

**DOEE SEALCOAT QC PROTOCOL
PAHS IN PARKING LOT SEALCOAT CERTIFICATION**

APPENDIX A

EXAMPLE DATA PACKAGE

ANALYTICAL & CASE NARRATIVE SUMMARY

The format used by laboratories to report results will vary from one lab to another. The laboratory report used in this example will look different than results performed by another lab, but all laboratories should report the same information.

The lab report should include a summary page detailing the test performed, project ID and sample ID numbers and may include a Table of Contents.

The Project or Case Narrative page is signed by the laboratory and identifies any flags/qualifiers or other outliers observed that exceed the quality control limits.

ANALYTICAL SUMMARY

REPORT DATE: 3/9/2021				WORK ORDER NUMBER: 21B0465	
				PROJECT NUMBER: 2102100	
PROJECT LOCATION:	PAHs in Parking Lot Sealcoats				
FIELD SAMPLE #	LAB ID:	MATRIX	SAMPLE DESCRIPTION	TEST	SUB LAB
SEALCOAT 1	21B0465-02	Sludge		SM 2540G SW-846 8270D-E	

CASE NARRATIVE SUMMARY

All reported results are within defined laboratory quality control objectives unless listed below or otherwise qualified in this report.

Per client request, QC limits have been changed for method 8270E. BS/BSD/MS/MSD recovery limits set to 60%-140%. Surrogate recovery limits set to 70%-130%.

For method 8270E, only PAHs were requested and reported.

FLAG/QUALIFIER RESULTS

The laboratory describes any flags/qualifiers observed that exceed the quality control limits in more detail.

In this example, several PAH compounds and surrogate compounds spiked in the samples that exceed the limits for SEALCOAT 1 are listed.

Qualifications: SW-846 8270D-E

MS-07A

Matrix spike and spike duplicate recovery is outside of control limits. Analysis is in control based on laboratory fortified blank recovery. Possibility of matrix effects that lead to low bias or non-homogeneous sample aliquot cannot be eliminated.

Analyte & Samples(s) Qualified:

Benzo(g,h,i)perylene

21B0465-02 [SEALCOAT 1], B276580-MS2, B276580-MSD2

Dibenz(a,h)anthracene

21B0465-02 [SEALCOAT 1], B276580-MS2, B276580-MSD2

MS-22

Either matrix spike or MS duplicate is outside of control limits, but the other is within limits. RPD between the two MS/MSD results is within method specified criteria.

Analyte & Samples(s) Qualified:

Benzo(k)fluoranthene

B276580-MS2

RL-12

Elevated reporting limit due to matrix interference.

Analyte & Samples(s) Qualified:

21B0465-02 [SEALCOAT 1]

S-26

Surrogate outside of control limits.

Analyte & Samples(s) Qualified:

2-Fluorobiphenyl

21B0465-01 [SEALCOAT 1] B276580-MS2, B276580-MSD2

Nitrobenzene-d5

21B0465-02 [SEALCOAT 1] B276580-BS1, B276580-BSD1, B276580-MS2, B276580-MSD2

FLAG/QUALIFIER RESULTS - *CONTINUED*

FLAG/QUALIFIER SUMMARY

*	QC result is outside of established limits.
†	Wide recovery limits established for difficult compound.
‡	Wide RPD limits established for difficult compound.
#	Data exceeded client recommended or regulatory level
ND	Not Detected
RL	Reporting Limit is at the level of quantitation (LOQ)
DL	Detection Limit is the lower limit of detection determined by the MDL study
MCL	Maximum Contaminant Level
	Percent recoveries and relative percent differences (RPDs) are determined by the software using values in the calculation which have not been rounded.
	No results have been blank subtracted unless specified in the case narrative section.
MS-07A	Matrix spike and spike duplicate recovery is outside of control limits. Analysis is in control based on laboratory fortified blank recovery. Possibility of matrix effects that lead to low bias or non-homogeneous sample aliquot cannot be eliminated.
MS-22	Either matrix spike or MS duplicate is outside of control limits, but the other is within limits. RPD between the two MS/MSD results is within method specified criteria.
RL-12	Elevated reporting limit due to matrix interference.
S-26	Surrogate outside of control limits.

SAMPLE EXTRACTION METHOD USED

The sample extraction method, or prep method, should also be reported in addition to the analysis method. In this example, the laboratory used method 3546 and reported the weight of sample (2 grams) used for extraction.

A variety of different extraction methods exist for 8270D analysis, all of which use methylene chloride solvent to extract a small aliquot of sample (1 to 5 grams is recommended) using different technologies. The most common methods are listed below. These methods perform well and exhibit low detection limits.

