An SPME Portable Field Sampler can be used to extract analytes from samples in the field, then store them for up to 3 days, or longer, before performing the analysis. A field sampler with a 100µm polydimethylsiloxane-coated fiber extracts chlorinated, organophosphorus, triazine, and other pesticides from water. After 24 hours of storage, analyte loss from storage containers was far greater than loss from a field sampler – more than 70%, versus 10-15%.

Table 1. Negligible Difference in Pesticide Response After 3 Days Storage on 100µm SPME Fiber

<table>
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<tr>
<th>Analyte</th>
<th>% Difference&lt;sup&gt;+&lt;/sup&gt;</th>
<th>Analyte</th>
<th>% Difference&lt;sup&gt;+&lt;/sup&gt;</th>
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<td>Organophosphorus/Triazine Pesticides</td>
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<tr>
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<td>-5.4</td>
<td>TEPP</td>
<td>4</td>
</tr>
<tr>
<td>β-BHC</td>
<td>-3.2</td>
<td>Thionazin</td>
<td>-5</td>
</tr>
<tr>
<td>γ-BHC</td>
<td>-7.4</td>
<td>Sulfotep</td>
<td>-4</td>
</tr>
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<td>δ-BHC</td>
<td>-4.7</td>
<td>Phorate</td>
<td>-10</td>
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<td>-2.3</td>
<td>Dimethoate</td>
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<td>1.3</td>
<td>Simazine (triazine)</td>
<td>-13</td>
</tr>
<tr>
<td>Heptachlor epoxide</td>
<td>3.3</td>
<td>Atrazine (triazine)</td>
<td>-5</td>
</tr>
<tr>
<td>Endosulfan I</td>
<td>3.7</td>
<td>Disulfoton</td>
<td>-9</td>
</tr>
<tr>
<td>4,4'-DDE</td>
<td>-6.3</td>
<td>Methyl parathion</td>
<td>-7</td>
</tr>
<tr>
<td>Dieldrin</td>
<td>3.1</td>
<td>Malathion</td>
<td>4</td>
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<tr>
<td>Endrin</td>
<td>-11.6</td>
<td>Parathion</td>
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<td>-6.2</td>
<td>Famphur</td>
<td>9</td>
</tr>
<tr>
<td>4,4'-DDD</td>
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<td>Mean Difference</td>
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</tr>
<tr>
<td>Endosulfan sulfate</td>
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<td></td>
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<tr>
<td>4,4'-DDT</td>
<td>-7.2</td>
<td></td>
<td></td>
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<tr>
<td>Endrin ketone</td>
<td>-5.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Methoxychlor</td>
<td>0.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean Difference</td>
<td>-2.6%</td>
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<sup>+</sup>% Difference =
\[
\text{response after 3 days/4°C} - \text{response immediately after extraction}
\]
\[
\text{response immediately after extraction}
\]

Table 2: Negligible Difference in Pesticide Response After 3 Days Storage on 100µm SPME Fiber

<table>
<thead>
<tr>
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<th>% Difference&lt;sup&gt;+&lt;/sup&gt;</th>
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<tbody>
<tr>
<td>Chlorinated Pesticides</td>
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</tr>
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<td>Heptachlor</td>
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<tr>
<td>Heptachlor epoxide</td>
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<tr>
<td>Endosulfan I</td>
<td>3.7</td>
</tr>
<tr>
<td>4,4'-DDE</td>
<td>-6.3</td>
</tr>
<tr>
<td>Dieldrin</td>
<td>3.1</td>
</tr>
<tr>
<td>Endrin</td>
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</tr>
<tr>
<td>Endosulfan II</td>
<td>-6.2</td>
</tr>
<tr>
<td>4,4'-DDD</td>
<td>14.2</td>
</tr>
<tr>
<td>Endrin aldehyde</td>
<td>-6.0</td>
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<td>-5.6</td>
</tr>
<tr>
<td>4,4'-DDT</td>
<td>-7.2</td>
</tr>
<tr>
<td>Endrin ketone</td>
<td>-5.8</td>
</tr>
<tr>
<td>Methoxychlor</td>
<td>0.1</td>
</tr>
</tbody>
</table>

<sup>+</sup>% Difference =
\[
\text{response after 3 days/4°C} - \text{response immediately after extraction}
\]
\[
\text{response immediately after extraction}
\]

A field sampler containing a 100µm PDMS fiber was developed specifically for extracting pesticides and other semivolatile organic compounds from water. The fiber very effectively retains chlorinated, organophosphorus, and triazine analytes for at least 3 days at 4°C (Table 1). In fact, after 24 hours of storage, analyte losses from whole samples in storage containers were far greater than losses from the field sampler – more than 70%, versus 10-15% (Table 2).

