Supelco®

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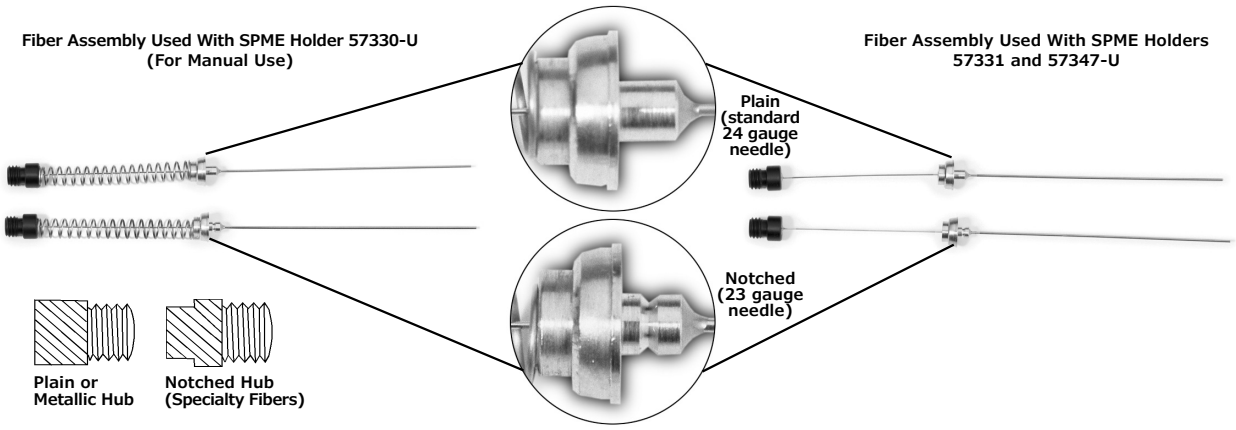
Solid Phase Microextraction Fiber Assemblies



Conditioning and Thermal Cleaning of SPME Fibers

See notes prior to starting thermal conditioning and using SPME Fibers.

Note 1: When conditioning in the GC injection port, be sure to open splitter to reduce the amount of impurities entering the column. Always ramp the oven temperature after fiber conditioning to remove any contaminants that may have entered the column.

Note 2: Make sure that the injection port contains an appropriate liner. We strongly recommend that you insert a liner designed for SPME use with a narrow I.D. (between 0.75 mm and 1.0 mm I.D.) designed for that particular GC inlet. Do not insert an SPME fiber into a liner containing glass wool. If the fiber contacts the wool, the coating could be damaged.

1. Follow the conditioning guidelines in **Table 1** to thermally clean the SPME fibers before use.
2. Insert the fiber into the injection port at the appropriate needle depth by adjusting the black needle guide/depth gauge so that the top is between 3.5 to 4 on the vernier gauge on the holder.
3. If a fiber becomes contaminated after use, these steps can be repeated if necessary. If the contamination is severe you can thermally clean the fibers for an extended period of time at a temperature 20 °C below the listed conditioning temperature in **Table 1**.
4. If this does not clean the fibers, solvent cleaning can be attempted. Please follow the guidelines for solvent cleaning of specific fiber coatings (see back).

Fiber Coating and Thickness	Fiber Core/Assembly Type	Hub Description	Sampling and Analysis Mode			
			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	Nitinol/SS	Blue Metallic	57916-U	57921-U	57923-U	57931-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	—
Carboxen®/Polydimethylsiloxane (CAR/PDMS)						
75 µm CAR/PDMS	Fused Silica/SS	Black/plain	57344-U	57318	57343-U	57319
75 µm CAR/PDMS	Nitinol/SS	Black Metallic	57901-U	57904-U	57907-U	57908-U
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
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 fiber assemblies are provided 1/pk.
***PDMS overcoated (OC)—see back side for description.

