

Evaluation of hair hydrolysis and MEPS columns for the detection of amphetamines derivatives in hair

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ORIGINAL ARTICLE

ABSTRACT

Introduction: Different extraction and cleanup procedures have been used to identify amphetamines in hair samples. The microextraction by packed sorbent (MEPS), a miniaturized approach of the classic solid-phase extraction (SPE), has only been used so far to determine the presence of amphetamine (AMP) and methamphetamine (MAMP) in hair. The aim of this work was to evaluate different hair sample extraction conditions, namely hydrolysis, to maximize the detection of AMP, MAMP, MDA, MDMA, MDE, and MBDB in combination with MEPS and GC-MS.

Methodology: Pulverized authentic hair (50mg) was incubated with 500 μ L of NaOH 1M at 80°C for 1H, 60°C overnight and 45°C overnight¹. After incubation, the extracts were neutralized with 50 μ L of 10M HCl and subsequently centrifuged. The supernatant was tested with several conditions to access the most suitable when paired with MEPS clean-up. The studied conditions were as follows: the addition of 500 μ L of H₂O; supernatant filtration followed by addition of H₂O; and precipitation with cold ACN followed by centrifugation. After clean-up, 30 μ L of MBTFA were added for evaporation under nitrogen gas. After being fully dried, 50 μ L of MBTFA was added and derivatization was done with a microwave oven. Finally, the extract was injected into a GC-MS and analysed.

Results: Results obtained from these tests indicated that the best hydrolysis for this purpose was obtained with 500 μ L NaOH 1M at 45°C overnight, with the addition of H₂O before MEPS clean-up. The clean-up procedure was previously optimized by design of experiments (not being the scope of this study). Furthermore, M1 and C18 MEPS sorbents were tested to check which one would provide cleaner extracts and greater analyte signals. A concentration of 2.00 ng/mg of AMP, MAMP, MDA, MDMA, MDE, and MBDB was used for this test.

Discussion: Considering the results, both columns presented similar recoveries for each amphetamine, when using equal hydrolysis and MEPS conditions.

Keywords: hair analysis, amphetamines, hydrolysis.

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