Appendix B - Waste Analysis Plan 2017

# 1.0 General Waste Analysis – §264.13

Wynnewood Refining Company, LLC ("WRC" or the "Facility") is a generator of hazardous waste and does not receive hazardous waste generated off site. Tank 2007 ("T-2007") is used to store API Separator Sludge (K051) and Dissolved Air Flotation Float (K048) generated by onsite operations. API Separator Sludge (K051) is a dark, oily, water-based sludge with solids that are generally heavier than the aqueous phase. That waste is produced in the Facility's API Separators, also known as oil/water separators. Process wastewaters flow to the API Separators where suspended solids and heavier oils settle out as sludge. The sludge is periodically pumped from the API Separators for storage in Tank 2007 ("T-2007") prior to off-site disposal. Dissolved Air Flotation ("DAF") is an alternative clarification process that uses micro air bubbles to attach and float flocculated particles and suspended solids, referred to as DAF Float (K048), to the water's surface for removal.

# 1.1 Analytical Methods - §264.13(b)(2)

The basis for listing both K048 and K051 hazardous wastes is because of the possible presence of hexavalent chromium and lead, as found in 40 CFR §261 Appendix VII. Treatment standards under the Land Disposal Restrictions are based on constituent concentrations in the waste or waste extract rather than on specified technologies. Those concentrations are presented in Attachment 1 to this Waste Analysis Plan. Analytical requirements for the waste stored in T-2007 are limited to information required by the TSDF for proper treatment and disposal. Analytical parameters will include flash point, pH, total cyanide, extractable sulfide, TCLP lead, TCLP chromium, TCLP nickel, BETX, and semi-volatile organic compounds. Analytical methods are as follows:

Analytical Parameter	Analytical Method
Flash Point	SW-846 Test Method 1010A
рН	SW-846 Test Method 9040/9045D
Total Cyanide	SW-846 Test Method 9014 or 9213
Extractable Sulfide	SW-846 Test Method 9034 or 9215
Lead (TCLP)	SW-846 Test Method 6010B/6020A
Chromium (TCLP)	SW-846 Test Method 6010B/6020A
Nickel (TCLP)	SW-846 Test Method 6010B/6020A
BETX (benzene, ethylbenzene, toluene and xylene)	SW-846 Test Method 8260B/8021B
Semi-Volatile Organic Compounds	SW-846 Test Method 8270C

Analytical Data on the K048 DAF float and K051 API Separator sludge are included in Attachment 2 to this Waste Analysis Plan.

# 1.2 Sampling Methods - §264.13(b)(3)

Samples are obtained in such a manner as to ensure that it is representative of all waste stored in T-2007. Samples are managed to ensure the identity and integrity of the sample stays intact, including thorough labeling and use of chain-of-custody. Samples for analysis are obtained from the "deepwell", which is a sump at the API Separator, using a coliwasa or similar sampling device.

# 1.3 Frequency of Analysis - §264.13(b)(4)

The wastes accumulated in T-2007 are listed wastes with consistent physical properties and chemical compositions. A sample of the combined API Separator sludge and DAF float will be obtained at least annually for analysis by a contract laboratory. Although the processes generating the wastes, and therefore the physical properties and chemical compositions of the wastes, are not expected to change, should WRC be notified, or have reason to believe, that the process or operation generating the hazardous wastes, has changed, analyses would be repeated.



#### Wynnewood Refining Company Waste Analysis Plan Attachment 1 Land Disposal Restriction Treatment Standards

		Regulated Hazardous Constitue	ent	Wastewater	Nonwastewater
Waste Code		Common name	CAS number	Concentration in mg/L	Concentration in mg/kg unless noted as "mg/L TCLP"
	Dissolved air flotation (DAF) float from the petroleum refining industry.	Benzene	71-43-2	0.14	10
		Benzo(a)pyrene	50-32-8	0.061	3.4
		bis(2-Ethylhexyl)phthalate	117-81-7	0.28	28
		Chrysene	218-01-9	0.059	3.4
		Di-n-butyl phthalate	84-74-2	0.057	28
		Ethylbenzene	100-41-4	0.057	10
		Fluorene	86-73-7	0.059	NA
		Naphthalene	91-20-3	0.059	5.6
		Phenanthrene	85-01-8	0.059	5.6
		Phenol	108-95-2	0.039	6.2
		Pyrene	129-00-0	0.067	8.2
		Toluene	108-88-33	0.080	10
		Xylenes-mixed isomers (sum of o-, m-, and p- xylene concentrations)	1330-20-7	0.32	30
		Chromium (Total)	7440-47-3	2.77	0.60 mg/L TCLP
		Cyanides (Total)	57-12-5	1.2	590
		Lead	7439-92-1	0.69	NA
		Nickel	7440-02-0	NA	11 mg/L TCLP

#### Wynnewood Refining Company Waste Analysis Plan Attachment 1 Land Disposal Restriction Treatment Standards

		Regulated Hazardous Constituent	Regulated Hazardous Constituent				
Waste Code	Waste Description	Common name	CAS number	Concentration in mg/L	Concentration in mg/kg unless noted as "mg/L TCLP"		
K051	API separator sludge from the petroleum refining industry.	Acenaphthene	83-32-9	0.059	NA		
		Anthracene	120-12-7	0.059	3.4		
		Benz(a)anthracene	56-55-3	0.059	3.4		
		Benzene	71-43-2	0.14	10		
		Benzo(a)pyrene	50-32-8	0.061	3.4		
		bis(2-Ethylhexyl)phthalate	117-81-7	0.28	28		
		Chrysene	218-01-9	0.059	3.4		
		Di-n-butyl phthalate	105-67-9	0.057	28		
		Ethylbenzene	100-41-4	0.057	10		
		Fluorene	86-73-7	0.059	NA		
		Naphthalene	91-20-3	0.059	5.6		
		Phenanthrene	85-01-8	0.059	5.6		
		Phenol	108-95-2	0.039	6.2		
		Pyrene	129-00-0	0.067	8.2		
		Toluene	108-88-3	0.08	10		
		Xylenes-mixed isomers (sum of o-, m-, and p-xylene concentrations)	1330-20-7	0.32	30		
		Cyanides (Total)	57-12-5	1.2	590		
		Chromium (Total)	7440-47-3	2.77	0.60 mg/L TCLP		
		Lead	7439-92-1	0.69	NA		
		Nickel	7440-02-0	NA	11 mg/L TCLP		

