



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5 CHICAGO REGIONAL LABORATORY
536 SOUTH CLARK STREET
CHICAGO, ILLINOIS 60605

Date: 8/21/2014
Subject: Review of Region 5 Data for SE Chicago Petcoke
To: Air Division, US EPA Region 5
77 West Jackson Boulevard
Chicago, IL 60605
From: Troy Stroock, Analyst
US EPA Region 5 Chicago Regional Laboratory

The data transmitted under this cover memo successfully passed CRL's data review procedures as documented in the current Quality Management Plan and applicable Standard Operating Procedures. In accordance with EPA's *Guidance on Environmental Data Verification and Data Validation* (Document EPA QA/G-8), CRL verified and validated the data but does not perform data quality assessment based on project plans.

This report was reviewed and the information provided herein accurately represents the analysis performed.

X _____

Please contact the analyst with any technical report issues, Amanda Wroble at (312)-353-0375 for sample project concerns, and Sylvia Griffin at (312)-353-9073 with data transmittal questions. Thank you.

Attached are Results for: SE Chicago Petcoke

Data Management Coordinator and Date Transmitted

Analyses included in this report:

SVOA PAHs in solids by press. fluid extr. (SIM)



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Chicago IL, 60605

Project: SE Chicago Petcoke
Project Number: [none]
Project Manager: Motria Caudill

Reported:
Aug-21-14 14:51

ANALYSIS CASE NARRATIVE

Analyst Phone number: 312.353.8362

General Information

2 filter samples were received by the Chicago Regional Laboratory (CRL) on 7/1/2014, and two new filters were purchased by the CRL on 7/8/2014, all for extraction and analysis by Standard Operating Procedure (SOP) MS026 rev. 8 (based on SW-846 Method 8270D). These samples were analyzed for polycyclic aromatic hydrocarbons (PAHs), including the target analytes listed in Table A.3.7 in the appendix of the SOP, along with perylene, an additional PAH of interest to the data user. All holding times were met for sample preparation and analysis.

Pen and ink change MS026 R8.0-PI02 applies to this data set, which addresses analysis and reporting of PAHs in the filter material. That pen and ink change states that PAHs will be reported in units of $\mu\text{g}/\text{kg}$, but the client has since requested results to be reported in the same units (ng/wipe) as used for wipe samples for this project. Clean matrix QC samples (method blank, RL verification, and blank spike QC samples) were prepared with Ottawa sand and hydromatrix, as described in SOP MS026 Rev 8 for solids, not with clean wipes as described in Pen and ink change MS026 R8.0-PI01 for preparation and analysis of dust wipe samples.

The filter samples received for work order 1407001 were cut approximately in half with metal shears before sample preparation in order to reserve the other portion for possible split analysis off-site. For sample 1407001-15, the filter dimensions tested were approximately 3.75"x6", and the mass of the filamentous material pulled off the metal backing that was tested was measured at 1.415 g. For sample 1407001-16, the filter dimensions tested were approximately 4.75"x 4.5", and the mass of the filamentous material pulled off the metal backing that was tested was measured at 1.49 g.

Filter sample 1407004-01 and associated matrix spike QC samples were cut from a single filter purchased new from a store, as was filter sample 1407004-02 and associated matrix spike QC samples. Subsamples were cut in dimensions of approximately 3.5" x 7", and masses of the filamentous material pulled from the metal backing for testing ranged from 0.825 g to 1.119 g.

Extracts were filtered through 0.45 μm particle filters twice during concentration of extracts to remove particulates observed in the extracts after extraction or observed to form during nitrogen blow-down of extracts.

The mass spectrometer was operated in selected ion monitoring (SIM) mode, which is more sensitive than full scan. Mass spectra acquired in SIM mode are limited to a couple of ions per analyte, resulting in poorer confirmation of peak identification by mass spectrum in unknown samples, as mass spectra acquired in SIM cannot be searched against a mass spectral library.



