Solid Phase Microextraction: Theory and Optimization of Conditions

Solid phase microextraction, a simple, effective adsorption/desorption technique, eliminates the need for solvents or complicated apparatus for concentrating volatile or nonvolatile compounds in liquid samples or headspace. SPME is compatible with analyte separation/detection by gas chromatography or HPLC, and provides linear results for wide concentrations of analytes. By controlling the polarity and thickness of the coating on the fiber, maintaining consistent sampling time, and adjusting several other extraction parameters, an analyst can ensure highly consistent, quantifiable results from low concentrations of analytes.

Key Words:
- solid phase microextraction • SPME
- sample preparation • sample extraction

Analyses of volatile or semivolatile organic environmental pollutants, flavor or fragrance components, and many other samples usually begin with concentrating the analytes of interest through liquid-liquid extraction, purge-and-trap, headspace, or various other techniques. These procedures typically require excessive time, complicated equipment, and/or extravagant use of organic solvents. Solid phase microextraction, or SPME,* an adsorption/desorption technique developed at the University of Waterloo (Ontario, Canada), eliminates the need for solvents or complicated apparatus for concentrating volatile or nonvolatile compounds in liquid samples or headspace. SPME provides linear results over wide concentrations of analytes (1-4), is compatible with any packed column or capillary gas chromatograph or gas chromatograph-mass spectrometer system, and can be used with split/splitless or direct/packed injectors. An SPME/HPLC interface allows the technique to be combined with analysis by HPLC, expanding the applications for the extraction technique to detection of surfactants in water, pharmaceuticals in biological fluids, and many other analyses.

In analyses of chlorinated pesticides, for example, the pesticides in environmental, food, or other samples usually are extracted by liquid-liquid extraction, which takes 4 to 18 hours, or with solid phase extraction cartridges or disks, which takes 1 to 2 hours total time and 20 to 45 minutes of handling time per sample. In addition, liquid-liquid and solid phase extraction procedures can carry contaminants into the final sample, along with the analytes of interest, producing a high background in the analysis. In contrast, SPME is faster (15 minutes) and much less labor intensive (about 3 minutes of handling time per sample) than liquid-liquid extraction or solid phase extraction, and requires only small amounts of sample and no organic solvents. SPME reduces interfering background in pesticide or other analyses, making analyte identification and quantification more reliable. Relative responses for a series of ten extractions of chlorinated pesticides from water show that SPME is very consistent in routine use (Table 1).

