SPME for GC Analysis

Getting Started with Solid Phase Microextraction
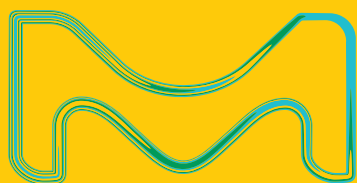
Supelco®

Analytical Products

SAMPLE PREP MADE EASY

Solid Phase Microextraction (SPME)

Solid Phase Microextraction (SPME) is an innovative, solvent-free sample prep technology that is fast, economical, and versatile.

How it Works

SPME uses a fiber coated with an extraction phase comprising a pure polymer (liquid coating) or adsorptive particles embedded in a polymer (adsorbent/particle coatings). The fiber coating extracts the compounds from the sample. The SPME fiber is then inserted directly into the GC injector for thermal desorption and analysis.

SPME has gained widespread acceptance as a preferred technique for many applications in the areas of food & beverage, flavors and fragrances, forensics and toxicology, and environmental volatiles (VOC) testing, to name a few.

During the SPME process, equilibria are established for the analytes between the sample, or the headspace above the sample, and the fiber. The analytes on the fiber are then directly thermally desorbed in the GC injector and transferred to the GC column. SPME concentrates analytes on the fiber, and the desorption step delivers them rapidly to the column. Thus, detection limits are minimized. SPME is compatible with analyte separation/detection by gas chromatography or HPLC, and provides quantitative results for a wide range and concentrations of analytes.

For more information visit
SigmaAldrich.com/spme

SPME Advantages

- Solvent-free microextraction technique for nearly any sample or matrix
- Easy to automate
- Compatible with GC instruments
- Reusable
- Inexpensive
- Fast
- Advantageous sample prep technology compared to static headspace gas chromatography (GC) and solid phase extraction (SPE)
- Directly interfaced with GC analysis for desorption into the GC injection port.
- Non-destructive to sample

SAMPLING AND SAMPLE PREPARATION IN ONE STEP ON A SMALL FIBER

How To Get Started with SPME

For more information visit
SigmaAldrich.com/spme

Optimization of SPME Extraction Conditions

Fiber Coating Selection

Types of SPME Fiber Coatings

There are two types of SPME coatings: polymeric films for absorption of analytes, and particles embedded in polymeric films for adsorption of analytes **(Table 1)**. The absorbent type (film) fibers include those coated with polydimethylsiloxane (PDMS), polyacrylate (PA), and polyethylene glycol (PEG).

The adsorbent type (particle) fibers contain porous particles such as divinylbenzene (DVB), Carboxen® adsorbent (CAR), or a combination of both. Usually, PDMS is used as the binder.

Typically the adsorption on a particle is a stronger and more efficient extraction mechanism, making the particle fibers more suitable for trace analysis methods at lower concentrations. However, as they have a finite surface on the particle, the linear range is typically smaller than the one from the film fibers.

Coating	Polarity
Films – Absorption	
7 µm Polydimethylsiloxane (PDMS)	Nonpolar
30 µm PDMS	Nonpolar
100 µm PDMS	Nonpolar
85 µm Polyacrylate (PA)	Polar
60 µm CARBOWAX™-Polyethylene Glycol (PEG)	Polar
Particles – Adsorption	
85 µm Carboxen®/PDMS	Bipolar
65 µm PDMS/DVB	Bipolar
55 µm/30 µm DVB/CAR/PDMS	Bipolar

Table 1. SPME Fiber Coating Types and Polarities

When selecting the appropriate SPME fiber, consider the physical and chemical properties of the compounds being analyzed. **Figure 1** illustrates fiber coating selection based on the molecular weight (MW) as an indicator for the volatility of the analytes. **Table 2** provides more specific guidance for fiber selection based on analyte type.

In the case of the adsorptive fiber types, the Carboxen®/PDMS fiber works well for low molecular weight, highly volatile compounds. Regarding the PDMS-DVB fibers, the more macro- and mesoporous DVB makes them better suited for higher MW compounds than the Carboxen®, providing efficient extraction and desorption properties The DVB-Carboxen® fibers cover both areas and expand the MW range that can be analyzed with the same fiber; however, they have slightly lower capacity for lighter or heavier compounds in comparison to the single sorbent fibers.

In the case of the absorptive PDMS fiber, the thinner film is better suited for higher MW analytes than the 100 µm PDMS fiber. Heavier analytes will be easier to desorb off of the 7 µm compared to the 100 µm fiber.
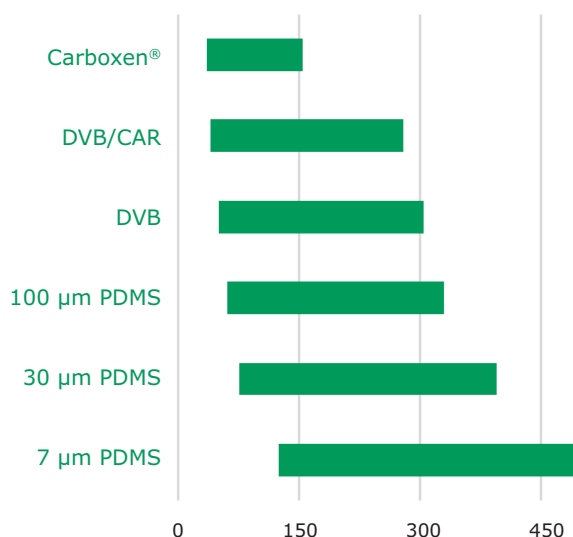

Figure 1. Analyte Molecular Weight Range (g/mol) for SPME Fibers

Optimization of SPME Extraction Conditions

Fiber Coating Selection

Analyte Type	Molecular Weight Range (g/mol)	Recommended Fiber
Gases and Low Molecular Weight Compounds	30 - 225	75 µm/85 µm CAR/PDMS
Volatiles	60 - 275	100 µm PDMS
Volatiles, Amines, and Nitro-aromatic Compounds	50 - 300	65 µm PDMS/DVB
Polar Semi-volatiles	80 - 300	85 µm PA
Non-polar High Molecular Weight Compounds	125 - 600	7 µm PDMS
Non-polar Semi-Volatiles	80 - 500	30 µm PDMS
Alcohols and Polar Compounds	40 - 275	60 µm CARBOWAX® (PEG)
Flavor Compounds: Volatiles and Semi-volatiles, C3 - C20	40 - 275	50 µm/30 µm DVB/CAR on PDMS on a StableFlex™ fiber
Trace Compound Analysis (ppb)	40 - 275	50 µm/30 µm DVB/CAR on PDMS on a 2 cm StableFlex™ fiber
Amines and Polar Compounds (HPLC use only)		60 µm PDMS/DVB

Table 2. Fiber Coating Selection Based on Analyte Properties

Fiber Core Type

There are currently three fiber core types: fused silica, StableFlex™ (SF), metal alloy, and nitinol. Fused silica is the original support for SPME, and numerous methods have been developed on these types of fibers. Fused silica has the advantage of being highly inert; however, the major disadvantage of fused silica is that it is fragile.

Currently, adsorbent type fibers are now available on StableFlex™ (SF) cores in addition to standard fused silica cores. A thin coat of polymer on the fused silica makes the SF core more flexible/rugged than the original fused silica because the coating also tends to bond more tightly to the SF core. This creates a more stable and durable fiber phase. In addition, due to automated fiber production, SF fibers tend to give better reproducibility than the original fused silica versions.

All metal alloy fiber assembies and fibers with a nitinol-core are even more flexible than the SF fibers. Comprised of a nickel/titanium alloy, the nitinol-core fibers are not only more durable but also highly reproducible. Nitinol is also known to be a highly inert as it is frequently used in the medical device industry in products such as stents. Additionally, the selectivity of nitinol-core (NIT) fibers is equivalent to that of the fused silica (FS) core fibers of the same coating type lending to simplified method transfer from FS to NIT fibers. The metal alloy design includes a thicker, flexible plunger that is much less likely to kink or break, and helps to reinforce the also superelastic needle, especially when used in an autosampler with a sample agitator.

Fiber Assembly Configuration

There are fiber assemblies designed for use with the manual holder as well as fiber assemblies designed for use with automation. The manual fiber assemblies contain a spring; while those designed for automation do not contain a spring **(Figure 2)**. Assemblies designed for automation may be used in a holder designed for automation or a manual holder. There are two needle gauge (Ga) sizes as well: 23 Ga and 24 Ga. The original SPME fibers were manufactured with 24 Ga needles, and these continue to work very well for manual sampling. More recently, SPME fibers with 23 Ga needles have been developed and are highly recommended to be used for all applications utilizing an autosampler. The 23 Ga needles also work well with the Merlin MicroSeal™ septum system, as well as other septum-less seals. Try to avoid using the 23 Ga needles with standard silicone septa, as they may core the septum.

