B15 Comparison of Extraction Procedures for Organic Impurity Profiling of Seized MDMA Tablets

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The goal of this presentation is to optimize and compare two alternative extraction procedures with conventional liquid-liquid extraction (LLE) for organic impurity profiling of seized tablets containing 3,4-methylenedioxy methamphetamine (MDMA).

This presentation will impact the forensic community by potentially identifying an effective method for extracting organic impurities from MDMA. This allows for the production of an MDMA impurity profile, which has potential in aiding law enforcement in monitoring drug trafficking.

Organic impurity profiling of seized synthetic drugs is used to potentially identify common production sources. In the case of MDMA, similarities among impurity profiles can indicate a common synthetic route and similar levels of the same impurities potentially indicate a common production batch. Currently, LLE is typically used to extract organic impurities from tablets with the extract subsequently analyzed by gas chromatography-mass spectrometry (GC-MS) to generate the impurity profile. This study aims to investigate two alternative extraction procedures; headspace-solid phase microextraction (HS- SPME) and microwave-assisted extraction (MAE), both of which offer numerous advantages compared to LLE procedures.

HS-SPME offers more selective extractions than LLE based on choice of fiber type, and requires less sample than LLE. Impurities are pre-concentrated on the SPME fiber, which is advantageous for trace impurity determinations. In addition, the low volatility of the MDMA salt prevents efficient partitioning into the headspace. Hence, profiles obtained with HS-SPME are not dominated by MDMA, as is often the case with LLE procedures. However, HS-SPME has many variables, such as fiber type, extraction time, and extraction temperature that must be optimized to afford efficient extractions.

Microwave-assisted extraction offers highly efficient extractions using small volumes of solvent, and requires fewer steps than LLE. However, the efficiency of the extraction may also prove problematic since other compounds in the sample are extracted and may interfere with subsequent instrumental analysis. Hence, MAE is often followed by HS-SPME for the selective extraction of the target analytes. MAE has not yet been considered as an extraction procedure for organic impurity profiling.

In this study, an HS-SPME procedure was optimized based on extraction time and extraction temperature using a circumscribed central composite experimental design. A similar approach was also used to optimize the MAE procedure in terms of solvent type, solvent volume, extraction time, and extraction temperature. The MAE procedure was then used in combination with the optimized HS-SPME to selectively extract impurities from the MAE extract. Both the HS-SPME and the MAE/HS-SPME procedures were compared with a conventional LLE procedure in terms of the number of impurities extracted from a homogenized batch of seized MDMA, as well as the repeatability and reproducibility of the extraction.

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Impurity Profiling, Head Space-Solid Phase Microextraction, Microwave-Assisted Extraction

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