<u>EPA SW-846</u>	<u>Description</u>
Method 3546:	Microwave oven extraction
Method 3540C:	Soxhlet extraction
Method 3541:	Auto soxhlet extraction
Method 3550C:	Ultrasonic extraction

EPA Method 3580A by Waste Dilution should be avoided. This method has high detection limits & other limitations.

Sample Extraction Data

Prep Method: SW-846 3546 Analytical Method: SW-846 8270D-E

Lab Number [Field ID]	Batch	Initial [g]	Final [mL]	Date
21B0465-02 [SEALCOAT-1]	B276580	2.00	1.00	02/17/21

SAMPLE RESULTS

PAHs in this sample are all Non Detect. All compounds were below the 9.4 ppm Reporting Limit. This sealcoat would qualify for DOE's Gold certification level (Total PAHs <1,000 ppm).

Surrogate compounds spiked in the sample are within DOE's 70%-130% Recovery Limits.

The elevated reporting limit due to matrix interference (Flag RL-12) is normal and not problematic. In this case, all RLs are less than 30 ppm (or ½ the Gold's Project Quantitation Limit of 500 ppm).

Project Location: PAHs in Parking Lot Sealcoats

Work Order: 21B0465

Date Received: 2/11/2021

Field Sample #: SEALCOAT 1

Sampled: 2/10/2021 15:00

Sample ID: 21B0465-02

Sample Matrix: Sludge

Sample Flags: RL-12

Semivolatile Organic Compounds by GC/MS

Analyte	Results	RL	DL	Units	Dilution	Flag/Qual	Method	Date Prepared	Date/Time Analyzed	Analyst
Acenaphthene	ND	9.4	5.0	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Acenaphthylene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Anthracene	ND	9.4	5.0	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Benzo(a)anthracene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Benzo(a)pyrene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Benzo(b)fluoranthene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Benzo(g,h,i)perylene	ND	9.4	5.5	mg/Kg dry	2	MS-07A	SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Benzo(k)fluoranthene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Chrysene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Dibenz(a,h)anthracene	ND	9.4	5.5	mg/Kg dry	2	MS-07A	SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Fluoranthene	ND	9.4	5.5	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Fluorene	ND	9.4	6.7	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Indeno(1,2,3-cd)pyrene	ND	9.4	6.7	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
2-Methylnaphthalene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Naphthalene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Phenanthrene	ND	9.4	5.0	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Pyrene	ND	9.4	6.1	mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR
Summation of 17 PAHs	0.0			mg/Kg dry	2		SW-846 8270D-E	2/17/21	2/18/21 14:27	IMR

Surrogates	% Recovery	Recovery Limits	Flag/Qual
Nitrobenzene-d5	71.0	70-130	2/18/21 14:27
2-Fluorobiphenyl	74.0	70-130	2/18/21 14:27
p-Terphenyl-d14	91.2	70-130	2/18/21 14:27

PERCENT SOLIDS ANALYSIS

%Solids analysis is performed to convert wet weight sample concentrations to dry weight. The water content in sealcoat samples can settle to the top over time. Instruct the laboratory to shake or mix sample prior to use.

This laboratory performed a %Solids duplicate . The Quality Control table compares the duplicate result to the original sample result (or source result). Relative Percent Difference is within DOE's RPD Limit $\leq 10\%$.

Project Location: PAHs in Parking Lot Sealcoats Work Order: 21B0465
 Date Received: 2/11/2021
 Field Sample #: SEALCOAT 1 Sampled: 2/10/2021 15:00
 Sample ID: 21B0465-02
 Sample Matrix: Sludge

Conventional Chemistry Parameters by EPA/APHA/SW-846 Methods (Total)

Analyte	Results	RL	Units	Dilution	Flag/Qual	Method	Date Prepared	Date/Time Analyzed	Analyst
% Solids	54.1		% Wt	1		SM 2540G	2/18/21	2/18/21 22:39	AVF

QUALITY CONTROL

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
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Batch B276684 - % Solids

Duplicate (B276684-DUP2)	Source: 21B0465-02	Prepared & Analyzed: 02/18/21			
% Solids	54.1	% Wt	54.1	0.0686	10

MATRIX SPIKE RESULTS

Three PAH compounds are outside DOE's 60%-140% Recovery Limit. However, %Recovery for Total PAH is 64% (not shown) and within DOE's Limit. The low recovery is normal and is caused by matrix interference. The spiked PAH compounds would pass if compared to the lab's standard QC limits of 40%-140%.

