IMPACTED SOIL)								
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3. Additional Descriptions for Materials Listed Above (indicate waste stream	m Approval # below)	14. Sp	ecial Handlir	g Procedure	s for W	astes Listed	Above
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. CL								
5. Special Handling Instructions and Additional I	nformation					04	fice Lles O	nly
Emergency Contact: Eric Gruber 50	7-351-4116					Lo	ad #	12851
6. GENERATOR'S CERTIFICATION: herebv d	eclare that the	contents of this c	onsianme	ent are fully	and accu	atelv o	described a	bove bv
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3. Transporter 2 Acknowledgement of Receipt of	Materials	VID	7711	N			- 9	r 3 C
Printed/Typed Name		Signature	s:				Mont	h Dav Va
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uscrepancy indication Space								
. Facility Owner or Operator: Certification of rec	eipt of non-ha	ardous materials	s covered	by this Ma	nifest exce	pt as r	noted in ite	m 19.
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From:	Paddock, Jeffrey J - DNR
Sent:	Monday, August 30, 2021 12:53 PM
То:	matthew.turner@huskyenergy.com
Cc:	Paddock, Jeffrey J - DNR
Subject:	Wisconsin DNR Spill Responsible Party Notification for SERTS ID
-	20210830NO16-1

Hi Matt,

Here is the notification for the crude oil spill that you reported this morning. Please submit the 45-day report with photos when the clean-up has been completed.

Thank you and let me know if you have additional questions.

Jeff Paddock

RR-5538 Wisconsin DNR Spill Electronic Reporting and Tracking System (SERTS) Responsible Party Notification

This notification contains information for the Responsible Party of the spill referenced below. Included is important legal information and links to spill response resources.

This document is intended solely as guidance and does not contain any mandatory requirements except where requirements found in statute or administrative rule are referenced. Any regulatory decisions made by the Department of Natural Resources in any matter addressed by this guidance will be made by applying the governing statutes and administrative rules to the relevant facts.

August 30, 2021

Spill Occurred: 2021-08-30 10:00 Spill Reported: 2021-08-30 12:13 Substance(s): Crude Oil SERTS ID: 20210830NO16-1

Spill Location: 2407 Stinson Ave Superior, WI Douglas County

Responsible Party: Superior Refining Company Llc [NO RP CONTACT NAME] [NO RP CONTACT TITLE] [NO RP ADDRESS 1] [NO RP ADDRESS 2] [RP CITY NOT ENTERED], [RP STATE NOT ENTERED] [RP ZIP NOT ENTERED]

Notice to Responsible Party

The person identified as the "Responsible Party" pursuant to <u>Wis. Admin. Code § NR 700.03(51)</u> is obligated to take the necessary response actions to address the hazardous substance discharge or environmental pollution under Wis. Stat. ch. 292.

Obligations

Your legal responsibilities are defined in Wis. Stat. ch. 292 and Wis. Admin. Code chs. NR 700-754. In particular, <u>the hazardous substances spill law</u> states:

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

<u>Wis. Admin. Code chs. NR 700 - 754</u> establish requirements for actions to be taken by responsible parties to restore the environmental to the extent practicable; protect public health, safety, welfare and the environment; and establishes documentation requirementsassociated with these response actions, where a hazardous substance discharge or environmental pollution has occurred. <u>Wis. Admin.Code ch.</u> <u>NR 708</u> contains requirements for immediate actions following a hazardous substance discharge.

Steps to Take

<u>Wis. Admin. Code § NR 708.05</u> requires responsible parties to take immediate action to halt a hazardous substance discharge or environmental pollution and minimize the harmful effects of the discharge or environmental pollution to the air, lands and waters of the state, unless otherwise directed by the DNR.

Below are initial actions that should be taken to address a hazardous substance discharge or environmental pollution:

Obtain the services of an environmental response contractor and/or an environmental consultant to help ensure that proper immediate actions are taken and documented. Information about selecting <u>Environmental Consultants</u> and <u>Spill Response Contractors</u> is available at dnr.wi.gov search for environmental consultants and spills.

Review, along with your contractor or consultant, <u>Wis. Admin. Code § 708.05</u>, which describes spill response actions for both emergency and non-emergency immediate actions.

<u>Wis. Admin. Code § NR 708.05(6)</u> requires the submittal of written documentation to the DNR of immediate actions taken and the outcome of those actions, within 45 days after the hazardous substance discharge notification to the DNR.

Comply with <u>Wis. Admin. Code § NR 708.09</u>, which specifies the requirements for the preparation and submittal of a final report to the DNR documenting the actions taken to respond to the hazardous substance discharge and environmental pollution. Reports may be submitted to the appropriate DNR regional spill coordinator, listed below

Review the remainder of <u>Wis. Admin. Code § NR 708</u> to ensure that all immediate response action requirements have been complied with.

DNR Determination

The DNR will provide a cursory review of the Wis. Admin. Code ch. NR 708 reports, if submitted without a review fee. If no further action is necessary, the DNR will note that in the Bureau for Remediation and Redevelopment (BRRTS) database. If you want a written response from the DNR related to a No Further Action decision, or any other determination, please fill out and submit <u>DNR Form 4400-237</u> with the appropriate fee.

If, however, groundwater wells are affected by the hazardous substance discharge or environmental pollution, if free product removal is required, if there is evidence that contaminated soil may be in contact with groundwater or residual contamination poses a threat to public health or the environment, the DNR shall require additional action per Wis. Admin. Code § NR708.09(2).

Please contact me if you have any questions regarding this notification or you would like to discuss your specific situation in more detail.

DNR Regional Spill Coordinator:

Jeffrey Paddock 715-828-8544 jeffrey.paddock@wisconsin.gov