Application Note

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Increasing Throughput for Forensic Screening of Raw Case Samples Using the Agilent QuickProbe GC/MS System

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Abstract

Forensic analysis prescreening using the Agilent QuickProbe GC/MS system allows a simple and fast analysis workflow that does not require sample preparation. The technique of ultrafast chromatographic separation resulting in library-searchable mass spectra permits the development of a forensically sound screening process. Because the QuickProbe GC/MS eliminates the need for preparation steps and reagent-based assays prior to confirmation testing, laboratory productivity can be significantly increased. The Alabama Department of Forensic Science (ADFS) requires reviewable data at all phases of the forensic analysis to increase defensibility in court, and spectra resulting from QuickProbe GC/MS screening satisfy this requirement.

Introduction

Criminal justice laboratories traditionally have used a sample analysis workflow that includes visual examination, weight measurement, and a number of presumptive tests prior to any subsequent confirmation through extraction and GC/MS analysis. In response to the NAS report and in preparation for accreditation under ISO 17025, the ADFS rewrote operating procedures to require reviewable data at all phases of the forensic analysis. Evaluation of the QuickProbe GC/MS (Figure 1) provided a means of prescreening seized samples with resulting mass spectra without any extraction required, which could lead to maximized throughput.

Figure 1. Agilent QuickProbe unit (G3971A) mounted on an Agilent 5977 GC/MS system.

Experimental

The QuickProbe unit is installed on the detector slot on top of the GC instrument 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 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. A glass probe was first inserted into the probe holder, then, while in loading position (Figure 3), was dipped into a liquid sample, or scraped along a solid sample or plant material. Powdered or granular samples were sampled using pocket-tip probes, which have a small indentation in the tip for a cupped design. Sample introduction was performed by first retracting the

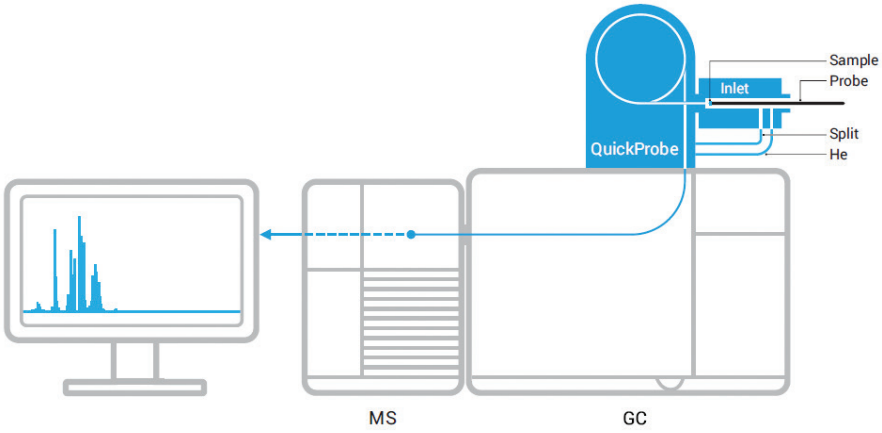

Figure 2. Schematic of QuickProbe direct insertion GC/MS instrument.

Figure 3. Sample probes in touchless packaging (A) and probe holder (B).

glass probe into the holder. The start button on the QuickProbe unit and the plunger on the probe holder were simultaneously depressed to start the run and to position the probe into the hottest part of the inlet. Insertion time was generally five seconds, but this time could be varied as required. Compound identification was achieved through searches performed through standard GC/MS data analysis packages: Agilent ChemStation, MassHunter Qualitative Analysis, Quantitative Analysis, and Unknowns Analysis software. A minimum match factor of 80 was used for the American Academy of Forensic Sciences (AAFS) and Cayman Chemical (Cayman) drug library matches.

