GLOBAL ANALYTICAL SOLUTIONS

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An Automated Sorptive Extraction-Thermal Desorption GC-MS System for the Analysis of Aqueous Samples

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Introduction

An alternative system for the enrichment of aqueous samples is presented based on the extraction of the analytes into a sorption phase followed by thermal desorption and GC-MS analysis.

Analytes are partitioned into the extraction phase and retained in the bulk of the sorption material.

This is in sharp contrast with most alternative extraction materials where analytes are adsorbed on an active surface (e.g. SPE).

Advantages of this new analytical approach are:

- Sorption mechanism (dissolution) instead of adsorption
- Highly inert retaining phase (similar to GC column)
- Retention properties well-known from Gas Chromatography
- Thermally stable
- Degradation products can easily be recognized with the use of MS
- Sorption isotherms are linear up to high concentration levels
- Desorption occurs thermally, ensuring transfer of all sample analytes onto the column
  --> very high sensitivity !

In practice two sorption materials are applied:

- Commercially available polydimethylsiloxane (PDMS) tubes
- Experimental polyacrylate tubes which have an improved performance for polar solutes

EXPERIMENTAL

The experimental setup consists of:

- a thermal desorption unit (TDS-2, Gerstel, Mülheim an der Ruhr, Germany)
- a cryotrap (CIS-4, Gerstel)
- a TDS on-line rack autosampler (Gerstel)
- a GC/MS system (6890/5972 GC/MSD, Hewlett Packard, Little Falls, DE, USA)

A schematic view of the set-up is given below:

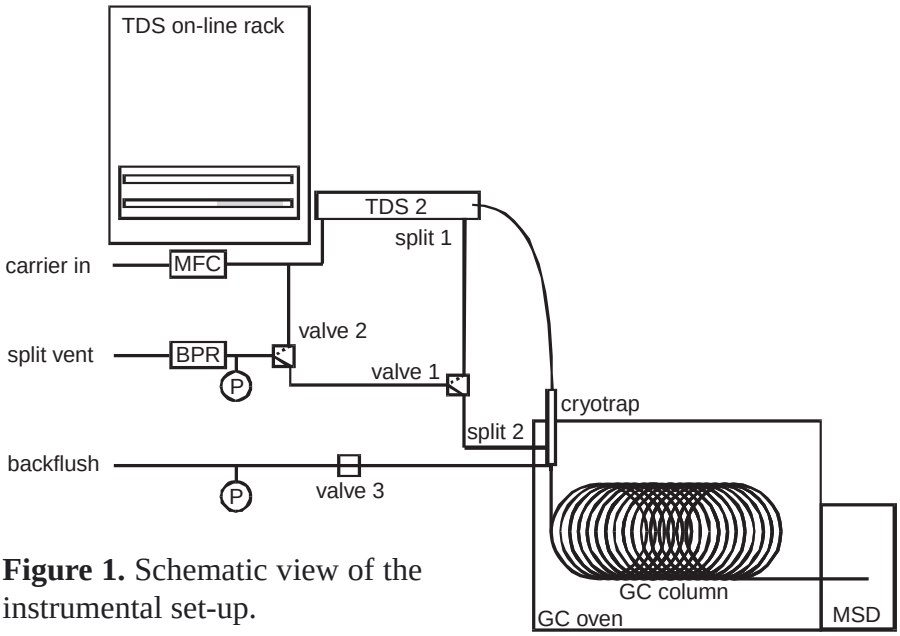


Figure 1. Schematic view of the instrumental set-up.