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PO Box 305					
Wynnewood, OK 73098-					
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	1532194				
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	DAF Float				
Administrative					
	* - -		•		
Reactivity	Review				
				- I	
EPA 9045D,					
Corrosivity (Solids by pH)	Non-Corrosive				
Distillations					
ASTM D 4978-95/SW 9031,					
Sulfide Screen	POSITIVE				
					······································
EPA 9014, mg/kg					
Total Cyanide	<8.37	· · · · · · · · · · · · · · · · · · ·			
Gravimetrics	-				
SM2540 G-1997 /MOD, %	-				
Total Solids for Dry Wt	16.8				
Metals					
EPA 6020A, mg/kg	-				
Lead, Total (7439-92-1)	76.2				
Barium (7440-39-3)	498				
Arsenic, Total (7440-38-2)	16.0				
Chromium, Total (7440-47-3)	85.1				
Selenium, Total (7782-49-2)	10.1				
Silver, Total (7440-22-4)	0.744				
Cadmium, Total (7440-43-9)	0.714				
EDA 6020A mall					······································
EPA 6020A, mg/L	-0.050				
TCLP Arsenic (7440-38-2) TCLP Barium (7440-39-3)	<0.050				
TCLP Cadmium (7440-39-3)	1.06				
TCLP Chromium (7440-47-3)	<0.005 0.0937				
TCLP Lead (7439-92-1)	<0.050				
TCLP Selenium (7782-49-2)	<0.050				
TCLP Silver (7440-22-4)	<0.050				
EPA 7470A, mg/L					
TCLP Mercury (7439-97-6)	<0.002				
EPA 7471A, mg/kg					
EFA 7471A, 1197Kg Mercury (7439-97-6)	1 49				
Organics	1.48				
OVERING					

	Ana-Lab Corp	. P.O. Box 9	000	Kilgore	, TX 75	5663	
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	<b>Project</b> ]	Recult (	2111	nmar			Page 2 of 3
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Wynnewood Refining Co				WYN	N K		758780
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906 South Powell					De	əfault	
PO Box 305							
Wynnewood, OK 73098-	····	4					-
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Organics							
EPA 8260B, mg/L			1				
TCLP 1,2-Dichloroethane (107-06-2)	<0.010						
TCLP 1,1-Dichloroethene (75-35-4)	<0.010		1				
TCLP 1,4 Dichlorobenzene (106-46-7)	<0.010						
TCLP Benzene (71-43-2)	0.622						
TCLP Carbon tetrachloride (56-23-5)	<0.010						
TCLP Chlorobenzene (108-90-7)	<0.010		<u> </u>				
TCLP Chloroform (67-66-3) TCLP MEK (78-93-3)	<0.010 <0.050		ļ				
TCLP Tetrachloroethylene (127-18-4)	<0.030						
TCLP Trichloroethylene (79-01-6)	<0.010						
TCLP Vinyl chloride (75-01-4)	<0.010						
EPA 8260B, ug/kg							
Benzene (71-43-2)	483000						
Carbon Tetrachloride (56-23-5) Chlorobenzene (108-90-7)	<2980 <2980		<u> </u>				· · · · · · · · · · · · · · · · · · ·
Chloroform (67-66-3)	<2480		ļ			· · ·	
p-Dichlorobenzene (106-46-7)	<2980					<u></u>	
1,2-Dichloroethane (107-06-2)	<5950						
1,1-Dichloroethylene (75-35-4)	<2980						
Methyl ethyl ketone (Butanone) (78-93	<14900						
Tetrachloroethylene (127-18-4)	<2980						
Trichloroethylene (79-01-6)	<2980						
Vinyl chloride (75-01-4)	<2980						
EPA 8270C, mg/L							
TCLP bis(2-Chloroethyl)ether (111-44-	<0.010						
TCLP 3&4-Methylphenol (m&p-Creso	0.142				<del></del>		
TCLP 2-Methylphenol (o-Cresol)	<0.100						
TCLP 2,4-Dinitrotoluene (121-14-2)	<0.020						
TCLP Hexachlorobenzene (118-74-1)	<0.010						
TCLP Hexachlorobutadiene (87-68-3)	<0.0103						
TCLP Hexachloroethane (67-72-1)	<0.020						
TCLP Nitrobenzene (98-95-3)	<0.010			· .			
TCLP Pentachlorophenol (87-86-5)	<0.050						
TCLP Pyridine (Reg. Limit 5) (110-86-1 TCLP Total Cresols (Reg Lim 200) (10)	<0.0135						
TCLP 10tal Cresols (Reg Lim 200) (10) TCLP 2,4,6-Trichlorophenol (88-06-2)	<b>0.142</b>						
TCLP 2,4,5-Trichlorophenol (88-06-2)	<0.020	······································					
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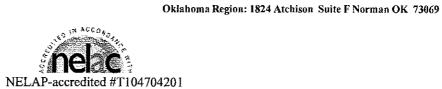
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	DAF Float					
Organics						
EPA 8270C, ug/kg						
Bis(2-chloroethyl)ether (111-44-4)	<9940					
3&4-Methylphenol (m&p-Cresol) (MEP	<26200					
2-Methylphenol (o-Cresol) (95-48-7)	<99400					
2,4-Dinitrotoluene (121-14-2)	<12800					
Hexachlorobenzene (118-74-1)	<9940					
Hexachlorobutadiene (87-68-3)	<9940					
Hexachloroethane (67-72-1)	<9940					
Nitrobenzene (98-95-3)	<9940					
Pentachlorophenol (87-86-5)	<11700			l		
Pyridine (110-86-1)	<16800					
2,4,6-Trichlorophenol (88-06-2)	<26600					
2,4,5-Trichlorophenol (95-95-4)	<9940					
Wet Bench						
Ana-Lab Method, Reactivity with Water	NON-REACTIVE					
EPA 1010A, Degrees F Flash Point (Reg. Limit 140.0 F)	>201					
EPA 9031, mg/kg Extractable Sulfide	7740					
EPA 9045D, SU pH Measured in Water	8.80					