Results and discussion

Case samples used in this study are those which had been previously analyzed, adjudicated, and turned into research samples. Sampling handling is critical to forensically sound analysis and probe-loading technique varies with sample type and the desired screen. The suggested workflow is: 1) run system blank, 2) run probe blank, 3) run sample, and 4) run blank. After system blanks are completed, the user may perform probe holder blanks to ensure absence of contamination of the holder tip, which may be rinsed with water or solvent for cleaning. Sample types including bulk materials and seized drugs are diverse in nature and a given screen may require a logistical approach. For example, sampling both the interior and exterior of a tablet may provide information regarding tablet components, previous handling, and storage environment. In certain cases, scraping the exterior surface with a scalpel can expose the interior ingredients.

Plant materials, such as cannabis and hemp, may be sampled by simply scraping or rubbing the material with the tip of the probe. In the case of cannabis, THC is normally the most intense peak in the chromatogram. THC and cannabidiol can be distinguished, however further sample evaluation would be required to determine whether plant material is industrial hemp or marijuana (the latter being defined as ≥0.3% THC by dry weight). The QuickProbe GC/MS can separate and lead to identification of the major component, THC, however it is not yet clear if it would be useful for a more accurate, quantitative purpose.

Due to the static nature of certain powdered samples such as cocaine, the probe holder was rinsed with water in between samples to prevent contamination. Alternatively, powders could be dissolved in 1 mL of methanol. Selected results for seized samples are shown in Figures 4 and 5.

Figure 4A. Seized sample of powdered cocaine, which was sampled using a pocket tip probe. The probe was then placed against a piece of clean weigh paper, pocket tip facing down, and rinsed twice with methanol. Once the probe dried, it was inserted into the probe holder, which was previously cleaned with water and dried with a laboratory wipe.

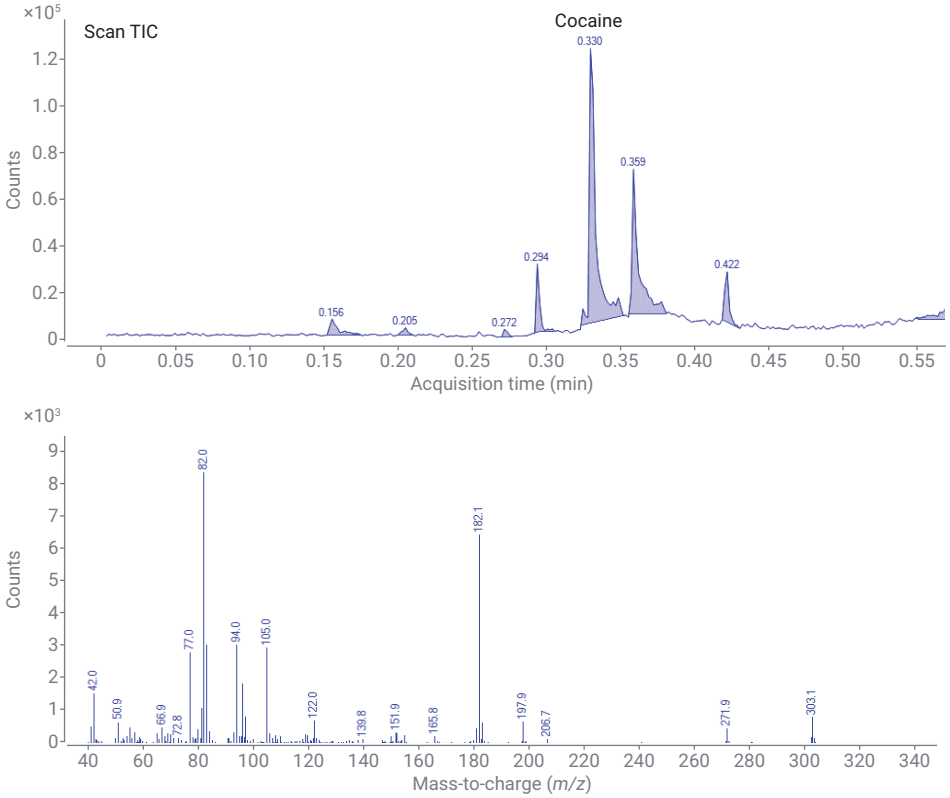

Figure 4B. Chromatographic and spectral results for the same sample of powdered cocaine. The cocaine peak elutes at 0.330 minutes. and the AAFS library match score is 99.

