



Ultraviolet Fluorescence Method for Polycyclic Aromatic Hydrocarbons Using Methanol

STANDARD OPERATING PROCEDURE: ASPHALT-PAHS-TRILOGY-SOPV1, PAGE 1 of 2

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Supplies & Equipment Required for Analysis:



Sample Test Kit:
Part No. EXTR010-20-PAHS

- Supplies for 20 samples.
- Use with methanol (solvent not included). Use with HPLC grade methanol, CAS #67-56-2.



Sitelab UVF-Trilogy PAH Analyzer:
Part No. 7200-004-PAHS

- Includes PAHS UV Module and factory calibrations for testing PAHs or Benzo[a]Pyrene.
- Quartz glass cuvettes, scale, micro-pipette, solvent dispenser bottle and other lab equipment needed is included.

Calibration Products (Optional):

- PAH Calibration Kit, Part No. CAL-060M
- Benzo[a]Pyrene Standard, Part No. CAL-BAP-COALTAR

Sample Extraction Procedure for Reclaimed Asphalts & Coal Tar Contaminates

Samples prepared using materials supplied in EXTR010-20-PAHS test kit. Solvent dispenser bottle, digital scale, pipette and other tools shown are included with UVF-Trilogy analyzer.

1 Sample Extraction Vial (40 mL Capacity) Metal Spatula

Sample

Digital Scale

Weight: 5 grams or 2 grams

Samples should be homogenized prior to use. Asphalts and sealcoats should be crushed and sieved to 4 mm particle size. Using a glass sample extraction vial, remove the cap and place vial onto the scale and tare the weight to zero. Scoop out and weigh 5.0 g of sample into the vial. Use 2.0 g for highly contaminated samples. Be precise (within +/- 0.1 g).

2 Plastic Graduated Test Tube

Methanol

5 g Sample creates a 4X Extract
2 g Sample creates a 10X Extract

Add 20 mL

Add methanol to the solvent dispenser bottle. Squirt 10 mL of methanol into a plastic graduated test tube. Pour contents into the sample extraction vial. Add another 10 mL to vial for a total of 20 mL of solvent. Keep track of the dilution created in the extract (4X or 10X). Tighten cap and shake vial periodically for 10 minutes, no more than 20 minutes.

3 Filter Extract after 10 Minutes

Syringe

Filter Contents into a Small Extract Vial

Add Filter

4X or 10X

Once the sample has been extracted for 10 minutes, remove the cap, dip a syringe into the vial and suck up the Extract into the syringe. Screw on a filter, push down on the plunger and filter the Extract into a small glass extract vial. Label the vial with "4X" or "10X." Samples extracted for 24 hours will produce higher concentrations and is recommended more for coal tars.

4 Prepare Dilutions for Analysis

Filtered Extract

Pipette

Test Tube

Pipette Extract	Add Solvent To	4X Extract Dilution	10X Extract Dilution
200 µL	5 mL line	= 100X	= 250X
100 µL	5 mL line	= 200X	= 500X
50 µL	5 mL line	= 400X	= 1,000X
40 µL	10 mL line	= 1,000X	= 2,500X

Attach a tip to the adjustable pipette, transfer aliquots of the Filtered Extract into a plastic graduated test tube and dilute with methanol using examples shown above. Prepare a 1,000X Dilution first, recommended for most samples.

Test Sample using UVF-Trilogy Hydrocarbon Analyzer

Use with analyzer factory calibrated to Target PAHs using standards supplied in Sitelab CAL-060M. Record results manually using a log book.

Test Dilution from Step 4

Glass Cuvette

Tissue Wipes

Insert into Analyzer with PAH UV Module

Fill glass cuvette about ½ full with the Dilution made in Step 4. Clean cuvette using a tissue to remove any liquids or fingerprints, place into the analyzer with the UV fluorescence module installed. Select the "PAHS 060M" test from the calibration menu screen and press the green MEASURE FLUORESCENCE button. Readings are in PPM units (mg/Kg).

