



# UVF-500D TEST PROCEDURES FOR PAHs IN COAL TARs

## Ultraviolet Fluorescence Method for Polycyclic Aromatic Hydrocarbons Using Methanol

STANDARD OPERATING PROCEDURE: COALTARS-PAHS-500D-SOPV1, PAGE 1 of 2

### 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 U VF-500D Analyzer: Part No. 50200

- Use the 8mm Cuvette Adapter
- Use on Channel A optics only

#### Lab Equipment and Other Parts Needed, Sold Separately:

- U VF-500D Starter Kit, Part No. 90300
- Soil Accessory Kit, Part No. 90300-SAK
- 8mm Round Glass Cuvettes, 400/Pack, Part No. 50957
- U VF-500D PAH Calibration Kit, Part No. CAL-061M-500D

### Sample Extraction Procedure for Coal Tar Contaminates, Asphalts and Sealcoats, Creosotes, Soils from MGP Sites

Samples prepared using materials supplied in EXTR010-20-PAHS test kit. Solvent dispenser bottle, digital scale, pipette and other tools needed are included in U VF-500D accessories.

**1** Sample Extraction Vial (40 mL Capacity) Metal Spatula

Sample

Digital Scale

Weigh: 5 grams or 2 grams

Samples should be homogenized prior to use. Asphalts or 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. For asphalts and soil samples, extract for 10 minutes.

**3** Filter Extract to Stop Extraction

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 for coal tar sealcoats.

**4** Prepare Dilutions for Analysis

Filtered Extract

Pipette

Add

Test Tube

Pipette Extract	Solvent To Add	Dilution	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 U VF-500D Hydrocarbon Analyzer

Use with analyzer calibrated to 500 ppb PAH standard supplied in Sitelab CAL-061M-500D. Record results manually using a log book.

**Test Dilution from Step 4**

Glass Cuvette

Tissue Wipes

Cuvette Adapter

Insert into Analyzer

PPM 1

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 cuvette adapter, insert into analyzer and press the READ button. The U VF-500D displays PPM units only, but PAHs are calibrated and analyzed at PPB concentrations. Divide readings by 1,000 to convert to PPB.

**Calculate PAH Concentration:**  
Example: Test a 1,000X Dilution  
120 ppb x 1,000X = 120,000 ppb ÷ 1,000 = 120 ppm (mg/Kg)

**Detection Range & Limits:**  
Lower limit = 25 ppb (µg/Kg)  
Upper limit = 2,000 ppb (µg/Kg)

Multiply the reading by dilution made to report final concentration. Divide result by 1,000 to convert back to PPM units. Press READ again to check repeatability. If the 1,000X dilution reads below 25 ppb, report sample as "Non-Detect <25 mg/Kg". Empty and rinse test tube with methanol to prepare and test higher or lower dilutions, as needed, using the Extract.

### Quality Control Tests

The U VF-500D does not need to be calibrated each time its used. The 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: 120 ppm vs. 130 ppm  
24 Hours: 19,000 ppm vs. 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, U VF 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

0.0

495

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 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.



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### 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 Cupvette Tissue Wipes Cuvette Adapter 0.620 x DIL = 62,200 ppm

Pour the dilution in Step 5 into a glass cuvette, place into the analyzer and press the READ 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 PAHs 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 Samples**

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, bitumen without coal tars, 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 STD VAL feature to estimate BaP content

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

500 ppb PAH Standard Response  
Fluoresces Same as  
25 ppb BaP Coal Tar Response

CAL-BAP-COALTAR

The UVF-500D can measure BaP, similar to using Sitelab's BaP coal tar standard without the need to recalibrate.

Press the **STD VAL** button and use the arrow keys to decrease the concentration from 500 to 25. BaP sample readings will be about 20 times lower compared to PAH sample readings. BaP is 5% PAH.

**The DIAG %FS values do not change!**

### PAH Calibration Kit

Part No. CAL-061M-500D

- 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-060M used with UVF-Trilogy analyzers.

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

**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 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%	30.9	495
500	16.2%	30.9	495

**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

**UVF-Trilogy Key Features**

- Benchtop instrument
- Factory calibrated to PAHs, BaP or both
- 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 too 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) vs Concentration (PPM)

Analyzer can be calibrated to PPB or PPM units

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**  
Phone: +45 20315771





# UUVF-500D CALIBRATION INSTRUCTIONS

Using Sitelab CAL-061M-500D for **Polycyclic Aromatic Hydrocarbons**

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Visit: UUVF-500D.COM

Need help? Call Toll Free 877-SITELAB or Dial (USA) 978-363-2299

CAL-061M-500D-SOPV1

## Equipment Required

### Sitelab PAH Calibration Kit Part No. CAL-061M-500D



Use to calibrate for Polycyclic Aromatic Hydrocarbons, detects Heavy PAHs in the C14 to C40 carbon range. Includes:

- 3 Calibration standards
- 2 Reference standards
- 20 Cuvettes + other supplies



**WARNING!** This product contains methanol solvent (highly flammable, CAS #67-56-2). Use in ventilated area, handle with care, store at room temperature.

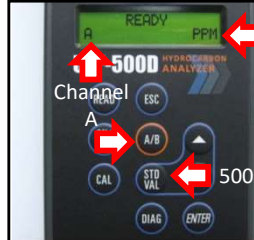


**UUVF-500D, Part No. 50200** with 8mm Cuvette Adapter & Solvent Dispenser Squirt Bottle

#### Other Items Needed:

- 8mm Round Glass Cuvettes, Part No. 50957, 400/Pack
- Methanol, Use "HPLC" Grade

## 1. Set up Analyzer



The UUVF-500D displays PPM units, used for TPH analysis.

