Good morning John and Phil,

Per my recent phone call with Phil, here is an update on the slop oil spill that occurred at the Calumet Superior refinery on 5/2/2017.

At roughly 14:00 on 5/2/2017, an oil stain was discovered in a grass covered pervious area that is used primarily as a rain water sump location. The water that is collected in this area is normally pumped to our API separator/on-site WWTP system. The affected area is roughly 12' x 20'. Immediately after discovery, a cleanup/recovery effort was started. The contaminated soil was placed into 55 gallon drums, brought to our 90-day waste storage building and will be properly disposed of off-site at a later date. Any free product/contaminated water was recovered with a vacuum truck and put into our re-refining process. Once the initial soil removal was complete, the cleanup of the contaminated surfaces (cement vaults, pumps, piping, etc.) commenced. This was done using hand scrapers and ACE-10 Plus (degreaser cleaning solution) and followed up with a pressure steam wash. As of the time of this email, we believe that the affected pervious surfaces have been cleaned to the fullest extent possible. We are still investigating how oil accumulated in the area causing the release.

Here's what our plan is going forward.

Still yet today (5/5/2017) we intend to do another round of soil removal to clean up any soil that may have come in contact with the water from the surface cleaning. Once that is done, we intend to gather soil samples to be sent off for analysis by a third party laboratory. We will compare the results to the Soil-to-Groundwater RCLs that have been provided to us by the WDNR. If the results come back under tolerance, we will then move to close the sight.

Please don't hesitate to call me with any questions or comments. As we are in the middle of a small turnaround here at the facility, if I do not answer my office phone, please call my cell phone.

Thanks

Matt Turner Environmental Engineer Calumet Specialty Products Superior, Wisconsin Refinery Office: (715) 398-8434 Cell: (715) 969-4873





2407 Stinson Avenue Superior, WI 54880 Phone: 715-398-3533 Fax: 715-398-8209 www.calumetspecialty.com

June 14, 2017

John Sager Hydrogeologist Program Coordinator Remediation and Redevelopment Program Wisconsin Department of Natural Resources 1701 N 4th St Superior, WI 54880

Re: Slop Oil Release SERTS ID 20170502NO16-1

Dear Mr. Sager,

Please find the attached report regarding the immediate action taken in response to the slop oil release reported the WDNR on Tuesday, May 2, 2017 as required under NR 708.05(6)(a).

It should be noted that Calumet is not requesting no further action at this time. Calumet has conducted soil sampling at the site and is awaiting the results. Once, desired results are obtained, Calumet will then request no further action be taken.

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

Thank you,

Matt Turner

Environmental Engineer

NR708.05(6)(a) Immediate Action Report

708.05(6)(a) Unless par. (b) is applicable or unless otherwise directed by the department, responsible parties shall prepare and submit written documentation to the department describing the immediate actions taken at their site or facility and the outcome of those actions, within 45 days after the initial hazardous substance discharge notification is given to the department in accordance with the requirements of ch. NR 706.

1. 708.05(6)(c)1 A statement expressing the purpose of the submittal and the desired department action or response.

The purpose of this submittal is to comply with the written documentation requirements under NR 708.05(6) describing the immediate action taken following a hazardous substance discharge. Calumet requests that the department deem this report acceptable to fulfill the requirements of NR 708.05(6).

All of the visible contamination has been removed and any affected surfaces have been cleaned and decontaminated. Calumet is awaiting the results from soil sampling conducted at the site. Calumet is requesting the WDNR allow the spill to remain open until confirmation sampling results are obtained to ensure the site is clean. Once the immediate response action is completed, Calumet will submit the required report under NR 708.09.

2. 708.05(6)(c)2 Name, address and telephone number of the responsible parties.

Name: Calumet Superior, LLC

Address: 2407 Stinson Ave., Superior, WI 54880

Phone: (715) 398-3533

708.05(6)(c)3 Location of the site or facility, or discharge incident, including street address; quarter-quarter section, township, range, and county; and the location information specified in s. NR 716.15 (5) (d); latitude and longitude, and legal description of lot, if located in platted area.

Street Address: 2407 Stinson Ave., Superior, WI 54880

Coordinates: NW ¼ of the NW¼ of Section 36, Township 49 North, Range 14 West, Superior Township of Douglas County.

WTM Coordinates: X:361,687 Y:692,875

4. 708.05(6)(c)4 Any information required under ch. NR 706 that has not been provided to the department previously.

Information required under NR 706 was supplied to the Division of Emergency Management with the Initial Discharge Notification required under NR 706.05(1)(b). To ensure all information is provided, please see attachment A for a summary of the information initially reported.

5. 708.05(6)(c)5 The type of engineering controls, treatment or both and the effluent quality of any permitted or licensed discharge.

The discharge occurred in a constructed low lying area designed to capture stormwater and was controlled utilizing earthen berms required by the Spill Prevention, Control, and Countermeasure (SPCC) regulation as required by Title 40 of the Code of Federal Regulation Part 112.

Effluent stormwater is regulated under the Department of Natural Resources general permit for Tier 1 industrial Facilities (WPDES Permit No. WI-S067849-3) and under the facilities WWTP (Permit No. 0003085-08-0).

Any deviations from permitted conditions and/or limits under either permit are reported as required by the respective permit.

6. 708.05(6)(c)6 The type, total volume and final disposition of the discharged hazardous substance and contaminated materials generated as part of the immediate action, including legible copies of manifests, receipts and other relevant documents.

Type of Material Spilled:

Slop Oil Total Volume: 100 Gallons

Final Disposition:

Calumet is currently awaiting sampling results to confirm that the cleanup efforts of the area have been sufficient. Once, desired results are obtained, Calumet will then request no further action be taken.

