Application Note

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Fast Analysis Workflow with No Sample Preparation for Forensic Applications Using QuickProbe GC/MS

Author

Luis A. Cuadra-Rodriguez
Agilent Technologies, Inc.

Abstract

This Application Note describes a straightforward, quick workflow for drug analysis using the Agilent QuickProbe GC/MS system without sample preparation. Sub-minute analysis was possible for complicated samples of seized drugs, including edible cannabis, black tar heroin, and magic mushroom.

Introduction

The need for fast analysis for the identification of compounds in a variety of samples has been increasing over time, especially for seized drugs.[1,2] Positive identification of drugs and other chemicals in bulk samples is critical during screening in crime laboratories. Conventional drug analysis often requires sample preparation that includes dissolution, dilution, and several reagent-based assays to classify the type of drugs, followed by gas chromatography/mass spectrometry (GC/MS) analysis or other techniques for confirmation.[3,4] The QuickProbe GC/MS demonstrates a simple and fast analysis workflow that does not require sample preparation.

Experimental

Samples

A variety of drug samples were analyzed including prescription drugs in tablet form (Oxycodone and Vicodin) and seized drugs from criminal cases such as black tar heroin, magic mushrooms, and a cannabis edible (cookie) (Figure 1).

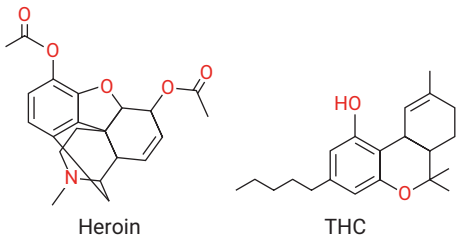

Figure 1. Chemical structures of two drug compounds tested in this study.

Instrument

QuickProbe is installed on the detector slot on top of the GC of a GC/MS system (Figure 2). It consists of a heated inlet open to the atmosphere, with a constant helium flow that prevents air intrusion. The system uses a short capillary column (Agilent J&W DB-1ht, ~1.5 m × 0.25 mm, 0.10 µm) that is rapidly heated (up to 16 °C/s or 960 °C/min), allowing for basic chromatographic separation in under one minute. Individual samples (liquid, solid, and powder) were touched with a glass probe (Figure 3) and introduced into the QuickProbe inlet for three to six seconds for vaporization prior to data acquisition with the GC/MS. Little to no sample preparation was required. Compound identification was achieved through searches performed through standard GC/MS data analysis packages (Agilent MassHunter Qualitative Analysis, Quantitative Analysis, and Unknowns Analysis software) and against the NIST or Wiley libraries.

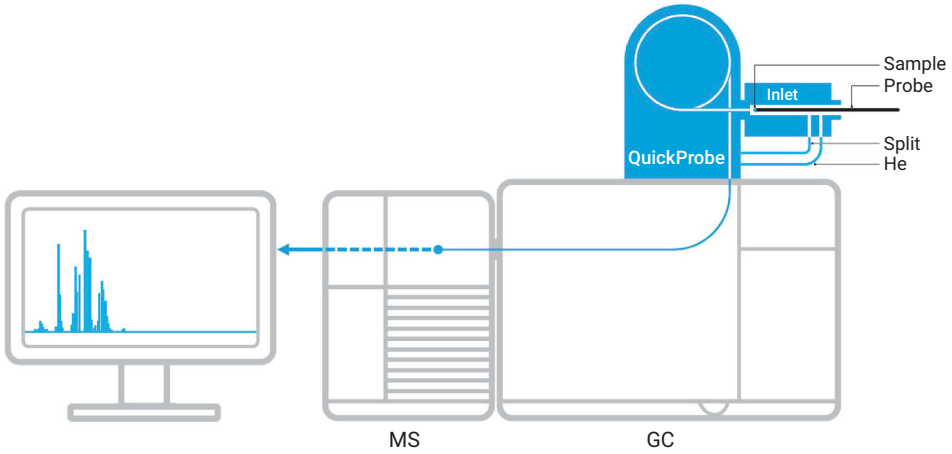

Figure 2. Schematic for the Agilent QuickProbe GC/MS system configuration.