Two surrogate compounds are outside DOE's 70%-130% Recovery Limits. The low recovery is normal and is caused by matrix interference. The surrogates would pass if compared to the lab's standard QC limits of 30%-130%.

QUALITY CONTROL

Semivolatile Organic Compounds by GC/MS - Quality Control

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B276580 - SW-846 3546										
Matrix Spike (B276580-MS2)	Source: 21B0465-02			Prepared: 02/17/21 Analyzed: 02/18/21						
Acenaphthene	29.6	9.4	mg/Kg dry	46.2	ND	64.0	60-140			
Acenaphthylene	31.1	9.4	mg/Kg dry	46.2	ND	67.2	60-140			
Anthracene	32.3	9.4	mg/Kg dry	46.2	ND	70.0	60-140			
Benzo(a)anthracene	30.3	9.4	mg/Kg dry	46.2	ND	65.6	60-140			
Benzo(a)pyrene	28.7	9.4	mg/Kg dry	46.2	ND	62.2	60-140			
Benzo(b)fluoranthene	29.8	9.4	mg/Kg dry	46.2	ND	64.4	60-140			
Benzo(g,h,i)perylene	23.8	9.4	mg/Kg dry	46.2	ND	51.6	*	60-140		MS-07A
Benzo(k)fluoranthene	27.1	9.4	mg/Kg dry	46.2	ND	58.6	*	60-140		MS-22
Chrysene	30.5	9.4	mg/Kg dry	46.2	ND	66.0	60-140			
Dibenz(a,h)anthracene	24.7	9.4	mg/Kg dry	46.2	ND	53.4	*	60-140		MS-07A
Fluoranthene	31.4	9.4	mg/Kg dry	46.2	ND	67.9	60-140			
Fluorene	31.0	9.4	mg/Kg dry	46.2	ND	67.0	60-140			
Indeno(1,2,3-cd)pyrene	29.5	9.4	mg/Kg dry	46.2	ND	63.9	60-140			
2-Methylnaphthalene	32.2	9.4	mg/Kg dry	46.2	ND	69.6	60-140			
Naphthalene	29.5	9.4	mg/Kg dry	46.2	ND	63.8	60-140			
Phenanthrene	32.2	9.4	mg/Kg dry	46.2	ND	69.7	60-140			
Pyrene	32.5	9.4	mg/Kg dry	46.2	ND	70.2	60-140			
Summation of 17 PAHs	506		mg/Kg dry		0.00					
Surrogate: Nitrobenzene-d5	59.0		mg/Kg dry	92.4		63.9	*	70-130		S-26
Surrogate: 2-Fluorobiphenyl	62.1		mg/Kg dry	92.4		67.2	*	70-130		S-26
Surrogate: p-Terphenyl-d14	69.4		mg/Kg dry	92.4		75.1		70-130		

MATRIX SPIKE DUPLICATE RESULTS

Two spiked PAH compounds are outside DOE's 60%-140% Recovery Limit. However, %Recovery for Total PAH is 66% (not shown) and within DOE's Limit. The low recovery is normal and is caused by matrix interference. The spiked PAH compounds would pass if compared to the lab's standard QC limits of 40%-140%.

Two surrogate compounds are outside DOE's 70%-130% Recovery Limits. The low recovery is normal and is caused by matrix interference. These surrogates would pass if compared to the lab's standard QC limits of 30%-130%.

Relative Percent Difference comparing the MS and MSD results for Total PAHs (506 ppm and 525 ppm) is 3.7%. This value is within DOE's RPD Limit \leq 30%.