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Paul Zhang, Ph.D., Quality Director

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		Re	sults					
1532	194 DAF Float					Rec	eived: 10/1	2/2016
	hemical Mat			<i>////</i>	17.0			
iona a C		Collected by: Client	Aj	ffiliation: W	ynnewood Refin	ing C	10/11/2016	11:00:00
		Prepared:	10/21/2016	16:46:04	Calculated		10/21/2016	16:46:04 CA
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bottle
N	Reactivity	Review				0		
Ana-L	ab Method	Prepared: 686946	10/13/2016	11:25:00	Analyzed 686.	946	10/13/2016	11:25:00 TD
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bottle
	Reactivity with Water	NON-REAC	TIVE					02
ASTM	1 D 4978-95/SW 9031	Prepared: 686775	10/13/2016	13:20:00	Analyzed 686	775	10/13/2016	13:20:00 GH
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bottle
	Sulfide Screen	POSITIVE		1.00	Ŭ	500		01
EPA 1	1010A	Prepared: 687259	10/17/2016	10:05:00	Analyzed 687.	259	10/17/2016	10:05:00 Nh
	Parameter	Results	Units	RL	Flags	MAL		Bottle
N	Flash Point (Reg. Limit 140.0 F)	>201	Degrees					01
EPA 6	020A	Prepared: 686734	10/13/2016	11:30:00	Analyzed 6870	096	10/14/2016	20:37:00 CL
	Parameter	Results	Units	RL	Flags	MAL		Bottle
N	Arsenic, Total	16.0 *	mg/kg	0.586	0	41.0	7440-38-2	03
N	Barium	498 *	mg/kg	0.293			7440-39-3	03
N	Cadmium, Total	0.714 *	mg/kg	0.293		39.0	7440-43-9	03
N	Chromium, Total	85.1 *	mg/kg	0.293			7440-47-3	03
N	Lead, Totai	76.2 *	mg/kg	0.293		300	7439-92-1	03
N	Selenium, Total	10.1 *	mg/kg	0.881		100	7782-49-2	03
N	Silver, Total	0.744 *	mg/kg	0.293			7440-22-4	03
EPA 6		Prepared: 687060	10/14/2016	15:31:00	Analyzed 6872	281	10/17/2016	17:01:00 CL
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bottle
N	TCLP Arsenic	<0.050	mg/L	0.050		5,00	7440-38-2	11
N	TCLP Barium	1.06	mg/L	0.050		100	7440-39-3	11
N	TCLP Cadmium	<0.005	mg/L	0.005		1.00	7440-43-9	11
N	TCLP Chromium	0.0937	mg/L	0.050		5.00	7440-47-3	11
N	TCLP Lead	<0.050	mg/L	0.050		5.00	7439-92-1	11
N	TCLP Selenium	<0.050	mg/L	0.050		1.00	7782-49-2	11
N	TCLP Silver	<0.050	mg/L	0.050		5.00	7440-22-4	11
	* Dry Weight Basis							

Corporate Shipping: 2600 Dudley Rd. Kilgore, TX 75662



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Results

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Received: 10/12/2016

#### 1532194 DAF Float

lid & Cl	hemical Mat	Collected by: Client	Aţ	filiation: W	ynnewood Re	fining C	10/11/2016	11:00:00	
EPA 7	7470A	Prepared: 686953	10/14/2016	10:45:00	Analyzed 6	87184	10/17/2016	11:59:00	LF
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bo	ttle
Ν	TCLP Mercury	<0.002	mg/L	0.002	Ū	0.200	7439-97-6	08	
EPA 7	7471A	Prepared: 686873	10/14/2016	08:30:00	Analyzed 6	87224	10/17/2016	13:35:00	LP.
	Parameter	Results	Units	RL	Flags	MAL	CAS	Во	ttle
N	Mercury * Dry Weight Basis	1.48 *	mg/kg	0.116		17.0	7439-97-6	07	
EPA 8	3260B	Prepared: 687230	10/17/2016	14:45:00	Analyzed 6	87528	10/18/2016	15:57:00	JRI
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bo	ttle
N	TCLP 1,1-Dichloroethene	<0.010	mg/L	0.010	_	0.700	75-35-4	20	
N	TCLP 1,2-Dichloroethane	<0.010	mg/L	0.010		0.500	107-06-2	20	
N	TCLP 1,4 Dichlorobenzene	<0.010	mg/L	0.010		7 50	106-46-7	20	
Ν	TCLP Benzene	0.622	mg/L	0.010		0.500	71-43-2	20	
N	TCLP Carbon tetrachloride	<0.010	mg/L	0.010		0.500	56-23-5	20	
N	TCLP Chlorobenzene	<0.010	mg/L	0.010		100	108-90-7	20	
Ν	TCLP Chloroform	<0.010	mg/L	0.010		6.00	67-66-3	20	
N	TCLP MEK	<0.050	mg/L	0.050		200	78-93-3	20	
N	TCLP Tetrachloroethylene	<0.010	mg/L	0.010		0.700	127-18-4	20	
N	TCLP Trichloroethylene	<0.010	mg/L	0.010		0.500	79-01-6	20	
N	TCLP Vinyl chloride	<0.010	mg/L	0.010		0.200	75-01-4	20	
EPA 8	260B	Prepared: 688007	10/20/2016	15:52:00	Analyzed 6	88007 .	10/20/2016	15:52;00	JRI
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bo	ttle
Ņ	1,1-Dichloroethylene	<2980 *	ug/kg	2980	(	50.1	75-35-4	01	
Ν	1,2-Dichloroethane	<5950 *	ug/kg	5950	(	13.7	107-06-2	01	
Ν	Benzene	483000 *	ug/kg	2980	(	26.0	71-43-2	01	
Ν	Carbon Tetrachloride	<2980 *	ug/kg	2980	(	61.9	56-23-5	01	
Ν	Chlorobenzene	<2980 *	ug/kg	2980	(	1090	108-90-7	01	
Ν	Chloroform	<2480 *	ug/kg	2480	(	417	67-66-3	01	
Ν	Methyl ethyl ketone (Butanone)	<14900 *	ug/kg	14900	(	29300	) 78-93-3	01	
Ν	p-Dichlorobenzene	<2980 *	ug/kg	2980	(	5.00	106-46-7	01	
N	Tetrachloroethylene	<2980 *	ug/kg	2980	(	50.2	127-18-4	01	
N	Trichloroethylene	<2980 *	ug/kg	2980	(	33.6	79-01-6	01	
N	Vinyl chloride * Dry Weight Basis	<2980 *	ug/kg	2980	(	22.3	75-01-4	01	
EPA 8	270C	Prepared: 687178	10/17/2016	10:30:00	Analyzed 6	88389 1	0/24/2016	12:43:00	SLC
	Parameter	Results	Units	RL	Flags	MAL	CAS	Boi	tle
N	2,4,5-Trichlorophenol	<9940 *	ug/kg	9940		33800	) 95-95-4	14	
N	2,4,6-Trichlorophenol	<26600 *	ug/kg	2 <b>6</b> 600		594	88-06-2	14	
N	2,4-Dinitrotoluene	<12800 *	ug/kg	12800		5.32	121-14-2	14	
Ν	2-Methylphenol (o-Cresol)	<99400 *	ug/kg	99400		7120	95-48-7	14	
Ν	3&4-Methylphenol (m&p-Cresol)	<26200 *	ug/kg	26200		632	MEPH34	14	
N	Bis(2-chloroethyl)ether	<9940 *	ug/kg	9940		2.11	111-44-4	14	