Table 1. Temperature, pH and Conditioning Guidelines for SPME Fiber Coatings

Fiber Coating	Film Thickness	pH	Maximum Temperature (°C)	Recommended Operating Temperature (°C)	Conditioning Temperature (°C)	Conditioning Time (Hrs)
PDMS	100 µm	2–10	280	200–280	250	0.5
PDMS	30 µm	2–11	280	200–280	250	0.5
PDMS	7 µm	2–11	340	220–320	320	1
PDMS/DVB (+OC)	65 µm (+10 µm)	2–11	270	200–270	250	0.5
Polyacrylate	85 µm	2–11	300	220–280	280	0.5
Carboxen®/PDMS	All	2–11	320	250–310	300	0.5
PEG	60 µm	2–9	250	200–250	240	0.5
DVB/CAR/PDMS	50/30 µm	2–11	270	230–270	270	0.5

General Precautions, Solvent Cleaning and Compatability for all SPME Fiber Coatings

Note 1: Do not soak any SPME fiber in chlorinated solvents.

Note 2: All SPME fiber coatings are bonded; however, bonded fibers will still swell in certain solvents. In some cases the swelling is sufficient that when the fiber is retracted into the needle, the needle can strip off the fiber coating. The swelling may occur in both headspace and immersion modes. In some samples the organics 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.

PDMS (Polydimethylsiloxane) Absorbent Fiber Coatings

1. For solvent cleaning, PDMS fibers can be immersed in water-soluble organic solvents such as methanol, acetonitrile, acetone or ethanol, or 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.
2. 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.
3. 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 Coating

1. For solvent cleaning PEG fibers, place the fibers in a 1% methanol:water solution containing a minimum of 15% NaCl for 15 to 30 min. It is important to have the salt present when soaking to reduce swelling of the PEG coating.
2. PEG fibers can be immersed in hydrocarbon solvents and will not swell.
3. It is highly recommended that PEG fibers not 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.
4. 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.
5. 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 that may be present. Solvent cleaning (see Note 1) is usually sufficient for removal of the inhibitor from the fiber coating.

Polyacrylate Fiber Coating

1. 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.
2. The polyacrylate fiber can be immersed in aliphatic hydrocarbon solvents (e.g. hexane, heptane) without swelling.
3. 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 coloration will be lessened.

Adsorbent/Particle Type Fibers

1. Carboxen® 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 greatly swell in water-soluble organic solvents to an appreciable degree.
2. 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 PDMS overcoat provides a barrier to non-volatile matrix components, lessens matrix deposition, prevents damage of the tip of the fiber coating from wicking, and increases the durability of the adsorbent coating on the fiber. The PDMS overcoat can often double or triple the life of the fiber coating, depending upon the matrix.

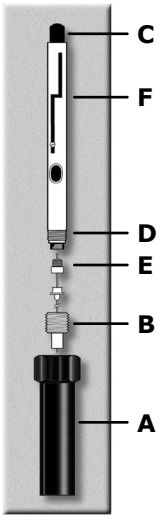
To help further lengthen the fiber coating life, the fiber coating should be rinsed after each extraction by dipping the fiber into clean water for 10–30 seconds prior to desorption. The rinse step will help remove sugars and other water-soluble, non-volatile components. The length of the rinse time is dependent upon the volatility of the components 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 is very subtle.

Attaching the Fiber Assembly to the Holder

To begin to use SPME, attach the fiber assembly to the SPME holder. Follow these simple steps.

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 (F) 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 (F) 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 (F) and draw it back.



The fiber assembly is attached the same way to the autosampler holder. Remove hexagonal nut and push black plunger down to expose threaded port for fiber assembly attachment. No spring is used with the autosampler 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.

Blank Analysis

Prior to running a fiber blank analysis, be sure that the GC column has been thermally cleaned to the desired upper temperature of your method.

1. Create an appropriate GC method for SPME analysis of your samples. This same program will be used to run a blank analysis. The starting temperature should be a maximum of 50 °C. After 1.5–5 minutes at the starting temperature, the column oven temperature can be ramped at rate(s) necessary to achieve the analyte separation. For splitless injections, set the vent to open after 1 min.
2. Insert the SPME fiber into the injection port at the appropriate depth and start the GC.
3. Run GC program until completed
4. There will typically be 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. (if not, consider an additional conditioning)
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 your peaks are too large.

For more resources, please visit **SigmaAldrich.com/spme**

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