All excavated material is stockpiled at Calumets' Solid Waste Storage Facility (License No. 4062). It is stored there until adequate quantities are reached to schedule shipment.

All recovered product is re-inserted into the refining process.

All recovered water is treated on-site at the facilities WWTP (Permit No. 0003085-08-0).

Attachment A

NR 706.05 Hazardous Substance Discharge Notification Form

NR 706.05 Hazardous Substance Discharge Notification Form

(c)(1) <u>Perse</u>	on Reporting	
	Name:	Matt Turner
	Address	2407 Stinson Ave, Superior, WI 54880
	Phone:	715-398-8434
(c)(2) <u>Owne</u>	er/Discharger	
	Name:	Calumet Superior, LLC
	Address	2407 Stinson Ave, Superior, WI 54880
	Phone:	715-398-3533
(c)(3) <u>Disch</u>	narge Information	<u>)</u>
	Date:	5/2/2017
	Time:	14:15
	Duration:	Unknown
(c)(3m) <u>Dis</u>	charge Location	(WTM Coordinates)
	X:	361,687
	Y:	692,875
(c)(4) <u>Disch</u>	narge Material	
	Identity:	Slop Oil
	Physical State:	Solid
	Quantity:	100 Gallons
(c)(5) <u>Disch</u>	harge Characteris	stics
	Physical:	Solid
	Chemical:	Hydrocarbons
ļ	Hazardous:	Combustable
-	Toxilogical:	Low Toxicity
(c)(6) <u>Caus</u>	<u>se</u>	
	Cause:	Faulty check valve on stormwater sump discharge line
(c)(7) <u>Resp</u>	onse Action	
	Action Taken:	Cleaned up impacted soil and water
	Contractor(s):	WCS & In-Line
(c)(8) <u>Move</u>	ement	
	Source:	Faulty check valve on stormwater sump discharge line
:	Speed:	<1 MPH
	Destination:	Contained within Seconday Contaiment
(c)(9) <u>Impa</u>	<u>icts</u>	
	Human Health:	None known at this time
	Environmental:	Contaminated soil
	Water Supplies:	None known at this time

(c)(10) Weather

Precipitation:	None
Wind Direction:	NE
Velocity	5 MPH

(c)(11) Other Agencies on-scene N/A

Agencies:



2407 Stinson Avenue Superior, WI 54880 Phone: 715-398-3533 Fax: 715-398-8209 www.calumetspecialty.com

September 1, 2017

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

Re: Slop Oil Release SERTS ID 20110502NO16-1

Dear Mr. Sager,

Please find the attached report regarding the immediate action taken in response to the Slop Oil release reported the WDNR on May 2, 2017 as required under NR 708.09(1). Based on the conclusions and recommendations of this report, we are requesting no further action for this release.

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

Sincerely,

Matt Turner Environmental Engineer

Enclosure



Immediate Response Action Report

Slop Oil Release SERTS ID 20170502NO16-1

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

9/1/2017

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1.0 INTRODUCTION

This report contains a summary of the immediate response actions at the Calumet Superior, LLC Superior, WI refinery in response to a Slop Oil release on May 2nd, 2017. The site location is shown in Figure 1. The response was initiated on May 2nd, 2017 after the release was discovered. The following report was prepared in accordance with Wisconsin Administrative Code NR 708 final report criteria under NR 708.09 for no further response action.

2.0 <u>Type of Hazardous Substance Discharged, Toxicity, Mobility and Volume- NR</u> <u>708.09 (1)(a)</u>

The slop oil spill occurred in an area located south of the facility's main "wash slab" and east of an area called the "slop manifold". The area in which the release occurred functions as a stormwater accumulation area that collects rain water from the surrounding vicinity in a cement vault prior to being pumped to our API separator. The volume of the release was estimated to be 100 gallons. The release had increased mobility due to the presence of rain water which moved the product from containment and onto a pervious surface. Weather conditions are shown in appendix B.

The spill did not reach any water bodies, and did not migrate from the impacted area. A site map is included in Figure 2. Spill site conditions are depicted in the photographs which are included in Appendix A. WTM coordinates of the spill are included in section 15.0.

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

The duration of the discharge is unknown; however, a rain event during the evening of May 1^{st} , 2017 most likely caused the sump to overflow and move the slop oil contained inside of it onto a pervious surface. The aforementioned rain event measured 0.26 inches. A summary of local weather conditions from May 2^{nd} , 2017 – June 17th, 2017 is located in Appendix B.

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

As the release occurred in a low lying area used to collect stormwater, the material pooled and was contained at the release site. In order to eliminate the suspected source of the discharge, the stormwater vault was vacuumed out using a vacuum truck on May 2nd, 2017. On May 2nd, 2017, cleanup of the spill was initiated by means of hand excavation.

5.0 MITIGATION EFFORTS THAT MAY HAVE ACCELERATED MIGRATION OF POLLUTION OR

HAZARDOUS SUBSTANCES NR 708.09(1)(d)

As contaminated soil and water were removed from the release site, it became apparent that the issue could repeat itself since the cause was not initially known. The cleanup commenced immediately but there was a chance that it could have been re-contaminated, undoing the cleanup effort. Steps were taken to isolate the potential causes and the issue did not repeat itself.

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

Weather conditions on the day of the release were typical for that time of year. The mean temperature was 46° F, there was a 10 mph N wind, and there was no measured precipitation. A summary of local weather conditions from May 2^{nd} , 2017 – June 17th, 2017 is located in Appendix B.

