

Analysis of Polycyclic Aromatic Hydrocarbons in Polyurethane Foam (PUF) and Diethylene Glycol/Water Samples using Pressurized Liquid Extraction, Semi Automated Clean Up and Automated Solid Phase Extraction

Introduction

Polycyclic Aromatic Hydrocarbons (PAHs) are routinely measured in various environmental samples. This includes stack emissions of municipal waste incinerator facilities around the world. In this application note we describe methods to analyze PAHs that have been collected during sampling of incinerator stacks using Polyurethane Foam (PUF) and diethylene glycol /water mixtures (used to trap PAHs not collected on PUFs).

Instrumentation

- FMS, Inc. PLE®
- FMS, Inc. EZPrep®
- FMS, Inc. TurboTrace®
- FMS, Inc. SuperVap® 6 Concentrator
- FMS, Inc. SuperVap® 12 Concentrator
- FMS, Inc. 250 mL concentrator tubes (1 mL termination)
- Thermo Trace GC w/DSQ MS and AS3000 Autosampler

Consumables

- FMS, Inc. PLE Teflon end caps
- FMS, Inc. 6 g neutral silica column
- FMS, Inc. 1 g C18 cartridge
- Ottawa Sand®
- Fisher Dichloromethane pesticide grade
- Fisher Hexane pesticide grade
- Fisher Methanol pesticide grade
- Restek native PAHs spiking standard and deuterated PAHs internal standard

PLE

- Large PUF plug (ca 6 cm OD x 7 cm length) was spiked with native PAH standards
- Sample placed in 40 mL extraction cell and void space filled with Ottawa Sand
- Capped with disposable Teflon end caps
- Heated with 50% Dichloromethane/50% Hexane for 20 min at 100 °C and 1500 psi
- 20 min cool down
- Nitrogen flush to transfer analytes and extract to 250 mL collection tubes

SuperVap Concentration

- Pre-heat temperature: 45 °C
- Pre-heat time: 15 min
- Heat in Sensor mode: 45 °C
- Nitrogen Pressure: 6-8 psi
- Solvent exchange to hexane

EZPrep Clean Up

Stage 1:

- Assemble 6 g neutral silica columns with EZPrep set-up
- Syringe vial at top is used for conditioning and sample loading.
- Condition silica column with 20 mL hexane (vacuum, waste).

Stage 2:

- Dilute sample extract to 9 mL hexane.
- Load sample extract onto silica column
- Elute column with 10 mL hexane (waste)
- Purge aliphatic fraction line (if used) with 5 mL hexane
- Elute column with 35 mL dichloromethane, collecting aromatic fraction (PAHs).
- Purge aromatic fraction line (if used) with 5 mL Dichloromethane.



SuperVap step (above)

- Reduce sample to 1 mL final volume.

Solid Phase Extraction TurboTrace

- Acidify 10% Diethylene glycol/water samples with 6 N Hydrochloric Acid to pH < 2
- Add surrogates if so desired
- Load samples on automated system
- Install C-18 cartridges
- Fill rinse bottles with 35 mL dichloromethane
- Condition cartridges: 10 mL dichloromethane 20 mL methanol and 35 mL water (waste)
- Load samples under vacuum across Cartridges (waste)
- Dry with nitrogen gas for 3 min
- Rinse sample bottles with 35 mL dichloromethane and load across cartridges for elution and collect
- Elute 5 mL, another 5 mL, and another 2 mL of dichloromethane for soaking and elute and collect
- Blow out fraction lines with nitrogen and collect
- Dry samples over sodium sulfate

SuperVap Concentration

- Same as above but no solvent exchange needed. Reduce final volume to 1 mL dichloromethane and add internal deuterated PAHs standards prior to analysis.



Figure 1. Pressurized Liquid Extraction Apparatus.



Figure 2. EZPrep with 6 g neutral Silica columns for PAH clean up.



Recoveries in percent	PUF	SPE
naphthalene	65.4	85.6
acenaphthylene	72.8	108.1
acenaphthene	75.4	100.2
fluorene	81.0	106.7
phenanthrene	86.1	114.1
anthracene	82.4	113.6
fluroanthene	82.7	123.6
pyrene	85.1	127.0
chrysene	79.9	129.0
benzo[a]anthracene	75.2	127.0
benzo[b]fluoranthene	86.6	117.2
benzo[k]fluoranthene	76.4	109.9
benzo[a]pyrene	79.7	115.6
indeno[1,2,3-cd]pyrene	82.8	122.2
dibenzo[a,h]anthracene	85.0	130.0
benzo[g,h,i]perylene	83.8	111.5

Table 1. PAHs recoveries for PUF and for SPE samples in 10% diethylene glycol/water

Discussion

Results for PUF extraction and clean up and for Solid Phase Extraction of 10% diethylene glycol/water samples in given in Table 1. Excellent recoveries were obtained. The PUF method and SPE method are very suitable when analyzing PAHs collected on PUF and in diethylene glycol/water traps.



Figure 3. TurboTrace for SPE of diethylene glycol/water samples.

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