A187 Differentiation of MDMA Exhibits Using Liquid-Liquid Extraction, Headspace Solid Phase Microextraction, and Gas Chromatography-Mass Spectrometry

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After attending this presentation, attendees will have learned about impurity profiling procedures for 3,4-methylenedioxymethamphetamine (MDMA). This research compares liquid-liquid extraction and headspace solid phase microextraction procedures, as well as different GC temperature programs.

This presentation will impact the forensic science community by investigating the effect of extraction procedure and GC temperature program on the association and discrimination of MDMA tablets based on organic impurities. Determining the more effective extraction procedure and GC temperature program would be a step towards standardizing the procedures used in forensic laboratories for this application. This would allow better comparison of data collected in different laboratories which will be necessary for the successful identification of drug trafficking routes.

MDMA is a Schedule I controlled substance, which is often found as an active ingredient in ecstasy tablets. Surveys such as the 2010 Monitoring the Future Survey indicate that abuse of controlled substances, including MDMA, continues to be a growing trend in the United States, with a significant increase in MDMA use for all ages questioned since the previous study five years prior. This increase has created a greater need for quick and easy identification methods for illicit tablets, as well as improved methods for the identification and prevention of drug trafficking.

Impurity profiling can be used to determine the synthesis method of MDMA in illicit tablets, potentially linking tablets to a common production source, based on the impurities present. Profiling is conventionally done using liquid-liquid extraction (LLE) and gas chromatography-mass spectrometry (GC/MS), but recent literature has also shown the potential benefits of using headspace solid phase microextraction (HS-SPME) as an alternative extraction procedure. However, reports in the literature use different GC temperature programs to analyze extracts and different statistical methods are used to evaluate the impurity profiles.

The objective of this research is to investigate the effect of the extraction procedure and GC temperature program on the association and discrimination of MDMA tablets based on the organic impurities. Determining the more effective extraction procedure and GC temperature program would be a step towards standardizing the procedures used in forensic laboratories for this application. This would allow better comparison of data collected in different laboratories, which will be necessary for the successful identification of drug trafficking routes.

Three different MDMA exhibits were used for this preliminary study. Tablets from each exhibit were homogenized to minimize variation within each exhibit due to the clandestine manufacturing process. Samples from each exhibit were extracted using both LLE and HS-SPME. Extracts were then analyzed in replicate (n=5) using four different GC temperature programs that ranged in the number of temperature ramps (one-, two-, and three-step ramps), as well as the total analysis time (36-53 minutes).

The resulting total ion chromatograms from each extraction/GC temperature program combination were treated as separate data sets and subjected to principal components analysis (PCA). This multivariate statistical procedure is used to identify sources of variance within a data set, while reducing dimensionality to allow simpler visualization. In this case, the PCA scores plots were used to assess association of replicates of the same exhibit and discrimination among exhibits. In addition, the PCA loadings plots were also used to identify those impurities contributing most to the variance in the data set. Such impurities are potentially useful chemical markers for discrimination of MDMA exhibits. Additional statistical procedures, such as Pearson product moment correlation (PPMC) coefficients and student t-tests, were used to evaluate the association and discrimination of exhibits observed in the PCA scores plots. Association of replicates from the same exhibit and discrimination among exhibits was possible irrespective of GC temperature program and extraction procedure.

MDMA, Impurity Profiling, Multivariate Statistics

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