

Solid Phase Microextraction for HPLC Analysis of Carbamate and Urea Pesticides

SPME is a viable alternative to time-consuming and expensive solvent-based extraction techniques often used in HPLC analyses of pesticides. A 60 μ m polydimethylsiloxane/divinylbenzene SPME fiber proved effective for concentrating carbamate and urea pesticides.

Key Words:

- carbamate pesticides • urea pesticides
- solid phase microextraction • SPME • HPLC

Pesticides traditionally are extracted from water samples by liquid-liquid extraction or by solid phase extraction. These methods, while reliable, can be labor intensive and require large volumes of sample and solvents.

Solid phase microextraction (SPME)* is showing great potential as an extraction method for GC analyses of pesticides. With the development of an SPME/HPLC interface, the HPLC analyst now can take advantage of the time and solvent savings offered by SPME.

The interface permits transfer of collected analytes from the SPME fiber to an HPLC column. This unit consists of a six-port injection valve and a desorption chamber that replaces the injection loop in the HPLC system. The SPME fiber is introduced into the desorption chamber under ambient pressure.

Our SPME fiber coated with 60 μ m of polydimethylsiloxane/divinylbenzene (PDMS/DVB) is flexible, rugged, and compatible with the organic solvents used in HPLC mobile phases. It can be used repeatedly with consistent results. We used a 60 μ m PDMS/DVB fiber to extract carbamate and urea pesticides at 8ppb from a water sample spiked with a standard 15-pesticide mix. Figure A1 shows the result of direct injection of the standard mixture, using the SPME/HPLC interface rather than an injection loop (total volume ~60 μ L). In contrast, SPME concentrated the last six carbamates, thereby increasing the sensitivity of the analysis (Figure A2). This enabled us to monitor these compounds reliably at levels as low as 1.5ppb (Figure B). Addition of salt slightly improved extraction.

The PDMS/DVB fiber is excellent for concentrating this and other classes of pesticides and herbicides. For further information on SPME pesticides applications, contact our Technical Service chemists (phone 800-359-3041 or 814-359-3041), or your local Supelco representative.

Figure A. Carbamate and Urea Pesticides at 8ppb from Water

Sample: 3mL water containing 8ng/mL of each analyte (except: 100ng/mL carbofuran, 80ng/mL propoxur, 20ng/mL carbaryl) in 10% NaCl
 Direct Injection: 60 μ L sample through interface
 SPME Fiber: PDMS/DVB, 60 μ m film
 Cat. No.: 57317
 Extraction: immersion, 40 min, rapid stirring
 Desorption: static, 5 min in acetonitrile:water (65:35); dynamic, valve open during run
 Column: SUPELCOSIL™ LC-8, 15cm x 4.6mm ID, 5 μ m particles
 Cat. No.: 58220-U
 Mobile Phase: acetonitrile:water (18:82 to 65:35 in 9 min, hold 3 min)
 Flow Rate: 2.0mL/min
 Temp.: 35°C
 Det.: UV, 240nm

