SPME Tips

Figure 1. SPME LC Tips in Tray to Well-Plate

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Figure 2. SPME LC Tips in Vials

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SPME LC Tips are designed for extraction of non-volatile analytes out of aqueous-based matrices. The fiber tips are SPME fibers sealed in a low volume micropipette tip. Because the tips are commonly used with biological fluids, it is recommended that each tip only be used for one sample. The tips can be manipulated by either robotic equipment or manually. The SPME LC tips are contained in a 96-well format tray. The tray containing the tips can be transferred between well plates during extraction and desorption process as shown in Figure 1. If only a few samples are being analyzed, the tips can be moved individually into small vials for extraction of the analytes then transferred to other low volume tapered vials for solvent desorption as shown in Figure 2. Vials can be placed in a tray so that multiple tips could be used at one time. It is not necessary to cap sample vial during the extraction process for non-volatile compounds, as depicted in Figure 1. **SPME LC Tips are not designed for GC thermal desorption applications.**

Process of Use for LC Tips

1. **The fiber coating on the LC tips must be pre-wet to solvate the stationary phase.** Place the LC tips in a solution of 50% methanol:water or 50% acetonitrile:water for a minimum of 20 minutes. Well-plates or vials can be used for this process. Solvated fibers will take on a dark gray appearance, indicating that the fibers are conditioned and ready for extraction.
2. Prepare samples for extraction by putting solutions either into well-plates or sample vials. Transfer the tips from the wetting solution to the well-plate or vials containing the samples. **Be sure NOT to allow the fiber coating on the tips to dry (fiber color turns from gray to white) during the transfer process. If the coating dries, the extraction efficiency will be reduced.** If necessary, a secondary wetting solution of higher aqueous content can be utilized to minimize desolvation.
3. Sample extraction times vary depending on target analyte and sample matrix, but typical times range from 5-60 minutes. Continuous sample agitation is recommended to increase sample extraction efficiency, and to improve extraction consistency. Mechanical agitation is recommended; either orbital shakers or table-top shakers are recommended.

4. After extraction is complete, the LC tips can be transferred to other low volume vials or well plates for desorption, typically 100 µL conical vials or tapered well-plates are utilized. Desorption volumes will vary depending on the geometry of the desorption vessel. Sufficient volume is necessary to fully cover the fiber coating. Depending on the type of desorption vessel, volumes as little as 50 µL can be sufficient to fully desorb the fibers. Desorption solvents will vary depending on the target analytes. Typical solvents utilized are methanol or acetonitrile. Desorption solvent must be optimized depending on target analytes. Mechanical agitation is recommended during the desorption process, both to increase analyte recovery and improve consistency. The faster the agitation speed, the shorter the desorption time. With moderate agitation, such as 120 rpm, a typical desorption time would be 30 minutes. With rapid agitation, such as 500-700 rpm, desorption time can be reduced to 5-10 minutes. To compensate for evaporation of solvent, an internal standard should be spiked into the desorption solvent prior to dispensing into the vials or well-plate. Table 1 shows compatibility with water soluble (reverse-phase) solvents. The tips are not compatible with normal phase solvents, especially aromatic hydrocarbon and chlorinated solvents. These solvents will soften the binder used to hold the fiber in the tip that will eventually lead to leaking of sample into the tip.

Table 1. Solvent Compatibility

Solvent	Compatible	Not Compatible
Reverse-Phase Solvents		
Methanol	X	
Acetonitrile	X	
Water	X	
Ethyl acetate	X	
Ethanol	X	
Isopropanol	X	
100% Acetone		X
Acetone with minimum 20% water	X	

5. After desorption is complete, the tips can be removed and the vials capped for analysis. Depending upon the solvent used for analyte desorption, it may be necessary to evaporate and re-dissolve the sample into a more suitable solvent prior to analysis.

Alternatives to Solvent Desorption

With the advent of direct ionization sources for mass spectrometers, there have been several applications developed that interface the tips directly with both DESI systems and DART systems. This consists of inserting the tips directly into the ionization stream to desorb the analytes directly off of the fibers and into the mass spectrometers. With direct interface, analysis time can be greatly reduced allowing for real-time screening of samples.

Ordering Information

Description	Cat. No.
PDMS/DVB SPME Tips, pk of 96	57248-U
SPME LC Tips, C18, pk of 96	57234-U