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Relationship between methamphetamine use history and segmental hair analysis findings of MA users

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ABSTRACT

The aim of this study was to investigate the relationship between methamphetamine (MA) use history and segmental hair analysis (1 and 3 cm sections) and whole hair analysis results in Korean MA users in rehabilitation programs. Hair samples were collected from 26 Korean MA users. Eleven of the 26 subjects used cannabis with MA and two used cocaine, opiates, and MDMA with MA. Self-reported single dose of MA from the 26 subjects ranged from 0.03 to 0.5 g/one time. Concentrations of MA and its metabolite amphetamine (AP) in hair were determined by gas chromatography mass spectrometry (GC/MS) after derivatization. The method used was well validated.

Qualitative analysis from all 1 cm sections (n = 154) revealed a good correlation between positive or negative results for MA in hair and self-reported MA use (69.48%, n = 107). In detail, MA results were positive in 66 hair specimens of MA users who reported administering MA, and MA results were negative in 41 hair specimens of MA users who denied MA administration in the corresponding month. Test results were false-negative in 10.39% (n = 16) of hair specimens and false-positive in 20.13% (n = 31) of hair specimens. In false positive cases, it is considered that after MA cessation it continued to be accumulated in hair still, while in false negative cases, self-reported histories showed a small amount of MA use or MA use 5–7 months previously.

In terms of quantitative analysis, the concentrations of MA in 1 and 3 cm long hair segments and in whole hair samples ranged from 1.03 to 184.98 (mean 22.01), 2.26 to 89.33 (mean 18.71), and 0.91 to 124.49 (mean 15.24) ng/mg, respectively. Ten subjects showed a good correlation between MA use and MA concentration in hair. Correlation coefficient (r) of 7 among 10 subjects ranged from 0.71 to 0.98 (mean 0.85). Four subjects showed a low correlation between MA use and MA concentration in hair. Correlation coefficient (r) of 4 subjects ranged from 0.36 to 0.55. Eleven subjects showed a poor correlation between MA use and MA concentration in hair. Correlation between MA use and MA concentration in hair of remaining one subject could not be determined or calculated.

In this study, the correlation between accurate MA use histories obtained by psychiatrists and well-trained counselors and MA concentrations in hair was shown. This report provides objective scientific findings that should considerably aid the interpretation of forensic results and of the results of trials related to MA use.

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1. Introduction

Researchers have conducted hair analysis and segmental hair analysis to detect drug use, and the statistical results of drug levels in hair have been used for interpretation in forensic investigations and trials. Studies on the hair analysis results of drug users in rehabilitation programs have been conducted in other countries [1–12], but no such study has been performed in Korea. In our previously published papers [13,14], we examined MA concentrations in the hair of American MA users in rehabilitation programs, and in suspected South Korean MA users.

The purpose of previous hair analyses conducted on MA-dependent individuals in USA was to investigate the correlation between drug histories and MA concentrations in hair and to determine whether such results can assist in the estimation of doses, frequencies, and patterns of drug use in the forensic field [13]. We conducted statistical research on whether MA levels in 1 cm long segments of hair samples from MA users were related to history of MA use, and a good correlation was found between MA concentrations in 1 and 4 cm long segmental hair and MA use history. However, it was difficult to apply these results to South Korean drug users because routes of administration and MA purity can differ.

Hair analysis on MA suspects in South Korea has been conducted to investigate whether the results of segmental hair analysis can be used to determine pattern of MA use [14]. However, accurate drug use history cannot be obtained from MA suspects, who generally tend to underreport MA use and they call themselves one time users or consumers of only small amounts (0.03 g per administration). MA suspects were classified as light, moderate, and heavy users. However, it was difficult to find correlation between MA use history as determined by police officers and MA concentration in hair [14].

In this study, we examined the relationship between MA concentrations in the hair of MA users in rehabilitation programs and their MA use history to overcome the limitations of our previous researches, and provide reliable information for forensic investigations and trials. To the best of our knowledge, this is first research to be conducted on hair segment analysis of Korean MA users in rehabilitation centers who offered self-report of MA use.

2. Methods

Hair collection procedures, hair analysis, and GC/MS conditions have been previously described [13–15].