QUALITY CONTROL

Semivolatile Organic Compounds by GC/MS - Quality Control

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B276580 - SW-846 3546										
Matrix Spike Dup (B276580-MSD2)	Source: 21B0465-02		Prepared: 02/17/21 Analyzed: 02/18/21							
Acenaphthene	30.5	9.5	mg/Kg dry	46.7	ND	65.3	60-140	2.92	30	
Acenaphthylene	31.2	9.5	mg/Kg dry	46.7	ND	66.8	60-140	0.468	30	
Anthracene	32.9	9.5	mg/Kg dry	46.7	ND	70.4	60-140	1.57	30	
Benzo(a)anthracene	32.4	9.5	mg/Kg dry	46.7	ND	69.4	60-140	6.52	30	
Benzo(a)pyrene	30.8	9.5	mg/Kg dry	46.7	ND	66.1	60-140	7.12	30	
Benzo(b)fluoranthene	31.7	9.5	mg/Kg dry	46.7	ND	68.0	60-140	6.44	30	
Benzo(g,h,i)perylene	23.4	9.5	mg/Kg dry	46.7	ND	50.1	* 60-140	1.98	30	MS-07A
Benzo(k)fluoranthene	30.8	9.5	mg/Kg dry	46.7	ND	66.0	60-140	12.9	30	
Chrysene	31.5	9.5	mg/Kg dry	46.7	ND	67.4	60-140	3.16	30	
Dibenz(a,h)anthracene	25.7	9.5	mg/Kg dry	46.7	ND	55.1	* 60-140	4.17	30	MS-07A
Fluoranthene	31.4	9.5	mg/Kg dry	46.7	ND	67.4	60-140	0.177	30	
Fluorene	32.2	9.5	mg/Kg dry	46.7	ND	68.9	60-140	3.77	30	
Indeno(1,2,3-cd)pyrene	30.2	9.5	mg/Kg dry	46.7	ND	64.7	60-140	2.25	30	
2-Methylnaphthalene	33.9	9.5	mg/Kg dry	46.7	ND	72.6	60-140	5.22	30	
Naphthalene	30.3	9.5	mg/Kg dry	46.7	ND	64.9	60-140	2.68	30	
Phenanthrene	33.0	9.5	mg/Kg dry	46.7	ND	70.6	60-140	2.32	30	
Pyrene	33.2	9.5	mg/Kg dry	46.7	ND	71.1	60-140	2.19	30	
Summation of 17 PAHs	525		mg/Kg dry		0.00			3.66		
Surrogate: Nitrobenzene-d5	59.2		mg/Kg dry	93.3		63.4	* 70-130			S-26
Surrogate: 2-Fluorobiphenyl	64.7		mg/Kg dry	93.3		69.3	* 70-130			S-26
Surrogate: p-Terphenyl-d14	72.4		mg/Kg dry	93.3		77.6	70-130			

METHOD BLANK RESULTS

PAHs in the blank are all Non Detect. All compounds were below the 0.17 ppm Reporting Limit.

The surrogate compounds spiked in the solvent blank are within DOE's 70%-130% Recovery Limit.

QUALITY CONTROL

Semivolatile Organic Compounds by GC/MS - Quality Control

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
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Batch B276580 - SW-846 3546

Blank (B276580-BLK1)

Prepared: 02/17/21 Analyzed: 02/18/21

Acenaphthene	ND	0.17	mg/Kg wet							
Acenaphthylene	ND	0.17	mg/Kg wet							
Anthracene	ND	0.17	mg/Kg wet							
Benzo(a)anthracene	ND	0.17	mg/Kg wet							
Benzo(a)pyrene	ND	0.17	mg/Kg wet							
Benzo(b)fluoranthene	ND	0.17	mg/Kg wet							
Benzo(g,h,i)perylene	ND	0.17	mg/Kg wet							
Benzo(k)fluoranthene	ND	0.17	mg/Kg wet							
Chrysene	ND	0.17	mg/Kg wet							
Dibenz(a,h)anthracene	ND	0.17	mg/Kg wet							
Fluoranthene	ND	0.17	mg/Kg wet							
Fluorene	ND	0.17	mg/Kg wet							
Indeno(1,2,3-cd)pyrene	ND	0.17	mg/Kg wet							
2-Methylnaphthalene	ND	0.17	mg/Kg wet							
Naphthalene	ND	0.17	mg/Kg wet							
Phenanthrene	ND	0.17	mg/Kg wet							
Pyrene	ND	0.17	mg/Kg wet							
Summation of 17 PAHs	0.0		mg/Kg wet							
Surrogate: Nitrobenzene-d5	2.52		mg/Kg wet	3.31		76.1	70-130			
Surrogate: 2-Fluorobiphenyl	2.68		mg/Kg wet	3.31		80.9	70-130			
Surrogate: p-Terphenyl-d14	2.94		mg/Kg wet	3.31		88.7	70-130			

LCS RESULTS

All 17 spiked PAH compounds are within DOEE's 60%-140% Recovery Limit. The %Recovery for Total PAH is 73% (not shown) and is also within DOEE's Limit.

One surrogate compound is outside DOEE's 70%-130% Recovery Limit. The low recovery is normal and is caused by matrix interference. This surrogate would pass if compared to the lab's standard QC limits of 30%-130%.