Figure 2.
Automated Fiber Assembly (Left)
Manual Fiber Assembly (Right)

For more information visit
SigmaAldrich.com/spme

Fiber Installation

To begin using SPME, attach the fiber assembly to the SPME holder. The steps below describe the process for inserting a fiber to a manual holder.

1. Unscrew the black cylinder-like depth gauge from the holder **(A)**.

2. Unscrew the threaded end-cap **(B)** on the end of the holder.

3. Push the black plunger **(C)** forward through the Z-slot on the base of the holder to expose the end of the plunger. Note internal threads inside of the plunger **(D)** will accept the threaded fiber assembly **(E)**.

4. Screw the fiber assembly into the end of the plunger.

5. Retract the plunger by pulling it back through the Z-slot and slide the threaded end-cap over the needle. Screw the threaded end-cap tightly onto the end of holder.

6. Screw the black depth gauge onto the end of the holder over the threaded end-cap.

7. Test the holder/fiber by pushing the plunger forward until the fiber is exposed from the protective needle. Stop at the Z-slot **(F)** to hold the fiber in the exposed position during sampling and injection in the GC.

8. To retract the fiber, move the plunger out of the Z-slot and draw it back.

The fiber assembly attaches in the same way to the auto-sampler holder. Remove the hexagonal nut and push the black plunger down to expose threaded port for fiber assembly attachment. No spring is used with the auto-sampler fiber assemblies because the autosampler controls the movement of the plunger and fiber. Autosampler fiber assemblies can be used with manual holders, but the manual fiber assemblies cannot be used with the autosampler holders because of the spring.

To see a video of fiber installation, visit SigmaAldrich.com/spme-videos and select "Installing an SPME Fiber."
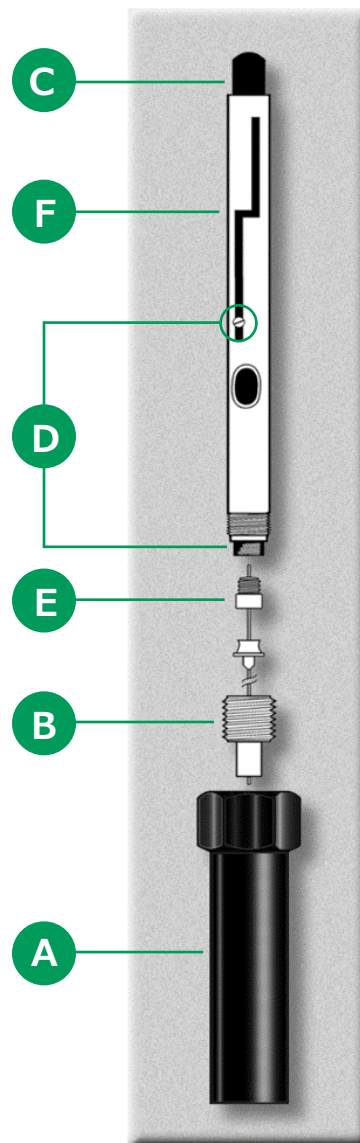

Figure 3. Diagram Illustrating Attachment of the SPME Fiber Assembly to the Holder

Optimization of SPME Extraction Conditions

GC Injector Setup

To reduce inlet septa coring, we strongly recommend using molded Thermogreen® LB-2 septa with injection hole. Also, make sure that the injection port contains an appropriate liner. The use of a liner designed specifically for SPME with a narrow I.D. (0.75 mm - 1.0 mm), compatible with the respective GC instrument, is strongly recommended. This will reduce band broadening to sharpen analyte peaks. Refer to the "SPME Product Offering" section for recommended inlet liners for various instruments. Do not insert an SPME fiber into a liner containing glass wool. If the fiber contacts the wool, the fiber's coating could be damaged or the fiber can break.

Precondition the Fiber

It is essential to precondition/thermally clean the SPME fiber before use. In Gas Chromatography (GC), this is performed thermally by exposing the fiber in the GC injection port (alternatively, in a conditioning station of the autosampler). When conditioning, be sure to open the splitter to reduce the amount of impurities entering the column.

For temperatures and times, follow the conditioning guidelines in **Table 3**. Insert the fiber into the injection port at the appropriate needle depth by adjusting the black needle guide so the top is between 3.5 to 4 on the vernier gauge on the holder (suitable for most instruments). If a fiber becomes contaminated after use, these steps can be repeated. If the contamination is severe, the fibers can be thermally cleaned for an extended period of time at a temperature 20 °C below the listed conditioning temperature in **Table 3**. If this does not clean the fibers, solvent cleaning can be attempted. Please follow the guidelines for solvent cleaning of specific fiber coatings in the SPME Troubleshooting section. After fiber conditioning, always ramp the oven temperature to remove any contaminants that may have entered the column and perform a blank run afterwards.

A conditioned fiber should be stored clean (e.g. in an empty closed vial with clean nitrogen) to prevent unnecessary contamination.

Fiber Coating	Film Thickness	Cond. Temp. (°C)	Cond. Time (h)
PDMS	100 µm	250	0.5
PDMS	30 µm	250	0.5
PDMS	7 µm	320	1
PDMS/DVB(+OC)	65 µm (+10 µm)	250	0.5
Polyacrylate	85 µm	280	0.5
Carboxen®/PDMS	all	300	0.5
PEG	60 µm	240	0.5
DVB/CAR/PDMS	50/30 µm	270	0.5

Table 3. Temperature and Conditioning Guidelines for SPME Fiber Coatings

Extraction Mode

When choosing the extraction mode, the physical and chemical properties of the analytes and the composition of the matrix must be considered. Headspace (HS) and direct immersion (DI) are the two extraction modes for SPME. If the analytes of interest are reasonably volatile, HS is the best choice. HS is the cleaner of the extraction modes because the fiber does not come in contact with the matrix or non-volatile components. It is exposed only to the air surrounding the matrix. HS extraction mode is useful when sampling from solid or very dirty matrices. Often matrix modifications must be performed to help drive analytes into the headspace (see "Matrix Modification" sections).

For compounds having low to medium volatility and high to medium polarity, DI is the preferred extraction mode. When performing DI, the fiber is submerged directly into the matrix. In most cases, the extraction efficiency is much higher in DI as compared to HS. Please refer to **Table 4** to determine the right mode for the application of interest.

When performing DI in very dirty or complex matrices, a fiber that is more physically robust and less prone to fouling is preferred. The overcoated SPME fiber (SPME-OC), also known as the matrix-compatible PDMS/DVB/PDMS fiber, has an extra outer layer of PDMS to make it compatible with complex matrices. For more information on this fiber, please visit SigmaAldrich.com/spme-ocf.

	Direct Immersion (DI)	Headspace (HS)
Analyte Properties	Low-to-medium volatility High-to-medium polarity	High-to-medium volatility Low-to-medium volatility
Sample Matrix	Simple or complex liquid	Simple and complex liquid; solid
Advantages	Higher extraction efficiency	Very helpful with dirty and complex matrices, long fiber lifetime
Disadvantages	Fouling, shorter fiber lifetime, sample pretreatment	Sample modification needed to improve mass transfer in HS

Table 4. Choosing an Extraction Mode Based on Application

Agitation Method

Agitation/stirring of the sample facilitates the mass transport between the sample and the fiber coating (improving the kinetics), and therefore may provide shorter extraction times as well as greater sensitivity in pre-equilibrium extractions (see "Extraction Time" section). There are a variety of agitation methods, each having advantages and disadvantages. For reproducible results, it is essential to maintain the same agitation method and agitation intensity. Please refer to **Table 5** to choose the one best suited for the application. Ultra-sonication is not recommended as it heats the sample uncontrollably and damages the fiber.

Agitation Method	Advantages	Disadvantages
Magnetic Stirring	• No sophisticated equipment	• Stirring plate can cause heating of the vial • Stir bar must be inserted in the sample
Needle Vibration	• Useful for trace analysis (ppb level)	• Excessive stress on the needle • Applicable only to relatively small sample volumes
Vortex Stirring	• Useful for trace analysis (ppb level) • Small sample volume limitation does not apply	

Table 5. SPME Agitation Methods with Advantages and Disadvantages

Extraction Time

The extraction time is a critical parameter in the SPME sampling process. **Figure 4** shows the typical relationship of extraction time to analyte absorbed on the fiber.

The optimum extraction time depends on the objective(s) of the analysis. If the main goal is high throughput, choose the shortest extraction time possible, and work under pre-equilibrium conditions. In this case, it is imperative to keep the extraction time and agitation exactly the same for each sampling. If the time that the fiber is exposed during sampling varies, the concentration of the analyte on the fiber will also vary, resulting in poor reproducibility. Therefore, when working under pre-equilibrium conditions, it is highly recommended to utilize SPME automation in order to attain good reproducibility.