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

All visually impacted soil and free product was removed from the site. Samples taken after cleanup indicated results above the soil-to-groundwater RCLs established by the WDNR. Stormwater from the release area is collected in a cement sump vault and pumped to the API separator at the facility.

Because of the relatively impermeable surficial clay at the refinery, releases tend to migrate more horizontally along the ground surface. Based on the very low groundwater velocities and absence of any groundwater receptors, there is literally no groundwater exposure risk at the refinery. All water and oil recovered by means of vacuum truck after the release was recovered/treated in the refineries No. 1 Oil/Water Separator/WWTP.

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

The release resulted from a faulty check valve on the discharge line leading from the cement sump vault to the API separator. The valve became stuck in the open position, allowing slop oil from the API separator to migrate backwards passed the valve and into the vault, overfilling it. The valve has been replaced with a properly functioning model. An additional sump pump and piping will be added to the vault to act as a redundancy in order to prevent future overflows from the vault.

On May 2nd, 2017 hand excavation of the visibly contaminated soil commenced. In addition to the excavation, a number of surfaces impacted by the release material were cleaned with a combination of steam and soapy water. Those combined efforts continued until April 10th, 2017. A total of 6,400 pounds of soil were removed, placed into 55 gallon steel drums, and sent to a proper TSDF for disposal.

The excavation took up an area approximately 28 feet by 25 feet but had a number of structural impediments present. The area was excavated to a depth between four and eight inches and the product did not seem to leech deep into the soil. Spill site conditions and remediation efforts are depicted in the photographs which are included in Appendix A. The area of the spill and excavation are depicted in Figure 2.

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

Four laboratory samples were collected after the initial excavation was complete to confirm the removal of contaminated soil. All soil samples were analyzed petroleum volatile organic compounds (PVOCs). Three of the four samples came back above the soil-to-groundwater residual contaminant levels (RCLs) established by the WDNR.

Soil sample locations are displayed in Figure 2. A summary of the analytical results is contained in Table 1. Full laboratory analytical results are included in Appendix C.

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

Visual and olfactory evidence of the slop oil contamination was present upon arrival to the site and during the excavation. The visual extent of contamination was excavated and no odor was present following the excavation. As slop oil is black in color, the most effective means to determine the extent of contamination is visually. It would have been unnecessary and unsafe to excavate further for risk of undermining the concrete foundations for the sump vault, a stormwater junction box, foundations for a pipe rack, and a pump slab foundation.

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

The contaminated material collected during the excavation of the spill area was placed into several 55 gallon steel drums. Disposal of this material ultimately occurred at the Clean Earth AES Environmental, LLC (EPA ID: KYD985073196) hazardous waste TSDF in Calvert City, KY. It is expected that the slop oil did not penetrate beyond the depth of the excavation due to visual observations after the release. Potential environmental impacts are minimal. The spill was contained on-site and did not run off into other areas, and was restricted to the area of the excavation; therefore, the actual or potential environmental impacts are minimal.

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

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

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

The land is presently used as a stormwater accumulation area for an oil refinery. The refinery was constructed in 1951 and has remained in the same use since that time. There is no anticipation the land will be used for another purpose in the future.

14.0 EVALUATE IF ROUTES OF EXPOSURE ARE PROTECTIVE AND ENVIRONMENT HAS BEEN

RESTORED TO THE EXTENT PRACTICABLE - NR 708.09 (1)(m)

A good faith effort was undertaken to remove all newly contaminated material from the release site. No off site receptors were impacted by the release. Given the amount of slop oil released, the site conditions at the time of the release, and the confirmation sampling results, there is little chance the slop oil migrated laterally beyond what has been excavated. Confirmation samples indicate the spill has not been remediated to below soil-to-groundwater residual contaminant levels but it is Calumet's intention to install a poly liner on top of the affected area. Doing so will eliminate the possibility of direct contact with the remaining contaminated soil.

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

The site is located in the NW ¼ of the NW ¼ of Section 36, Township 49 North, Range 14 West, City of Superior, Douglas County, WI. The WTM coordinates for the spill site are 361689, 692876. A site vicinity map is included in Figure 1.

16.0 CONCLUSION AND RECOMMENDATIONS

Based on laboratory results indicating the presence of contaminants above the NR720 soil-togroundwater RCL's, the spill has not been remediated to allow for clean closure. Therefore, it is Calumet's intention to install a poly liner on top of the affected area and the area immediately surrounding it. This "cap" will serve as a barrier to prevent direct contact with the material as well as another line of defense in case the sump malfunction occurs again. It is also Calumet's intention to install a redundant pump in the sump vault as a further precaution to prevent overfilling. Once the liner and new pump system are installed, it is recommended that no further response action is necessary at the site and that it be added to the facility wide GIS and ERP registry.

TABLES

Table 1Soil Sample Analytical Results

Soil Sample Results (mg/kg)						
Analyte	S1	S2 S3		S4		
Benzene	0.00507	0.00788	2.22000	0.02140		
Toluene	0.00000	0.00000	0.00000	0.00000		
Ethylbenzene	0.00000	0.00513	16.30000	0.04160		
m&p-Xylene	0.00887	0.01280	11.50000	0.11300		
o-Xylene	0.00000	0.00000	6.32000	0.27500		
Methyl tert-butyl ether	0.00000	0.00898	3.22000	0.00000		
Naphthalene	0.00000	0.00000	0.00000	0.00000		
1,3,5-Trimethylbenzene	0.00683	0.00518	6.63000	0.00000		
1,2,4-Trimethylbenzene	0.00695	0.02210	18.80000	0.00000		
TPH (GC/FID) Low Fraction	0.00	1.10	2160.00	317.00		

