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## Application of ion mobility spectrometry to the rapid screening of methamphetamine incorporated in hair

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### Abstract

Using ion mobility spectrometry (IMS), a simple, sensitive and rapid screening for methamphetamine (MA) incorporated in user's hair has been developed. To completely unbind MA from hair matrix and to achieve its effective vaporization for the IMS detection, the hair sample was digested in 5 M NaOH (methanol–water, 4:1, v/v) solution prior to IMS measurement. MA in hair was semi-quantitatively detected by monitoring the digested hair sample employing dibenzylamine (DBA) as internal standard. The minimum amount of hair sample required was 2 mg and its digested sample was ample for four IMS measurements. The detection limit of MA in hair was 0.5 ng mg<sup>-1</sup>. This proposed method was applicable to the semi-quantitative detection of MA in users' hair samples, and to the sectional analysis for MA in a limited amount of user's hair. The IMS results obtained were in good agreement with their GC–MS determination.

**Keywords:** Ion mobility spectrometry; Methamphetamine

### 1. Introduction

MA abuse accounts for well over 80% of drug-related offences in Japan [1], and is becoming a more serious problem in Asian and the Pacific countries including the United States. Therefore it is of great importance for drug enforcement to detect MA not only in urine or blood but also in hair. Hair analysis for detecting MA is usually conducted by GC–MS [2,3] after elaborate sample preparation. So far there have been no convenient, reliable and rapid screening methods for MA in hair, and the GC–MS procedure includes time-consuming pretreatment and requires skilled handling. Therefore, the development

of a simple, reliable and sensitive screening for MA incorporated in hair is of great importance not only for forensic toxicologists but also for drug enforcement officers.

In drug enforcement departments, on the other hand, ion mobility spectrometry (IMS) has been becoming an indispensable tool for detecting trace amounts of drugs in mail, imported articles, suspects' clothing, luggage and in their nail dirt and so on [4–8]. IMS is a mass-selective technique in which the analytes are vaporized, ionized, and with given initial velocity are moved along a drift region towards a collector electrode. The drift time needed to reach the collector electrode is proportional to the mass of the ions [9,10]. The primary advantages of IMS are the very short analysis time (around 5 s),

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detection limits in the ng to pg range, and the various options for sample introduction including applying a solution on the filter and trapping particulate matter on the filter [11].

Previously the authors analyzed various drug users' hair samples directly by IMS without any sample pretreatment. Those experiments presented sufficient sensitivity for detection in externally contaminated hair, usually found on drug dealers, but poor results were obtained for drugs incorporated in users' hair [12]. De Zeeuw et al. also directly monitored cocaine users' hair by IMS. Their results did not reflect the cocaine concentration probably due to the insufficient desorption of cocaine from hair matrix with direct IMS measurement [13]. In this paper, the authors describe a simple and rapid screening by IMS for MA incorporated in user's hair after digesting the hair with an alkali solution in order to sufficiently unbind MA from hair matrix and to achieve its effective vaporization.

## 2. Principles

A schematic diagram of the IONSCAN used for IMS measurement is shown in Fig. 1. The sample, collected on a filter, is thermally vaporized by the desorber and carried through the heated transfer line

to the ionization/reaction chamber by a stream of dried and purified air. Ionization is initiated by high energy electrons emitted from a  $^{63}\text{Ni}$  beta-ray source. Every 20 ms, positive or negative ions are gated with a pulse width of 0.02 ms into the heated drift region. Under the influence of an electric field and against a counterflow of ambient air drift gas, the ions move to the collector electrode. In the positive mode used for drug detection, the drift gas contains trace amounts of nicotinamide (NA), acting as both calibrant and reactant. The drift time ( $t_d$ ) required to reach the electrode is inversely proportional to the characteristic reduced ion mobility ( $K_0$ ) of the ion and is the means by which the ions are identified. Drift gas and NA are constantly flowing in and are being ionized in the ionization/reaction chamber when the instrument is in the ready mode waiting for a sample to be introduced. The dominant ion in the ready mode is  $(\text{NA})\text{H}^+$ . In the analyze mode, when a sample is introduced, the dominant sample ionization process is proton transfer from protonated NA to the sample molecule M:  $(\text{NA})\text{H}^+ + \text{M} \rightarrow \text{NA} + \text{MH}^+$ . This reaction only proceeds if the proton affinity of M is greater than the one of NA. This is the case for most drug molecules which are therefore detectable as  $\text{MH}^+$  under IMS conditions.

Any potential interferences with proton affinities less than that of NA are suppressed.

