PROMOCHROM TECHNOLOGIES

A Robust and Simple Method for Solid Phase Extraction of Polycyclic Aromatic Hydrocarbons in Water

Summary

Polycyclic aromatic hydrocarbons (PAH) are one of the most widespread organic pollutants. Their analysis in water plays an important role for effective monitor and control of their pollution. Due to the requirement of very low detection limit (sub ppb to ppt level), extensive sample preparation is required before instrumental analysis using GC or HPLC. This application describes a reliable and simple methods for extraction of PAHs in water using automated solid phase extraction. Thanks to the highly inert flow path and user friendly design of SPE-03 automated SPE system, recoveries above 90% are achieved using a simple elution that involves only one solvent. The trapped analytes on SPE columns can be washed down using only 2.5 mL methanol, making the final evaporation faster or unnecessary.

Polycyclic aromatic hydrocarbons (PAH) are one of the most widespread organic pollutants. Due to the requirement of very low detection limit (sub ppb to ppt level), extensive sample preparation is required before instrumental analysis. Conventional procedures for sample preparation involves extracting 500-2000 mL of water sample using solid phase extraction, concentrating the extracted sample from 5-10 mL to 0.5-1 mL, and analyzing concentrated samples using GC or HPLC.

Due to the extremely low concentration in water sample and a very strong hydrophobicity of PAHs, great care is required in selection of solvents for SPE column activation and for elution of trapped PAHs on the SPE columns. The inertness of the flow path in the SPE apparatus and the SPE columns is also critical to avoid adsorption loss of the analytes.

SPE-03 multi-channel SPE system uses PromoChrom's flow path integration techniques. The system has a much simpler and shorter flow path than other automated SPE instruments, and thus the adsorption and retention of analytes are minimized. Based on its SPE-03 SPE system and the specially deactivated SPE column, a simple and reliable method for PAH extraction has been developed.

Materials and methods

1. Instrument. SEP-03 multi channel SPE system

2. SPE column. PromoChrom deactivated C18 SPE column, 500 mg/6 mL, part number 18-050-6C.

3. Elution solvents. Methanol, ethyl acetate, reagent grade.

4. Procedures for PAH extraction.

Use methanol as solvent 1, water as solvent 2, and ethyl acetate as solvent 3. Run a system clean method as below:

No.	Action	Flow rate (mL/min)	Volume (mL)	Remarks
0	Clean 2	20	4.0	Clean the sample lines using water
1	Clean 1	20	4.0	Clean the sample lines using methanol
2	Elute 3	20	1.0	Elute column using ethyl acetate
3	Collect 1	20	5.0	Clean the tubing to fraction 1 with ethyl acetate
4	Collect 2	20	5.0	Clean the tubing to fraction 2 with ethyl acetate

The system clean procedure takes around 10 minutes.

Place the sample tubing into water samples with volume 500-1000 mL. If the collected fraction is not to be further evaporated, a 5-mL graduated test tube may be used for the collection. The exact volume can be used for final quantitation. Run the following method for PAH extraction:

No.	Action	Flow rate (mL/min)	Volume (mL)	Remarks
0	Elute 1	10	10.0	Activate and clean SPE column with methanol
1	Add samp	10	500.0	Load sample to SPE column
2	Elute 1	10	0.2	Purge SPE column with small volume methanol
3	Blow air	20	20.0	Dry SPE column with air
4	Elute 1*	10	0.2	Change elution solvent for collection to methanol
5	Collect 1	10	2.5	Collect 2.5 mL fraction

*If the final analysis is by GC and further evaporation is necessary, use Elute 3 for ethyl acetate.

The procedure for extraction of 1000 mL water will take around 2.5 hours.

5. Instrumental analysis.

The collected fraction may be analyzed directly using GC or HPLC without further evaporation. If the method

detection limit requirement cannot be satisfied, volume of collected fraction can be reduced by blowing nitrogen. A graduated centrifuge tube is very convenient for the evaporation and final volume adjustment.

Results and discussion

PAHs are highly hydrophobic compounds. They tend to be adsorbed by the surface of the flow path and SPE columns. At ppb/ppt level, the adsorption loss can have very significant effect on the final results. SPE-03 adopts flow path integration techniques that simplifies and shorten flow path significantly. It helps to reduce the adsorption to hydrophobic analytes.

The strong adsorption of SPE column to PAH is another cause of low recovery of PAHs. To avoid the problem, a elution involves several solvent are normally required. In this application, the C18 SPE column is specially deactivated to allow fast elution of PAHs from the column sorbent. The elution volume for fraction collection is only 2.5 mL and it requires only one simple solvent. The recovery of PAHs are above 95%. In other PAH methods, the fraction volume is normally 10 mL and requires several solvents. The reduced fraction volume makes the fraction evaporation faster or unnecessary.

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