Amount Below RCL Level (mg/kg)						
Analyte	S1	S2	S3	S4		
Benzene	0.00005	-0.00276	-2.21488	-0.01628		
Toluene	1.10720	1.10720	1.10720	1.10720		
Ethylbenzene	1.57000	1.56487	-14.73000	1.52840		
m&p-Xylene	2 05112	3.94720	-13.86000	3.57200		
o-Xylene	5.95115					
Methyl tert-butyl ether	0.02702	0.01804	-3.19298	0.02702		
Naphthalene	0.65818	0.65818	0.65818	0.65818		
1,3,5-Trimethylbenzene	1 26920	1 25 470	24 04702	1 20207		
1,2,4-Trimethylbenzene	1.30829	1.55479	-24.04793	1.56207		

Soil-to-Groundwater RCLs (mg/kg)				
Benzene	0.00512			
Toluene	1.10720			
Ethylbenzene	1.57000			
m&p-Xylene	3 06000			
o-Xylene	5.90000			
Methyl tert-butyl ether	0.02702			
Naphthalene	0.65818			
1,3,5-Trimethylbenzene	1 20207			
1,2,4-Trimethylbenzene	1.56207			

FIGURES

Figure 1 Site Vicinity Map

Figure 2 Site Map



Lined Area	Wash Slab	Figure 2 - Site Map Wash Slab		
Lined Area	Affected Area		Elevated Building	Not to Scale
Lined Area	Out of Service Pump Foundation	Affected Area	Stormwater Sump	
Lined A	Area Affected A	Affected Area	Affected Area Pipe Rack Footing	

APPENDICES

Appendix A	Photographs
Appendix B	Historical Weather Information

Appendix C Laboratory Analytical Report

Appendix A – Photographs



Photo 1: Looking north at the release site.



Photo 2: Looking north at the release site. The water seen entering the release site is runoff from the road located uphill and south of the release site. A sand dam and absorbent boom were placed at the edge of the road to prevent further water from entering the site. This can be seen in Photo 6.



Photo 3: Looking west at the release site.



Photo 4: Looking west at the release site.



Photo 5: Looking northwest at the release site.



Photo 6: Looking north at the cleanup effort. As mentioned in Photo 2, a sand dam and absorbent booms were placed along the edges of the road to prevent further storm water from entering the site. Absorbent pads were placed on the excavated soil to soak up any potential contaminants that may have been missed after the initial excavation.



Photo 7: Looking west at the cleanup effort. In addition to soil removal, all contaminated surfaces were cleaned as well, which can be seen here. Steam pressure washers were used in combination with soap and hand tools to clean the affected surfaces.



Photo 8: Looking southeast at the scraped and exposed visually clean soil. The grass line marks the extent of the release.



Photo 9: Looking south at the scraped and exposed visually clean soil. The grass line marks the northern extent of the release.



Photo 10: Looking south at the sample location of S1 (red dot).



Photo 11: Looking northwest at the sample location of S2 (red dot).



Photo 12: Looking south at the sample location of S3 (red dot).



Photo 13: Looking north at the sample location of S4 (red dot).

Appendix B - Weather History

	Max	Avg	Min	Sum
Temperature				
Max Temperature	91 °F	65 °F	45 °F	
Mean Temperature	70 °F	52 °F	38 °F	
Min Temperature	54 °F	39 °F	27 °F	
Degree Days				
Heating Degree Days (base 65)	28	13	0	326
Cooling Degree Days (base 65)	4	0	0	7
Growing Degree Days (base 50)	20	4	0	111
Dew Point				
Dew Point	68 °F	43 °F	21 °F	
Precipitation				
Precipitation	0.44 in	0.02 in	0.00 in	0.62 in
Snowdepth	-	-	-	-
Wind				
Wind	31 mph	6 mph	0 mph	
Gust Wind	36 mph	21 mph	9 mph	
Sea Level Pressure				
Sea Level Pressure	30.18 in	29.89 in	29.34 in	