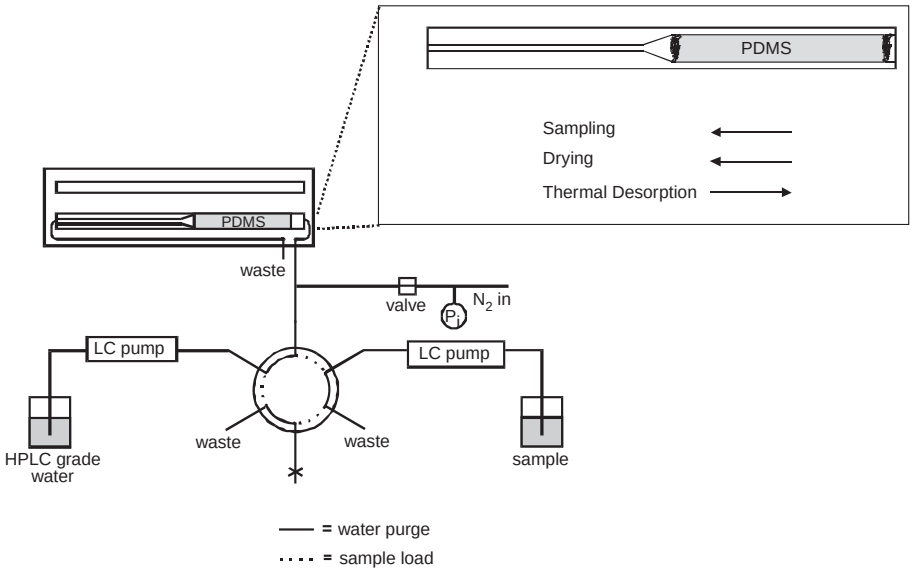


Figure 2. System used for loading the liquid samples onto the PDMS-traps.