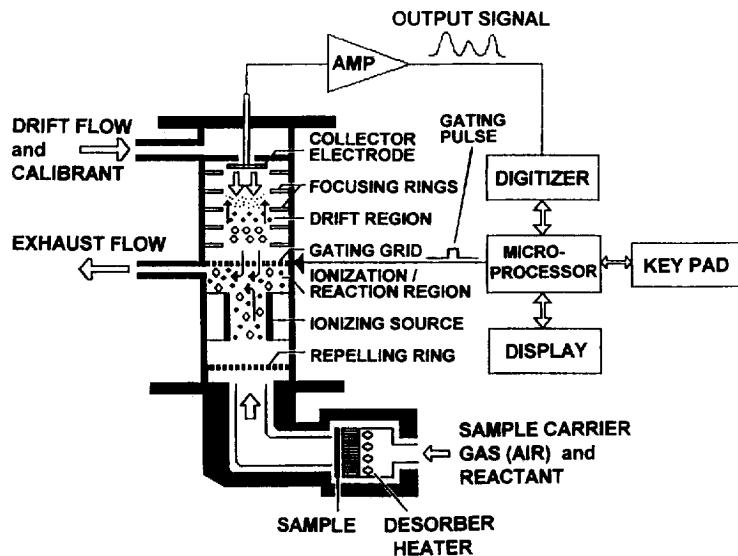


Fig. 1. Schematic diagram of the IONSCAN.

Each ion has a characteristic mobility  $K$ , which is dependent on the temperature  $T$  (K) and pressure  $P$  (Torr). To compare results obtained under different conditions, the reduced mobility  $K_0$  is defined as  $K_0 = 273 \cdot K \cdot T^{-1} \cdot P^{-1} \cdot 760$ , and in which  $K$  is the observed mobility, defined as  $K = d \cdot t^{-1} \cdot E^{-1}$ , where  $d$  is the drift region in cm,  $t$  is the  $t_d$  in s and  $E$  the applied field in  $V \text{ cm}^{-1}$ . As a result,  $K_0$  is given in  $\text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ . The calibrant is used to calculate  $K_0$  for a certain ion from the observed  $t_d$  values using the equation:  $K_{0,\text{ion}} = t_{d,\text{calibrant}} \cdot K_{0,\text{calibrant}} / t_{d,\text{ion}}$ . Although  $K_0$  is not solely dependent on ion mass, but also on ion geometry and collisional and ionic interactions, a reasonably straight line is observed by plotting  $t_d$ , or the inverse of  $K_0$ , against the mass of ions whose structure is known [11,13].

### 3. Experimental

#### 3.1. Specimens

Human scalp hair specimens A,B,C and D were obtained from volunteers who admitted being MA users. Human hair specimen E was sampled according to the standard procedure [14] from the scalp of the 25-year-old woman who admitted using MA for about 2 years or longer. Her hair was about 25 cm long.

#### 3.2. Chemicals

Amphetamine (AM) sulfate, hydrochlorides of *d*-MA, *d*-ephedrine, *d*-pseudoephedrine, phentermine, 3,4-methylenedioxymphetamine (MDA) and 3,4-methylenedioxymethamphetamine (MDMA) were obtained from Sigma (St. Louis, MO, USA). *d*-Methylephedrine hydrochloride was obtained from Kansai Yakuhin (Osaka, Japan). Chlorophentermine, phenfluramine and ethylamphetamine were extracted from commercially available medicines Teramine, Darby (Rockville Center, NY, USA), Ponderal, Sezviver (Meyrin, Switzerland) and Apetinil, Drosapharm (Basel, Switzerland), respectively. Hemisulfate of mescaline was provided by Japanese Ministry of Health and Welfare. Dimethylamphetamine hydrochloride was synthesized from methylephedrine according to the method of Nagai [15]. Hydrochlorides

of chloroephedrine and chloromethylephedrine were synthesized from ephedrine and methylephedrine respectively, according to the method of Emde [16]. DBA, ( $\pm$ )- $\alpha$ -methylbenzylamine, and hydrochlorides of 2-phenylethylamine and norephedrine were obtained from Aldrich (Milwaukee, WI, USA). The other amine compounds were obtained from Merck (Darmstadt, Germany). All organic solvents and inorganic reagents are of analytical-reagent grade, and all of them were obtained from Merck. Deionized, distilled water was used throughout.

#### 3.3. Instruments

##### 3.3.1. IMS

An IONSCAN Model 250 ion mobility spectrometer (Barringer, Rexdale, Canada) was used in the positive ion mode. Teflon filters with a thickness of 0.25 mm were used as filter substances. The operation conditions are listed in Table 1.