If sensitivity is the most important factor, make sure to operate at, or close to, equilibrium conditions. Use an extraction time after which the uptake of analyte onto the fiber remains constant. Furthermore, the equilibrium can be shifted to provide higher extraction efficiencies by variation of temperature and sample modification (ionic strength and pH).

If reproducibility is the main objective, operate at or very close to equilibrium conditions (if performing the extraction manually), or in pre-equilibrium conditions using an autosampler.
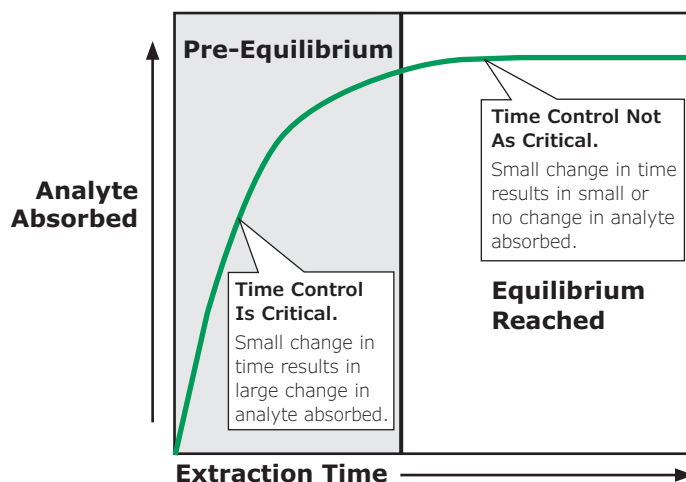

Figure 4. Effect of Extraction Time on Amount of Analyte Absorbed

Optimization of SPME Extraction Conditions

Sample Volume

Figure 5 shows the equation governing the equilibrium between analytes extracted by SPME and the initial concentration of analytes in the sample. This distribution is dependent on different parameters: the partition coefficient of the analyte between the coating and the sample, the volume of the sample, and the volume of the coating. In the case of large sample volumes (greater than 10 mL), the amount of analyte extracted is not influenced by sample volume changes anymore. Therefore, the amount of analyte extracted increases with the sample size up to a point, after which the sensitivity does not increase with further increases in sample volume. This important aspect of SPME allows for field analysis and *in vivo* analysis without having to know the precise sample volume.

When selecting an appropriate sample volume, take into account the following:

- Sample availability
- System compatibility, in the case of automation (e.g. vial size, extraction conditions, etc.)
- Sufficient volume to cover the coating reproducibility for direct immersion extraction
- Sufficient headspace, in the case of headspace extraction, to avoid fiber spilling (sample droplets on the fiber)

$$n_e = \frac{K_{fs} V_f V_s C_o}{K_{fs} V_f + V_s}$$

If

$$V_s \gg K_{fs} V_f$$

then

$$V_s / (K_{fs} V_f + V_s) = 1$$

1. **Large Samples**
2. ***in vivo* Samples**
3. **Analytes with small K_{fs}**

$$V_s \gg K_{fs} V_e \longrightarrow n_e = K_{fs} V_e C_o$$

n_e = number of moles of analyte extracted at equilibrium

V_f = fiber coating volume

V_s = sample volume

C_o = initial sample concentration of a target analyte

K_{fs} = distribution constant fiber/sample

Figure 5. Correlation between the Amount of Analytes Extracted at Equilibrium by SPME and Initial Concentration of Analytes in the Sample

For more information visit
SigmaAldrich.com/spme

Sample Matrix Modification

pH

Sensitivity of SPME is maximum when extracting neutral analytes that are not disassociated. Thus, pH modification can improve the method's sensitivity for basic or acidic compounds. When dealing with acidic compounds, select a pH that is less than the pKa of the compound (minus two units or less). When dealing with basic compounds, select a pH that is greater than the pKa of the compound (plus two units or more).

When performing headspace SPME (HS-SPME), the matrix can be adjusted to any pH value without damaging the fiber. If performing direct immersion SPME (DI-SPME), care must be taken when adjusting the pH. Very low and very high pH levels may damage the fiber coating. **Table 6** illustrates the recommended pH range of operation for the respective SPME fibers when performing DI-SPME.

Fiber Coating	Film Thickness	pH
PDMS	100 µm	2 to 10
PDMS	30 µm	2 to 11
PDMS	7 µm	2 to 11
PDMS/DVB (+OC)	65 µm (+ 10 µm)	2 to 11
Polyacrylate	85 µm	2 to 11
Carboxen®/PDMS	all	2 to 11
PEG	60 µm	2 to 9
DVB/CAR/PDMS	50/30 µm	2 to 11

Table 6. Recommended pH Range of Operation for the Respective SPME Fibers when Performing Direct Immersion SPME (DI-SPME)

Ionic Strength

In certain applications, the addition of salt to an aqueous solution minimizes the variability in ionic strength in the sample, helping to normalize the results obtained. Increasing the ionic strength of a sample induces an effect referred to as the salting out effect. This is mostly beneficial for compounds with log P < 3. It is recommended to saturate with salt to ensure same ionic strength from sample-to-sample, ensuring reproducibility.

Adding salt to the sample matrix may:

- Improve sensitivity in most applications by driving analytes toward the fiber

- Promote the mass transfer of analytes to the headspace, in the case of headspace analysis

- Improve reproducibility for samples containing salt.

Although sodium chloride (NaCl) is the most commonly used salt to adjust ionic strength, it is wise to explore the use of other salts. Other salts may have different abilities to salt out analytes, especially when dealing with complex matrices such as food.

Cautions to take when adding salt to the sample:

- Increasing the ionic strength may decrease solubility of some analytes.

- For some analytes, the addition of salt may also decrease the amount of analyte extracted; something to be especially aware of with analytes that are very polar.

- While salt may improve the SPME extraction of the desired analytes, it could also cause co-extraction of more matrix interferences or undesired compounds.

Sample Dilution

For complex matrices, sample dilution may prove to be beneficial. Sample dilution can help to release the analytes from the matrix, increasing analyte extraction and method sensitivity. Dilution will also help to minimize fouling of the coating and provide a longer fiber lifetime when performing extractions by direct immersion. It also helps to avoid saturation of adsorptive coatings (especially for complex matrices). However, one must be careful to avoid excessive dilution as it will essentially increase the detection limit by diluting the amount of analyte in the sample, leading to less sensitivity.

Sample Temperature

There are a number of factors to keep in mind when deciding upon the best temperature to perform an SPME extraction. Increasing the extraction temperature has a positive affect on the overall extraction kinetics by increasing the analyte diffusion coefficient, the headspace capacity, and the extraction rate and speed (decreasing the time at which equilibrium is reached). Conversely, increasing the temperature decreases the partition coefficients as well as the amount of analyte extracted at equilibrium when working with absorptive fibers. Summarizing, one must consider the priorities of the experiment when selecting an appropriate temperature (e.g. a temperature profiling is advisable).

Sample Matrix Modification

Organic Solvent Content

When adding spiking solutions in organic solvent to samples prior to SPME, keep the amount of organic solvent added to samples to a minimum; less than 5% v/v is recommended. The ideal value is ≤ 1% v/v. Also, try to keep the amount of organic solvent added to samples and standards constant, as wide variations can negatively affect reproducibility. If alcoholic beverages are investigated, a dilution to a lower alcohol content is advisable to achieve better extraction results.

Analyte Derivatization

Because derivatization reagents can bring additional sources of interference and errors into the system, derivatization should only be carried out when strictly necessary.

Analytes may be derivatized to:

- Make compounds more amenable to a particular mode of analysis or detection
- Enhance extraction efficiency
- Enhance detection sensitivity

Different options include derivatization of analytes prior to the extraction, during the extraction, or post-extraction. Derivatization prior to or during the extraction can affect extraction amounts, chromatographic behavior, and detection. Post-extraction derivatization can affect only chromatographic behavior and detection.

Desorption Condition Optimization

Desorption In GC Analysis

When desorbing analytes into the GC instrument, factors to take into consideration include carrier gas flow rate, desorption temperature, and desorption time. A high carrier gas flow rate, as well as the use of narrow bore liners in splitless mode during desorption is recommended for the best results. Please see the "SPME Product Offering" section for recommended inlet liners.

Regarding the desorption temperature, **Table 7** lists the recommended temperature for each fiber coating type. Typically particle fibers should be desorbed at high temperatures to ensure immediate release of the volatile analytes. Determine the optimal desorption time by performing a desorption time profile, making sure to desorb quantitatively and reproducible. Also, make sure to test carry over by performing a consecutive desorption of the coating and monitor for any targeted peak detected.

