



Development and Validation of an Analytical Method for the Simultaneous Determination of Benzoylecgonine and Ecgonine Methyl Ester in Human Urine by Gas Chromatography/Mass Spectrometry

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SUMMARY. A simple and rapid procedure has been developed and validated for the simultaneous determination of benzoylecgonine and ecgonine methyl ester in human urine samples. After extraction and sample cleanup by solid-phase extraction (SPE), the extracts were derivatized with 50 μ L of MTBSTFA and analyzed by gas chromatography/mass spectrometry (GC/MS). The limit of detection (LOD) was 50 ng/mL for both metabolites. The recoveries were 81% and 75.3% for benzoylecgonine and ecgonine methyl ester, respectively. The intra-assay precision (% relative standard deviation) at two different added amounts (200 and 1000 ng/mL) in urine matrix varied from 4.8 to 8.4 and from 4.4 to 5.7, respectively. The mass spectra obtained for each compound have many diagnostics ions with relative abundance in accordance with the WADA requirements.

KEY WORDS: Benzoylecgonine, Ecgonine methyl ester, Doping control, Gas chromatography/mass spectrometry.

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