##### 3.3.2. GC-MS

GC-MS analysis was performed on a Hewlett-Packard (HP) 5890 Series II gas chromatograph equipped with a quadrupole mass spectrometer model HP 5972. Confirmation and quantitative analyses were conducted in the scan mode and in the selected ion monitoring (SIM) mode, respectively with electron impact ionization (70 eV). A 30 m  $\times$  0.25 mm I.D. Optima 5 cross-linked fused-silica capillary column (5% phenyl, 95% methyl silicone) with a 0.25  $\mu\text{m}$  film thickness (Macherey-Nagel, Düren, Germany) was used with helium as the carrier gas at a septum purge flow-rate of 1.0  $\text{ml min}^{-1}$  and nitrogen as the make-up gas at a

Table 1  
Operation conditions for the IONSCAN

Electric field	210 $\text{V cm}^{-1}$
Desorber temperature	180°C
Inlet temperature	290°C
Drift tube temperature	235°C
Width of the spectrum	20 ms
Number of scan	20
Drift gas flow	300 $\text{ml min}^{-1}$
Sample gas flow	200 $\text{ml min}^{-1}$
Drift gas	Dried and purified air
Carrier gas	Dried and purified air
Calibrant ion	Nicotinamide

flow-rate of  $4.0 \text{ ml min}^{-1}$ . A Model 7673 HP automatic sampler was used for injection in the splitless mode. The operating conditions for the analyses were: injection port temperature,  $200^\circ\text{C}$ ; initial oven temperature,  $70^\circ\text{C}$  for 2 min; programming:  $15^\circ\text{C min}^{-1}$  to final temperature of  $220^\circ\text{C}$ ; hold for 3 min.

### 3.4. Procedure

#### 3.4.1. Pretreatment of specimens

Each specimen was cut into about 5 mm lengths and placed in a test tube. Every specimen was washed with 0.1% SDS (sodium dodecyl sulfate) under ultrasonication for 1 min. The specimen was then washed four times with distilled water ( $4 \times 1 \text{ ml}$ ) and methanol ( $4 \times 1 \text{ ml}$ ) in turn by applying ultrasonication for 1 min each. The first half of the water and methanol washings were combined and stored. The second half of the washings was also stored. These washings were subjected to both IMS and GC-MS evaluation.

After complete drying, each specimen was sandwiched in a piece of filter paper and cut into 2–3 mm pieces. These were then mixed thoroughly, and precisely weighed portions were subjected to further pretreatment for both IMS and GC-MS analyses.

#### 3.4.2. Sample preparation for IMS measurement

To each of the hair samples (2.0 mg) placed in a 5-ml stoppered glass test tube was added 200  $\mu\text{l}$  of 5  $M$  NaOH (methanol–water, 4:1, v/v) solution and 20  $\mu\text{l}$  of 1 ng/ $\mu\text{l}$  methanolic DBA as internal standard (I.S.). Ultrasonication at  $45^\circ\text{C}$  for 20 min resulted in the complete digestion of the sample.

To be used for the optimization of IMS conditions, a digested standard 10 ng MA  $\text{mg}^{-1}$  hair sample was prepared as follows: to 10 mg of control human scalp hair placed in a 5-ml stoppered glass test tube was successively added 10 ng/ $\mu\text{l}$  MA methanolic solution (10  $\mu\text{l}$ ), 1.0  $\mu\text{l}$  of 5  $M$  NaOH (methanol–water, 4:1, v/v) solution and 1 ng/ $\mu\text{l}$  methanolic DBA solution (100  $\mu\text{l}$ ). Ultrasonication at  $45^\circ\text{C}$  for 20 min resulted in a homogenous sample solution. Known concentrations (blank and 0.2–120 ng MA  $\text{mg}^{-1}$  hair) of digested hair samples were also prepared by the same process for constructing the calibration curve and determining the detection limit. The control hair used was obtained from a volunteer who

had never come in contact with MA, and was washed beforehand in the same way as were MA users' hair specimens.

#### 3.4.3. IMS measurement

A digested hair sample solution (50  $\mu\text{l}$ ) was applied to a Teflon membrane filter in a plane circle shape with a diameter of about 15 mm. The applied sample was immediately dried for 60 s under a gentle stream of warm air from a hair drier at about  $50^\circ\text{C}$  at a working distance of 20 cm. After drying, the sample was immediately measured by IMS.

