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Determination of methamphetamine in human hair by ultra high performance liquid chromatography/tandem mass spectrometry

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Overview

A method for quantitative analysis of methamphetamine in human hair by liquid chromatography mass spectrometry was developed. The lower limit of quantitation was 0.1 ng/mL and results of other parameters for method validation were good.

Introduction

Methamphetamine is a neurotoxin and potent psychostimulant of the phenethylamine and amphetamine classes that is used to treat attention deficit hyperactivity disorder and obesity. Long-term use of methamphetamine can reduce dopamine activity in the brain. And substituted amphetamines abuse, such as methamphetamine, may cause lots of social problem. In public security system, blood and urine sample were collected usually to detect the narcotics level of the abuser. However, it is difficult to sample from the uncoordinated abuser. Hair sample can be used to detect the level of the methamphetamine as well as to conjecture abuse period.

Methods

Sample Preparation

(1) Collect hair sample with length of 2 cm from the root and cut into small pieces with length of not more than 1 mm. Accurately weigh 100 mg of the small pieces.
(2) Add 1 ml of 12 mol/L HCl, allowing hydrolysis in 100 °C water bath for more than 1 h, cooling.
(3) Adjust pH to 9.0 with $NaHCO_3$.
(4) Extract with 1 mL of ethyl acetate.
(5) Centrifuge to obtain supernate, then blow-dry with N_2 at normal temperature.
(6) Dissolve residue with 1 mL of mobile phase, filter with 0.22 μm microfiltration membrane.

LC-MS/MS Analysis

The analysis was performed on a Shimadzu Nexera UHPLC instrument (Kyoto, Japan) equipped with LC-30AD pumps, CTO-30A column oven, DGU-30A_5 on-line degasser, and SIL-30AC autosampler.
The separation was carried out on Shim-pack ODS II (2.0 mmi.d. x 75 mmL.) with the column temperature at 40 °C. A triple quadruple mass spectrometer (Shimadzu LCMS-8040, Kyoto, Japan) was connected to the UHPLC instrument via an ESI interface.

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Analytical conditions

HPLC (Nexera UHPLC system)	
Column	: Shim-pack ODS II (2.0 mmi.d. x 75 mmL., 2.2 µm)
Mobile Phase A	: water with 0.1% formic acid
Mobile Phase B	: acetonitrile
Flow Rate	: 0.4 mL/min
Column Tem.	: 40 °C
Injection Volume	: 5 µL
MS (LCMS-8040 triple quadrupole mass spectrometer)	
Ionization	: ESI
Polarity	: Positive
Interface Voltage	: +4.5 kV
Nebulizing Gas Flow	: 3.0 L/min
Drying Gas Flow	: 15.0 L/min
Heat Block Temperature	: 450 °C
DL Temperature	: 250 °C
Dwell Time	: 30 ms
Pause Time	: 3 ms
Mode	: MRM

Table 1 MRM parameters

Compound	Precursor *m/z*	Product *m/z*	Q1 Pre Bias (V)	CE (V)	Q3 Pre Bias (V)
Methamphetamine	150.10	91.05*	-16	-18	-16
		119.20	-16	-14	-20

