04-16-562147

SFILL ID# ZUTSTZZONUTO-T

		State Su	of Wiscons bstance Re Repo	in - Dep lease N ort creat	artment o otification ted on 06/	f Natural Reso Report (SERT 12/2014	ources "S)			
Incident Date & Time:         Reported Date           12/26/2013 11:50         12/26/2013 12				Time: BRRTS No:				Spill ID; 20131226NO16-1		
DATCP Reported? DATCP Transferre	? No ed? No	NFA No	Letter Sent?		ERP Tran	nsferred?		Incident CI Yes : 06/1	osed? 2/2014	
				Lov	nation					
Region: NO	County: Douglas			Municip	cality: RIOR, CITY	Y OF				
Facility/Property N CALUMET SUPE 2400 STINSON /	Name and Street Ad ERIOR AVE	dress:			Descripti EAST O	ion: F COOLING TO	OWER #2			
Facility Type:	Bulk Petroleum St	orage (Tar	nk Farm/Ter	minal/Re	efinery)	3				
Lat/Long:			PLSS:				<b>WTM</b> : X 36171	1 Y 6929	28	
Weather Condition	ns:					1928()	1			
			R	lespons	ible Partie	es out out		0.111.0		
CALUMET SUPE 2400 STINSON / SUPERIOR, WI -	: ERIOR LLC AVE	DAVI ENVI (715)	act: ID BEATTIE IRONMENT ) 398-3533 x	AL ENG primary	INEER	Other Contac	t:	Spill Pa	скет:	
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-	Method						Description	n		
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			Clea	nup Act	ion comm	Terris		v v		

	Contractors Hired	Wards - Frank and
Name	Description	
	Waste Destinations	
Locátion	Description	
Age	ncies Notified / On Scene	E. Caller
Agency	Notified	On Scene
ONR	Х	
	Additional Comments	

State of Wisconsin - Department of Natural Resources

# State of Wisconsin - Department of Natural Resources Substance Release Notification Report (SERTS) Report created on 06/12/2014

REPAIRS WERE MADE AND CLEANUP OF CONTAMINATED SOIL WILL BEGIN ON 12/27/13. SAGER SENT PETER FREDMAN A REQUEST FOR AN UPDATE VIA EMAIL ON 1/9/14. SAGER RECEIVE EMAIL RESPONSE FROM FEDMAN AT 1019HRS ON 1/9/14. SAGER SPOKE TO PETER FEDMAN AT APPROXIMATELY 1330HRS ON 1/29/14. CALUMET HAS EXCAVATED TO THE EXTENT POSSIBLE CONSIDEREING THE WEATHER. CALUMET WANTS TO WAIT UNTIL SPRING TO FINISH THE CLEANUP. SAGER VISITED THE SITE ON 1/30/14. VERY SMALL AMOUNTS OF FREE PRODUCT VISIBLE UNDER PIPE RACK. CALUMET RECOVERED ALL THE OIL THAT IS PRACTICAL TO RECOVER NOW. ADDITIONAL CONTAMIANTED SOIL WILL BE REMOVED WHEN WEATHER CONDITIONS IMPROVE. SAGER RECEIVED AN IMEDIATE ACTION REPORT FROM CALUMET ON FEBRUARY 7, 2014. CALUMET WILL SEND IN A FINAL REPORT UPON COMPLETION OF IMMEDIATE ACTIONS. SAGER RECEIVED AN EMAIL STATUS UPDATE FROM CALUMET ON APRIL 29, 2014. ADDITIONAL FREE PRODUCT WAS COLLECTED AND CONTAMINATED SOIL WAS EXCAVATED ON 4/22-23/14. SAGER REQUESTED CONFIRMATION SAMPLES FROM THE SPILL AREA ON 4/29/14. SAGER RECEIVED AN DIDITIONAL INFORMATION FROM CALUMET ON JUNE 5, 2014. TRANSFERRED TO ERP 01-16-562123 PENDING. Enforcement Action/Citation? No											
Case Activity Reports:		1. A.									
	Person Repo	orting									
Name	Representing / Address	Primary Phone		Sec	Secondary Phone						
DAVID BEATTIE	CALUMET SUPERIOR LLC	(715) 398-3533 x									
	Contractors	Hired									
	Name / Address			Zone	Zone Contractor Hired by						
				No							
The design of the press of	Contact	S		Carlo State							
Role	Name	8	Office P	hone	Date	Time					
Prepared By:	JOHN SAGER	1	(715) 365-	8959 x	12/26/2013						
Person Notified:	NOR SPILL COORDINATOR JOHN SA	AGER	(715) 365-	8959 x	12/26/2013						
Investigated By:	JOHN SAGER				12/26/2013						
Incident Commander:											
Spill Coordinator:	JOHN SAGER, NO Region		(715) 365-	8959 x	06/12/2014						
	Electronic Attach	ments (list)		No. And And	State States	A DECK					
Na	Name Type										
20131226NO16-1_RP_Docum	entation_Immeidate_Action_Repedr <u>t</u> ab	dfDocument Format									

From: Sent: To: Cc: Subject: Sager, John E - DNR Thursday, June 12, 2014 13:30 Peter Fredman (Peter.Fredman@calumetspecialty.com) Endsley, Erin A - DNR December 26 2013 spill east of cooling tower #2 SERTS ID 20131226NO16-1

Peter,

Attached is the DNR's spill report for the spill near cooling tower # 2. I transferred this spill to a pending ERP site until the negotiated agreement is finalized. At that time the spill will be transferred to the facility-wide ERP site BRRTS ID 02-16-559511. When the transfer occurs and a facility-wide GIS package is submitted the required GIS Registry information concerning this spill will need to be submitted for addition to the Registry. Please contact me if you have any questions.

1



20131226NO16-...

John Sager

Emergency Response Coordinator / Hydrogeologist Remediation and Redevelopment Program Wisconsin Department of Natural Resources 1701 North 4<sup>th</sup> Street Superior, WI 54880 (2) phone: (715) 365-8959 (2) fax: (715) 392-7990 (3) e-mail: john.sager@wi.gov

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From:	Peter Fredman <peter.fredman@calumetspecialty.com></peter.fredman@calumetspecialty.com>
Sent:	Thursday, January 09, 2014 10:20
То:	Sager, John E - DNR
Subject:	RE: December 26th spill
Attachments:	122613 6 Oil Spill Map.pdf

## Hi John,

Attached is a map with the location of the spill. As of Thursday 1/2/14, we had excavated all soil that could be reached with our mini excavator. We were able to excavate some soil by hand under the pipe racks at that time as well. Since Friday 1/3/14, the temperatures have been very cold and the #6 oil has solidified to the point it cannot be excavated by hand. We have tried warming the area with insulation; however, temperatures have been so low, the insulation has not been effective. We could put steam hoses under the insulation but are concerned it will melt the frost and allow the spill to absorb into the soil. Our current plan it to try hand excavation again tomorrow afternoon when temperatures have increased 50+ degrees from last week to see if the remaining free product (about 5-10 gallons) is able to be excavated. Current totals are 10 yards of snow and 10 yards of soil have been excavated from the area. Let me know if you have any further questions.

Peter

From: Sager, John E - DNR [mailto:John.Sager@wisconsin.gov] Sent: Thursday, January 09, 2014 9:19 AM To: Peter Fredman Subject: December 26th spill

Hi Peter,

Can you please send me an update of the spill response for the spill on 12/26? I exchanged voice mail messages with Dave at the time at is seemed like everything was under control. With your update can you please also send a location of the spill. Thanks.