2017		Temp. (°F	·)	De	ew Point ((°F)	Н	lumidity (%)	Sea L	evel Pres	s. (in)	V	isibility (n	ni)	V	Vind (mpl	h)	Precip. (in)	Events
May	high	avg	low	high	avg	low	high	avg	low	high	avg	low	high	avg	low	high	avg	high	sum	
<u>2</u>	58	46	34	33	30	26	91	58	30	29.97	29.79	29.54	10	10	10	18	10	24	0	
<u>3</u>	67	48	30	42	35	30	99	60	29	30.02	29.99	29.96	10	10	10	20	5	31	0	
<u>4</u>	58	47	36	44	40	37	100	75	51	30.1	30.06	30.01	10	9	1	16	5	23	0	
5	47	39	31	40	37	32	100	91	75	30.07	29.95	29.82	10	8	0	20	6	26	0	Fog
<u>6</u>	49	38	29	40	34	26	100	79	44	30.06	30.01	29.9	10	8	0	13	4	18	0	Fog
<u>7</u>	48	38	27	33	27	21	100	65	36	30.17	30.11	30.05	10	10	7	12	6	16	0	
<u>8</u>	45	40	35	33	29	23	88	64	47	30.16	30.08	30	10	10	10	20	10	31	0	
<u>9</u>	61	49	34	37	28	23	80	52	37	30	29.91	29.81	10	10	10	10	5	-	0	
<u>10</u>	73	52	31	44	38	29	96	58	36	30.01	29.88	29.83	10	10	10	16	5	24	0	
<u>11</u>	65	50	36	38	34	32	95	56	29	30.12	30.05	30.01	10	10	10	8	5	-	0	
<u>12</u>	72	52	32	42	36	32	100	63	30	30.07	29.98	29.85	10	10	10	17	3	23	0	
<u>13</u>	55		41																0	
<u>14</u>	46		42																0	
<u>15</u>	46		42																0.35	Rain
<u>16</u>	47		41																1.35	Rain
<u>17</u>	52		43																0.49	Rain
<u>18</u>	54		41																0.2	Rain
<u>19</u>	48		38																0	
<u>20</u>	43		39																0.61	Rain
<u>21</u>	49		39																0.17	Rain
<u>22</u>	57		46																0	
<u>23</u>	51		44																0	
<u>24</u>	49		45																0	
<u>25</u>	50		42																0	
<u>26</u>	49		44																0	
<u>27</u>	76		45																0	Deia
28	00		52																0.3	Rain
29	61		54																0.01	
<u>30</u>	72		48																0.01	
<u>31</u>	12		40	D	Deint ((0)				0	aural Dana	- (in)	V	- 11- 11-4 <i>(</i>	- :)		Mins al Anna an	-)		
2017	high	Temp. (F		bigh	ew Point (⊓ biab	umidity (/o)	Sea L	ever Pres	is. (in)	high	Sibility (I	ni)	hiah	vina (mpi	n) biab	Precip. (in)	Events
Jun	nign	avy	1000	nign	avy	10 W	nign	avy	10 W	nığıı	avy	IOW	nign	avy	10 10	nign	avy	nign	Sulli	
<u>1</u>	80		42																0	
<u>2</u>	61	50	42	47	4.4	20	02	70	EQ	20.97	20.02	20.77	10	10	10	7	2		0	
<u><u>3</u></u>	83	62	41	+1 53	44	40	90 100	73	22	23.01	29.00	29.11	10	10	10	20	5	- 28	0.01	Rain
<u>4</u>	62	52	38	55	40	38	100	8/	60	29.93	29.02	29.13	10	6	0	16	5	20	0.01	Fog
<u> </u>	68	51	30	 7	+1 ⊿2	30	100	70	<u>⊿</u> ∩	30.12	30.03	20.00	10	10	0	13	3	<u> </u>	0	Fog
<u>0</u> 7	72	54	36	54	+∠ 45	36	100	70	50	30.10	30.13	30.03	10	a	1	7	1	_	0	iuy
<u>/</u>	56	52	48	53	40	46	97	02	84	29.86	29.77	29.67	10	10	10	10	Q	-	0	
<u>2</u> 10	91	70	48	68	57	45	95	69	45	29.66	29.47	29.34	10	10	10	31	8	36	0.01	Rain
11	64	56	48	60	55	48	100	91	71	29.88	29.74	29.65	10	9	1	9	3	-	0.16	Rain
12	79	62	46	62	55	47	100	81	38	29.92	29.85	29.78	10	8	0	9	4	-	0	Fog
13	57	52	47	50	46	43	83	80	74	29.92	29.87	29.81	10	10	10	23	17	32	0	109
14	54	50	46	50	47	44	100	94	80	29.86	29.77	29.69	10	7	0	22	9	32	0.44	Fog . Rain
15	79	62	46	63	53	47	100	74	42	29.75	29,72	29.7	10	6	0	20	4	30	0	Fog, Rain
16	82	66	51	63	56	50	97	67	38	29.71	29.64	29.57	10	10	10	17	7	24	0	-3,
<u>17</u>	78	66	54	60	57	53	98	77	47	29.6	29.58	29.54	10	10	10	13	4	17	0	Rain









L916839

06/17/2017



Calumet Specialty Products

Sample Delivery Group:

Samples Received:

Project Number:

Description:

Wash Slab Sump

Report To:

Matt Turner 2407 Stinson Avenue Superior, WI 54880

Entire Report Reviewed By: John V Haulins

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.

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SAMPLE SUMMARY

ONE LAB. NATIONWIDE.

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			Collocted by	Collocted date/time	Received date/time
S1 L916839-01 Solid			Justin Boucher	06/16/17 13:36	06/17/17 08:45
Method	Batch	Dilution	Preparation	Analysis	Analyst
			date/time	date/time	
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	WG992683	51	06/16/17 13:36	06/27/17 20:14	LRL
			Collected by	Collected date/time	Received date/time
S2 L916839-02 Solid			Justin Boucher	06/16/17 13:40	06/17/17 08:45
Method	Batch	Dilution	Preparation	Analysis	Analyst
			date/time	date/time	
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	WG992683	51.5	06/16/17 13:40	06/27/17 20:38	LRL
S3 L916839-03 Solid			Collected by Justin Boucher	Collected date/time 06/16/17 13:44	Received date/time 06/17/17 08:45
Method	Batch	Dilution	Preparation date/time	Analysis date/time	Analyst
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	WG992683	980	06/16/17 13:44	06/27/17 21:01	LRL
			Collected by	Collected date/time	Received date/time
S4 L916839-04 Solid			Justin Boucher	06/16/17 13:49	06/17/17 08:45
Method	Batch	Dilution	Preparation	Analysis	Analyst
			date/time	date/time	
Volatile Organic Compounds (GC) by Method 8021B/WI(95) GRO	WG992683	198	06/16/17 13:49	06/27/17 21:25	LRL

CASE NARRATIVE

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All sample aliquots were received at the correct temperature, in the proper containers, with the appropriate preservatives, and within method specified holding times. 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 Technical Service Representative