RESULTS WATER-TD-GC

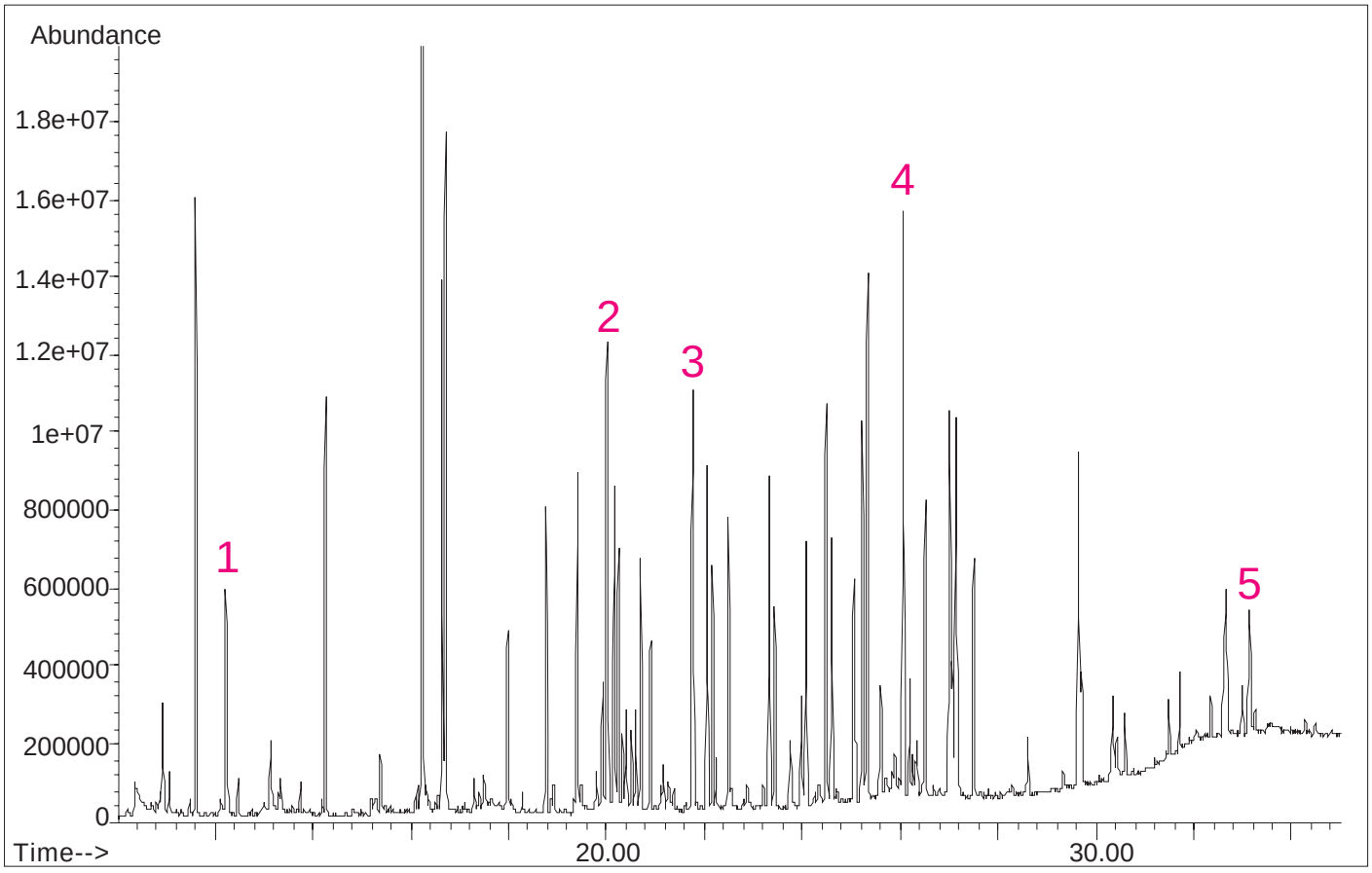
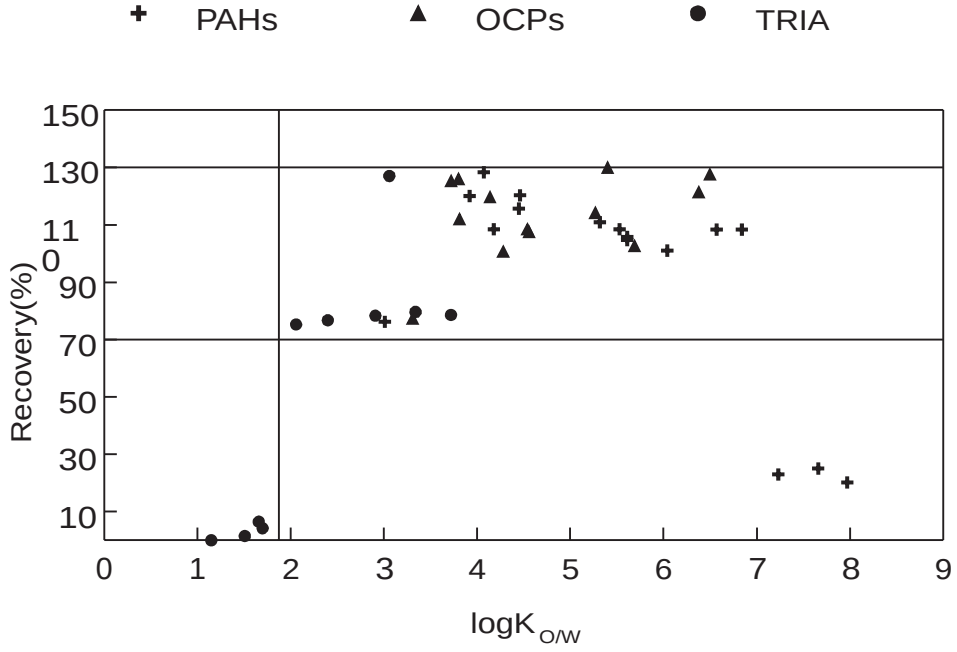