S John Sager

Emergency Response Coordinator / Hydrogeologist Remediation and Redevelopment Program Wisconsin Department of Natural Resources 107 Sutliff Avenue Rhinelander, WI 54501 (2) phone: (715) 365-8959 (2) fax: (715) 365-8932 (...) e-mail: john.sager@wi.gov

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From: Sent: To: Subject: Attachments: Peter Fredman <Peter.Fredman@calumetspecialty.com> Friday, February 07, 2014 13:55 Sager, John E - DNR SERTS ID 20131226NO16-1 122613 NR708 Immediate Action Report (final).pdf

1

John,

Attached is the Immediate Action Report required under NR708.05. I will submit the final report required under NR708.09 once the final cleanup is completed this spring. Thanks, Peter Fredman

Environmental Engineer Phone: 715-398-8434 Fax: 715-398-8209





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

February 7, 2014

John Sager Emergency Response Coordinator / Hydrogeologist Remediation and Redevelopment Program Wisconsin Department of Natural Resources 107 Sutliff Avenue Rhinelander, WI 54501

Re: #6 Fuel Oil Release SERTS ID 20131226NO16-1

Dear Mr. Sager,

Please find the attached report regarding the immediate action taken in response to the #6 fuel oil release reported the WDNR on December 26, 2013 as required under NR 708.05(6)(a).

It should be noted that Calumet is not requesting no further action at this time. It is Calumet's intention to complete addition clean-up activities as weather/site conditions permit in 2014.

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

Thank you,

**Peter Fredman** 

**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.

 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).

Due to the current weather/site conditions at and subsequent to the time of discharge, including the depth of frost at the site, it has been impracticable to eliminate all visual traces of #6 fuel oil at the site of the spill. Calumet is requesting the WDNR allow the spill to remain open until weather/site conditions allow excavation to be completed in areas where structural impediments prohibit the use of heavy equipment. 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; quarterquarter 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:361,711 Y:692,928

 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 was controlled utilizing an earthen berm 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. 0003085-07-0).

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: #6 Fuel Oil

Total Volume:

100 Gallons

# Final Disposition:

Due to the weather/site conditions at and subsequent to the time of discharge, there are still trace amounts of #6 fuel of remaining at the site of the discharge.

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. 0003085-07-0).

# Attachment A

NR 706.05 Hazardous Substance Discharge Notification Form

# (c)(1) Person Reporting Name: David Beattie Address 2407 Stinson Ave, Superior, WI 54880 Phone: 715-398-8455 (c)(2) Owner/Discharger Name: Calumet Superior, LLC Address 2407 Stinson Ave, Superior, WI 54880 Phone: 715-398-3533 (c)(3) Discharge Information Date: 12/26/2013 Time: 11:50 Duration: On-going (c)(3m) Discharge Location (WTM Coordinates) 361,711 X: 692,928 Y: (c)(4) Discharge Material Identity: #6 Fuel Oil Physical State: Solid 100 Gallons Quantity: (c)(5) Discharge Characteristics Physical: Solid Chemical: C18 + Hydrocarbons Hazardous: Combustable Toxilogical: Low Toxicity (c)(6) Cause Leaking Thermal Relief Line Cause: (c)(7) <u>Response Action</u> Action Taken: Clean Snow and Impacted Soil JR Jensen, In-Line Contractor(s): (c)(8) Movement **Thermal Relief Line** Source: Speed: <1 MPH Contained within Seconday Contaiment Destination: (c)(9) Impacts Human Health: None know at this time Environmental: Contaminated soil Water Supplies: None know at this time

# NR 706.05 Hazardous Substance Discharge Notification Form

(c)(10) Weather

Precipitation:	None	
Wind Direction:	WNW	
Velocity	5 MPH	

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

Agencies:

From:	Peter Fredman <peter.fredman@calumetspecialty.com></peter.fredman@calumetspecialty.com>
Sent:	Tuesday, April 29, 2014 13:07
То:	Sager, John E - DNR
Subject:	RE: December 26th 2013 spill SERTS ID 20131226NO16-1

Hi John,

On April 22<sup>nd</sup> and 23<sup>rd</sup> we completed additional remediation at the site.

This response included excavation under the pipe rack area by hand, a backhoe to excavate open areas, and a vacuum truck to recover water in the area.

The remaining free product was recovered (estimated 10 gallons) and visual evidence of impacted soil was removed. An additional 50 cubic yards of soil was removed from the area.

Please advise if the WDNR requests confirmation sampling for this spill. Due to the low lying nature of the release area and frequent precipitation since 4/23/14, a couple days without precipitation is needed to collect accurate samples. Thanks,

Peter Fredman Environmental Engineer Phone: 715-398-8434 Fax: 715-398-8209



From: Sager, John E - DNR [mailto:John.Sager@wisconsin.gov] Sent: Tuesday, April 29, 2014 9:26 AM To: Peter Fredman Subject: December 26th 2013 spill SERTS ID 20131226NO16-1

Hi Peter,

Do you have an update on this spill. I am trying to catch up on my open spills from 2013. Thanks.

John Sager

Emergency Response Coordinator / Hydrogeologist Remediation and Redevelopment Program Wisconsin Department of Natural Resources 1701 North 4<sup>th</sup> Street Superior, WI 54880 (2) phone: (715) 365-8959 (2) fax: (715) 392-7990 (...) e-mail: john.sager@wi.gov

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From:	Peter Fredman <peter.fredman@calumetspecialty.com></peter.fredman@calumetspecialty.com>
Sent:	Wednesday, April 30, 2014 08:25
То:	Sager, John E - DNR
Subject:	RE: December 26th 2013 spill SERTS ID 20131226NO16-1

I'll work on a plan to get some samples. Thanks, Peter

From: Sager, John E - DNR [mailto:John.Sager@wisconsin.gov]
Sent: Tuesday, April 29, 2014 4:44 PM
To: Peter Fredman
Subject: RE: December 26th 2013 spill SERTS ID 20131226NO16-1

Thank you for the update Peter. Waiting for a few days with no precipitation may be a giant hurdle but I think we should collect a few confirmation samples analyzed for PVOC and naphthalene.

Thanks.

🚔 John Sager

Emergency Response Coordinator / Hydrogeologist Remediation and Redevelopment Program Wisconsin Department of Natural Resources 1701 North 4<sup>th</sup> Street Superior, WI 54880 (2) phone: (715) 365-8959 (2) fax: (715) 392-7990 (I) e-mail: john.sager@wi.gov We are committed to service excellence. Click here to evaluate how I did.

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Thanks, Peter Fredman Environmental Engineer Phone: 715-398-8434 Fax: 715-398-8209



From: Sager, John E - DNR [mailto:John.Sager@wisconsin.gov] Sent: Tuesday, April 29, 2014 9:26 AM To: Peter Fredman Subject: December 26th 2013 spill SERTS ID 20131226N016-1

Hi Peter,

Do you have an update on this spill. I am trying to catch up on my open spills from 2013. Thanks.

