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Sub-minute Analysis for Samples of Forensic Applications

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Introduction

The last two decades has seen a steady increase in the need for fast and accurate identification of compounds in a variety of samples and mixtures, especially in forensic laboratories. Positive identification of chemical components in bulk samples is critical during the screening process. Conventional analysis often requires sample preparation that includes dissolution, dilution and physical characterization followed by GC/MS analysis for confirmation. A simple and fast screening analysis that requires little to no sample preparation is demonstrated with a unique direct insertion GC/MS system. Compound identification of prescription drugs and standards is achieved through the routinely used NIST library search when using a single quadrupole (SQ) mass spectrometer.

Experimental

Instrument

The QuickProbe (QP) GC/MS system (Figure 1) consists of a heated inlet and a short analytical high temperature column (1.5m x 0.250mm x 0.10µm). Typically a ~400-600 °C/min temperature ramp was used that allowed for chromatographic separation in under 1 minute. The QP column is attached to the mass spectrometer through a ultimate union with a restrictor column (~0.6m x 0.18mm x 0.18µm).

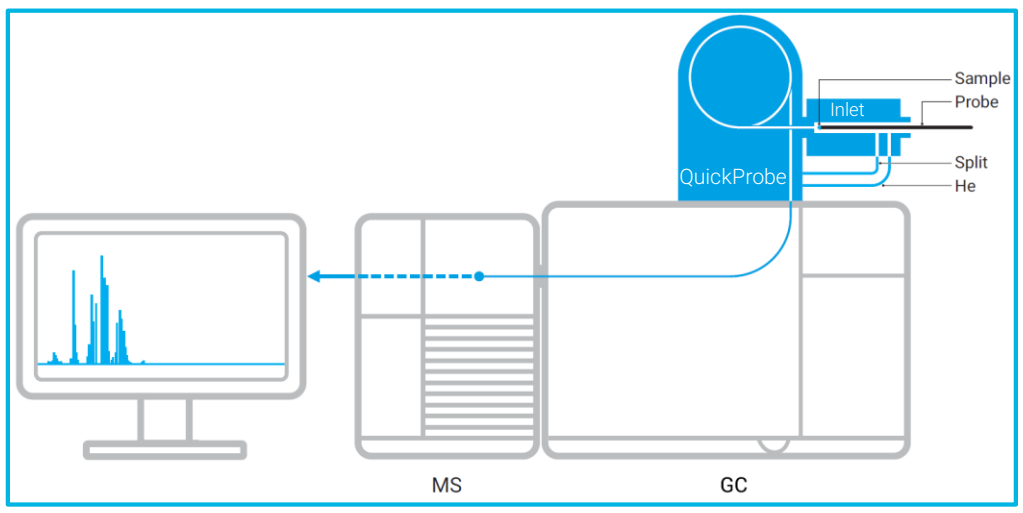

Figure 1. Schematic for the QuickProbe GC/MS system configuration.

Sample Analysis

Individual samples (liquid, solid, powder) were touched (Figure 2) with a glass probe and introduced into the QuickProbe GC/MS system (with SQ) for 3-6 seconds vaporization prior to data acquisition. Compound identification is achieved through NIST library search using existing GC/MS data analysis software. Spectra deconvolution within MassHunter Quant Unknown Analysis was used for the data shown herein. Custom and other existing libraries can also be used.

Experimental

Samples

A variety of samples were analyzed including;

- Prescription tablets or capsules. Capsules need to be opened in order to access the active ingredients.
- Seized drugs from criminal cases including: black tar heroin, magic mushrooms and a cannabis edible.