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Sample Analysis and Results

Many of the target PAHs were measured above the RL in the filter samples in work order 1407001, and a few PAHs were also observed in the filter samples in work order 1407004. Perylene was qualified as potentially high bias (“K”) in sample ID 1407001-16 due to poor peak shape and a qualifier to quantification ion ratio that was much higher than in the calibration standard. Full scan analysis of the extract showed that a large hydrocarbon peak elutes at the expected perylene retention time, so this target analyte measurement is suspected to be high bias or false positive.

The data reported herein meet the Data Quality Objectives referenced in the SE Chicago Petcoke QAPP dated 12/16/2013 and addendum named SE Chicago Petcoke QAPP HVAC filter update 063014.docx, with the exception that PAH concentrations were reported for sample ID 1407001-15 from a 10-fold dilution, resulting in reporting of some results as non-detects at 10-fold higher reporting limits (RLs) than listed in the QAPP.

Quality Controls

Initial Calibration:

Most of the target analytes were contained in the primary and second source standards used for calibration of the instrument and initial calibration verification, respectively. However, no certified second source standard was analyzed or evaluated for 1-methylnaphthalene, benzo(e)pyrene, or perylene. Response factors for these analytes were similar to structural isomers that met criteria for initial calibration verification.

Continuing Calibration Verification (CCV):

One of the CCVs bracketing analysis of the method blank, RL verification, and blank spike QC samples associated with the extraction batch slightly exceeded the % difference criteria of $\leq \pm 25\%$ relative to the expected concentration for pyrene. The concentration or RL of this target analyte in these QC samples is qualified as estimated (“J”) for measurements above and below the RL, respectively. One or both CCVs bracketing analysis of the 10-fold dilution of sample ID 1407001-15 exceeded the % difference criteria of $\leq \pm 25\%$ relative to the expected concentration for nitrobenzene-d5, 2-methylnaphthalene, 1-methylnaphthalene, phenanthrene, and fluorene. Of these target analytes, only phenanthrene was measured in this sample above the RL. The concentration of phenanthrene is qualified as estimated in this field sample, and the RLs for the other non-detected target analytes that did not meet the CCV criteria are qualified as estimated (“J”).

Internal Standards:

Internal standard responses in all reported field samples and associated batch QC samples were within SOP criteria of 50-200% of the associated peak responses in the continuing calibration verification (CCV) injections. However,



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responses of some internal standards in one of the two CCV injections bracketing analysis of all of the field samples and matrix spike QC samples reported for this work order except for the 10-fold dilution of sample 1407001-15 were slightly low relative to their responses in the midpoint initial calibration standard. Internal standard responses in one of the CCVs bracketing reported analytical runs of sample ID 1407004-01, 1407004-02, and all matrix spike QC samples ranged between 37.2 and 50.4% of the response in the midpoint initial calibration standard, and internal standard responses in one of the CCVs bracketing the reported analytical run of the 1 mL extract volume of sample ID 1407001-16 ranged between 47.8 and 72.3% of the midpoint initial calibration standard. One of the two CCVs bracketing analysis of these samples met the internal standard performance criteria, and the CCVs with low internal standard responses met the % difference criteria for all target analytes and surrogates, so recalibration of the instrument was deemed unnecessary. No field sample data is qualified as a result of low response observed in 1 of the 2 CCVs bracketing the samples.

Surrogates Spike Recovery:

The non-ionizable surrogates listed in section 8.2 of the SOP (nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14) were used for evaluation of successful preparation and analysis of each field sample and QC sample listed in this report. Nitrobenzene-d5 was not recovered in filter sample 1407001-15, and nitrobenzene-d5 recovery was low in one of the matrix spike QC samples (in B407046-MS2, nitrobenzene recovery was 47.5%,; lower control limit is 50%), but the other two surrogates were recovered acceptably in both of these samples, so no data is qualified due to surrogate recovery.