John Sager

Emergency Response Coordinator / Hydrogeologist Remediation and Redevelopment Program Wisconsin Department of Natural Resources 1701 North 4<sup>th</sup> Street Superior, WI 54880 (2) phone: (715) 365-8959 (2) fax: (715) 392-7990 (E) e-mail: john.sager@wi.gov

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2

From:	Peter Fredman <peter.fredman@calumetspecialty.com></peter.fredman@calumetspecialty.com>
Sent:	Wednesday, May 28, 2014 15:12
То:	Sager, John E - DNR
Subject:	RE: December 26th 2013 spill SERTS ID 20131226NO16-1
Attachments:	052214 Sample Map.pdf; 052214 CT2 6OIL Spill.pdf
Follow Up Flag:	Follow up
Flag Status:	Completed

John,

I was finally able to collect samples. The results are attached. The two samples with high readings of naphthalene (S-3 and S-5) were outside the area free product was encountered in. Give me a call to discuss the next step. Thanks, Peter

From: Sager, John E - DNR [mailto:John.Sager@wisconsin.gov] Sent: Tuesday, April 29, 2014 4:44 PM To: Peter Fredman Subject: RE: December 26th 2013 spill SERTS ID 20131226NO16-1

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Sohn Sager

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Company Name/Address:		E	Billing Information:				Analysis/Container/Preservative				ervative	-	Chain of Custody Page of		
2407 Stinson Avenue Superior.WI 54880		David Beattie 2407 Stinson Avenue Superior,WI 54880				/Me OH	res			and the second se			L-A-B 5-C	SC I-E-N-C-E-S anon Road	
Report to: Peter Fredmann		E	mail to:	Iman @	Int. (or	M	- W	001						Mt. Juliet	TN 37122
Project Description: CT2 #6 (	21		City/Sate Collected	une nor/	WI		1	<						Phone: (80 Phone: (61	0) 767-5859 5) 758-5858
Phone: (715) 398-8455 FAX: (715) 398-8209	Client Project	<b>#</b> :	ESC Key:				moc	LR.						Fax: (61	5) 758-5859
Collected by: (print) Josh VanHoraurder	Site/Facility ID	#:	P.O.#:	67981				5					No.		
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Sample ID		Matrix*		Date	Time	Cntrs	VC	S					Sh	arks/Contaminant	Sample # (lab only)
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12065 Lebanon Rd. Mt. Juliet, TN 37122 (615) 758-5858 1-800-767-5859 Fax (615) 758-5859

Tax I.D. 62-0814289

Est. 1970

Peter Fredman Calumet Specialty Products 2407 Stinson Avenue Superior, WI 54880

# Report Summary

Wednesday May 28, 2014

Report Number: L700815 Samples Received: 05/23/14

Client Project: CT2 6 Oil

Description: CT2 6 Oil

The analytical results in this report are based upon information supplied by you, the client, and are for your exclusive use. If you have any questions regarding this data package, please do not hesitate to call.

Entire Report Reviewed By:

John Hawkins

ohn Hawkins , ESC Representative

### Laboratory Certification Numbers

A2LA - 1461-01, AIHA - 100789, AL - 40660, CA - 01157CA, CT - PH-0197, FL - E87487, GA - 923, IN - C-TN-01, KY - 90010, KYUST - 0016, NC - ENV375/DW21704/BIO041, ND - R-140. NJ - TN002, NJ NELAP - TN002, SC - 84004, TN - 2006, VA - 460132, WV - 233, AZ - 0612, MN - 047-999-395, NY - 11742, WI - 998093910, NV - TN000032011-1, TX - T104704245-11-3, OK - 9915, PA - 68-02979, IA Lab #364, EPA - TN002

Accreditation is only applicable to the test methods specified on each scope of accreditation held by ESC Lab Sciences.

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Page 1 of 9



Calumet Specialty Products 2407 Stinson Avenue Superior, WI 54880 12065 Lebanon Rd. Mt. Juliet, TN 37122 (615) 758-5858 1-800-767-5859 Fax (615) 758-5859

Tax I.D. 62-0814289

Est. 1970

REPORT OF ANALYSIS

May 28,2014

Date Received : May Description : CT2 6	23, 2014 Oil		ESC	: Sample # :	L700815-01	
Sample ID · S-1 6	STN		Sit	e ID :		
	111		Pro	ject # : CT2	6 Oil	
Collected By : Josh V Collection Date : 05/22/	, 14 10:47					
Parameter	Dry Result	Det. Limit	Units	Method	Date	Dil.
Total Solids	74.7		00	2540 G-2011	05/24/14	1
PVOCGRO						
Benzene	0.36	0.34	mg/kg	8021	05/24/14	505
Toluene	BDL	3.4	mg/kg	8021	05/24/14	505
Ethylbenzene	BDL	0.34	mg/kg	8021	05/24/14	505
m&p-Xylene	BDL	0.68	mg/kg	8021	05/24/14	505
o-Xylene	BDL	0.34	mg/kg	8021	05/24/14	505
Methyl tert-butyl ether	BDL	0.68	mg/kg	8021	05/24/14	505
Naphthalene	9.5	3.4	mg/kg	8021	05/24/14	505
1,3,5-Trimethylbenzene	1.5	0.68	mg/kg	8021	05/24/14	505
1,2,4-Trimethylbenzene	0.86	0.68	mg/kg	8021	05/24/14	505
TPH (GC/FID) Low Fractio	n 120	68.	mg/kg	8015	05/24/14	505
Surrogate recovery-% a,a,a-Trifluorotoluene(P	'ID) 98.0		% Rec.	8021	05/24/14	505

Results listed are dry weight basis. BDL - Below Detection Limit Det. Limit - Practical Quantitation Limit(PQL)

Note:

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Page 2 of 9



Peter Fredman

Calumet Specialty Products 2407 Stinson Avenue Superior, WI 54880 12065 Lebanon Rd. Mt. Juliet, TN 37122 (615) 758-5858 1-800-767-5859 Fax (615) 758-5859

Tax I.D. 62-0814289

Est. 1970

REPORT OF ANALYSIS

May 28,2014

Date Received	:	May 23,	2014		ESC	C Sample # :	L700815-02	2
Description	•	CT2 6 011			Sit	e ID :		
Sample ID	:	S-2 GIN			Due		C 011	
Collected By Collection Date	:	Josh V 05/22/14 11:14	1		Pro	oject # : CT2	6 011	
Parameter			Dry Result	Det. Limit	Units	Method	Date	Dil.
Total Solids			77.6		ę	2540 G-2011	05/24/14	1
PVOCGRO								
Benzene			BDL	0.13	mg/kg	8021	05/25/14	200
Toluene			BDL	1.3	mg/kg	8021	05/25/14	200
Ethylbenzene			1.5	0.13	mg/kg	8021	05/25/14	200
m&p-Xylene	42		1.5	0.26	mg/kg	8021	05/25/14	200
o-Xylene			1.4	0.13	mg/kg	8021	05/25/14	200
Methyl tert-bu	ity]	l ether	BDL	0.26	mg/kg	8021	05/25/14	200
Naphthalene			24.	1.3	mg/kg	8021	05/25/14	200
1,3,5-Trimethy	ylbe	enzene	13.	0.26	mg/kg	8021	05/25/14	200
1,2,4-Trimethy	ylbe	enzene	7.1	0.26	mg/kg	8021	05/25/14	200
TPH (GC/FID) ]	Low	Fraction	620	26.	mg/kg	8015	05/25/14	200
Surrogate recover	ery-	-00						
a,a,a-Trifluon	roto	oluene (PID)	98.7		% Rec.	8021	05/25/14	200