Typically with SPME the initial oven temperature of the GC is low (< 50 °C) to allow a refocussing of the semi- and less volatile analytes at the column entrance (hold for a minimum of 1.5 min). For more volatile compounds, thicker GC column films (> 1 µm) are recommended to ensure good peak shapes. Also a narrow inlet liner (ID 0.75-0.8 mm) support a narrow peak shape for volatile compounds, that do not refocuss at the column entrance.

Fiber Coating	Film Thickness	Recommended Desorption Temperature (°C)
PDMS	100 µm	200 to 280
PDMS	30 µm	200 to 280
PDMS	7 µm	220 to 320
Polyacrylate	85 µm	220 to 280
PEG	60 µm	200 to 250
PDMS/DVB (+OC)	65 µm (+ 10 µm)	200 to 270
CAR/PDMS	all	250 to 310
DVB/CAR/PDMS	50/30 µm	230 to 270

Table 7. Recommended Desorption Temperatures for SPME Fiber Coatings

Advances in SPME

Supelco® Smart SPME Fibers for PAL3 Series II Autosampler

The full range of Supelco® SPME fibers is now available for use with the PAL3 Series II autosampler system. Supelco® Smart SPME fibers combine SPME coating expertise and innovations, including our Carboxen®, dual-coated, and overcoated fibers, with Smart technology for seamless sample preparation. Supelco® Smart fibers are equipped with a unique Smart chip that offers the following benefits:

- **Traceability:** monitor usage, stroke count, dates of operation, and temperature exposure.

- **Ease-of-use:** automatic application of correct parameters for each specific SPME fiber coating.

- **Increased productivity:** fully automated for high throughput sample preparation.

Figure 6. Assembly with Color-Coded Smart Chip for Traceability, Ease-of-Use and Increased Productivity.

Read more on **SigmaAldrich.com/SmartSPME**

Nitinol-core (NIT) SPME Fibers

Supelco® SPME fibers with Carboxen®/ Polydimethylsiloxane (CAR/PDMS) and Polydimethylsiloxane/ Divinylbenzene (PDMS/DVB) coatings are now available on a nitinol-core (NIT) in a stainless steal assembly for improved fiber performance. Selectivity of the CAR/PDMS and PDMS/ DVB SPME fibers has been maintained for seamless transition of methods from fused-silica core (FS) fibers to the improved NIT technology. NIT fibers offer the following benefits:

- Enhanced lot-to-lot and fiber-to-fiber reproducibility stemming from more stringent coating conditions **(Figure 8)**

- Greater fiber durability due to nitinol-core mechanical strength and flexibility **(Figure 7)**

- Maintained selectivity and core inertness compared to fused silica core fibers for method consistency

Read more on **SigmaAldrich.com/NITSPME**
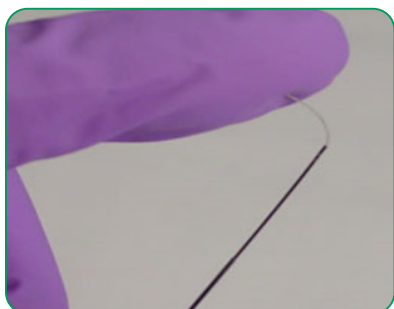
Figure 7. 90° Flexion of a Nitinol-core Fiber

CAR/PDMS

	Intralot Variability		Interlot Variability
Fused silica	6.1%		9.3%
Nitinol	4.0%		7.4%

PDMS/DVB

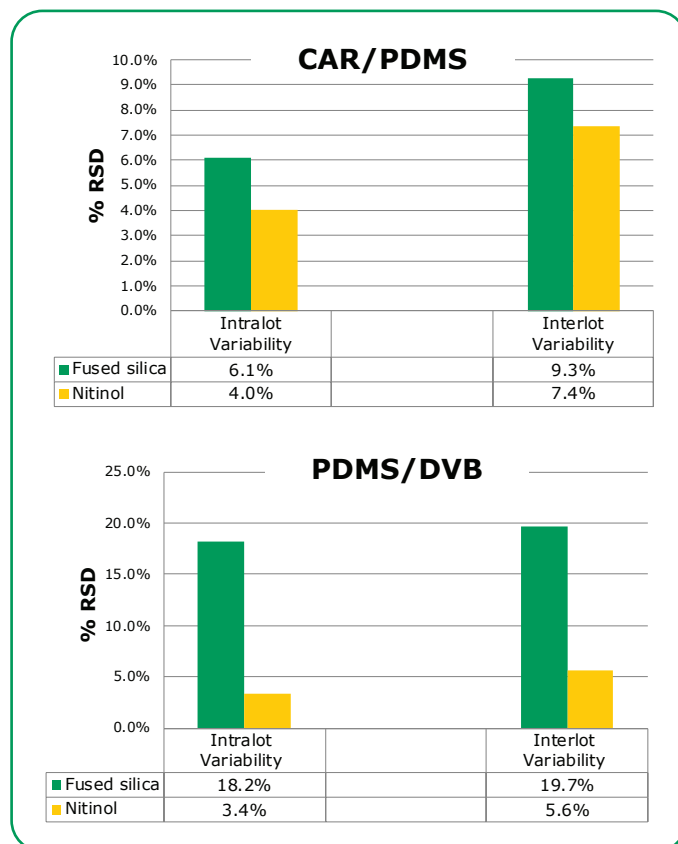|	Intralot Variability		Interlot Variability
Fused silica	18.2%		19.7%
Nitinol	3.4%		5.6%

Figure 8. Inter-lot and Intra-lot Reproducibility Testing Comparing NIT and FS SPME Fibers

Overcoated SPME (Matrix Compatible PDMS/DVB/PDMS SPME)

Complex matrices, such as many foods, pose a challenge to direct immersion SPME (DI-SPME) due to the presence of fats, sugars, pigments, and other matrix-specific compounds. These compounds can stick to the SPME fiber, in particular to particle fibers, and reduce its lifetime or be transferred to the GC where they may interfere with chromatographic analysis.

A new development in SPME fiber technology allows DI-SPME in complex matrix samples. Overcoated SPME, also known as matrix compatible PDMS/DVB/PDMS SPME, incorporates an additional protective PDMS overcoating on the fiber.

The PDMS overcoating seals the tip of the fiber, preventing wicking of matrix components and analytes into the fiber core. It also seals the connection of the fiber to the assembly, preventing fiber detachment due to corrosion by the sample. As a result, the overcoated fiber is physically more robust, and typically lasts two to three times longer than the conventional SPME equivalent.

The overcoating repels unwanted matrix components and allows for an efficient and quick wash step before analyte desorption into the GC. Washing or wiping the matrix components off of the fiber gives fewer interferences and decreases system fouling.

Overcoating also reduces competition between the analytes and matrix components for adsorption sites on the fiber's particle surface, resulting in increased fiber capacity/analyte extraction and improved sensitivity.
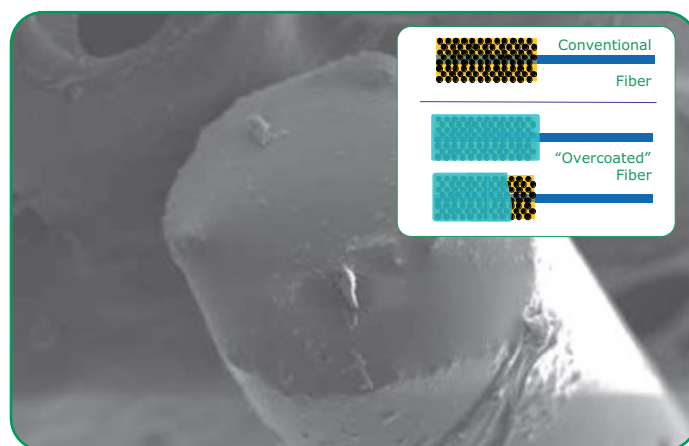
Figure 9. Overcoated SPME Fiber Assembly PDMS/DVB/PDMS (SEM Image Courtesy of E.A Sousa-Silva, University of Waterloo, Canada)

For more information on overcoated SPME, please visit
SigmaAldrich.com/spme-ocf

SPME Portable Field Sampler

The SPME portable field sampler provides an efficient and economical way of extracting and transporting volatile and semivolatile compounds from the field. Extracted compounds on the fiber are safely sealed off by a replaceable septum. The sampler can be reused up to 50-100 times (depending on the sample matrix), and is disposed of when the fiber is no longer usable. The portable field sampler also efficiently collects organic compounds from air. Three fiber phases are available: general purpose polydimethylsiloxane (PDMS); PDMS/Carboxen® for trace levels of volatiles; and PDMS/DVB for semi-volatiles and larger volatiles.