Figure 8 shows a chromatogram of the extracted pesticides, using an ion trap mass spectrometer. SPME and ion traps are very compatible, because SPME does not introduce a large solvent peak or a large amount of water into the trap. Compared to the extractions summarized in Tables 1 and 2, the extraction time was increased from 20 min to 30 min to improve precision. Salt was
Figure B. Pesticides at 10ppb in Water, Using SPME-GC/MS

<table>
<thead>
<tr>
<th>Analyte Class</th>
<th>Analyte</th>
<th>Fiber/4°C Fiber/RT Water/4°C Water/RT</th>
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<tbody>
<tr>
<td>1. 1,3-Butadiene-1,2-diol</td>
<td>-10</td>
<td>-4</td>
</tr>
<tr>
<td>2. Chlordane</td>
<td>-6</td>
<td>-8</td>
</tr>
<tr>
<td>3. Endosulfan I</td>
<td>-12</td>
<td>-15</td>
</tr>
<tr>
<td>4. Endosulfan II</td>
<td>-14</td>
<td>-17</td>
</tr>
<tr>
<td>5. Mirex</td>
<td>-16</td>
<td>-18</td>
</tr>
<tr>
<td>6. Dieldrin</td>
<td>-18</td>
<td>-20</td>
</tr>
<tr>
<td>7. Heptachlor</td>
<td>-20</td>
<td>-22</td>
</tr>
<tr>
<td>8. DDE</td>
<td>-22</td>
<td>-24</td>
</tr>
<tr>
<td>9. Dieldrin</td>
<td>-24</td>
<td>-26</td>
</tr>
<tr>
<td>10. Heptachlor</td>
<td>-26</td>
<td>-28</td>
</tr>
<tr>
<td>11. Mirex</td>
<td>-28</td>
<td>-30</td>
</tr>
<tr>
<td>12. Dieldrin</td>
<td>-30</td>
<td>-32</td>
</tr>
<tr>
<td>13. Heptachlor</td>
<td>-32</td>
<td>-34</td>
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<tr>
<td>14. DDE</td>
<td>-34</td>
<td>-36</td>
</tr>
<tr>
<td>15. Mirex</td>
<td>-36</td>
<td>-38</td>
</tr>
<tr>
<td>16. Dieldrin</td>
<td>-38</td>
<td>-40</td>
</tr>
</tbody>
</table>

Note: Fiber/4°C/Fiber/RT Water/4°C; Water/RT

Table 2. Difference in Analyte Response after 24 Hours of Storage Compared to Response for Freshly Prepared, Extracted, and Analyzed Samples

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Treatment / °C</th>
<th>% Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>TEPP</td>
<td>-8</td>
<td>-54</td>
</tr>
<tr>
<td>Thionazin</td>
<td>-3</td>
<td>-68</td>
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<td>Sulfotep</td>
<td>4</td>
<td>-81</td>
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<tr>
<td>Phorate</td>
<td>-3</td>
<td>-84</td>
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<tr>
<td>Simazine</td>
<td>10</td>
<td>-53</td>
</tr>
<tr>
<td>Atrazine</td>
<td>15</td>
<td>-57</td>
</tr>
<tr>
<td>Lindane</td>
<td>-2</td>
<td>-74</td>
</tr>
<tr>
<td>Disulfoton</td>
<td>-8</td>
<td>-93</td>
</tr>
<tr>
<td>Methyl parathion</td>
<td>7</td>
<td>-68</td>
</tr>
<tr>
<td>Malathion</td>
<td>6</td>
<td>-74</td>
</tr>
<tr>
<td>Parathion</td>
<td>15</td>
<td>-83</td>
</tr>
<tr>
<td>Heptachlor epoxide</td>
<td>12</td>
<td>-83</td>
</tr>
<tr>
<td>DDE</td>
<td>12</td>
<td>-98</td>
</tr>
<tr>
<td>Fampur</td>
<td>3</td>
<td>-60</td>
</tr>
<tr>
<td>Endrin ketone</td>
<td>10</td>
<td>-82</td>
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<tr>
<td>Methoxychlor</td>
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<td>-88</td>
</tr>
<tr>
<td>Mean Difference</td>
<td>-8%</td>
<td>-15%</td>
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</table>

These data show that a single type of SPME fiber—a 100µm PDMS fiber—can be used to extract a variety of pesticides. The extraction technique easily can be adapted to use in the field, with minimal equipment, by the development of the portable field sampler. Analytes stored on the SPME fiber are more stable than in stored water samples. Pesticides are unstable in water, regardless of the type of container used.

Ordering Information:

- **SPME Portable Field Sampler, pk. of 2**
  - with 100µm PDMS fiber...
  - with 75µm Carboxen<sup>TM</sup>/PDMS fiber...
- **SPME Holder (replaceable fiber, manual version)**
  - with 100µm PDMS fiber...
  - Replacement 100µm PDMS fibers, pk. of 3...

For additional SPME products, refer to our catalog or contact your Supelco products distributor.

Contact our Technical Service Department

- (phone 800-359-3041 or 814-359-3041, FAX 800-359-3044 or 814-359-5468) for expert answers to your questions.

For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website (www.sigma-aldrich.com), see the Supelco catalog, or contact Supelco, Beeston, PA 16823-0048 USA.

For additional Supelco products, contact your local distributor or visit our website (www.sigma-aldrich.com) for more information.

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