SUPELCO®

595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Telephone 800-247-6628 ● 814-359-3441
Fax 800-447-3044 ● 814-359-3044
email: supelco@sial.com
sigma-aldrich.com/supelco

Bulletin 928A

# Solid Phase Microextraction Troubleshooting Guide

## How to Locate & Solve Problems

***Solid phase microextraction (SPME) is an innovative, solvent free technology that is fast, economical, and versatile. SPME has gained wide spread acceptance as the technique of preference for many applications. As with any analytical process however, problems occur on occasion. The most important step in correcting a problem when it occurs is identifying the root cause of the problem without wasting time. The systematic approach to troubleshooting described in this guide will allow you a quick solution to many problems. The guide contains helpful tips to prevent problems before they occur, as well as, a troubleshooting table listing the symptoms of the common problems, the possible causes, and suggested remedies. By following these recommendations, you can save valuable time and money.***

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## Troubleshooting Suggestions

Make troubleshooting faster and easier by closely observing and keeping complete records of your analytical conditions. Understanding the system performance history, as related to the fiber, sampling, desorption, inlet, column, detector response, etc., is important for effective troubleshooting. Thorough documentation of system maintenance (fiber changes, inlet liner changes, etc.) are equally important when determining what variables have changed and when.

Troubleshooting is more effective when you have on hand the following items:

- New backup fibers
- New backup column
- Pre-tested or control fiber with known performance
- Pre-tested or control column with known performance
- Spare injection port septa and liners
- Spare sampling vials and septa
- Spare SPME holder
- All associated product instruction sheets and instrument manuals

## Isolating the Problem Source

### Establish a Systematic Approach

Carefully note the symptoms you are encountering (e.g., no peaks detected, extraneous peaks detected, etc.), then find these in the troubleshooting table (page 6). Next to each symptom is listed several possible causes for what is being observed. Review the possible causes and systematically address each one through the remedy listed. Start your elimination process with the most probable cause based upon your specific situation, then work through the remedies systematically. A shotgun approach to applying the remedies, while seeming to be the fastest approach, is usually the least effective. Understanding the cause and effect relationship with the changes made and determining the actual root cause of the problem is the most beneficial and effective approach.

The troubleshooting table contains most of the problems you will encounter with SPME, but we cannot anticipate every situation or application. If you experience a problem not covered in the table, you can still determine the cause and remedy by systematically isolating the problem into one of four areas: sampling, desorption, analysis, or product. The following is a general scheme for isolating the problem:

**Step 1:** Eliminate the sampling, desorption, and SPME fiber from your analytical process by directly injecting a known reference standard containing your compounds of interest. This will give you an analysis focused test that provides valuable data on the chromatographic system's performance.

If the results show the problem persists, then focus your attention on the injection port, column, or detector (maintenance, replacement, etc.)

If the problem disappears and your normal operating performance returns, then go on to step two.

**Step 2:** Next, eliminate the sample matrix from the process by sampling a clean matrix (reagent water or sand) spiked with a known reference standard. Sample this control with the identical sampling conditions used previously.

**Step 3:** Analyze the sampled fiber under the identical desorption and instrument conditions used previously.

If the results show the problem persists, then proceed to step four.

If the problem disappears, you have demonstrated that the SPME fiber and desorption process are not the likely causes of the problem. The most likely cause of the problem is the sample matrix and its effect on the equilibrium or fiber. Experiment with your sampling conditions (headspace vs. immersion, time, temperature, pH, salt, agitation, etc.) to determine the optimal parameters for that matrix. Just as with any sample preparation technique, changes in the sample matrix you work with will influence your precision, accuracy, and chromatographic results.

**Step 4:** If the problem persists after removing the sample matrix from the process, then start systematically removing the remaining possible sources of the problem. These include:

- Sampling vial - change the vial and associated septum and make sure you are working with baked or pre-cleaned sampling vials and septa.
- Fiber - change to a control fiber with known performance if you have one or switch to a new, conditioned fiber. In addition, you can eliminate the sampling step and directly desorb the fiber to isolate the problem as it relates to product versus sampling.
- Fiber position - make sure the fiber is positioned properly during the headspace or immersion sampling step.

*If you are having difficulty isolating the problem, contact one of our Technical Service chemists at 800-359-3041 / 814-359-3014 or techservice@sial.com for further assistance.*

