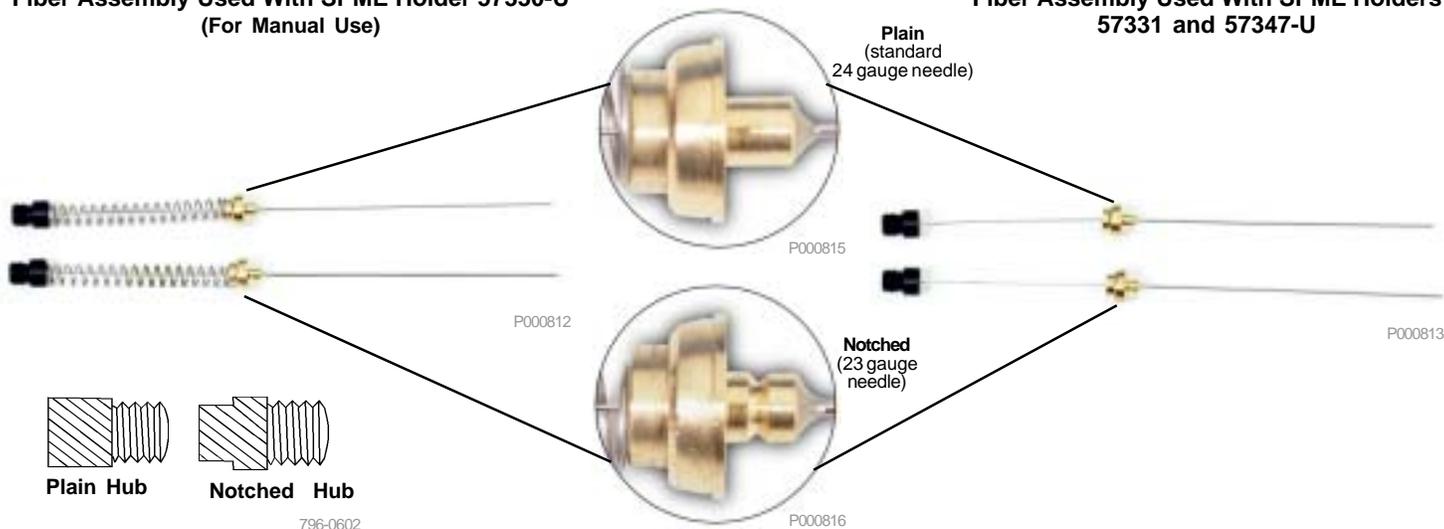


## Solid Phase Microextraction Fiber Assemblies

Fiber Assembly Used With SPME Holder 57330-U  
(For Manual Use)

Fiber Assembly Used With SPME Holders  
57331 and 57347-U



| Stationary Phase/<br>Film Thickness                           | Description | Hub<br>Description   | Recommended<br>Use | Used with<br>SPME Holder 57330-U |               | Used with SPME<br>Holders 57331 and 57347-U |               |
|---|-------------|----------------------|--------------------|----------------------------------|---------------|---|---------------|
|   |             |                      |                    | Std. Needle                      | 23 Ga. Needle | Std. Needle                                 | 23 Ga. Needle |
| <b>Polydimethylsiloxane (PDMS)</b>                            |             |                      |                    |                                  |               |   |               |
| 100µm   | Non-bonded  | Red / plain          | GC/HPLC            | 57300-U                          | 57342-U       | 57301                                       | 57341-U       |
| 30µm  | Non-bonded  | Yellow / plain       | GC/HPLC            | 57308                            | —             | 57309                                       | 57289-U       |
| 7µm   | Bonded      | Green / plain        | GC/HPLC            | 57302                            | —             | 57303                                       | 57291-U       |
| <b>Polydimethylsiloxane/Divinylbenzene (PDMS/DVB)</b>         |             |                      |                    |                                  |               |   |               |
| 65µm  | Bonded      | Blue / plain         | GC                 | 57310-U                          | 57346-U       | 57311                                       | 57345-U       |
| 60µm ♦  | Bonded      | Brown / notched      | HPLC               | —                                | —             | 57317                                       | —             |
| <b>StableFlex™</b>  |             |                      |                    |                                  |               |   |               |
| 65µm  | Bonded      | Pink / plain         | GC                 | 57326-U                          | —             | 57327-U                                     | 57293-U       |
| <b>Polyacrylate</b>   |             |                      |                    |                                  |               |   |               |
| 85µm  | Bonded      | White / plain        | GC/HPLC            | 57304                            | —             | 57305                                       | 57294-U       |
| <b>Carboxen™/Polydimethylsiloxane (CAR/PDMS)</b>              |             |                      |                    |                                  |               |   |               |
| 75µm  | Bonded      | Black / plain        | GC                 | 57318                            | 57344-U       | 57319                                       | 57343-U       |
| <b>StableFlex</b>   |             |                      |                    |                                  |               |   |               |
| 85µm  | Bonded      | Lt. Blue / plain     | GC                 | 57334-U                          | —             | 57335-U                                     | 57295-U       |
| <b>Carbowax®/Divinylbenzene (CW/DVB)</b>                      |             |                      |                    |                                  |               |   |               |
| 65µm  | Bonded      | Orange / plain       | GC                 | 57312                            | —             | 57313                                       | 57296-U       |
| <b>StableFlex</b>   |             |                      |                    |                                  |               |   |               |
| 70µm  | Bonded      | Yellow-green / plain | GC                 | 57336-U                          | 57338-U       | 57337-U                                     | 57339-U       |
| <b>Carbowax/Tempated Resin (CW/TPR)</b>                       |             |                      |                    |                                  |               |   |               |
| 50µm ♦  | Bonded      | Purple / notched     | HPLC               | —                                | —             | 57315                                       | —             |
| <b>StableFlex Divinylbenzene/Carboxen/PDMS (DVB/CAR/PDMS)</b> |             |                      |                    |                                  |               |   |               |
| 50/30µm   | Bonded      | Gray / plain         | GC                 | 57328-U                          | —             | 57329-U                                     | 57298-U       |
| 50/30µm   | Bonded      | Gray / notched       | GC                 | 57348-U •                        | 57299-U •     | 57348-U •                                   | 57299-U •     |