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	Result	Qualifier	MDL	RDL	Dilution	Analysis	Batch	
Analyte	mg/kg		mg/kg	mg/kg		date / time		2
Benzene	0.00507	J	0.00449	0.0149	51	06/27/2017 20:14	WG992683	² Tc
Toluene	U		0.00821	0.0274	51	06/27/2017 20:14	WG992683	
Ethylbenzene	U		0.00464	0.0155	51	06/27/2017 20:14	WG992683	³ Sc
m&p-Xylene	0.00887	J	0.00785	0.0262	51	06/27/2017 20:14	WG992683	55
o-Xylene	U		0.00490	0.0163	51	06/27/2017 20:14	WG992683	4
Methyl tert-butyl ether	U		0.00816	0.0272	51	06/27/2017 20:14	WG992683	Cr
Naphthalene	U		0.0530	0.177	51	06/27/2017 20:14	WG992683	
1,3,5-Trimethylbenzene	0.00683	J	0.00418	0.0139	51	06/27/2017 20:14	WG992683	⁵ Sr
1,2,4-Trimethylbenzene	0.00695	J	0.00546	0.0182	51	06/27/2017 20:14	WG992683	
TPH (GC/FID) Low Fraction	U		0.561	1.87	51	06/27/2017 20:14	WG992683	6
(S) a,a,a-Trifluorotoluene(PID)	100			80.0-200		06/27/2017 20:14	WG992683	ଁ Q(

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	Result	Qualifier	MDL	RDL	Dilution	Analysis	Batch				
Analyte	mg/kg		mg/kg	mg/kg		date / time		2			
Benzene	0.00788	J	0.00453	0.0151	51.5	06/27/2017 20:38	WG992683	Tc			
Toluene	U		0.00829	0.0277	51.5	06/27/2017 20:38	WG992683				
Ethylbenzene	0.00513	J	0.00469	0.0156	51.5	06/27/2017 20:38	WG992683	³ S c			
m&p-Xylene	0.0128	J	0.00793	0.0264	51.5	06/27/2017 20:38	WG992683	55			
o-Xylene	U		0.00494	0.0165	51.5	06/27/2017 20:38	WG992683	4			
Methyl tert-butyl ether	0.00898	J	0.00824	0.0274	51.5	06/27/2017 20:38	WG992683	Cn			
Naphthalene	U		0.0536	0.179	51.5	06/27/2017 20:38	WG992683				
1,3,5-Trimethylbenzene	0.00518	J	0.00422	0.0141	51.5	06/27/2017 20:38	WG992683	⁵ Sr			
1,2,4-Trimethylbenzene	0.0221		0.00551	0.0184	51.5	06/27/2017 20:38	WG992683				
TPH (GC/FID) Low Fraction	1.10	J	0.566	1.89	51.5	06/27/2017 20:38	WG992683	6			
(S) a,a,a-Trifluorotoluene(PID)	101			80.0-200		06/27/2017 20:38	WG992683	ČQc			

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	Result	Qualifier	MDL	RDL	Dilution	Analysis	Batch	
Analyte	mg/kg		mg/kg	mg/kg		date / time		- L
Benzene	2.22		0.0862	0.287	980	06/27/2017 21:01	WG992683	
Toluene	U		0.158	0.526	980	06/27/2017 21:01	WG992683	L
Ethylbenzene	16.3		0.0892	0.297	980	06/27/2017 21:01	WG992683	
m&p-Xylene	11.5		0.151	0.503	980	06/27/2017 21:01	WG992683	
o-Xylene	6.32		0.0941	0.314	980	06/27/2017 21:01	WG992683	Γ
Methyl tert-butyl ether	3.22		0.157	0.522	980	06/27/2017 21:01	WG992683	
Naphthalene	U		1.02	3.40	980	06/27/2017 21:01	WG992683	L
1,3,5-Trimethylbenzene	6.63		0.0804	0.268	980	06/27/2017 21:01	WG992683	
1,2,4-Trimethylbenzene	18.8		0.105	0.350	980	06/27/2017 21:01	WG992683	
TPH (GC/FID) Low Fraction	2160		10.8	36.0	980	06/27/2017 21:01	WG992683	
(S) a,a,a-Trifluorotoluene(PID)	111			80.0-200		06/27/2017 21:01	WG992683	

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	Result	Qualifier	MDL	RDL	Dilution	Analysis	Batch	
Analyte	mg/kg		mg/kg	mg/kg		date / time		2
Benzene	0.0214	J	0.0174	0.0580	198	06/27/2017 21:25	WG992683	Tc
Toluene	U		0.0319	0.106	198	06/27/2017 21:25	WG992683	
Ethylbenzene	0.0416	J	0.0180	0.0600	198	06/27/2017 21:25	WG992683	³ Sc
m&p-Xylene	0.113		0.0305	0.102	198	06/27/2017 21:25	WG992683	53
o-Xylene	0.275		0.0190	0.0634	198	06/27/2017 21:25	WG992683	4
Methyl tert-butyl ether	U		0.0317	0.106	198	06/27/2017 21:25	WG992683	Cn
Naphthalene	U		0.206	0.687	198	06/27/2017 21:25	WG992683	
1,3,5-Trimethylbenzene	U		0.0162	0.0541	198	06/27/2017 21:25	WG992683	⁵ Sr
1,2,4-Trimethylbenzene	U		0.0212	0.0707	198	06/27/2017 21:25	WG992683	51
TPH (GC/FID) Low Fraction	317		2.18	7.27	198	06/27/2017 21:25	WG992683	6
(S) a,a,a-Trifluorotoluene(PID)	100			80.0-200		06/27/2017 21:25	WG992683	ČQc