To see a video of the portable field sampler, visit
SigmaAldrich.com/spme-videos
and watch **"Portable Field Sampler"**

Figure 10. SPME Portable Field Sampler

SPME Troubleshooting

The following section gives some helpful troubleshooting suggestions to combat factors that may prove to be problematic in SPME extractions. Review these suggestions before performing method development work. For a more complete troubleshooting guide, please visit SigmaAldrich.com/SPME-troubleshoot

General Precautions for Using SPME Fibers

1. Keep in mind that there are a number of factors that may affect reproducibility, including:

   - Variable extraction time

   - Variable salt content

   - Variable pH

   - Variable content of organic solvent in spiked samples

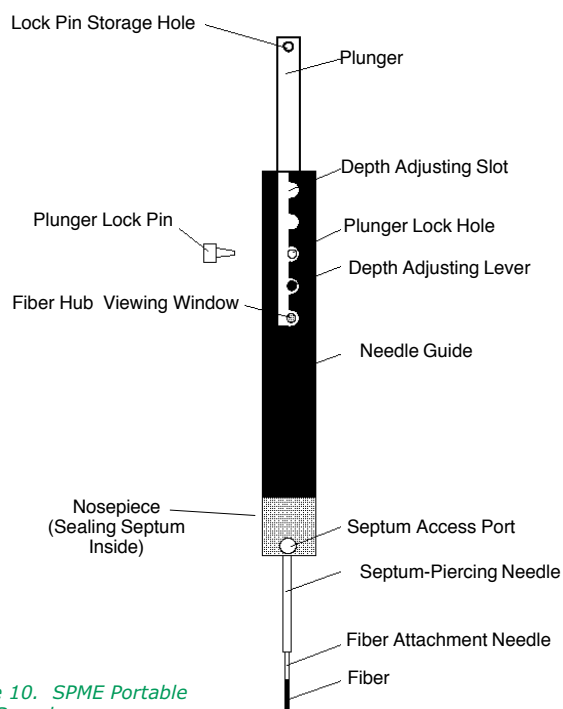   - Variable vial shape and headspace volumes

   - Inconsistent agitation speed during extraction

   - Heating of the stirring plate when using magnetic stirrers

   - Incorrect or irreproducible sampling depth inside a vial

2. Help to maintain reproducibility by:

   - Using an internal standard for extraction (add the internal standard to sample before extraction)

   - Keeping the organic solvent content the same for all samples when optimizing the method and constructing a calibration curve

   - Using the same type of vial for all samples in a batch and always piercing the vial in the same position (use recommended SPME vials)

   - Keeping sample agitation constant during extraction of one sample or multiple samples in a batch

   - Isolating the vial from the stirring plate by placing a septum between a vial and stirrer surface

   - Keeping the fiber sampling depth in the vial constant for all samples (coating exposure)

   - Avoiding splashing of the coating in HS-SPME during agitation

   - Immersing the coating entirely in the sample for DI-SPME (avoid piercing needle to contact the sample)

   - Avoiding sample heat from other ambient sources

3. Do not soak any SPME fiber in chlorinated solvents.

4. All SPME fiber coatings are bonded; however, bonded fibers will still swell in certain solvents. Polar fibers (e.g. polyacrylate) will swell more in the presence of polar solvents and, likewise, nonpolar fibers (e.g. PDMS) in the presence of nonpolar solvents. In some cases, the swelling can be enough that the coating will strip off when the fiber is retracted back into the needle. The swelling may occur in headspace and in immersion modes. For some samples, the organic compounds can be concentrated in the headspace and swell the fiber even more than if the fiber was immersed. It is important to determine compatibility of samples with the fiber coatings.

   If a fiber becomes contaminated after use, repeat the cleaning procedure listed in the "Preconditioning" section. If the contamination is severe, the fibers can be thermally cleaned for an extended period of time 20 °C below the listed conditioning temperature in **Table 3**. If this does not clean the fibers, solvent cleaning can be attempted. Please follow the guidelines for solvent cleaning of specific fiber coatings in the troubleshooting section entitled "Fiber Specific Precautions, Solvent Cleaning, and Compatibility."

5. To prevent fiber breakage when autosamplers are used:

   - Frequently check for proper alignment of the fiber needle with the injector of the GC-MS, vial trays, and vials. Improper alignment is one of the main causes of fiber breakage/damage.

   - Avoid stress of the needle by keeping agitation speed into the agitator/heater module of the autosampler below 600 rpm. Higher speeds can be used if the extraction time is short (< 10 min).

   - Be sure that the tension chords contained in some autosamplers are at the proper tension and functioning properly.

   - It is highly recommended to use screw capped vials with 1.3 to 1.5 mm silicone septa.

   - Make sure that the insertion depths into the injection port and vials are properly set.

   - It is recommended to use assemblies with 23 gauge needles for increased strength and less bending.

Blank Analysis

After conditioning the SPME fiber, it is advisable to run a fiber blank to gauge the amount of background. Prior to running a fiber blank analysis, be sure that the GC column has been thermally conditioned to the desired upper temperature of the method.

1. Create an appropriate GC method for SPME analysis of the samples. This same program will be used to run a blank analysis. The starting temperature of the oven program should be low enough to help focus the analytes onto the column (generally a temperature of < 50 °C works in most cases). After 1.5 - 5 minutes at the starting temperature the column oven temperature can be ramped at a rate(s) necessary to achieve the desired separation of analytes. For splitless injections (which are commonly used in SPME), set the vent to open after 1 - 3 minutes.

2. Insert the SPME fiber into the injection port at the appropriate depth and start the GC.

3. Run the GC program until completed.

4. There are typically some extraneous peaks in the initial runs.

5. Repeat the step again to see if there is a reduction in the size and number of peaks.

6. Depending upon the sensitivity of the instrument, the peaks may be extremely low in intensity. It is important to run a known sample to determine if the background peaks are relevant for the analysis.

7. Please note the fibers may introduce oxygen and water into the GC which may produce extraneous peaks.

8. Contact technical service to obtain assistance if the peaks are too large.

9. Also consider running a vial blank by performing SPME on a capped, empty vial. Use the same SPME parameters as for the samples. This will help to gauge the level of background coming from the septa used on the sample vials.

10. Store conditioned fibers clean (not in the box) by flushing a vial with clean nitrogen and close with a septum cap. "Inject" the fiber assembly into the vial without exposing the fiber. The opening of the fiber will only be exposed to clean nitrogen. If this is not possible, a small cap of any inert material e.g. Teflon® can be placed at the tip of the fiber assembly to protect the inner SPME coating. Before using a stored fiber, perform a blank run to recondition and check for cleanliness. If the fiber assembly has not been used for several hours, it is best to desorb the fiber with the split vent open for several minutes prior to extraction (potentially perform a blank run also).

SPME Troubleshooting

Fiber Specific Precautions, Solvent Cleaning, and Compatibility

PDMS (Polydimethylsiloxane) Absorbent Fiber Coatings

- For solvent cleaning, PDMS fibers can be immersed in water-soluble organic solvents such as methanol, acetonitrile, acetone or ethanol, especially if it is a mixture of water with the organic solvent. The addition of water helps to reduce swelling. Usually 15 - 30 min is sufficient to clean the fibers.

- Keep in mind that PDMS fibers may swell when exposed to silylating reagents.

- Do not place PDMS fibers in non-polar solvents or samples containing high levels of non-polar solvents such as hexane, methylene chloride and diethyl ether.

- Heated headspace extraction of samples with high concentration (>100 ppm) of non-polar solvents and terpenes can swell PDMS coatings. The 30 µm PDMS is less likely to be stripped than the 100 µm PDMS when the fiber is retracted. Consider this fiber as an option when evaluating such samples.

PEG (Polyethylene glycol, CARBOWAX®) Fiber Coatings

- For solvent-cleaning PEG fibers, place the fibers in a 1% methanol:water solution containing a minimum of 15% sodium chloride (NaCl) for 15 to 30 minutes. It is important to have the salt present when soaking to reduce swelling of the PEG coating.

- PEG fibers can be immersed in hydrocarbon solvents and will not swell.

- It is highly recommended that PEG fibers are not to be immersed in aqueous samples with water-soluble organic concentrations above 1% (total water soluble organic) unless the sample contains at least 15% NaCl or other salts. The degree of swelling will vary depending upon the solvent(s) in the water. In many cases there will not be sufficient swelling to damage the fiber, but in some cases the fiber coating can be stripped or damaged when the fiber is retracted.

- It is recommend that the PEG fiber should not be exposed to the headspace of samples with a water-soluble organic concentration higher than 2% v/v. The organic analytes will be concentrated in the heated headspace and can swell the phase that can result in stripping when the fiber is retracted into the needle.

- Methanol may be produced when the fiber is exposed to acidic samples. This is due to the presence of an inhibitor in the starting material. Most of the inhibitor has been removed, but several additional extractions in an acidic solution will remove any remaining inhibitor. Solvent cleaning (first point) is usually sufficient for removal of the inhibitor from the fiber coating.

