Determination of polycyclic aromatic hydrocarbons in water

Syncore® Analyst R-12 with Flushback module and SPE advanced module, Vacuum Pump V-700, Vacuum Controller V-855, Recirculating Chiller F-108: Determination of PAH traces in waste water

Metabolites of polycyclic aromatic hydrocarbons (PAH) have been shown to act as potent carcinogens, mutagens and teratogens. Thus, the determination of PAH is of great importance. A simple and fast method for their determination in waste water is introduced below. The sample is eluted with dichloromethane (DCM) directly into the Syncore® Analyst sample tube after being extracted over a solid phase extraction (SPE) cartridge. Subsequently, the DCM is evaporated to a residual volume for GC-MS analysis. The combined extraction-evaporation sample preparation method proceeds without cross-contamination.

1. Introduction

PAH are molecules based on aromatic rings containing only carbon and hydrogen atoms. PAH are natural components of fossil fuels; they are released to the environment during incomplete combustion of organic matter. [1]

Once emitted, PAH adsorb on soot (nano)particles and eventually enter the food chain. Next to the oral ingestion, PAH enter the body *via* the respiratory tract and the dermal tissue. It has been shown that oxidized PAH (epoxides) can act as carcinogens, mutagens and/or teratogens interfering with the DNA and altering the genetic makeup. Following health risks might be the consequence: [2]

- impaired fertility and fetal/reproductive damage
- infant developmental defects
- carcinogenic effects (benzo[*a*]pyrene (Figure 1) verified; other PAH in category 2 (probably))
- weakening of the immune system and disorder of the central nervous system
- skin damage (through defatting)

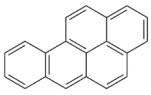

Figure 1: Chemical structure of benzo[a]pyrene

Therefore, authorities enforced many regulations to avoid as much as possible the use and the emission of PAH. In the 80s, the United States Environmental Protection Agency (US EPA) defined 16 PAH to be representative for the laboratory analysis of the over hundreds of components (Table 1).

Some examples of Swiss regulatory limits for PAH in water are:

- drinking water: 0.2 µg/kg (or 0.2 µg/L) sum of (*) in Table 1 [3]
- groundwater intended for drinking water after application of simple regeneration process: 0.1 µg/L per EPA PAH component [4]

Table 1: 16 priority PAH according to US EPA

Acenaphthene(°)	Chrysene
Acenaphthylene(°)	Dibenz[*a*,*h*]anthracene
Anthracene	Fluoranthene(*)
Benz[*a*]anthracene	Fluorene(°)
Benzo[*a*]pyrene(*)	Indeno[1,2,3-*cd*]pyrene(*)
Benzo[*b*]fluoranthene(*)	Naphthalene(°)
Benzo[*ghi*]perylene(*)	Phenanthrene
Benzo[*k*]fluoranthene(*)	Pyrene

2. Experimental

Equipment: Syncore® Analyst R-12 with Flushback module and SPE advanced module / Vacuum Pump V-700 / Vacuum Controller V-855 / Recirculating Chiller F-108 / phenomenex® SPE cartridge: strata® PAH, 1.5 g/6 mL.

Sample: PAH standard (Sigma Aldrich®, 10 µg/mL each component in acetonitrile, certified reference material).

Determination: 1.2 mL of the sample was diluted to 100 mL with a water/acetonitrile solution (75/25 v/v, HPLC grade) and transferred to the conditioned (DCM/ethanol/water) cartridge with the aid of a tube connected to an adapter. After washing (methanol/water solution, 50/50 v/v) and drying the cartridge, the PAH were eluted with DCM directly into the Syncore® Analyst sample tube. The elution was concentrated under vacuum for GC-MS analysis (Supelco®, EPA 525, 525.1 Internal Standard Mix, 500 µg/mL each component in acetone).

Pure DCM was poured directly in a fresh Syncore® Analyst sample tube and concentrated parallel to the PAH sample to check for cross-contamination during evaporation.

3. Results

Excellent PAH recoveries were obtained (*e.g.*, 93.7 % for benzo[*a*]pyrene (Figure 1)), except for the low boilers ((°) in Table 1). No PAH residues were found in the (pure) DCM reference, meaning that no cross-contamination of samples occurred.

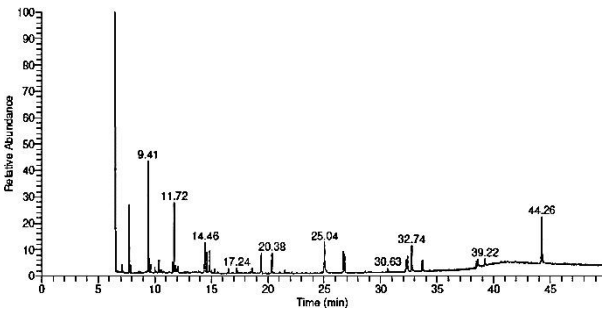

Figure 2: GC chromatogram of the PAH standard sample

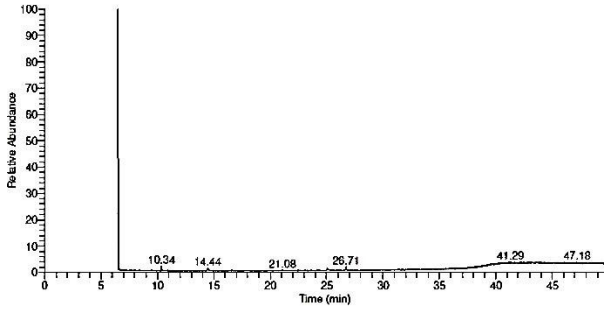

Figure 3: GC chromatogram of the DCM reference sample

4. Conclusion

The Syncore® Analyst with the SPE advanced module using standard SPE cartridges provides a simple and fast method of sample preparation for the PAH determination in water. Very high recoveries were found for most compounds. An optimization of the SPE method with lower heating temperature and longer drying time is expected to lead to better results for the low boilers as well.

Analogically, with appropriate cartridges, also samples containing polychlorinated biphenyls (PCBs) can easily be prepared for GC/LC-MS analysis.

Thus, the Syncore® Analyst is a versatile and economic product for the parallel sample preparation of water samples containing several contaminants.

5. Acknowledgment

Labor Veritas AG (Zurich, Switzerland) is kindly acknowledged for the analytical PAH determination.

6. References

[1] Bundesamt für Gesundheit (BAG): PAK Factsheet
[2] Bundesamt für Bauten und Logistik (BBL): Weisung Schadstoffvorkommen in zivilen Bundesbauten – Anhang I – Vorgehen und Grundlagen
[3] Gewässerschutzverordnung (GSchV 814.201)
[4] Fremd- und Inhaltsstoffverordnung (FIV 817.021.23)