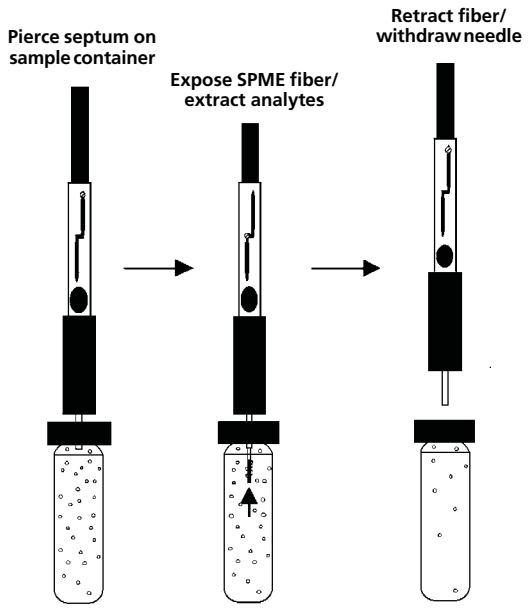
## Tips for Problem Prevention

### Sampling Procedure

***Extraction time*** is critical for the sample to establish equilibrium with the SPME fiber coating. Extractions typically take 15-20 minutes, but can be as short as 30 seconds. Headspace extractions are usually shorter than immersion. The extraction time will depend on the size of the compounds, fiber coating, type of extraction used and sample concentration. Extraction times can be shorter when you are:

- analyzing small compounds (<150 MW)
- using thinner, absorbent type fiber coatings
- using the headspace technique
- working with high concentration samples (high ppb or ppm range).

### Extraction Procedure

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***Temperature*** of the sample is critical for accurate quantitation of the sample. You must use a constant temperature for all extractions to obtain good precision. The use of heat during headspace extractions will help release the analyte from the sample, improve sensitivity, and shorten the extraction time. Note that SPME headspace sampling requires lower temperatures than standard headspace applications. If the temperature is too high with SPME, you can drive the analytes out of the fiber and reduce your overall sensitivity. This is particularly true when using liquid phase, absorbent type fibers such as PDMS. You generally do not need to heat the sample for immersion extractions. For some applications with non-volatile or high boiling semivolatile compounds, a small amount of heat applied to the sample can shorten the equilibration time.

***Agitation*** of the sample is important to reduce the equilibrium time and improve your accuracy and precision. This is crucial when analyzing semivolatile compounds by immersion sampling. Maintain a consistent agitation between all extractions for good precision. Stirring, sonication, and vibration are all suitable methods to agitate the sample. You can also use agitation with headspace extractions. The agitation will usually shorten the headspace extraction time and assure better precision across the sample analyses.

***Adjusting the pH or adding salt*** can improve the extraction efficiency by changing the solubility of the analytes in the sample. The addition of 25-30% (wt./vol.) sodium chloride will increase the ionic strength of the sample, which reduces analyte solubility. The addition of salt is especially helpful when analyzing polar analytes in water. You should buffer the pH of the sample to decrease analyte solubility, improve volatility of bases and acids, and to assure constant pH between extractions. Acidic analytes can be buffered down to pH 2 and basic analytes up to pH 11. The Carbowax-DVB fiber coating should not be used at a pH above 9. Do not use mineral acids or hydroxide salts when you adjust the sample pH. Phosphate buffers at 0.1 M are suitable to obtain a pH buffering range of 2 to 11.

***Headspace sampling*** sensitivity is best when the headspace volume is small. We recommend keeping the headspace volume between 30% and 50% of the vial. When the headspace volume is small the fiber extracts more sample, faster, and with greater efficiency. You can use a larger headspace volume in some cases with high concentration samples. It is extremely important to keep the headspace volume and the vial size constant. You should position the fiber at the same depth in the headspace every time to improve reproducibility. If the matrix contains proteins such as serum and blood, it is advisable to deproteinate the sample before headspace extraction.

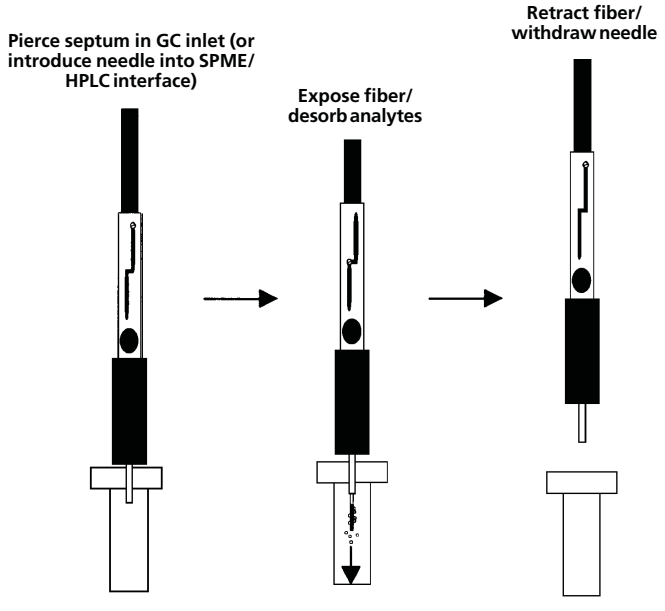
***Immersion sampling*** sensitivity is improved by filling the sampling vial to a minimum of 80%. The recommended sample volumes are 1 mL to 5 mL and use the same volume for all extractions to achieve reproducible results. Immersion sampling works best for low concentration water based sample matrices. When using immersion for samples containing sugars, proteins, and particulate matter, rinse the fiber in water before desorption. This will extend fiber life and reduce injection port contamination. It is best not to immerse the fiber in oils, but if needed, you can wipe the fiber lightly before desorption. Position the fiber just below the sample surface for immersion sampling and maintain this position consistently for all extractions.