Resulting spectra of raw or dissolved samples provided excellent library match scores against AAFS and Cayman drug libraries. Therefore, any extraction steps could be eliminated in the screening process. Sample throughput was significantly increased since typical analysis time for a sample is one minute, while the total analysis time is three to four minutes, including analysis of appropriate blanks (system, probe, and any solvent if used).

Implementation of the QuickProbe GC/MS in a drug chemistry lab will result in quantifiable benefits such as the generation of reviewable spectra for case screening and the possibility of screening samples for which there had been no prior screening technique available, such as synthetic cannabinoids.

Table 1 provides results for case samples, including library match scores that were evaluated in this study. Results highlighted in yellow indicate that identified compounds did not match the known identification of the case samples (which had previously been adjudicated and turned into test samples for this study). Some missed identifications are readily explained. For example, clonazepam elutes late and is challenging by GC/MS. GHB must be derivatized to perform GC/MS analysis, and methamphetamine in lighter fluid is overwhelmed by the propane peak. Nicotinamide is a cutting agent for pseudoephedrine (some of the latter may be observed in a sample). However, QuickProbe correctly identified 83% of the samples studied.

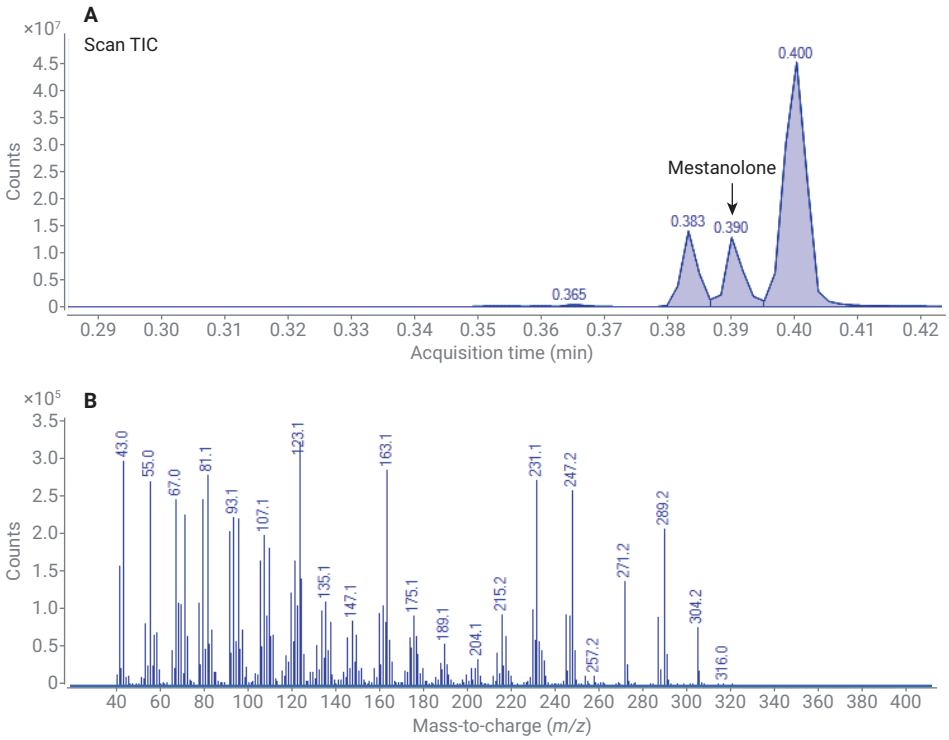

Figure 5. Chromatographic (zoomed in) and spectral results for a sample containing mestanolone. The mestanolone peak elutes at 0.390 minutes. and the AAFS library match score is 91.

Table 1. Case sample (previously adjudicated) results, including library match scores.