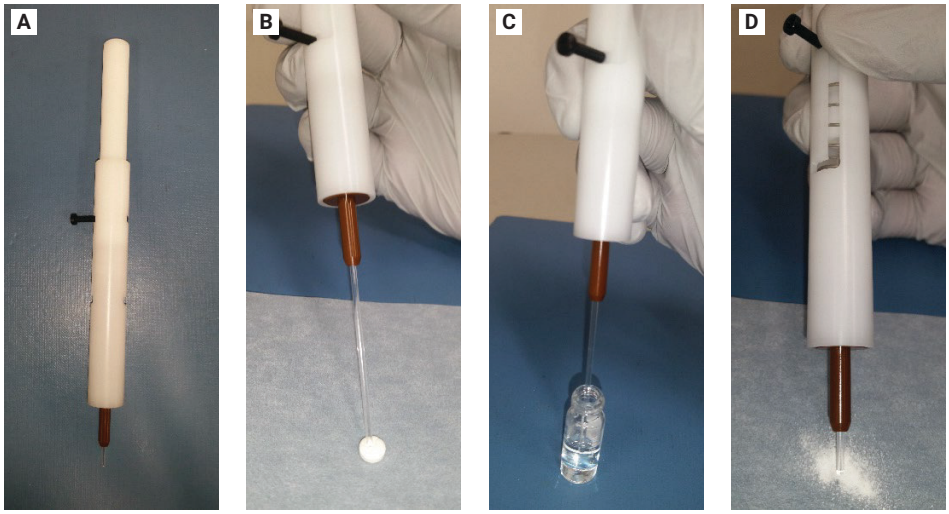

Figure 3. Sample preparation consists of touching the sample with a glass probe in a probe holder (A) as shown for B) solid (tablet), C) liquid, or D) powder (pulverized tablet).

Results and discussion

The analysis of a pulverized Vicodin tablet (5:300 mg of hydrocodone:acetaminophen) with no sample preparation in under one minute resulted in chromatographic separation of the two main components, namely acetaminophen and hydrocodone, thereby demonstrating the capabilities of QuickProbe GC/MS (Figure 4). Additionally, the two active ingredients were identified with a NIST library match of >90 even when the hydrocodone accounted to <2% by weight of acetaminophen.

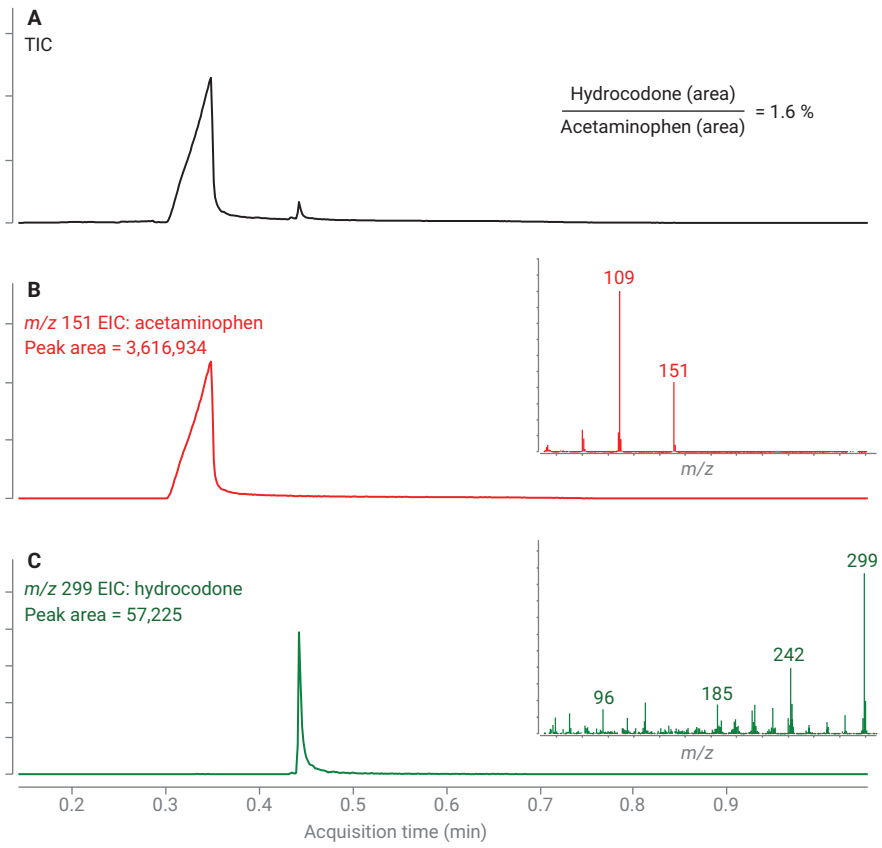

Figure 4. Pulverized Vicodin tablet (5:300 mg of hydrocodone:acetaminophen) analysis in ~one minute. A) Total ion chromatogram (TIC). Extracted ion chromatograms (EIC) for acetaminophen *m/z* 151 (B) and hydrocodone *m/z* 299 (C). NIST library match >90 for both components.

The combination of fast analysis with minimal sample preparation, basic chromatographic separation, and library searches allows for the development of simple, forensically sound workflows (Figure 5). Fast screening workflow analysis in under five minutes includes the following steps:

1. System blank
2. Probe blank
3. Sample
4. System blank

Figure 5 shows the analysis of an Oxycodone tablet using the workflow. Blank runs show background peaks such as phthalates and organic acids. The sample extracted spectrum was identified as Oxycodone with a NIST library match of 93. The final system blank shows the system is back to normal background levels, and is ready for the next screening analysis.