QUALITY CONTROL

Semivolatile Organic Compounds by GC/MS - Quality Control

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B276580 - SW-846 3546										
LCS (B276580-BS1)				Prepared: 02/17/21 Analyzed: 02/18/21						
Acenaphthene	1.13	0.17	mg/Kg wet	1.66		68.3	60-140			
Acenaphthylene	1.17	0.17	mg/Kg wet	1.66		70.5	60-140			
Anthracene	1.29	0.17	mg/Kg wet	1.66		77.4	60-140			
Benzo(a)anthracene	1.26	0.17	mg/Kg wet	1.66		75.9	60-140			
Benzo(a)pyrene	1.18	0.17	mg/Kg wet	1.66		70.8	60-140			
Benzo(b)fluoranthene	1.22	0.17	mg/Kg wet	1.66		73.2	60-140			
Benzo(g,h,i)perylene	1.15	0.17	mg/Kg wet	1.66		69.2	60-140			
Benzo(k)fluoranthene	1.18	0.17	mg/Kg wet	1.66		71.0	60-140			
Chrysene	1.30	0.17	mg/Kg wet	1.66		78.6	60-140			
Dibenz(a,h)anthracene	1.21	0.17	mg/Kg wet	1.66		72.8	60-140			
Fluoranthene	1.27	0.17	mg/Kg wet	1.66		76.7	60-140			
Fluorene	1.17	0.17	mg/Kg wet	1.66		70.5	60-140			
Indeno(1,2,3-cd)pyrene	1.26	0.17	mg/Kg wet	1.66		75.7	60-140			
2-Methylnaphthalene	1.20	0.17	mg/Kg wet	1.66		72.1	60-140			
Naphthalene	1.09	0.17	mg/Kg wet	1.66		65.4	60-140			
Phenanthrene	1.23	0.17	mg/Kg wet	1.66		74.2	60-140			
Pyrene	1.16	0.17	mg/Kg wet	1.66		69.5	60-140			
Summation of 17 PAHs	20.5		mg/Kg wet							
Surrogate: Nitrobenzene-d5	2.23		mg/Kg wet	3.32		67.2	* 70-130			S-26
Surrogate: 2-Fluorobiphenyl	2.43		mg/Kg wet	3.32		73.0	70-130			
Surrogate: p-Terphenyl-d14	2.54		mg/Kg wet	3.32		76.4	70-130			

LCS DUPLICATE RESULTS

All 17 spiked PAH compounds are within DOEE's 60%-140% Recovery Limit. The %Recovery for Total PAH is 78% (not shown) and is also within DOEE's Limit.

One surrogate compound is outside DOEE's 70%-130% Recovery Limit. The low recovery is normal and is caused by matrix interference. This surrogate would pass if compared to the lab's standard QC limits of 30%-130%.

Relative Percent Difference comparing the LCS and LCSD results for Total PAHs (20.5 ppm and 21.6 ppm) is 5.2%. This value is within DOEE's RPD Limit \leq 30%.

QUALITY CONTROL

Semivolatile Organic Compounds by GC/MS - Quality Control

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B276580 - SW-846 3546										
LCS Dup (B276580-BSD1)										
					Prepared: 02/17/21 Analyzed: 02/18/21					
Acenaphthene	1.19	0.17	mg/Kg wet	1.64		72.6	60-140	5.09	30	
Acenaphthylene	1.23	0.17	mg/Kg wet	1.64		74.9	60-140	5.03	30	
Anthracene	1.35	0.17	mg/Kg wet	1.64		82.3	60-140	5.12	30	
Benzo(a)anthracene	1.32	0.17	mg/Kg wet	1.64		80.4	60-140	4.72	30	
Benzo(a)pyrene	1.27	0.17	mg/Kg wet	1.64		77.1	60-140	7.53	30	
Benzo(b)fluoranthene	1.31	0.17	mg/Kg wet	1.64		79.5	60-140	7.26	30	
Benzo(g,h,i)perylene	1.17	0.17	mg/Kg wet	1.64		71.2	60-140	1.89	30	
Benzo(k)fluoranthene	1.30	0.17	mg/Kg wet	1.64		78.9	60-140	9.55	30	
Chrysene	1.38	0.17	mg/Kg wet	1.64		83.9	60-140	5.54	30	
Dibenz(a,h)anthracene	1.30	0.17	mg/Kg wet	1.64		79.3	60-140	7.63	30	
Fluoranthene	1.35	0.17	mg/Kg wet	1.64		82.4	60-140	6.13	30	
Fluorene	1.24	0.17	mg/Kg wet	1.64		75.5	60-140	5.86	30	
Indeno(1,2,3-cd)pyrene	1.32	0.17	mg/Kg wet	1.64		80.0	60-140	4.53	30	
2-Methylnaphthalene	1.23	0.17	mg/Kg wet	1.64		75.0	60-140	2.87	30	
Naphthalene	1.10	0.17	mg/Kg wet	1.64		67.1	60-140	1.60	30	
Phenanthrene	1.32	0.17	mg/Kg wet	1.64		80.1	60-140	6.71	30	
Pyrene	1.24	0.17	mg/Kg wet	1.64		75.3	60-140	6.99	30	
Summation of 17 PAHs	21.6		mg/Kg wet					5.57		
Surrogate: Nitrobenzene-d5	2.20		mg/Kg wet	3.29		66.8	* 70-130			S-26
Surrogate: 2-Fluorobiphenyl	2.48		mg/Kg wet	3.29		75.4	70-130			
Surrogate: p-Terphenyl-d14	2.69		mg/Kg wet	3.29		81.9	70-130			