Results listed are dry weight basis. BDL - Below Detection Limit Det. Limit - Practical Quantitation Limit(PQL) Note: This report shall not be reproduced, except in full, without the written approval from ESC. The reported analytical results relate only to the sample submitted Reported: 05/28/14 08:59 Printed: 05/28/14 08:59

Page 3 of 9



Peter Fredman

Calumet Specialty Products 2407 Stinson Avenue Superior, WI 54880 12065 Lebanon Rd. Mt. Juliet, TN 37122 (615) 758-5858 1-800-767-5859 Fax (615) 758-5859

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Est. 1970

REPORT OF ANALYSIS

May 28,2014

Data Decidend	1.0	No	2014		ESC	C Sample # :	L700815-03	3
Description	:	May 23, CT2 6 Oil	2014					
		a 0 (au)			Sit	e ID :		
Sample 1D	•	S-3 61N			Pro	piect # · CT2	6 Oil	
Collected By Collection Date	:	Josh V 05/22/14 11:34				,jeee " . err	0 011	
Parameter			Dry Result	Det. Limit	Units	Method	Date	Dil.
Total Solids			78.4		Ŷ	2540 G-2011	05/28/14	1
PVOCGRO								
Benzene			0.023	0.021	mg/kg	8021	05/25/14	32.5
Toluene			BDL	0.21	mg/kg	8021	05/25/14	32.5
Ethylbenzene			BDL	0.021	mg/kg	8021	05/25/14	32.5
m&p-Xylene			BDL	0.041	mg/kg	8021	05/25/14	32.5
o-Xylene			0.022	0.021	mg/kg	8021	05/25/14	32.5
Methyl tert-b	uty	l ether	BDL	0.041	mg/kg	8021	05/25/14	32.5
Naphthalene			BDL	0.21	mg/kg	8021	05/25/14	32.5
1,3,5-Trimeth	ylbe	enzene	BDL	0.041	mg/kg	8021	05/25/14	32.5
1,2,4-Trimeth	ylbe	enzene	0.064	0.041	mg/kg	8021	05/25/14	32.5
TPH (GC/FID)	Low	Fraction	6.5	4.1	mg/kg	8015	05/25/14	32.5
Surrogate recov	ery-	- %						
a,a,a-Trifluo	roto	oluene(PID)	97.8		% Rec.	8021	05/25/14	32.5

Results listed are dry weight basis. BDL - Below Detection Limit Det. Limit - Practical Quantitation Limit(PQL) Note: This report shall not be reproduced, except in full, without the written approval from ESC. The reported analytical results relate only to the sample submitted Reported: 05/28/14 08:59 Printed: 05/28/14 08:59

Page 4 of 9



Peter Fredman

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Calumet Specialty Products

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Tax I.D. 62-0814289

Est. 1970

REPORT OF ANALYSIS

May 28,2014

Date Received : May 23		ESC	Sample # :	L700815-04	1	
			Sit	e ID :		
Sample ID : S-4 6IN			Dre	vioct # · · · · ·	e oil	
Collected By : Josh V Collection Date : 05/22/14 11:	50		FIC	Ject # . 012	. 0 011	
Parameter	Dry Result	Det. Limit	Units	Method	Date	Dil.
Total Solids	78.5		Dio	2540 G-2011	05/28/14	1
PVOCGRO						
Benzene	0.020	0.020	mg/kg	8021	05/25/14	31.5
Toluene	BDL	0.20	mg/kg	8021	05/25/14	31.5
Ethylbenzene	0.020	0.020	mg/kg	8021	05/25/14	31.5
m&p-Xylene	BDL	0.040	mg/kg	8021	05/25/14	31.5
o-Xylene	BDL	0.020	mg/kg	8021	05/25/14	31.5
Methyl tert-butyl ether	BDL	0.040	mg/kg	8021	05/25/14	31.5
Naphthalene	1.4	0.20	mg/kg	8021	05/25/14	31.5
1,3,5-Trimethylbenzene	BDL	0.040	mg/kg	8021	05/25/14	31.5
1,2,4-Trimethylbenzene	0.13	0.040	mg/kg	8021	05/25/14	31.5
TPH (GC/FID) Low Fraction	9.8	4.0	mg/kg	8015	05/25/14	31.5
a,a,a-Trifluorotoluene(PID)	98.6		% Rec.	8021	05/25/14	31.5

Results listed are dry weight basis. BDL - Below Detection Limit Det. Limit - Practical Quantitation Limit(PQL) Note: This report shall not be reproduced, except in full, without the written approval from ESC. The reported analytical results relate only to the sample submitted Reported: 05/28/14 08:59 Printed: 05/28/14 08:59

Page 5 of 9

	ESC				12065 Lebar Mt. Juliet, (615) 758-5 1-800-767-5 Fax (615) 7	non Rd. 7 TN 37122 5858 5859 758-5859	
					Tax I.D. 62	2-0814289	
YOUR	LAB OF CHOICE				Est. 1970		
	Peter Fredman Calumet Specialty Products 2407 Stinson Avenue Superior, WI 54880	REPORT	OF ANALYSIS	Мау	7 28,2014		
	Date Received : May 23, Description : CT2 6 Oil	2014		ESC	Sample # :	L700815-05	
	Sample ID : S-5 6IN			Sit	e ID :		
	Collected By : Josh V Collection Date : 05/22/14 12:0	8		Pro	oject # : CT2	6 011	
	Parameter	Dry Result	Det. Limit	Units	Method	Date	Dil.
	Total Solids	73.6		00	2540 G-2011	05/28/14	1
	PVOCGRO Benzene Toluene Ethylbenzene m&p-Xylene o-Xylene Methyl tert-butyl ether Naphthalene 1,3,5-Trimethylbenzene 1,2,4-Trimethylbenzene TPH (GC/FID) Low Fraction Surrogate recovery-%	1.8 BDL 5.4 1.0 1.8 BDL 39. 13. 6.2 980	$\begin{array}{c} 0.50 \\ 5.0 \\ 0.50 \\ 1.0 \\ 0.50 \\ 1.0 \\ 5.0 \\ 1.0 \\ 1.0 \\ 1.0 \\ 1.0 \\ 1.0 \end{array}$	mg/kg mg/kg mg/kg mg/kg mg/kg mg/kg mg/kg mg/kg mg/kg	8021 8021 8021 8021 8021 8021 8021 8021	05/25/14 05/25/14 05/25/14 05/25/14 05/25/14 05/25/14 05/25/14 05/25/14 05/25/14	735 735 735 735 735 735 735 735 735 735
	a, a, a-Trifluorotoluene (PID)	98.0		% Rec.	8021	05/25/14	735

Results listed are dry weight basis. BDL - Below Detection Limit Det. Limit - Practical Quantitation Limit(PQL) Note: This report shall not be reproduced, except in full, without the written approval from ESC. The reported analytical results relate only to the sample submitted Reported: 05/28/14 08:59 Printed: 05/28/14 08:59