#### 3.4.4. Confirmation and quantification by GC-MS

Each of precisely weighed hair samples (about 8 mg), placed in a glass test tube was extracted with 2 ml of methanol–5  $M$  HCl (20:1, v/v) solution [2,3] for 16.5 h at room temperature applying ultrasonication (only for the first 1 h and the last 30 min of the extraction). The mixture was centrifuged and the supernatant was passed through a disposable Pasteur glass pipette sealed with a piece of cotton in the tip. For maximum recovery, the test tube was washed again with the same solvent (1 ml), and this washing was also treated in the same way with the same pipette, and was then added to the filtrate. The filtrate was evaporated to dryness at  $60^\circ\text{C}$ . Ethyl acetate (100  $\mu\text{l}$ ) and trifluoroacetic anhydride (100  $\mu\text{l}$ ) were added to the residue and it was derivatized for 30 min at  $55^\circ\text{C}$ . After evaporation of excessive reagent and solvent, the residue was reconstituted in 50  $\mu\text{l}$  of ethyl acetate containing 1  $\mu\text{g ml}^{-1}$  of diphenylmetane (I.S.). A 1- $\mu\text{l}$  volume of this solution was injected into the GC-MS operated in the SIM mode. The calibration curves for MA and AM were drawn by measuring the peak areas of  $m/z$  154 for MA,  $m/z$  140 for AM and  $m/z$  168 for diphenylmetane. After concentration to about 20–30  $\mu\text{l}$ , a 2- $\mu\text{l}$  volume of the solution was then analyzed in the scan mode for the confirmation of MA and AM.

## 4. Results and discussion

### 4.1. Optimization of IMS

#### 4.1.1. Selection of internal standard

Without employing any I.S., the repeatability of MA peak intensity was only moderate although the

IMS measurements were done under optimized conditions [12]. This was probably due to loss of MA, which is highly volatile in a strongly basic solution [17,18], during the drying on the filter. It was also found that sample contamination with trace amount of single textile or paper fibers originating from ambient air may cause loss in peak intensity. In addition, other drugs which may be present in the hair sample compete with MA in the ionization process, causing loss in MA peak intensity. These facts led us to employ a proper I.S., though this is not generally done in IMS. The I.S. should have similar proton affinity and volatility under basic conditions, and its  $K_0$  value should be similar to that of MA. Furthermore, it should not be an endogenous hair component or an ingredient of hair treatment agents. The I.S. should also be clearly distinguished from any other major drugs. Twenty-six amines were tested [19], among which DBA was found to be the substance of choice, since it fulfilled the necessary conditions of an I.S. mentioned above, even though DBA may not be the best choice for all other amphetamines. The growing curves of DBA and MA are shown in Fig. 2. Optimum response for MA and DBA was received by averaging their peak intensities in the window 7–12 (corresponding to measurement time from 2.3–4.6 s). The plasmagrams of the digested standard 10 ng MA  $\text{mg}^{-1}$  hair sample and a digested control hair (without I.S.) are presented in Fig. 3a and b, respectively.

#### 4.1.2. Desorber temperature

Fig. 4 illustrates the peak intensity of MA at various desorber temperatures. The best results were obtained at 180°C. A desorber temperature higher than 220°C caused baseline drift and some interfering peaks with MA and DBA originated probably from burning of hair constituents. No interfering peaks were observed at 200°C or below. All of the other IMS conditions were set as Table 1 according to our previous basic experiments [12].

#### 4.1.3. Drying time of the digested sample on the filter

Fig. 5 illustrates the peak intensities of MA and DBA, together with the peak ratio MA/DBA for various drying periods (0–180 s at 50°C) on the filter. A drying time up to 20 s caused poor responses for MA and DBA probably due to the

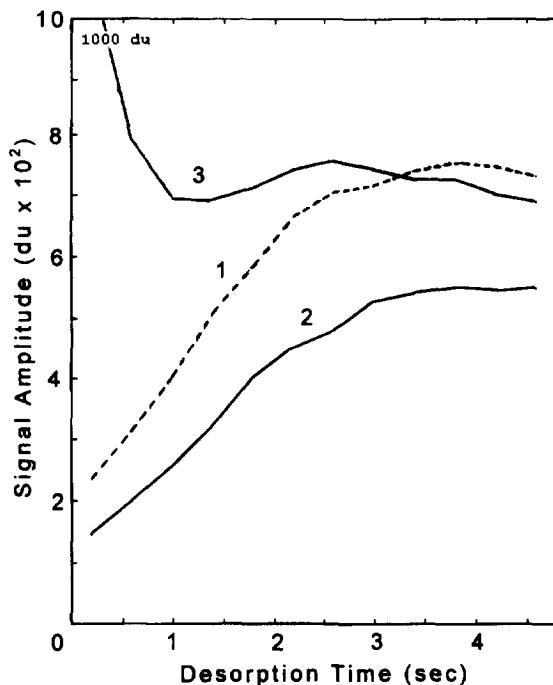


Fig. 2. Growing curves of methamphetamine (MA) and di-benzylamine (DBA). Curves: 1=MA; 2=DBA; 3=NA (calibrant/reactant). A 50- $\mu\text{l}$  volume of the digested standard 10 ng MA  $\text{mg}^{-1}$  hair sample was measured by the IONSCAN on the operation conditions listed in Table 2.

consumption of the heat for the evaporation of the solvent. A remarkable decrease of peak intensities was also observed for drying times longer than 90 s, though the peak intensity ratio MA/DBA was nearly constant over the range of 40 to 120 s. A drying time of 60 s was found to be best for the conditions already mentioned in the Section 3.4.3.