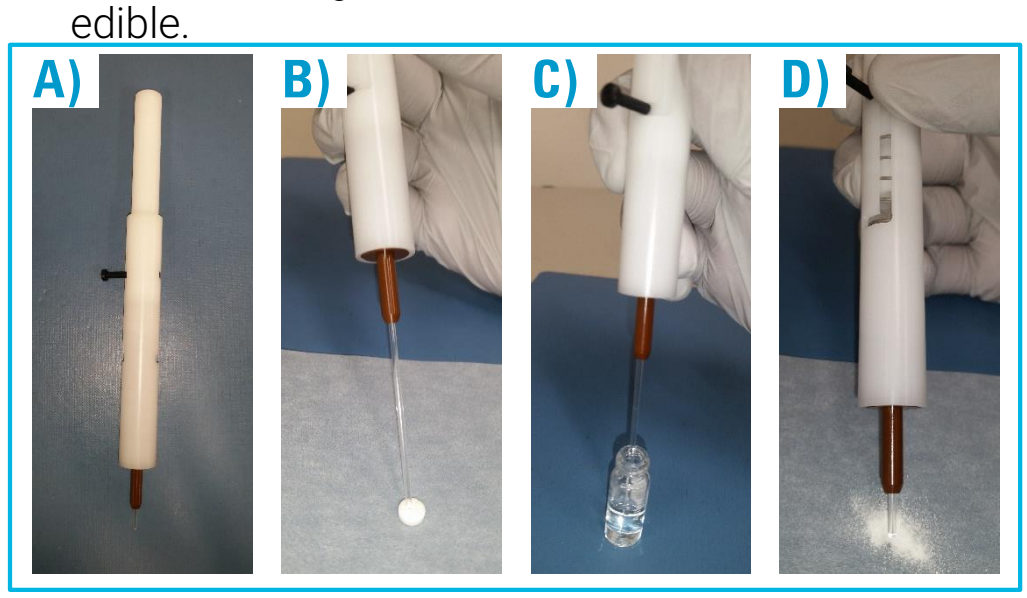

Figure 2. Sample preparation consists of touching the sample with a glass probe in a probe holder (A) as shown for B) solid (tablet), C) liquid or D) powder (pulverized tablet).