Figure 3. Determination of organochlorine pesticides(18), PAHs(16) and triazines(13) in tap water (spiked, ca. 1 µg/l).

Sample volume: 10 ml (sampled at 1 ml/min)
Desorption: Thermal 225°C (5 min)
Detection: Mass Selective Detector (SCAN 40-400 amu, 2.2 scans/s)



loss of polars: lack of retention
loss of apolars: system adsorbtion
theory: $\log K_{O/W}=1.8$

Figure 4. Recovery vs $\log K_{O/W}$

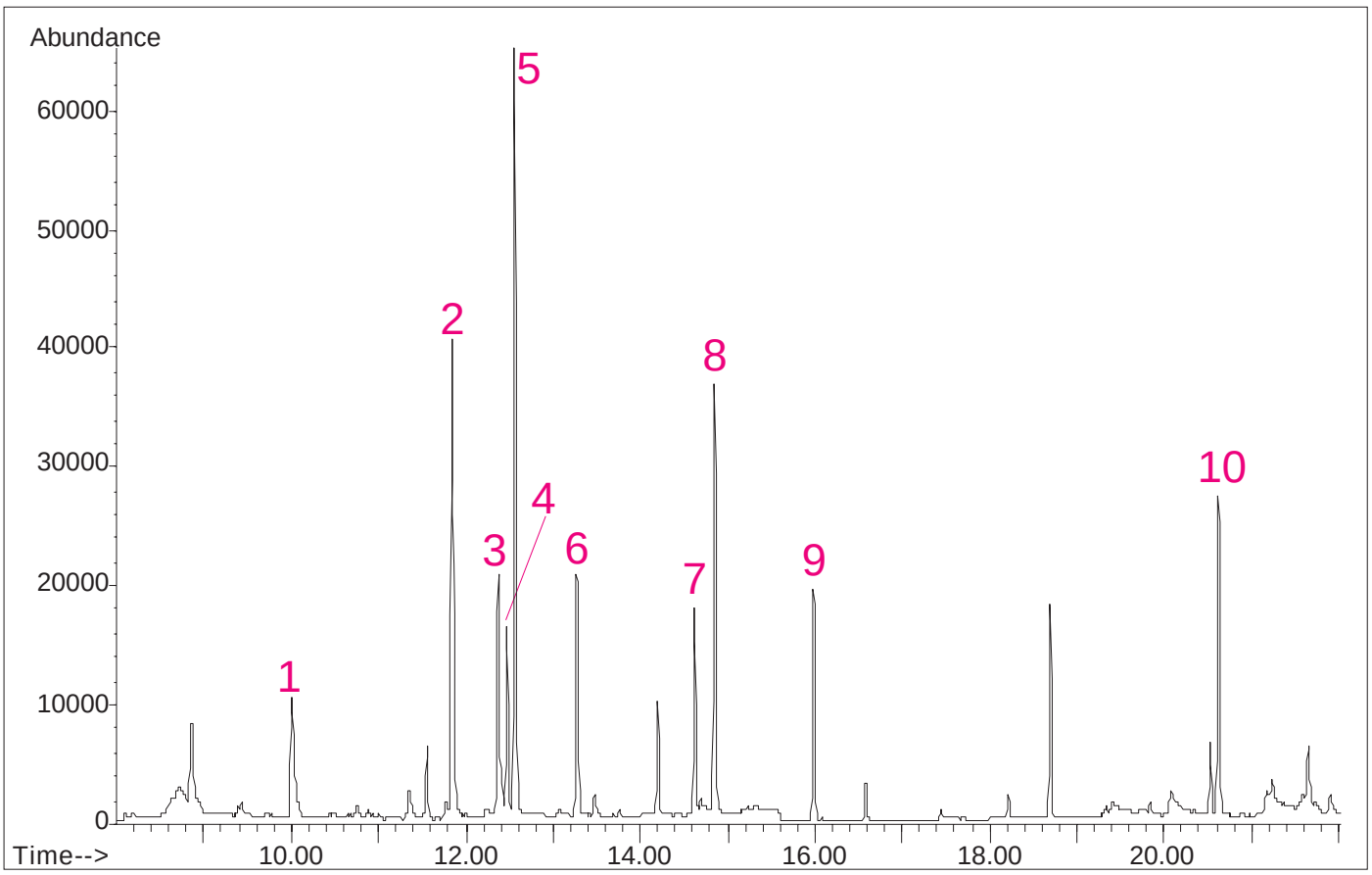


Figure 5. Determination of phenols in tap water (spiked at 0.1 mg/l, acetylated with acetic anhydride).

List of compounds:

1 Phenol
2 4-Methylphenol
3 2-Chlorophenol
4 2,6-Dimethylphenol
5 2-Ethylphenol
6 p-Isopropylphenol
7 2,4-Dichlorophenol
8 2,3,5-Trimethylphenol
9 2,4,6-Trichlorophenol
10 Pentachlorophenol

Sample volume: 10 ml (sampled at 1 ml/min)
Desorption: Thermal 225°C (5 min)
Detection: Mass Selective Detector (SIM 2 ions/component)

