

Polynuclear Aromatic Hydrocarbons In Water By Automated Solid Phase Extraction and HPLC Analysis



Introduction

Produced as the byproducts of fuel combustion, polynuclear aromatic hydrocarbons (PAHs) consist of fused aromatic rings. As pollutants, they are of concern because some polynuclear compounds have been identified as carcinogenic, mutagenic, and teratogenic. PAHs are lipophilic and therefore found in the environment, primarily in soil, sediment, and oily substances. They also appear in surface and ground water, indicating a source of pollution.

Instrumentation and Consumables

- FMS, Inc. TurboTrace™ SPE system (solid phase extraction)
- FMS, Inc. SuperVap™ Concentrator
- FMS, Inc. 1 gram PAH C₁₈ cartridge
- Waters Alliance 2695 HPLC, UV254

Method Summary

TurboTrace SPE

1. Condition cartridge with 15 mL dichloromethane
2. Condition cartridge with 15 mL methanol
3. Condition cartridge with 35 mL water
4. Load 1 liter of water sample
5. Dry cartridge for 20 minutes
6. Rinse sample bottle with 35 mL dichloromethane
7. Elute sample from cartridge with bottle rinse
8. Elute sample with additional 25 mL dichloromethane

SuperVap Concentrator

1. Pre-heat temp: 60 °C
2. Pre-heat time: 20 minutes
3. Heat in Sensor mode: 60 °C
4. Nitrogen pressure: 9 PSI

Procedure

Three, one liter samples were acidified to pH 2 with 6N hydrochloric acid and spiked with 5 µL of Restek 31458 MA EPH aromatic hydrocarbon solution. Samples were then loaded onto the FMS TurboTrace SPE system using vacuum adjusted to -10 psi and passed across an FMS PAH C₁₈ cartridge. After sample loading, the cartridges were automatically dried using a stream of nitrogen until no residual water remained. The cartridges were subsequently eluted using dichloromethane from both the automatic sample bottle rinse and from the elution solvent reservoir. The extracts were concentrated to 1.0 mL and 5 mL acetonitrile was added for solvent exchange. The sample was concentrated to a final volume of 1 mL for HPLC analysis.



Figure 1: TurboTrace SPE and SuperVap Concentrator systems.



Results

Compound	Amount Spiked µg/L	Mean Recovery µg/L	SD	Percent Recovery
Naphthalene	5.00	3.57	0.67	71%
2-methylnaphthalene	5.00	3.95	0.70	79%
Acenaphthylene	5.00	3.71	0.78	74%
Acenaphthene	5.00	4.08	0.71	82%
Fluorene	5.00	4.19	0.59	84%
Phenanthrene	5.00	4.71	0.44	94%
Anthracene	5.00	4.79	0.41	96%
Fluoranthene	5.00	5.22	0.33	104%
Pyrene	5.00	5.30	0.31	106%
Benzo(a)anthracene	5.00	5.34	0.35	107%
Chrysene	5.00	5.36	0.35	107%
Benzo(b)fluoranthene	5.00	5.35	0.38	107%
Benzo(k)fluoranthene	5.00	5.36	0.29	107%
Benzo(a)pyrene	5.00	5.35	0.37	107%
Indeno(1,2,3-cd)pyrene	5.00	5.22	0.41	104%
Dibenzo(a,h)anthracene	5.00	5.31	0.42	106%
Benzo(g,h,i)perylene	5.00	5.26	0.40	105%

Conclusions

The FMS TurboTrace SPE system delivers consistent reliable, reproducible results for polynuclear aromatic hydrocarbons in water. The FMS PAH SPE cartridge can achieve high recovery extraction in water. Results reported from the TurboTrace™ SPE and SuperVap™ Direct-to-Vial Concentration system (Table 1) demonstrate high levels of precision and accuracy for the parallel extraction and concentration of PAHs using the FMS integrated sample-to-vial systems. With expanded modules, the TurboTrace™ SPE system enables labs to simultaneously extract eight samples hands free. Compared to manual extraction

processes, this automated technique frees up chemists to focus on increasing sample throughput. The addition of direct-to-GC vial concentration vessels eliminates the need to manually transfer extract from the concentration tubes to the vials which reduces operator error.

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