DUPLICATE RESULTS FOR SAMPLE WITH ELEVATED PAHS



This example compares the results for a different sealcoat sample containing Total PAHs in the 2,100 to 2,300 ppm range. This sealcoat would qualify for DOE's Silver certification level (Total PAHs <10,000 ppm).

The Lab Duplicate result is compared to the original sample result (or source result) for each PAH compound and for Total PAH. The Relative Percent Difference values are shown for each. The lab did not report the RPD Limit in this case, but all values are within DOE's RPD Limit $\leq 50\%$.

One surrogate compound is outside DOE's 70%-130% Recovery Limit. The low recovery is normal and is caused by matrix interference. This surrogate would pass if compared to the lab's standard QC limits of 30%-130%.

QUALITY CONTROL

Semivolatile Organic Compounds by GC/MS - Quality Control

Analyte	Result	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit	Notes
Batch B270764 - SW-846 3540C										
Duplicate (B270764-DUP1) SEALCOAT 2				Prepared: 11/12/20 Analyzed: 11/15/20						
Acenaphthene	88	55	mg/Kg dry		75			15.3		
Acenaphthylene	ND	55	mg/Kg dry		ND			NC		
Anthracene	160	55	mg/Kg dry		140			12.7		
Benzo(a)anthracene	140	55	mg/Kg dry		120			13.3		
Benzo(a)pyrene	110	55	mg/Kg dry		93			15.0		
Benzo(b)fluoranthene	120	55	mg/Kg dry		120			2.52		
Benzo(g,h,i)perylene	59	55	mg/Kg dry		50			16.1		
Benzo(k)fluoranthene	50	55	mg/Kg dry		45			10.3		J
Chrysene	130	55	mg/Kg dry		110			17.4		
Dibenz(a,h)anthracene	ND	55	mg/Kg dry		ND			NC		
Fluoranthene	340	55	mg/Kg dry		310			8.60		
Fluorene	110	55	mg/Kg dry		94			15.5		
Indeno(1,2,3-cd)pyrene	65	55	mg/Kg dry		55			16.7		
2-Methylnaphthalene	ND	55	mg/Kg dry		ND			NC		
Naphthalene	84	55	mg/Kg dry		75			11.8		
Phenanthrene	560	55	mg/Kg dry		490			13.3		
Pyrene	340	55	mg/Kg dry		260			26.7		
Summation of 17 PAHs	2300		mg/Kg dry		2100			11.0		
Surrogate: Nitrobenzene-d5	142		mg/Kg dry	214		66.1	* 70-130			S-26
Surrogate: 2-Fluorobiphenyl	169		mg/Kg dry	214		78.9	70-130			
Surrogate: p-Terphenyl-d14	201		mg/Kg dry	214		93.8	70-130			

J Detected but below the Reporting Limit (lowest calibration standard); therefore, result is an estimated concentration (CLP J-Flag).

S-26 Surrogate outside of control limits.

CHAIN OF CUSTODY FORM

A sample chain of custody form (COC) must be completed and submitted with the sample for analysis. These forms are available on the laboratory websites and can look different from the chain of custody form shown here.

This example lists all the important information to add on the form. Be sure to include a copy of the DOEE QA protocol with your sample for the laboratory to review. A scanned copy of the COC with laboratory receipt is provided in the lab report.

