The impurity characteristics of methamphetamine synthesized by Emde and Nagai method

BEOM JUN KO[1,2], SUNGILL SUH[1], YONG JUN SUH[1], MOON KYO IN[1], SUNG-HONG KIM[3]

1 Drug Signature Analysis Center, Supreme Prosecutors' Office, Seoul 137-730, Korea

2 School of Agricultural Biotechnology, Seoul National University, San 56-1, Sillim-Dong, Gwanak-gu, Seoul, 151-742, Korea

3 Korea Basic Science Institute, Daegu branch, 1370, Sankyuk-Dong, Puk-gu, Daegu, 702-701, Korea

AIMS: Most of illicit methamphetamine in Korea are thought to be synthesized by Nagai method or Emde method, because ephedrine or pseudoephedrine is commonly detected as one of organic impurities in seized methamphetamine samples. It would be very useful that the synthetic method of a seized methamphetamine can be determined for the illicit drug investigation. In this study, methamphetamines were synthesized by Nagai method or Emde method, and analyzed by GC-MS to find characteristic features of organic impurities depending on their synthetic methods. Chloroephedrine, an intermediate of Emde method, was also synthesized and analyzed.

METHODS: Methamphetamines were synthesized from ephedrine by Emde method and Nagai method. Chloroephedrine, the intermediate of Emde method was also synthesized. These 3 synthesized materials and 52 samples of seized methamphetamine were analyzed by GC-MS. Approx. 100mg of sample was dissolved in 2.0ml of 0.1M potassium phosphate buffer (pH 7.0) with 0.25ml-10% Na_2CO_3 and extracted 0.2ml of ethyl acetate containing 50μg/ml of n-nonadecane ($C_{29}H_{60}$). The mixture was centrifuged for 10 min at 3,000rpm and the organic layer was transferred to a vial for GC-MS analysis. The GC-MS analysis was performed on Jeol JMS700M station interfaced to HP6890 plus GC. An analysis condition was as followed. The column was a fused-silica capillary column, DB-1 (0.25mm × 30m i.d., film thickness 0.25μm). The GC was operated in the splitless mode for 1.0 min and sample injection volume was 1.0μl. The injector, detector and source temperature were maintained at 250°C, 300°C and 280°C, respectively. The oven temperature program was as followed: initial temperature at 40°C for 2 min, increase at 10°C/min to 280°C, and hold for 4 min. The carrier gas was He at a flow rate of 1.0ml.

RESULTS: The methamphetamine synthesized by Emde method has one unique impurity, which is characterized by the peak of retention time 12.2 minutes (m/z=120, base peak). The impurity (m/z=120) is also detected in chloroephedrine synthesized in our laboratory. Therefore, it could be concluded that the impurity (m/z=120) is originated from chloroephedrine, which is an intermediate of Emde method. The methamphetamine synthesized by Nagai method has 1,3-dimethyl-2-phenylnaphthalene and 1-benzyl-3-methylnaphthalene as its own unique impurities, and these two impurities are not detected in the methamphetamine synthesized by Emde method.

According to these characteristics of organic impurities, 52 seized methamphetamines were classified into 4 Types; Nagai Type that is probably synthesized by Nagai method, Chloroephedrine Type by Emde method, Undetermined Type I which has no characteristic impurities and Undetermined Type II which is almost pure methamphetamine with very small amount of common impurities such as N-formylmethamphetamine, N-acetylmethamphetamine, etc. 27 of 52 seized methamphetamines are Chloroephedrine Type, 16 of them are Nagai Type, 3 of them are Undetermined Type I, and the rests are Undetermined Type II.

CONCLUSIONS: There are distinct differences in organic impurities between two kinds of methamphetamine synthetic methods, Emde method and Nagai method. Methamphetamine synthesized by Emde method is characterized by the peak of retention time 12.2 minutes (m/z=120, base peak). Methamphetamine synthesized by Nagai is characterized 1,3-dimethyl-2-phenylnaphthalene and 1-benzyl-3-methylnaphthalene. These three impurities could be used as criteria for classification based on synthetic methods of methamphetamine.

KEYWORDS: *Methamphetamine, Emde method, Nagai method, Chloroephedrine*

Corresponding author: pinus@spo.go.kr