

DRAFT

**District Department of Energy and Environment
Pavement Sealant Quality Assurance Protocol**

Prepared for:

Department of Energy and Environment

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Table of Contents

Table of Contents	1
List of Acronyms and Abbreviations	2
Problem Definition	3
Measurement Performance Criteria	4
Reference Limits and Evaluation Tables	6
Sample Containers, Preservation, and Hold Times	9
Analytical Method and Reporting Requirements	10

List of Acronyms and Abbreviations

°C	Celsius degree
CBT	Chesapeake Bay Trust
COC	Chain of Custody
CRM	Certified Reference Material
DC DOEE	District of Columbia Department of Energy and Environment
DOEE	Department of Energy and Environment
DQI	Data Quality Indicator
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EPA	Environmental Protection Agency
g	Gram
L	Liter
LCS	Laboratory Control Sample
LCS D	Laboratory Control Sample Duplicate
LD	Laboratory Duplicate (also called Duplicate)
LOQ	Limit of Quantitation (equivalent for this project to the QL)
MB	Method Blank
MDE	Maryland Department of the Environment
MDL	Method Detection Limit
mg	Milligram
mg/kg	milligram per kilogram (ppm)
µg/kg	microgram per kilogram (ppb)
mL	Milliliter
MM/DD/YY	Month/Day/Year
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NEH	New Environmental Horizons, Inc.
NELAP	National Environmental Laboratory Accreditation Program
PAH	Polycyclic Aromatic Hydrocarbon
ppm	parts per million
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
QL	Quantitation Limit
RPD	Relative Percent Difference
RTU	Ready to Use
SDG	Sample Delivery Group
SOPs	Standard Operating Procedures
TAT	Turnaround Time
TBD	To Be Determined
TMDL	Total Maximum Daily Load
USGS	United States Geological Survey

Problem Definition

The problem to be addressed by the project:

The District Department of Energy and Environment (DOEE) has established two certification levels for pavement sealant products. Those with Total Polycyclic Aromatic Hydrocarbon (PAH) content $\leq 1,000$ mg/kg (ppm) fall into the Gold certification level, those that contain Total PAH content $> 1,000$ mg/kg but $\leq 10,000$ mg/kg are in the Silver certification level. Those pavement sealants with $> 10,000$ mg/kg Total PAH content will not be certified.

Each pavement sealant Manufacturer participating in this program must demonstrate the accurate Total PAH content of their product, which must be verified biennially to maintain certification.

Polycyclic Aromatic Hydrocarbon (PAH) Measurement Performance Criteria

Matrix	Sealcoat				
Analytical Group	Target PAHs & Total PAHs				
Concentration Level	High				
Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Manufacturer SOP for Ready to Use Product ¹	SW-846 Method 8270D (or updated version)	Sensitivity	< QL	Method Blank	A
		Accuracy/Precision	LCS/LCSD Recoveries: 60-140% ² LCS/LCSD: RPD ≤ 30%	LCS/LCSD	A
		Accuracy	Recoveries within 70-130%	Surrogates	A
		Accuracy/Precision	MS/MSD Recoveries: 60-140% ² MS/MSD: RPD ≤ 30%	MS/MSD	A
		Precision/Representativeness	RPD ≤ 50% for results > 2x QL	Lab Duplicate (LD)	A
		Completeness	100% sample collection 100% laboratory analysis	Data Completeness Check	S & A

Notes:

1. Samples submitted for analysis must be representative of what is sold on the market.
2. Data packages with qualifiers falling outside but close to stated ranges (at least above 50% recovery) may still be submitted without reanalysis for consideration by DOEE if all other QC data is found within acceptable ranges. Visit Section III of the User's Guide for more information.

Percent Solids Measurement Performance Criteria

Matrix	Sealcoat				
Analytical Group	Percent Solids ¹				
Concentration Level	High				
Sampling Procedure	Analytical Method/SOP	DQIs	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
NA	SM 2540G or equivalent	Precision	Sample/LD RPD ≤ 10%	Laboratory Duplicate (LD)	A
		Completeness	100% sample collection 100% laboratory analysis	Data Completeness Check	S & A

Notes:

1. The water content in sealcoat products may vary. All products tested must be reported on a dry weight basis (mg/kg).

Polycyclic Aromatic Hydrocarbon (PAH) Reference Limits and Evaluation Tables

Matrix: Sealcoat

Analytical Group: 17 Target PAHs plus Total PAHs (SW-846 8270D or updated version)

Concentration Level: Low / Medium / High

Analyte	CAS Number	Units	Project Action Level ¹	Project QL (ppm) ²	Analytical Method		Achievable Laboratory Limits ³	
					MDL	Method QL	MDL ⁴	QL
Naphthalene	91-20-3	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
2-Methylnaphthalene	91-57-6	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Acenaphthylene	208-96-8	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Acenaphthene	83-32-9	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Fluorene	86-73-7	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Phenanthrene	85-01-8	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Anthracene	120-12-7	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Fluoranthene	206-44-0	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Pyrene	129-00-0	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Benzo[a]anthracene	56-55-3	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Chrysene	218-01-9	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Benzo[b]fluoranthene	205-99-2	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD

