

Detection of methylenedioxypyrovalerone (MDPV), 4-methylmethcathinone (mephedrone), and 4-methoxymethcathinone (methedrone) by gas chromatography-chemical ionization tandem mass spectrometry

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ABSTRACT

Bath salts have recently gained popularity among recreational drug users. Thus, bans are beginning to occur for the compounds commonly found in the products. The determination and development of the most suitable method for the identification of common bath salts ingredients mephedrone, methedrone, and MDPV using gas chromatography-mass spectrometry was achieved.

Samples were analyzed on a Varian 8400 GC coupled to a Varian 1200 MS capable of single and triple quadrupole analysis in electron ionization or chemical ionization mode. Chromatograms and mass spectra were obtained in EI GC-MS, EI GC-MS/MS, CI GC-MS, and CI GC-MS/MS modes, then compared to determine the most definitive results. Triple quadrupole analysis was performed as a full scan or multiple reaction monitoring. CI GC-MS/MS analysis in MRM mode sufficiently characterized the studied compounds.

In order to accommodate the nature of rapidly-evolving designer drugs, this method is designed to incorporate new compounds as they are introduced on the market.

INTRODUCTION

As of July 2011, U.S. poison control centers have received 3,740 exposure reports for the year, compared to only 303 calls in 2010 regarding synthetic cathinones. This outbreak has led many states to control or ban many of the synthetic cathinones. Kansas is one such state, and the synthetic cathinones have been controlled as a class of compounds. Through this legislation, MDPV, mephedrone, and methedrone, among others, have been banned.

Synthetic cathinones have been sold as "bath salts" or "plant food." Product names include Ivory Wave, Vanilla Sky, Energy1, White Lightning, and Hurricane Charlie. Mephedrone, methedrone, and MDPV are some of the most common compounds seen in "bath salts." They are structurally similar to the stimulants cathinone and methcathinone. Many other synthetic cathinones are also structurally similar to each other. As a result, they have similar fragmentation patterns when characterized by traditional GC-EIMS methods; therefore, the EI mass spectra are not particularly useful for identification.

In this study, EI and CI, and single and triple quadrupole (QqQ) mass spectrometry were evaluated in order to determine the most definitive method for identification of synthetic cathinones.

MATERIALS AND METHODS

- Mephedrone (Cerilliant)
- Methedrone (Cerilliant)
- MDPV (Cayman Chemical) [5 ng/µL]

[100 ng/µL] [100 ng/µL]

- Varian CP-3800 GC/1200 MS
 - Column: VF-5ms FactorFour (30m x 0.25mm x 0.25µm) - Injection volume: 1µL
 - Reagent gas (CI): methane
 - Source pressure (CI): 3.5 Torr
 - Filament current: 50µA (EI), 250µA (CI) – CID gas: argon
 - CID pressure: 1.5 mTorr
- Research Temperature Program
 Final Temperature Program - Initial: 80°C (3 min) - Initial: $100^{\circ}C$ (1 min)
- Ramp: 10°C/min
- Final: 280°C (3 min)
- Total run time: 26 min
- Ramp: 200°C, 20°C/min
- Final: 290°C, 30°C/min (1 min)
- Total run time: 10 min



caffeine, ketamine), Interference Mix 2 (methamphetamine, pseudoephedrine, MDA, BZP, TFMPP, MDMA, cocaine, codeine, diazepam, hydrocodone, oxycodone alprazolam, clonazepam, heroin), inositol, phenethylamine, and cathinone.

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DISCUSSION

2: MDPV is distinguishable from the other two compounds, but ot be definitively identified. Mephedrone and methedrone tra were very similar, with strong m/z 58 ions and little else. QqQ: MDPV exhibited a more characteristic spectrum. The r two remained indistinguishable from one another due to lowil fragmentation, but the abundance of the ions was increased. Q: Soft ionization produced low-detail fragmentation in all ctra. Each spectrum included a protonated molecular ion. QqQ: Selecting the [M+H]⁺ for each compound produced ctra that allowed for differentiation and identification. Unique gments were detected due to the soft ionization and sensitivity n QqQ analysis. Fragments were then chosen from each full to create a more sensitive and selective MRM method, where h compound produced a clean peak and unique spectrum. interference occurred during the CI QqQ MRM analysis of 23 pounds with similarities in physical appearance or structure.

CONCLUSION

le some useful data can be obtained for mephedrone, edrone, and MDPV using EI and CI in single and triple rupole analyses, the developed method utilizing CI QqQ des the most definitive results for the identification of the ed compounds. It, too, is most likely to provide such conclusive for other synthetic cathinones encountered in the future. CI ces protonated molecular ions of high enough intensity to be ole precursor ions. Triple quadrupole analysis allows for the on and detection of ions unique to a given compound, dless of similar structures or chromatographic retention times.

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