Results and Discussion

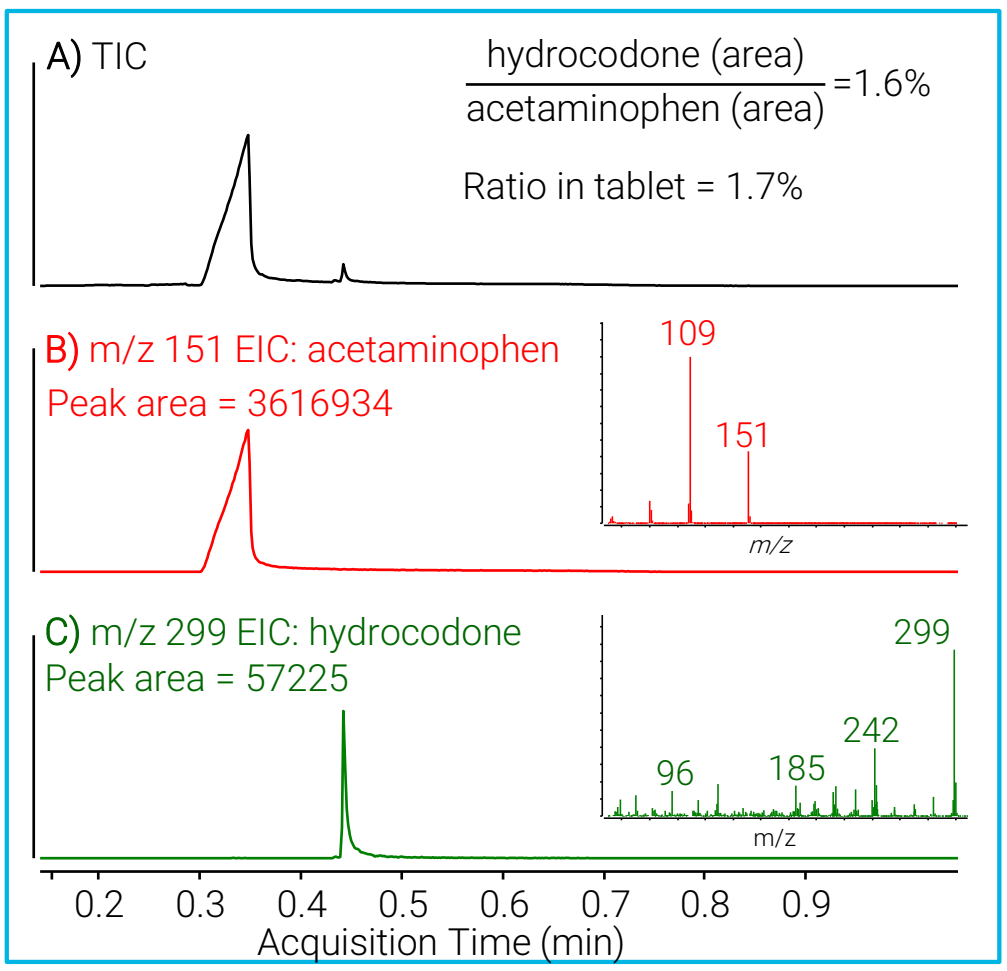

Figure 3. Pulverized Vicodin tablet (5:300 mg of hydrocodone:acetaminophen) analysis in ~1 min. A) Total ion chromatogram (TIC). Extracted ion chromatograms (EIC) for acetaminophen m/z 151 (B) and hydrocodone m/z 299 (C). Excellent agreement with the components ratio in the pill.