Target Compounds Spike Recovery:

Recovery of 1-methylnaphthalene is evaluated against control limits provided in the SOP for 2-methylnaphthalene, and recovery of perylene and benzo(e)pyrene is evaluated against control limits for benzo(a)pyrene. Once sufficient data are acquired, historical spike recovery statistics for these analytes will be used to generate more appropriate recovery criteria. An RL study has been completed for benzo(e)pyrene and 1-methylnaphthalene and is summarized in the SOP in Attachment 3 table A.3.7. A similar RL study has not been completed for perylene, so the concentration of this analyte is qualified as estimated ("J") in all field samples until it is included in the SOP.

One of the blank spike (LCS) QC samples, B407047-BSD1, was lost during pressurized fluid extraction due to an equipment malfunction. The other LCS QC sample, B407047-BS1, was analyzed successfully, so no further actions were taken due to loss of one of the LCS QC samples in the batch.

4 replicate matrix spike QC samples were prepared and analyzed for each of field samples 1407004-01 and -02 and are reported as B407047-MS1 through -MS8. In spite of background observed in the unspiked samples for a few target analytes, all target analytes were recovered acceptably in all matrix spikes except for phenanthrene in B407047-MS3 (63.4% recovery; lower control limit of 68.1%), fluorene in B407047-MS4 (45.4% recovery; lower control limit 57.1%), and a recovery of a number of target analytes was low in B407047-MS8. Part of the extract of



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B407047-MS8 was lost during the second particle filtration (approximately 2 mL of 10 mL), resulting in slightly low bias recovery for most of the target analytes. The target analytes in the other 3 replicate matrix spike QC samples were recovered acceptably, so no data is qualified as a result of matrix spike recovery.

RL Verification:

For this analysis, RL verification QC samples were prepared at two (nominal) spike levels: 20 ng/wipe (B407047-MRL1) and 100 ng/wipe (B407047-MRL2), and the same acceptance criteria are used to evaluate these QC samples as used for the blank spikes. All target analytes were recovered at their respective RLs within the acceptance criteria. Results are only reported for the individual RL verification QC sample corresponding to that analyte's RL.

All other quality controls not mentioned here meet the criteria in the SOP.

Signature _____, Date _____



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ANALYTICAL REPORT FOR SAMPLES

Sample ID	Laboratory ID	Matrix	Date Sampled	Date Received
15	1407001-15	Wipe	Jul-01-14 14:16	Jul-01-14 15:25
16	1407001-16	Wipe	Jul-01-14 14:26	Jul-01-14 15:25



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Semivolatiles by GC/MS, EPA 8270D (modified) US EPA Region 5 Chicago Regional Laboratory

15 (1407001-15) Wipe Sampled: Jul-01-14 14:16 Received: Jul-01-14 15:25

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Naphthalene	U			1000	ng/Wipe	10	B407047	Jul-10-14	Jul-31-14
2-Methylnaphthalene	U	J		1000	"	"	"	"	"
1-Methylnaphthalene	U	J		1000	"	"	"	"	"
Acenaphthylene	U			200	"	"	"	"	"
Acenaphthene	U			1000	"	"	"	"	"
Fluorene	U	J		200	"	"	"	"	"
Phenanthrene	1660	J		1000	"	"	"	"	"
Anthracene	U			200	"	"	"	"	"
Fluoranthene	1750			1000	"	"	"	"	"
Pyrene	1290			1000	"	"	"	"	"
Benzo (a) anthracene	428			200	"	"	"	"	"
Chrysene	1360			200	"	"	"	"	"
Benzo(b)fluoranthene	2070			200	"	"	"	"	"
Benzo(k)fluoranthene	447			200	"	"	"	"	"
Benzo (e) pyrene	1810			200	"	"	"	"	"
Benzo(a)pyrene	621			200	"	"	"	"	"
Perylene	U	J		200	"	"	"	"	"
Indeno(1,2,3-cd)pyrene	993			200	"	"	"	"	"
Dibenz(a,h)anthracene	401			200	"	"	"	"	"
Benzo(g,h,i)perylene	1920			200	"	"	"	"	"

