

TheReporter

Reprinted from Volume 16, No. 1, 1997

T297051

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SPME/GC Analyses of Sulfur Gases and VOCs, Using a New Carboxen/PDMS Fiber

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Sample extraction of sulfur compounds and VOCs by traditional methods can be difficult and expensive. Typically, sulfur analysis requires concentration of the gas using adsorbent tubes containing carbon adsorbents. Analysis by such means, however, requires the use of an expensive thermal desorption unit to heat the tubes before transferring the gases to a gas chromatograph. Analysis of VOCs likewise can be cumbersome.

Our new 75 μ m Carboxen™/polydimethylsiloxane (PDMS) solid phase microextraction (SPME) fiber* is specifically designed for extracting gases and other small and low molecular weight analytes, and effectively adsorbs very volatile compounds. This fiber is coated with Carboxen-1006** adsorbent, a porous carbon with a surface area of 1200m²/g, widely used in air sampling.

We used the new fiber to extract 1 ppm of a sulfur gas mixture and analyzed the sample by gas chromatography/mass spectrometry (Figure A). A sulfur-specific detector can easily detect the sulfur compounds at ppb levels.

Figure A. Sulfur Gases in Air at 1ppm

Sample: sulfur gases in air at 1ppm, 250mL bulb
 SPME Fiber: Carboxen/PDMS, 75 μ m film
 Cat. No.: 57318
 Extraction: headspace, 5 min, ambient temp.
 Desorption: 2 min, 250°C
 Column: Supel-Q™ PLOT, 30m x 0.32mm ID
 Cat. No.: 24242
 Oven: 45°C (0.75 min) to 250°C at 25°C/min
 Carrier: helium, 25cm/sec
 Injection: splitless (closed 2 min), 0.75mm ID liner
 Detector: GC/MS quadrupole, m/z = 32 - 125, 0.6sec/scan

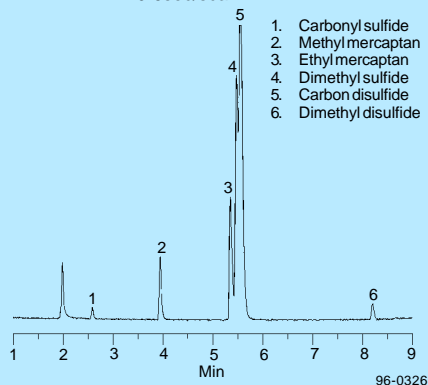


Table 1. Response Factors for Headspace Sampling of VOCs*

Component	Ion Scan	Carboxen/ PDMS	PDMS/ Divinylbenzene	100 μ m PDMS
Dichlorofluoromethane	85	3	0	0.0
Chloromethane	49	9	ND	ND
Vinyl chloride	62	60	1	0.1
Bromomethane	94	172	23	ND
Chloroethane	49	24	1	ND
Trifluoromethane	101	10	1	0.1
1,1-Dichloroethene	96	28	2	0.3
Methylene chloride	84	118	8	0.6
Chloroform	117	48	11	0.7
Bromochloromethane	130	127	162	0.9
Carbon tetrachloride	117	8	5	0.6
Benzene	78	23	10	0.6
Bromodichloromethane	83	28	18	0.9
Toluene	91	7	9	0.9
1,1,2-Trichloroethane	97	33	26	0.9
Tetrachloroethylene	166	8	6	1.0
Dibromochloromethane	129	11	21	0.9
Ethylbenzene	91	5	6	1
Bromoform	173	17	26	2
1,3-Dichlorobenzene	146	3	5	2

*Responses relative to direct immersion response using a 100 μ m PDMS fiber.

Analyte Concentration: 5ppb; Detection: ion trap MS.

ND = not detected.

The Carboxen/PDMS fiber also can be used to concentrate highly volatile compounds, such as those currently regulated in drinking water and wastewater. We extracted a spiked water sample of US EPA Method 524 VOCs at 0.5ppb. The Carboxen/PDMS fiber yielded a higher response for the VOCs than other fiber types (Table 1). We used headspace sampling, rather than direct immersion, to eliminate possible complications caused by dirty samples and to reduce sampling time.

Although the data are not given here, the extraction of polar solvents from a spiked water sample demonstrates the ability of the Carboxen/PDMS fiber to remove low levels of hard-to-extract alcohols and carbonyl compounds from a water matrix. These compounds are representative of many flavor compounds used in food products.

Other possible applications yet to be developed using the Carboxen/PDMS fiber include characterization of soil gas composition, monitoring hydrocarbon streams, and analysis of off-gas products from petroleum-based products.

Ordering Information:

Description	Cat. No.
SPME Fibers, 75 μ m 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
Supel-Q™ PLOT Column 30m x 0.32mm ID	24242

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Fused silica columns manufactured under HP US Pat. No. 4,293,415.

*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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