***Sampling vials and septa*** can be a source of contaminants and mistaken frequently as a fiber related problem. We recommend baking out septa at 150 °C for two hours before use and always use pre-cleaned vials for your SPME sampling.

***Prevent the needle from bending*** when doing manual SPME sampling by following this procedure:

- Adjust the SPME needle to the 0.2 depth gauge setting on the plunger (first tick mark). This will expose ~3 mm of the needle through the end of the black holder.
- Hold the SPME assembly on top of the sampling vial with the bottom of the black holder flush with the top of the vial cap.
- Hold the sampling vial and black SPME holder base securely with one hand.
- Twist the stainless steel plunger clockwise with the other hand.
- Keep turning the plunger until the desired depth setting is achieved (you will usually hear a pop when the needle pierces the septum).
- Expose the fiber and perform sampling as usual.

## Desorption Procedure




### Desorption Procedure

***Splitless injection*** is required with SPME to focus the analytes on the chromatographic column. Close the splitter vent valve for at least two minutes during sample injection.

***Narrow-bore inlet liners*** (0.75 mm I.D.) will reduce peak broadening in the chromatographic analysis by minimizing the dead volume during analyte transfer onto the column.

***Pre-drilled, low bleed septa*** (Thermogreen™ LB-2) or a ***septumless injector system*** such as the Merlin Microseal™** is recommended to reduce or eliminate coring of the injection port septa during sample desorption. Septa coring can result in interfering peaks, poor chromatography, and fiber breakage.

***Prevent the needle from bending*** when doing manual SPME desorption by following this procedure,

- Adjust the SPME needle to the 0.2 depth gauge setting on the plunger (first tick mark). This will expose ~3 mm of the needle through the end of the black holder.
- Position the SPME assembly on top of the GC injection port or the SPME inlet guide with the bottom of the black holder flush with the top of the injector or guide.
- Hold the black SPME holder base securely with one hand.
- Twist the stainless steel plunger clockwise with the other hand.
- Keep turning the plunger until the desired depth setting is achieved (you will usually hear a pop when the needle pierces the septum).
- Expose the fiber and perform desorption as usual.

### Analysis Procedure

We suggest reviewing the Capillary GC Troubleshooting Guide (T112853) or HPLC Troubleshooting Guide (T100826) when you isolate the problem to the injection port, column, or detector. These guides provide helpful tips to improve your analysis procedure.

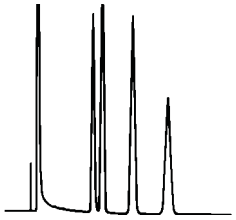
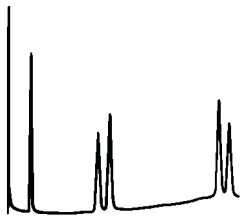
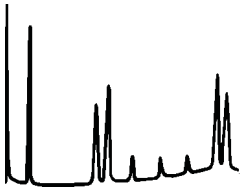
### SPME Product Related

SPME fiber breakage is a potential problem when you apply excessive stress to the fiber during sampling or analysis. We recommend the use of StableFlex™ fibers when possible for your application. We make StableFlex fibers with a flexible fused silica core, which is more durable and less likely to break if stressed.

SPME accessories such as the inlet guide, sampling stand, heat/stir plate, magnetic stirring bars, and thermometer will improve the reproducibility and ease of the sampling and desorption steps.

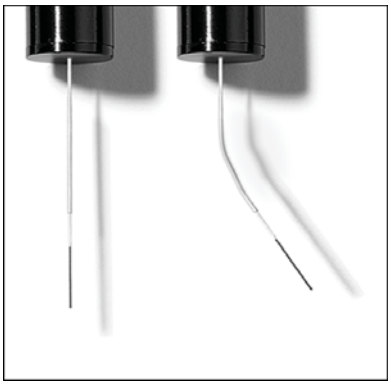
Do not expose PDMS coated fibers to non-polar solvents and do not expose Carbowax coated fibers to polar solvents. The fiber coating will swell and cause damage to the fiber upon retraction into the holder. The damage may include breakage, grooving, or stripping of the fiber coating. To avoid this problem, dilute the sample with water before extraction to reduce the organic solvent percentage to less than three percent (<3%).

