

APPLICATION NOTE

Solid Phase Microextraction (SPME) and HAPSITE ER: Detection and Identification of Volatile Organic Compounds (VOCs) in the Headspace Above a Water Sample

SUMMARY

HAPSITE® ER is a rugged, person-portable GC/MS instrument for the detection of volatile and semi-volatile organic compounds (VOCs and SVOCs) in air, water, and soil. The HAPSITE ER SPME Sampling System, designed for sampling and detection of SVOCs, can also be used for direct aqueous or headspace sampling of VOCs, thereby providing the user with an additional tool complementing INFICON Headspace and SituProbe sampling accessories. A variety of VOCs found in the field, including chemical warfare agents, fumigants, water disinfection by-products (THMs), and chlorinated hydrocarbon solvents can easily be extracted and pre-concentrated from headspace samples using SPME techniques. The sample can then be introduced into HAPSITE ER via the SPME Sampling System for GC separation, followed by MS vapor detection and identification. This application note outlines a procedure for SPME headspace vapor extraction and HAPSITE ER detection of a 15-compound mixture (see Table 1) of disinfection by-products and chlorinated solvents.

EXPERIMENTAL

A 20 mL aliquot of distilled water was added to a 40 mL VOA vial containing a stir bar. A 5.0 gram sample of sodium chloride was then added to the water to produce a 25% w/v NaCl solution. The added salt was used to maximize the amount of VOCs in the headspace above the aqueous solution. The vial was then sealed with a PTFE septum held on with a screw cap which served to create a 20 mL headspace above the water. Using a syringe, the compound sample mix was

injected through the septum into the salt water solution. The resulting solution concentration was 200 ng/mL (200 ppb) per analyte. A Carboxen/PDMS (black) fiber, attached to a SPME fiber holder, was first conditioned in the SPME Sampling System prior to sampling using a default conditioning method specifically for that fiber type. The fiber holder needle, with retracted fiber, was then inserted through the PTFE septum of the sample vial and the fiber exposed for 10 minutes to the headspace above the continuously-stirred water sample solution. Following exposure, the SPME fiber was carefully retracted, removed from the vial, and brought to the HAPSITE ER for analysis. The fiber holder was inserted into the SPME Sampling System desorption chamber. The fiber was then exposed inside the 250 °C desorption chamber which is attached to the injection port of the HAPSITE ER. A 10 minute 40 second sample separation and analysis run was carried out with the mass spectrometer scanning from 45 to 300 amu at a rate of 1.0 scan/sec. Method conditions and the resulting chromatogram are shown in Figure 1.

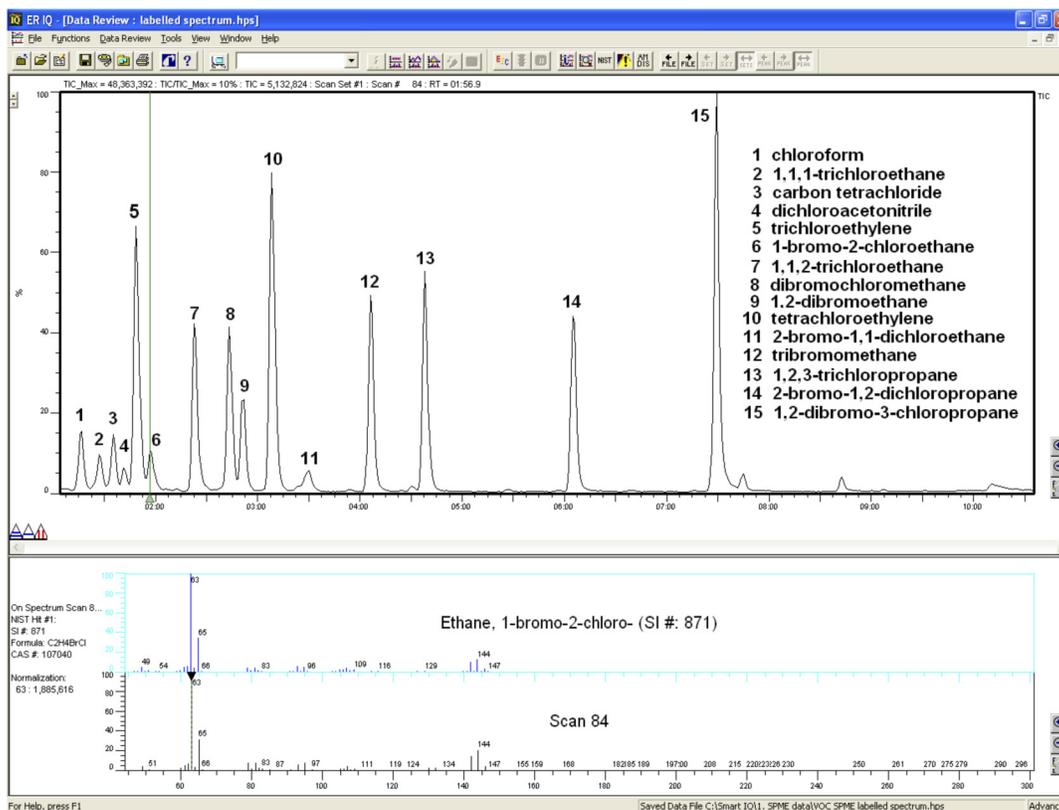
CONCLUSIONS

This study demonstrates the increased versatility of HAPSITE ER with the addition of the SPME Sampling System. VOCs in the headspace above an aqueous solution can now be extracted with a SPME fiber and introduced into the HAPSITE ER for analysis. The HAPSITE user now has yet another choice of VOC sampling methods that can be used in addition to purge and trap methods.

Table 1: Chlorinated Solvents and Disinfection By-products in Study

	Analyte Name	CAS Number	Retention Time		Analyte Name	CAS Number	Retention Time
1	chloroform	67-66-3	01:16.6	9	1,2-dibromoethane	106-93-4	02:51.7
2	1,1,1-trichloroethane	71-55-6	01:26.9	10	tetrachloroethylene	127-18-4	03:08.3
3	carbon tetrachloride	56-23-5	01:35.2	11	1,1,1-trichloro-2-propanone	918-00-3	03:28.9
4	dichloroacetonitrile	3018-12-0	01:41.1	12	bromoform	75-25-2	04:06.1
5	trichloroethylene	79-01-6	01:48.6	13	1,2,3-trichloropropane	96-18-4	04:38.1
6	1-bromo-2-chloroethane	107-04-0	01:56.9	14	2-bromo-1,2-dichloropropane	17759-88-5	06:04.7
7	1,1,2-trichloroethane	79-00-5	02:22.7	15	1,2-dibromo-3-chloropropane	96-12-8	07:29.4
8	dibromochloromethane	124-48-1	02:43.3				

Figure 1: Total Ion Chromatogram (TIC) of Chlorinated Solvents and Disinfection Byproducts



Column: Rtx-1MS (15 m x .25 mm x 1.0 μ m)
 Column temperature program: 60 °C held for 1.0 minute, ramp at 6 °C/min. to 80 °C,
 ramp at 12 °C/min. to 120 °C, ramp at 20 °C to 180 °C.



www.inficon.com reachus@inficon.com

Due to our continuing program of product improvements, specifications are subject to change without notice. HAPSITE is a registered trademark of INFICON.

diae16a1-a ©2009 INFICON