K1 Effects of Hair Bleachers in the Analysis of Amphetamine(s) and Bath Salt Drugs

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After attending this presentation, attendees will learn about the effects of hair bleaching in the analysis of amphetamines and synthetic cathinones in hair using readily available Solid Phase Extraction (SPE) cartridges and tandem mass spectrometry.

This presentation will impact the forensic science community by offering analysts operating in forensic facilities information about the impact of bleaching materials used on hair analyzed for amphetamines and synthetic cathinones (bath salt) drugs analyzed by Liquid Chromatography with Tandem Mass Spectrometry (LC/MS/MS) and solid phase extraction.

Method: Samples of decontaminated hair (10mg) containing amphetamines and bath salt-type drugs (amphetamine, methamaphetamine, methylenedioxyamphetamine (MDA), methylenedioxymethamphetamine (MDMA), butylone, ethylone, flephedrone, mephedrone, methylone, methedrone, methcathinone (4-MEC), methylenedioxypyravalerone (MDPV), and pyravalerone were treated with 10% aqueous sodium hypochlorite solution (bleach), 3% aqueous hydrogen peroxide solution, or 3% aqueous ammonium hydroxide solution for 2 hr before being removed, washed, and dried. The samples were then digested in 0.1M NaOH (containing deuterated analogues) for 0.5 hr at room temperature. Each solution was adjusted to pH6 with 0.1M phosphate buffer (4mL) and applied to a conditioned SPE column. The samples were extracted on commercially available SPE columns (C8/SCX). After loading the sample, the sorbent was washed with deionized water, acetic acid (0.1M), and methanol (3mL of each, respectively). Each SPE column was dried and eluted with 3mL of a solvent consisting of methylene chloride/isopropanol/ammonium hydroxide (78:20:2). After elution, 200µL of mobile phase was added to the collection tube. The samples were then evaporated to the mobile phase for analysis by LC/MS/MS in positive Multiple Reaction Monitoring mode (MRM). Data is presented for MRMs of amphetamine, methamphetamine, MDA, and MDMA, butylone, ethylone, flephedrone, mephedrone, methylone, methedrone, methcathinone (4-MEC), methylenedioxypyravalerone (MDPV), and pyravalerone (and deuterated analogues), respectively.

Liquid chromatography was performed in gradient mode employing a 50 x 2.1mm C_{18} analytical column and a mobile phase consisting of acetonitrile and 0.1% aqueous formic acid. The gradient was programmed to run from 5% to 90% acetonitrile in 4.0 min and then back to 5% for re-injection. The total run time for each analysis was less than five minutes.

Tandem mass spectrometry was performed in using positive MRM mode. The following transitions were monitored (quantification ions underlined): Amphetamine m/z: 136.1 to 91.0, 65.0, Amphetamine-d_5: m/z 141.1 to 124.0, 93.0, Metamphetamine: m/z 150.1 to 91.1, 119.1 Methamphetamine-d_5: m/z 155.2 to 92.1, 121.2, MDA: m/z 180.2 to 163.1, 105.1, MDA-d_5: m/z 185.2 to 168.1, 110.1, MDMA: m/z 194.2 to 163.1. 105.1. MDMA-d_5: m/z 199.2 to 165.1, 106.8, Butylone: m/z 222.1 to 174.2, 204.2, Ethylone: m/z 222.1 to 174.2, 204.2, Flephedrone: m/z 182.1 to 164.2, 149.1, Mephedrone: m/z 178.1 to 145.1, 160.1, Methylone: m/z 208.1 to 160.1, 132.1, Methedrone: m/z 194.1 to 176.2, 161.1, Methylethylcathinone (4-MEC): m/z 192.1 to 174.2, 144.1, Methylenedioxypyravalerone (MDPV): m/z 276.2 to 135.1, 126.1 and Pyravalerone: m/z 246.2 to 105.1, 175.2, respectively. In this presentation, representative chromatograms are shown to illustrate the efficiency of the chromatography and analysis of amphetamine and synthetic cathinones

Results: The limits of detection/quantification for the SPE method were determined to be 0.05ng/mg and 0.1ng/mg, respectively for the amphetamines (amphetamine, methamphetamine, MDA, and MDMA) and synthetic cathininones (butylone, ethylone, flephedrone, mephedrone, methylone, methedrone, methcathinone (4-MEC), methylenedioxypyravalerone (MDPV), and pyravalerone). The method was found to be linear from 0.1ng/mg to 10ng/mg (r^2>0.999). Data is presented to show that recoveries of amphetamine were found to be greater than 90%. Interday and Intraday analysis of amphetamine were found to <7% and <10%, respectively. Matrix effects were determined to be <5%. Degradation of the amphetamines ranged from 77% to 45%, while the degradation of the synthetic cathinones ranged from 25% to 100% for the bleaching agents.

Conclusion: The use of the information given in this new procedure for the analysis of amphetamine and synthetic cathinones will be of great use to analysts in the field of forensic hair analysis as it demonstrates the use of SPE/LC/MS/MS to provide valuable data from about the effects of bleaching agents in hair analysis.

Hair, LC/MS/MS, SPE

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