## Troubleshooting Table

| Problem / Symptom | Possible Cause | Remedy |
|---|---|---|
| **1. No peaks seen in GC analysis**<br>  <br>Normal<br> <br>Problem | 1. Instrument problems (A) | 1. Inject standard mixture to verify detector response, see GC troubleshooting guide for help |
| | 2. The splitter vent was left open (D) | 2. Run splitless injection for 2 min. |
| | 3. The analyte concentration is too low to be detected (S) | 3. Start with known concentration (1ppm) of analyte in de-ionized water mixture. Optimize extraction by adjusting extraction time, temperature, and chemical condition of pH and salt |
| | 4. Solvents present in sample competing with SPME extraction (S) | 4. Minimize solvents in the sample to <3% by dilution in water |
| | 5. Headspace volume too large to establish equilibrium with fiber (S) | 5. Reduce headspace to 50% or less, agitate sample vigorously, or increase the sampling temperature. |
| | 6. Coating on fiber deteriorated (P) | 6. Replace fiber. Fibers are reusable and will last for 50 injections on average. |
| | 7. Incorrect SPME fiber used for extraction (P) | 7. This is beyond the scope of this guide. Please contact technical service (800-359-3041 / 814-359-3041) for assistance if you are experiencing problems selecting the appropriate fiber for your application |
| | 8. There is a leaking injection port (septum or connection) (D) | 8. Replace septum and tighten nut properly |
| | 9. There is a leaking sample vial (S) | 9. Replace vial septum and seal cap properly |
| | 10. Loss during transport from the field (S) | 10. Move the depth adjusting lever of the portable field sampler to the top-most locking slot, so the end of the septum-piercing needle is totally withdrawn into the sealing septum of the sampler. If the fiber will be stored for more than one day we recommend that it be stored at subambient temperature. This reduces the chance of breakdown and oss of sample that could occur at higher temperatures. |
| **2. Extraneous peaks in analysis**<br>  <br>Normal<br>  <br>Problem | 1. Septa used in sampling vial or injection port is outgassing organic contaminants (S/A) | 1. Prebake the vial septa for 2 hours at 68°C prior to use. Use low-bleed LB-2 septa to minimize injection port septum bleed. |
| | 2. Fiber is not preconditioned prior to sampling (P) | 2. Precondition fiber at the recommended conditioning temperature in the fiber instruction sheet. Once fiber is preconditioned, only 1-2 minutes is required to clean the fiber prior to sampling. |
| | 3. Inlet liner is contaminated or contains septa particles (D) | 3. Replace the inlet liner. Use pre-drilled septum or a septumless injector system (e.g. Merlin Microseal) |
| | 4. GC column is collecting analytes on the front of the column because it is not heated high enough in sample analysis (A) | 4. Complete GC analysis temperature program before injecting another SPME extract and keep column at 150°C when not in use. |
| | 5. Interfering peaks coelute with analytes of interest (A) | 5. Change GC column or temperature program rate |
| | 6. Carryover from previous analysis of the fiber (P) | 6. Bake out fiber at the recommended conditions for several additional minutes |
| | 7. Cross-contamination from laboratory air (S) | 7. Do not expose the fiber to the laboratory environment at any time during the sampling or injection steps. Analyze control blanks using the same handling process as the sample to determine if technique or laboratory cross-contamination is present. |
| | 8. Cross-contamination during transport from the field (S) | 8. Move the depth adjusting lever of the portable field sampler to the top-most locking slot, so the end of the septum-piercing needle is totally withdrawn into the sealing septum of the sampler. If the fiber will be stored for more than one day we recommend that it be stored at subambient temperature. This reduces the chance of sample cross-contamination. |

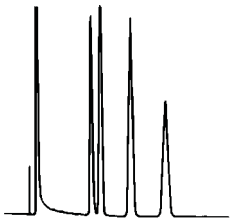
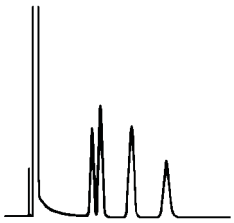
A=Analysis related D=Desportion related P=Product related S=Sampling related

## Troubleshooting Table

| Problem / Symptom | Possible Cause | Remedy |
|---|---|---|
| **3. Fiber will not retract or sticks in holder needle** | 1. The end of the needle is plugged with a piece of septum (D) | 1. Injection port septum nut is overtightened. Loosen the nut slightly to allow for improved injection. Use pre-drilled injection port septa or a septumless injector system (e.g. Merlin Microseal) |
| | 2. The fiber was exposed to solvents that caused swelling of coating (S) | 2. Do not expose PDMS coated fibers to non-polar solvents such as pentane, methylene chloride, or diethyl ether. Do not expose Carbowax fibers to polar solvents. |
| | 3. The top screw in the holder assembly is too tight (P) | 3. Loosen the top screw on the holder assembly slightly to allow for free movement of the plunger. |
| **4. Needle bends during injection into sample vial or GC injection port**  | 1. Improper manual sampling technique (S) | 1. To prevent the needle from bending when doing manual SPME sampling, follow this procedure:<br>• Adjust the SPME needle to the 0.2 depth gauge setting on the plunger (first tick mark). This will expose about 3 mm of the needle through the end of the black holder.<br>• Hold the SPME assembly on top of the sampling vial with the bottom of the black holder flush with the top of the vial cap.<br>• Hold the sampling vial and black SPME holder base securely with one hand<br>• Twist the stainless steel plunger clockwise with the other hand<br>• Keep turning the plunger until the desired depth setting is achieved (you will usually hear a pop when the needle pierces the septum).<br>• Expose the fiber and perform sampling as usual |
| | 2. Improper manual desorption (injection) technique (D) | 2. To prevent the needle from bending when doing manual SPME injections, follow this procedure:<br>• Adjust the SPME needle to the 0.2 depth gauge setting on the plunger (first tick mark). This will expose about 3 mm of the needle through the end of the black holder.<br>• Position the SPME assembly on top of the GC injection port or in the SPME inlet guide with the bottom of the black holder flush with the top of the injector or guide.<br>• Hold the black SPME holder base securely with one hand<br>• Twist the stainless steel plunger clockwise with the other hand<br>• Keep turning the plunger until the desired depth setting is achieved (you will usually hear a pop when the needle pierces the septum).<br>• Expose the fiber and perform sample desorption as usual |
| | 3. Vial or injection port septum is too tight (S/D) | 3. Loosen slightly the vial closure or injection port nut |
| | 4. Septa in sample vial/injection port is too thick or coated with thick Teflon® coating (S/D) | 4. Use LB-2 septa for injection port or silicone septa with <10 mil Teflon on sampling vials. Shorten the amount of exposed needle on SPME holder to 0.5 inch or (~1 cm) before puncturing the vial septa. Adjust the holder needle setting to the desired depth for sampling. Do not use butyl rubber style septa. |
| | 5. GC inlet liner is too narrow or packed with adsorbent material (D) | 5. Use larger splitless inlet liners (0.75 mm I.D. or larger) without glass wool or adsorbents. |
| Needle bends with automated injection systems | 6. Needle is out of alignment with injection port or sample vial (S/D) | 6. Reference autoinjector manual on alignment |

