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If you have questions about applying methodology described in this article to a current application, please contact our technical service chemists.

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Field Sampling for Pesticides, Using Solid Phase Microextraction/Capillary GC

R. Shirey, Sample Handling, Supelco, Bellefonte , PA, USA

The SPME Portable Field Sampler is a simple, reliable sampling unit that can eliminate the need to fill, transport, and ultimately dispose of bulky vials or bottles – with better analyte recovery.

By optimizing extraction conditions according to the matrix and the class of analytes, solid phase microextraction (SPME)* can be used to monitor chlorinated pesticides, organophosphorus pesticides, carbamates, and other pesticides in water, soil, and food samples. A newly designed SPME apparatus, the SPME Portable Field Sampler (Figure A), enables environmental analysts to extract samples in the field, store them on the SPME fiber for up to 3 days – or longer – and analyze the adsorbed analytes without additional processing after returning to the lab. The field sampler is simple to use: expose the SPME fiber to the sample to be tested, then retract the fiber within a septum seal in the handle. Ultimately, when the fiber loses its analyte-extracting ability (typically 50-100 samples), replace the entire sampler.

Figure A. SPME Portable Field Sampler

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A field sampler with a 100µm polydimethylsiloxane (PDMS) fiber very effectively retains chlorinated, organophosphorus, and triazine analytes for at least 3 days at 4°C. In fact, after 24 hours of storage, analyte losses in glass or plastic storage containers were far greater than losses from the field sampler – more than 70%, versus 10-15%.

Table 1 shows recovery values for extracted analytes stored on the SPME fiber for 24 hours, then analyzed, and for analytes in water samples stored for 24 hours, followed by extraction and analysis. The results, particularly the losses of chlorinated pesticides, were surprising. These data show that storing water samples – even at 4°C – may produce unreliable results.

For most of the analytes, recovery from silanized glass containers was only slightly better than from non-silanized glass containers (Table 1). Plastic containers were better for some pesticides, but were dramatically worse for others.

Losses were smaller for analytes stored at 4°C than at room temperature, but the differences were not great. As expected, the more volatile organophosphorus analytes appeared to be more affected by temperature. Thus, when possible, an SPME fiber containing extracted analytes should be stored in a controlled environment, such as in a cooler with an ice pack. The storage temperature showed even less effect on the water samples, but precautions always should be taken to minimize analyte loss on storage.

In summary, these data show that it is best to extract samples in the field, rather than store them whole. *Pesticides stored on a 100µm PDMS fiber in an SPME Portable Field Sampler are more stable than analytes in whole samples stored in glass or plastic containers.*

Table 1. Difference in Analyte Response after 24 Hours of Storage Compared to Response for Freshly Prepared, Extracted, and Analyzed Samples▼

	Treatment▼▼ / % Difference						
Analyte	Fiber/ 4°C	Fiber/ RT	Water/ 4°C	Water/ RT	Silanized	Non-Silanized	Plastic
TEPP	- 8	- 33	- 54	- 67	- 54	- 58	- 65
Thionazin	- 3	9	- 68	- 75	- 68	- 71	- 52
Sulfotep	4	- 23	- 81	- 88	- 81	- 81	- 97
Phorate	- 3	- 27	- 84	- 95	- 83	- 89	- 96
Disulfoton	- 8	- 34	- 93	- 95	- 92	- 94	- 99
Methyl parathion	- 7	- 7	- 68	- 73	- 66	- 67	- 58
Malathion	- 6	- 6	- 74	- 78	- 73	- 81	- 76
Parathion	- 15	- 26	- 83	- 84	- 78	- 84	- 89
Famphur	- 3	2	- 60	- 61	- 59	- 66	- 51
Simazine◆	- 10	3	- 53	- 50	- 48	- 56	- 43
Atrazine◆	- 15	1	- 57	- 55	- 52	- 59	- 44
Lindane●	- 2	- 12	- 74	- 75	- 71	- 79	- 69
Heptachlor epoxide●	- 12	- 21	- 83	- 90	- 79	- 77	- 97
DDE●	- 12	- 23	- 98	- 94	- 84	- 80	- 98
Endrin ketone●	- 10	- 15	- 82	- 82	- 80	- 87	- 88
Methoxychlor●	-14	- 25	- 88	- 89	- 87	- 88	- 99
Mean Difference	**- 8%**	**- 15%**	**- 75%**	**- 78%**	**- 72%**	**- 76%**	**- 76%**

▼% Difference = $\frac{\text{(response after storage) – (response immediately after extraction)}}{\text{response immediately after extraction}}$

▼▼RT = room temperature. Analytes stored in silanized glass at 4°C unless otherwise stated.

SPME Fiber: **100µm PDMS**
Cat. No.: **504823** (portable field sampler)
Sample: Pesticides in water / 25% NaCl, pH = 8
4mL sample in 4mL vial
Extraction: 20 min (direct immersion, constant stirring)
Desorption: 10 min, 260°C

●Chlorinated pesticide
◆Triazine
All others - organophosphorus pesticides

Ordering Information:

Description	Cat. No.
SPME Portable Field Sampler, pk. of 2 with 100µm PDMS fiber	**504823**
with 75µm Carboxen™/PDMS fiber	**504831**

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

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