Corporate Shipping: 2600 Dudley Rd. Kilgore, TX 75662



Oklahoma Region: 1824 Atchison Suite F Norman OK 73069

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Page 3 of 6

#### 1532194 DAF Float

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EPA 8	270C	Prepared: 687178	10/17/2016	10:30:00	Analyzed 688	3389	10/24/2016	12:43:00	SL
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bc	ottle
N	Hexachlorobenzene	<9940 *	ug/kg	9940	U	1100	118-74-1	14	
N	Hexachlorobutadiene	<9940 *	ug/kg	9940		1370	87-68-3	14	
N	Hexachloroethane	<9940 *	ug/kg	9940		1840	67-72-1	14	
Ν	Nitrobenzene	<9940 *	ug/kg	9940		87.9	98-95-3	14	
N	Pentachlorophenol	<11700 *	ug/kg	11700	)	3.58	87-86-5	14	
N	Pyridine	<16800 *	ug/kg	16800	)	69.0	110-86-1	14	
EPA 82	270C	Prepared: 687197	10/17/2016	12:00:00	Analyzed 688	360	10/19/2016	16:36:00	SL
	Parameter	Results	Units	RL	Flags	MAL	CAS	Ba	ottle
N	TCLP 2,4,5-Trichlorophenol	<0.050	mg/L	0.050	•	1.00	95-95-4	17	
N	TCLP 2,4,6-Trichlorophenol	<0.020	mg/L	0.020		2.00	88-06-2	17	
N	TCLP 2,4-Dinitrotoluene	<0.020	mg/L	0.020		0.130		17	
N	TCLP 2-Methylphenol (o-Cresol)	<0.100	mg/L	0,100		200		17	
N	TCLP 3&4-Methylphenol (m&p-Creso	0.142	mg/L	0.080		200		17	
N	TCLP bis(2-Chloroethyl)ether	<0.010	mg/L	0.010		0.100	) 111-44-4	17	
N	TCLP Hexachlorobenzene	<0.010	mg/L	0.010		0.130	) 118-74-1	17	
N	TCLP Hexachlorobutadiene	< 0.0103	 mg/L	0.010		0.500		17	
N	TCLP Hexachloroethane	<0.020	mg/L	0.020		3.00	67-72-1	17	
N	TCLP Nitrobenzene	<0.010	mg/L	0.010		2.00	98-95-3	17	
N	TCLP Pentachlorophenol	<0.050	mg/L	0.050		100	87-86-5	17	
N	TCLP Pyridine (Reg. Limit 5)	<0.0135	mg/L	0.013		5.00	110-86-1	17	
EPA 82		Prepared: 687197	10/17/2016	12:00:00	Calculated 688	360	10/24/2016	13:22:11	CA
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bo	
N	TCLP Total Cresols (Reg Lim 200) * Dry Weight Basis	0.142	mg/L	0.080	_	200	108-39-4,ec		
EPA 90	014	Prepared: 688011	10/21/2016	09:40:00	Analyzed 688	203	10/21/2016	14:47:00	ML
	Parameter	Results	Units	RL	Flags	MAL	CAS	Bo	
N	Total Cyanide	<8.37	mg/kg	8.37	P	250	0110	21	
		-0627	mg/kg	0.57	1	200			
EPA 90	031	Prepared: 687599	10/17/2016	07:45:00	Analyzed 687	599	10/17/2016	07:45:00	BA.
	Parameter	Results	Units	RL	Flags	MAL	CAS	Boi	ttle
N	Extractable Sulfide * Dry Weight Basis	7740 *	mg/kg	119	Р			01	
EPA 90	045D	Prepared:	10/20/2016	13:10:25	Calculated		10/20/2016	13:10:25	CA
	Parameter	Results	Units	RL	Flags	MAL	CAS	· Boi	ttle
N	Corrosivity (Solids by pH)	Non-Corros	ive		~				
EPA 90		Prepared: 686992	10/14/2016	09:20:00	Analyzed 686	992 I	10/14/2016	09:20:00	SEI
	Parameter	Results	Units	RL	Flags	MAL	CAS	Boi	
N	pH Measured in Water	8.8 @ 21 C	SU	2.00				01	
SM254	0 G-1997 /MOD	Prepared: 686835	10/13/2016	10:00:00	Analyzed 686	835	10/13/2016	10:00:00	JW
	Parameter	Results	Units	RL	Flags	MAL	CAS	Boi	ttle
oorate SI	hipping: 2600 Dudley Rd. Kilgore, TX 75662	1			na Region: 182		on Suite F Noi	rman OK	7306!



