Using a Solid Phase Micro Extraction Fiber Conditioner in High Throughput Trace Materials Analysis

Sample Preparation Challenges for Trace Materials Analysis

Trace materials analysis methods involve the extraction and concentration of compounds in air, aqueous solutions, soil, slurry and other complex matrices. Limits of trace detection for target analytes are typically of the order of parts per billion (ppb). Scientists very often face challenges in precise, timely and reliable sample preparation, both in a laboratory and at sample sites. Sample preparation for trace analysis can easily extend to over 80% of the total analysis time, therefore effective sample preparation is a high priority for high throughput projects.

A technique that provides effective solutions to sample preparation in trace analysis is the use of solid phase micro-extraction (SPME) fibers. SPME fiber sampling has been shown to be effective for trace detection in many complex matrices, including:

- chemical warfare agents in environmental samples and on clothes
- environmental pollution in soil and water
- food and beverage product flavors
- forensics investigations of fire accelerants, gunshot residue and post-blast explosions
- odors from drugs at clandestine labs and ports
toxicology cases involving blood alcohol and drugs in urine and serum

**Solid Phase Micro Extraction (SPME) Method**

SPME is a low cost, quick and flexible adsorption/desorption method of extracting compounds from samples. The technique integrates sampling, extraction, concentration and sample introduction into a single step, enabling solvent-free extraction using a fused silica or stainless steel fiber coated with a thin film polymer. The fiber acts as the solvent during the extraction of compounds. The fiber is mounted on syringe-like device for extraction of analytes from complex matrices and introduction to a chromatography system. Figure 1. shows how the SPME fiber sampling process works.

The extraction principle is based on an equilibrium process. Compounds can be extracted by immersing the fibers in a liquid sample or exposing those with significant vapor pressure to the
headspace of a solid, liquid or gas sample. The analytes adsorb to the polymer coating on the fibers. The adsorbed analytes are then thermally desorbed in the injector of a gas chromatograph (GC) for separation.

Selecting the most appropriate type of SPME fiber for analytes depends on the volatility and molecular weight (MW) together with the polarity of each target analyte. Fibers that have polar phases, usually polyacrylate and Carbowax (CW) coatings are best for polar analytes. Volatile analytes are best collected with a thicker 100 µm film. These fibers can also be used for less volatile compounds using longer extraction times. Small molecule analytes are best collected with porous fibers with Carboxen (CAR) or divinylbenzene (DVB) coatings. For large molecule analytes, thin film fibers with 7 µm and 30 µm polydimethylsiloxane (PDMS) coatings are more efficient.

The thickness of absorbent fiber coatings is also a consideration. Diffusion of an analyte from the sample matrix or headspace into the coating on the fiber is proportional to the thickness of the coating. A thicker film retains volatile compounds and transfers them to the GC injection port without loss. For higher boiling compounds, a thin film ensures fast diffusion and release of the analyte during thermal desorption. A thick film will effectively remove high boiling compounds from the sample matrix, but the desorption rate will be prolonged, and analytes could be carried over to the next extraction.

**SPME Fiber Conditioner**

At typically over US$100 each, SMPE fibers are expensive but they can be reconditioned between applications. The injector of a GC unit can be used for desorbing residues and contaminants off a fiber. With some SPME fibers, a high temperature is necessary to desorb high boiling residues completely. Often the GC injector cannot be set to a high enough temperature because a column with a low temperature limit is installed. Since SPME fibers can take up to four hours to condition/recondition, using the GC injection port wastes valuable instrument time and resources. Figure 2. shows a SPME fiber conditioner, a dedicated device for reconditioning the different types of fibers, one or more at the same time.

Using the Field Forensics SPME Conditioner X1 Model CN303R unit, the fibers can be reconditioned separately from the GC, with the recommended specifications for each. During this cleaning process a fiber is heated up to 350°C and is purged with an inert gas flow. In a second heated and purged port a new fiber can be prepared already offline for following analyses. The Model CN 303R can support up to four (4) separate ports, or more, to support a timely flow of multiple reconditioned fibers for high throughput trace materials analysis. Table 1. provides a summary of reconditioning settings for different SPME fibers.

