A115 Optimization of a Microwave Assisted Extraction/Headspace Solid - Phase Microextraction (MAE/HS - SPME) Procedure for Organic Impurity Profiling of Seized 3,4-Methylenedioxymethamphetamine (MDMA) Tablets

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After attending this presentation, attendees will be familiar with the application of microwave assisted extraction (MAE) and headspace solid - phase microextraction (HS-SPME) for organic impurity profiling of 3,4-methylenedioxymethamphetamine (MDMA) tablets.

This presentation will impact the forensic community by offering an extraction procedure that overcomes limitations in commonly used liquid-liquid extraction procedures and yields more informative organic impurity profiles. With more informative organic impurity profiles, greater confidence is achieved in the association and discrimination of organic impurity profiles from different MDMA exhibits.

Several different methods are used by clandestine laboratories to synthesize MDMA. Because these labs typically do not employ quality control measures, impurities resulting from starting materials and by-products of the reaction are often present in the final MDMA product. Typically, organic impurities are extracted from MDMA using a LLE procedure, and the extract is analyzed by gas chromatography-mass spectrometry (GC-MS), generating an impurity profile of the tablet. Based on the impurities present, the synthetic route used to manufacture the MDMA may be determined since some impurities are route specific. Furthermore, similar levels of the same impurities may imply that the tablets originated from a common clandestine lab. However, the LLE procedure commonly used for impurity extraction requires a relatively large sample mass and often efficiently extracts MDMA which can mask the presence of trace level impurities in the final chromatogram. Due to these limitations, alternative extraction procedures may be more useful to obtain informative organic impurity profiles.

In the presented research, MAE followed by HS-SPME is investigated as a possible alternative to LLE. MAE is first used to extract the organic compounds of the sample into a buffer solution. Because MAE provides highly efficient extractions, all components in the sample that are soluble in the buffer are extracted. The HS-SPME step after the MAE allows for the selective extraction of impurities. Due to the low volatility of the salt form (which is typically present in tablets), MDMA is not efficiently extracted by HS-SPME. In addition, HS-SPME offers the advantage of pre-concentrating impurities on the fiber which is especially important for impurities present at trace levels. Therefore, the resulting organic impurity profile is likely to be more informative.

A factorial experimental design is used to screen for the important parameters in the MAE and to evaluate the interaction of the different parameters. The parameters of the microwave extraction screened in the design include extraction time, extraction temperature, ramp rate, and sample mass. A central composite design is used to optimize the important parameters determined by the screening design. The HS- SPME procedure utilized was previously developed in our lab. The development of the MAE/HS-SPME procedure for the extraction of organic impurities from seized MDMA will be presented and compared to conventional LLE procedures based on the number of impurities extracted and the repeatability and reproducibility of the extraction, as well as the overall chromatography.

3,4-Methylenedioxymethamphetamine (MDMA), Microwave Extraction, Impurity Profile

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