#### 4.1.4. Selection of filter

The glass-fiber filter (thickness of 0.13 mm) was the best for the direct measurement of MA in hair [12], but this filter was found to be ineffective for digested hair samples. Not only did the filter not show a clear MA peak even with the digested standard 10 ng MA  $\text{mg}^{-1}$  hair sample, but also the filter caused serious baseline drift and interfering peaks throughout the  $t_d$  range of the plasmagram. Teflon filter produced clear peaks of MA and DBA, probably due to good heat conduction.

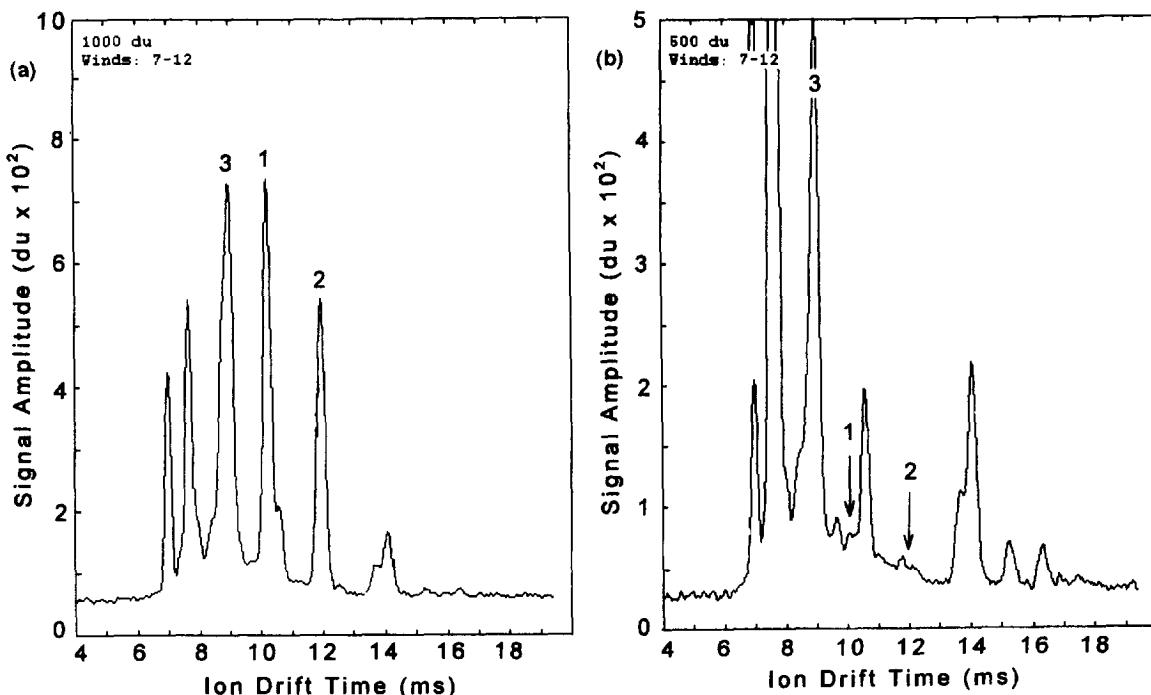


Fig. 3. Plasmagrams of the digested standard  $10 \text{ ng MA mg}^{-1}$  hair sample (a) and a digested control hair sample (b). Peaks: 1=MA; 2=DBA (I.S.); 3=NA (calibrant/reactant). A  $50\text{-}\mu\text{l}$  volume of the digested standard  $10 \text{ ng MA mg}^{-1}$  hair sample or a digested control hair (without I.S.) was measured by the IONSCAN on the operation conditions listed in Table 2. The plasmagrams were received by averaging the peak intensities in the window 7–12.

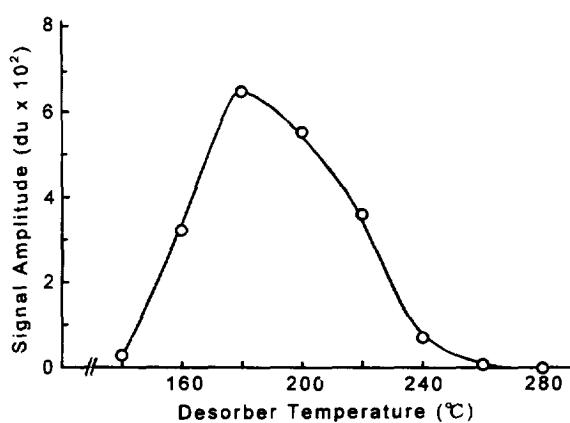


Fig. 4. Peak intensity of methamphetamine (MA) at various desorber temperatures. A  $50\text{-}\mu\text{l}$  volume of the digested standard  $10 \text{ ng MA mg}^{-1}$  hair sample was measured by the IONSCAN at various desorber temperatures ( $140\text{--}280^\circ\text{C}$ ). The amplitude of the peak was received by averaging the peak intensities in the window 7–12.

