Application Note

Polynuclear Aromatic Hydrocarbons In Water By Semi-Automated Solid Phase Extraction using the FMS EZSpe® and HPLC Analysis via EPA Method 550.1

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 EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Waters Alliance 2695 HPLC, UV254
- FMS, Inc. 1 g PAH C18 cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Fisher 6 N Hydrochloric Acid
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Dichloromethane
- Restek 31458 MA EPH aromatic hydrocarbon standards

Procedure

- 6 samples (1L water each) are prepared and acidified with 2 mL HCl till pH ~ 2
- Spike with MA EPH aromatic hydrocarbon standards
- Put sample bottles in place and fill rinse bottles with 12 mL dichloromethane
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 4 x10 mL dichloromethane (drain), 4 x10 mL methanol (drain) and 2 x 10 mL water (keep wet)
- Samples are loaded across cartridges under vacuum
- Cartridges are flushed with 10 mL of water and then dried under vacuum for 10 min (no nitrogen)
- Sample bottles are automatically rinsed from the rinse bottles with 12 mL dichloromethane

Stage 2:

- Dichloromethane from sample bottles is loaded across the cartridges (2 x 6 mL, 1 min soak) and the eluent is collected for analysis into Direct to GC Vial Collection Vessels
- Extracts are dried over sodium sulfate or in line cartridges can be used downstream from C18 cartridges

FMS SuperVap®

- Pre-heat temp: 60 ºC
- Pre-heat time: 20 minutes
- Heat in Sensor mode at 60 ºC under nitrogen (9 psi)
- Direct to LC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

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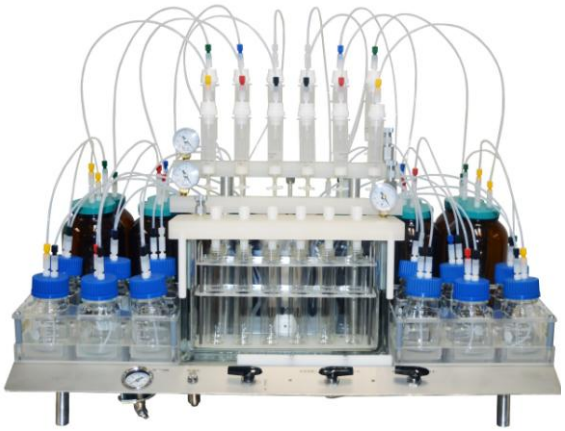
Table 1 with recoveries for a number of 550.1 PAHs

Compound	%Recovery	Stdev
Naphthalene	71	0.67
2-methylnaphthalene	79	0.70
Acenaphtylene	74	0.78
Acenaphthene	82	0.71
Fluorene	84	0.59
Phenanthrene	94	0.44
Anthracene	96	0.41
Fluoranthene	104	0.33
Pyrene	106	0.31
Benzo(a)anthracene	107	0.35
Chrysene	107	0.35
Benzo(b)fluoranthene	107	0.38
Benzo(k)fluoranthene	107	0.29
Benzo(a)pyrene	107	0.37
Indeno(1,2,3-cd)pyrene	104	0.41
Dibenzo(a,h)anthracene	106	0.42
Benzo(g,h,i)perylene	105	0.40

Conclusions

The FMS EZSpe® 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 supported from the EZSpe® 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. Compared to manual extraction processes, this semi-automated technique frees up chemists to focus on increasing sample throughput. The addition of direct-to-LC vial concentration vessels eliminates the need to manually transfer extract from the concentration tubes to the vials, which reduces operator error.



FMS EZSpe® system

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