Polyacrylate Fiber Coatings

- For cleaning, soak the fiber in a water miscible organic solvent (e.g. methanol for 30 min), followed by immersion in water to reduce any swelling. It is best to place the fiber in water prior to retracting the fiber.

- The polyacrylate fiber can be immersed in aliphatic hydrocarbon solvents (e.g. hexane, heptane) without swelling.

- Polyacrylate coating may darken with use. This is not unusual and does not affect fiber performance unless the coating becomes black. This indicates that oxygen is present in the injection port. If the fiber is desorbed at 280 °C or lower, the discoloration will be lessened.

Adsorbent/Particle Type Fibers

- Carboxen® adsorbent containing SPME fibers can retain solvents in the micropores, so it is generally not advisable to soak this fiber in solvents. It could take multiple desorption cycles to remove the solvent. Fibers will not swell in water-soluble organic solvents to an appreciable degree. To quickly clean the fiber from strong contamination, one may expose it to the headspace above a solvent (e.g. benzene or toluene), wait for 10-15 min, and then perform repeated thermal desorption.

- For PDMS/DVB fibers, follow the guidelines for PDMS fibers. Methanol is the best option.

Overcoated (OC) SPME Fiber Assemblies

- OC fiber assemblies have a thin layer of PDMS applied over the adsorbent coating. The overcoat provides a barrier to non-volatile matrix components and increases the durability of the adsorbent coating on the fiber.

- To help further lengthen the fiber coating life, the fiber coating should be rinsed after each extraction by dipping the fiber into clean water for 10 - 30 seconds prior to desorption. For very complex matrices, it is recommended to wipe the fiber with clean water. The rinse/wipe step will help remove sugars and other water-soluble, non-volatile components. The length of the rinse time is dependent upon the volatility of the analytes that are being sampled.

- The overcoat changes the extraction properties of the adsorbent coating by slightly reducing fiber affinity/selectivity for polar analytes and slightly increasing equilibrium time. For many analytes, the changes in extraction properties between the OC and standard adsorbent coatings are very subtle.

SPME in Official Methods
(a Selection)

International Standards/Methods

ASTM D6438-05 (2015)
Standard Test Method for Acetone, Methyl Acetate, and Parachlorobenzotrifluoride Content of Paints, and Coatings by Solid Phase Microextraction-Gas Chromatography

ASTM D6520-18
Standard Practice for the Solid Phase Micro Extraction (SPME) of Water and its Headspace for the Analysis of Volatile and Semi-Volatile Organic Compounds

ASTM D6889-03 (2017)
Standard Practice for Fast Screening for Volatile Organic Compounds in Water Using Solid Phase Microextraction (SPME)

ASTM D7363-13a
Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using SPME and GC-MS in Selected Ion Monitoring Mode

ASTM E2154-15a
Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with SPME

ISO 17943:2016
Water Quality – Determination of Volatile Organic Compounds in Water – Method using Headspace Solid Phase Microextraction (HS-SPME)- followed by Gas Chromatography- Mass Spectrometry (GC-MS)

ISO 27108:2010
Water Quality – Determination of Selected Plant Treatment Agents and Biocide Products – Method using Solid Phase Microextraction (SPME)- followed by Gas Chromatography- Mass Spectrometry (GC-MS)

Standard Methods – 6040D and E
Solid-phase microextraction (SPME) techniques for taste- and odor-causing compounds (6040 CONSTITUENT CONCENTRATION BY GAS EXTRACTION (2017)", Standard Methods For the Examination of Water and Wastewater, DOI: 10.2105/ SMWW.2882.117)

National Standards/Methods

EPA Method 8272 (USA)
Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water by SPME GC/MS

GB/T 24572.4-2009 (China)
Standard practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples. Part 4: Solid Phase Microextraction (SPME)

GB/T 24281-2009 (China)
Textiles - Determination of Volatile Organic Compounds. SPME / Gas Chromatography/Mass Spectrography

GB/T 32470-2016 (China)
Organic Compounds in Drinking Water—Test Methods of Geosmin and 2-Methylisoborneol

SN/T 3626-2013 (China)
Determination of Hexanal in Edible Oil for Export. Headspace-SPME-GC Method

OENORM A 1117, 2004-05-01 (Austria)
Determination of Volatile Compounds in Cellulose-Based Materials by Solid Phase Micro Extraction (SPME

UNICHIM 2237:09 (Italy)
Ambienti di Lavoro – Determinazione delle aldeidi aerodisperse - Metodo per microestrazione su fase solida (SPME) ed analisi mediante gascromatografia accoppiata a spettrometria di massa (GC-MS)

(Determination of air borne aldehydes by SPME/GC-MS using derivatisation on fiber with PFBHA)

SPME Product Offering and Related Products

SPME Fiber Holders

Description	Cat. No.
SPME Fiber Holders	
For use with manual sampling	**57330-U**
For use with Non-CTC CombiPAL™ Type Autosampler	**57331**
For use with CTC CombiPAL™, GERSTEL® MPS2 and Thermo® TriPlus Autosamplers	**57347-U**

SPME Fibers

In packs of 3, if not indicated differently.

Fiber Coating and Thickness	Fiber Core/ Assembly Type#	Hub Description	Sampling Mode and Needle Size			
			Manual Holder (w/spring)		Autosampler	
			23 Ga*	24 Ga*	23 Ga*	24 Ga*
Polydimethylsiloxane (PDMS)						
7 µm PDMS	Fused Silica/SS	Green/plain	—	57302	57291-U	57303
30 µm PDMS	Fused Silica/SS	Yellow/plain	—	57308	57289-U	57309
100 µm PDMS	Metal alloy/Metal alloy**	Red/plain	—	—	57928-U	—
100 µm PDMS	Fused Silica/SS	Red/plain	57342-U	57300-U	57341-U	57301
Polyacrylate						
85 µm Polyacrylate	Fused Silica/SS	White/plain	—	57304	57294-U	57305
Polyethylene Glycol (PEG)						
60 µm PEG	Metal alloy/SS	Purple/plain	57355-U	—	57354-U	—
Polydimethylsiloxane/Divinylbenzene (PDMS/DVB)						
65 µm PDMS/DVB	Metal alloy/Metal alloy**	Pink/plain	—	—	57902-U	—
65 µm PDMS/DVB	Fused Silica/SS	Blue/plain	57346-U	57310-U	57345-U	57311
65 µm PDMS/DVB	StableFlex™/SS	Pink/plain	—	57326-U	57293-U	57327-U
65/10 µm PDMS/DVB-OC***	StableFlex™/SS	Pink/notched	—	—	57439-U	—
65 µm PDMS/DVB	Nitinol/SS	Blue metallic	57916-U	57921-U	57923-U	57931-U
Carboxen®/Polydimethylsiloxane (CAR/PDMS)						
75 µm CAR/PDMS	Fused Silica/SS	Black/plain	57344-U	57318	57343-U	57319
85 µm CAR/PDMS	Metal alloy/Metal alloy**	Lt. Blue/plain	—	—	57906-U	—
85 µm CAR/PDMS	StableFlex™/SS	Lt. Blue/plain	—	57334-U	57295-U	57335-U
75 µm CAR/PDMS	Nitinol/SS	Black Metallic	57901-U	57904-U	57907-U	57908-U
Divinylbenzene/Carboxen/Polydimethylsiloxane						
50/30 µm DVB/CAR/PDMS	Metal alloy/Metal alloy (1 cm)**	Gray/plain	—	—	57912-U	—
50/30 µm DVB/CAR/PDMS	Metal alloy/Metal alloy (2 cm)**	Gray/notched	—	—	57914-U	—
50/30 µm DVB/CAR/PDMS	StableFlex™/SS (1 cm)	Gray/plain	—	57328-U	57298-U	57329-U
50/30 µm DVB/CAR/PDMS	StableFlex™/SS (2 cm)	Gray/notched	—	—	57299-U	57348-U
Bare Fused Silica						
Bare Fused Silica	Fused Silica/SS	Orange/plain	—	57316-U	—	—

* Ga – Needle gauge.
** Metal alloy/Matal alloy fiber assemblies are provided 1/pk.
*** PDMS overcoated.
\# SS - Stainless Steel.