#### 4.1.5. Reliability of MA detection

The authors measured  $t_d$  of MA and 20 of its analogues. The analogues tested are listed in Table 2 together with their  $t_d$  and  $K_0$  values calculated from  $t_d$ . Most adjacent peaks to the MA peak are the ones of AM and norephedrine. Although these three peaks are not clearly separated, the reproducibilities of their  $t_d$  values observed were within  $\pm 12 \mu\text{s}$ . The IONSCAN clearly discriminates MA from these two analogues by setting the detection windows at  $20 \mu\text{s}$ .

The minimum allowable amplitude value of the threshold for the detection of MA by IONSCAN is 2 du (data unit). The authors set the threshold at 2 du, and measured a total of 50 control hair samples according to the procedure described in Section 3.4.2. No positive result for MA was obtained with a threshold value set at 2 du. Although some control hair samples showed a very small peak near the  $t_d$  of MA as shown in Fig. 3b, the IMS data processor

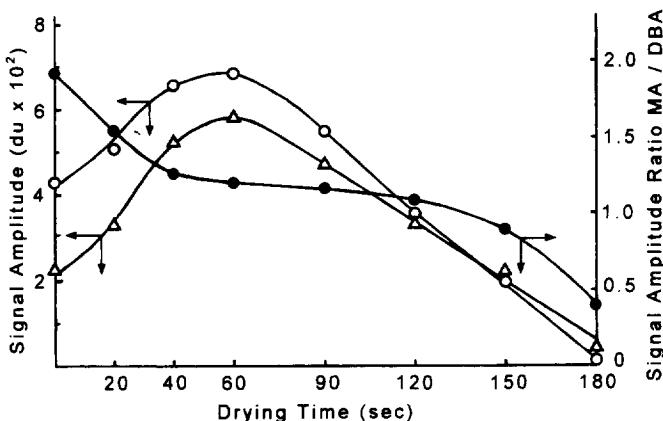


Fig. 5. Peak intensities of methamphetamine (MA) and dibenzylamine (DBA) together with their peak ratios MA/DBA after various drying periods. ○=MA; ●=DBA (I.S.); △=Ratio MA/DBA. A 50- $\mu$ l volume of the digested standard 10 ng MA mg<sup>-1</sup> hair sample was measured by the IONSCAN immediately after various drying period (0–180 s) at 50°C on the filter. The amplitude of the peak was received by averaging the peak intensities in the window 7–12.

could discriminate this false peak from the real peak by the differences in half-wave width and  $t_d$ .

Although MA-negative results were obtained for all of the MA analogues tested and also for a total of 50 of control hair samples, it should be noted that this IMS screening method for MA will not hold for identification in every unknown hair sample. There may be some substances other than MA analogues that may appear in hair with  $t_d$  values close to MA peak. Therefore, every MA-positive result by IMS should be confirmed by a reliable analysis such as GC-MS. There is however, a certain advantage in using IMS technology in which no cross-reactivity with another MA analogue is observed, such as immunoassay-based methods.

#### 4.1.6. Calibration curve and reproducibility

The calibration curve for MA in hair was obtained by IMS assay of digested control hair samples spiked with known amounts of MA (0, 0.5, 1, 2, 5, 10, 20, 40, 70, 100, 120 ng mg<sup>-1</sup> hair) and of I.S. (10 ng mg<sup>-1</sup> hair). The calibration curve was constructed by plotting of the peak-height ratios MA/I.S. against the concentrations of MA. It was linear over the concentration range of 0.5–120 MA mg<sup>-1</sup> hair ( $y = -11.02 + 8.99x$ ,  $r^2 = 0.998$ ), though the IMS calibration curve for MA without employing I.S. was a quadratic curve [12]. Generally, the intensity of IMS