Analyte	CAS Number	Units	Project Action Level ¹	Project QL (ppm) ²	Analytical Method		Achievable Laboratory Limits ³	
					MDL	Method QL	MDL ⁴	QL
Benzo[k]fluoranthene	207-08-9	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Benzo[a]pyrene	50-32-8	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Indeno[1,2,3-cd]pyrene	193-39-5	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Dibenzo[a,h]anthracene	53-70-3	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Benzo[g,h,i]perylene	191-24-2	mg/kg – dry wt	NA	29.4	Per Lab SOP	Per SW-846 8270D	<QL	TBD
Total PAHs	NA	mg/kg – dry wt	1000 (Gold) 10000 (Silver)	500	Per Lab SOP	Per SW-846 8270D	<QL	TBD

Notes:

1. Project Action Level (PAL) is established for Total PAHs, which is defined as the sum of the concentrations of the detected PAHs shown in this Worksheet (non-detects have zero contribution to the Total PAH). The “Gold” level certification PAL is 1,000 mg/kg (ppm) and “Silver” level certification is 10,000 mg/kg (ppm).
2. The Project Quantitation Limit (QL) has been set to ½ the PAL for “Gold” (500 mg/kg). Sensitivity for analysis will be considered acceptable if non-detect PAH has a dry-weight concentration < QL.
3. The lab will report detected results between the MDL and QL, qualified as estimated "J" data. Non-detected results will be reported at the QL.
4. MDL should be established by lab following demonstration of capability.

Percent Solids Reference Limits and Evaluation Tables

Matrix: Pavement Sealant

Analytical Group: Percent Solids (SM 2540G or equivalent)

Concentration Level: Low / Medium / High

Analyte	CAS Number	Units	Project Action Level	Project QL	Analytical Method		Achievable Laboratory Limits	
					MDL	Method QL	MDL	QL
Percent Solids	NA	%	NA	0.1%	Lab SOP	Lab SOP	TBD	TBD

Notes:

Sample Containers, Preservation, and Hold Times

Analytical Group	Matrix ¹	Analytical and Preparation Method/SOP Reference	Accreditation Expiration Date	Containers (number, size, and type)	Preservation Requirements	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
PAH	Wet Sealant RTU	Method 8270D (or updated method)	Lab chosen for analysis must be accredited by NELAP	1 8 oz. jar or can	Store at room temperature. Do not freeze sealcoats.	14 days	Extracts stored at < 6°C; analysis within 40 days of extraction	28 days

Notes:

1. RTU = Ready to Use. Product submitted for analysis must be representative of what is sold on the market. Collect and submit samples in 8 oz or pint size containers (jars, cans) with a tight-fitting lid. Only collect Ready to use (RTU) samples of pavement sealant and not emulsions or concentrates. Include a copy of the product’s safety data sheet (SDS) with your sample. Label the sample container with “Shake sample prior to use” and instruct the laboratory to store at room temperature and to not freeze the samples (including transit). The samples must be submitted to the laboratory under Chain-of-Custody (COC) [see example in Appendix 1]. Provide the laboratory with a copy of this DOEE Pavement Sealant QA Protocol.

Analytical Methods and Reporting Requirement Information

Samples must be submitted to a NELAP-certified laboratory under Chain-of-Custody and you should provide the laboratory with a copy of this DOEE Pavement Sealant QA Protocol.

This certification program was developed through testing various types of pavement sealants by a variety of analytical extraction and analysis protocols. Based on this testing, SW-846 Method 8270D (or updated version) is required for analysis of the 17 PAHs and Total PAHs and percent solids determination must be performed. Labs may use any of the following extraction procedures: SW-846 Methods 3540C (Soxhlet Extraction), 3541 (Automated Soxhlet Extraction), 3546 (Microwave Extraction), and 3550C (Ultrasonic Extraction). Waste Dilution (SW-846 Method 3580A) is not an acceptable preparation procedure for this certification.

Sample Preparation

- The contents of the container received from the Manufacturer should be shaken prior to use
- Spike surrogates into the sample for extraction prior to mixing any drying agents (e.g., sodium sulfate) with the sample prior to extraction.
- Use enough sample to meet Recovery Limit of 29.4 ppm for each PAH, ideally 1-5 g of material for extraction

Analytical Reporting (see Appendix A for an example data package)

- All results should be reported in units of mg/kg on a dry weight basis.
- The project narrative must clearly explain all of the sample preparation, cleanup, and analysis steps taken in the reporting of results. Any QC deviations (e.g. spike recoveries outside control, instrument calibration exceedances) must also be documented in the narrative.
- A summary data package including:
 - Project Narrative
 - Copy of Chain-of-Custody form
 - Extraction method used
 - Sample (including LD) results with surrogate recoveries
 - Sample/LD RPDs
 - Method Blank results with surrogate recoveries
 - LCS/LCSD results with PAH recoveries, and LCS/LCSD RPDs
 - MS/MSD results with PAH recoveries, and MS/MSD RPDs (only MS results and PAH recoveries are required if the lab chose not to perform MSD analysis because the level of Total PAHs was too high for the spike to be meaningful)

Raw data including preparation benchesheets, instrument runs (chromatograms and mass spectra), and maintenance logs should be kept on-file at the laboratory and available if requested.

Reporting to DOEE

- The Manufacturer must include a copy of the safety data sheet (SDS) for each product tested with the sample results.
- DOEE form signed by Manufacturer attesting to the accuracy of the sample submitted for analysis.
- The analytical data must be submitted in its entirety to DOEE in the format received directly from the laboratory. It is the Manufacturer's responsibility to ensure that the laboratory was in compliance with the requirements of this program and it is expected that the Manufacturer will carry out any corrective actions required (e.g., fixing submission errors as outlined above) prior to submission of the data to DOEE. See Appendix A for an example data package and Appendix B for general guidance in reviewing the data package for quality.