*Transition for quantitation

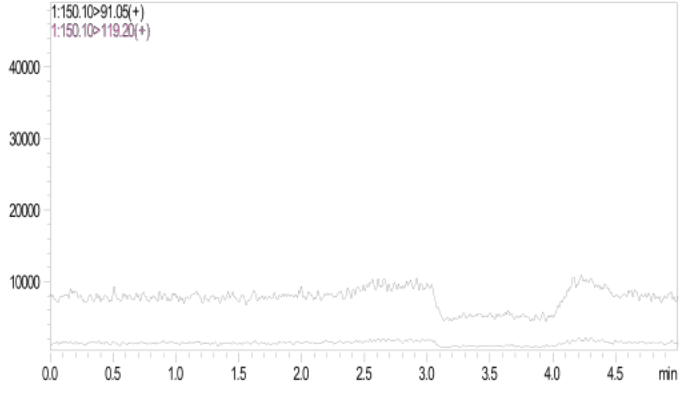

Figure 1 Representative MRM chromatograms of blank human hair

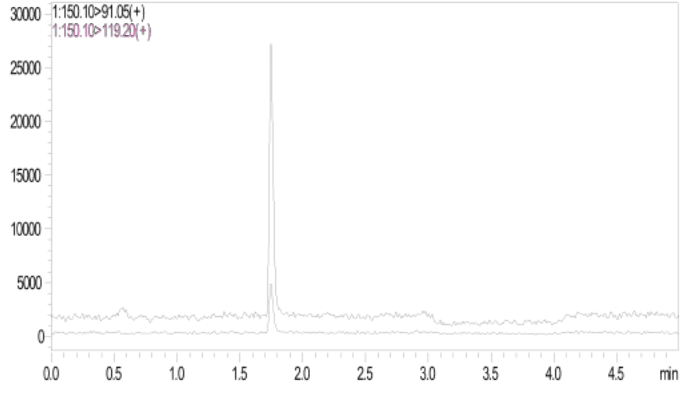

Figure 2 Representative MRM chromatograms of human hair (methamphetamine: 1 ng/mL)

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Results

Human hair samples containing methamphetamine ranging from 0.1 to 500 ng/mL were prepared and analyzed by LC-MS/MS. MRM chromatograms of methamphetamine (1 ng/mL) is presented in Fig. 1 (blank) and Fig. 2 (spiked). The linear regression for methamphetamine was found to be >0.9999. The calibration curve with human hair as the matrix were shown in Fig. 3. Excellent precision of the retention time and peak area of 1 ng/mL methamphetamine sample were maintained. Recovery of the method was 53.7% at concentration 10 ng/mL. Two abuser hair samples were detected by this UHPLC-MS/MS method. The hair methamphetamine concentration of abuser A and abuser B were 5.17 ng/mg and 18.46 ng/mg respectively. The public security bureau confirmed that the abuse period of abuser A was earlier than B. MRM-triggered product ion scan show that the main peak of the abuser samples analysis was methamphetamine. The false positive result was excluded.

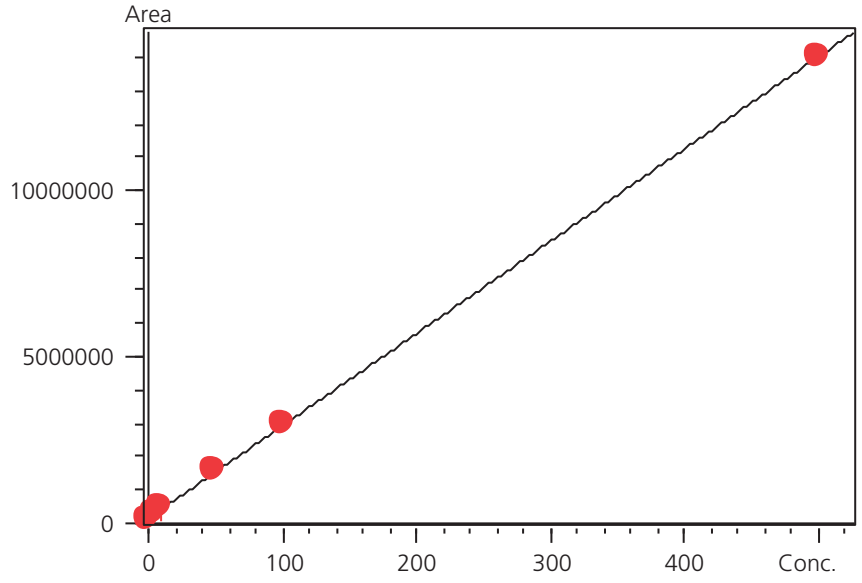

Figure 3 Calibration curve of methamphetamine in human plasma

Table 2 Calibration curve parameters

Compound	Calibration Curve	Linear Range (ng/mL)	r
Methamphetamine	$Y = (2.77\times10^{4})X + (1.53\times10^{5})$	0.1~500	0.9999

