

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 1 mL urine sample is sealed in a 12 mL vial, internal standard (5 µg pentadeuterated methamphetamine, prepared according to reference 2) and 0.7 g 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 100 µm 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 d₅-MA, were 0.9999 for concentrations from 0.2 to 10 mg/liter and 0.9970 for concentrations from 5 to 100 mg/liter (Figure B). Coefficients of variation for 5 mg/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, 0.5 mL 1N NaOH is used to drive the analytes into the headspace.

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

Sample: 1 mL urine + 100 µg each analyte, 5 µg d₅-methamphetamine, 0.7 g K₂CO₃ in 12 mL vial
SPME Fiber: **100 µm 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), 15 m x 0.53 mm ID, 2.0 µm film
Oven: 110 °C
Carrier: nitrogen, 25 mL/min
Det.: FID, 250 °C
Inj.: splitless, 250 °C

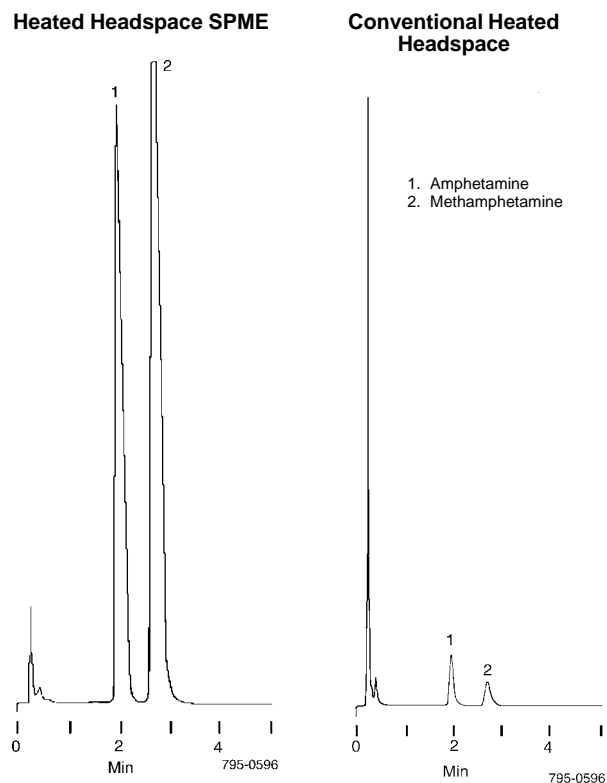
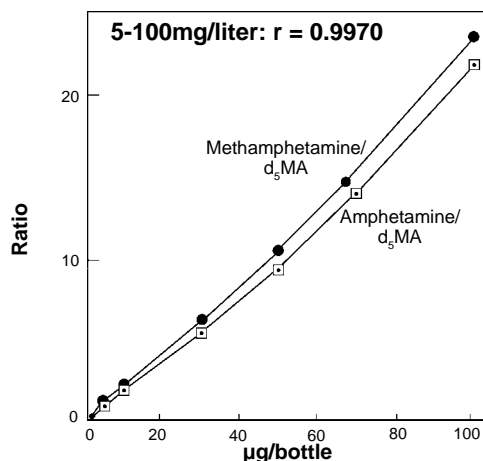
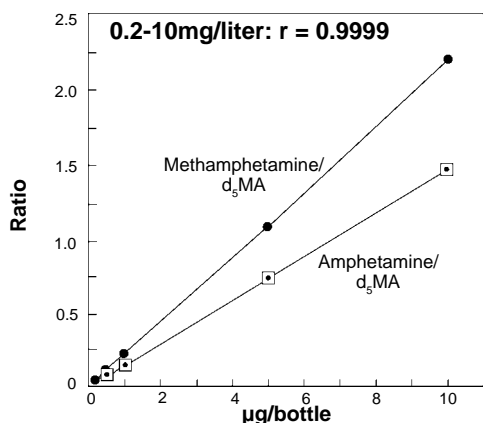


Figure provided by M. Yashiki, T. Kojima, T. Miyazaki, N. Nagasawa, and Y. Iwasaki, Hiroshima University School of Medicine, Hiroshima, Japan and K. Hara, Fukuoka University School of Medicine, Fukuoka, Japan.

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Figure B. SPME Extraction of Methamphetamine and Amphetamine Is Linear

Extraction and chromatography described in Figure A.



995-0597, -0598

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.

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Acknowledgment

This investigation was conducted by Mikio Yashiki, Tohru Kojima, Tetsuji Miyazaki, Nobuyuki Nagasawa, and Yasumasa Iwasaki, Department of Legal Medicine, Hiroshima University School of Medicine, 1-2-3 Kasumi, Minami-ku, Hiroshima 734, Japan and Kenji Hara, Department of Legal Medicine, Fukuoka University School of Medicine, 7-45-1 Nanakuma, Jonan-ku, Fukuoka 814-01, Japan.

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