Impurity Profiling of Crystalline Methamphetamine (‘Ice’) by Automated Solid-Phase Microextraction (SPME) Coupled with Gas Chromatography-Flame Ionization Detection

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AIMS: Impurities of methamphetamine (MA) show different patterns under various conditions of synthesis, and they can be utilized for investigation of their origins and smuggling routes. Generally, there are only traces of impurities in crystalline MA (‘Ice’). Specific extraction methods are required to selectively identify impurities in MA. Liquid-liquid extraction (LLE) method has been widely used for impurity profiling for its stability and compatibility, but it is limited by the high concentration of MA that co-extracts. Headspace solid-phase microextraction (SPME) and thermal desorption (TD) methods have been developed as complementary techniques for selective extraction of trace impurities. The aim of this work was to develop an automated SPME method coupled with gas chromatograph-flame ionization detector (GC-FID) to improve reproducibility and reliability of the profiling result.

METHODS: Areas of impurity peaks were normalized by using solid nonadecane (C19) diluted with potassium bromide (KBr) powder as an internal standard. A bi-polar SPME fiber coated with divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) was used for extraction of various impurities, and the extraction condition was optimized at 85°C for 20 min after incubation for 5 min. The suitability for impurity profiling was examined by comparing extraction efficiency of the SPME with the LLE method. LLE methods developed by National Research Institute of Police Science (NRIPS) and the United Nations International Drug Control Programme (UNDCP) are most widely used recently for extraction of MA impurities. The NRIPS method was used in this work.

RESULTS: Intensities of volatile and some of semi-volatile impurities extracted by SPME method were much higher than those extracted by LLE method, and interferences of MA and its artifacts were not observed. Highly reliable profiling results could be obtained by using SPME method for confirmation of similarity, which comes from high selectivity for impurities and differences in impurity patterns from LLE method. By cluster analysis of 11 MA samples of different origins, 9 samples were classified by both methods, but 2 samples could be classified only by SPME method.

CONCLUSIONS: Improved reliability of the profiling result obtained by SPME method will contribute to efficient investigation on the origins and smuggling routes of MA seizures.

Keywords: Impurity profiling, Methamphetamine, Solid-phase microextraction (SPME)