Surogate	Result		%REC	%REC Limits	Batch	Prepared	Analyzed
<i>Surrogate: Nitrobenzene-d5</i>	0.00	J	%	50-94.9	"	"	"
<i>Surrogate: 2-Fluorobiphenyl</i>	227		90.6%	48.1-108	"	"	"
<i>Surrogate: Terphenyl-d14</i>	223		89.4%	59.4-127	"	"	"

16 (1407001-16) Wipe Sampled: Jul-01-14 14:26 Received: Jul-01-14 15:25

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Naphthalene	U			100	ng/Wipe	1	B407047	Jul-10-14	Jul-25-14
2-Methylnaphthalene	U			100	"	"	"	"	"
1-Methylnaphthalene	U			100	"	"	"	"	"
Acenaphthylene	U			20.0	"	"	"	"	"
Acenaphthene	U			100	"	"	"	"	"



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Semivolatiles by GC/MS, EPA 8270D (modified) US EPA Region 5 Chicago Regional Laboratory

16 (1407001-16) Wipe Sampled: Jul-01-14 14:26 Received: Jul-01-14 15:25

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluorene	58.5			20.0	ng/Wipe	1	B407047	Jul-10-14	Jul-25-14
Phenanthrene	374			100	"	"	"	"	"
Anthracene	42.8			20.0	"	"	"	"	"
Fluoranthene	315			100	"	"	"	"	"
Pyrene	228			100	"	"	"	"	"
Benzo (a) anthracene	227			20.0	"	"	"	"	"
Chrysene	518			20.0	"	"	"	"	"
Benzo(b)fluoranthene	403			20.0	"	"	"	"	"
Benzo(k)fluoranthene	94.6			20.0	"	"	"	"	"
Benzo (e) pyrene	241			20.0	"	"	"	"	"
Benzo(a)pyrene	291			20.0	"	"	"	"	"
Perylene	162	J, K		20.0	"	"	"	"	"
Indeno(1,2,3-cd)pyrene	207			20.0	"	"	"	"	"
Dibenz(a,h)anthracene	79.3			20.0	"	"	"	"	"
Benzo(g,h,i)perylene	278			20.0	"	"	"	"	"

Surrogate	Result	%REC	%REC Limits	Batch	Prepared	Analyzed
<i>Surrogate: Nitrobenzene-d5</i>	189	75.5%	50-94.9	"	"	"
<i>Surrogate: 2-Fluorobiphenyl</i>	191	76.4%	48.1-108	"	"	"
<i>Surrogate: Terphenyl-d14</i>	213	85.4%	59.4-127	"	"	"



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Semivolatiles by GC/MS, EPA 8270D (modified) - Quality Control US EPA Region 5 Chicago Regional Laboratory

Batch B407047 - Solvent Extraction

Blank (B407047-BLK1)

Prepared: Jul-10-14 Analyzed: Jul-23-14

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Naphthalene	U			100	ng/Wipe						
2-Methylnaphthalene	U			100	"						
1-Methylnaphthalene	U			100	"						
Acenaphthylene	U			20.0	"						
Acenaphthene	U			100	"						
Fluorene	U			20.0	"						
Phenanthrene	U			100	"						
Anthracene	U			20.0	"						
Fluoranthene	U			100	"						
Pyrene	U	J		100	"						
Benzo (a) anthracene	U			20.0	"						
Chrysene	U			20.0	"						
Benzo(b)fluoranthene	U			20.0	"						
Benzo(k)fluoranthene	U			20.0	"						
Benzo (e) pyrene	U			20.0	"						
Benzo(a)pyrene	U			20.0	"						
Perylene	U			20.0	"						
Indeno(1,2,3-cd)pyrene	U			20.0	"						
Dibenz(a,h)anthracene	U			20.0	"						
Benzo(g,h,i)perylene	U			20.0	"						
Surrogate: Nitrobenzene-d5	170				"	250.0		68.1%	50-94.9		
Surrogate: 2-Fluorobiphenyl	167				"	250.0		66.6%	48.1-108		
Surrogate: Terphenyl-d14	214				"	250.0		85.4%	59.4-127		

Blank (B407047-BLK2)

