K7 A Validated Analytical Method for Simultaneous Detection of ATS in Human Urine Using SPME-GC/MS

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After attending this presentation, attendees will understand the benefits of using Solid-Phase Microextraction-Gas Chromatographic/Mass Spectrometry (SPME-GC/MS) to analyze Amphetamine-Type Stimulants (ATS) in human urine and the quantification results of authentic urine samples.

This presentation will impact the forensic science community by presenting a validated analytical method for simultaneous detection of ATS in human urine.

The Fluorescence Polarization Immunoassay (FPIA) as a preliminary test sometimes gives false positive results due to structural similarity between ATS. A rapid and easy SPME-GC/MS screening method has been developed and validated for simultaneous detection of nine ATS in human urine. These drugs include methamphetamine, amphetamine, Methylenedioxymethamphetamine (MDMA), and 3,4-methylenedioxyamphetamine (MDA) which are popular abused drugs in Korea. Phentermine, phenmetrazine, ephedrine, pseudoephedrine, and norephedrine/norpseudoephedrine also show cross-reactivity by amphetamine FPIA.

The conditions of SPME extraction time, temperature, and deposition time were optimized to yield the highest peak area. The extraction condition using 100µm Polydimethylsiloxane (PDMS) fiber was 80°C for 25min with 250rpm and deposition time was 4min in the GC injector at 280°C with split mode (ratio 5:1). To prevent carryover, the fiber was baked out pre- and post-injection into the GC/MS. The GC separation was performed using temperature program of 100-300°C at 10°C/min.

The data was collected by extracted ion chromatogram for each drug from total ion chromatograms by full scan mode and calculated by peak area ratios (peak area drug/peak area internal standard). All nine ATS showed good resolution with retention times from 4.45min to 9.33min. Methamphetamine and ephedrine were especially easy to discriminate by retention time. High concentrations of ephedrine and low concentrations of methamphetamine in urine are difficult to distinguish from each other because of similar retention time and similar fragmented ions by Pentafluoropropionic Acid Anhydride (PFPA) derivatization. The calibration curve showed acceptable linearity for each drug with R^2>0.99. The results of the intra-and inter-day precision and accuracy were satisfactory: <10% for precision and within ±10% for accuracy at three different concentrations (167, 333, 1,000ng/mL). In this analytical method, no significant matrix effect was observed and high recoveries (>92%) were achieved. Thus, SPME-GC/MS has the advantages of an easy sample preparation with acceptable accuracy and precision for the simultaneous quantification. It will be a useful method for a simple and rapid screening analysis of urines for ATS abuse.

SPME-GC/MS, Amphetamine Type Stimulants, Validation

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