To facilitate safe and secure sample collection and storage of SPME fibers, Field Forensics also provides two important accessories, the TuffSyringe Model TS100 and the SafePorter Model SP200. The TuffSyringe Model TS100 the most durable manual SPME manipulator available, constructed of stainless steel. The end caps are made of titanium; each unit has a slim design and is easily carried in a shirt or laboratory coat pocket. The titanium end caps protect the units during storage and transport and the Teflon insert envelopes the SPME needle and prevents loss or contamination of the sample or contamination of the SPME fiber.

The SafePorter Model SP200, is a field transport container constructed of machined aluminum, for SPME manual sample holders, syringes, and other samplers. It has a hermetic seal to preserve and protect the SPME sample. A septum in one end allows for sampling of the internal space before opening as a precaution against possible contamination of the manual sample holder.

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**Figure 2.** Field Forensics SPME Multi-Fiber Conditioner
Conclusions

To facilitate high throughput trace materials analysis utilizing SMPE techniques, Field Forensics provides a small, portable, cost-effective SPME fiber conditioner and storage accessories with a capacity for reconditioning one, two, three, four or more fibers at the same, using different processing settings. This unit operates independently of a GC-MS unit and can be set for optimum, simultaneous reconditioning of multiple and different types of SPME fibers. There is no need to tie up an expensive GC-MS with maintenance tasks.

About Field Forensics

Founded in 2001, Field Forensics, Inc. (FFI) is a US-owned developer and manufacturer of forensics sampling and identification devices for forensics, law enforcement, homeland security, public health and military customers. FFI has several product lines, which address market needs in field chemical extraction, explosives detection and substance identification, explosives screening, covert operations, human intelligence and source handling. Research and development in these and related technologies are continuously underway. FFI's current products are based on four technologies: (1) Colorimetric Chemistry, (2) Solid Phase Micro Extraction (SPME and PSPME), (3) Thin-Layer Chromatography, and (4) Raman Spectroscopy.

FFI offers SPME products for the laboratory and for the field. The TuffSyringe™ uses Supelco SPME fiber assemblies and offers users a very rugged manual SPME sampler for a very reasonable price. The SafePorter™ is designed to protect the Supelco Manual Sample Holder but can also be used with other types of devices. The Conditioner 1X™ Model CN303R, is the first commercially available portable and cost-effective SPME conditioner.

<table>
<thead>
<tr>
<th>Analyte (Polarity and Molecular Weight)</th>
<th>SPME Fiber Coating Type</th>
<th>Coating Thickness</th>
<th>Conditioning Temperature</th>
<th>Conditioning Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gasses and low MW compounds MW range 30-225</td>
<td>CAR/PDMS Adsorption</td>
<td>75/85 μm</td>
<td>260 °C</td>
<td>0.5 hours</td>
</tr>
<tr>
<td>Non-polar compounds MW range 125-600</td>
<td>PDMS Absorption</td>
<td>7 μm</td>
<td>320 °C</td>
<td>2 – 4 hours</td>
</tr>
<tr>
<td>Non-polar semi volatile compounds MW range 80-500</td>
<td>PDMS Absorption</td>
<td>30 μm</td>
<td>250 °C</td>
<td>1 hour</td>
</tr>
<tr>
<td>Polar semi-volatiles MW range 80-300</td>
<td>Polyacrylate Absorption</td>
<td>85 μm</td>
<td>300 °C</td>
<td>2 hours</td>
</tr>
<tr>
<td>Trace compound analysis MW range 40-275</td>
<td>DVB/CAR/PDMS Adsorption</td>
<td>50/30 μm</td>
<td>270 °C</td>
<td>4 hours</td>
</tr>
<tr>
<td>Volatile polar alcohols MW range 40-275</td>
<td>CW/DVB Adsorption</td>
<td>65 μm</td>
<td>250 °C</td>
<td>0.5 hours</td>
</tr>
<tr>
<td>Volatile polar amines MW range 50-300</td>
<td>PDMS/DVB Adsorption</td>
<td>65 μm</td>
<td>250 °C</td>
<td>0.5 hour</td>
</tr>
<tr>
<td>Volatiles and low MW compounds MW range 60-275</td>
<td>PDMS Absorption</td>
<td>100 μm</td>
<td>250 °C</td>
<td>1 hour</td>
</tr>
</tbody>
</table>

Table 1. Conditioning temperatures and times for SPME fiber types