Prepared: Jul-10-14 Analyzed: Jul-23-14

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Naphthalene	U			100	ng/Wipe						
2-Methylnaphthalene	U			100	"						
1-Methylnaphthalene	U			100	"						
Acenaphthylene	U			20.0	"						
Acenaphthene	U			100	"						



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Batch B407047 - Solvent Extraction

Blank (B407047-BLK2)

Prepared: Jul-10-14 Analyzed: Jul-23-14

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC Limits	RPD	RPD Limit
Fluorene	U			20.0	ng/Wipe					
Phenanthrene	U			100	"					
Anthracene	U			20.0	"					
Fluoranthene	U			100	"					
Pyrene	U	J		100	"					
Benzo (a) anthracene	U			20.0	"					
Chrysene	U			20.0	"					
Benzo(b)fluoranthene	U			20.0	"					
Benzo(k)fluoranthene	U			20.0	"					
Benzo (e) pyrene	U			20.0	"					
Benzo(a)pyrene	U			20.0	"					
Perylene	U			20.0	"					
Indeno(1,2,3-cd)pyrene	U			20.0	"					
Dibenz(a,h)anthracene	U			20.0	"					
Benzo(g,h,i)perylene	U			20.0	"					
Surrogate: Nitrobenzene-d5	178				"	250.0		71.2% 50-94.9		
Surrogate: 2-Fluorobiphenyl	171				"	250.0		68.4% 48.1-108		
Surrogate: Terphenyl-d14	215				"	250.0		86.0% 59.4-127		

LCS (B407047-BS1)

Prepared: Jul-10-14 Analyzed: Jul-23-14

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC Limits	RPD	RPD Limit
Naphthalene	123			100	ng/Wipe	250.0		49.3% 43.1-108		
2-Methylnaphthalene	139			100	"	250.0		55.6% 45.5-117		
1-Methylnaphthalene	135			100	"	250.0		53.9% 45.5-117		
Acenaphthylene	149			20.0	"	250.0		59.7% 52.7-117		
Acenaphthene	152			100	"	250.0		60.8% 51.7-116		
Fluorene	176			20.0	"	250.0		70.5% 57.9-120		
Phenanthrene	200			100	"	250.0		80.2% 68.1-114		
Anthracene	175			20.0	"	250.0		70.0% 69.7-116		
Fluoranthene	212			100	"	250.0		84.8% 70.8-122		
Pyrene	213	J		100	"	250.0		85.3% 71.8-117		
Benzo (a) anthracene	210			20.0	"	250.0		84.1% 67.6-115		



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Batch B407047 - Solvent Extraction

LCS (B407047-BS1)

Prepared: Jul-10-14 Analyzed: Jul-23-14

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC Limits	RPD	RPD Limit
Chrysene	213			20.0	ng/Wipe	250.0		85.1%	68.5-117	
Benzo(b)fluoranthene	220			20.0	"	250.0		88.0%	68.9-128	
Benzo(k)fluoranthene	217			20.0	"	250.0		86.9%	62.8-135	
Benzo (e) pyrene	212			20.0	"	250.0		85.0%	68.9-133	
Benzo(a)pyrene	179			20.0	"	250.0		71.8%	68.9-133	
Perylene	190			20.0	"	250.0		76.2%	68.9-133	
Indeno(1,2,3-cd)pyrene	208			20.0	"	250.0		83.3%	70-129	
Dibenz(a,h)anthracene	211			20.0	"	250.0		84.5%	69.1-131	
Benzo(g,h,i)perylene	192			20.0	"	250.0		77.0%	53.9-139	
<i>Surrogate: Nitrobenzene-d5</i>	<i>129</i>				"	<i>250.0</i>		<i>51.7%</i>	<i>50-94.9</i>	
<i>Surrogate: 2-Fluorobiphenyl</i>	<i>144</i>				"	<i>250.0</i>		<i>57.6%</i>	<i>48.1-108</i>	
<i>Surrogate: Terphenyl-d14</i>	<i>210</i>				"	<i>250.0</i>		<i>84.2%</i>	<i>59.4-127</i>	