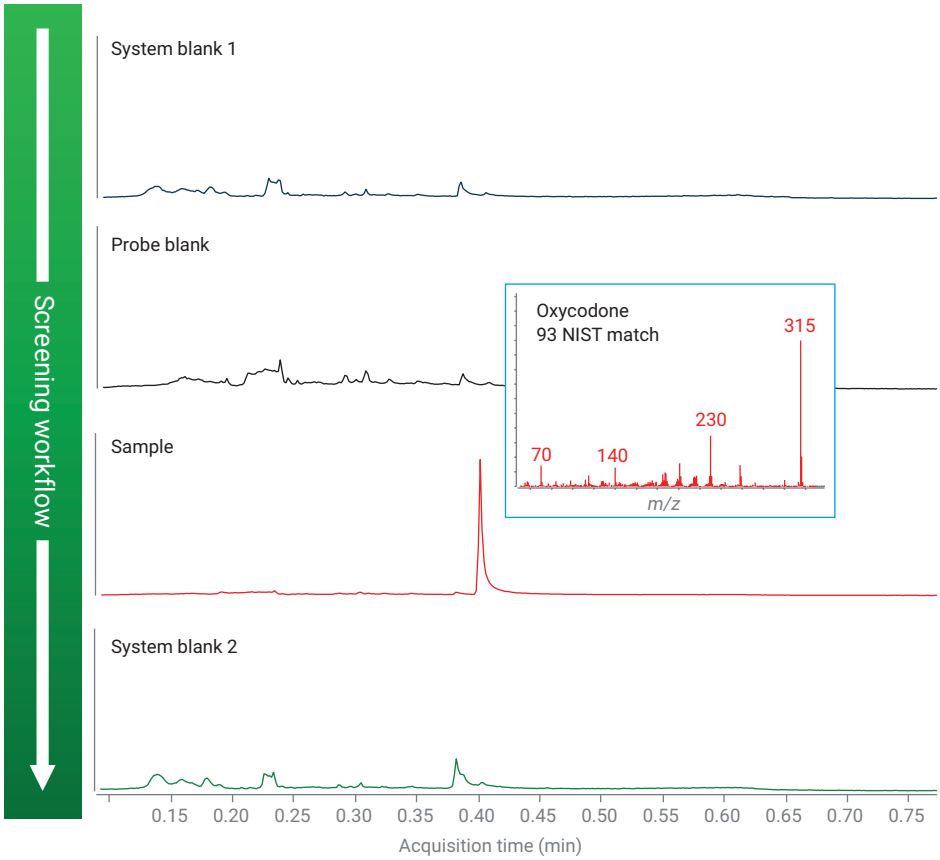

Figure 5. Screening workflow analysis of an Oxycodone tablet in <five minutes.

The fast workflow was applied to seized drug samples from forensic cases. Figure 6 shows the analysis of a cannabis edible (cookie), black tar heroin and "magic" mushroom using MassHunter Unknowns Analysis software. Note that major and minor components were identified with high NIST library match scores. For example, the analysis of the cannabis edible showed THC/dronabinol, cannabichromene, and cholesterol in the sample. Heroin, acetylcodeine, 6-monoacetylmorphine, papaverine, and noscapine were identified in the black tar heroin sample. In the "magic" mushroom analysis, psilocin was identified, even though it was not a major peak. Table 1 shows the summary of results of a variety of seized drug samples and tablets.

Table 1. Summary of the screening analysis results of forensic case samples and prescription drugs.

Sample	Compound	NIST Library Match	Formula
Back Tar Heroin	Acetylcodeine	97	$C_{20}H_{23}NO_4$
	6-Monoacetylmorphine (6-MAM)	98	$C_{19}H_{21}NO_4$
	Diacetylmorphine (Heroin)	98	$C_{21}H_{23}NO_5$
	Papaverine	93	$C_{20}H_{21}NO_4$
	Noscapine	98	$C_{22}H_{23}NO_7$
Cannabis Edible	Dronabinol	99	$C_{21}H_{30}O_2$
	Cannabichromene	89	$C_{21}H_{30}O_2$
"Magic" Mushroom	Psilocin	90	$C_{12}H_{16}N_2O$
Cocaine Powder	Cocaine	98	$C_17H_{21}NO_4$
	Tetramisole	97	$C_{11}H_{12}N_2S$
Red Tablet	Sildenafil (Viagra)	92	$C_{22}H_{30}N_6O_4S$
Tablet (Alprazolam)	Alprazolam	99	$C_{17}H_{13}ClN_4$
Tablet (Oxycodone)	Oxycodone	97	$C_{18}H_{21}NO_4$
Tablet (Vicodin)	Acetaminophen	99	$C_8H_9NO_2$
	Hydrocodone	96	$C_{18}H_{21}NO_3$

