EPA Method 8310: Solid Phase Extraction of Polynuclear Aromatic Hydrocarbons (PAHs) in Water

UCT Part Numbers

ECUNIPAH
ENVIRO-CLEAN® Unendcapped C18
2000mg/Universal Cartridge

ECUNIMSS
ENVIRO-CLEAN®
Muffled sodium sulfate
20g/Universal Cartridge

ECUCTADP
ENVIRO-CLEAN®
Glass Cartridge Adaptor

ECUNIBHD
ENVIRO-CLEAN®
White Bottle Holder

ECUCTVAC6
ENVIRO-CLEAN®
6 Station Vacuum Manifold Stainless Steel System

Summary:

Polynuclear aromatic hydrocarbons (PAHs) are consisted of a large group of organic compounds with two or more fused aromatic rings. Hundreds of different PAHs may be generated from incomplete combustion or pyrolysis of organic materials, smoked or grilled food, vehicle exhaust and cigarettes [1]. PAHs may also be emitted from natural activities, such as forest fire, volcanoes and hydrothermal processes [2]. Some PAHs undergo metabolic activation to diol epoxides which may bind to DNA, resulting with errors in DNA replication and mutations that start the carcinogenic process in mammals [3]. EPA method 8310 determines 16 PAHs including acenaphthene, acenaphthylene, anthracene, benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[g,h,i]perylene, chrysene, dibenzo[a,h]anthracene, fluoranthene, fluorene, indeno[1,2,3-c,d]pyrene, naphthalene, phenanthrene and pyrene [4], among which benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, chrysene, dibenzo[a,h]anthracene, and indeno[1,2,3-c,d]pyrene are probable human carcinogens. EPA method 8310 is a performance based hazardous waste test method (SW-846) regulated under the Resource Conservation and Recovery Act (RCRA), which allows analysts modify sampling and analytical approaches flexibly to meet the measurement requirements [5].

This application note outlines a solid phase extraction (SPE) procedure using C18 SPE universal cartridges to extract 16 PAHs in water. The target PAHs are retained on the C18 sorbent, and are later eluted with acetone and dichloromethane (DCM). Sodium sulfate drying cartridges are used to remove any residual water in the SPE eluates. The dried eluates are concentrated, exchanged to acetonitrile, and analyzed by HPLC with ultraviolet (UV) or fluorescence detectors (FLD).

ENVIRO

SPE Procedure:

1. **Cartridge Conditioning**
   a) Place the glass adapters (**ECUCTADP**) on the 6-station manifold (**ECUCTVAC6**), attach the SPE cartridges (**ECUNIPAH**) to the glass adapters, then the bottle holders (**ECUNIBHD**) to the top of the SPE cartridges.
   b) Add 10 mL DCM to the SPE cartridges by rinsing the bottle holders and the SPE cartridges, let DCM wet and soak the SPE sorbent for 1 min before drawing to waste. Leave full vacuum on for 1 min.
   c) Add 10 mL methanol (MeOH) to the SPE cartridges, let MeOH wet and soak the SPE sorbent for 2 min, then pull MeOH through (or by gravity) leaving a thin layer above the frit.
   d) Add 20 mL reagent water to the SPE cartridges, pull water through leaving a water layer of about 1 cm above the frit.

2. **Sample Extraction**
   a) Dechlorinate the samples with 50 mg/L sodium sulfite if free chlorine present.
   b) Add surrogate and target analyte spiking solutions prepared in water miscible solvents, such as MeOH.
   c) Add 5 mL MeOH to the sample bottles and mix well.
   d) Load the sample bottles to the bottle holders, adjust vacuum for a fast dropwise flow (about 30 mL/min).

3. **Cartridge Drying**
   a) Dry the SPE cartridges under full vacuum for 10 min. Move the cartridges from the manifold during drying, and shake the cartridges to remove excess water from the bottom of the SPE cartridges.

4. **Analyte Elution**
   a) Insert glass vials (40 or 50 mL) into the SPE manifold to collect the SPE eluates.
   b) Rinse the sample bottles with 5 mL of acetone, add the rinsates to the SPE cartridges, let elution solvents wet and soak the sorbent for 1 min before drawing slowly to the collection vials. Leave full vacuum on for 1 min.
   c) Repeat the above step with 2 x 10 mL DCM.

