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Fast, Accurate Detection of Amphetamines in Urine, Using Solid Phase Microextraction/ Capillary GC

In monitoring methamphetamine and amphetamine in urine, heated headspace SPME offers a 20-fold increase in sensitivity, relative to conventional heated headspace sampling. Correlation coefficients for methamphetamine and amphetamine were 0.9999 for 0.2-10mg/liter concentrations and 0.9970 for 5-100mg/liter concentrations.

Key Words:
- amphetamines • drug abuse
- solid phase microextraction • SPME

Staff members of the Department of Legal Medicine, Hiroshima University School of Medicine and the Department of Legal Medicine, Fukuoka University School of Medicine have developed an accurate, simple, and rapid method for analyzing urine for methamphetamine (MA) and its principal metabolite, amphetamine (AP), using heated headspace solid phase microextraction (SPME•) (1). A 1mL urine sample is sealed in a 12mL vial, internal standard (5µg pentadeuterated methamphetamine, prepared according to reference 2) and 0.7g potassium carbonate are added, and the sample is heated at 80°C for 20 minutes in a block heater. An SPME fiber coated with a 100µm film of polydimethylsiloxane is exposed to the headspace above the sample for 5 minutes, then introduced into the injection port of a capillary gas chromatograph. In a system equipped with mass spectrometry/chemical ionization selected ion monitoring (GC-MS/CI-SIM), this analysis was 20 times as sensitive as a method incorporating conventional headspace extraction (Figure A). Correlation coefficients for MA and AP, based on d_5-MA, were 0.9999 for concentrations from 0.2 to 10mg/liter and 0.9970 for concentrations from 5 to 100mg/liter (Figure B). Coefficients of variation for 5mg/liter of AP and MA in urine were 7.0% and 5.1%, respectively.

In addition to speed, simplicity, and accuracy, the headspace SPME method can, under some circumstances, reduce the potential for interference by co-administered drugs. In an immunoassay for MA/AP, chlorpromazine and its metabolites can cause a false positive result (3), but the headspace SPME extraction was not interfered with by these compounds.

NOTE: These authors have developed a similar procedure for monitoring amphetamines in blood (N. Nagasawa, M. Yashiki, Y. Iwasaki, K. Hara, and T. Kojima, *Rapid Analysis of Amphetamines in Blood Using Head Space-Solid Phase Microextraction and Selected Ion Monitoring* in *Forensic Science International* **78** (2), 1996). In place of potassium carbonate, 0.5mL 1N NaOH is used to drive the analytes into the headspace.

Figure A. SPME is Effective for Detecting Methamphetamine and Amphetamine in Urine

Sample:	1mL urine + 100µg each analyte, 5µg d_5-methamphetamine, 0.7g K_2CO_3 in 12mL vial
SPME Fiber:	**100µm polydimethylsiloxane**
Cat. No.:	**57300-U** (manual sampling)
Extraction:	headspace, 80°C, 5 min (after 20 min sample warming period)
Desorption:	3 min, 250°C
Column:	poly(dimethylsiloxane), 15m x 0.53mm ID, 2.0µm film
Oven:	110°C
Carrier:	nitrogen, 25mL/min
Det.:	FID, 250°C
Inj.:	splitless, 250°C