Figure A. Solid Phase Microextraction

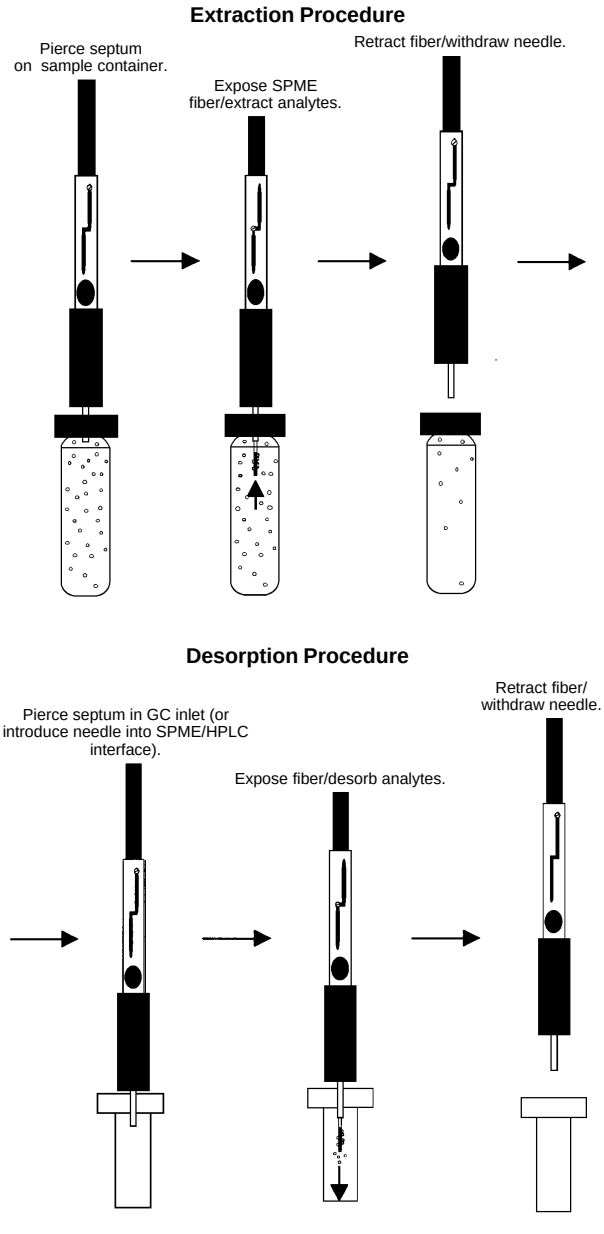

Table 1. Precise Relative Responses for Chlorinated Pesticides

Analyte	Relative Response (n = 10) Mean	Std. Dev.	% RSD
□-BHC	0.72	0.07	9.2
□-BHC	0.06	0.01	19.1
□-BHC	0.53	0.06	10.5
□-BHC	0.28	0.03	11.9
Heptachlor	1.01	0.10	10.2
Aldrin	1.25	0.06	4.8
Heptachlor epoxide	0.92	0.12	13.2
□-Chlordane	0.97	0.12	9.9
Endosulfan I	0.87	0.10	11.1
□-Chlordane	0.92	0.11	10.1
4,4'-DDE	0.92	0.07	8.1
Dieldrin	0.83	0.08	9.5
Endrin	0.68	0.06	9.1
Endosulfan II	0.72	0.09	13.0
4,4'-DDD	0.69	0.06	8.9
Endrin aldehyde	0.13	0.04	28.6
Endosulfan sulfate	0.54	0.06	11.9
4,4'-DDT	0.51	0.08	15.2
Endrin ketone	0.57	0.06	10.7
Methoxychlor	0.26	0.04	16.2

Sample: chlorinated pesticides in 4mL water, 50ppt each
SPME: 100µm polydimethylsiloxane-coated fiber, immersion sampling, 15 min
Column: poly[5% diphenyl/95% dimethylsiloxane] phase, 15m x 0.20mm ID, 0.20µm film
Oven: 120°C (1 min) to 180°C at 30°C/min, then to 290°C at 10°C/min
Carrier: helium, 37cm/sec, set at 120°C
Det.: ECD, 300°C
Inj.: splitless (closed 3 min), 260°C

How SPME Works

The solid phase microextraction process is shown in Figure A. A 1cm length of fused silica fiber, coated with a polymer, is bonded to a stainless steel plunger and installed in a holder that looks like a modified microliter syringe. The plunger moves the fused silica fiber into and out of a hollow needle. To use the unit, the analyst draws the fiber into the needle, passes the needle through the septum that seals the sample vial, and depresses the plunger, exposing the fiber to the sample or the headspace above the sample. Organic analytes adsorb to the coating on the fiber. After adsorption equilibrium is attained, usually in 2 to 30 minutes, the fiber is drawn into the needle, and the needle is withdrawn from the sample vial. Finally, the needle is introduced into the gas chromatograph injector, where the adsorbed analytes are thermally desorbed and delivered to the GC column, or into the SPME/HPLC interface. Results compare very favorably to results for other sample preparation methodology (Table 2).

In SPME, equilibria are established among the concentrations of an analyte in the sample, in the headspace above the sample, and in the polymer coating on the fused silica fiber. The amount of analyte adsorbed by the fiber depends on the thickness of the polymer coating and on the distribution constant for the analyte. Extraction time is determined by the length of time required to obtain precise extractions for the analytes with the highest distribution constants. The distribution constant generally increases with increasing molecular weight and boiling point of the analyte. Selectivity can be altered by changing the type of polymer coating on the fiber, or the coating thickness, to match the characteristics of the analytes of interest. In general, volatile compounds require a thick coating, and a thin coating is most effective for adsorbing/desorbing semivolatile analytes.

Table 2. SPME Compares Well with Other Sample Preparation Techniques

Detection Limit (MS)	Precision (% RSD)	Expense	Time	Solvent Use	Simplicity
Purge & Trap					
ppb	1-30	high	30 min	none	no
Stripping					
ppt	3-20	high	2 hr	none	no
Headspace					
ppm		low	30 min	none	yes
Liquid-Liquid Extraction					
ppt	5-50	high	1 hr	1000mL	yes
Solid Phase Extraction					
ppt	7-15	medium	30 min	to 100mL	yes
SPME					
ppt	**<1-12**	**low**	**5 min**	**none**	**yes**

Table provided by J. Pawliszyn, University of Waterloo, Waterloo, Ontario, Canada.

For liquid polymeric SPME coatings, the amount of analyte adsorbed by the coating at equilibrium is directly related to the concentration of the analyte in the sample (5):

$$n = \frac{K_{fs}V_fC_0V_s}{K_{fs}V_f + V_s}$$

where n = mass of analyte adsorbed by coating
C_0 = initial concentration of analyte in sample
K_{fs} = partition coefficient for analyte between coating and sample matrix
V_f = volume of coating
V_s = volume of sample

This equation shows the relationship between the initial concentration of analyte in the sample and the amount of analyte adsorbed by the coating is linear.

Because the coatings used in SPME are selected to have strong affinities for the organic compounds they are intended to extract, K_{fs} values for these analytes are large. Consequently, SPME has a very effective concentrating effect and leads to good sensitivity. K_{fs} values usually are not sufficiently large to exhaustively extract the analyte from the matrix, however; hence the statement that SPME is an equilibrium sampling method. Through proper calibration, SPME can be used to accurately determine the concentrations of analytes of interest in a sample.

