Impurity Profiling of Amphetamine-Type Stimulant Tablets Seized in Japan by GC-MS

Hiroyuki Inoue*, Yuko T. Iwata, Kenji Kuwayama, Kenji Tsujikawa, Tatsuyuki Kanamori, and Hajime Miyaguchi
National Research Institute of Police Science, Kashiwa, Chiba, Japan

AIMS: There has been a considerable increase in the number of seizures of amphetamine-type stimulant (ATS) tablets in Japan. Characterization and classification of seized ATS tablets can provide very useful information in criminal investigations aimed at identifying drug traffic routes, the sources of supply and relationship between seizures. In the present study, chemical profiling of organic impurities in ATS tablets was examined by gas chromatography-mass spectrometry (GC-MS).

METHODS: ATS tablets were ground to a powder, and 25 mg of the homogeneous sample was dissolved in 1 mL of 1 M Tris-HCl buffer, pH 8.0. The suspension was vortexed for 5 min and centrifuged. The supernatant was filtered through a membrane filter (0.45 µm). The filtrate was extracted with 0.5 mL of dichloromethane (containing n-docosane as IS). After centrifugation, the lower organic layer was subjected to GC-MS. The column was a DB-5 capillary column (length, 30 m; i.d.; 0.32 mm, film thickness, 1.0 µm). The oven temperature was programmed as follows: 50°C for 1 min, 10°C/min to 300°C. The injector and ion source temperatures were set at 240°C and 230°C, respectively. Helium was used as the carrier gas at a constant column flow-rate of 2 mL/min. Injection of 2 µL of the extract was made in the splitless mode.

RESULTS: In the early stage of this study, optimization was made for extraction conditions of organic impurities in ATS tablets. As pH was increased, extraction efficiencies of basic impurities became better, while those of neutral impurities not impacted. Higher pH also enlarged peaks of main components (MDMA and MDA) in tablets, leading to the overload of samples into the instrument. As a compromise, pH 8.0 was adopted for further experiments. Dichloromethane efficiently extracted impurities under pH 8.0 among organic solvents tested (ethyl acetate, diethyl ether, t-butylmethyl ether, toluene, dichloromethane and 1-chlorobutane). Fifteen impurity peaks were selected for comparative analysis of tablets. Twenty-two samples were classified into at least 3 groups by a hierarchical cluster analysis. One group was composed of MDA tablets, and another 2 were composed of MDMA tablets, which might indicate the different routes of MDMA synthesis.

CONCLUSIONS: Impurity profiling of ATS tablets seized in Japan was examined by GC-MS after optimization of extraction conditions. ATS tablets were classified into at least 3 groups (one MDA and two MDMA groups). The method would provide useful information about relationship between tablets.

Keywords: Amphetamine-type stimulants, Impurity profiling, GC-MS