A=Analysis related D=Desportion related P=Product related S=Sampling related

## Troubleshooting Table

| Problem / Symptom | Possible Cause | Remedy |
|---|---|---|
| 5. Fiber breaks | 1. The fiber was not retracted into the protective needle after removal from sample vial or injection port (S/D) | 1. Retract fiber into protective needle during insertion into vial/injection port and removal |
| | 2. The end of the needle is plugged with a piece of septum (D) | 2. Injection port septum nut is overtightened. Loosen the nut slightly to allow for improved injection. Use pre-drilled injection port septa or a septumless injector system (e.g. Merlin Microseal) |
| 6. Reproducibility is poor | 1. Time and temperature variations during sampling (S) | 1. Time of extraction and temperature are the two most critical conditions to control. Use timing device and calibrated thermometer to ensure reproducible results. Remember that room temperature fluctuations will influence the ambient sample temperature. |
|  Normal | 2. Not consistently positioning the fiber at the same depth during sampling (S) | 2. Position fiber just below sample surface for immersion sampling and at a consistent position above the sample during headspace sampling. |
| | 3. pH or salt conditions varying during sampling (S) | 3. Apply uniformly across all extractions any pH or salt adjustments made to the samples. |
| | 4. Equilibrium is not reached during extraction (S) | 4. Determine minimum time for equilibrium using a standard mixture and controlled extraction conditions. Note that full equilibrium is not required to be reached for all applications to achieve reproducible results. |
|  Problem | 5. Varying organic content in the samples (S) | 5. Dilute samples to minimize solvent interference or use headspace sampling to minimize solvent effect. Use internal standards, surrogates, or the standard addition technique to compensate for variations in sample matrix. |
| | 6. Varying headspace in sample vials during headspace extraction (S) | 6. Minimize headspace volume to 50% or less and agitate the sample. Maintain the same headspace volume and agitation conditions across all extractions. |
| | 7. Solid samples not releasing analytes for extraction (S) | 7. Grind solid into small particles, add to water, and apply heat and agitation. |
| | 8. Competing analyte displaces compound of interest/or interferes (S) | 8. Reduce the extraction time to minimize displacement/or interference |
| | 9. Not reproducing desorption conditions (D) | 9. Verify that the fiber position (depth), desportion time, temperature, and splitless conditions are consistent. Use an automated SPME system to improve reproducibility. |
| | 10. Not using agitation during sampling or apply it inconsistently (S) | 10. Use a stir bar or soncication system to agitate the sample during sampling. Maintain consistent agitation conditions for all standards and samples. |
| | 11. Sample volumes are inconsistent (S) | 11. Maintain consistent volumes for all standards and samples. |