Sample	Major Identified Compound(s)	Identification
13HQ00031-1	Amobarbital (91), secobarbital (91)	Amobarbital Sodium
13HQ00048-1	Chlordiazepoxide (87)	Chlordiazepoxide
13HQ00114-1	Hexanedioic acid (93)	Plant material
13HQ00128-1	Stearic acid (89)	Clonazepam
13HQ00170-1	Lorazepam (99)	Lorazepam
13HQ00180-1	Oxazepam (72)	Oxazepam
13HQ00289-1	JWH250 (80)	JWH 250
13HQ00411-1	Morphine (98)	Morphine
13HQ00434-1	Phenobarbital (96)	Phenobarbital
13HQ00505-1	Pentobarbital (72)	Pentobarbital
13HQ00522-1	PCP (phencyclidine) (92), cyclohexene (95)	Phencyclidine
13HQ00525-1	Ketamine (97)	Meth, ketamine
13HQ00552-1	Dimethyl sulfone (90), nicotinamide (96), methamphetamine (64)	Meth, nicotinamide
13HQ00581-1	Temazepam (99)	Temazepam
13HQ00584-1	PCP (phencyclidine) (96)	Phencyclidine
13HQ00591-1	Pentobarbital (91)	Pentobarbital
13HQ00611-1	Benzoic acid (90), cocaine (99), anhydroecgonine methyl ester(98), benzoylecgonine (96)	Cocaine
13HQ00615-1	Cocaine (99)	Cocaine HCl
13HQ00622-1	Dimethyl sulfone (76), nicotinamide (96), methamphetamine (93)	Methamphetamine, nicotinamide
13HQ00866-2	Hexanedioic acid (47)	$GHB/H_2O/CHCl_3$
13HQ00869-1	Pseudoephedrine (83)	Pseudoephedrine

Sample	Major Identified Compound(s)	Identification
13HQ00881-1	Guaiphenesin (98)	Guaphenesin
13HQ00893-1	Mestanolone (91)	Mestanolone
13HQ00912-1	Hexanedioic acid (86)	Sodium bicarbonate
13HQ00933-1	Testosterone propionate (99), testosterone enanthate (72)	Steroid
13HQ00938-1	Diisoctyl adipate (72), hexanedioic acid (80)	Hydromorphone
13HQ00949-1	Dodecanoic acid (86)	Trenbolone
13HQ00970-1	Nandrolone decanoate (99)	Nandrolone decanoate
13HQ00972-1	Bezyl benzoate (98), testosterone cypionate (99)	Testosterone
13HQ00975-1	Nicotinamide (98)	Pseudoephedrine
13HQ01188-1	Codeine(97)	Codeine sulfate
13HQ01192-1	Hydromorphone (99)	Dilaudid
13HQ01193-1	Ketamine (97)	Ketamine
13HQ01195-1	heroin(99)	Heroin
13HQ01198-1	Benzophenone (91), nordiazepam (99)	Clorazepate
13HQ01217-1	AM2201 (99)	AM2201
13HQ01231-1	AM2201 (99)	AM2201
13HQ01238-1	Hexanedioic acid (86)	Negative
14HQ00003-1	1,4-Butanediol (83)	1,4-Butanediol
15HQ00097-1	Heroin (99)	Heroin and lactose
16HQ00095-1	Propane (78)	Meth in lighter fluid
16HQ00197-1	Caffeine (97)	Suspected kratom
16HQ00197-8	Hexanedioic acid (62)	Suspected kratom
18HQ00193-1	1-Di-Cyclobutanol (72)	Plant material
18HQ00195-1	Cannabidiol (90)	Relax gummies
18HQ00203-1	Hexanedioic acid (91)	Brownie
18HQ00204-1	Theobromine (95), glycerine (83)	Kush cakes
18HQ00216-1	Cannabidiol (93)	Pharxma
18HQ00218-1	Propylene glycol (86)	CBD Drip Gold
19HQ00057-1	Acetaminophen (87)	Hydrocodone/APAP syrup
HW250	Cannabidiol (98)	Cannabidiol
HW750	Cannabidiol (97)	Cannabidiol