The equation also shows that if V_s is very large, the amount of analyte extracted by the fiber coating is not related to sample volume. This makes SPME ideally suited for field sampling and analysis. Because the fiber can provide accurate information on target analytes simply by exposing it to air or dipping it into a lake, river, or well, SPME can significantly reduce field analysis time by combining sampling, extraction, concentration, and injection into a single process.** The kinetics of SPME are discussed in more detail in references 1 and 5.

In addition to changing the coating type or thickness, an analyst can improve analyte recovery, or alter selectivity in favor of more volatile or less volatile compounds, by changing analytical conditions: adding an electrolyte to the sample, changing the pH, or sampling the headspace rather than the sample (or vice versa). These changes will be discussed in more detail later in this bulletin.

Sampling with SPME

For high accuracy and precision from SPME, consistency in sampling time and other sampling parameters is more important than full equilibration. It also is important to keep constant the vial size and the sample volume and, when sampling by immersion, the depth to which the fiber is immersed in the sample. (An adjustable needle guide/depth gauge on the fiber holder helps ensure consistent depth of immersion of the fiber.) Because immersion and headspace sampling methods differ in kinetics, the two approaches have been considered complementary (6). For a given sampling time, Yang and Peppard (6) found immersion SPME was more sensitive than headspace SPME for analytes predominantly present in the liquid. The reverse was true for analytes that were primarily in the headspace. Increasing the sample volume from 200µL to 3mL, while keeping the ratio of liquid to headspace constant (1:1), increased analyte adsorption by either immersion or headspace SPME (6). For higher sensitivity from headspace SPME, the sample headspace should be as small as is practical (7). Equilibrium is attained more rapidly in headspace SPME than in immersion SPME, because the analytes can diffuse more rapidly to the coating on the fiber. These characteristics can be manipulated to advantage to selectively adsorb sample components, as appropriate. Headspace SPME is ideal for minimizing interferences with an analysis, and can prolong the lifetime of the SPME fiber. For example, Alan Harmon of McCormick & Co. used SPME coupled with capillary GC to solve a particularly difficult analytical problem – analyzing punch flavor in the presence of glycerin. Headspace SPME eliminated the glycerin peak and revealed 13 additional flavor components that had been obscured in direct split injection (8).

Desorption of an analyte from an SPME fiber depends on the boiling point of the analyte, the thickness of the coating on the fiber, and the temperature of the injection port. Cryogenic cooling sometimes is required to focus slowly desorbed compounds at the inlet of the capillary column. Alternatively, an inlet liner with a narrow internal diameter (e.g., 0.75mm ID, compared to conventional 2mm ID liners) sharpens the peaks and often can eliminate the need for cooling (Figure B). In HPLC applications, the SPME/HPLC interface enables an analyst to introduce the analyte-bearing fiber into a moving stream of mobile phase (dynamic desorption), or to allow more strongly bound analytes to be desorbed from the fiber in a standing volume of mobile phase (static desorption).

SPME can be used to rapidly screen samples or, when used under consistent extraction conditions and with internal standards, can be a reliable part of a formal, quantitative analyses. The combination of extraction by SPME and analysis on a short, narrow bore capillary column, such as the 10m x 0.20mm ID x 1.20µm columns used to obtain the data in Table 3, can greatly increase the number of samples processed in a day. The number of extractions that can be performed with a single SPME fiber assembly is governed by the care given the assembly and the nature of the components in the samples being analyzed. Under most conditions, an assembly can provide 50 to 100 extractions.

Figure B. A Reduced Inlet Liner Volume Improves SPME/GC Analyses of Volatile Analytes