Disregard PPM, enter PAH STD value in PPB!

Press the ON/OFF button to turn on. The READY screen – or home screen - appears and should display the "A" Channel is selected. If not, press the A/B button to switch from B to A. Next, press the STD VAL button to check and confirm the PAH standard's concentration is set to 500 ppb (shown as 500 ppm). If not, use the arrow keys to adjust the standard value and then press ENTER. Once the standard value and Channel are set, press the CAL button to begin the calibration process. Press ESC button to abort the calibration at any time.

## 2. Use Methanol for Solvent Blank

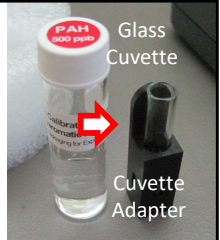


Solvent Dispenser Squirt Bottle

Fill Cuvette ½ Full with Methanol

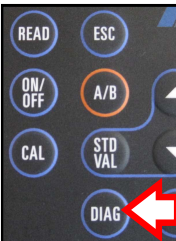
Always use clean solvent to blank or "zero" the analyzer during the calibration process. Use the same solvent used to extract samples. Fill the solvent dispenser bottle with methanol and squirt into a glass cuvette about half full. Wipe the outside glass with a tissue wipe to remove any liquids or fingerprints. Place the blank into the cuvette adapter, insert into the analyzer and press the ENTER button when ready to test the blank. The analyzer will read the blank for a few moments, settle and then prompt you to the next step. Remove the blank from the cuvette adapter and empty contents into a waste jar.

## 3. Use the 500 ppb PAH Standard



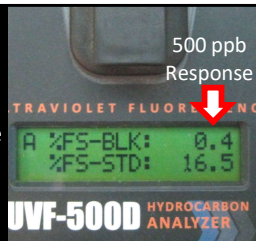
Pour the 500 ppb PAH Calibration Standard into a new cuvette about half full. Or use a pipette to transfer contents (less messy). Wipe the outside glass clean with a tissue wipe, place into the cuvette holder, insert into analyzer and press the ENTER button. The analyzer will read the standard for a few moments and will prompt you to press ENTER again when calibration is complete. When finished, remove the standard from the cuvette adapter. Add a plug cap to cuvette to save and reuse later or pour contents back into the vial, the standards are reusable. Throw out cuvette or rinse cuvette with hexane to clean and reuse.

## Check & Record Calibration Diagnostic Data



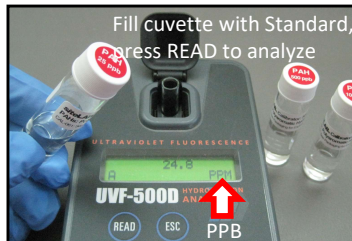
Record the %FS values of the blank and the standard.

%FS values should be close/similar each time the analyzer is calibrated to PAHs.



After calibrating the analyzer, press the "DIAG" button to record the percent fluorescence scale (%FS or voltage) for the blank and the standard. These sensitivity values are very important and should be recorded. The %FS-BLK value should be below 1 and close to zero. The %FS-STD value using the Standard should be in the 12 to 20 range (each analyzer varies) and should produce similar %FS-STD values each time its used, within 10%, no more than 20% RPD. If not, a new standard should be used. See the Certificate of Analysis for details. Once the UUVF-500D is calibrated, it's stable for very long periods of time. The detector does not drift. Only recalibrate if necessary.

## Check Standards for Quality Control



Fill cuvette with Standard, press READ to analyze

#### Acceptance Criteria

- 500 ppb Standard: Reads 450 to 550 ppb
- 100 ppb Standard: Reads 80 to 120 ppb
- 25 ppb Standard: Reads 20 to 30 ppb

Periodically check analyzer for accuracy and precision by testing the 500 ppb Standard. Readings should be close to 500 ppb. Press READ button again to check repeatability. Next, test the 100 ppb and 25 ppb standards to confirm the analyzer is linear at the lower end of the curve. Readings should be close, within 10%, no more than 20% off, for best performance. If readings are outside the acceptance criteria, test new, fresh standards. Avoid testing standards past their 6-month expiration date. Test a solvent blank too to confirm the methanol is clean. This should be done on a more frequent basis, especially when new solvent is used.

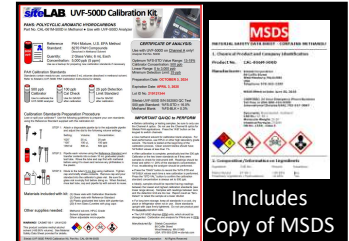
## Solid Standard Available



The UUVF-500D includes a 100 ppm TPH Solid Standard. This device reads the same as the 100 ppm TPH standard used to factory calibrate the analyzer.

The Adjustable Solid Standard can be used and tuned to read "500 ppb", after the analyzer is calibrated to BaP using the 500 ppb Standard. Use the device for extra QC test or to calibrate. Once tightened, readings are always stable.

## Certificate of Analysis



Sitelab's PAHs Calibration Kit includes a Certificate of Analysis. This document has important information about the product, including the preparation date, expiration date (6 months) and lot ID number. Instructions are provided to make new calibration standards using two Reference Standards provided. Use of a micro pipette is required.