Smart SPME Fibers

1 Fiber / Pack

Fiber Coating and Thickness	Fiber Core/ Assembly Type#	Hub Description	Sampling Mode and Needle Size
			Autosampler
			23 Ga
Polydimethylsiloxane (PDMS)			
7 μm PDMS	Fused Silica/SS	Light Green/Smart	**548552-U**
30 μm PDMS	Fused Silica/SS	Yellow/Smart	**548553-U**
100 μm PDMS	Fused Silica/SS	Red/Smart	**548575-U**
Polyacrylate			
85 μm Polyacrylate	Fused Silica/SS	Silver/Smart	**548652-U**
Polyethylene Glycol (PEG)			
60 μm PEG	Metal alloy/SS	Purple/Smart	**548676-U**
Polydimethylsiloxane/Divinylbenzene (PDMS/DVB)			
65 μm PDMS/DVB	Fused Silica/SS	Blue/Smart	**548576-U**
65 μm PDMS/DVB	StableFlex™/SS	Light Red/Smart	**548650-U**
65/10 μm PDMS/DVB-OC*	StableFlex™/SS	Gold/Smart	**548651-U**
Carboxen®/Polydimethylsiloxane (CAR/PDMS)			
75 μm CAR/PDMS	Fused Silica/SS	Black/Smart	**548550-U**
85 μm CAR/PDMS	StableFlex™/SS	Light Blue/Smart	**548551-U**
Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS)			
50/30 μm DVB/CAR/PDMS	StableFlex™/SS	Dark Gray/Smart	**548653-U**
50/30 μm DVB/CAR/PDMS, 2 cm Fiber	StableFlex™/SS	Light Gray/Smart	**548675-U**

* PDMS overcoated.
\# SS - Stainless Steel

More information and resources can be found on SigmaAldrich.com/SPME

Fiber Selection Guide

Performance Tips

SPME Videos and Webinars

Literature

Frequently Asked Questions

Custom Fiber Request

SPME Product Offering and Related Products

SPME Fiber Assortment Kits

The SPME fiber assortment kits consist of 1 fiber each of the types listed below.

Description		Cat. No.
SPME Fiber Assortment Kit 1: For Volatiles and Semi-volatiles		
• 85 µm polyacrylate coated fiber • 100 µm PDMS coated fiber • 7 µm PDMS coated fiber	For use with manual holder, needle size 24 ga	**57306**
	For use with autosampler, needle size 24 ga	**57307**
	For use with autosampler, needle size 23 ga	**57285-U**
SPME Fiber Assortment Kit 2: For Volatile or Polar Organics in Water		
• 75 µm Carboxen®/PDMS coated fiber • 65 µm PDMS/DVB coated fiber • 85 µm Polyacrylate coated fiber	For use with manual holder, needle size 24 ga	**57320-U**
	For use with autosampler, needle size 24 ga	**57321-U**
	For use with autosampler, needle size 23 ga	**57286-U**
SPME Fiber Assortment Kit 3		
• 60 µm PDMS/DVB coated fiber • 85 µm Polyacrylate coated fiber • 100 µm PDMS coated fiber	For use with autosampler, needle size 24 ga	**57323-U**
SPME Fiber Assortment Kit 4: For Flavors and Odors		
• 100 µm PDMS coated fiber • 65 µm PDMS/DVB coated fiber • 75 µm Carboxen®/PDMS coated fiber	For use with manual holder, needle size 24 ga	**57324-U**
	For use with autosampler, needle size 24 ga	**57325-U**
	For use with autosampler, needle size 23 ga	**57287-U**
SPME Fiber Assortment Kit 5: For Flavors and Odors		
• 100 µm PDMS coated fiber • 65 µm PDMS/DVB coated fiber • 85 µm Carboxen®/PDMS coated fiber • 50 µm/30 µm DVB/PDMS coated fiber	For use with autosampler, needle size 23 ga	**57362-U**
SPME StableFlex™ Fiber Assortment Kit		
• 65 µm PDMS/DVB coated fiber • 50 µm/30 µm DVB/Carboxen®/PDMS coated fiber • 85 µm Carboxen®/PDMS coated fiber • 85 µm Polyacrylate coated fiber	For use with manual holder, needle size 24 ga	**57550-U**
	For use with autosampler, needle size 24 ga	**57551-U**
	For use with autosampler, needle size 23 ga	**57284-U**

SPME Portable Field Samplers and Accessories

Description	Cat. No.
Portable Field Samplers (see also p. 15 for more details)	
Coating 100 µm Polydimethylsiloxane (PDMS)	**504823**
Coating 85 µm Carboxen®/Polydimethylsiloxane (CAR/PDMS)	**504831**
Coating 65 µm Polydimethylsiloxane/Divinylbenzene (PDMS/DVB)	**57359-U**
Thermogreen® LB-2 Replacement Septa, solid discs diam. 5 mm (3/16 in.), pkg of 50	**20638**

SPME Accessories

GC Inlet Liners

Description	Cat. No.
Agilent®/HP Inlet Liners, Direct (SPME) Type	
Straight Design (unpacked), 78.5 mm length x 6.5 mm O.D. x 0.75 mm I.D., pkg of 1	2637501
Straight Design (unpacked), 78.5 mm length x 6.5 mm O.D. x 0.75 mm I.D., pkg of 5	2637505
Straight Design (unpacked), 78.5 mm length x 6.5 mm O.D. x 0.75 mm I.D., pkg of 25	2637525
PerkinElmer® Inlet Liners, Direct (SPME) Type	
Straight Design (unpacked), 92 mm length x 6.35 mm O.D. x 0.75 mm I.D., pkg of 5	2631205
Shimadzu™ Inlet Liners, Direct (SPME) Type	
Straight Design (unpacked), 95 mm length x 5.0 mm O.D. x 0.75 mm I.D., pkg of 1	2633901
Straight Design (unpacked), 95 mm length x 5.0 mm O.D. x 0.75 mm I.D., pkg of 5	2633905
Straight Design (unpacked), 95 mm length x 5.0 mm O.D. x 0.75 mm I.D., pkg of 25	2633925
Straight Design (unpacked), 99 mm length x 5.0 mm O.D. x 0.75 mm I.D., pkg of 1	2633501
Straight Design (unpacked), 99 mm length x 5.0 mm O.D. x 0.75 mm I.D., pkg of 5	2633501
Straight Design (unpacked), 127 mm length x 5.0 mm O.D. x 0.75 mm I.D., pkg of 1	2632901
Straight Design (unpacked), 127 mm length x 5.0 mm O.D. x 0.75 mm I.D., pkg of 5	2632901
Varian® Inlet Liners, Direct (SPME) Type	
Straight Design (unpacked), 54 mm length x 5.0 mm O.D. x 0.8 mm I.D., pkg of 1	2637801
Straight Design (unpacked), 54 mm length x 5.0 mm O.D. x 0.8 mm I.D., pkg of 5	2637805
Straight Design (unpacked), 54 mm length x 4.6 mm O.D. x 0.8 mm I.D., pkg of 1	2636401
Straight Design (unpacked), 54 mm length x 4.6 mm O.D. x 0.8 mm I.D., pkg of 5	2636405
Straight Design (unpacked), 54 mm length x 4.6 mm O.D. x 0.8 mm I.D., pkg of 25	2636425
Straight Design (unpacked), 74 mm length x 6.35 mm O.D. x 0.75 mm I.D., pkg of 1	2635801
Straight Design (unpacked), 74 mm length x 6.35 mm O.D. x 0.75 mm I.D., pkg of 5	2635805
Straight Design (unpacked), 74 mm length x 6.35 mm O.D. x 0.75 mm I.D., pkg of 25	2635825

GC Septa

Description	Cat. No.
Molded Thermogreen® LB-2 Septa with Injection Hole	
diam. 9.5 mm, pkg of 50	28331-U
diam. 9.5 mm, pkg of 250	28332-U
diam. 10 mm, pkg of 50	28333-U
diam. 10 mm, pkg of 250	28334-U
diam. 11 mm, pkg of 50	28336-U
diam. 11 mm, pkg of 250	28338-U
diam. 11.5 mm, pkg of 50	29446-U
diam. 11.5 mm, pkg of 250	29448-U
diam. 17 mm, pkg of 50	29452-U
diam. 17 mm, pkg of 250	29453-U