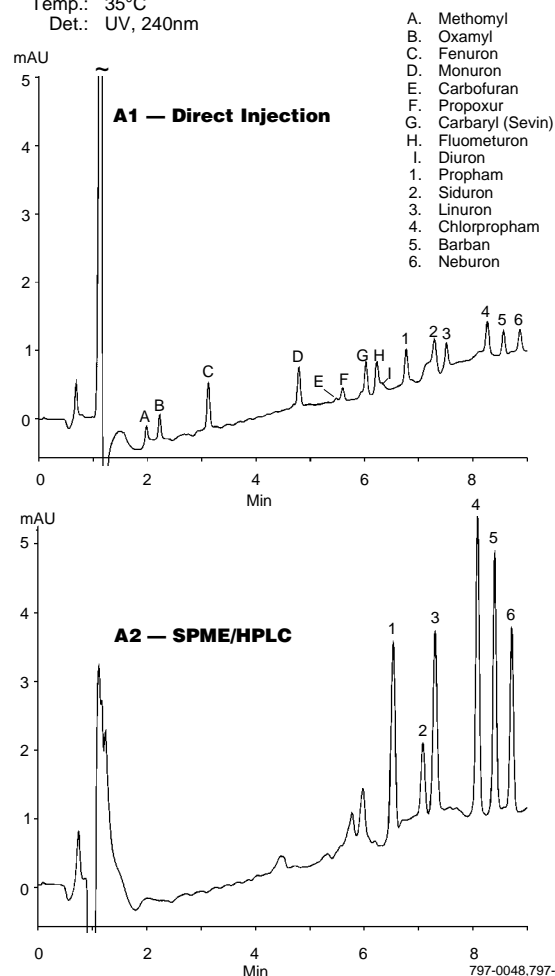
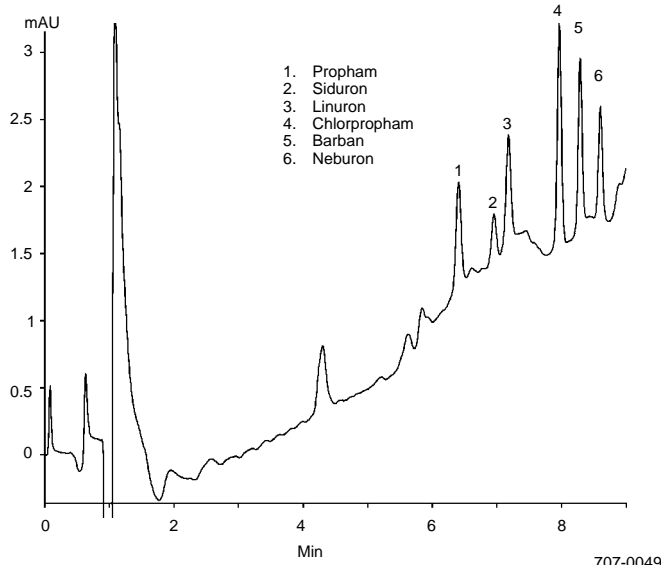


Figure B. Carbamate and Urea Pesticides at 1.5ppb from Water

Sample: 3mL water containing 1.5ng/mL of each analyte in 10% NaCl
 SPME Fiber: PDMS/DVB, 60µm film
 Cat. No.: 57317
 Extraction: immersion, 60 min, rapid stirring
 Desorption: static, 5 min in acetonitrile:water (65:35); dynamic, valve open during run
 Column: SUPELCOSIL LC-8, 15cm x 4.6mm ID, 5µm particles
 Cat. No.: 58220-U
 Mobile Phase: acetonitrile:water (18:82 to 65:35 in 9 min, hold 3 min)
 Flow Rate: 2.0mL/min
 Temp.: 35°C
 Det.: UV, 240nm



SPME Sampling Stand



995-0250

Holds eight 4mL vials while supporting the SPME syringe for consistent fiber immersion. Order heat/stir plate separately.

Ordering Information:

Description	Cat. No.
SPME Fiber Assembly, pk. of 3 60µm PDMS/DVB	57317
SPME Holder** For HPLC	57331
SPME/HPLC Interface with Valco® valve with Rheodyne® valve	57350-U 57353
SPME Sampling Stand (see photo)	57333-U
Hot Plate 110VAC 220VAC	Z11,0914 Z26,2007
SUPELCOSIL LC-8 HPLC Column 15cm x 4.6mm ID, 5µm particles 15cm x 4.6mm ID, 3µm particles	58220-U 58983

For a list of more than 800 pesticide and pesticide metabolite chemical standards, refer to our catalog or request our pesticides product specification brochure (697002).

* Solid phase microextraction technology licensed exclusively to Supelco. US patent pending; European patent #0523092.

** Initially you must order both holder and fiber assembly. Holder is reusable indefinitely.

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Trademarks

Rheodyne – Rheodyne, Inc.
 SUPELCOSIL — Sigma-Aldrich Co.
 Valco – Valco Instruments Co., Inc.

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.

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