Calculate PAH Results:
Example:
0.12 ppm x 1,000X Dilution = 120 ppm (mg/Kg)

Detection Range & Limits:
Lower limit = 0.05 ppm (or 50 ppb)
Upper limit = 1.5 ppm (or 1,500 ppb)

Multiply the reading by dilution made to report final concentration. Press measure again to check repeatability. If the 1,000X dilution reads below 0.05 ppm, report sample as "Non-Detect <50 mg/Kg". Empty and rinse test tube with methanol to prepare and test higher or lower dilutions, as needed, using the Extract. Always rinse glass cuvette in between running samples.

Quality Control Tests

The UVF-Trilogy does not need to be calibrated each time its used. QC tests described below are recommended to help validate results and confirm analyzer performs properly.

QC Check for Sample Quenching
1,000X Dilution Result = 120 ppm vs. 500X Dilution Result = 112 ppm
Good, Similar Results! RPD = 7%

Monitor Extraction Efficiency Over Time

	10 Minutes:	24 Hours:
Soil vs.	= 120 ppm	= 130 ppm
Sealcoat	= 19,000 ppm	= 62,000 ppm

"Quenching" can occur when the detector is swamped by too many hydrocarbons which can produce low or poor results. Test a sample at multiple dilutions to confirm readings are linear and results are <20% RPD. Depending on the matrix, UVF can over quantify or under quantify PAH content due to differences in extraction efficiency, but this data can be useful.

QC Test Your Solvent & Perform Calibration Checks

Methanol Blank

PAH Standards

Fill the cuvette ½ full with methanol and test a blank to make sure the solvent is clean. Readings in the blank should be close to zero ppm. If available, test the PAH calibration standards to check the analyzer for accuracy or drift. Readings should be close to each standard's concentration, <20% RPD. See calibration instructions for more details.



UVF-TRILOGY TEST PROCEDURES FOR PAHs IN COAL TARs

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STANDARD OPERATING PROCEDURE: ASPHALT-PAHS-TRILOGY-SOPV1, PAGE 2 of 2

Highly Contaminated Coal Tars Requiring 24 Hour Extraction

Prepare and analyze high dilutions. May not be practical for quick screening purposes.

5 Pipette Add Methanol

1,000X Dilution

Pipette Volume	Add Solvent To	New Dilution Created
250 µL x2	5 mL line	= 10,000X
250 µL	5 mL line	= 20,000X
100 µL	5 mL line	= 50,000X
50 µL	5 mL line	= 100,000X
50 µL	10 mL line	= 200,000X

Use the adjustable pipette and a second test tube to prepare very high dilutions, if necessary, using the dilution in Step 4 diluted further in methanol. Examples shown here uses the 1,000X Dilution with new dilutions created.

6 Example Testing a 100,000X Dilution

Glass Cuvette, Tissue Wipes

0.62 x 100,000X DIL = 62,000 ppm (mg/Kg)

Pour the dilution in Step 5 into the glass cuvette, place into the analyzer and press the MEASURE FLUORESCENCE button. Multiply the reading by the dilution factor to report the final concentration.

Use this protocol for coal tar driveway sealcoats, oily soil samples or DNAPL. The 24 hour extraction time performs best compared to BaP using lab GC methods.

Additional Tests to Perform

Use this option for qualitative analysis

Photograph Extract Colors

Coal Tar Asphalt

Interference in Coal Tar Free Asphalts

Asphalt Bitumen:

- 10 Minute Extraction UVF PAHs = 100 ppm
- 24 Hour Extraction UVF PAHs = 400 ppm

Unfiltered extracts yellow in color indicate high concentrations of PAHs. This can be useful deciding which dilution to make or for observing the color change over time.

Clean asphalts, like binders with no BaP, will fluoresce due to other PAHs in the sample which are not detected by the lab GC/MS methods. Monitoring background levels can help with false positive results.

Need to Test Benzo[a]Pyrene?