2.1. Recruitment of subjects

We recruited volunteers who met the hair criteria and wanted to participate in the research. Volunteers who had at least 4 cm length of hair with at least 1 month of MA use history were enrolled for study subjects. The subjects provided signed consent after being provided with information on the purpose, schedule, risks, and benefits of the study. They completed a questionnaire and provided hair samples. The questionnaire inquired about their frequency and doses of MA use by day, week and month as accurate as possible. Hair were collected from the Korean Association Against Drug Abuse, a probation office, and psychiatry departments at hospitals. The Duksung Women's University Institutional Review Board approved this study.

2.2. Reagents and standards

AP sulfate, MA HCl, AP-d_5, and MA-d_5 were obtained from Cerilliant Corporation (Round Rock, TX, USA), trifluoroacetic anhydride from Sigma-Aldrich (St. Louis, MO, USA), and 2-fritted reservoir (3 ml) from Varian (Harbor City, CA, USA). Working solution of the internal standard AP-d_5 and MA-d_5 (1 μg/ml) and of standard solutions AP and MA (1 μg/ml) were diluted with methanol and stored in the dark at −20 °C. Acetone, methanol, and hydrochloric acid were of analytical grade.

2.3. Hair collection and hair analysis

Hair samples from the vertex region of the scalp were collected from 26 Korean users who wanted to rehabilitate and consult with doctors or counselors. MA users provided information on their drug use, such as MA dose per day, and month, as well as the frequency of MA use.

The detailed sample preparation method has been previously described [13–15]. The hair segments were prepared in two segmenting patterns that measured 1 cm and 3 cm each from hair roots in order to correspond to individual month and the recent three months. A total of 154 segments in 1 cm length, 24 segments in 3 cm length, and 25 whole hairs were analyzed for investigating the relationship between the MA use history and MA concentration in hair.

About 3 mg of hair (mean = 3.60) was weighed and washed two times with methanol and distilled water. After cutting the hair into small size less than 1 mm, the hair was incubated in the mixture of 1 ml methanol containing 10% hydrochloric acid and 50 μg of each internal standard, MA-d_5 and AP-d_5 for 18 h. The extracts were evaporated under the stream of nitrogen for 25 min. A total of 30 μl of ethyl acetate and 30 μl of TFAA were added and the eluent was kept in the oven for 30 min at 65 °C. After evaporating excess TFAA under nitrogen gas for 4 min, 50 μl of methanol was added for reconstitution.

2.4. GC/MS analysis

The procedure has previously been described [13–15]. GC/MS system was equipped with a Hewlett Packard 7683 series injector, HP 6890N series GC system (Wilmington, DE, USA), and HP5975 inert XL mass selective detector. The column was silica capillary column (HP-5MS, 30.0 m × 0.25 m, Agilent Technologies, Foster, CA, USA). A total of 1 μl of the extract was injected in the splitless mode and temperature of injector and transfer line were 250 and 280 °C. The initial oven temperature was 60 °C and held for 1 min, increased to 260 °C (20 °C/min), and held at 260 °C for 10 min. The analysis was performed in a mode of selective ion monitoring (SIM) and the ionization energy was set at 70 eV. The m/z of TFAA-derivatized MA, AP, MA-d_5 (internal standard), and AP-d_5 (internal standard) was as follows: MA, m/z 154, 118, 110, 91; AP, m/z 140, 118, 91; MA-d5, m/z 158, 122; AP-d5, m/z 144, 122 (the underlined ions were used for quantification). The cut-off value for the positive results of MA in hair defined as 0.5 ng/mg.

2.5. Method validation

Method validation was achieved by evaluating linearity, limit of detection (LOD), accuracy and precision in intra-assay and inter-assay, and percent recovery in extraction. Linearity was calculated from the calibration curve with six sets of calibrators at a range of 0.5–20 ng/mg by using 10 mg of blank hair. The samples with decreasing concentration of the compounds were used to estimate the LOD and LOQ, where the response of qualifying ions was equivalent to three and ten times the background noise, respectively. Blank hair samples spiked with 4, 8, 16 ng/mg of MA and AP were prepared to validate precision and accuracy in intra-assay and inter-assay, and the experiment was replicated for 6 consecutive days. Percentage recovery was determined by adding 25, 50, and 100 ng of standards to 10 mg of pulverized drug-free