On-site Illicit Drug Identification by Portable GC-MS and SPME

Analysis of illicit drugs on a portable GC-MS, the TRIDION®-9. The instrument was used to analyze underivatized illicit drugs including amphetamine type stimulants (ATS) and cocaine. The addition of ammonium hydroxide to form free base drugs improved the chromatography of the samples. The TRIDION-9 coupled with CUSTODION® solid phase microextraction (SPME) proved effective for analysis and identification.

Introduction

Gas chromatography-mass spectrometry (GC-MS) is commonly used in forensic laboratories with applications including explosives, illicit drugs, chemical warfare agents and hydrocarbons in fire debris. Together, gas chromatography and mass spectrometry provide an ideal analytical tool. In a forensic environment, a benefit of on-site analysis is the ability to screen samples which allows only positive samples to be forwarded to the forensic laboratory for full analysis, thereby decreasing the overall sample workload for both time and cost savings.

GC-MS is an established method for the analysis and identification of illicit drugs[1-2], and is the preferred analytical method in most forensic laboratories due to its ability to detect and identify a broad range of sample components including drug impurities and by-products[3]. This is particularly important for impurity profiling. Currently, impurity profiling via GC-MS allows investigators to identify precursors, intermediates, by-products and excipients by producing a chemical 'fingerprint'. These impurities are often the result of products from side reactions, poor chemical handling during synthesis, inadequate purification techniques, and contamination of reactants, materials, or packaging. As a result of these factors during the manufacturing process, illicit substances are rarely pure. Links can be made between drug seizures, drug sources and also drug trafficking routes using this chemical 'fingerprint'.

Sample Preparation and Analysis

The analysis of illicit drugs including amphetamine type stimulants (ATS), various precursors, heroin and cocaine, was performed. The illicit substances analyzed are listed in Table 1 below.

Each analytical standard was diluted with water to approximately 200 ppm. Cocaine was available in dichloromethane solution and was not diluted, but was sampled using a coiled wire filament (CWF) sampler. Samples were not derivatized as this is impractical for on-site analyses.

Table 1. List of illicit drugs analyzed on the TRIDION-9 GC-MS

Compound	Chemical Formula
Caffeine	$C_8H_{10}N_4O_2$
Amphetamine	$C_9H_{13}N$
1,3-Benzodioxole, 5-(2-propenyl)- (Safrole)	$C_{10}H_{10}O_2$
3,4-Methylenedioxy-amphetamine (MDA)	$C_{10}H_{13}NO_2$
Methamphetamine	$C_{10}H_{15}N$
Pseudoephedrine	$C_{10}H_{15}NO$
N-methyl-3,4-methylenedioxy-amphetamine (MDMA, Ecstasy)	$C_{11}H_{15}NO_2$
3,4-Methylenedioxy-n-ethylamphetamine (MDEA)	$C_{12}H_{17}NO_2$
Ketamine	$C_{13}H_{16}ClNO$
Cocaine	$C_{17}H_{21}NO_4$
6-Dimethylamino-4,4-diphenyl-3-heptanone (Methadone)	$C_{21}H_{27}NO$

