



SPE Application Note for PAHs from Water

This method was developed for the extraction of polycyclic aromatic hydrocarbons (PAHs) from natural waters with a high concentration of humic acids, which can be problematic during analysis.

EXTRACTION PROCEDURE

ISOLUTE® SPE Column: ISOLUTE PAH 1.5 g/6 mL Part # 927-0150-C

Pre-treatment: To a 1L water sample, add 10% isopropanol (v/v), and mix thoroughly. If an internal standard is to be used, add at this stage.

Solvation: Solvate the column with isopropanol (5 mL) at a flow rate of 5 mL/min.

Equilibration: Equilibrate the column with deionized water containing 2% (v/v) isopropanol (5 mL) at a flow rate of 5 mL/min.

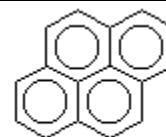
Sample application: Load the sample (1 L) at a flow rate of 20 mL/min. Rinse the sample bottle with acetone (10 mL) dilute this to 100 mL with deionized water, and apply to the extraction column. Repeat this step if necessary to prevent losses of the analytes to the walls of the sample bottle.

Interference elution: Rinse the column with 90:10 (v/v) deionized water:isopropanol (5 mL). Dry the column thoroughly by vacuum aspiration for 15-20 mins (use -20"Hg vacuum). If losses of the more volatile PAHs are found at this stage, consider drying the column by centrifugation.

Analyte elution: Elute analytes with 2 x 3 mL THF/hexane (50:50, v/v). Use a soak step of up to 2 min between aliquots to maximize recoveries of the analytes.

Evaporate the extract at low temperature under nitrogen to concentrate further if required to improve detection limit. Care should be taken at this stage to prevent losses of the more volatile analytes, and the extract should NOT be taken to dryness. A keeper solvent may be of use in this situation.

Structure Various. Pyrene is shown as an example.



**Structural considerations**

These compounds are extremely non-polar and the smaller PAHs can be extremely volatile. These properties of the analytes can lead to problems during extraction and analysis. Losses of the analytes can occur during loading of the sample, as analytes can remain on the walls of the sample container, and during concentration of the sample during evaporation.

Matrix considerations

Natural waters (e.g. river water) often contain humic and fulvic substances which can be co-extracted with the analytes. This leads to interferences during subsequent analysis steps. The column used in this procedure minimizes the co-elution of humic substances by utilising a layer of aminopropyl sorbent on which they are retained.

Analytical method GC, HPLC**Reagents****General comments**

1. The method described in this application note can be directly transferred to the glass column (927-0150-L) if the sample is loaded at 10 mL/min
2. Dichloromethane is a suitable alternative elution solvent. The THF/hexane elution solvent was developed and optimized in order to replace dichloromethane as necessary.

ISOLUTE column part numbers represent the product configuration of choice for use with a vacuum sample processing station. For 96-well and alternative column configurations compatible with any SPE automation system, please contact Biotage.

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