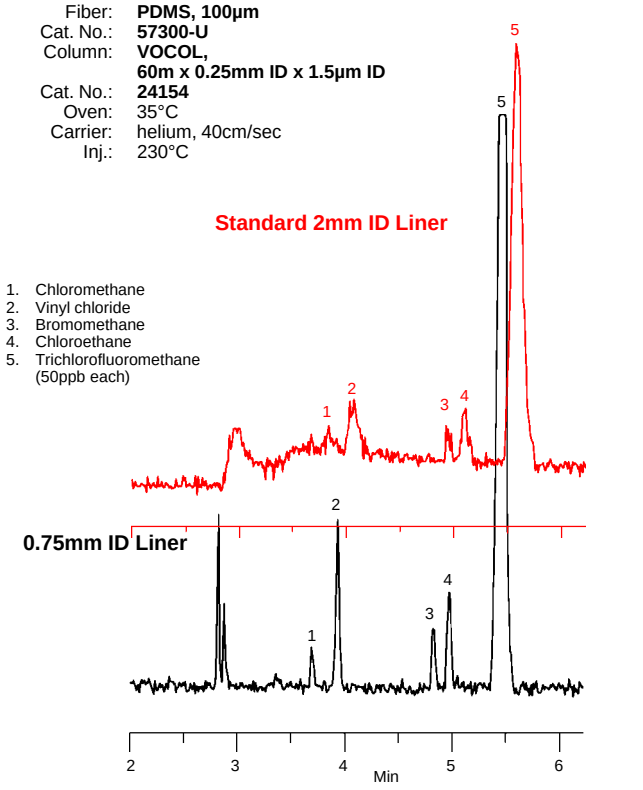

Table 3. Linear Responses for Difficult-to-Extract Volatile Compounds

Analyte	9 concentration points, 25ppb - 10ppm Mean	% RSD
Chloromethane	0.021*	17.6
Vinyl chloride	0.667*	21.0
Bromomethane	0.024*	11.8
Chloroethane	0.226*	16.8
Trichlorofluoromethane	0.023*	8.7
1,1-Dichloroethene	0.345**	12.1
Methylene chloride	0.039**	15.8
trans 1,2-Dichloroethene	0.350**	14.0
1,1-Dichloroethane	0.277**	8.8
Chloroform	0.105**	11.1
1,1,1-Trichloroethane	0.376*	4.7
Carbon tetrachloride	0.079**	12.0
1,2-Dichloroethane	0.179*	8.6
Benzene	1.959*	4.7
Trichloroethene	0.336**	3.7

$$\text{Response Factor} = \frac{(\text{area counts}_{\text{analyte}}) \times (\text{concentration}_{\text{int. std.}})}{(\text{area counts}_{\text{int. std.}}) \times (\text{concentration}_{\text{analyte}})}$$

Sample: volatile organic compounds listed in US EPA Method 624 in water + 25% NaCl (only first 15 eluting compounds are shown)
SPME Fiber: 100µm polydimethylsiloxane-coated fiber
Cat. No.: **57300-U**
Extraction: immersion, 5 min
Column: 10m x 0.20mm ID, 1.20µm film
Cat. No.: SPB-1: **24134-U**; VOCOL: **24129-U**
Oven: 40°C (0.75 min) to 160°C at 20°C/min
Carrier: helium, 40cm/sec
Det.: FID, 230°C
Inj.: splitless, 230°C

* SPB™-1 column.
**VOCOL™ column.

Optimizing SPME

Analysts who are not familiar with SPME often express concern about obtaining consistent results and reliable detection of low concentrations of analytes. The polarity and thickness of the coating on the fiber, the sampling method (headspace sampling or fiber immersion), the volume pH, and salt content of the sample, sample agitation, and other factors affect results from SPME. Remember that in SPME neither complete extraction of analytes nor full equilibrium is necessary, but consistent sampling time, temperature, and fiber immersion depth are critical.

Fiber Polarity, Porosity/Surface Area Nonpolar analytes are most effectively extracted with a nonpolar fiber coating and polar analytes are most effectively extracted with a polar coating (Table 4), just as nonpolar or polar analytes are most effectively analyzed on a gas chromatography column of like polarity. In SPME however, because only 1cm of fiber is exposed to the sample matrix, the fiber coating must be either nonpolar or strongly polar in nature. The small differences in stationary phase polarity that are useful in gas chromatography (a 5% diphenylsiloxane/95% dimethylsiloxane phase versus a 100% dimethylsiloxane phase, for example) will not produce appreciable selectivity differences in SPME. What can be beneficial, however, is the addition of an adsorbent material to the coating, such as coating strongly polar Carbowax® PEG onto divinylbenzene polymer. The polymer increases the available surface area and thus improves extractions of small polar molecules, as shown in Table 4. The small pores in Carboxen™ particles make this carbon molecular sieve particularly effective for extracting small molecules (Table 4).

Table 4. Effects of SPME Fiber Coating Polarity on Analyte Extraction

	Area Counts				
Analyte*	Nonpolar 100µm PDMS	Polar 85µm polyacrylate	Particles/ Nonpolar 65µm PDMS/DVB	Particles/ Polar 65µm Carbowax/DVB	Carboxen Particles/ PDMS
Methanol	0	170	30	75	630
Ethanol	35	180	110	130	5250
Acetonitrile	140	230	160	130	6500
Isopropanol	180	360	600	250	97700
n-Propanol	220	1200	1200	450	53400
Acetone	400	260	640	250	83000
Ethyl acetate	1500	2700	14000	4700	449500
2-Me-3-propanone	4000	2100	48000	13000	821000

*Listed in order of decreasing polarity

Our current selection of SPME fibers, and their recommended applications, are described in the **Ordering Information** section of this bulletin. For additional information on choosing the best fiber for a particular application, request free Application Notes 11, 56, and 143 (SPME of volatile analytes), 6, 17, and 81 (SPME of semivolatiles), and/or 58, 85, 94, and 143 (SPME of pesticides and herbicides). If you prefer to experiment to determine the best fiber for your application, we offer several fiber kits, each consisting of a selection of fiber types (see **Ordering Information**).