♦ This fiber is more durable than the others listed. It contains no epoxy

• Special 2cm length. Does not contain spring.

**Non-bonded phases** are stable with some water-miscible organic solvents, but slight swelling may occur. NEVER use or rinse with nonpolar organic solvents.

**Bonded phases** are stable with all organic solvents. Slight swelling may occur when used with some nonpolar solvents.

**Note:** For solvent/fiber compatibility, refer to Supelco application notes and bulletins that describe SPME/HPLC analyses.

**Note:** Fibers for autosampler holders (57331 & 57347-U) **can be** used with manual holder (57330-U).

**CAUTION:** CHLORINATED SOLVENTS MAY DISSOLVE THE EPOXY THAT HOLDS THE FIBER.

USE EXTRA CAUTION WHEN HANDLING PDMS/DVB AND CW/DVB FIBERS. THE COATING CAN BE INADVERTENTLY STRIPPED OFF.

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**Table 1. Temperature and Conditioning Recommendations for GC Use and pH Guidelines**

| Stationary Phase | Film Thickness | pH   | Maximum Temperature | Recommended Operating Temp. | Conditioning Temperature | Time (Hrs.) |
|------------------|----------------|------|---------------------|-----------------------------|--------------------------|-------------|
| PDMS             | 100µm          | 2-10 | 280°C               | 200-280°C                   | 250°C                    | 0.5         |
|                  | 30µm           | 2-11 | 280°C               | 200-280°C                   | 250°C                    | 0.5         |
|                  | 7µm            | 2-11 | 340°C               | 220-320°C                   | 320°C                    | 1           |
| PDMS/DVB         | 65µm           | 2-11 | 270°C               | 200-270°C                   | 250°C                    | 0.5         |
| Polyacrylate     | 85µm           | 2-11 | 320°C               | 220-310°C                   | 300°C                    | 2           |
| CAR/PDMS         | 75µm           | 2-11 | 320°C               | 250-310°C                   | 300°C                    | 1-2         |
| CW/DVB           | 65µm           | 2-9  | 260°C               | 200-250°C                   | 220°C                    | 0.5         |
| DVB/CAR/PDMS     | 50/30µm        | 2-11 | 270°C               | 230-270°C                   | 270°C                    | 1           |

## Attaching the Fiber Assembly to the Holder

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

1. Unscrew the black cylinder like depth gauge from the holder.
2. Unscrew the threaded end-cap on the end of the holder.
3. Push the black plunger 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 will accept the threaded fiber assembly.
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 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.



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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.