	Ana-Lal	o Corp. P.O.	Box 9000	) Kilş	gore, TX 7	5663		
ANALAR OKP. HE COMPLETE SERVICE	Resul	-0551 FAX 903/984 Employee Owned		Caring	a-lab.com Continual Imp nted: 10/24/2016		Page 4	4 of (
1532194 DAF I	rloat				R	eceived: 10/	12/2016	
olid & Chemical Mat		Collected by: Client	Aj	ffiliation: V	Vynnewood Refining	C 10/11/2016	11:00:00	
SM2540 G-1997 /MOL	)	Prepared: 686835	10/13/2016	10:00:00	Analyzed 686835		10:00:00	JWF
Parameter N Total Solids	for Dry Wt	Results 16.8	Units %	<i>RL</i> 0.010	÷	AL CAS	<i>Bo</i> 02	ottle
		Sample P	reparation	1				<b>373800002</b> 0
1532194 DAF I	float		· · ·		R	eceived: 10/	12/2016	
Calculation		Prepared:	10/24/2016	14:38:11	Calculated	10/24/2016	14:38:11	CAL
As Received	I to Dry Weight Basis	Calculated			·			
EPA 3510C		Prepared: 686806	10/13/2016	14:45:00	Analyzed 687197	10/17/2016	12:00:00	KRR
TCLP Liquic	-Liquid Extract	1/100	ml				06	
EPA 1311		Prepared: 686806	10/13/2016	14:45:00	Analyzed 686806	10/13/2016	14:45:00	TDL
N TCLP Extrac	tion Non-Volatile	SOLID EXT	Г 2				02	
EPA 1311ZHE		Prepared: 687230	10/17/2016	14:45:00	Analyzed 687230	10/17/2016	14:45:00	TDL
N TCLP Extrac	tion ZHE Volatiles	100.0% SO	L <b>ID</b>		·		02	
EPA 200.2 2.8		Prepared: 686734	10/13/2016	11:30:00	Analyzed 686734	10/13/2016	11:30:00	TES
N Solid Metals	Digestion	50/5.08	grams				01	
EPA 3005A		Prepared: 686806	10/13/2016	14:45:00	Analyzed 687060	10/14/2016	15:31:00	ALH
N Metals Diges	stion TCLP Extract	50/10	ml				05	
EPA 3550B		Prepared: 687178	10/17/2016	10:30:00	Analyzed 687178	10/17/2016	10:30:00	CRG
N Sonic Extrac	tion	5/30	grams				01	
EPA 7470A		Prepared: 686806	10/13/2016	14:45:00	Analyzed 686953	10/14/2016	10:45:00	ALB
N Metals Diges	stion TCLP 7470	50/2.5	mt				05	

Corporate Shipping: 2600 Dudley Rd. Kilgore, TX 75662

Oklahoma Region: 1824 Atchison Suite F Norman OK 73069



### Ana-Lab Corp. P.O. Box 9000 Kilgore, TX 75663

Phone 903/984-0551 FAX 903/984-5914 e-Mail corp@ana-lab.com

Employee Owned Integrity

Phone 903/984-055

Caring Continual Improvement Printed: 10/24/2016

Page 5 of 6

#### 1532194 DAF Float

THE COMPLETE SERVICE LAB

Corp.

Received: 10/12/2016

EPA	7471A	Prepared: 686873	10/14/2016	08:30:00	Analyzed 68	36873	10/14/2016	08:30:00	ALB
N	Solid Metals Digestion Hg	50/0.5127	grams					01	
EPA	8260B	Prepared: 687230	10/17/2016	14:45:00	Analyzed 68	7528	10/18/2016	15:57:00	JRH
N EPA	MS TCLP Volatile Analysis 8260B	Entered Prepared: 688007	10/20/2016	15:52:00	Analyzed 68	8007	10/20/2016	20 15:52:00	JRH
N	Volatiles as Totals	Entered	-		(			01	
EPA i	8270C	Prepared: 687178	10/17/2016	10:30:00	Analyzed 68	8389	10/24/2016	12:43:00	SLC
N EPA a	Semivolatile Exp 8270C	Entered Prepared: 687197	10/17/2016	12:00:00	Analyzed 68	8360	10/19/2016	14 <i>16:36:00</i>	SLC
N	MS TCLP Semivolatile Analysis	Entered						17	
EPA S	9010 C MOD	Prepared: 688011	10/21/2016	09:40:00	Analyzed 68	8011	10/21/2016	09:40:00	GWA
	Reactivity Distillation	10/0.1195	grams					01	
SM 25	540 G-1997	Prepared: 686565	10/13/2016	10:00:00	Analyzed 68	6565	10/13/2016	10:00:00	JWK
N	Total Solids Start Code	Started							

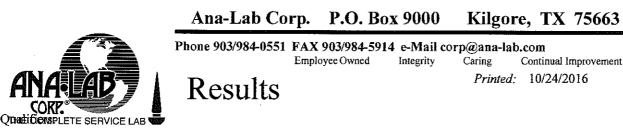
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Oklahoma Region: 1824 Atchison Suite F Norman OK 73069



Page 6 of 6

(- Sample from Bulk Container P - Spike recovery outside control limits due to matrix effects

We report results on an 'As Received' or wet basis unless marked 'Dry Weight' Unless otherwise noted, testing was performed at Ana-lab's corporate laboratory that holds the following Federal and State certificates Texas Department of Health Lead Firm Certificate 2110076, US Department of Agriculture Soil Import Permit S-37592, Texas Commisson on Environmental Quality Drinking Water Laboratory Certificate TX219, Texas Commission on Environmental Quality NELAP TI04704201, Oklahoma Department of Environmental Quality Drinking Water Certification Lab ID# D9913, EPA Lab Number TX00063, USEPA Approved Perchlorate Testing Lab, Oklahoma Department of Environmental Quality Laboratory Certificate8125, Arkansas Department of Environmental Quality Certification #03-070-0, Louisiana Department of Environmental Quality Laboratory Certification(NELAP, LELAP) #02008, Louisiana Department of Health and Hospitals Drinking Water (NELAP) # LA030020, US Department of Energy Approved, State of Kansas Department of Health and Environment Waste Water and Solid/Hazardous Waste Cert. E-10365. The Accredited column designates accreditation by N -- NELAC, or z -- not covered under NELAC scope of accreditation.