MRL Check (B407047-MRL1)

Prepared: Jul-10-14 Analyzed: Jul-23-14

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC Limits	RPD	RPD Limit
Acenaphthylene	15.0			20.0	ng/Wipe	20.00		75.1%	52.7-117	
Fluorene	18.4			20.0	"	20.00		91.9%	57.9-120	
Anthracene	17.2			20.0	"	20.00		86.0%	69.7-116	
Benzo (a) anthracene	18.9			20.0	"	20.00		94.6%	67.6-115	
Chrysene	17.7			20.0	"	20.00		88.5%	68.5-117	
Benzo(b)fluoranthene	18.8			20.0	"	20.00		94.2%	68.9-128	
Benzo(k)fluoranthene	18.3			20.0	"	20.00		91.4%	62.8-135	
Benzo (e) pyrene	18.2			20.0	"	20.00		91.0%	68.9-133	
Benzo(a)pyrene	18.3			20.0	"	20.00		91.4%	68.9-133	
Perylene	16.6			20.0	"	20.00		82.9%	68.9-133	
Indeno(1,2,3-cd)pyrene	16.5			20.0	"	20.00		82.4%	70-129	
Dibenz(a,h)anthracene	16.9			20.0	"	20.00		84.6%	69.1-131	
Benzo(g,h,i)perylene	16.4			20.0	"	20.00		82.0%	53.9-139	
<i>Surrogate: Nitrobenzene-d5</i>	<i>180</i>				"	<i>250.0</i>		<i>72.1%</i>	<i>50-94.9</i>	
<i>Surrogate: 2-Fluorobiphenyl</i>	<i>176</i>				"	<i>250.0</i>		<i>70.2%</i>	<i>48.1-108</i>	
<i>Surrogate: Terphenyl-d14</i>	<i>214</i>				"	<i>250.0</i>		<i>85.6%</i>	<i>59.4-127</i>	



Environmental Protection Agency Region 5
Chicago Regional Laboratory

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 Phone:(312)353-8370 Fax:(312)886-2591

Air Division, US EPA Region 5
 77 West Jackson Boulevard
 Chicago IL, 60605

Project: SE Chicago Petcoke
 Project Number: [none]
 Project Manager: Motria Caudill

Reported:
 Aug-21-14 14:51

Semivolatiles by GC/MS, EPA 8270D (modified) - Quality Control
US EPA Region 5 Chicago Regional Laboratory

Batch B407047 - Solvent Extraction

MRL Check (B407047-MRL2)

Prepared: Jul-10-14 Analyzed: Jul-23-14

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC Limits	RPD	RPD Limit
Naphthalene	68.7			100	ng/Wipe	100.0		68.7%	43.1-108	
2-Methylnaphthalene	73.7			100	"	100.0		73.7%	45.5-117	
1-Methylnaphthalene	67.5			100	"	100.0		67.5%	45.5-117	
Acenaphthene	71.1			100	"	100.0		71.1%	51.7-116	
Phenanthrene	87.7			100	"	100.0		87.7%	68.1-114	
Fluoranthene	88.6			100	"	100.0		88.6%	70.8-122	
Pyrene	88.4	J		100	"	100.0		88.4%	71.8-117	
<i>Surrogate: Nitrobenzene-d5</i>	<i>171</i>				"	<i>250.0</i>		<i>68.5%</i>	<i>50-94.9</i>	
<i>Surrogate: 2-Fluorobiphenyl</i>	<i>168</i>				"	<i>250.0</i>		<i>67.3%</i>	<i>48.1-108</i>	
<i>Surrogate: Terphenyl-d14</i>	<i>213</i>				"	<i>250.0</i>		<i>85.2%</i>	<i>59.4-127</i>	



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Notes and Definitions

- K The identification of the analyte is acceptable; the reported value may be biased high. The actual value is expected to be less than the reported value.
- J The identification of the analyte is acceptable; the reported value is an estimate.
- U Not Detected
- NR Not Reported