Page 6 of 9

# Summary of Remarks For Samples Printed 05/28/14 at 08:59:42

TSR Signing Reports: 341 R4 - Rush: Three Day

Sample: L700815-01 Account: MUROILSWI Received: 05/23/14 10:00 Due Date: 05/29/14 00:00 RPT Date: 05/28/14 08:59 Sample: L700815-02 Account: MUROILSWI Received: 05/23/14 10:00 Due Date: 05/29/14 00:00 RPT Date: 05/28/14 08:59 Sample: L700815-03 Account: MUROILSWI Received: 05/23/14 10:00 Due Date: 05/29/14 00:00 RPT Date: 05/28/14 08:59 Sample: L700815-04 Account: MUROILSWI Received: 05/23/14 10:00 Due Date: 05/29/14 00:00 RPT Date: 05/28/14 08:59 Sample: L700815-05 Account: MUROILSWI Received: 05/23/14 10:00 Due Date: 05/29/14 00:00 RPT Date: 05/28/14 08:59

# LAB S.C.I.E.N.C.E.S

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Calumet Specialty Products Peter Fredman 2407 Stinson Avenue

Superior, WI 54880

12065 Lebanon Rd. Mt. Juliet, TN 37122 (615) 758-5858 1-800-767-5859 Fax (615) 758-5859

Tax I.D. 62-0814289

Est. 1970

# Quality Assurance Report Level II

L700815

May 28, 2014

		Laboratory	Blank			
Analyte	Result	Units	% Rec	Limit	Batch	Date Analyzed
Total Solids	< .1	9		8	WG722800	05/24/14 11:13
1,2,4-Trimethylbenzene	< .001	mg/kg			WG722805	05/24/14 14:29
1,3,5-Trimethylbenzene	< .001	mg/kg			WG722805	05/24/14 14:29
Benzene	< .0005	mg/kg			WG722805	05/24/14 14:29
Ethylbenzene	< .0005	mg/kg			WG722805	05/24/14 14:29
m&p-Xylene	< .001	mg/kg			WG722805	05/24/14 14:29
Methyl tert-butyl ether	< .001	mg/kg			WG722805	05/24/14 14:29
Naphthalene	< .005	mg/kg			WG722805	05/24/14 14:29
o-Xylene	< .0005	mg/kg			WG722805	05/24/14 14:29
Toluene	< .005	mg/kg			WG722805	05/24/14 14:29
TPH (GC/FID) Low Fraction	< .1	mg/kg			WG722805	05/24/14 14:29
a,a,a-Trifluorotoluene(PID)		% Rec.	99.60	80-120	WG722805	05/24/14 14:29
Total Solids	< .1	00			WG722853	05/28/14 06:21

			Duplicate				
Analyte	Units	Result	Duplicate	RPD	Limit	Ref Samp	Batch
Total Solids	8	82.6	81.7	1.12	5	L700755-05	WG722800
Total Solids	00 00	75.6	76.0	0.521	5	L700855-04	WG722853

		Laboratory Con	ntrol Sample			
Analyte	Units	Known Val	Result	% Rec	Limit	Batch
Total Solids	8	50	50.0	100.	85-115	WG722800
1,2,4-Trimethylbenzene	mg/kg	.05	0.0512	102.	80-120	WG722805
1,3,5-Trimethylbenzene	mg/kg	.05	0.0507	101.	80-120	WG722805
Benzene	mg/kg	.05	0.0525	105.	80-120	WG722805
Ethylbenzene	mg/kg	.05	0.0518	104.	80-120	WG722805
m&p-Xylene	mg/kg	.1	0.105	105.	80-120	WG722805
Methyl tert-butyl ether	mg/kg	.05	0.0527	105.	80-120	WG722805
Naphthalene	mg/kg	.05	0.0509	102.	80-120	WG722805
o-Xylene	mg/kg	.05	0.0507	101.	80-120	WG722805
Toluene	mg/kg	.05	0.0503	101.	80-120	WG722805
a,a,a-Trifluorotoluene(PID)				100.0	80-120	WG722805
TPH (GC/FID) Low Fraction	mg/kg	.5	0.469	93.7	80-120	WG722805
a,a,a-Trifluorotoluene(PID)				100.0	80-120	WG722805
Total Solids	8	50	50.0	100.	85-115	WG722853

121 2 3		haborator	y concror of	ampie bupite	acc		- 2 - N	
Analyte	Units	Result	Ref	%Rec	Limit	RPD	Limit	Batch
1,2,4-Trimethylbenzene	mg/kg	0.0516	0.0512	103.	80-120	0.630	20	WG722805
1,3,5-Trimethylbenzene	mg/kg	0.0516	0.0507	103.	80-120	1.84	20	WG722805
Benzene	mg/kg	0.0541	0.0525	108.	80-120	3.11	20	WG722805
Ethylbenzene	mg/kg	0.0533	0.0518	107.	80-120	2.89	20	WG722805
m&p-Xylene	mg/kg	0.107	0.105	107.	80-120	1.39	20	WG722805
Methyl tert-butyl ether	mg/kg	0.0529	0.0527	106.	80-120	0.390	20	WG722805
Naphthalene	mg/kg	0.0536	0.0509	107.	80-120	5.24	20	WG722805
o-Xylene	mg/kg	0.0517	0.0507	103.	80-120	2.01	20	WG722805

\* Performance of this Analyte is outside of established criteria. For additional information, please see Attachment A 'List of Analytes with QC Qualifiers.'

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# L.A.B S.C.I.E.N.C.E.S

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Tax I.D. 62-0814289

Est. 1970

#### Quality Assurance Report Level II

L700815

May 28, 2014

		Laboratory	Control	Sample Dupl	icate				
Analyte	Units	Result	Ref	%Rec		Limit	RPD	Limit	Batch
Toluene a.a.a-Trifluorotoluene(PID)	mg/kg	0.0517	0.0503	103.		80-120 80-120	2.78	20	WG72280 WG72280
TPH (GC/FID) Low Fraction	mg/kg	0.448	0.469	90.0		80-120	4.51	20	WG72280
a,a,a-Trifluorotoluene(PID)				98.50		80-120			WG72280
			Matrix S	spike					
Analyte	Units	MS Res	Ref Re	es TV	% Rec	Limit		Ref Samp	Batch
1,2,4-Trimethylbenzene	mg/kg	1.16	0.0076	.05	92.0	80-120		L700389-01	WG72280
1,3,5-Trimethylbenzene	mg/kg	1.14	0.0054	.05	91.0	80-120		L700389-01	WG72280
Benzene	mg/kg	1.19	0.0	.05	95.0	32-137		L700389-01	WG72280
Ethylbenzene	mg/kg	1.17	0.0033	.05	93.0	10-150		L700389-01	WG72280
m&p-Xylene	mg/kg	2.37	0.0074	.1	94.0	14-141		L700389-01	WG72280
Methyl tert-butyl ether	mg/kg	1.08	0.0050	.05	86.0	24-151		L700389-01	WG72280
Naphthalene	mg/kg	1.01	0.0117	.05	80.0	80-120		L700389-01	WG72280
o-Xylene	mg/kg	1.14	0.0044	2 .05	91.0	10-157		L700389-01	WG72280
Toluene	mg/kg	1.13	0.0045	.05	90.0	20-142		L700389-01	WG72280
a,a,a-Trifluorotoluene(PID)	1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 - 1990 -				101.0	80-120			WG72280
TPH (GC/FID) Low Fraction	mg/kg	11.9	0.0016	.5	95.0	80-120		L700389-01	WG72280
a,a,a-Trifluorotoluene(PID)					101.0	80-120			WG72280
		Matr	ix Spike	Duplicate					
Analyte	Units	MSD	Ref	%Rec	Limit	RPD	Limit	Ref Samp	Batch
1,2,4-Trimethylbenzene	mg/kg	1.28	1.16	102.	80-120	9.88	20	L700389-01	WG72280
1,3,5-Trimethylbenzene	mg/kg	1.26	1.14	100.	80-120	9.99	20	L700389-01	WG72280
Benzene	mg/kg	1.31	1.19	104.	32-137	9.60	39	L700389-01	WG72280
Ethylbenzene	mg/kg	1.29	1.17	103.	10-150	9.74	44	L700389-01	WG72280
m&p-Xylene	mg/kg	2.61	2.37	104.	14-141	9.84	44	L700389-01	WG72280
Methyl tert-butyl ether	mg/kg	1.26	1.08	100.	24-151	15.8	37	L700389-01	WG72280
Naphthalene	mg/kg	1.20	1.01	95.2	80-120	17.1	20	L700389-01	WG72280
o-Xylene	mg/kg	1.26	1.14	100.	10-157	9.87	44	L700389-01	WG72280
Toluene	mg/kg	1.25	1.13	99.9	20-142	9.88	42	L700389-01	WG72280
a, a, a-Trifluorotoluene (PID)	1000 at 1100 a			101.0	80-120				WG72280
TPH (GC/FID) Low Fraction	mg/ka	12.9	11.9	103.	80-120	7.86	20	L700389-01	WG72280
a a a-Trifluorotoluene(PID)				101 0	80-120				WG72280