These analytical results relate to the sample tested. This report may NOT be reproduced EXCEPT in FULL without written approval of Ana-Lab Corp. Unless otherwise specified, these test results meet the requirements of NELAC

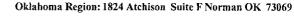
RL is the Reporting Limit (sample specific quantitation limit) and is at or above the Method Detection Limit (MDL). CAS is Chemical Abstract Service number. RL is our Reporting Limit, or Minimum Quantitation Level The RL takes into account the Instrument Detection Limit (IDL), Method Detection Limit (MDL), and Practical Quantitation Limit (PQL), and any dilutions and/or concentrations performed during sample preparation (EQL). Our analytical result must be above this RL before we report a value in the 'Results' column of our report (without a 'J' flag). Otherwise, we report ND (Not Detected above RL), because the result is "<" (less than) the number in the RL column. MAL is Minimum Analytical Level and is typically from regulatory agencies Unless we report a result in the result column, or interferences prevent it, we work to have our RL at or below the MAL

Dela

Paul Zhang, Ph.D., Quality Director



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Panhandis Oktahoma North TX Central TX 808.385.3558 dt5.282.8650 817.261.6404 512.621.0045 Rio Grand Valley Loufelana Guif Coast Alebama 958.831.6457 318.218.8300 281.333.9414 256.851.7564	alysis Requested	14 515/4 11/4	1\$1 PW 1910 1910	1414 1917 583	1919 19 8 I-, 10 5/	172 172 157 157 157 157	L X L X 		Semples Received on leaf. Dives Diverse Dispinent Data Dealer (1976 Star DUPS CHand delivered Difforme Difference (2000, 2000) Cooler/Samples Society Press Dives Dispinent Data Star DuPS CHand delivered Difforme Difforme Content Homments Ana Lab personnel collect samples as specified by Ana-Lab SOP #200232. Samples analysis will be provided according to Ana-Lab's Standard Terms and Conditions of Agreement, evaluable upon request and at workana-Any other terms proposed by Cliem
Enhande Panhande area assa Rio Grand Vali 958.831.6437	Billing Address (It different)	-	City Stans	Phone Fax	Affiliation	Dette 71:00 Mathfk Contrainers	10/11/14 11am 3/4.42 - 2 ~	Athlation Perma Name	Method of ShipmentBusEddFXLone StarUPS Tracking or Shipping Mumber
	Company names		CHY WYNNELLOOD BINE OK ZIP 73098	iS Fax	Bampler Signature	Leo Number Do Not Use Transformer	153219U	Upple 120 CSC 200	Samples Received on Ico? These DNo Method of Shipment Cooler/Sample Socure? Kee DNo Method of Shipping Mumb Requested TAT UbGoutins D3 day D2 Day D2 Hy. D24 Hu. D24 Hu. D24 Hu. D24 Hu. Day D3 day D2 Day D34 Haduested TAT UbGoutins D3 day D2 Day D34 Haduested TAT Ubgoutins D3 day D2 Day D34 Haduested TAT Ubgouting to Ana-Lab Standard Terms a are deemed material altoration after a altoration at the provided according to Ana-Lab Standard Terms a

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#### 758780 CoC Print Group 001 of 001

Airbill No. 23364749 SHIP TO: From: MONICA TORRES ANA-LAB CORPORATION 1824 ATCHISON DR. LOGIN ANA-LAB CORPORATION SUITE F NORMAN, OK 73069 2600 DUDLEY RD. KILGORE, TX 75663 4055902533 9039840551 LSO ECONOMY NEXT DAY 3:00 IN MOST AREAS LATER IN REMOTE AREAS PRINT DATE: 10/3/2016 QUICKCODE: LAB REF 1: 1D00V,0000 WEIGHT: 10.00LBS Fold on above line and place shipping label in pouch on package. Please be sure the barcodes and addresses can be read and scamed. Shipping Instructions ٨ 1. Fold this page along the horizontal line above. 2. Place this Airbill in the shipping label pouch on the package you are shipping. Please be sure the barcodes and addresses can be read and scanned. 3. To locate a drop box near you, click on Find A Drop Box from the home page main menu. 4. To schedule a pickup, click on Request Pickup. WARNING: Use only the printed original label for shipping. Using a photocopy of this label for shipping purposes is fraudulent and could result in additional billing charges, along with the cancellation of your Lone Star Overnight account number. This label is valid for use for 3 months from the date printed. Use of expired labels may result in delayed billing and / or additional research charges. LIMIT OF LIABILITY: We are not responsible for claims in excess of \$100 for any reason unless you: 1) declare a greater value (not to except S25,000); 2) pay an additional fee; 3) and document your actual loss in a timely manner. We will not pay any claim in excess of the actual loss. We are not liable for any special or consequential damages. Additional limitations of liability are contained in our current Service Guide. If you ask is to deliver a package without obtaining a delivery signature, you release us of all liability for claims resulting from such service. NO DELIVERY SIGNATURE WILL BE OBTAINED FOR 8:30 AM DELIVERIES OR RESIDENTIAL DELIVERIES.

A/10

EPA laboratory code: OK00921

Oklahoma DEQ Certification No. 8304 7211 8316

### **Certificate of Analysis**

SN4393 - Refinery Sludge

Date Received: 12/5/2012

Report Date: 12/18/2012

ERT Lab	Sample	Date	Analysis		Analyzed				
Log #	Identification	Sampled	Date	Time	By	Parameter	Results	Units	Method
WW1212060	T2007 K051	12/04/12	12/06/12	18:31	SM	TCLP Chromium	0.061	mg/L	200.7/60108
			12/06/12	18:31	SM	TCLP Lead	<0.001	mg/L	200.7/60108
			12/06/12	18:31	SM	TCLP Nickel	0.778	mg/L	200.7/60108
			12/12/12	11:21	PM	Benzene	9.71	mg/Kg	8021B
			12/12/12	11:21	PM	Toluene	58.2	mg/Kg	8021B
			12/12/12	11:21	PM	Ethylbenzene	50.0	mg/Kg	8021B
			12/12/12	11:21	PM	Xylene (m,p & o)	170	mg/Kg	8021B
			12/11/12	13:08	CDH	Acenaphthene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Anthracene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Benzo(a)anthracene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Benzo(a)pyrene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Chrysene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Fluorene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Naphthalene	95.8	mg/Kg	8070D
			12/11/12	13:08	CDH	Phenanthrene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Phenol	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Pyrene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	bis(2-Ethylhexyl)phthalate	<160	mg/Kg	8070D
			12/11/12	13:08	CDH	di-n-Butylphthalate	<80	mg/Kg	8070D
			12/14/12	15:45	AL	BTU	3680	BTU/Ib	BTU
			12/12/12	10:00	DS	Cyanide	<0.500	mg/Kg	9010C

Laboratory Authorized Signature

MDL = Method Detection Limit.