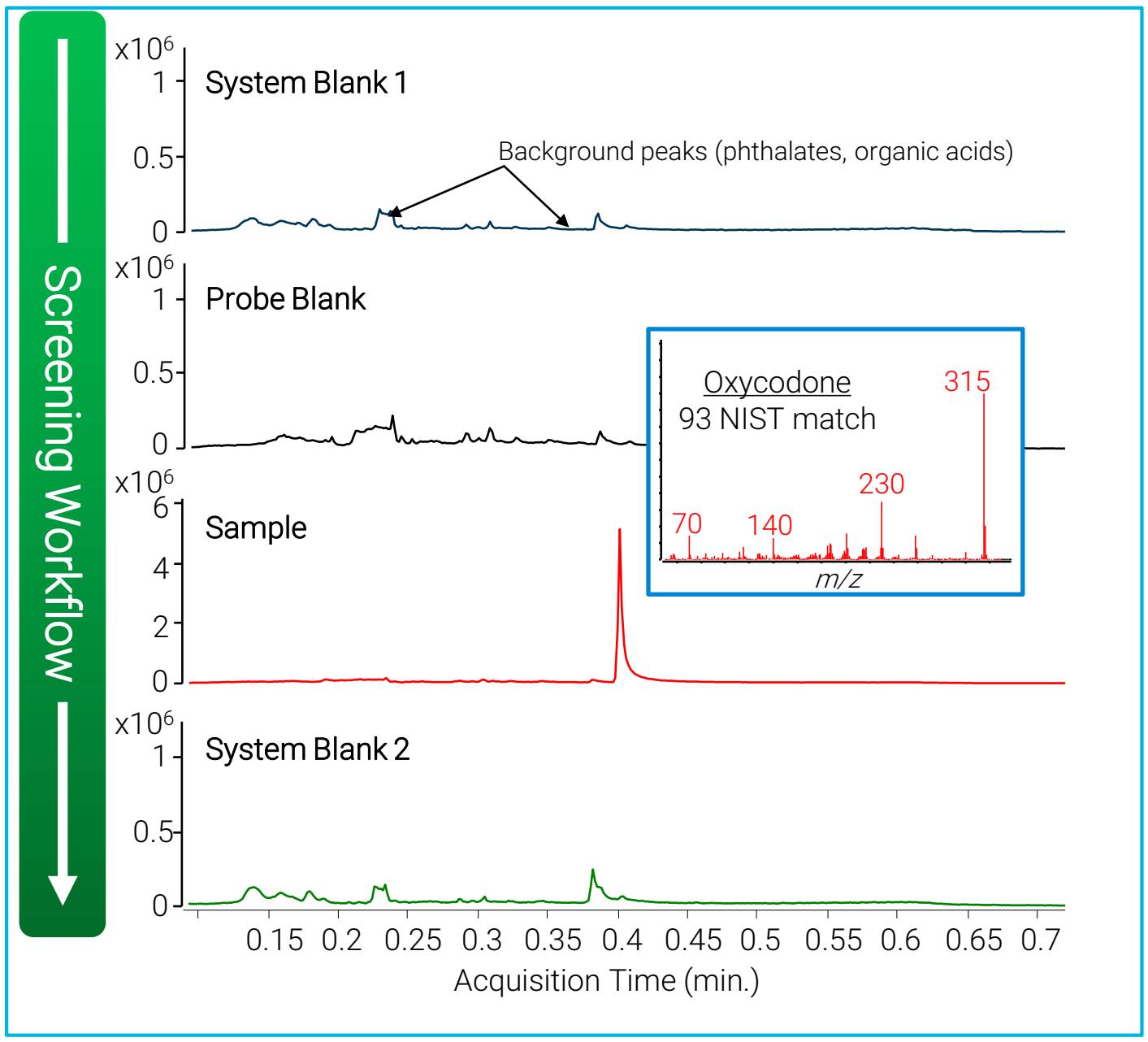

Figure 4. Screening workflow of an oxycodone tablet in < 5 minutes.

Fast screening workflow analysis (Figure 4) in under 5 minutes with the following steps: 1) System Blank, 2) Probe Blank, 3) Sample and 4) System Blank. Minimal or no sample preparation is required. Blank runs show background peaks such as phthalates and organic acids. The sample extracted spectrum is 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.

Spectra show the capabilities of using 70 eV electron ionization for compound identification (Table 1) coupled with Unknown Analysis spectra deconvolution analysis and NIST library searches. Figure 5 shows typical screening analysis of tablets while Figure 6 shows the analysis of seized drug samples. The positive identification of the main components can guide the analyst to specific sample preparation procedures and/or confirmation methods as in the tablets analysis examples. Additionally, extensive time savings were achieved in the sub-minute screening analysis of seized drugs for complicated samples (cannabis edible, black tar heroin, "magic" mushroom).