As a result of evaluating the QuickProbe screening method, a complete workflow may be recommended for drug analysis case work. The diagram in Figure 6 shows that cases would be triaged by a screen team prior to analysis, instead of each case screened separately by the analyst as in traditional case analyses. QuickProbe GC/MS can be the screening technique used for any sample containing powders, liquids, or unknown plant materials. Following triage, a different analyst would perform the drug screening. This new workflow would limit the tasks necessary for each analyst, allowing the screen team to sort cases into batches for the analyst prior to the run. This, in turn, would add redundancy to the analysis and reduce employee workload.

The transition from a single analyst to a team of analysts per case would improve productivity. Cases could be sorted effectively into batches following the first sampling without extraction, allowing the confirmation analyst to test an entire batch of cases in sequence. QuickProbe screening generates reviewable data that can be recorded and stored with the batch for further reference. Since a minimum of two scientists would be involved in each case, there is increased confidence in analyte determination as an additional scientist would now confirm the evidence description.

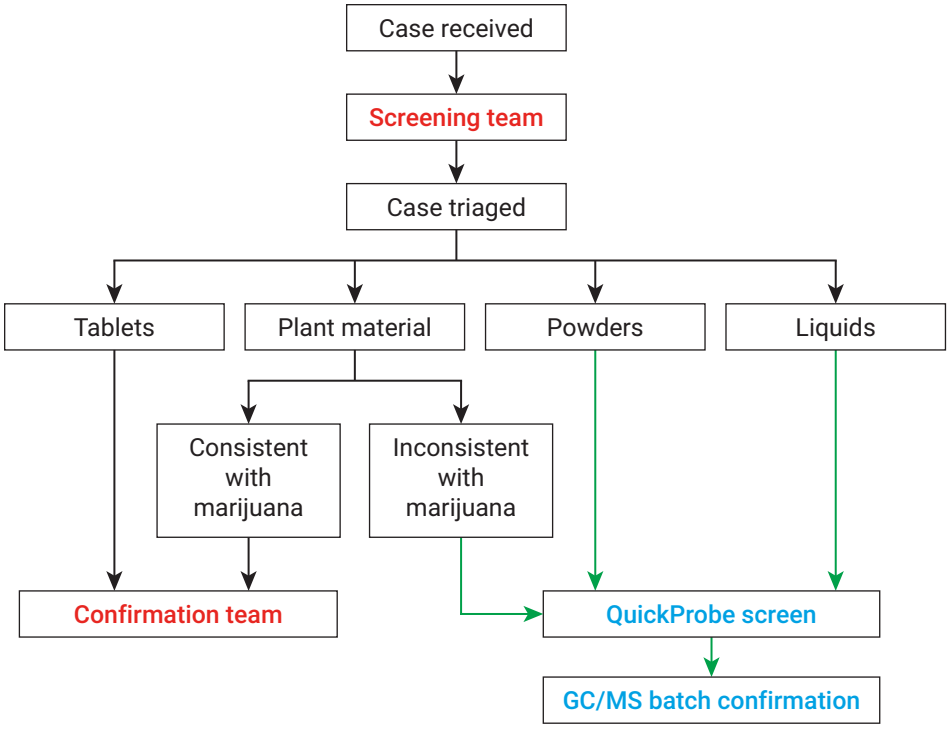

Figure 6. Recommended laboratory workflow.

Conclusion

It has been demonstrated that the QuickProbe GC/MS system screening method provides advantages over traditional screening techniques in forensic drug chemistry labs. The method provides a single screening test for a wide range of analytes, as well as reviewable data that can be recorded and stored with the case records. It also provides the ability to screen for emerging analytes that do not have a traditional screening technique, such as synthetic cannabinoids.

Using the QuickProbe GC/MS system as the screening choice increases confidence in the results since library-searchable mass spectra are generated with every run. The workflow developed as a result of this method improves productivity and confidence in analytical results.

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