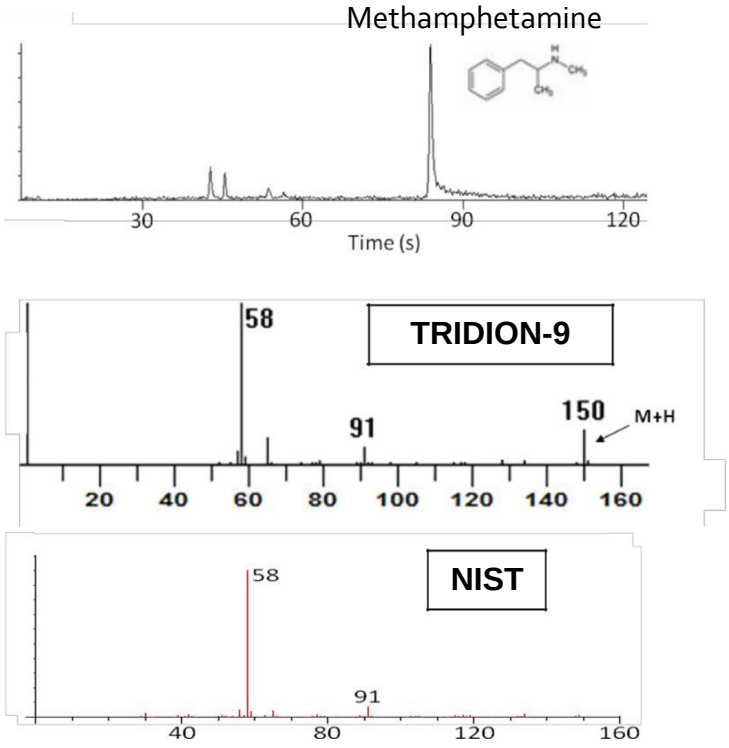

Figure 1. Chromatogram and mass spectrum of underivatized methamphetamine in water after a 20 s immersion using a PDMS/DVB SPME fiber.

A CUSTODION SPME syringe was used and consisted of a 1cm length of silica fiber coated with a liquid polymer film of polydimethylsiloxane (PDMS) and divinylbenzene (DVB). Samples were analyzed on the TRIDION-9 GC-MS using an MXT-5 GC column (5m x 0.1mm, 0.4µm film thickness). All analytes (except cocaine) were extracted using a PDMS/DVB SPME fiber that was directly immersed in the solution for approximately 20 seconds, with gentle manual agitation. As most of the samples were hydrochloride salts, 1 drop per mL of concentrated ammonium hydroxide was added to form the free base prior to analysis to improve chromatographic peak shape.

After sampling, the SPME fiber was introduced into the injection port on the TRIDION-9 where the analytes were desorbed from the fiber into the low thermal mass capillary GC column, followed by a rapid GC temperature program. The low thermal mass GC is directly interfaced to a toroidal ion trap mass spectrometer (TMS) with a mass range from 43-500 Da. Overall, this method is rapid with a total SPME sampling time of 20 sec and GC-MS analysis time of ~2 min.

Experimental Conditions

Sampling:	Solid phase microextraction (SPME)
SPME Phase:	Polydimethylsiloxane/divinylbenzene (PDMS/DVB)
GC Inj. Temp:	270°C
GC Column:	MXT-5, 5 m x 0.1 mm, 0.4 µm d_f
GC Carrier Gas:	Helium, constant pressure
GC Column Temp:	50°C (hold 10s), 50-300°C at 2°C/s, hold 300°C (70s)
Transfer Line:	270°C
Injection Split:	20:1 after 4 sec splitless
Mass Analyzer:	Toroidal ion trap (TMS)
TMS Mass Range:	43-500 Da
Ionization Mode:	In-trap electron impact (EI)
Detector:	Electron multiplier
Vacuum:	Roughing and turbo molecular pumps
Resolution:	Less than unit mass to 230 amu, nominal unit mass to 500 amu

Results

The following illicit drugs were successfully detected and identified according to their retention times and mass spectra: methamphetamine, amphetamine, MDEA, MDMA, MDA, ketamine, caffeine, pseudoephedrine, safrole, methadone and cocaine. All substances were detected with a total analysis time (including sampling) of less than four minutes.

Conclusions

Analysis of underivatized illicit drugs is possible using the TRIDION-9 GC-MS. A total of 11 illicit substances were successfully analyzed including various amphetamine type stimulants, precursors and cocaine. SPME sampling was successful using a 65µm PDMS/DVB for amphetamine type stimulants (in compatible solvents), while the CWF was used to successfully sample cocaine dissolved in dichloromethane.[4]

References

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