Batch number /Run number / Sample number cross reference

WG722800: R2927667: L700815-01 02 WG722805: R2928832: L700815-01 02 03 04 05 WG722853: R2929088: L700815-03 04 05

\* Calculations are performed prior to rounding of reported values.
 \* Performance of this Analyte is outside of established criteria.
 For additional information, please see Attachment A 'List of Analytes with QC Qualifiers.'



Calumet Specialty Products Peter Fredman 2407 Stinson Avenue

Superior, WI 54880

#### Quality Assurance Report Level II

#### L700815

The data package includes a summary of the analytic results of the quality control samples required by the SW-846 or CWA methods. The quality control samples include a method blank, a laboratory control sample, and the matrix spike/matrix spike duplicate analysis. If a target parameter is outside the method limits, every sample that is effected is flagged with the appropriate qualifier in Appendix B of the analytic report.

> Method Blank - an aliquot of reagent water carried through the entire analytic process. The method blank results indicate if any possible contamination exposure during the sample handling, digestion or extraction process, and analysis. Concentrations of target analytes above the reporting limit in the method blank are qualified with the "B" qualifier.

> Laboratory Control Sample - is a sample of known concentration that is carried through the digestion/extraction and analysis process. The percent recovery, expressed as a percentage of the theoretical concentration, has statistical control limits indicating that the analytic process is "in control". If a target analyte is outside the control limits for the laboratory control sample or any other control sample, the parameter is flagged with a "J4" qualifier for all effected samples.

> Matrix Spike and Matrix Spike Duplicate - is two aliquots of an environmental sample that is spiked with known concentrations of target analytes. The percent recovery of the target analytes also has statistical control limits. If any recoveries that are outside the method control limits, the sample that was selected for matrix spike/matrix spike duplicate analysis is flagged with either a "J5" or a "J6". The relative percent difference (%RPD) between the matrix spike and the matrix spike duplicate recoveries is all calculated. If the RPD is above the method limit, the effected samples are flagged with a "J3" qualifier.

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May 28, 2014

DATE:	5-22-14
011011/011	

SHEET			OF	
BY: Riley	Loolos	- 5:1	Sample	Lachions

# MURPHY OIL USA, INC. Superior Refinery



From:	Peter Fredman <peter.fredman@calumetspecialty.com></peter.fredman@calumetspecialty.com>
Sent:	Thursday, June 05, 2014 15:35
То:	Sager, John E - DNR
Subject:	SERTS ID 20131226NO16-1 Update
Attachments:	060314 CT2 6OIL Spill Round 2.pdf
Follow Up Flag:	Follow up
Flag Status:	Flagged

John,

We completed additional remediation including excavation of 5 cubic yards of soil in the area of S-5 on 6/3/14. The sample results taken at the new lower depth are included.

In addition, a geoprobe was used in conjunction with an 11.7eV PID on 6/5/14 to determine the extent of contamination.

Let me know if you have any questions.

Peter Fredman Environmental Engineer Phone: 715-398-8434 Fax: 715-398-8209



Company Name/Address: Calumet Specialty Pr	oducts	Bill	ing Informa	tion:			Analysis/Container/Preservative					rvative	Chain of Custody Page 1 of E111	
2407 Stinson Avenue Superior.WI 54880			Superior, WI 54880			nb/MeDt	o Pres	10 Pres					SC	
Report to: Peter Fredman Project Description: CT2 #6 0:1 Phone: (715) 398-8455 FAX: (715) 398-8209 Client Project #:		Ema	Enail to: Peter, Fredman @ Clmt. com Collected Superior / WI ESC Key:				60ml A	CLR- A			12065 Let ML Juliet Phone: (80 Phone: (61 Fax: (61	Nanon Road , TN 37122 0) 767-5859 5) 758-5858 5) 758-5859		
Collected by: (print)	Site/Facility ID#	¥:	P.O.#: 67981			02	2							
Collected by (signature):	Rush? (La Sa Ne Tw	b MUST Be I me Day xt Day o Day	Be Notified )         Date Results Needed			No. of	OCG	20					CoCode MUROILSW (lab use only) Template/Prelogin	
Sample ID	Th Comp/Grab	Matrix*	25% Depth	Date	Time	Cntrs	Ne	T					Remarks/Contaminant	Sample # (lab only)
5-5-2	Grab	55	5'6"	6-3-2014	14:10	2	X	X					PID 50ppmv	L702393-01
														品語言語。
							-							
											-		-	
				-										
										36				
														The second second
Matrix: SS Soil/Solid GW - Ground	iwater MAN - V	VasteWater F	JW - Drinkir	Mater OT - (	Other		1.111	1				nH	Ter	
Remarks:	Mater WWW-V		Ju - Dinika	grader of - c	2010	52-	14 8	.784	29	17		Flow	Pti	ner
Relinquished by: (Signature)	Date:	Time:	Rece	nyed by: (Signat	ure)				Sam DFe	ples retu dEx 🛛	urned via: Courier		Condition:	(lab use only)
Relinguished by: (Signature)	Date:	-14 Time:	Rece	ived by: (Signat	ure)		Temp: 7			Temp: Bottles Receive		Ved: CoC Seals Intact	YNNA	
Relinquished by: (Signature)	Date:	Time:	Rec	eived for lab by	(Signature)	市場			Date	a: 4-14	Tim	ne: 1900	pH Checked:	NCF:



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Peter Fredman Calumet Specialty Products 2407 Stinson Avenue Superior, WI 54880

# Report Summary

Thursday June 05, 2014

Report Number: L702393 Samples Received: 06/04/14

Client Project:

Description: CT2 #6 Oil

The analytical results in this report are based upon information supplied by you, the client, and are for your exclusive use. If you have any questions regarding this data package, please do not hesitate to call.