A=Analysis related D=Desportion related P=Product related S=Sampling related

## Troubleshooting Table

| Problem / Symptom | Possible Cause | Remedy |
|---|---|---|
| 7. Fiber discolored | 1. Fiber is oxidized during fiber conditioning or sample injection into GC (S/D)<br>2. Heating during injection (D/P) | 1. Minimize oxygen in carrier gas, condition fibers in oxygen free gas flow. Reduce the injection port temperature to the recommended maximum setting. Carbowax/DVB coatings are especially sensitive to temperature (<260 °C is recommended).<br>2. Does not usually affect the performance of the fiber. Always minimize the oxygen content in the GC carrier gas to avoid oxidizing the fiber coating. The polyacrylate coated fiber will discolor above 280 °C. Carbowax/DVB may slightly darken during use, however, if the fiber turns brown, lower the injection port temperature (265 °C is the recommended maximum) and check the system for leaks. |
| 8. Number of injections from the fiber is less than previously obtained | 1. Fiber is oxidized during fiber conditioning or sample injection into GC (S/D)<br>2. Coating on fiber deteriorated (P)<br>3. Fiber was exposed to solvents that cause swelling of coating (S) | 1. Minimize oxygen in carrier gas, condition fibers in oxygen free gas flow. Reduce injection port temperature to the recommended fiber maximum.<br>2. Replace fiber. Fibers are reusable and will last for 50 injections on average.<br>3. Do not expose PDMS coated fibers to non-polar solvents such as pentane, methylene chloride, or diethyl ether. Do not expose Carbowax fibers to polar solvents. |

**Patents**

*Solid Phase Microextraction (SPME) Technology licensed exclusively to Supelco. U.S. patent #5,691,206; European patent #523092.

**Merlin Instrument Co., US Patent #4,954,149.

**Trademarks**

Carbowax — DOW Chemical

Carboxen, StableFlex, and Thermogreen — Sigma Aldrich

Microseal — Merlin Instrument Company

Teflon, VESPEL — E.I. dupont de Nemours & Co., Inc.

A=Analysis related D=Desportion related P=Product related S=Sampling related

## Ordering Information:

| | Fiber Core/ Assembly Type | Hub Description | Sampling and Analysis Mode | | | |
|---|---|---|---|---|---|---|
| | | | Manual Holder (w/ spring) | | Autosampler (w/o spring) | |
| | | | 23 Ga* | 24 Ga* | 23 Ga* | 24 Ga* |
| **Carboxen/Polydimethylsiloxane** | | | | | | |
| 75 µm Carboxen/PDMS | Fused Silica/SS | Black/plain | **57344-U** | **57318** | **57343-U** | **57319** |
| 85 µm Carboxen/PDMS | Metal alloy/Metal alloy** | Lt. Blue/plain | | | **57906-U** | |
| 85 µm Carboxen/PDMS | Stableflex/SS | Lt. Blue/plain | | **57334-U** | **57295-U** | **57335-U** |
| **Polydimethylsiloxane (PDMS)** | | | | | | |
| 7 µm PDMS | Metal alloy/Metal alloy** | Green/plain | | | **57919-U** | |
| 7 µm PDMS | Fused Silica/SS | Green/plain | | **57302** | **57291-U** | **57303** |
| 30 µm PDMS | Metal alloy/Metal alloy** | Yellow/plain | | | **57922-U** | |
| 30 µm PDMS | Fused Silica/SS | Yellow/plain | | **57308** | **57289-U** | **57309** |
| 100 µm PDMS | Metal alloy/Metal alloy** | Red/plain | | | **57298-U** | |
| 100 µm PDMS | Fused Silica/SS | Red/plain | **57342-U** | **57300-U** | **57341-U** | **57301** |
| **Polydimethylsiloxane/Divinylbenzene (PDMS/DVB)** | | | | | | |
| 60 µm PDMS/DVB | Stableflex/SS | Brown/notched | | | | **57317** |
| 65 µm PDMS/DVB | Metal alloy/Metal alloy** | Blue/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** |
| **Polyacrylate** | | | | | | |
| 85 µm Polyacrylate | Fused Silica/SS | White/plain | | **57304** | **57294-U** | **57305** |
| **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 | | **57348-U** | **57299-U** | |
| **Polyethylene Glycol (PEG)** | | | | | | |
| 60 µm PEG | Metal alloy/SS | Purple/plain | **57355-U** | | **57354-U** | |

* Ga - Needle gauge.
** Metal alloy fiber assemblies are provided 1/pk.

### Fiber Assortment Kits

| Name | Description of Fiber Coatings (1 ea.) | Recommended Use | Automated/HPLC | | |
|---|---|---|---|---|---|
| | | | Manual | Std. Needle | 23 Ga. Needle |
| Kit # 1 | 85 µm polyacrylate, 100 µm PDMS, 7 µm PDMS | GC | **57306** | **57307** | **57285-U** |
| Kit # 2 | 75 µm Carboxen/PDMS, 65 µm PDMS/DVB, 85 µm polyacrylate | GC | **57320-U** | **57321-U** | **57286-U** |
| Kit # 3 | 60 µm PDMS/DVB, 85 µm polyacrylate, 100 µm PDMS | HPLC | – | **57323-U** | – |
| Kit # 4 | 100 µm PDMS, 65 µm PDMS/DVB, 75 µm Carboxen/PDMS | GC | **57324-U** | **57325-U** | **57287-U** |
| StableFlex Kit | 65 µm PDMS/DVB, 50/30 µm DVB/Carboxen/PDMS | GC | **57550-U** | **57551-U** | – |
| Kit # 5 | 100 µm PDMS, 65 µm PDMS/DVB, 85 µm Carboxen/PDMS | GC | **57324-U** | **57325-U** | **57287-U** |
| | 50/30 µm DVB/Carboxen/PDMS | GC | | | **57362-U** |