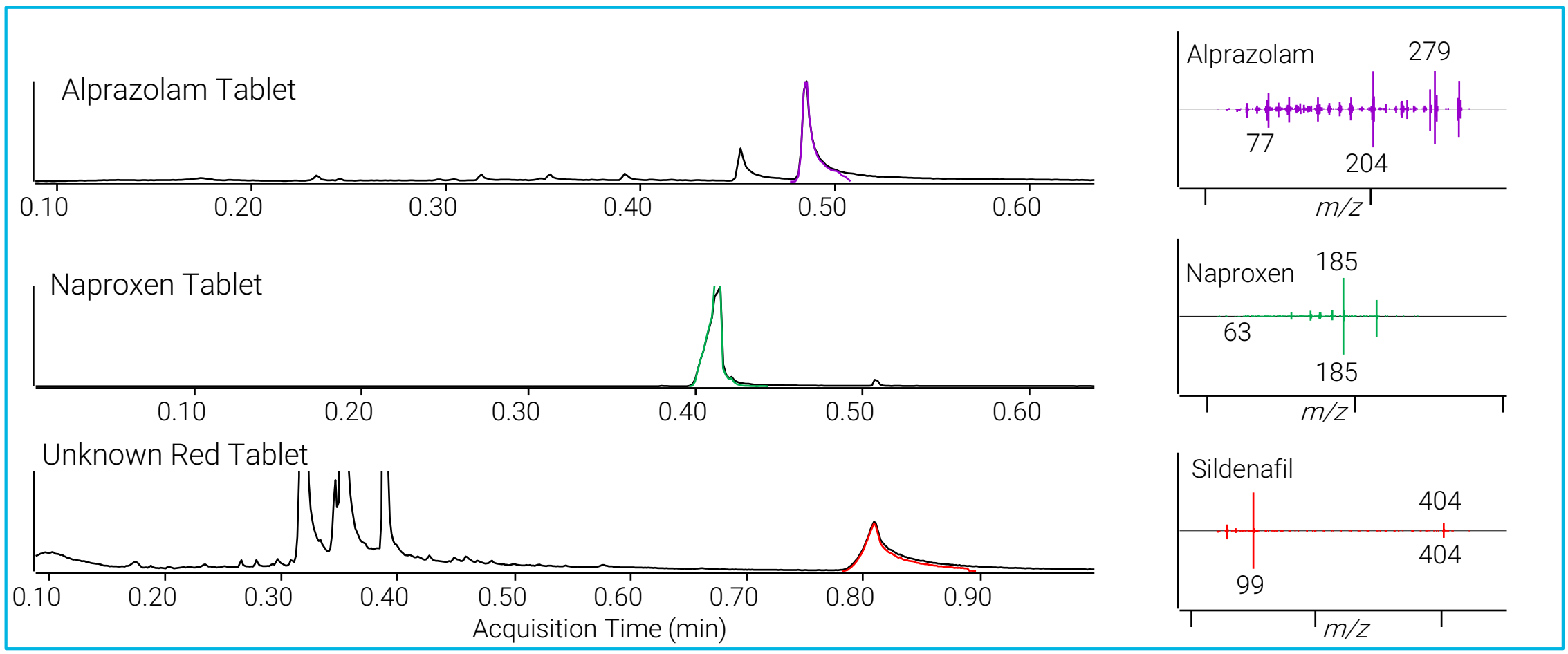

Figure 5. Examples of QuickProbe GC/MS screening analysis of tablets. Comparison spectra (head-to-tail) are shown for the main component in each sample.

Results and Discussion

Table 1. Summary of the screening analysis results of tablets and forensic cases

Sample	Compound Name	NIST library match	Formula
Tablet (Oxycodone)	Oxycodone	97	$C_{18}H_{21}NO_4$
Tablet (Vicodin)	Acetaminophen	99	$C_8H_9NO_2$
	Hydrocodone	96	$C_{18}H_{21}NO_3$
Tablet (Alprazolam)	Alprazolam	99	$C_{17}H_{13}ClN_4$
Red Tablet	Sildenafil (Viagra)	92	$C_{22}H_{30}N_6O_4S$
Capsule (Diltiazem)	Diltiazem	93	$C_{18}H_{36}O_2$
	Triethyl citrate	92	$C_{12}H_{20}O_7$
Tablet (Naproxen)	Naproxen	97	$C_{14}H_{14}O_3$
Tablet (Prednisone)	Androsta-1,4-diene-3,11,17-trione	96	$C_{19}H_{22}O_3$
Tablet (Lovastatin)	Simvastatin	97	$C_{25}H_{38}O_5$
Tablet (Metronidazole)	Metronidazole	99	$C_6H_9N_3O_3$
Tablet (Levothyroxine)	Not Detected	-	-
Tablet (Glimepiride)	Not Detected	-	-
Tablet (Metformin)	Not Detected	-	-
Back tar heroin	Acetylcodeine	97	$C_{20}H_{23}NO_4$
	6-MAM	98	$C_{19}H_{21}NO_4$
	Diacetylmorphine (Heroin)	98	$C_{21}H_{23}NO_5$
Cannabis edible	Dronabinol/THC	99	$C_{21}H_{30}O_2$
Mushroom	Psilocin	90	$C_{12}H_{16}N_2O$
Cocaine powder	Cocaine	98	$C_{17}H_{21}NO_4$
	Tetramisole	97	$C_{11}H_{12}N_2S$