CHAIN OF CUSTODY PAGE <u>1</u> OF <u>1</u>						Date Rec'd in Lab:		Job #:						
Lab Name Lab Address Lab Phone#		Project Information				Report Information - Data Deliverables				Billing Information				
		Project Name: <i>PAHs in Parking Lot Sealcoat</i>				<input type="checkbox"/> FAX <input type="checkbox"/> EMAIL <input type="checkbox"/> ADEx <input type="checkbox"/> Add'l Deliverables				<input type="checkbox"/> Same as Client info PO #:				
Client Information		Project Location:				Regulatory Requirements/Report Limits								
Client: <i>Enter Manufacturer's Name</i>		Project #:				State /Fed Program Criteria								
Address:		Project Manager:				<i>See DOEE QA Protocol</i>								
Phone:		Turn-Around Time				<div style="display: flex; justify-content: space-between;"> <div style="writing-mode: vertical-rl; transform: rotate(180deg);">ANALYSIS</div> <div style="writing-mode: vertical-rl; transform: rotate(180deg);">17 PAHs by Method 8270D</div> <div style="writing-mode: vertical-rl; transform: rotate(180deg);">%SOLIDS by Method 2540G</div> <div style="writing-mode: vertical-rl; transform: rotate(180deg);">MATRIX CODES GW: Ground Water DW: Drinking Water A: Air S: Soil SL: Sludge/Waste O: Other</div> <div style="writing-mode: vertical-rl; transform: rotate(180deg);">TOTAL # BOTTLES</div> </div>								
Fax:		<input type="checkbox"/> Standard <input type="checkbox"/> RUSH <small>(only confirmed if pre-approved)</small>												
Email:		Date Due: Time:												
<input type="checkbox"/>														
Other Project Specific Requirements/Comments/Detection Limits:														
<i>Report results in dry weight. Report 17 Compounds + Total PAH. Use 1 to 5 grams for extraction. Sample contains asphalt (CAS# 8052-42-4) with low/no PAHs expected. Use low detection limits if possible. Please hold sample if Lab Duplicate (LD) is needed.</i>														
Lab ID (Lab Use Only)	Sample ID	Collection		Sample Matrix	Sampler's Initials	ANALYSIS					Sample Specific Comments	TOTAL # BOTTLES		
	<i>SEALCOAT1 (Enter Product Name)</i>	Date	Time											
		1-31-21	12:00	SL	SPG	X	X						<i>MS and MSD Required</i>	1
													<i>Report Blank, LCS + LCS Dup</i>	
<i>Note: Do not refrigerate or freeze sample!</i>					Container Type	JAR							Please print clearly, legibly and completely. Samples can not be logged in and turnaround time clock will not start until any ambiguities are resolved.	
					Preservative	NO								
Relinquished By:			Date/Time	Received By:			Date/Time							
<i>Enter Your Name, Date and Time</i>														

**DOEE SEALCOAT QC PROTOCOL
PAHS IN PARKING LOT SEALCOAT CERTIFICATION**

APPENDIX B

DATA REVIEW PROCESS

SUMMARY OF DATA REVIEW PROCESS

Data Review is performed to determine whether or not the results reported by the laboratory are biased and whether the data can be used for project decisions (e.g., whether the Total PAHs are < 1,000 mg/kg or < 10,000 mg/kg on a dry weight basis). To accomplish this goal, the Quality Control (QC) associated with the sample results needs to be evaluated in a systematic manner.

Samples are analyzed within Quality Control (QC) Batches (group of up to 20 field samples extracted at the same time with "Batch" QC). When reviewing the data, you want to first make sure that you understand which Batch QC Samples (e.g., Method Blank and LCS/LCSD) are associated with your samples. The Method Blanks and LCS usually are reported with a QC Batch designation while MS/MSD and LD (also called "Dup") must reference the parent sample used to create these QC samples.

All QC criteria given in this Appendix are based on the requirements provided in the DOEE Sealcoat QA Protocol. As you are performing your review, note any QC deviations so that once you've concluded your review, you can understand the overall potential QC issues with your results. The basic steps in Data Review for Usability are shown below. Please refer to EPA QA/G-8 for additional guidance on environmental data verification and data validation.

