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If you have questions about applying methodology described in this article to a current application, please contact our technical service chemists.

SUPELCO

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SPME/Capillary GC Analysis of Solvents from Water at Low ppb Levels

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The new Carboxen/polydimethylsiloxane SPME fiber retains small analytes, enabling detection at very low concentrations.

Extraction of small polar analytes from water samples is extremely difficult. Many commonly used extraction techniques are not suitable for polar compounds such as solvents. In liquid-liquid extraction and solid phase extraction, the solvents used in the process often are the same materials as the solvents being analyzed. With static and dynamic headspace extraction (purge and trap), some solvents of interest can be analyzed but, because of their solubility in water, they can be detected only to parts-per-million (ppm) concentrations.

Figure A. Solvents in Water at 20ppb

Sample:	solvents at 20ppb in 4mL water + 25% NaCl in 4mL vial
SPME Fiber:	**Carboxen/PDMS, 75µm film**
Cat. No.:	**57318**
Extraction:	direct immersion, 10 min, rapid stirring
Desorption:	3 min, 260°C
Column:	**SPB™-1 SULFUR, 30m x 0.32mm ID, 4.0µm film**
Cat. No.:	**24158**
Oven:	50°C (2 min) to 150°C at 10°C/min
Carrier:	helium, 30cm/sec
Injection:	splitless (closed 2 min), 260°C, 0.75mm ID liner
Detector:	FID

1. Methanol (solvent for dioxane)
2. Ethanol
3. Acetonitrile
4. Acetone
5. Isopropanol
6. n-Propanol
7. Ethyl acetate
8. 3-Methyl-2-butanone
9. 1,4-Dioxane

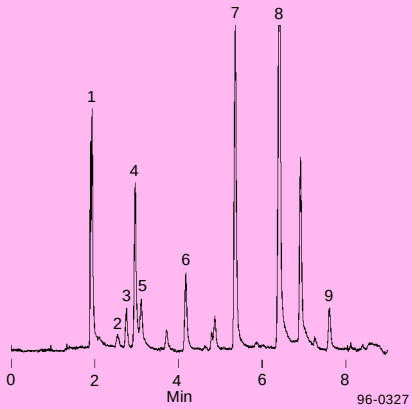

Table 1. Area Counts for Solvents at 500ppb, Using Various SPME Fibers

Compound	100µm PDMS	Carbowax/ DVB[1]	PDMS/ DVB[1]	Polyacrylate	Carboxen/ PDMS
Methanol	0	76	34	190	640
Ethanol	35	130	110	200	5300
Acetonitrile	140	140	170	230	6500
Acetone	410	250	640	260	98000
Isopropanol	180	250	610	260	53000
n-Propanol	220	450	1200	360	83000
Ethyl acetate	1600	4700	15000	1200	450000
3-Methyl-2-butanone	4000	13000	48000	2700	820000

[1] DVB = divinylbenzene.

Solid phase microextraction (SPME)* is a viable alternative to the traditional extraction techniques. SPME is fast, simple, and uses no solvents. SPME methods have been developed for analyzing a variety of samples and compounds with detection limits as low as parts-per-billion (ppb) concentrations.

We recently developed an SPME fiber with a Carboxen™/polydimethylsiloxane (PDMS) layer. This fiber is designed specifically for extraction of small, low molecular weight analytes at trace levels. Carboxen-1006** adsorbent is a highly porous carbon with a surface area of 1200m^2/g. The high porosity enables the fiber to strongly retain small analytes and provide a high sample capacity.

We used the Carboxen/PDMS fiber to extract solvents at 20ppb (Figure A). The fiber exhibited excellent extraction capability. Our sampling was by direct immersion for 10 minutes, but the extraction also can be accomplished using heated headspace at 50°C.

Compared with other SPME fibers, the Carboxen/PDMS fiber exhibits superior solvent recovery (Table 1). The small pores enable this fiber to extract analytes at higher orders of magnitude than other SPME fibers, including the more polar Carbowax®/ divinylbenzene and polyacrylate fibers.

Ordering Information:

Description	Cat. No.
SPME Fibers, 75µm Carboxen/PDMS, pk. of 3	
Manual sampling	**57318**
AutoSampler	**57319**
SPME Holder* **	
Manual sampling	**57330-U**
AutoSampler	**57331**
SPME Stand	
Holds eight 4mL vials, supports SPME syringe	**57333-U**
SPB-1 SULFUR Capillary Column	
30m x 0.32mm ID, 4.0µm film	**24158**

*Solid phase microextraction technology licensed exclusively to Supelco. US patent #5,691,206; European patent #0523092.

**US Pat. No. 4,839,331.

***Initially you must order both holder and fiber assembly. Holder is reusable indefinitely. Use Cat. No. 57331 with Varian 8100/8200 AutoSampler (requires Varian SPME upgrade kit, available from Varian), or with Supelco™ SPME/HPLC interface.

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