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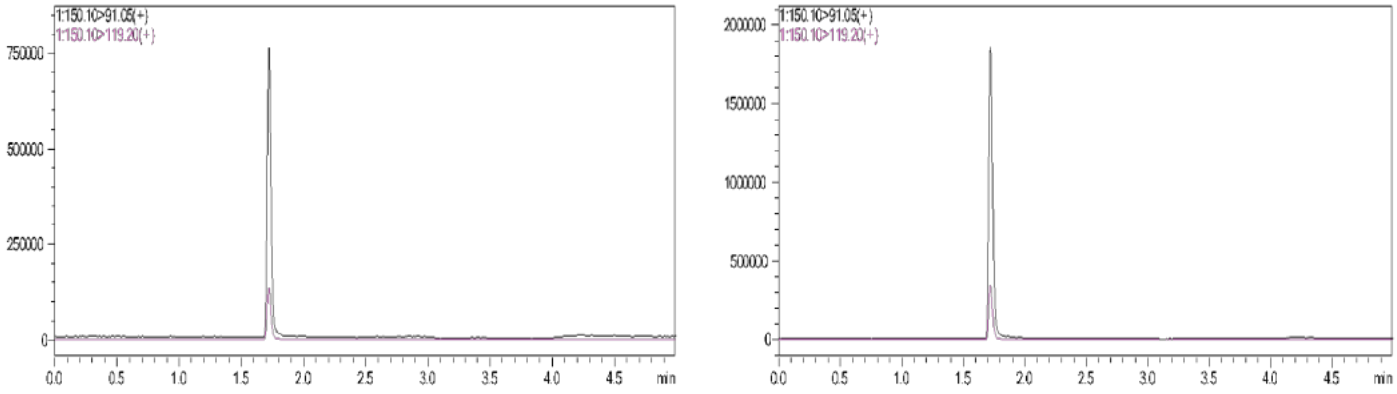

Figure 4 MRM chromatograms of two abusers hair samples

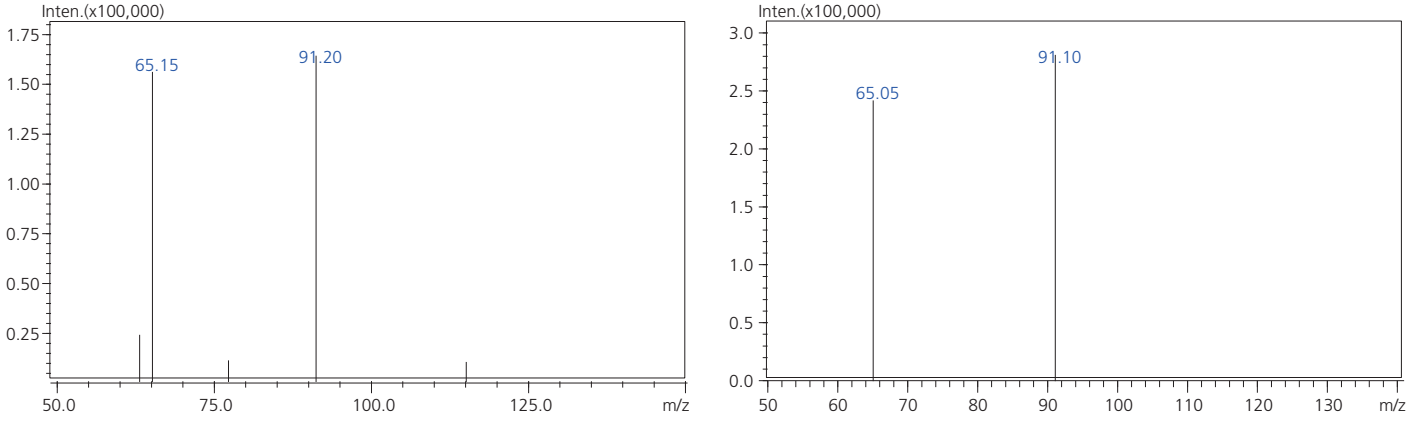

Figure 5 Representative product ion scan mass spectrum of abuser (left) and standard substance (right)

Table 3 Quantitative analysis result of abusers

Abuser	Date of last abuse	Position of test	Concentration (ng/mg)
A	1st May	Root	5.17
B	11th May	Root	18.46

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Conclusions

Results of parameters for method validation such as dynamic range, linearity, precision, recoveries were good. The sensitive LC-MS/MS technique provides a powerful tool quickly quantitation, trace analysis, and narcotics abuse review.

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