response is only linear in a small range when compared to the sample concentration [11]. Furthermore, in case of some IMS-sensitive substances like MA and DBA present together in a sample, these substances compete for the reactant NA. This leads to poor reproducibility of peak intensity on IMS measurement. The R.S.D.s of peak intensity of MA at the concentration of 1.0 ng mg<sup>-1</sup> and 20 ng mg<sup>-1</sup> were 23.8% and 17.9%, respectively ( $n=5$ ). These are in accordance with R.S.D. values for other compounds already published [11]. In contrast, our method employing the I.S. resulted in much better reproducibility. The R.S.D. values of MA/DBA ratio were 5.2% and 3.6% respectively at the MA concentrations of 1.0 ng mg<sup>-1</sup> and 20 ng mg<sup>-1</sup>. The detection limit of MA in hair was determined by using a series of known concentrations (0.2, 0.3, 0.5, 0.7, 1.0 ng MA mg<sup>-1</sup> hair) of digested hair samples. The detection limit was 0.5 ng mg<sup>-1</sup> hair, which was determined by the indication of 'fail' (i.e. a positive judgment) in the IMS result table. It should be noted that the detection limit can be affected by coexisting drugs or substances which may compete with MA and DBA in the ionization process. Although the authors have not experienced a noticeable decrease in sensitivity caused by coexisting substances, attention should be paid to the peak intensities of DBA (I.S.) as well as that of MA on every measurement.

Table 2

Drift times ( $t_d$ ) and  $K_0$  values of methamphetamine analogues measured by the IONSCAN

Substance	$t_d^a$ (ms)	$K_0^b$ ( $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ )
N-Methylbenzylamine	9.370 $\pm$ 0.005	1.781
3-Phenyl-1-propylamine	9.903 $\pm$ 0.006	1.685
Amphetamine	9.959 $\pm$ 0.005	1.676
Methamphetamine	10.187 $\pm$ 0.006	1.640
Norephedrine	10.318 $\pm$ 0.006	1.617
Phentermine	10.400 $\pm$ 0.007	1.601
$\alpha$ -Methylbenzylamine	10.457 $\pm$ 0.007	1.592
2-Phenylethylamine	10.480 $\pm$ 0.008	1.592
Dimethylamphetamine	10.488 $\pm$ 0.007	1.591
Ethylamphetamine	10.523 $\pm$ 0.008	1.586
Ephedrine	10.625 $\pm$ 0.007	1.572
Pseudoephedrine	10.710 $\pm$ 0.007	1.558
Methylephedrine	10.851 $\pm$ 0.007	1.538
Methoxyphenamine	10.921 $\pm$ 0.008	1.528
MDA	11.237 $\pm$ 0.007	1.487
Chlorophentermine	11.385 $\pm$ 0.008	1.466
MDMA	11.527 $\pm$ 0.008	1.449
Chloroephedrine	11.649 $\pm$ 0.007	1.433
Chloromethylephedrine	12.085 $\pm$ 0.008	1.381
Phenfluramine	12.437 $\pm$ 0.008	1.342
Mescaline	12.569 $\pm$ 0.007	1.328
Nicotinamide (calibrant)	8.939 $\pm$ 0.005	1.867
Dibenzylamine (I.S.)	11.865 $\pm$ 0.007	1.407

<sup>a</sup> Mean $\pm$ S.D. ( $n=6$ ). Drift time was measured by repeated (six) analyses of  $5.0 \mu\text{g ml}^{-1}$  ethanol solution of the substance;  $10 \mu\text{l}$  of the solution was applied on the Teflon filter and measured as described in Section 3.

<sup>b</sup> Reduced mobility. Mean ( $n=6$ ) of  $K_0$  values which were automatically calculated from the drift time observed and indicated by the IONSCAN.

#### 4.1.7. Comparison of IMS and GC-MS results of MA users' hair specimens

The thoroughly washed MA users' hair specimens A, B, C and D were submitted to both IMS and GC-MS quantification. These results are listed in Table 3. A typical plasmagram of the specimen A is shown in Fig. 6. In every specimen, MA concentrations determined by IMS and GC-MS showed good agreement.

Unfortunately, the IMS measurement could not clearly separate the peak of AM from MA, because the concentration of AM in hair is only around 10% of MA in average [2,3,20] and the difference of their  $t_d$  values is only 228  $\mu\text{s}$ . The width of both MA and AM detection windows was set at 20  $\mu\text{s}$  in com-

Table 3

Comparison of IMS and GC-MS quantification of methamphetamine (MA) and amphetamine (AM) in the users' hair samples

Sample	IMS <sup>a</sup> ( $\text{ng mg}^{-1}$ hair)		GC-MS <sup>b</sup> ( $\text{ng mg}^{-1}$ hair)	
	MA	AM	MA	AM
A	26.3	Nm <sup>c</sup>	27.4	2.2
B	8.3	Nm	7.1	0.7
C	2.8	ND <sup>d</sup>	2.1	ND
D	16.3	Nm	17.7	2.0

<sup>a</sup> Mean;  $n=4$ .

<sup>b</sup> Mean;  $n=3$ .

