K11 Parameters Optimization Associated With the Analysis of Methylenedioxymethamphetamine (MDMA) and Related Compounds in Biological Matrices

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The learning objectives of this presentation are to characterize and evaluate parameters that are pertinent to the analysis of methlenedioxymethamphetamine (MDMA) and related compounds in biological specimens.

With increasing report on MDMA abuse and required analysis, a systematic evaluation on parameters associated with the analysis of MDMA and related compounds is undertaken, including methylenedioxyamphetamine (MDA), amphetamine (AM), and methamphetamine (MA). Parameters studied included (a) three solid-phase adsorbents; (b) five derivatization reagents; and (c) four deuterated internal standards.

Ions resulting from the use of various derivatization reagents that are potentially useful for selected-ion-monitoring (SIM) for qualitative and quantitative determination of MDMA, MDA, MA, and AM are listed in Tables 1 and 2. TMSand TCA-derivatives do not generate adequate number of qualified ion-pairs as required in common SIM practice. Among those generating adequate number of qualified ionpairs, HFB-derivatives appear to produce higher ion intensities (ionization efficiencies). Some of the ion-pairs selected from the HFBderivatives have low relative intensities in their respective spectra; however, this unfavorable factor appears to be adequately compensated for by the enhanced ionization efficiency and desirable limits of quantitation and detection still can be achieved.

HFB-derivatives of the analytes and internal standards were used to evaluate the effectiveness of internal standards. Ions adapted to designate MDMA/MDMA-d_5, MDA/MDA-d_5, MA/MA-d_8, and AM/AMd_8 are: *m/z* 254/258, 162/167, 254/261, and 240/243. Integrated SIM intensities of these ions are used for further statistical analysis. In this study, four sets of standard solutions containing all four compounds at five low concentrations (2, 5, 10, 20, and 40 ng/mL) were prepared with all four internal standards (10 ng/mL). Another four sets with analytes at higher concentrations (100, 250, 500, 1000, and 2000 ng/mL) were also prepared (internal standard concentrations = 500 ng/mL). The first set of the four was first used as the calibrators for the calculation of analyte concentrations in the other three sets. The same process was followed by using the second, the third, and the fourth sets as the calibrators. This same process was applied to both the low and the higher concentration sets. In these calculations, MDMA-d_5 and MDA-d_5 were sequentially used as the internal standards to calculate the concentrations of MDMA and MDA. Similarly, MA-d_8 and AM-d_8 were sequentially used as the internal standards to calculate the concentrations of MA and AM. Statistical methods were then used to determine whether analyte concentrations resulting from the use of different internal standard were statistically different. Relevant statistical data are shown in Table, while the interpretation of these data are summarized in Table 4. Further studies are currently in progress to further understand the performance characteristics exhibited by the AM/MA and the MDMA/MDA pairs. These characteristics will be evaluated along with what has been observed [1] in an earlier study on the barbiturate system.

1. Liu RH, McKeehan AM, Edwards C, Foster GF, Bensley WD, Langner JG, Walia AS: Improved gas chromatography/mass spectrometry analysis of barbiturates in urine using centrifuge-based solidphase extraction, methylation, with d_5-pentobarbital as internal standard; *J Forensic Sci* 39:1501–1514; 1994.

MDMA, MDA, Internal Standard

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