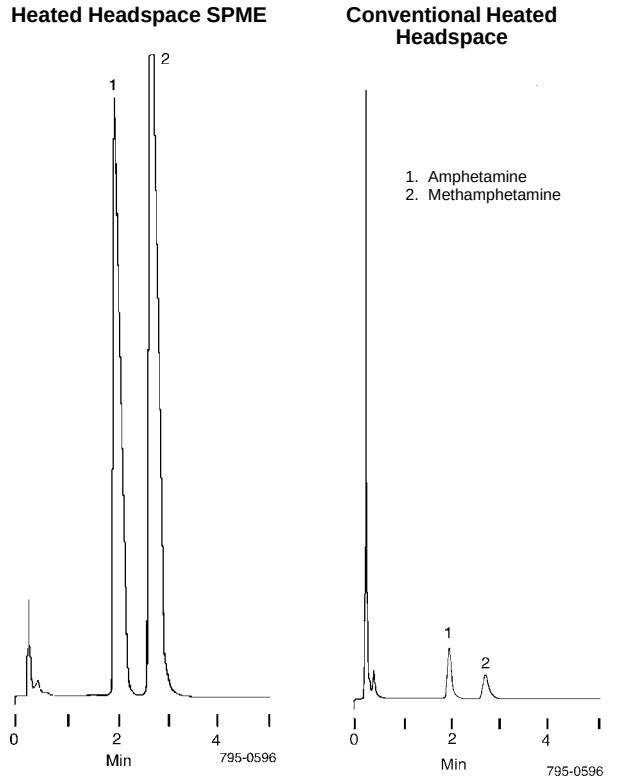
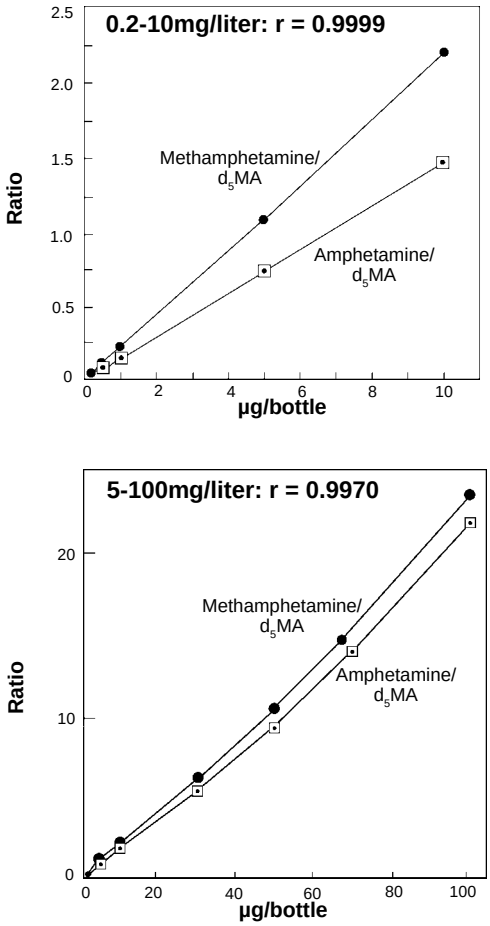


Figure provided by M. Yashiki, T. Kojima, T. Miyazaki, N. Nagasawa, and Y. Iwasaki, Hiroshima University School of Medicine, Hiroshima, Japan and K. Hara, Fukuoka University School of Medicine, Fukuoka, Japan.

• Technology licensed exclusively to Supelco. US patent 5,691,206; European patent #0523092.

Figure B. SPME Extraction of Methamphetamine and Amphetamine Is Linear

Extraction and chromatography described in Figure A.



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References

1. Yashiki, M., T. Kojima, T. Miyazaki, N. Nagasawa, Y. Iwasaki, and K. Hara, *Forensic Science International* **76:** 169-177 (1995).
2. Hara, K., T. Nagata, and K. Kimura, *Z. Rechtsmed.,* **96:** 93-104 (1986).
3. Olsen, K.M., M. Gulliksen, and A.S. Christophersen, *Clin. Chem.,* **38:** 611-612 (1992).

References not available from Supelco.

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Ordering Information:

Description	Cat. No.
SPB™-1 Capillary Column	
15m x 0.53mm ID, 2.0µm film	**available on request**
SPME Fiber Holder♦	
For manual sampling	**57330-U**
For Varian 8100/8200 AutoSampler (requires Varian SPME upgrade kit)	**57331**
SPME Fiber Assembly (pkg. of 3)	
7µm polydimethylsiloxane coating	
For manual sampling	**57302**
For Varian 8100/8200 AutoSampler	**57303**
100µm polydimethylsiloxane coating	
For manual sampling	**57300-U**
For Varian 8100/8200 AutoSampler	**57301**
30µm polydimethylsiloxane coating	
For manual sampling	**57308**
For Varian 8100/8200 AutoSampler	**57309**
85µm polyacrylate coating	
For manual sampling	**57304**
For Varian 8100/8200 AutoSampler	**57305**
SPME Fiber Assortment Kit	
One fiber each of 85µm polyacrylate coating and 100µm and 7µm polydimethylsiloxane coating	
For manual sampling	**57306**
For Varian 8100/8200 AutoSampler	**57307**
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Inserts, Varian SPI Models 1093-94SP1, Series 3000 GC	
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pk. of 5	**26364,05**
pk. of 25	**26364,25**
Inlet Liners, Splitless, 0.75mm ID■	
For Hewlett-Packard GC - 5700, 5800, 5890	
each	**26375,01**
pk. of 5	**26375,05**
pk. of 25	**26375,25**
For Varian GCs (all)	
each	**26358,01**
pk. of 5	**26358,05**
pk. of 25	**26358,25**

For additional SPME fibers and fiber kits, sampling vials, and other SPME accessories, refer to the Supelco catalog.

♦First time users must order both holder and fiber assembly. Holder is reusable indefinitely.

■You can reduce inlet volume by replacing a standard 2mm ID liner with this specially designed liner. This increases sensitivity and provides sharper peaks.

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