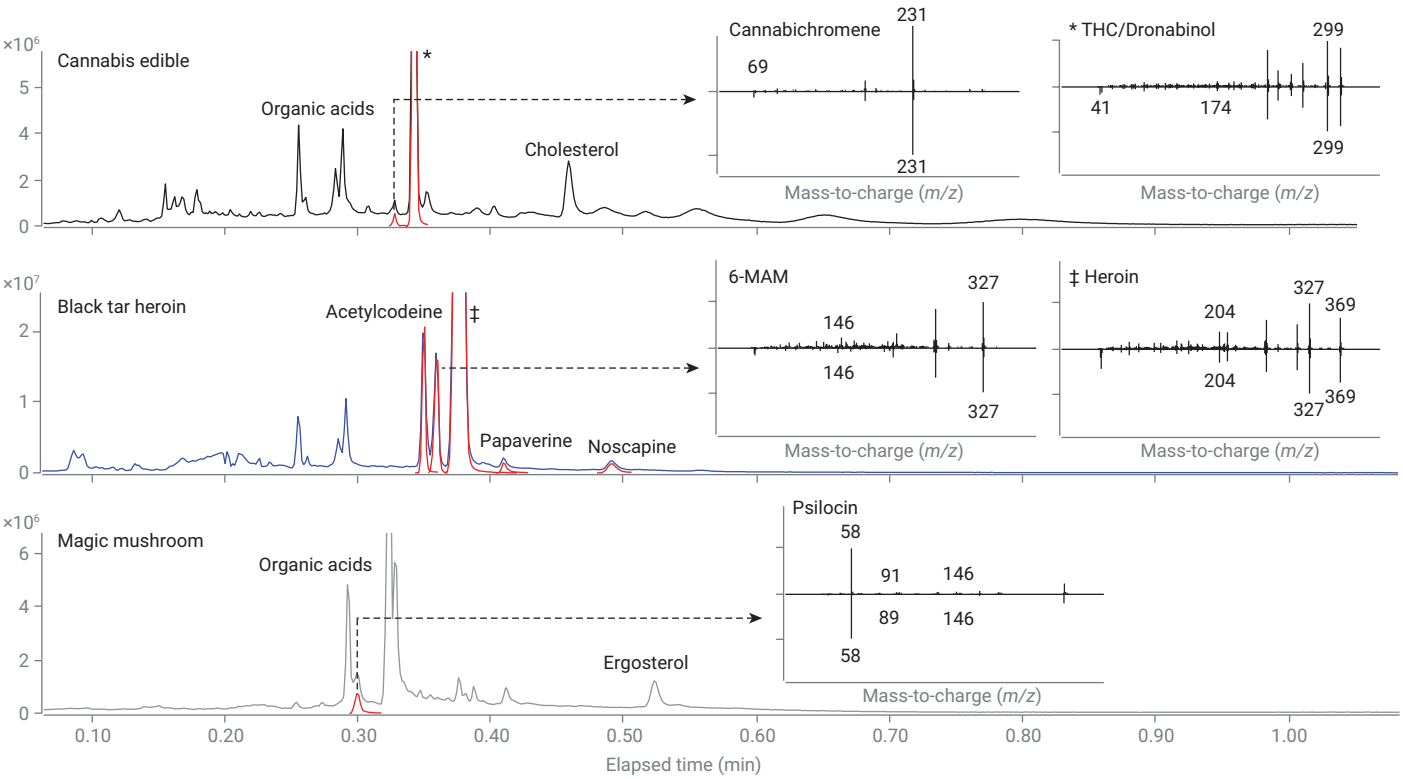

Figure 6. Sub-minute fast screening analysis (without sample preparation) of various forensic case samples including a cannabis edible, black tar heroin, and "magic" mushroom. Comparison spectra (head-to-tail) are shown for the main target compounds in each sample.

The positive identification of the main components can guide the analyst to specific sample preparation procedures or confirmation methods as in the tablets analysis examples. Furthermore, extensive time savings could be achieved in the sub-minute screening analysis of seized drugs for complicated samples (such as cannabis edible, black tar heroin, and "magic" mushroom).

Conclusion

Fast sample analysis with little to no sample preparation was demonstrated in the screening of tablets and seized drugs in bulk samples of different physical states (solid, gel, or powder). The analyses were performed rapidly with the positive identification of drug components by NIST library match and known origin. A fast and forensically sound analysis workflow was shown for screening that involved: 1) system blank; 2) probe blank; 3) sample; and 4) system blank.

References

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