Forensic Drugs Analysis by Thermal Desorption and Pyrolysis Combined with Direct Analysis in Real Time- Mass Spectrometry (TDP/DART-MS)

Hiroko Abe[1], Chikako Takei[2], Yasuo Shida[2,3], Motoshi Sakakura[4], Teruhisa Shiota[4], Kayako Suga[5], Daisuke Yajima[1], Hirotaro Iwase[1,6]

[1]University of Chiba, [2]BioChromato, Inc., [3]University of Yamanashi, [4]AMR, Inc., [5]AB SCIEX, [6]University of Tokyo

Introduction

Drugs in biological and autopsy specimens cannot be detected without first selecting the pretreatment and analytical conditions appropriate for the drugs. Thus, it is extremely important to investigate the analytical conditions suitable for specific compounds and samples. However, in recent years the situation in which new substances appear one after another, including New Psychoactive Substance (NPS) that threaten society, it is very difficult to examine individually the analytical conditions that are appropriate for each new substance. Thus, the comprehensive analysis system for drugs that require minimal investigation of sample preparation and analytical conditions are greatly desired. So ,we are investigating an analytical method for directly analyzing drugs in blood and urine that does not require any pretreatment. In this poster, we introduce about the results of drugs in urine.

Materials and Methods

◆Analytical systems and conditions

We conducted our study by assessing whether drugs could be detected by using this analytical system. Sample solutions were put into the POT (Fig.2) and samples were gradient heated by TDP device.

Mass Spec. : TripleTOF™ 5600 system (SCIEX)
Mass range : *m/z* = 100-1000,
MS measuring method : IDA (25IDA)
Ion Source : DART-SVP (IonSense)
Ionization gas : Helium
Helium gas temperature : 400℃
Thermal desorption : ionRocket (BioChromato)
Temperature program : RT → 300℃ (60℃/min)

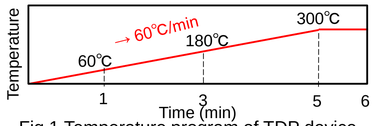

Fig.1 Temperature program of TDP device

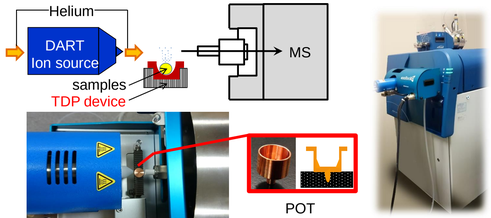

Fig.2 TDP/DART-MS system

◆Drugs mixture std.

α-PPP	AKB48	CUMYL-THPINACA	Acetylfentanyl
α-PVP	5fluoro-AMB	PCP	Acetylnorfentanyl
α-PHP	5fluoro-ADB	3-MeO-PCP	Amphetamine
α-PHPP	5-fluoro ADB-PINACA	Diphenidine	Methamphetamine
α-POP	5-fluoro AB-PINACA	Ketamine	MDMA

Results and Discussions

◆Analysis results of drugs mixture std.

As a results of drugs mixture standards, each drugs were separated and detected through thermal gradient heating for all samples. Moreover, the detected ions were correctly identified according to their measured accurate mass and product ion spectra.

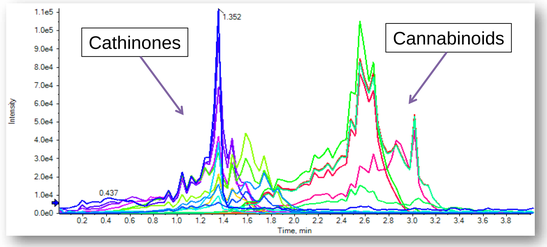

Fig.3 Extracted ion current gram (EIC) of 1μg/mL drugs mixture std. in ACN

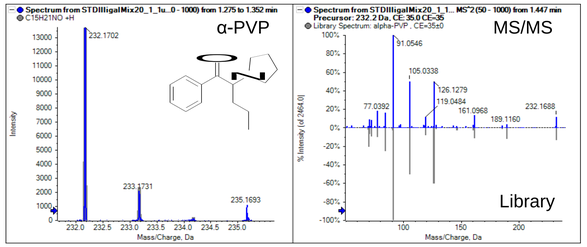

Fig.4 Accurate mass spectrum and MS/MS Spectrum of 1μg/mL std.