## SPME Fiber Holders

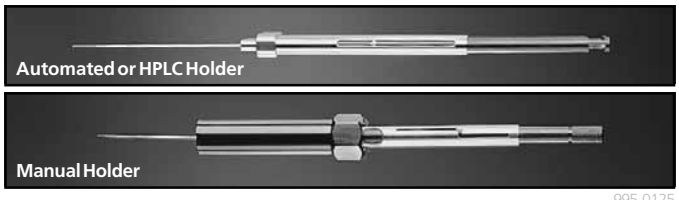
The holder protects the coated fiber, and controls exposure of the fiber during analyte adsorption and desorption. The holder is reusable indefinitely and accepts the replaceable fiber assembly. First time users must order both a holder and a fiber assembly.

*Fiber Holder for Manual Sampling*

An adjustable depth guide positions the fiber for sampling and for correct placement in the heated zone of the GC injection port. The fiber can be locked in the exposed position.

*Fiber Holder for Automated Sampling or HPLC Analysis*

Use this fiber holder with a Varian 8100/8200 AutoSampler or with our SPME/HPLC interface. An SPME upgrade kit is necessary for operation with the Varian AutoSampler – contact Varian Instrument Division for information concerning system requirements.




995-0125

### Fiber Holder for CTC, Gerstel, and Thermo Autosamplers▲

Use this holder with SPME fiber assemblies that are designed for automated sampling.

| Description | Cat. No. |
|---|---|
| **Fiber Holder** | |
| For Manual Sampling | **57330-U** |
| For Varian Autosampler or HPLC Analysis | **57331** |
| For CTC, Gerstel, and Thermo Autosamplers | **57347-U** |

▲ Autosamplers distributed by Varian, Leap, Gerstel, and Thermo.

## SPME Portable Field Sampler

**Concentrate and Store Analytes from Water; Sample Indoor Air** - The SPME portable field sampler is an efficient and economical way of extracting and transporting volatile and semivolatile compounds from field samples. Extracted compounds are safely sealed behind a replaceable septum. Table 1 shows that storage losses for pesticides extracted and stored using a portable field sampler were significantly lower than losses from stored whole water samples. The sampler can be reused 50-100 times, and is disposed of when the fiber is no longer usable.

The portable field sampler also efficiently detects organic compounds in air. In our studies, the sampler allowed us to monitor typical HPLC and GC solvents at ppb levels in laboratory air. Four fibers are available: a polydimethylsiloxane (PDMS)/Carboxen fiber for trace levels of volatiles, a general purpose PDMS fiber and, a PDMS/DVB fiber for semi-volatiles and larger volatiles.

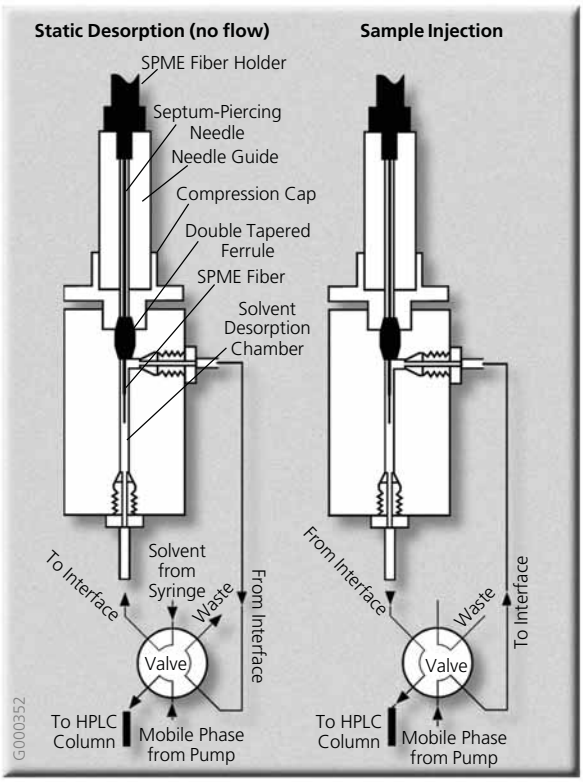
Five slots in the needle guide/depth gauge control the depth of needle insertion into a sample container, or into the injection port during fiber desorption.

| **SPME Portable Field Samplers, 24 Ga., pk. of 2** | |
|---|---|
| 75 µm PDMS/Carboxen Fiber | **504831** |
| 100 µm PDMS Fiber | **504823** |
| 65 µm PDMS/DVB StableFlex Fiber | **57359-U** |
| **Replacement Septa, pk. of 50** | **20638** |
| **SPME Septum Removing Tool** | **504858** |

## SPME/HPLC Interface for Easy HPLC Analyses with SPME

Investigators in several laboratories have shown that SPME can be effective for monitoring drugs and drug metabolites in biological fluids – and SPME also has pharmacological and food and beverage applications.