Fiber Coating Thickness A thick fiber coating will extract more of a given analyte than will a thin coating (Table 5). Consequently, a fiber with a thicker coating is used to retain volatile compounds and transfer them to the GC injection port without loss, but a thin coating is used to ensure fast diffusion and release of higher boiling compounds during thermal desorption. A thick coating will effectively remove high boiling compounds from the sample matrix, but the desorption rate will be prolonged, and analytes could be carried over to the next extraction.

Sample Agitation Sample agitation enhances extraction and reduces extraction time, especially for higher molecular weight analytes with high diffusion coefficients. Inconsistent stirring causes poor precision and is worse than no stirring. Sonication promotes analyte adsorption, but can add heat to the sample. This might be beneficial for vaporizing the analytes for headspace extraction.

Table 5. Effect of SPME Fiber Coating Thickness on Analyte Recovery

	Relative Recovery (%)		
Analyte*	100µm Coat	30µm Coat	7µm Coat
Benzene	2	1	<1
Toluene	5	1	<1
Ethylbenzene	6	4	1
1,3-Dichlorobenzene	15	5	1
Naphthalene	13	4	1
Acenaphthylene	19	8	3
Fluorene	29	18	6
Phenanthrene	37	27	16
Anthracene	49	38	32
Pyrene	69	54	47
Benzo(a)anthracene	105	91	96
Chrysene	100	100	100
Benzo(b)fluoranthene	104	111	120
Benzo(a)pyrene	119	127	131
Indeno(1,2,3-cd)pyrene	61	140	148
Benzo(ghi)perylene	61	117	122

*Listed in order from smaller to larger molecular weights

SPME: polydimethylsiloxane-coated fibers, immersion sampling, 15 min

Immersion versus Headspace Sampling; Effects of Salt and pH Analytes that exhibit a vapor pressure can be extracted by immersing the fiber into the sample, or by sampling the headspace above the sample. Analytes which exhibit no vapor pressure must be extracted by immersion. Adding 25-30% (wt./vol.) sodium chloride to the sample or changing the sample pH prior to extraction can increase the ionic strength of the solution and, in turn, reduce the solubility of some analytes.

The addition of salt to a sample greatly increases the extraction efficiency for many analytes, particularly polar compounds and volatiles. Salt should be added for trace analyses. Salt is not needed to enhance extraction of analytes with high distribution constants, however, and may introduce interfering peaks. Changing the pH can minimize the solubility of some analytes. Acidic and basic compounds are more effectively extracted at acidic and basic pH, respectively. A combination of salt and pH modification often enhances the extraction of analytes from the headspace (Table 6). Note that equilibration is faster in headspace sampling than in immersion sampling, because molecules move more rapidly in air than in water.

Other Factors Less subject to control, but influencing the extraction, is analyte concentration. At low concentrations, such as 50ppb or less for the volatile compounds in Table 6, changes in sample volume do not affect response, because equilibrium is concentration-dependent. At higher concentrations, changes in sample volume become significant. With a large sample (>5mL) containing a high concentration of analyte, the amount of analyte removed from the sample is not sufficient to change the concentration. Therefore, response throughout the calibration curve is exponential, not linear, especially for compounds with high distribution constants. Responses may be linear for low concentrations. Because analyte concentration often is not known, it is best to keep sample volumes between 1mL and 5mL, and always

Table 6. Added Salt, pH Change Improve Extraction of Phenols

	No Salt		Salt*	
Analyte	**pH 7**	**pH 2**	**pH 7**	**pH 2**
2-Chlorophenol	1800	2361	3952	14028
Phenol	810	1003	6425	6150
2-Methylphenol	761	882	5485	7434
3- & 4-Methylphenol	1795	1846	15337	19723
2-Nitrophenol	422	474	311	2315
2,4-Dimethylphenol	1344	1476	15000	20710
2,4-Dichlorophenol	5396	8138	19803	61664
2,6-Dichlorophenol	2991	5858	12511	48530
4-Chloro-3-methylphenol	2398	3137	24060	33529
2,4,5-Trichlorophenol	3115	11097	24270	96333
2,4,6-Trichlorophenol	9702	19307	35466	109492
2,4-Dinitrophenol	0	11	765	1182
4-Nitrophenol	626	730	11458	6536
2,3,4,6-Tetrachlorophenol	3108	27683	33938	70440
2-Methyl-4,6-Dinitrophenol	55	47	920	1685
Pentachlorophenol	2305	40582	22056	143905
Dinoseb	68	2123	6676	37744

*Sodium chloride, saturated solution. Values = area counts.

use the same volume for samples and calibration standards. If you anticipate extracting the analytes by using an immersion sampling technique, minimize the headspace in the sample vial.

Finally, the desorption parameters – injection port temperature, depth of fiber insertion in the injection port, and desorption time – also must be optimized for the analytes involved. Once established, these values should be used consistently.

By controlling the polarity and thickness of the coating on the fiber, consistently agitating the sample, and, when necessary, adjusting the pH and/or salt content of the sample, an analyst can be assured of highly consistent, quantifiable results from SPME.