Client Name:

Project:

Wynnewood Refining Co LLC

T2007 K051

BDL = Analyte was analyzed for but not detected above MDL. PM, DS, CDH = Subcontracted to ODEQ Lab #7211

AL = Subcontracted to ODEQ Lab #8316

OUR LETTERS AND REPORTS APPLY ONLY TO THE SAMPLE TESTED AND/OR INSPECTED, AND ARE NOT INDICATIVE OF THE QUANITIES APPARENTLY IDENTICAL OR SIMILAR PRODUCTS. UNLESS NOTIFIED IN WRITING, SAMPLES ARE DISPOSED OF 15 DAYS AFTER THE SAMPLE IS REPORTED.

EPA laboratory code: OK00921

Oklahoma DEQ Certification No. 8304 7211 8316

## **Quality Control Report**

lient Name	Wynnewood Refinin	ig Co LLC			Date Re	ceived:	12/5/2012
					Repor	t Date:	12/18/2012
Date				Duplicate %		Spike	Standard %
Sampled	Parameter	Method	MDL	Difference	BLANK	Recovery	Recovery
12/4/2012	TCLP Chromium	200.7/60108	0.001 mg/L	2.25	BDL	83.7	96.5
	TCLP Lead	200.7/6010B	0.001 mg/L	3.17	BDL	89.9	101.2
	TCLP Nickel	200.7/6010B	0.001 mg/L	2.00	BDL	90.3	104.7
	Benzene	8021B	0.025 mg/Kg	NA	BDL	107	96
	Toluene	80218	0.025 mg/Kg	NA	BDL	106	98
	Ethylbenzene	8021B	0.025 mg/Kg	NA	BDL	101	99
	Xylene (m,p & o)	8021B	0.025 mg/Kg	NA	BDL	102	99
	Phenoi	8070D	0.4 mg/Kg	NA	BDL	D	65
	Acenaphthene	8070D	0.4 mg/Kg	NA	BDL	D	72
	Pyrene	8070D	0.4 mg/Kg	NA	BDL	D	107
	Cyanide	9010 <b>C</b>	0.050 mg/Kg	NA	BDL	103	108

Laboratory Authorized Signature

MDL = Method Detection Limit.

BDL = Analyte was analyzed for but not detected above MDL.

D = Surrogate of Matrix Spike Diluted Out OUR LETTERS AND REPORTS APPLY ONLY TO THE SAMPLE TESTED AND/OR INSPECTED, AND ARE NOT INDICATIVE OF THE QUANITIES OF APPARENTLY IDENTICAL OR SIMILAR PRODUCTS. UNLESS NOTIFIED IN WRITING, SAMPLES ARE DISPOSED OF 15 DAYS AFTER THE SAMPLE IS REPORTED.

Client Name: Wynnewood Refining Lab Log # Date Sample Lab Log # Sample Sample Sample Taken Taken Taken Taken Taken Taken Taken Taken Taken	Environmental Resource Technologies 13526 CR 3530/131 Arlington Ada, Oklahoma 74820 Phone (580) 332-8808 Fax (580) 421-9110	CHAIN OF CUSTODY Project Name:T2007 K051	TimeMatrixGCClient I.D.TempNo. ofAnalysisSampleSampleWater (w)ROSample LocationC, FContainerRequestedPresv.TakenSoil (s)AMC, FContainer(p)=blasticPresv.	X 12007 K051			Date/Time. 12/04/12 12prus Received By & c~ 2 v cline Date/Time.	2. Olio 12.5-12 11:00 Received at Lay By. Date Time. Date Time.	Report to: EHIG unecukencigy.com Sendinvoice to:	Address:	
	Environmen 13526 CR 36 Phone		Matrix G Water (w) R Soil (5) A Studge (5)) B	Studge (SI)				ao: H		Address.	

EPA laboratory code: OK00921

Oklahoma DEQ Certification No. 8304 7211 8316

### **Certificate of Analysis**

SN4393 - Refinery Sludge

Date Received: 12/5/2012

Report Date: 12/18/2012

ERT Lab	Sample	Date	Analysis		Analyzed				
Log #	Identification	Sampled	Date	Time	By	Parameter	Results	Units	Method
WW1212060	T2007 K051	12/04/12	12/06/12	18:31	SM	TCLP Chromium	0.061	mg/L	200.7/60108
			12/06/12	18:31	SM	TCLP Lead	<0.001	mg/L	200.7/60108
			12/06/12	18:31	SM	TCLP Nickel	0.778	mg/L	200.7/60108
			12/12/12	11:21	PM	Benzene	9.71	mg/Kg	8021B
			12/12/12	11:21	PM	Toluene	58.2	mg/Kg	8021B
			12/12/12	11:21	PM	Ethylbenzene	50.0	mg/Kg	8021B
			12/12/12	11:21	PM	Xylene (m,p & o)	170	mg/Kg	8021B
			12/11/12	13:08	CDH	Acenaphthene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Anthracene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Benzo(a)anthracene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Benzo(a)pyrene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Chrysene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Fluorene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Naphthalene	95.8	mg/Kg	8070D
			12/11/12	13:08	CDH	Phenanthrene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Phenol	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	Pyrene	<80	mg/Kg	8070D
			12/11/12	13:08	CDH	bis(2-Ethylhexyl)phthalate	<160	mg/Kg	8070D
			12/11/12	13:08	CDH	di-n-Butylphthalate	<80	mg/Kg	8070D
			12/14/12	15:45	AL	BTU	3680	BTU/Ib	BTU
			12/12/12	10:00	DS	Cyanide	<0.500	mg/Kg	9010C

Laboratory Authorized Signature

MDL = Method Detection Limit.