5. **Eluate Drying**
   a) Remove the SPE cartridges and bottle holders from the glass adapters, and remove the collection vials from the manifold.
   b) Place the drying cartridges (**ECUNIMSS**) on the glass adapters, rinse with 10 mL of DCM.
   c) Insert new glass vials into the manifold, pass the eluates through the drying cartridges and collect.
   d) Rinse the collection vials with 10 mL DCM, pass the rinsates through and collect.

6. **Eluate Evaporation**
   a) Add 2 mL acetonitrile to the dried extracts and evaporate to 0.7 - 0.9 mL using TurboVap under a gentle stream of nitrogen (7 - 8 psi) at 40°C.
   b) Add internal standard and adjust the final volume to 1 mL using a small amount of acetonitrile.
   c) Transfer the concentrated extracts to 2-mL autosampler vials, and analyze by HPLC-UV or HPLC-FLD.

*Note: UCT's ENVIRO-CLEAN® Universal Cartridges can be used on Horizon SPE-DEX® 4790 Automated Extraction System directly, or on J.T. Baker® BAKERBOND Speedisk™ Expanded Extraction Station with the use of universal cartridge adapters (**ECBMADP**) and bottle holders (**ECUNIBHD**).*

ENVIRO

Results:

Average Recoveries of 4 Laboratory Fortified Blanks (LFB)
Compared to the QC Acceptance Criteria of EPA Method 8310

Compound Name	EPA Method 8310 Recovery QC*	Recovery% of UCT Method	
		LFB at 1 µg/L	LFB at 10 µg/L
Acenaphthene	D - 124	102.2	98.4
Acenaphthylene	D - 139	62.1	96.2
Anthracene	D - 126	92.3	98.3
Benz[a]anthracene	12 - 135	99.6	100.1
Benzo[a]pyrene	D - 128	100.2	89.8
Benzo[b]fluoranthene	6 - 150	87.4	106.7
Benzo[ghi]perylene	D - 116	104.0	89.3
Benzo[k]fluoranthene	D - 159	99.1	85.2
Chrysene	D - 199	123.5	82.3
Dibenz[a,h]anthracene	D - 110	102.5	90.3
Fluoranthene	14 - 123	114.7	101.8
Fluorene	D - 142	102.4	100.8
Indeno[1,2,3-cd]pyrene	D - 116	81.0	101.7
Naphthalene	D - 122	113.4	99.7
Phenanthrene	D - 155	109.1	96.3
Pyrene	D - 140	104.3	99.3

*: adopted from Table 3 in EPA method 8310

Conclusion:

A simple and efficient SPE method has been demonstrated for the extraction of 16 PAHs in water by EPA method 8310, a performance based method allowing for modifications with the QC acceptance criteria met. Excellent recoveries and RSD% (< 10%, n = 4) have been obtained using this SPE method, all parameters have passed the QC limits required by EPA method 8310, offering environmental testing labs a successful alternate extraction method that is more selective, consumes less organic solvents, and yields no emulsion which is a bottleneck when using liquid-liquid extraction for real world samples.

References:

[1] Polycyclic Aromatic Hydrocarbons in Food, Scientific Opinion of the Panel on Contaminants in the Food Chain, EFSA Journal 724 (2008) 1.
[2] D.L. Poster, M.M. Schantz, L.C. Sander, S.A. Wise, Anal. Bioanal. Chem. 386 (2006) 859.
[3] International Agency for Research on Cancer, Polycyclic Aromatic Compounds. Part I. Chemicals, Environment and Experimental Data, IARC Monographs on the Evaluation of Carcinogen Risk of Chemicals to Humans, 32 (1983) 453.
[4] https://www.epa.gov/sites/production/files/2015-12/documents/8310.pdf
[5]https://waste.zendesk.com/hc/en-us/articles/217452058-How-does-EPA-s-Performance-Based-Measurement-System-PBMS-approach-affect-SW-846-

UCT, LLC • 2731 Bartram Road • Bristol, PA 19007 • 800.385.3153 • 215.781.9255 •
www.unitedchem.com • Email: methods@unitedchem.com

ENVIRO

UCT