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References not available from Supelco.

* Technology licensed exclusively to Supelco. US patent pending; European patent #0523092.

**For information about SPME devices specifically designed for field sampling and prolonged storage of adsorbed analytes, request Product Specification 497105 and 497174.

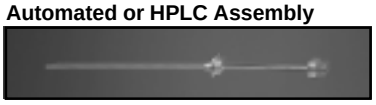
For new information on SPME products, applications, and literature, visit our website:

www.supelco.com

The SPME page is just a click away from our main menu.

Ordering Information:

Automated or HPLC Assembly



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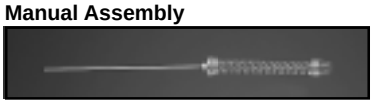
Manual Assembly



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SPME Fiber Assemblies

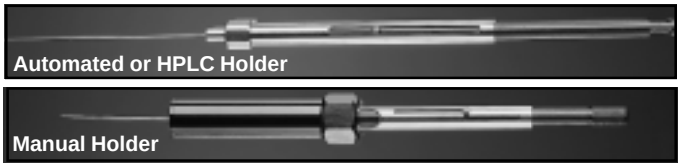
SPME fiber assemblies can be reused for up to 100 analyses, or more, depending on the particular application and the care that they are given. For reuse, simply condition with solvent or heat before and after every analysis.

Stationary Phase	Recommended Use	For Manual Holder (57330-U) Cat. No.	For Automated or HPLC Holder (57331) Cat. No.
Polydimethylsiloxane (PDMS)			
100µm / non-bonded	volatiles	**57300-U**	**57301**
30µm / non-bonded	nonpolar semivolatiles	**57308**	**57309**
7µm / bonded	moderately polar to nonpolar semivolatiles	**57302**	**57303**
Polydimethylsiloxane/Divinylbenzene (PDMS/DVB)			
65µm / partially crosslinked	polar volatiles	**57310-U**	**57311**
60µm / partially crosslinked	general purpose (for HPLC only)	**—**	**57317**
Polydimethylsiloxane/Carboxen (PDMS/Carboxen)			
75µm / partially crosslinked	trace-level volatiles	**57318**	**57319**
Carbowax/Divinylbenzene (CW/DVB)			
65µm / partially crosslinked	polar analytes	**57312**	**57313**
Carbowax/Templated Resin (CW/TPR)			
50µm / partially crosslinked	surfactants (for HPLC only)	**—**	**57315**
Polyacrylate			
85µm / partially crosslinked	polar semivolatiles	**57304**	**57305**
Fiber Assortment Kits			
Fiber Kit 1 (one fiber of each), 85µm polyacrylate, 100µm PDMS, 7µm PDMS		**57306**	**57307**
Fiber Kit 2 (one fiber of each), 65µm CW/DVB, 65µm PDMS/DVB, 75µm PDMS/Carboxen		**57320-U**	**57321-U**
Fiber Kit 3 (one fiber of each), 50µm CW/TPR, 60µm PDMS/DVB, 100µm PDMS		**—**	**57323-U**
Fiber Kit 4 (one fiber of each), 100µm PDMS, 65µm PDMS/DVB, 75µm PDMS/Carboxen		**57324-U**	**57325-U**

Non-bonded phases are stable with some water-miscible organic solvents, but slight swelling may occur. NEVER use or rinse with nonpolar organic solvents.
Bonded phases are stable with all organic solvents. Slight swelling may occur when used with some nonpolar solvents.
Partially crosslinked phases are stable in most water-miscible organic solvents. They may be stable in some nonpolar solvents, but slight swelling may occur.

SPME Fiber Holders

The holder protects the phase-coated fiber, and controls exposure of the fiber for sample adsorption and desorption. The holder is reusable indefinitely and accepts any of the replaceable fiber assemblies. First time users must order both a holder and a fiber assembly.



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Description	Cat. No.

Holder for Automated Sampling or HPLC Analysis

This holder can be used with a Varian 8100/8200 AutoSampler or an SPME/HPLC interface. (An SPME upgrade kit is necessary for operation with the Varian AutoSampler — contact Varian Instrument Division for information concerning system requirements). The needle moves freely for control by an automated system, and for depth regulation in the SPME/HPLC interface desorption chamber.

SPME Fiber Holder for Automated Sampling or HPLC Analysis	**57331**

Holder for Manual Sampling

An adjustable depth guide positions the fiber for sampling and for correct placement in the heat zone of a GC injection port. The fiber can be locked in the exposed position and easily retracted back into the needle.

SPME Fiber Holder for Manual Sampling	**57330-U**

SPME Portable Field Sampler

This manual-type holder stores the fiber after sampling by sealing it with an internal septum. It is ideal for field work and comes with a PDMS/Carboxen fiber for trace-level volatile analysis, or a PDMS fiber for concentrating polar analytes.