1 Review the Data Package for completeness to ensure the following are in the data package and make yourself familiar with how the data are presented:

- Project or case narrative
- All samples on COC have the 17 PAHs + Total PAHs and %Solids results reported
- The analysis method, extraction method and %Solids method are reported
- All Method Blanks required are reported (check QC Batch on sample data to QC Batch on QC)
- All LCS/LCSD Reported (check QC Batch on sample data to QC Batch on QC)
- LD Reported, if requested (check Sample ID and match to your sample)
- MS or MS/MSD, if requested (check Sample ID and match to your sample)
- Executed (all signatures present) Chain-of-Custody (COC) present

2 Review the Project or Case Narrative

- If the lab raises a quality issue, keep this in mind as you review the data

3 Review Method Blank

- Are all compounds reported (all 17 PAHs + Total PAH)? If not, contact the lab.
- Are there any detected PAHs in the Blank? If yes, that PAH may be biased high in all samples run in the QC Batch

4 Review Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD)

- Are all compounds reported (all 17 PAHs + Total PAH)? If not, contact the lab.
- Were all compounds recovered within criteria (60-140%)? If not, the PAH(s) that are outside criteria may be biased (high recovery of LCS and LCSD indicate possible high bias and low recovery indicates possible low bias)
- Were LCS/LCSD RPDs within criteria ($RPD \leq 30\%$)? If not, those PAHs with $RPD > 30\%$ are uncertain in the samples
- Issues with LCS/LCSD affect all samples that were analyzed in the QC Batch
- If recoveries are very poor (< 10% recovery), contact the lab since the data should not have been reported and the entire QC Batch (samples + QC) may need to be redone

SUMMARY OF DATA REVIEW PROCESS - *CONTINUED*

5 Review Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

- Are all compounds reported (all 17 PAHs + Total PAH)? If not, contact the lab.
- Were all compounds recovered within criteria (60-140%)? If not, PAHs that are outside criteria may be biased (high recovery of MS/MSD indicate possible high bias and low recovery indicates possible low bias)
- Were MS/MSD RPDs within criteria (RPD \leq 50%)? If not, PAHs with RPD >50% are uncertain in the samples
- Issues with MS/MSD only affects the sample used for spiking (native sample)

6 Review Surrogate Recoveries

- Were surrogates reported for all samples and QC? If not, contact the lab.
- Were recoveries within criteria (70-130%)? If not, sample data may be biased (low recovery of surrogate(s) indicates possible low bias in all PAH results while high bias indicates a possible high bias for all detected results only – non-detects are not affected by a high bias)
- Issues only affect the samples with surrogates not within criteria

7 Review Field Samples - use information obtained above to determine impact on data, plus:

- Verify Holding Times (HT) compared to method / project requirements
 - Extraction HT = date of extraction – date of collection, if < 14 days, HT acceptable
 - Analysis HT = date of analysis – date of extraction, if < 40days, HT acceptable
 - If HT not acceptable all result for that sample may be biased low
- Understand Reporting Limits (RLs) or Limits of Quantitation (LOQ)
 - Are units correct for the matrix (mg/kg)? Are they sample-specific (different from method blank LOQs for solid samples) and dry-weight corrected (for soil/sediment)? If not, contact the lab
 - Are there results reported below the LOQ (i.e., “J” qualified data)?
 - Are the LOQs below the Project QL?
- Were the correct methods used (8270D or 8270E) and were all 17 PAHs + Total PAHs reported? If not, contact the lab.

If QC criteria are not met, this does not necessarily mean that the laboratory performed poorly. If the Method Blanks and LCS/LCSD have QC issues, this does point to a lab issue; however, if the MS/MSD or MS and LD don't meet criteria while the Method Blank and LCS/LCSD do meet criteria, then this is an indication that the QC exceedance is because of a problem with the sample matrix (e.g., imprecision can be an indication of sample heterogeneity).

Cumulative bias, which occurs if there are multiple QC exceedances, can be complicated to evaluate. If the QC that exceeded criteria are biased in the same direction (e.g., all are biased high or all biased low), then the cumulative bias for your sample data would simply be the bias for the QC (i.e., high or low). If however, your results have a mix of issues (e.g., low surrogate recovery + high LCS recovery + low MS recovery), then the bias is considered indeterminate (i.e., you can't tell whether your data are biased low or high but they do have uncertainty).

Usability of the data is determined by evaluating all of these QC elements and deciding if the data are biased or uncertain. If the QC doesn't meet the DOEE Sealcoat QA Protocol requirements, this does not necessarily mean the data are unusable but instead means that you, the data user (Manufacturer and DOEE), will need to evaluate whether the bias in the data is acceptable. If the bias is acceptable, then there is confidence that any decision made about the certification level for the sealcoat is accurate. For example, if your Total PAHs are < 500 mg/Kg-dry weight and you have a high bias in QC, you can be sure that your sealcoat sample is below the 1,000 mg/kg Gold certification limit. If however, your Total PAHs are 950 mg/Kg-dry weight with indeterminate bias, you cannot be sure that your sealcoat sample is actually below the 1,000 mg/kg Gold limit and further analyses may be required.