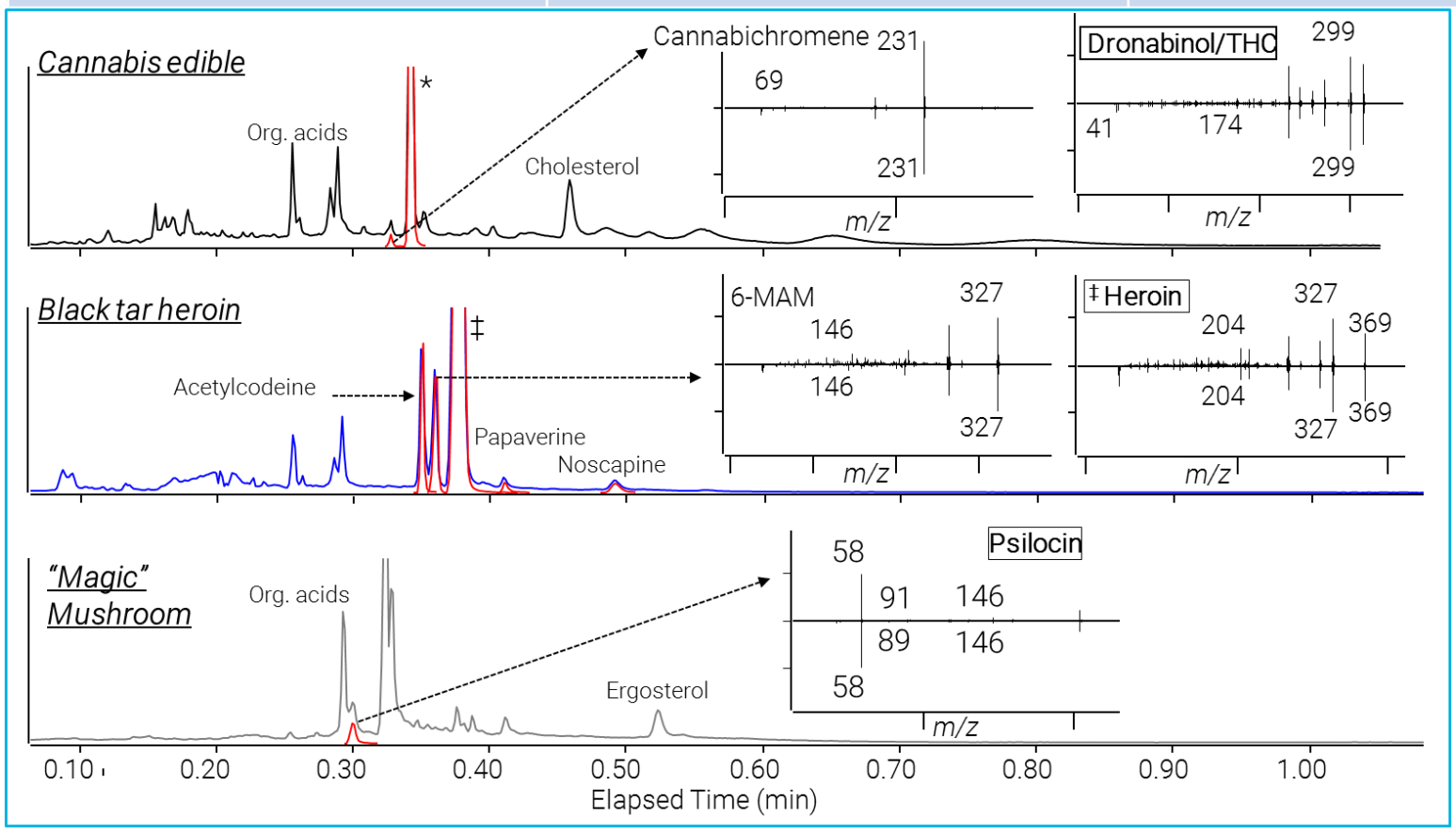

Figure 6. Sub-minute fast screening analysis with minimal sample preparation of various forensic case samples including a Cannabis edible, black tar heroin and "magic" mushroom. Comparison spectra (head-to-tail) are shown for some of the main target compounds in each sample.

Conclusions

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

For Forensic Use.

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