997-0046

Description	Cat. No.
SPME Portable Field Samplers, pk. of 2	
75µm PDMS/Carboxen	**504831**
100µm PDMS	**504823**
Accessories	
Replacement septa, pk. of 10	**20638**
SPME Septum-Removing Tool	**504858**

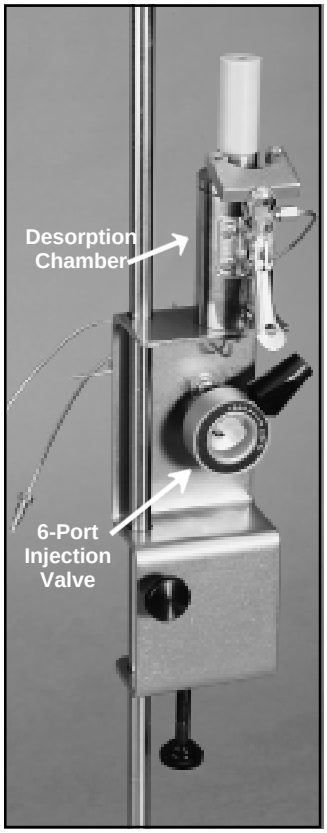
SPME/HPLC Interface

For HPLC Analyses with SPME

The SPME/HPLC interface allows mobile phase to contact the SPME fiber, remove the adsorbed analytes, and deliver them to the column for separation. Analytes can be removed via a stream of mobile phase (dynamic desorption) or, when analytes are more strongly adsorbed to the fiber, the fiber can be soaked in mobile phase before the material is injected onto the column (static desorption).

The interface consists of a six-port injection valve and a desorption chamber that replaces the injection loop in the HPLC system. The SPME fiber is introduced into the desorption chamber under ambient pressure. After the fiber is inserted through the ferrule, the unit is made leak-tight by closing the clamp and compressing the ferrule against the SPME needle. The ferrule maintains a leak-free seal at pressures up to 5000psi (35mPa). All surfaces which come in contact with the SPME fiber or the mobile phase are stainless steel or VESPEL®. Easily installed and removed, the desorption chamber includes a PEEK (polyetheretherketone) needle guide, a stainless steel body and compression cap, a double-tapered VESPEL ferrule, and a sealing clamp.

SPME/HPLC Interface



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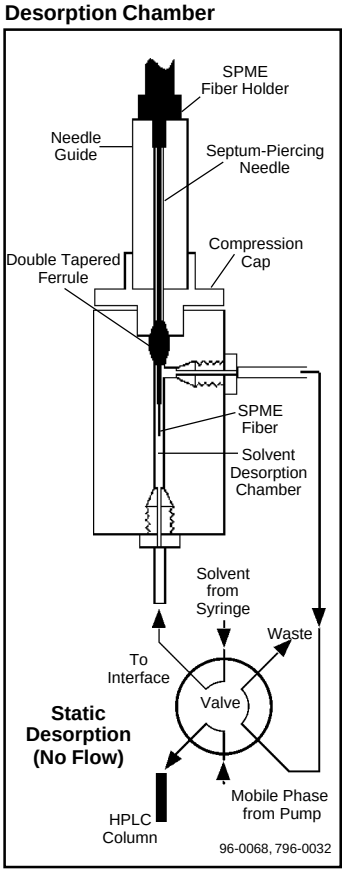
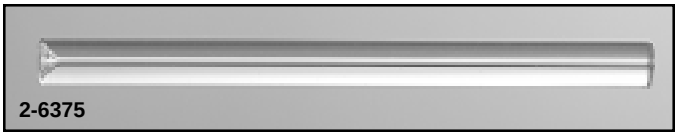
Desorption Chamber



Description	Cat. No.
SPME/HPLC Interface, includes 2 ferrules	
Valco® valve version	**57350-U**
Rheodyne® valve version	**57353**
Replacement parts	
Ferrules, pk. of 10	**57351**
Rotor, Valco model C6W	**57352**
Rotor seal for Rheodyne valve 7125	**58830-U**
Rhebuild kit for Rheodyne valve 7125	**55045**

SPME Inlet Liners

Achieve Sharper Peaks with SPME/GC



997-0137

Using a 0.75mm ID inlet liner in the GC injection port increases linear velocity through the liner, compared to a larger volume 2mm ID liner, and rapidly introduces analytes onto the column in a narrow band. Our proprietary, high-temperature technique thoroughly deactivates these liners to minimize adsorption of active sample components.