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

QUALITY CONTROL SUMMARY

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Method Blank (MB)

(MB) R3229458-3 06/27/17	09:57			
	MB Result	MB Qualifier	MB MDL	MB RDL
Analyte	mg/kg		mg/kg	mg/kg
Benzene	U		0.0000880	0.000293
Toluene	U		0.000161	0.000537
Ethylbenzene	U		0.0000910	0.000303
m&p-Xylene	U		0.000154	0.000513
o-Xylene	U		0.0000960	0.000320
Methyl tert-butyl ether	U		0.000160	0.000533
Naphthalene	U		0.00104	0.00347
1,3,5-Trimethylbenzene	U		0.0000820	0.000273
1,2,4-Trimethylbenzene	U		0.000107	0.000357
TPH (GC/FID) Low Fraction	U		0.0110	0.0367
(S) a,a,a-Trifluorotoluene(PID)	101			80.0-200

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

(LCS) R3229458-1 06/27/17 09:10 • (LCSD) R3229458-8 06/27/17 22:35												
	Spike Amount	LCS Result	LCSD Result	LCS Rec.	LCSD Rec.	Rec. Limits	LCS Qualifier	LCSD Qualifier	RPD	RPD Limits		
Analyte	mg/kg	mg/kg	mg/kg	%	%	%			%	%		
Benzene	0.0500	0.0440	0.0423	88.1	84.6	80.0-120			4.02	20		
Toluene	0.0500	0.0435	0.0419	86.9	83.7	80.0-120			3.76	20		
Ethylbenzene	0.0500	0.0470	0.0453	94.1	90.5	80.0-120			3.85	20		
m&p-Xylene	0.100	0.100	0.0958	100	95.8	80.0-120			4.26	20		
o-Xylene	0.0500	0.0474	0.0461	94.8	92.2	80.0-120			2.78	20		
Methyl tert-butyl ether	0.0500	0.0474	0.0463	94.9	92.5	80.0-120			2.49	20		
Naphthalene	0.0500	0.0473	0.0455	94.7	90.9	80.0-120			4.07	20		
1,3,5-Trimethylbenzene	0.0500	0.0491	0.0472	98.3	94.5	80.0-120			3.94	20		
1,2,4-Trimethylbenzene	0.0500	0.0475	0.0453	95.0	90.5	80.0-120			4.80	20		
(S) a,a,a-Trifluorotoluene(PID,)			98.5	98.2	80.0-200						

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

(LCS) R3229458-2 06/27/17 09:10 • (LCSD) R3229458-9 06/27/17 22:35												
	Spike Amount	LCS Result	LCSD Result	LCS Rec.	LCSD Rec.	Rec. Limits	LCS Qualifier	LCSD Qualifier	RPD	RPD Limits		
Analyte	mg/kg	mg/kg	mg/kg	%	%	%			%	%		
TPH (GC/FID) Low Fraction	0.550	0.498	0.467	90.5	85.0	80.0-120			6.33	20		
(S) a,a,a-Trifluorotoluene(PID)	98.5	98.2	80.0-200									

DATE/TIME: 06/28/17 15:25 QUALITY CONTROL SUMMARY

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L916799-01 Original Sample (OS) • Matrix Spike (MS) • Matrix Spike Duplicate (MSD)

(OS) L916799-01 06/27/17 17:06 • (MS) R3229458-4 06/27/17 15:55 • (MSD) R3229458-6 06/27/17 16:19

	Spike Amount (dry)	Original Result (dry)	MS Result (dry)	MSD Result (dry)	MS Rec.	MSD Rec.	Dilution	Rec. Limits	MS Qualifier	MSD Qualifier	RPD	RPD Limits
Analyte	mg/kg	mg/kg	mg/kg	mg/kg	%	%		%			%	%
Benzene	0.0532	ND	2.20	2.17	82.5	81.5	50	32.0-137			1.20	39
Toluene	0.0532	ND	2.18	2.16	82.0	81.0	50	20.0-142			1.24	42
Ethylbenzene	0.0532	ND	2.38	2.35	89.3	88.4	50	10.0-150			1.03	44
m&p-Xylene	0.106	ND	5.15	5.09	96.7	95.4	50	14.0-141			1.25	44
o-Xylene	0.0532	ND	2.42	2.39	91.1	89.9	50	10.0-157			1.36	44
Methyl tert-butyl ether	0.0532	ND	2.32	2.26	87.2	85.1	50	24.0-151			2.44	37
Naphthalene	0.0532	ND	2.29	2.23	86.2	84.0	50	80.0-120			2.58	20
1,3,5-Trimethylbenzene	0.0532	ND	2.43	2.41	91.2	90.5	50	80.0-120			0.780	20
1,2,4-Trimethylbenzene	0.0532	ND	2.44	2.42	91.8	91.0	50	80.0-120			0.880	20
(S) a,a,a-Trifluorotoluene(PID)				98.8	98.9		80.0-200				

L916799-01 Original Sample (OS) • Matrix Spike (MS) • Matrix Spike Duplicate (MSD)

(OS) L916799-01 06/27/17	17:06 • (MS) R3	229458-5 06	/27/17 15:55 • (N	/ISD) R322945	8-7 06/27/17 1	6:19						
	Spike Amount (dry)	Original Result (dry)	MS Result (dry)	MSD Result (dry)	MS Rec.	MSD Rec.	Dilution	Rec. Limits	MS Qualifier	MSD Qualifier	RPD	RPD Limits
Analyte	mg/kg	mg/kg	mg/kg	mg/kg	%	%		%			%	%
TPH (GC/FID) Low Fraction	0.586	ND	25.7	25.1	87.7	85.9	50	80.0-120			2.07	20
(S) a,a,a-Trifluorotoluene(PID)					98.8	98.9		80.0-200				

ACCOUNT: Calumet Specialty Products DATE/TIME: 06/28/17 15:25 PAGE: 10 of 13

GLOSSARY OF TERMS

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Abbreviations	and	Definitions
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SDG	Sample Delivery Group.
MDL	Method Detection Limit.
RDL	Reported Detection Limit.
U	Not detected at the Reporting Limit (or MDL where applicable).
RPD	Relative Percent Difference.
(dry)	Results are reported based on the dry weight of the sample. [this will only be present on a dry report basis for soils].
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.
(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.
Rec.	Recovery.
Qualifior	Description

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

ACCREDITATIONS & LOCATIONS

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.