◆Optimization of sample preparations

In order to optimize the way of sample pretreatment, we analyzed urine sample which was added drugs mixture standard (drug concentration: 0.1 μg / mL).
As a results of comparing non-treated and acetonitrile(ACN) deproteinization treated, the intensity of each drugs in ACN deproteinization treated urine sample were increased considerably.

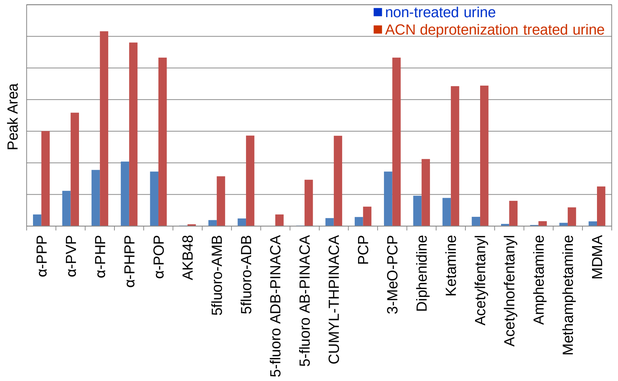

Fig.5 EIC peak area of each drugs in urine

◆Calibration curves

Calibration curves were prepared with urine added drugs at the concentrations ranging 0.01–1 μg/ml, the curves were linear in that range.

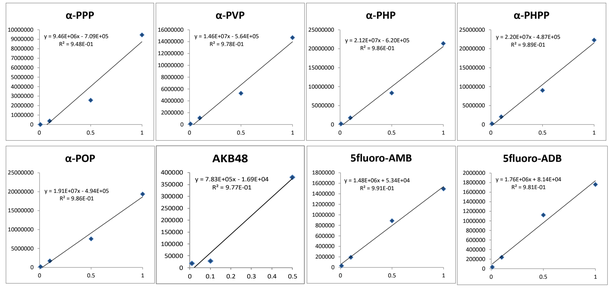

Fig. 6 Calibration curves

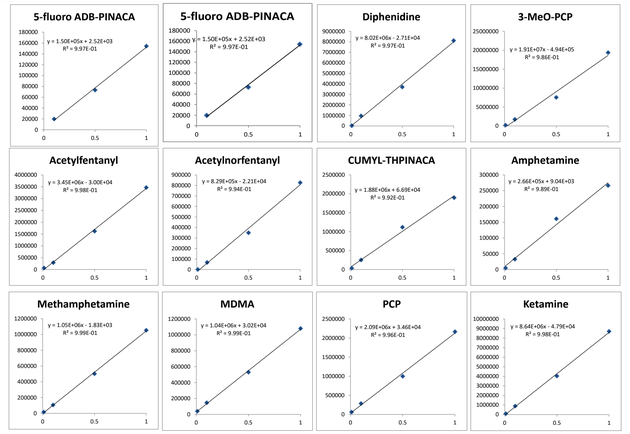

Fig. 6 Calibration curves

◆Application to forensic case

This method was successfully applied to real cases, allowing identification : α-PVP, α-PHP, α-PHPP, α-PVT, DL-4662 and 3MeO-PCP (post-mortem).

Table 3 The results of analysis in femoral vein by TDP/DART-MS

	α-PVP	α-PHP	α-PHPP	α-PNP	α-PVT	DL-4662	4-metyl buphedrone	3MeO-PCP
LC-ESI	0.24	0.74	0.12	detected	detected	1.1	0.25	0.72
TDP/DART-MS	detected	detected	detected	N.D.	detected	detected	N.D.	detected

(μg/mL)

Conclusions

- By using TDP/DART-MS for drugs in urine, each drugs were separated and detected through thermal gradient heating for all drugs. Moreover, the detected ions were correctly identified according to their measured accurate mass and product ion spectra.
- By using TDP/DART-MS, we can analyze drugs in urine directory, disregarding the ion suppression of urea.
- Deproteinization by ACN pretreatment is effective for improving the detection intensity of drugs in urine.
- The calibration curves were prepared with urine added drugs at concentrations ranging 0.01–1 μg/ml, the curves were linear in that range. Thus, TDP/DART-MS deemed to be useful method for quantitative analysis of drugs in biological and autopsy specimens. So, we are investigation to improve the detection intensity of drugs.

65th ASMS Conference