Entire Report Reviewed By:

Jimm Hunt , ESC Representative

### Laboratory Certification Numbers

A2LA - 1461-01, AIHA - 100789, AL - 40660, CA - 01157CA, CT - PH-0197, FL - E87487, GA - 923, IN - C-TN-01, KY - 90010, KYUST - 0016, NC - ENV375/DW21704/BIO041, ND - R-140. NJ - TN002, NJ NELAP - TN002, SC - 84004, TN - 2006, VA - 460132, WV - 233, AZ - 0612, MN - 047-999-395, NY - 11742, WI - 998093910, NV - TN000032011-1, TX - T104704245-11-3, OK - 9915, PA - 68-02979, IA Lab #364, EPA - TN002

Accreditation is only applicable to the test methods specified on each scope of accreditation held by ESC Lab Sciences.

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Peter Fredman

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Calumet Specialty Products

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Tax I.D. 62-0814289

Est. 1970

REPORT OF ANALYSIS

June 05,2014

Date Received :	June 04.	2014		ESC	C Sample # :	L702393-01	6	
Description :	CT2 #6 Oil			044	TD			
Sample ID :	S-5-2 5-6IN			SIT	ce ID :			
Collected By : Collection Date :	Cliff Wright 06/03/14 14:10			Project # :				
Parameter		Dry Result	Det. Limit	Units	Method	Date	Dil.	
Total Solids		76.6		00	2540 G-2011	06/05/14	1	
PVOCGRO								
Benzene		0.84	0.037	mg/kg	8021	06/04/14	56.75	
Toluene		BDL	0.37	mg/kg	8021	06/04/14	56.75	
Ethylbenzene		1.2	0.037	mg/kg	8021	06/04/14	56.75	
m&p-Xylene		0.12	0.074	mg/kg	8021	06/04/14	56.75	
o-Xylene		0.27	0.037	mg/kg	8021	06/04/14	56.75	
Methyl tert-but	yl ether	0.17	0.074	mg/kg	8021	06/04/14	56.75	
Naphthalene		BDL	0.37	mg/kg	8021	06/04/14	56.75	
1,3,5-Trimethvlbenzene		1.1	0.074	mg/kg	8021	06/04/14	56.75	
1,2,4-Trimethyl	benzene	0.73	0.074	mg/kg	8021	06/04/14	56.75	
TPH (GC/FID) Lo	w Fraction	130	7.4	mg/kg	8015	06/04/14	56.75	
Surrogate recover	y-%							
a,a,a-Trifluoro	toluene (PID)	98.9		% Rec.	8021	06/04/14	56.75	

Results listed are dry weight basis. BDL - Below Detection Limit Det. Limit - Practical Quantitation Limit(PQL) Note: This report shall not be reproduced, except in full, without the written approval from ESC. The reported analytical results relate only to the sample submitted Reported: 06/05/14 08:50 Printed: 06/05/14 08:51

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# Attachment A List of Analytes with QC Qualifiers

Sample Number	Work Group	Sample Type	Analyte	Run ID	Qualifier
L702393-01	WG724512	SAMP	Naphthalene	R2934106	J5
	WG724512	SAMP	1,3,5-Trimethylbenzene	R2934106	J5
	WG724512	SAMP	1,2,4-Trimethylbenzene	R2934106	J5
	WG724512	SAMP	TPH (GC/FID) Low Fraction	R2934106	J5

#### Attachment B Explanation of QC Qualifier Codes

Qualifier	Meaning					
	-					

J5

The sample matrix interfered with the ability to make any accurate determination; spike value is high

#### Qualifier Report Information

ESC utilizes sample and result qualifiers as set forth by the EPA Contract Laboratory Program and as required by most certifying bodies including NELAC. In addition to the EPA qualifiers adopted by ESC, we have implemented ESC qualifiers to provide more information pertaining to our analytical results. Each qualifier is designated in the qualifier explanation as either EPA or ESC. Data qualifiers are intended to provide the ESC client with more detailed information concerning the potential bias of reported data. Because of the wide range of constituents and variety of matrices incorporated by most EPA methods, it is common for some compounds to fall outside of established ranges. These exceptions are evaluated and all reported data is valid and useable "unless qualified as 'R' (Rejected)."

#### Definitions

- Accuracy The relationship of the observed value of a known sample to the true value of a known sample. Represented by percent recovery and relevant to samples such as: control samples, matrix spike recoveries, surrogate recoveries, etc.
- Precision The agreement between a set of samples or between duplicate samples. Relates to how close together the results are and is represented by Relative Percent Differrence.
- Surrogate Organic compounds that are similar in chemical composition, extraction, and chromotography to analytes of interest. The surrogates are used to determine the probable response of the group of analytes that are chemically related to the surrogate compound. Surrogates are added to the sample and carried through all stages of preparation and analyses.
- TIC Tentatively Identified Compound: Compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates.

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# Summary of Remarks For Samples Printed 06/05/14 at 08:51:10

TSR Signing Reports: 341 R1 - Rush: Sameday

Sample: L702393-01 Account: MUROILSWI Received: 06/04/14 09:00 Due Date: 06/05/14 00:00 RPT Date: 06/05/14 08:50

# L·A·B S.C.I.E.N.C.E.S

# YOUR LAB OF CHOICE

Calumet Specialty Products Peter Fredman 2407 Stinson Avenue

Superior, WI 54880

12065 Lebanon Rd. Mt. Juliet, TN 37122 (615) 758-5858 1-800-767-5859 Fax (615) 758-5859

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#### Quality Assurance Report Level II

L702393

June 05, 2014

Laboratory Blank								
Analyte	Result	Units	% Rec	Limit	Batch	Date Analyzed	d	
1,2,4-Trimethylbenzene	< .001	mg/kg		÷	WG724512	06/04/14 12:3	29	
1,3,5-Trimethylbenzene	< .001	mg/kg			WG724512	06/04/14 12:2	29	
Benzene	< .0005	mg/kg			WG724512	06/04/14 12:2	29	
Ethylbenzene	< .0005	mg/kg			WG724512	06/04/14 12:2	29	
m&p-Xylene	< .001	mg/kg			WG724512	06/04/14 12:2	29	
Methyl tert-butyl ether	< .001	mg/kg			WG724512	06/04/14 12:2	29	
Naphthalene	< .005	mg/kg			WG724512	06/04/14 12:2	29	
o-Xylene	< .0005	mg/kg			WG724512	06/04/14 12:2	29	
Toluene	< .005	mg/kg			WG724512	06/04/14 12:2	29	
TPH (GC/FID) Low Fraction	< .1	mg/kg			WG724512	06/04/14 12:2	29	
a,a,a-Trifluorotoluene(PID)		% Rec.	98.70	80-120	WG724512	06/04/14 12:2	29	
Total Solids	< .1	80			WG724509	06/05/14 07:4	40	

