B101 Solid-Phase Microextraction Based Approach for Enantiomeric Analysis of Amphetamines

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The goal of this presentation is to advance the development in solid-phase microextraction (SPME), specifically, to explore an one-step absorption/derivatization approach for enantiomeric analysis of amphetamines.

This presentation will benefit the forensic scientist's analytical knowledge of these SPME techniques.

As a solventless approach and with advances in fiber manufacturing technology, SPME has great potential in various sample pretreatment processes. Reported applications of this technology to the analysis of amphetamines included: (a) the inclusion of the derivatizing reagent in the sample matrix [1] or the chromatographic injection port [2]; (b) a two-step approach in sequentially placing the fiber in the headspace of the sampleand derivatizing reagent-containing vials [3]; and (c) an one-step procedure in placing the fiber in the headspace of the derivatizing reagent-containing vial, which was in turn placed in the headspace of the sample vial [4]. This current study represents a further progress in the application of the SPME technology. Specially, an onestep process is used to complete the absorption/derivatization process for the analysis of the enantiomeric compositions of amphetamines.

(S)-(-)-N-(Trifluoroacetyl)-prolyl chloride (l-TPC) was adapted as the chiral derivatizing reagent and added directly into the sample matrix. Temperature, absorption/desorption duration, and the amount of derivatizing reagent were studied to determine their effects on the yields of analytes on the fiber. The derivatization products resulting from this study show excellent desorption characteristics of the polydimethylsiloxane-coated fiber (100 µm) used in this study. For example, an onetime 5-min desorption leaves no detectable carry over. Optimal operational parameters (absorption: 70 ^{o}C for 10 minutes; injection: 250 ^{o}C for 5 minutes) cause minimal negative impact on the fiber, allowing repeated use (> 30 times) of the fiber.

This method was evaluated for its effectiveness (a) in quantitative determination of the enantiomeric pairs of amphetamine and methamphetamine — in terms of repeatability, linearity, and limits of detection and quantitation; and (b) by comparing its analytical findings with those derived from a conventional liquid-liquid extraction approache (Table 1).

Table 1. Comparison of methamphetamine/amphetamine enantiomeric compositions resulting from two sample preparation protocols (SPME and liquid-liquid extraction)

SPME (concentration in ng/mL)

Sample	d-Methamp	l-Methamp	d-Amp	l-Amp.
1	12,169	1,280	2,477	72
2	1,821	219	1,010	42
3	7,436	593	1,329	36
4	13,845	1,960	2,581	94
5	3,766	340	2,675	93
6	10,745	1,254	3,324	92
7	3,090	704	1,466	78
8	2,482	5,863	580	403
9	5,398	486	1,971	50
10	6,667	608	2,376	84
11	6,761	947	2,220	78
12	8,600	972	1,308	90

Liquid-liquid (concentration in ng/mL)

Sample	d-Methamp	l-Methamp	d-Amp	l-Amp.
1	10,638	1,564	2,702	63
2	1,427	146	976	32
3	7,768	429	1,318	37
4	11,753	1,233	2,545	67
5	3,310	252	2,763	72
6	9,931	1,315	2,973	79
7	2,564	591	1,381	79
8	2,010	6,317	482	538
9	4,680	435	1,928	43
10	5,649	543	2,230	66
11	5,632	449	2,100	73
12	6,276	950	1,276	58

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SPME, Enantiomeric Analysis , Amphetamines

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