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APPLICATION NOTE

Solid Phase Microextraction (SPME) followed by HAPSITE ER Detection and Identification of Semi-Volatile Polychlorinated Hydrocarbons in an Aqueous Mixture

INTRODUCTION

SPME Sampling System was designed to provide HAPSITE® ER with the added capability of detecting and identifying SVOCs. The HAPSITE ER SPME Sampling System, when attached to the HAPSITE ER via the universal interface, provides a means to introduce SVOCs into HAPSITE ER. An aqueous mixture of SVOCs, consisting of polycyclic aromatic hydrocarbons (PAHs), polychlorinated aromatic hydrocarbons (PCAHs) and polychlorinated hydrocarbons (PCHs) were sampled and introduced into the HAPSITE ER via the SPME Sampling System. PAHs found in oil, coal, and tar deposits are released into the environment as by-products of fuel burning. PCAHs are manufactured for use as fungicides or fungicide precursors and are sprayed on crop seeds. Both classes of compounds are considered carcinogenic to humans and animals. This application note describes a procedure developed for SPME extraction and HAPSITE ER identification of PAHs, PCAHs, and PCHs from a prepared aqueous solution (see Table 1).

EXPERIMENTAL

A 20 mL aliquot of distilled water was added to a 40 mL VOA vial containing a stir bar. 5.0 grams of sodium chloride was then added to the vial to produce a 25% w/v NaCl solution. The added salt was used to maximize the recovery of the SVOCs in the aqueous solution. The vial was then sealed with a PTFE septum which served to create a 20 mL headspace above the water sample. Using a syringe, the compound sample mix was injected through the septum into the salt water solution. The resulting solution concentration was 50 ng/mL (50 ppb) per analyte. A PDMS (red) fiber, attached to a SPME fiber holder, was conditioned in the SPME Sampling System prior to sampling, using a default conditioning method specifically for that fiber type. The fiber holder was then inserted through the PTFE septum of the sample vial and the fiber was exposed and immersed in the water sample for 10 minutes. Following exposure, the SPME fiber was retracted and the holder was removed from the vial. The fiber holder was then brought to the HAPSITE ER and inserted in the SPME Sampling System for analysis. The SPME fiber was exposed inside the 250 °C desorption chamber housed in the Sampling System. A 10 minute 40 second sample separation and analysis run was carried out with the mass spectrometer scanning from 43 to 300 amu at a rate of 1.0 scan/sec. Method conditions and the resulting chromatogram are shown in Figure 1.

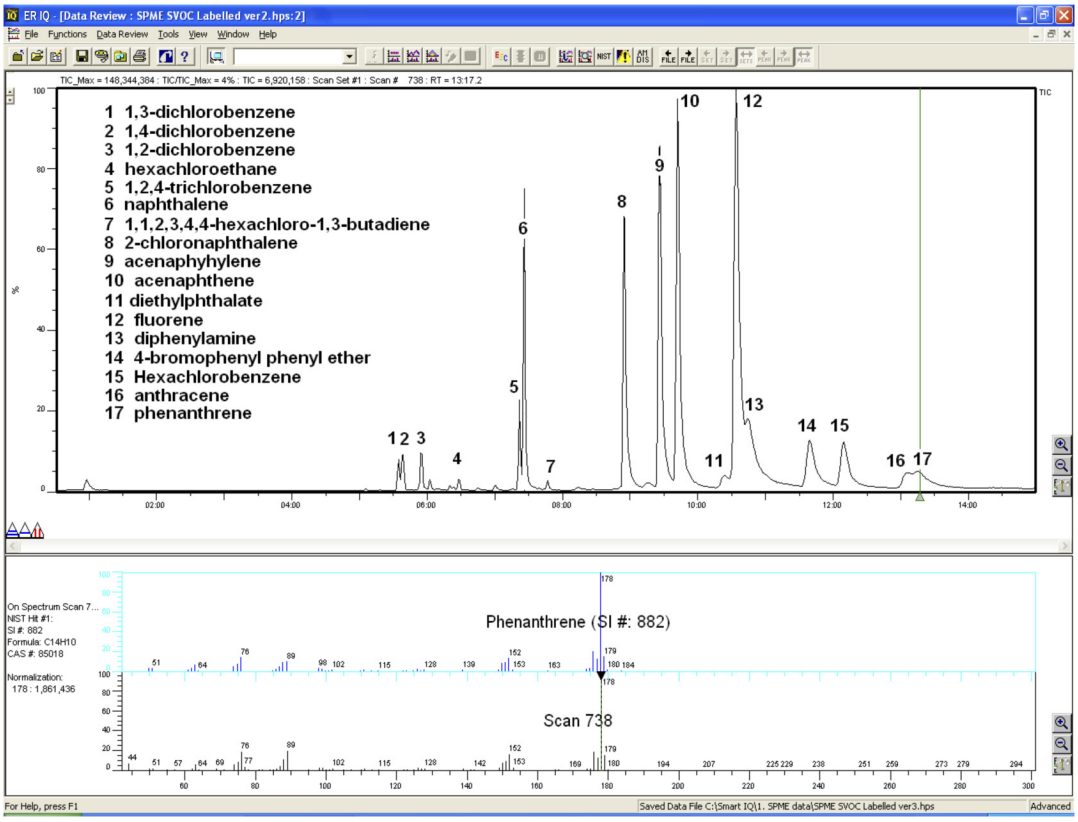
RESULTS AND SUMMARY

This study demonstrates the increased versatility of HAPSITE ER when used in conjunction with the SPME Sampling System, providing the user with the ability to sample, detect and identify SVOCs.

Table 1: PAHs, PCHs, and PCHs in Study

	Analyte Name	CAS Number	Retention Time		Analyte Name	CAS Number	Retention Time
1	1,3-dichlorobenzene	541-73-1	05:34.6	10	acenaphthene	83-32-9	09:42.0
2	1,4-dichlorobenzene	106-46-7	05:38.7	11	diethyl phthalate	84-66-2	10:23.6
3	1,2-dichlorobenzene	95-50-1	05:54.3	12	fluorene	86-73-7	10:34.0
4	hexachloroethane	67-72-1	06:27.6	13	diphenylamine	122-39-4	10:44.4
5	1,2,4-trichlorobenzene	120-82-1	07:21.7	14	4-bromophenyl phenyl ether	101-55-3	11:39.4
6	naphthalene	91-20-3	07:25.8	15	hexachlorobenzene	118-74-1	12:09.5
7	hexachloro-1,3-butadiene	87-68-3	07:46.6	16	anthracene	120-12-7	13:05.7
8	2-chloronaphthalene	91-58-7	08:54.1	17	phenanthrene	85-01-8	13:15.1
9	acenaphthylene	208-96-8	09:26.4				

Figure 1: Total Ion Chromatogram (TIC) of a Mixture of PAHs, PCAHs, and PCHs



Column: Rtx-1MS (15 m x .25 mm x 1.0 µm)
Column temperature program: 60 °C held for 2.0 minutes, ramp at 16 °C/min. to 120 °C, ramp at 24 °C/min. to 200 °C, hold for 5 minutes 55 seconds

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