State Accreditations

Alabama	40660	Nevada	TN-03-2002-34
Alaska	UST-080	New Hampshire	2975
Arizona	AZ0612	New Jersey-NELAP	TN002
Arkansas	88-0469	New Mexico	TN00003
California	01157CA	New York	11742
Colorado	TN00003	North Carolina	Env375
Conneticut	PH-0197	North Carolina ¹	DW21704
Florida	E87487	North Carolina ²	41
Georgia	NELAP	North Dakota	R-140
Georgia ¹	923	Ohio-VAP	CL0069
Idaho	TN00003	Oklahoma	9915
Illinois	200008	Oregon	TN200002
Indiana	C-TN-01	Pennsylvania	68-02979
lowa	364	Rhode Island	221
Kansas	E-10277	South Carolina	84004
Kentucky ¹	90010	South Dakota	n/a
Kentucky ²	16	Tennessee 14	2006
Louisiana	AI30792	Texas	T 104704245-07-TX
Maine	TN0002	Texas ⁵	LAB0152
Maryland	324	Utah	6157585858
Massachusetts	M-TN003	Vermont	VT2006
Michigan	9958	Virginia	109
Minnesota	047-999-395	Washington	C1915
Mississippi	TN00003	West Virginia	233
Missouri	340	Wisconsin	9980939910
Montana	CERT0086	Wyoming	A2LA
Nebraska	NE-OS-15-05		

Third Party & Federal Accreditations

A2LA – ISO 17025	1461.01	AIHA-LAP,LLC	100789
A2LA – ISO 17025 ⁵	1461.02	DOD	1461.01
Canada	1461.01	USDA	S-67674
EPA-Crypto	TN00003		

¹ Drinking Water ² Underground Storage Tanks ³ Aquatic Toxicity ⁴ Chemical/Microbiological ⁵ Mold ^{r/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.



ACCOUNT:
Calumet Specialty Products

SDG: L916839 DATE/TIME: 06/28/17 15:25

Sec			Billing lafor	ation:		Т		1	Analysis / Con	tainer /	Preservative			Chain of Custody	rage_of_
Aatt Turner Environmental Engineer Calumet Specialty Products Refinery 2407 Stinson Avenue Superior, Wisconsin 54800			Attn: Acc PO Box 24 Indianap	Attn: Accounts Payable Provide the second s			Aeok								
Report to: Email To: matt.tur				er@calumets								1980	Mount Juliet, TN 371 Phone: 615-758-5858 Phone: 800-767-5859 Fax: 615-758-5859	Iulier, TN 37122 615-758-5858 800-767-5859 C 758-5859	
Project Description: Wash Slab Sump				Collected: Supe	rior, WI								1	L# 7162	79
hone: 715-398-8434	Client Project #	•	14	Lab Project #										D19	19
USTEN BOUCHER	Site/Facility ID	#	1 270	P.O. #								1.3	Acctnum: MUROILSWI Template:		
ollected by (signature):	Interceed by (signature):		Notified) Day	Quote #		6							Prelogin:		ultime (Bara)
mmediately racked on Ice N y X			iy (Rad Only) Jay (Rad Only)	Date Resu	ilts Needed	No. of	Q	F					PB: Shipped Via:		wkins (341)
Sample ID	Comp/Grab	Matrix *	Depth	Date	Time	Cntr.	PVC							Remarks	Sample # (lab only)
1	Grab	SS	6"	6/16/17	13:36	1	×							-	0
2	Grab	SS	6"	6/16/17	13:40	1	×			-			-	-	57
3	Grab	SS	6"	6/16/17	13:44	1	X					-		-	dy
4	Grab	SS	6"	6/16/17	13:49	1	×								
2.						T									
					1000	1					4		-		
			1	n 2.5007.55	Constanting	+		-					-	- Sala	
	1.1				-	+	-							1 4	
the first		1	A PARA		1	1					1000	Harr	Seul	mple Receipt	ti NP Y
Matrix: 55 - Soil AIR - Air F = Filter	Remarks:	Remarks:						2 15 422 /244 - Temp					coc Signed/Accurate:		
GW - Groundwater B - Bioassay WW - WasteWater DW - Drinking Water	Samples returned via:				72 13 YO 12 1249				Flow	Y Flow Other			Correct bottles used: Sufficient volume sent: <u>If Applicable</u> VOA Zero Headspace: Y		
OT - Other		(I-	Time:	lature	1		Trip Blan	k Receiv	red: Yes / No HCL / MA	PT4	eserva	tion Correct/	necked:Y		
		6-17	6-17 13:57 Time: Received by: (Signatur)		Temp:	2 °(C Bottles Receiv	ed: If p	reserva	ation required by	ogin: Date/Time	
		_	Time:	Received for lab by: (Signature)				Date:	Date: Time:			ld:		NCF /	