Use the BaP calibration stored on analyzer

Coal Tars Contain 4 to 6% Benzo[a]Pyrene

1,000 ppb PAH Standard Response

Fluoresces Same as

50 ppb BaP Coal Tar Standard Response CAL-BAP-COALTAR

Select the "BAPCOALTAR" test from the calibration menu screen to measure Benzo[a]Pyrene calibrated to Sitelab's BaP calibration standards in methanol. BaP sample readings will be about 20 times lower compared to sample readings using the PAHS 060M test. BaP is 5% PAH.

Raw Fluorescence Units (RFU) values are similar. Press MODE to record RFU value.

PAH Calibration Kit

Part No. CAL-060M



- Contains mixture of US EPA regulated PAHs in methanol.
- Standards are reusable with 6-month expiration date.

This product is similar to PAH calibration kit CAL-061M-500D used with UVF-500D analyzers.



Use for screening PAH content in parking lot sealcoats to meet 1,000 ppm or 10,000 ppm PAH action limits, where regulated.

Key Features, Performance and Limitations Comparing UVF-Trilogy and UVF-500D Analyzers, both Suitable for PAHs in Coal Tars

UVF-Trilogy Key Features

- Benchtop instrument
- Factory calibrated to both PAHs and BaP
- Performs multi-point calibration curves
- Detects <5 ppb BaP
- Computer connection

Performance & Limitations

The analyzer uses a 255-nm light source and detects "Target PAHs". It has low detection limits. It is sensitive to PAHs in the C10 to C22 carbon range, including Benzo[a]Pyrene. It is suitable for testing a wider range of contaminants, including PAHs in gasoline, diesel and other refined fuel oils. When testing asphalts or coal tars, it's more susceptible to fluorescence quenching compared to the UVF-500D.

Analyzer Uses 'Snap In' UV Modules

Analyzer displays Raw Fluorescence Units (RFU) or voltage for the Blank and each Calibration Standard.

Modules for GRO, EDRO and TPH OIL are also available. All four modules are used for hydrocarbon fingerprinting analysis.

Example Showing PAH Calibration Curve

Standard (PPM)	Response (RFU)
0 (Blank)	3.1
0.1	595
0.5	2,672
1.0	4,998
1.5	6,880

Fluorescence (RFU)

Concentration (PPM)

Analyzer can be calibrated to PPM or PPB units

UVF-500D Key Features

- Rugged, handheld, low-cost instrument
- Two optical channels
- Detects TPH and PAHs
- Detects <5 ppb BaP
- Solid Standard available, useful for extra QC test

Performance & Limitations

The analyzer uses a 375-nm light source and detects "Heavy PAHs". It too has low detection limits and is very sensitive to Benzo[a]Pyrene, about the same as the UVF-Trilogy. PAH results in asphalts and coal tars are close, typically slightly lower compared to the UVF-Trilogy. It is not suitable for testing PAHs in gasoline, diesel or other refined fuel oils. Lighter PAH compounds fluoresce poorly.

Calibration Data Recorded

Analyzer records the Fluorescence Scale (%FS) or voltage for the Blank and Calibration Standard.

Standard (PPB)	Response (%FS)	Calibration Factor	QC Test 500 PPB Reading
0 (Blank)	0.3%		
500	16.2%	30.9	495

1-Point Calibration Curve

Lower & Higher Calibrations are Linear

Standard (PPB)	Response (%FS)	Calibration Factor	QC Test 500 PPB Reading
25	0.8%	31.3	496
100	3.2%	31.3	499
1,000	32.4%	30.9	485
2,000	64.5%	31.0	490
3,000	93.0%	32.3	503
4,000	OVER	RSD = 1.5%	RSD = 1.2%

OVER response indicates %FS exceeds 100% limit



The UVF-500D, Part No. 50200, and supplies available are sold online with Sitelab's partner Obstitech ApS in Denmark.

Visit web shop: UVF-500D.COM
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