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Automatic extraction and detection of PAHs in drinking water with SPDE-GC/MS

Introduction

PAHs (Polyaromatic hydrocarbons) form a class of diverse organic compounds each containing two or more fused aromatic rings of carbon and hydrogen atoms.

Most PAHs enter the environment via the atmosphere from a variety of combustion processes and pyrolysis sources. Owing to their low solubility and high affinity for particulate matter, they are not usually found in water in notable concentrations. The main source of PAH contamination in drinking water is usually the coal-tar coating of drinking water distribution pipes, used to protect the pipes from corrosion. Where there are elevated levels of contamination by coal-tar coatings of water pipes, PAH intake from drinking water could be equal to or even exceed that from food (grilling, roasting, frying or baking).

Evidence that mixtures of PAHs are carcinogenic to humans comes primarily from occupational studies of workers following inhalation and dermal exposure.

For the detection of PAHs in drinking water the Analytische Qualitätssicherung in Germany requires a method where the lower working level is below 0.005 μ g/L (5 ppt)[1].

Due to the combination of fully automated CHROMTECH SPDE-Option followed by mass spectrometric detection (SPDE-GC/MS) the determination of PAHs in drinking water down the low ppt range is possible in routine analysis without any "offline" sample extraction step.

In this application note we report the suitability of SPDE-GC/MS for the analysis of PAH spiked drinking water.

Methods

GC-Conditions: Agilent 6890N. column: Varian VF-5MS, 30 m x 0.25 mm x 0.25 µm. 40°C (3 min), 10° C/min \Rightarrow 320°C (5 min). s/slinjector (250°C), 1 min splitless, 30 mL/min purge flow. Helium (1.2 mL/min).

MS-Conditions: KODIAK 1500 with GC Interface (250° C). EI at -70 eV, 150 µA, 200° C. Scan 40-400 with 600 ms/Scan.

SPDE-Conditions: CHROMTECH SMM (Single Magnet Mixer) on CTC CombiPAL. 10 mL Sample in 20 mL headspace vial; temperature (syringe and oven): 35° C; 750 rpm agitator speed; 40 mm sample penetration; 90 extraction strokes à 1 ml sample; 0.5 mL desorption gas volume with 100 µL/s flow speed; 60 s pre-desorption time. Needle cleaning 5 x 2 mL water and at elevated temperature.

Results and Discussion

Figure 1 illustrates the principle of operation for SPDE. By this method the PAHs of the sample could be enriched inside the SPDE needle and thereafter injected directly into the split-/splitless injector of the GC/MS. The enrichment process needs about the same time as the gc analysis. Therefore it is possible to establish a continuous extraction /analysis process.

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Fig. 1: Principle of the automated SPDE extraction. The analyte is enriched by controlled pumping through the specially coated syringe needle.

Composition of PAH-standard:

Compound	Extracted ion	Ret. time
Acenaphthylene	152.1	15.25
Fluorene	166.1	16.98
Phenanthrene	178.1	19.16
Anthracene	178.1	19.28
Pyrene	202.1	22.52
Chrysen e	228.0	25.49
Benzo[b]fluoranthene	252.1	27.80
Benzo[k]fluoranthene	252.1	27.81
Benzo[a]pyrene	252.2	28.46
Indeno[1,2,3-cd]pyrene	276.2	30.57
Dibenzo[a,h]anthracene	278.2	30.63
Benzo[g,h,i]perylene	276.1	31.03

Figure 2 - **4** show very clearly some examples of PAHs at a concentration level of 10 ppt. The GC/MS data were obtained in scan mode to allow a quick identification of the compounds by library search. To maximize of sensitivity the use of SIM or quadrupole MS/MS may be useful.

The presented results show very clearly that identification and quantitation of PAHs is possible in drinking water by means of SPDE-GC/MS even in the low ppt range. The SPDE technique is characterized by complete automation and high sensitivity. Compared to other extraction procedures only a small quantity of sample (5-10 mL water) and no to-xic solvent are needed. The system is based on the **CTC CombiPAL** sampler, so no further laboratory space is needed. Due to the above



Fig. 2: 10 ppt Pyrene, Benzo[a]anthracen and Chrysene.



Fig. 3: 10 ppt Indeno[1,2,3-cd]pyrene and Benzo [g,h,i]-perylene.



Fig. 4: 10 ppt Benzo[b]fluoranthene, Benzo [k]fluoranthene and Benzo[a]pyrene

mentioned advantages **SPDE** could be the method of choice for the routine analysis of PAHs in water.

Literature: AQS-Ringversuch RV 2/04-Trinkwasser PAK - TW O3 (9.2.2004)