Inlet Liners for SPME

Description	Cat. No.
Hewlett-Packard® (5880, 5890 series, 6890)	
Each	**26375,01**
pk. of 5	**26375,05**
pk. of 25	**26375,25**
Varian	
1075/1077 Injectors	
Each	**26358,01**
pk. of 5	**26358,05**
pk. of 25	**26358,25**
1093/1094 SPI Injectors	
Each	**26364,01**
pk. of 5	**26364,05**
pk. of 25	**26364,25**
Perkin-Elmer® (Auto System Split/Splitless Injector)	
Each	**26312,01**
pk. of 5	**26312,05**
pk. of 25	**26312,25**
Shimadzu®	
GC Models 9A/15A/16 (SPL-G9/15 Injector)	
Each	**26329,01**
pk. of 5	**26329,05**
pk. of 25	**26329,25**
GC Models 14/15A/16 (SPL-14 Injector)	
Each	**26335,01**
pk. of 5	**26335,05**
pk. of 25	**26335,25**
GC Models 17A (SPL-17 Injector)	
Each	**26339,01**
pk. of 5	**26339,05**
pk. of 25	**26339,25**

913-0249

SPME Inserts

Description	Cat. No.
Flash On-Column Inserts for Varian SPME Injector	
pk. of 5	**26364,05**

For more information on SPME/HPLC analyses, request Application Notes: 98 on explosives, 99 on PAHs, 106 on surfactants, 110 on food antioxidants and preservatives, or 121 on carbamates.

SPME Accessories

995-0254

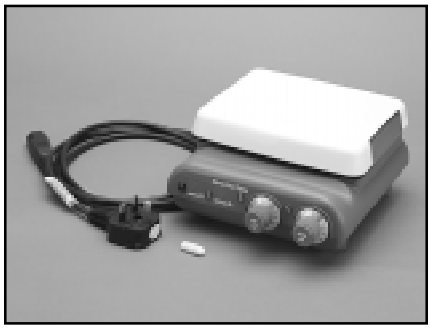
SPME Sampling Stand

Holds eight vials while supporting the SPME syringe for consistent fiber immersion. Accommodates 4mL vials only.

Description	Cat. No.
SPME Sampling Stand	**57333-U**

Heat/Stir Plates

Heat or stir samples when using the SPME sampling stand. The plate fits compactly on the base of the stand. Heating range is 40-550°C. Stirring range is 60-1200rpm. 240VAC unit is CE approved.



997-0102

Description	Cat. No.
Corning Heat/Stir Plate, 120VAC	**Z26,212-9**
Corning Heat/Stir Plate, 240VAC ■	**Z26,213-7**

Magnetic Stirring Bars

Fit 4mL vials. 10 x 3mm, pk. of 3.

Magnetic Stirring Bars	**Z11,887-7**

Thermometer

For monitoring the temperature of samples when using the SPME sampling stand and the heat/stir plate. 5" long.

Thermometer	**57332**

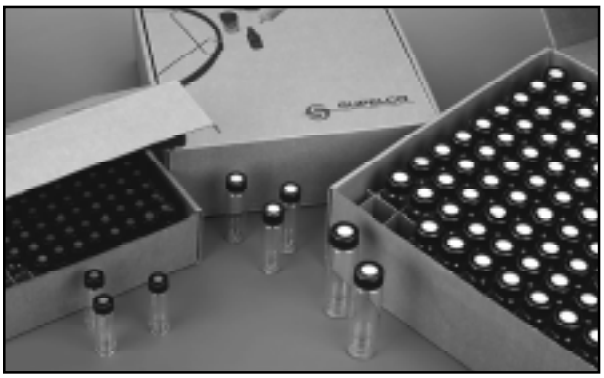
■ CE approved.

Pre-Drilled Septa for SPME

- Easier needle penetration and high puncture tolerance
- Reduces septum coring that can cause extraneous peaks
- Conditioned, ready-to-use
- Extremely low bleed over a wide range of inlet temperatures — from 100°C to 250°C
- Rubber formulation exclusive to Supelco

Description	Qty.	Cat. No.
Thermogreen™ LB-2 Septa, Pre-Drilled		
11mm	pk. of 25	**23167**
11mm	pk. of 50	**23168**
9.5mm	pk. of 25	**23161**
9.5mm	pk. of 50	**23162-U**

Screw-Top Vials with Closures



994-0420

PTFE silicone septa and open-top phenolic closures.

Description	Qty.	Cat. No.
Screw Top Vials for Varian 8100/8200 AutoSampler, 12mm OD x 32mm length		
Clear, 2mL	pk. of 100	**27124-U**
Amber, 2mL	pk. of 100	**27005**
Headspace Vials, 15mm OD x 45mm length		
Clear, 4mL	pk. of 10	**26901**
	pk. of 100	**27136**
Amber, 4mL	pk. of 10	**26930**
	pk. of 100	**27006**

Crimp-Seal Vials

Designed for use with thin septa — for easier penetration by an SPME needle.

Description	Qty.	Cat. No.
Headspace Vials for Varian 8100/8200 AutoSampler		
Clear, 10mL	pk. of 36	**27385**
	pk. of 144	**27386**
Closures and Septa for 10mL vial (20mm Viton®)		
	pk. of 36	**33146-U**
	pk. of 100	**27245**

For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website (www.sigma-aldrich.com), see the Supelco catalog, or contact Supelco, Bellefonte, PA 16823-0048 USA.

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