Duplicate									
Analyte	Units	Result	Duplie	cate I	RPD	Limit	:	Ref Samp	Batch
Total Solids	용	74.9	76.6	2	2.21	5		L702393-01	WG724509
		Labora	tory Contro	ol Samole					
Analyte	Units	Known	Val	Resul	lt	% Rec		Limit	Batch
					(4.174)				
1,2,4-Trimethylbenzene	mg/kg	.05		0.0484		96.8		80-120	WG724512
1,3,5-Trimethylbenzene	mg/kg	.05		0.0479		95.9		80-120	WG724512
Benzene	mg/kg	.05		0.0486		97.3		80-120	WG724512
Ethylbenzene	mg/kg	.05		0.0489		97.7		80-120	WG724512
m&p-Xylene	mg/kg	.1		0.0979		97.9		80-120	WG724512
Methyl tert-butyl ether	mg/kg	.05		0.0493		98.6		80-120	WG724512
Naphthalene	mg/kg	.05		0.0485		97.1		80-120	WG724512
o-Xvlene	mg/kg	.05		0.0476		95.2		80-120	WG724512
Toluene	mg/kg	.05		0.0468		93.7		80-120	WG724512
a.a.a-Trifluorotoluene(PID)						101.0		80-120	WG724512
TPH (GC/FID) Low Fraction	ma/ka	. 5		0.414		82.8		80-120	WG724512
a,a,a-Trifluorotoluene(PID)						101.0		80-120	WG724512
Total Solids	8	50		50.0		100.		85-115	WG724509
		Laboratory	Control San	nple Dupl	licate				
Analyte	Units	Result	Ref	%Rec		Limit	RPD	Limit	Batch
1,2,4-Trimethylbenzene	mg/kg	0.0515	0.0484	103.		80-120	6.21	20	WG724512
1,3,5-Trimethylbenzene	mg/kg	0.0506	0.0479	101.		80-120	5.48	20	WG724512
Benzene	mg/kg	0.0509	0.0486	102.		80-120	4.56	20	WG724512
Ethylbenzene	mg/kg	0.0515	0.0489	103.		80-120	5.17	20	WG724512
m&p-Xvlene	mg/kg	0.103	0.0979	103.		80-120	5.40	20	WG724512
Methyl tert-butyl ether	mg/kg	0.0524	0.0493	105.		80-120	6.14	20	WG724512
Naphthalene	mg/kg	0.0507	0.0485	101.		80-120	4.32	20	WG724512
o-Xvlene	mg/kg	0.0503	0.0476	100.		80-120	5.51	20	WG724512
Toluene	mg/kg	0.0493	0.0468	98.0		80-120	5.10	20	WG724512
a.a.a-Trifluorotoluene(PID)	2. 2			98.90		80-120			WG724512
TPH (GC/FID) Low Fraction	mg/kg	0.475	0.414	95.0		80-120	13.8	20	WG724512
a,a,a-Trifluorotoluene(PID)				98.90		80-120	0.000	1.0005	WG724512
			Matrix Spil	ke					
Analyte	Units	MS Res	Ref Res	TV	% Rec	c Limi	t	Ref Samp	Batch
1,2,4-Trimethylbenzene	mg/kg	4.47	0.559	.05	140.*	80-1	20	L702393-01	WG724512

\* Performance of this Analyte is outside of established criteria. For additional information, please see Attachment A 'List of Analytes with QC Qualifiers.'



Calumet Specialty Products Peter Fredman 2407 Stinson Avenue

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o-Xylene

Toluene

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L702393-01

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WG724512

WG724512

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WG724512

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Est. 1970

Quality Assurance Report Level II

L702393

June 05, 2014

			Matrix S	pike					
Analyte	Units	MS Res	Ref Re	s TV	% Rec	Limit	i.	Ref Samp	Batch
1,3,5-Trimethylbenzene	mg/kg	4.79	0.867	.05	140.*	80-12	20	L702393-01	WG724512
Benzene	mg/kg	3.14	0.636	.05	88.0	32-13	37	L702393-01	WG724512
Ethylbenzene	mg/kg	3.80	0.937	.05	100.	10-15	50	L702393-01	WG724512
m&p-Xylene	mg/kg	5.68	0.0913	.1	98.0	14-14	11	L702393-01	WG724512
Methyl tert-butyl ether	mg/kg	2.52	0.128	.05	84.0	24-15	51	L702393-01	WG724512
Naphthalene	mg/kg	6.62	0.0	.05	230.*	80-12	20	L702393-01	WG724512
o-Xylene	mg/kg	2.84	0.213	.05	93.0	10-15	57	L702393-01	WG724512
Toluene	mg/kg	2.80	0.216	.05	91.0	20-14	12	L702393-01	WG724512
a,a,a-Trifluorotoluene(PID)					101.0	80-12	20		WG724512
TPH (GC/FID) Low Fraction	mg/kg	136.	101.	.5	120.*	80-12	20	L702393-01	WG724512
a,a,a-Trifluorotoluene(PID)					101.0	80-12	20		WG724512
		Mat	rix Spike	Duplicate					
Analyte	Units	MSD	Ref	%Rec	Limit	RPD	Limi	t Ref Samp	Batch
1,2,4-Trimethylbenzene	mg/kg	4.66	4.47	144.*	80-120	4.02	20	L702393-01	WG724512
1,3,5-Trimethylbenzene	mg/kg	4.99	4.79	145.*	80-120	4.07	20	L702393-01	WG724512
Benzene	mg/kg	3.32	3.14	94.5	32-137	5.47	39	L702393-01	WG724512
Ethylbenzene	mg/kg	4.01	3.80	108.	10-150	5.27	44	L702393-01	WG724512
m&p-Xylene	mg/kg	5.98	5.68	104.	14-141	5.12	44	L702393-01	WG724512
Methyl tert-butyl ether	mg/kg	2.72	2.52	91.2	24-151	7.37	37	L702393-01	WG724512
Naphthalene	mg/kg	7.09	6.62	250.*	80-120	6.91	20	L702393-01	WG724512

2.84

136.

mg/kg

mg/kg

mg/kg 130.

2.98

2.95

97.4 96.3

103.

99.30

99.30

10-157

20-142

80-120

80-120

80-120

4.72

5.10

4.13

44

42

20

Batch number /Run number / Sample number cross reference

WG724512: R2934106: L702393-01 WG724509: R2934628: L702393-01

a,a,a-Trifluorotoluene(PID)

a,a,a-Trifluorotoluene(PID)

TPH (GC/FID) Low Fraction

\* \* Calculations are performed prior to rounding of reported values.

\* Performance of this Analyte is outside of established criteria.

For additional information, please see Attachment A 'List of Analytes with QC Qualifiers.'



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#### Quality Assurance Report Level II

#### L702393

The data package includes a summary of the analytic results of the quality control samples required by the SW-846 or CWA methods. The quality control samples include a method blank, a laboratory control sample, and the matrix spike/matrix spike duplicate analysis. If a target parameter is outside the method limits, every sample that is effected is flagged with the appropriate qualifier in Appendix B of the analytic report.

> Method Blank - an aliquot of reagent water carried through the entire analytic process. The method blank results indicate if any possible contamination exposure during the sample handling, digestion or extraction process, and analysis. Concentrations of target analytes above the reporting limit in the method blank are qualified with the "B" qualifier.

Laboratory Control Sample - is a sample of known concentration that is carried through the digestion/extraction and analysis process. The percent recovery, expressed as a percentage of the theoretical concentration, has statistical control limits indicating that the analytic process is "in control". If a target analyte is outside the control limits for the laboratory control sample or any other control sample, the parameter is flagged with a "J4" qualifier for all effected samples.

Matrix Spike and Matrix Spike Duplicate - is two aliquots of an environmental sample that is spiked with known concentrations of target analytes. The percent recovery of the target analytes also has statistical control limits. If any recoveries that are outside the method control limits, the sample that was selected for matrix spike/matrix spike duplicate analysis is flagged with either a "J5" or a "J6". The relative percent difference (%RPD) between the matrix spike and the matrix spike duplicate recoveries is all calculated. If the RPD is above the method limit, the effected samples are flagged with a "J3" qualifier. 12065 Lebanon Rd. Mt. Juliet, TN 37122 (615) 758-5858 1-800-767-5859 Fax (615) 758-5859

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