The SPME/HPLC interface enables HPLC analysts to take advantage of the time and cost savings offered by SPME. The interface allows mobile phase to contact the SPME fiber, remove the adsorbed analytes, and deliver them to the column for separation. The interface consists of a six-port injection valve and a desorption chamber that replaces the injection loop in the HPLC system. 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.




The SPME fiber is introduced into the desorption chamber with the injection valve in the "load" position. The unit is made leak-tight (to 5000 psi/35 mPa) by closing the clamp and compressing the ferrule against the SPME needle. All surfaces which contact the SPME fiber or the mobile phase are stainless steel or VESPEL.

Analytes often can be removed via a stream of mobile phase (dynamic desorption). 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).

**SPME/HPLC Interface, Replacement Parts, and Accessories**

| Description | Cat. No. |
|---|---|
| **SPME/HPLC Interface (includes 2 ferrules)** | |
| Rheodyne valve version | **57353** |
| **Ferrules, pk. of 10** | **57351** |
| **Rotor seal for Rheodyne valve 7125** | **58830-U** |
| **Rhebuild kit for Rheodyne valve 7125** | **55045** |
| **SPME holder for HPLC use** | **57331** |
| **Fiber assemblies for HPLC use** | |
| 60 µm polydimethylsiloxane/divinylbenzene, for drugs, vitamins, preservatives, general purpose | **57317** |
| 85 µm polyacrylate, for polar semivolatiles | **57305** |
| 100 µm polydimethylsiloxane, for volatiles | **57301** |
| **Fiber Kit 3 (one fiber of each)** | |
| 85 µm polyacrylate, 60 µm polydimethylsiloxane/divinylbenzene, 100 µm polydimethylsiloxane | **57323-U** |

## Manual SPME Sampling Stand

Holds eight vials while supporting the SPME syringe for consistent fiber immersion depth. Cat. No. **57333-U** accommodates 4 mL vials only; Cat. No. **57357-U** accommodates 15 mL vials. Order the 15 mL vial puck (Cat. No. **57358-U**) as a replacement for the 15 mL unit, or to use 15 mL vials with the 4 mL unit. Not for use with automated / HPLC fiber holders.

## Heat/Stir Plate

Fits compactly on the base of the SPME sampling stand. Heating range is 40-550 °C, stirring range is 60-1200 rpm.

## 40 mL Vial Holder

Use this aluminum block for heating/stirring during headspace SPME sampling of odors or other volatiles. Holds six 40 mL vials.

## Thermometer

For monitoring sample temperature when using the SPME sampling stand and a heat/stir plate.

| Description | Cat.No. |
|---|---|
| SPME sampling stand for 4 mL vials | **57333-U** |
| SPME sampling stand for 15 mL vials | **57357-U** |
| Vial puck for 15 mL vials | **57358-U** |
| 40 mL Vial Holder | **33313-U** |
| Thermometer, 5″ | **57332** |

## SPME Inlet Guide

Secures the SPME fiber holder in the injection port during the thermal desorption process. Interchangeable among Merlin Microseal sealing system and most Varian and Agilent/HP chromatographs.

| Description | Cat. No. |
|---|---|
| SPME inlet guide | **57356-U** |

## Merlin Microseal High Pressure Septa

**Eliminate siloxane background, prolong septum lifetime.** To eliminate septum coring during SPME injections, use the Merlin Microseal system, a patented long-life replacement for the standard septum and septum nut on a capillary or purged packed inlet system. Two sequential seals provide a much longer life than conventional septa. The new high pressure units allow operation at 2-100 psi. Use only with 23 gauge SPME fiber assembly.

| Description | Cat. No. |
|---|---|
| **For Agilent/HP GC Models 5800, 5900 series, 6890** | |
| 1 nut and 2 septa | **24814-U** |
| 1 nut and 1 septum | **24815-U** |
| 1 replacement septum | **24816-U** |
| **For Varian GC Models 3400, 3800 (1078, 1079 injectors)** | |
| 1 Varian nut, 1 septum, & 1 inlet adapter | **24817-U** |
| 1 replacement septum | **24818-U** |

## Pre-Drilled Thermogreen LB-2 Septa for SPME

Easier needle penetration and high puncture tolerance – ideal for autosamplers. Reduce septum coring that can cause extraneous peaks. Already conditioned, ready-to-use. Extremely low bleed over a wide range of inlet temperatures – from 100 °C to 350 °C. Rubber formulation exclusive to Supelco.

| Description | Cat. No. |
|---|---|
| 9.5 mm, pk. of 25 | **23161** |
| 9.5 mm, pk. of 50 | **23162-U** |
| 11 mm, pk. of 25 | **23167** |
| 11 mm, pk. of 50 | **23168** |

**Order/Customer Service** 800-247-6628, 800-325-3010
Fax 800-325-5052 ● E-mail supelco@sial.com

**Technical Service** 800-359-3041, 814-359-3041
Fax 800-359-3044, 814-359-5468 ● E-mail techservice@sial.com

**SUPELCO ● 595 North Harrison Road, Bellefonte, PA 16823-0048 ● 814-359-3441**

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