

Polycyclic Aromatic Hydrocarbons in Drinking Water by Solid Phase Extraction (SPE) with EPA 550.1



Introduction

PAHs are hazardous compounds found in petroleum and emissions from fossil fuel consumption. Physically, PAHs are neutral, non-polar organic molecules that comprise two or more aromatic rings arranged in various configurations. Members of this class of compounds have been identified as exhibiting toxic and hazardous properties, and for this reason the U.S. Environmental Protection Agency has included 16 PAHs on its list of priority pollutants to monitor in water and waste. The procedure here described follows EPA method 550.1.

Analytically, PAHs present challenges for testing labs. Their volatile nature can make them problematic to extract without loss using manual methods. This is especially true for the concentration of sample extracts.

The following procedure utilizes the FMS TurboTrace® SPE extraction system in combination with the SuperVap® concentrator (Figure 1) to extract PAHs from drinking water.

Instrumentation

- FMS, Inc. TurboTrace SPE (Solid Phase Extraction) System
- FMS, Inc. SuperVap Concentrator
- FMS, direct-to-vial concentrator tubes
- Waters Alliance 2695 HPLC, UV254

Consumables

- FMS Inc PAH 1 g cartridge
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Methylene Chloride
- Fisher Ultrapure DI Water
- Fisher Anhydrous Sodium Sulfate

Procedure

TurboTrace SPE

1. Cartridges conditioned with 4x 10 mL methylene chloride
2. Cartridges conditioned with 4 x 10 mL methanol
3. Cartridges conditioned with 2 x 10 mL water
4. Samples loaded across cartridges at ~75mL/min
5. Sample bottles rinsed with H₂O; Rinse loaded across cartridges.
6. Cartridges dried under vacuum for 10 minute each.
7. Cartridges eluted with 2 x 5 mL Methylene Chloride.
8. Dry extracts by loading across sodium sulfate columns and rinsing each columns with 2 mL methylene chloride

SuperVap

1. Preheat temp: 20 minutes at 30 °C
2. Sensor mode / temp: 30 °C
3. Nitrogen Pressure: 7-10 PSI

Analysis

- 1 Analyze samples with HPLC-UV



Figure 1: TurboTrace SPE system.



Results

Table 1: Results for five, 1 liter replicates spiked at 50 ppb.

| Compound | Mean Rec. | STD DEV |
|-------------------------|-----------|---------|
| Naphthalene | 86.6% | 4.6% |
| 2-Methylnaphthalene | 88.2% | 3.3% |
| 1-Methylnaphthalene | 83.4% | 4.3% |
| Acenaphthylene | 81.7% | 4.8% |
| Acenaphthene | 92.3% | 6.8% |
| Fluorene | 79.4% | 5.1% |
| Phenanthrene | 87.0% | 3.0% |
| Anthracene | 81.4% | 2.8% |
| Fluoranthene | 93.5% | 2.4% |
| Pyrene | 100.8% | 3.2% |
| Benzo[a]anthracene | 88.3% | 5.1% |
| Chrysene | 93.6% | 6.6% |
| Benzo[b]fluoranthene | 85.5% | 7.0% |
| Benzo[k]fluoranthene | 91.8% | 3.9% |
| Benzo[a]pyrene | 90.5% | 5.1% |
| Indeno[1,2,3-cd]pyrene | 88.9% | 4.0% |
| Dibenzo[a,h]anthracene | 92.3% | 5.2% |
| Benzo[g,h,i]perylene | 88.1% | 3.7% |
| Nitrobenzene-D5 (Surr) | 93.6% | 6.1% |
| 2-Fluorobiphenyl (Surr) | 96.4% | 4.9% |
| p-Terphenyl-d14 (surr) | 81.7% | 5.2% |

Conclusions

Results reported from the TurboTrace SPE and SuperVap Direct-to-Vial Concentration system (Table 1) show 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 the lab to simultaneously extract full batches of sample hands free. Compared to manual extraction processes, this auto-mated technique frees up chemists to focus on increasing sample throughput. The addition of direct to GC vial concentration tubes eliminates the need to manually transfer extract from the concentration tubes to the vials and reduces operator error.

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