SPME Product Offering and Related Products

SPME Accessories
Manual Sampling Accessories

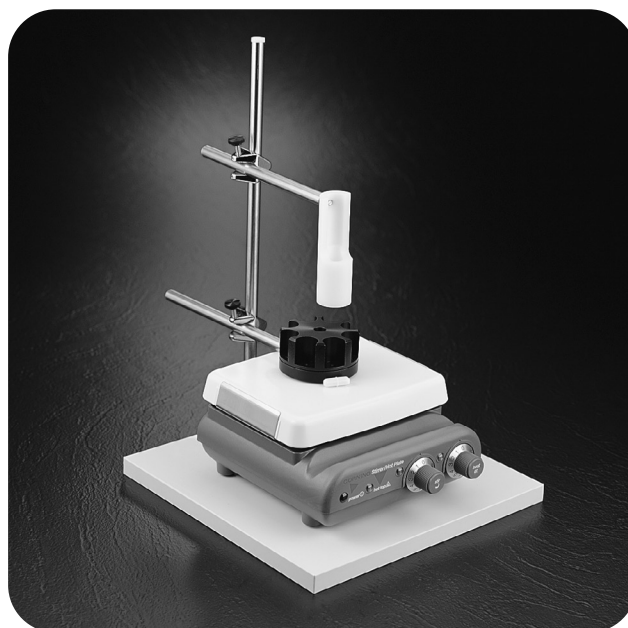| Description	Cat. No.
SPME Sampling Stand	
Corning® hotplate and stirrer with digital display AC input 120 V, US 3-pin plug, plate L × W 5 in. × 7 in.	**CLS6795420D**
Corning® hotplate and stirrer with digital display AC input 230 V AC (CEE7-7 plug), plate L × W 5 in. × 7 in.	**CLS6798420D**
Heater block for 28 mm diameter vials (40 mL)	**33313-U**
IKA® ETS-D5 temperature controller electronic contact thermometer with 3 operating modes, stainless steel, 1/cs	**Z645125**
Spinbar® magnetic stirring fleas blue, L × diam. 10 mm × 3 mm	**Z118877**
SPME Sampling Stand for use with 4 mL vials	**57333-U**
SPME Sampling Stand for use with 15 mL vials	**57357-U**
SPME sampling stand holder & rod assembly for use with SPME Sampling Stand	**57364-U**
Thermometer L 5 in., parameter -10-110 °C temperature	**57332**
Vial puck, 15 mL, for SPME Sampling Stand made to hold 8 × 15 mL vials	**57358-U**
Injection Support	
SPME Inlet Guide (supports manual holder during desoption)	**57356-U**

Figure 10. SPME Sampling Stand for 15mL vials Cat.No. 57357-U
(Spinbar, hotplate and stirrer not included, please order separately)

Vials

Description	Cat. No.
Vials - 2 mL, 10 mL (e.g. for Agilent® Autosamplers)	
Crimp Top	
Vials, crimp top, for Thin Seal volume 10 mL, clear glass vial, O.D. × H 23 mm × 46 mm, crimp top (0.125 in. thick) for thin septa, pkg of 36	**27385**
Crimp seals with Viton® septa silver aluminum seal, seal diam. 20 mm, black Viton® septum, thickness 0.76 mm, pkg of 36	**33146-U**
Crimp seals with Viton® septa silver aluminum seal, diam. 20 mm, open center, 8 mm center hole, black Viton® septum, thickness 0.76 mm, pkg of 100	**27245**
Crimp seals with Viton® septa aluminum seal, open center, seal diam. 20 mm, 8 mm center hole, black Viton® septum, diam. × thickness 20 mm × 0.76 mm, pkg of 288	**28298-U**
Screw Top	
Vials, screw top with black polypropylene hole cap (10-425 thread), large opening, pre-assembled volume 2 mL, clear glass vial, red PTFE/silicone septum, pkg of 100	**27531**
Vials, screw top with black polypropylene hole cap (10-425 thread), large opening, pre-assembled volume 2 mL, amber glass vial, red PTFE/silicone septum, pkg of 100	**27532**
Headspace Vials for CTC Autosampler - 10 mL, 20 mL	
Crimp Top	
Headspace vial, beveled top, rounded bottom volume 10 mL, clear glass vial beveled finish, O.D. × H 22.6 mm × 46 mm, round bottom, pkg of 100	**27294**
Headspace vial, beveled top, rounded bottom volume 20 mL, clear glass vial beveled finish, O.D. × H 22.6 mm × 75 mm, pkg of 100	**27296**
Vials, crimp top, for Thin Seal volume 20 mL, clear glass vial (flat top), closure type, crimp top vial, O.D. × H 22.5 mm × 75.5 mm, pkg of 100	**SU860104**
Crimp seals (magnetic) with PTFE/silicone septa, pkg/100 silver steel (magnetic, with 5 mm center hole), PTFE/silicone, seal diam. × total thickness 20 mm × 3 mm, pkg of 100	**27300**
Crimp seals with Viton® septa silver seal, magnetic (with 8 mm center hole), diam. × thickness 20 mm × 1.0 mm, black Viton® septum, pkg of 100	**SU860106**
Screw Top	
Headspace vial, screw top, rounded bottom (vial only) volume 10 mL, clear glass vial, thread 18, O.D. × H 22.5 mm × 46 mm, pkg of 100	**SU860099**
Headspace vial, screw top, rounded bottom (vial only) volume 10 mL, amber glass vial, thread 18, O.D. × H 22.5 mm × 46 mm, pkg of 100	**SU860100**
Headspace vial, screw top, rounded bottom (vial only) volume 20 mL, clear glass vial, thread 18, O.D. × H 22.5 mm × 75.5 mm, pkg of 100	**SU860097**
Headspace vial, screw top, rounded bottom (vial only) volume 20 mL, amber glass vial, thread 18, O.D. × H 22.5 mm × 75.5 mm, pkg of 100	**SU860098**
Magnetic Screw Cap for Headspace Vials, 18 mm thread PTFE/silicone septum (white PTFE/transparent blue silicone), septum thickness 1.3 mm, pkg of 100	**SU860101**
Magnetic Screw Cap for Headspace Vials, 18 mm thread PTFE/silicone septum (white PTFE/blue silicone), septum thickness 1.5 mm, pkg of 100	**SU860103**
Vials for SPME Sampling Stand - 4 mL, 15 mL	
Vials, screw top, amber glass (vial only) volume 4 mL, amber glass vial, thread 13-425, pkg of 100	**27115-U**
Vials, screw top, clear glass (vial only) volume 4 mL, clear glass vial, O.D. × H × I.D. 15 mm × 45 mm × 8 mm, thread 13-425, pkg of 10	**27111**
Vials, screw top, silane-treated volume 4 mL, clear glass vial, O.D. × H × I.D. 15 mm × 45 mm × 8 mm, thread 13-425, pkg of 100	**27114**
Vials, screw top, silane-treated volume 4 mL, amber glass vial, O.D. × H × I.D. 15 mm × 45 mm × 8 mm, thread 13-425, pkg of 100	**27217**
Vials, screw top, silane-treated volume 4 mL, clear glass vial, O.D. × H × I.D. 15 mm × 45 mm × 8 mm, thread 13-425, pkg of 1000	**27220-U**
Vials, screw top with phenolic open-top cap, pre-assembled volume 4 mL, clear glass vial, O.D. × H 15 mm × 45 mm, tan PTFE/silicone septum, pkg of 100	**27136**
Vials, screw top with phenolic open-top cap, pre-assembled volume 15 mL, clear glass vial, O.D. × H 21 mm × 70 mm, tan PTFE/silicone septum, pkg of 100	**27159**
Vials, screw top with phenolic open-top cap, pre-assembled volume 4 mL, amber glass vial, O.D. × H 15 mm × 45 mm, tan PTFE/silicone septum, pkg of 100	**27006**
Vials, screw top with phenolic open-top cap, pre-assembled volume 15 mL, amber glass vial, O.D. × H 21 mm × 70 mm, tan PTFE/silicone septum, pkg of 100	**27008**
Screw cap, phenolic, with open center black phenolic cap, for use with 4 mL vial with 13-425 thread, pkg of 100	**27120-U**
Septa, Viton black Viton®, diam. × thickness 11 mm × 0.060 in., for use with 4 mL vial, pkg of 100	**27351**
Septa, white PTFE/silicone diam. × thickness 11 mm × 0.075 in., for use with 4 mL vial, pkg of 100	**27356**

SPME Product Offering and Related Products

SPME Accessories

Description	Cat. No.
Vials for Heating Block for 28 mm diameter vials (33313-U) - 40 mL	
Vials, screw top, amber glass (vial only) volume 40 mL, amber glass vial, thread 24-400, pkg of 100	27185-U
Vials, screw top, clear glass (vial only) volume 40 mL, clear glass vial, thread 24-400, pkg of 100	27184
Vials, screw top with phenolic open-top cap, pre-assembled volume 40 mL, clear glass vial, O.D. × H 29 mm × 82 mm, tan PTFE/silicone septum, pkg of 100	27180
Vials, screw top with phenolic open-top cap, pre-assembled volume 40 mL, amber glass vial, O.D. × H 29 mm × 82 mm, tan PTFE/silicone septum, pkg of 100	27010-U
Screw cap, phenolic, with open center black phenolic hole cap, for use with 20 or 40 mL vial with 24-400 thread, pkg of 100	27187
Septa, tan PTFE/silicone white tan PTFE/silicone, diam. × thickness 22 mm × 0.100 in., for use with 20, 40 or 60 mL vial, pkg of 100	27188-U
Septa, Viton® black Viton®, diam. × thickness 22 mm × 0.060 in., for use with 20, 40, or 60 mL vial, pkg of 100	27355

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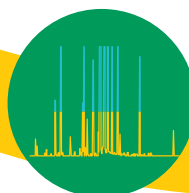
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