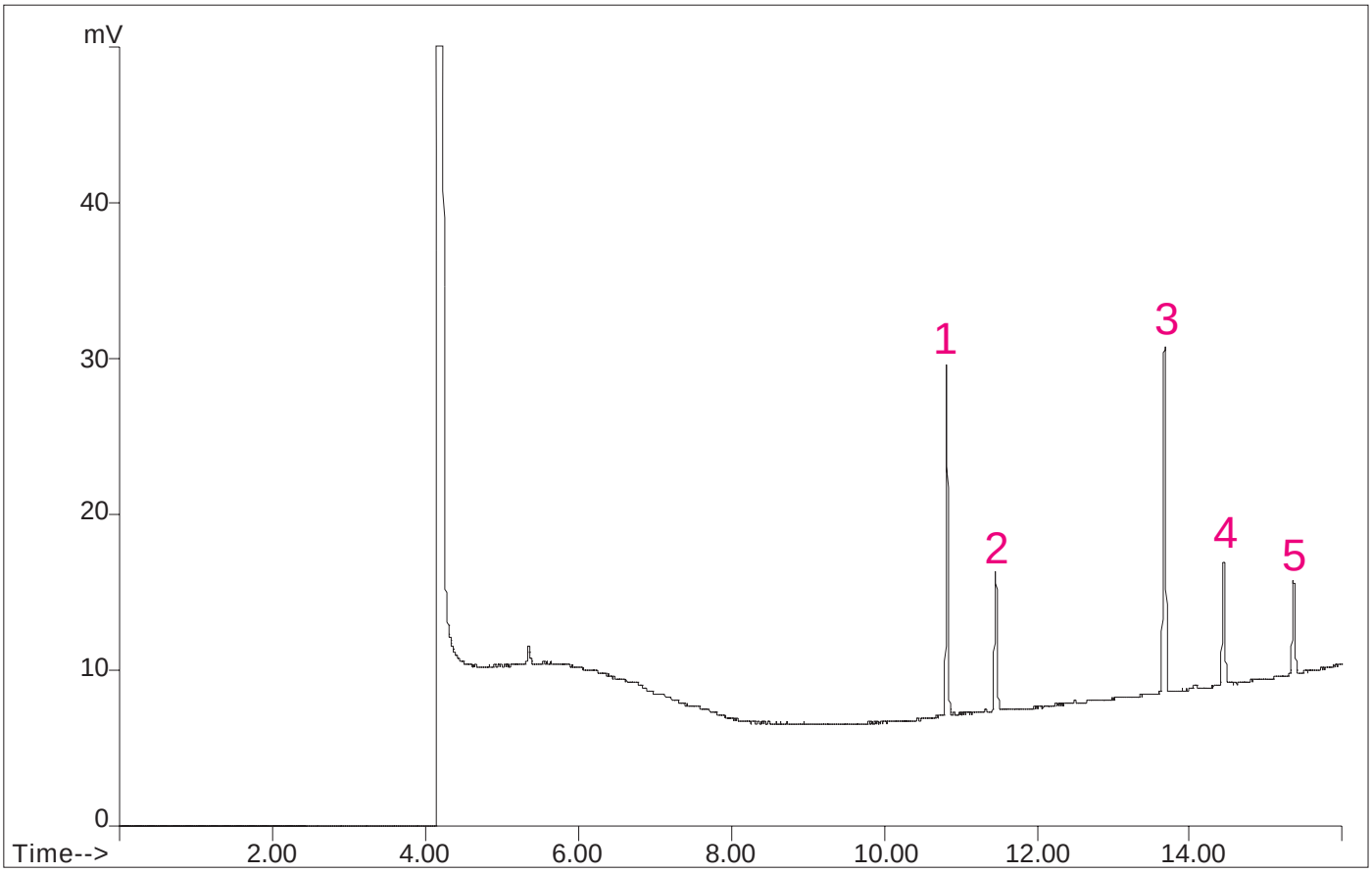


Figure 6. Determination of amines in water (spiked at 1-2 mg/l, derivatized with pentafluorobenzoylchloride).

List of compounds:

1 Dimethylamine
2 Ethylamine
3 Dipropylamine
4 Pentylamine
5 Hexylamine

Sample volume: 1 ml (sampled at 0.5 ml/min)
Desorption: Thermal 225°C (5 min)
Detection: Nitrogen Phosphorous Detector

CONCLUSIONS

- Sorptive enrichment was shown to perform well for the analysis of PAHs, pesticides, phenols and amines from water.
- PDMS degradation products do not interfere with the analysis of the target analytes.
- Derivatization of analytes widely extends application range.
- The system described here allowed fully automated, unattended water enrichment and analysis.
- Repeatability and recoveries were good at the 0.1 ppb concentration level.
- Using 10 mL samples and mass selective detection, detection limits were in the 1-10 ng/L range.

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