Client Name:

Project:

Wynnewood Refining Co LLC

T2007 K051

BDL = Analyte was analyzed for but not detected above MDL. PM, DS, CDH = Subcontracted to ODEQ Lab #7211

AL = Subcontracted to ODEQ Lab #8316

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EPA laboratory code: OK00921

Oklahoma DEQ Certification No. 8304 7211 8316

## **Quality Control Report**

lient Name	Wynnewood Refinin	ig Co LLC			Date Re	ceived:	12/5/2012
					Repor	t Date:	12/18/2012
Date				Duplicate %		Spike	Standard %
Sampled	Parameter	Method	MDL	Difference	BLANK	Recovery	Recovery
12/4/2012	TCLP Chromium	200.7/60108	0.001 mg/L	2.25	BDL	83.7	96.5
	TCLP Lead	200.7/6010B	0.001 mg/L	3.17	BDL	89.9	101.2
	TCLP Nickel	200.7/6010B	0.001 mg/L	2.00	BDL	90.3	104.7
	Benzene	8021B	0.025 mg/Kg	NA	BDL	107	96
	Toluene	80218	0.025 mg/Kg	NA	BDL	106	98
	Ethylbenzene	8021B	0.025 mg/Kg	NA	BDL	101	99
	Xylene (m,p & o)	8021B	0.025 mg/Kg	NA	BDL	102	99
	Phenoi	8070D	0.4 mg/Kg	NA	BDL	D	65
	Acenaphthene	8070D	0.4 mg/Kg	NA	BDL	D	72
	Pyrene	8070D	0.4 mg/Kg	NA	BDL	D	107
	Cyanide	9010 <b>C</b>	0.050 mg/Kg	NA	BDL	103	108

Laboratory Authorized Signature

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BDL = Analyte was analyzed for but not detected above MDL.

D = Surrogate of Matrix Spike Diluted Out OUR LETTERS AND REPORTS APPLY ONLY TO THE SAMPLE TESTED AND/OR INSPECTED, AND ARE NOT INDICATIVE OF THE QUANITIES OF APPARENTLY IDENTICAL OR SIMILAR PRODUCTS. UNLESS NOTIFIED IN WRITING, SAMPLES ARE DISPOSED OF 15 DAYS AFTER THE SAMPLE IS REPORTED.

Client Name: Wynnewood Refining Lab Log # Date Sample Lab Log # Sample Sample Sample Taken Taken Taken Taken Taken Taken Taken Taken Taken	Environmental Resource Technologies 13526 CR 3530/131 Arlington Ada, Oklahoma 74820 Phone (580) 332-8808 Fax (580) 421-9110	CHAIN OF CUSTODY Project Name:T2007 K051	TimeMatrixGCClient I.D.TempNo. ofAnalysisSampleSampleWater (w)ROSample LocationC, FContainerRequestedPresv.TakenSoil (s)AMC, FContainer(p)=blasticPresv.	X 12007 K051			Date/Time. 12/04/12 12prus Received By & c~ 2 v cline Date/Time.	2. Olio 12.5-12 11:00 Received at Lay By. Date Time. Date Time.	Report to: EHIG unecukencigy.com Sendinvoice to:	Address:	
	Environmen 13526 CR 36 Phone		Matrix G Water (w) R Soil (5) A Studge (5)) B	Studge (SI)				ao: H		Address.	

EPA laboratory code: OK00921

Oklahoma DEQ Certification No. 8304 7211

## **Certificate of Analysis**

Client Name: Wynnewood Refining Co LLC

Project: T2007 K051

Fiojeçi.	12007 6057						Rep	ort Date:	12/18/2012
ERT Lab	Sample	Date	Analysis		Analyzed	J			
Log #	Identification	Sampled	Date	Time	Ву	Parameter	Results	Units	Method
WW1212061	T2007 K051	12/04/12	12/12/12	10:57	PM	Benzene	10.4		8021B
			12/12/12	10:57	PM	Toluene	57.6		8021B
			12/12/12	10:57	PM	Ethylbenzene	46.5		8021B
			12/12/12	10:57	PM	Xylene (m,p & o)	168		8021B

Laboratory Authorized Signature

MDL = Method Detection Limit.

BDL = Analyte was analyzed for but not detected above MDL. PM = Subcontracted to ODEQ Lab #7211

Date Received:

12/5/2012

OUR LETTERS AND REPORTS APPLY ONLY TO THE SAMPLE TESTED AND/OR INSPECTED, AND ARE NOT INDICATIVE OF THE QUANITIES APPARENTLY IDENTICAL OR SIMILAR PRODUCTS. UNLESS NOTIFIED IN WRITING, SAMPLES ARE DISPOSED OF 15 DAYS AFTER THE SAMPLE IS REPORTED.

EPA laboratory code: OK00921

Oklahoma DEQ Certification No. 8304 7211

### **Quality Control Report**

Client Name	Wynnewood Refining	Co LLC			Date Re	ceived:	12/5/2012
					Repo.	rt Date:	12/18/2012
Date		<u> </u>	·····	Duplicate %		Spike	Standard %
Sampled	Parameter	Method	MDL	Difference	BLANK	Recovery	Recovery
12/4/2012	8enzene	8021B	0.025 mg/Kg	NA	BDL	107	96
	Toluene	8021B	0.025 mg/Kg	NA	BDL	106	98
	Ethylbenzene	8021B	0.025 mg/Kg	NA	BDL	101	99
	Xylene (m,p & o)	8021B	0.025 mg/Kg	NA	BDL	102	99

Laboratory Authorized Signature

MDL = Method Detection Limit. D = Surrogate or Matrix Spike Diluted Out

BDL = Analyte was analyzed for but not detected above MDL.

OUR LETTERS AND REPORTS APPLY ONLY TO THE SAMPLE TESTED AND/OR INSPECTED, AND ARE NOT INDICATIVE OF THE QUANITIES OF APPARENTLY IDENTICAL OR SIMILAR PRODUCTS. UNLESS NOTIFIED IN WRITING, SAMPLES ARE DISPOSED OF 15 DAYS AFTER THE SAMPLE IS REPORTED.

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