## For GC Use

### Choosing a Fiber

As a first step, identify the molecular weight range of the analytes to be extracted. Higher molecular weight compounds desorb easier from the surface of the 7 $\mu$ m or 30 $\mu$ m PDMS absorption fiber coatings. Smaller molecules are retained in the pores of the fibers containing adsorbents in the coating; e.g. Carboxen, divinylbenzene particles. Further, refine your choice by matching the fiber coating relative to analyte polarity.

StableFlex SPME fibers are coated on a flexible fused silica core. The coating partially bonds to the flexible core which results in a more stable coating and a less breakable fiber. The extraction selectivity of StableFlex fibers may be slightly different from the same coating on a standard fused silica core.

23 gauge needles are specifically designed to meet the specifications of Merlin Microseal septum system. The needle is also suitable for other septumless seals. The 23 gauge needles are not recommended for use with standard silicone septa as they severely core the septum.

2cm fibers are available in the StableFlex DVB/CAR/PDMS fibers. They do not contain springs and must be manually retracted. They are not recommended for autosampler use.

### Conditioning Instructions

Set the injection port temperature according to Table 1 on this sheet. Install a column into the GC and adjust carrier gas to the desired flow rate. Use a carrier gas purifier to reduce oxygen in the carrier gas to minimize oxidation of the fiber surface during conditioning. If a splitless/split injection port is used, open the splitter vent. Insert the SPME needle into the GC injection port, expose the fiber, and condition for the recommended time. Longer

than recommended conditioning times will not hurt the fiber. After conditioning is completed, retract the fiber and remove the needle from the injection port. Condition the column for an additional 30 minutes at the upper temperature of the program.

THE POLYACRYLATE FIBER WILL TURN BROWN AS A RESULT OF CONDITIONING. THIS WILL NOT AFFECT THE PERFORMANCE OF THE FIBER.

### Blank Analysis

After conditioning the fiber, set the injection port to the desired operating temperature and cool the GC oven to 50°C or lower. Set the detector attenuation at the sensitivity typical for the desired analysis. Close the split injection port valve, insert the SPME needle and start the GC temperature program. Desorb the fiber for 5 minutes in the injection port while programming the GC. Retract the fiber and remove the needle after 5 minutes and observe the chromatogram when the program is completed. There may be some extraneous peaks from the previous extraction or exposure of the fiber since the initial conditioning of the fiber. Redesorb the fiber, the intensity of the peaks should decrease after the second desorption step. Please contact our technical service department for further assistance if the background remains too high after following these steps.

### Cleaning Fibers

You can clean severely contaminated fibers by one of two methods. First, you can heat the fiber to 20°C below its recommended maximum temperature for one hour to overnight. Check the table of fibers to determine the recommended maximum temperature. A second alternative is to rinse the fiber by soaking it in a water miscible solvent for one hour, then thermally desorb it for thirty minutes. NEVER RINSE A FIBER IN CHLORINATED SOLVENTS. These solvents will damage the fibers.

### Fiber Selection

The fiber assemblies recommended for HPLC are crimped rather than use epoxy to attach the fiber in the fiber assembly. Four fibers are recommended for use with HPLC solvents: 100 $\mu$ m PDMS, 60 $\mu$ m PDMS/DVB, 50 $\mu$ m Carbowax/templated resin, and 85 $\mu$ m polyacrylate.

## For HPLC Use

### Conditioning Instructions

The effects of different solvents on an SPME fiber will vary. It is best to condition the fiber with the mobile phase or solvent to which it will be exposed. Referring to the instructions for using the SPME/HPLC interface, expose the fiber in the interface. Switch the valve from the LOAD position to the INJECT position, allowing the mobile phase to pass through the interface (dynamic mode). If a gradient is used, run the gradient through the entire cycle. The fiber should be conditioned for a minimum of 30 minutes.

If the fiber is desorbed in a solvent different than the mobile phase, switch the valve to the LOAD position, fill the valve desorption chamber with the solvent, and allow the fiber to soak in the solvent (static mode) for a minimum of 15 minutes. The background can be checked by switching the valve to the INJECT mode and monitoring baseline throughout the run cycle. The fiber may need additional conditioning if the background is not sufficiently clean. Minimal cleaning is usually required, but cleaning time often depends on the type of detection and sensitivity.

### Solvent Effects on Extraction of Samples

If organic solvents are present in the fiber, they may affect the extraction of the next sample. As a precaution, if the fiber has been desorbed in organic solvents, allow the fiber to dry for several minutes prior to starting the next extraction. You may determine that the drying step is not necessary for particular samples. The effect of solvents on the fiber can be determined by comparing results of two extractions — one with a dried fiber, and the other with a non-dried fiber.