<sup>c</sup> Not measurable. Quantification of AM could not be performed, though the peak of AM was observed as a shoulder peak, and the positive indication of Amphetamine ALARM! was displayed in the results table of the IONSCAN.

<sup>d</sup> Not detected.

parison with the difference of their  $t_d$  values. Although AM was barely detectable as a shoulder peak on the specimens A, B and D, the quantification of AM could not be performed by this IMS screening method. It was thought that the detection of MA

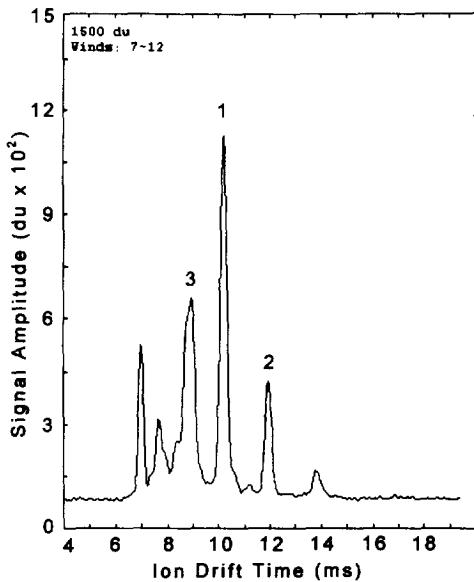


Fig. 6. Plasmagram of hair sample A. Peaks: 1=MA; 2=DBA (I.S.); 3=NA (calibrant/reactant). A 50- $\mu\text{l}$  portion of the digested sample was measured by the IONSCAN. The plasmagram was received by averaging the peak intensities in the window 7–12.

alone is sufficient for the screening of MA in users' hair, because the detection of AM is very easy when AM is the major drug compound in the hair sample.

#### 4.1.8. Application to the sectional analysis of MA user's hair

In the same manner, a sectional hair analysis for MA was conducted for a limited amount of hair using the hair specimen E. Although IMS measurement was possible for every 20-mm long section, every two consecutive sections had to be bundled for the GC-MS quantification due to lack of sample. Both IMS and GC-MS measurements were done not only for every hair specimen itself, but also for the alkali extracts of both the first and second halves of the combined hair washings. The results obtained are summarized in Table 4.

On all of the sections, good agreements were ensured between IMS and GC-MS results on both washed hair samples and their washings. It is demonstrated that this proposed IMS procedure is applicable to the comprehensive evaluation on hair analysis for MA, which includes quantitative detection of MA

in both decontaminated hair sample and in the washings.

#### 5. Conclusion

The most reliable and popular analytical method for MA in hair is GC-MS, however, it requires labor intensive and time-consuming pretreatment. For that reason, a simple, sensitive and rapid screening for MA in hair by IMS has been developed. MA in users' hair was semi-quantitatively detected by monitoring the digested hair sample employing DBA as I.S. Although the authors have not experienced any serious interference by coexisting substances in semi-quantitative MA detection, this IMS screening method will not hold for identification in any unknown hair samples. Therefore, confirmation and quantitative analysis by GC-MS should be performed for every IMS-positive sample.

IMS was found to have potential for detecting trace levels of various amphetamine-type drugs in a small amount of hair specimen. The authors have also successfully applied this IMS procedure to the

Table 4  
Sectional evaluation of methamphetamine (MA) in the user's hair comparison of IMS and GC-MS quantification

Section <sup>a</sup>	Washed hair		The first half of the washings		The second half of the washings		
	MA (ng mg <sup>-1</sup> hair)		IMS <sup>b</sup>	GC-MS <sup>c</sup>	MA (ng from mg hair)	IMS <sup>b</sup>	GC-MS <sup>c</sup>
	IMS <sup>b</sup>	GC-MS <sup>c</sup>					
1	32.5		1.0		ND <sup>d</sup>		
2	27.3	28.5	0.5	0.6	ND	0.2	
3	19.8		0.8		ND		
4	17.3	20.8	ND	0.8	ND	ND	
5	7.5		ND		ND		
6	5.5	7.4	ND	0.3	ND	0.1	
7	1.0		ND		ND		
8	0.5	0.8	ND	ND	ND	ND	
9	0.8		ND		ND		
10	ND	0.7	ND	ND	ND	ND	
11	0.8		ND		ND		
12	0.5	0.8	ND	0.4	ND	ND	

<sup>a</sup> 20-mm long sections from the scalp.

<sup>b</sup> Mean, n=4.

<sup>c</sup> GC-MS was performed for each of the bundled 2 consecutive sections; mean, n=3.

<sup>d</sup> Not detected.

detection of MDMA and MDEA (3,4-methyl-enedioxyethylamphetamine) in human hair [21].

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