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TITLE

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LEARNING OBJECTIVE and **OUTCOME**

TEXT

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

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~~(what the attendee can expect to learn, retain, or implement into their practice)~~

Learning Objectives: To characterize and evaluate parameters that are pertinent to the analysis of methylenedioxymethamphetamine (MDMA) and related compounds in biological specimens.

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

Recovery data (Table 1) resulting from the three commercial solid adsorbents studied are compatible, all superior over the liquid-liquid extraction approach.

TABLE 1—*Recoveries of liquid-liquid and solid-phase extraction using adsorbents from various commercial sources.*

Extraction	Drug	Recovery (mean of triplicates, standard deviation) at various concn (ng/mL) 100	250	500	1000	2000
Liquid-liquid	MDA	75.1, 0.12	74.7, 0.97	75.9, 2.97	77.0, 3.17	84.6, 3.47
	MDMA	79.3, 1.02	82.7, 1.37	78.2, 0.92	77.3, 1.48	92.2, 0.40
Isolute®	MDA	96.6, 1.95	97.3, 0.31	94.3, 2.12	96.4, 0.36	94.0, 2.04
	MDMA	96.4, 2.73	96.8, 2.02	97.0, 3.02	97.1, 1.87	94.2, 2.11
SPEC® Plus™	MDA	98.8, 1.20	95.0, 8.47	94.5, 0.81	86.9, 9.15	85.9, 4.10
	MDMA	101.4, 1.57	96.4, 5.38	95.6, 1.32	92.8, 7.61	94.8, 4.25
Bond-Elut® certify™	MDA	98.5, 4.63	96.9, 1.67	93.9, 1.76	93.1, 2.13	92.4, 2.03
	MDMA	97.3, 1.15	99.4, 1.38	93.8, 0.65	91.1, 3.89	92.7, 0.77

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 and MDA are listed in Table 2. Ions listed in this table meet the following criteria: the contribution of these ions' intensities by their isotopic analogs are < 1%. TMS- and TCA-derivatives do not generate adequate number of qualified ion-pairs as required in common SIM practice. Among those generating adequate number of qualified ion-pairs, HFB-derivatives appear to produce higher ion intensities (ionization efficiencies). Some of the ion-pairs selected from the HFB-derivatives 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.

The third part of this study evaluates the precision and accuracy of quantitation data resulting from the use of the following deuterated internal standards: amphetamine-d_8, methamphetamine-d_8, MDA-d_5, MDMA-d_5. For example, the precision and accuracy of MDA and MDMA data resulting from the use of MDA-d_5 or MDMA-d_5 are compared. If no significant difference is apparent, perhaps only one internal standard (MDA-d_5 or MDMA-d_5) is adequate for the quantitation of MDA and MDMA. As reported earlier [1], the use of a deuterated analog of the analyte does not necessary produce better results.

(Continued)

KEY TERMS Three

TABLE 2—*Cross-Contribution of ions designated for MDA and MDMA and their deuterated analogs (SIM data)*

Derivatization	3,4-Methylenedioxyamphetamine (MDA) Suitable ions* (% cross contribution by analog) Analyte	Int. std.	3,4-Methylenedioxymethamphetamine (MDMA) Suitable ions* (% cross contribution by analog) Analyte	Int. std.
TMS	116 (0.29%)	120 (0.32%)	130 (0.64%)	134 (0.19%)
TFA	275 (0.03% 162 (0.30%) 140† (0.06%)	280 (0.0%) 167 (0.0%) 144† (0.0%)	289 (0.0%) 154 (0.21%) 110 (0.31%)	294 (0.0%) 158 (0.0%) 113 (0.0%)
PFP	325 (0.0%) 190† (0.0%) 162 (0.22%)	330 (0.0%) 194 (0.0%) 167 (0.0%)	339† (0.0%) 204 (0.0%)	344† (0.0%) 208 (0.0%)
HFB	375‡ (0.0%) 240† (0.0%) 162 (0.23%)	380‡ (0.0%) 244† (0.0%) 167 (0.0%)	389‡ (0.0%) 254 (0.0%) 210 (0.40%)	394‡ (0.0%) 258 (0.0%) 213 (0.0%)
TCA	190 (0.30%) 162 (0.61%)	194 (0.63%) 167 (0.0%)		

* These ions meet the following two criteria: (a) Ions designated for the isotopic analog pair differ by 3 or more mass units; (b) intensity contributed by the isotopic analog is less than 1.0%.
† The relative intensities of these ions are low (in the 8–10% range).
‡ Relative intensities of these ions are very low (in the 2–4% range).

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 solid-phase extraction, methylation, with d_5-pentobarbital as internal standard; *J Forensic Sci* 39:1501–1514; 1994.

MDMA, MDA, Internal Standard, Quantitation, Solid-Phase Extraction, GC-MS