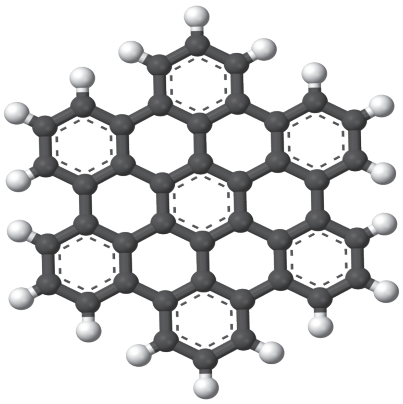
Polynuclear Aromatic Hydrocarbons (PAH) from a Water Matrix



UCT Part Numbers

ECUNIPAH
ENVIRO-CLEAN®
Universal PAH/DRO, 2000 mg
C18, 83 mL cartridge

ECSS25K
anhydrous. sodium sulfate

Summary:

This application note describes a procedure for the analysis of PAH compounds in water. Analysis is conducted by LC with UV or fluorescence, or GC/MS.

Polynuclear Aromatic Compounds			
PAH	**CASRN**	**PAH**	**CASRN**
Acenaphthene	83-32-9	Chrysene	218-01-9
Acenaphthylene	208-96-8	Dibenzo(a,h)anthracene	53-70-3
Anthracene	120-12-7	Fluoranthene	206-44-0
Benzo(a)anthracene	56-55-3	Fluorene	86-73-7
Benzo(a)pyrene	50-32-8	Indeno(1,2,3-cd)pyrene	193-39-5
Benzo(b)fluoranthene	205-99-2	Naphthalene	91-20-3
Benzo(g,h,i)perylene	191-24-2	Phenanthrene	85-01-8
Benzo(k)fluoranthene	207-08-9	Pyrene	129-00-0

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Procedure:

1. Condition Cartridge

a) Assemble an all glass vacuum manifold system **UCT ECUCTVAC1**, **ECUCTVAC3** or **ECUCTVAC6** (1,3 or 6 station)
b) Place a **ECUNIPAH** cartridge(s) on the vacuum manifold*
c) With vacuum turn off add 10 mL of methylene chloride to the cartridge
d) Let it soak for 1 minute
e) Turn on vacuum and draw through to waste
f) Add 10 mL of methanol to the cartridge
g) Let it soak for 1 minute
h) Draw the methanol to the level of the frit
i) Add 10 mL of deionized water to the cartridge
j) Let it soak for 1 minute
k) Draw most of the water to waste but do not allow the sorbent to dry

Note: Do not let the cartridge go dry after addition of methanol otherwise repeat starting at step 1. f)

2. Sample Addition

a) Add surrogates to the sample
b) Shake
c) Add the sample to the cartridge under vacuum. Draw the sample through the cartridge in approximately 20 – 30 minutes
d) Allow the cartridge to dry under full vacuum for 10 minutes**

3. Extract Elution

a) Place a collection tube or vial in the vacuum manifold
b) Rinse sample bottle with 10 mL of methylene chloride to remove any sample residue from the glass
c) Add the methylene chloride rinse to the cartridge
d) Allow to soak for 1 minute then draw through
e) Repeat this procedure three more times using 10 mL aliquots of methylene chloride
f) Dry the extract by passing it through 5 grams of **ECSS25K** anhydrous sodium sulfate
g) Thoroughly rinse the collection device with methylene chloride and add this solvent to the sodium sulfate

4. Concentration and Analysis

a) Using a standard analytical evaporator with gentle N_2 flow and low temperature, carefully concentrate the extract to a final volume for GC/MS analysis. Solvent exchange into acetonitrile and take to a 1 ml final volume for HPLC analysis

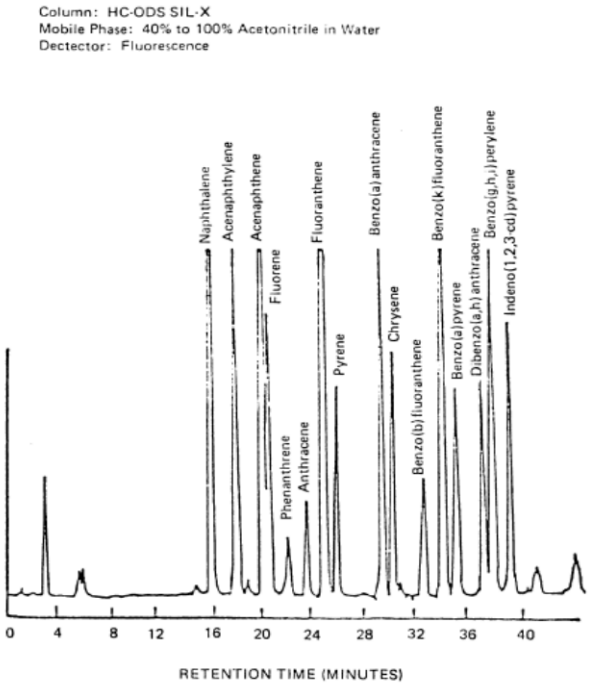
Note: Most analysis errors are caused by poor concentration technique. Do not concentrate below 0.5 mL or low recoveries may result

5. LC/UV Analysis[1]

HPLC conditions:

- Reverse phase HC-ODS Sil-X or equivalent, 5 micron particle size 250-mm x 2.6-mm I.D. stainless steel column
- Isocratic elution: acetonitrile/water (4:6)(v/v)
    - *5 min then linear gradient elution to 100% acetonitrile over 25 min*
    - *Flow rate: 0.5 mL/min*
- Fluorescence detector: For excitation at 280-nm and emission greater than 389-nm cutoff (Corning 3-75 or equivalent) Fluorometers should have dispersive optics for excitation and can utilize either filter or dispersive optics at the emission detector
- UV detector: 254 nm

Chromatogram from LC Analysis



5. GC/MS Analysis[2]

GC/MS	
Column	30 m x 0.25 mm ID (or 0.32 mm ID) 1 µm silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent)
Mass spectrometer	GC/MS operating conditions-Use the following recommendations as guidance:
Mass range	35-500 amu
Scan time	1 sec/scan
Initial temperature	40 °C, hold for 4 minutes
Temperature program	40-270 °C at 10 °C/min
Final temperature	270 °C, hold until benzo[g,h,i]perylene elutes
Injector temperature	250-300 °C
Transfer line temperature	250-300 °C
Source temperature	Use manufacturer's specifications
Injector	Grob-type, splitless
Injection volume	1-2 µL
Carrier gas	H_2 at 50 cm/sec or helium at 30 cm/sec
Ion trap only	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. The mass spectrometer must be capable of producing a mass spectrum for DFTPP.

GC/MS tuning standard--A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/µL each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store at -10EC or less when not in use. If a more sensitive MS is employed to achieve lower detection levels, a more dilute tuning solution may be used.

*The ENVIRO-CLEAN® Universal PAH cartridge can be used on standard vacuum manifolds (#**VMFF016GL**), standard disk manifolds (#**ECUCTVAC6**) (with adapter part #**ECUCTADP**). The cartridge is specifically designed to fit the Horizon SPE DEX 4790® made by Horizon Technologies, Inc.

**Faster drying results can be obtained by removing the cartridge during drying and shaking or tapping the excess moisture from the bottom of the cartridge. Drying times are approximate. Do not over dry as low recoveries may result.

References:

[1] Analytical conditions from EPA Method 8310, "Polynuclear Aromatic Hydrocarbons" - Revision 0

[2] Analytical conditions from EPA Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)" Revision 3, 1996

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