White Paper

Agilent
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Maximizing Efficiency of the Agilent QuickProbe GC/MS System

Using the QuickProbe GC/MS and consumables

Introduction

The Agilent QuickProbe GC/MS system is a novel system for quick analysis of various forensic samples including liquids, powders, tablets, plant materials, and plastics (Figure 1). The QuickProbe GC/MS provides fast separation and analysis of samples in under one minute for fast screening. It requires some unique consumables and techniques for successful and efficient incorporation into the laboratory environment. Samples are loaded onto probes and inserted into the QuickProbe inlet, but loading the probes requires some care to avoid overloading the GC/MS system (column and MS detector) or carryover. When used efficiently, the QuickProbe system and its consumables can quickly screen samples such as white powders or other suspected drugs of abuse, possibly reduce backlogs, and allow users to select which samples require confirmation.

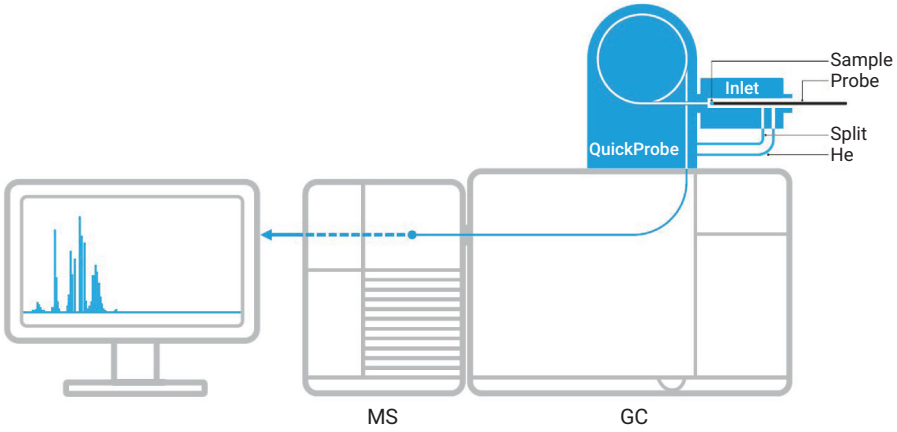

Figure 1. Schematic of the Agilent QuickProbe direct insertion GC/MS instrument.

The direct-insertion GC/MS system has a fast run time (<one minute), with adequate separation for peaks in the same compound class, and retains good peak shapes for compounds that normally tail in GC/MS analysis.

QuickProbe consumables

Table 1 contains the consumables necessary for sampling and running the QuickProbe GC/MS system.

Table 2 lists the recommended consumables to use with the carrier gas and split vent. Oxygen and moisture can degrade the columns and affect filament lifetime in the mass spectrometer. It is highly recommended to use the listed products to keep a clean system.

QuickProbe columns

The DB-1ht and DB-1ms column lengths were chosen to allow users to install these columns with extra length for initial column installation trim at each end of each column. The columns should be trimmed after insertion through the ferrules, as with normal column installation. The DB-1ht column must be installed from the Ultimate Union through the QuickProbe unit and end at the inlet, where the graphite Vespel ferrule is used. Other high-temperature columns may be used, but the columns must be high temperature-rated, due to the heater design of the QuickProbe unit. This column should be installed at ~1.5 m in length with short distance to the Ultimate Union to retain the fast separation and analysis time. See Figure 2 for the column connections in the GC oven from the QuickProbe through the Ultimate Union and to the MSD transfer line for an example of installation.

Table 1. Consumables and part numbers for the Agilent QuickProbe GC/MS system.

Consumable	Details	Part Number
Probe Holder	Use with glass probes	G3971-60200
Round Tip Insertion Probe	Convex rounded tip for use with liquids, tablets, and so forth sampling; 100 per pack	5190-5118
Pocket Tip Insertion Probe	Concave tip for powders, 100 per pack	5190-5113
TSP Vial Probe Holder	Use with TSP vial	G3971-20251
TSP Solid Probe Vial	Specialized vial for sampling solids like plastics, plant material, and so forth; 100 per pack	5190-3187
Agilent QuickProbe Ultra Inert Fritted Liner	Short fritted liner designed for use only with QuickProbe system	5190-5104
Agilent J&W DB-1ht QuickProbe GC Column	2 m × 0. 25 mm, 0.10 µm; high temperature column recommended for use in QuickProbe	G3903-61006
Agilent J&W DB-1ms Ultra Inert QuickProbe GC Column	1 m × 0.18 mm, 0.18 µm for connection to MSD	G3903-61007
Agilent Ultimate Union Kit	Connection point of two columns	G3182-61580
0.4 mm id Graphite Vespel Ferrules	Use on QuickProbe inlet and MSD transfer line (10 per pack)	5181-3323
0.4 mm id Flexible Metal Ferrules	Use for Ultimate Union connections	G3188-27501
MSD Transfer Line Self-Tightening Nut	Suggested nut for easier installation	5190-5233
CFT Capillary Internal Nut	For use with Ultimate Union and flexible metal ferrules	G2855-20530
GC Inlet Gold Seal	Used in QuickProbe inlet; includes washer	5188-5367

Table 2. Recommended consumables for gas lines exterior to GC/MS system.

Consumable	Details/Use	Part Number
External Split Vent Cartridge Kit	Use on the split line from QuickProbe inlet; includes trap and three cartridges	RDT-1020
External Split Vent Replacement Cartridges	Three per pack	RDT-1023
Gas Clean Carrier Gas Kit	Use on carrier gas line; includes 1 position 1/8 inch connecting unit and two carrier gas purifiers	CP17976
Gas Clean Carrier Gas Purifier	Replacement purifier	CP17973

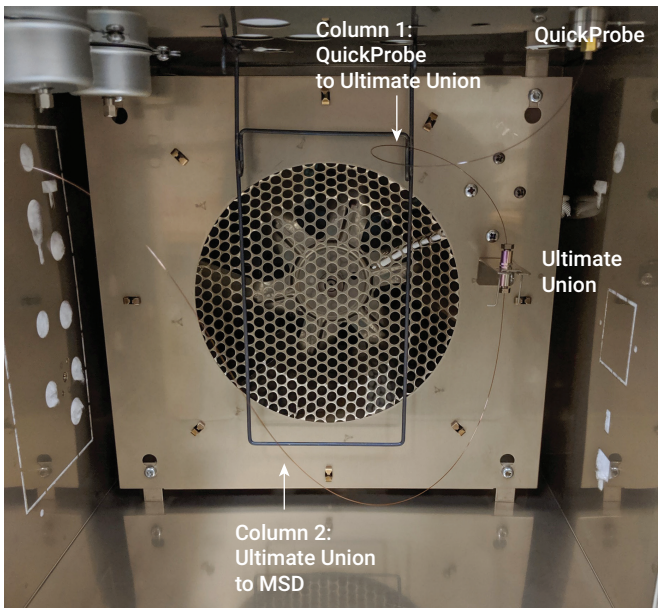

Figure 2. Interior of the oven showing the column 1 connection from the Agilent QuickProbe to the Ultimate Union, and the column 2 connection from the Ultimate Union to the MSD transfer line.

The DB-1ms column is installed from the Ultimate Union across the GC oven and into the MSD transfer line. Other columns may be used in this configuration, but it is encouraged that this length is kept to a shorter distance, ~0.7 mm, to retain the fast separation and analysis time (Figure 2).

QuickProbe liner

The QuickProbe fritted liner was developed specifically for the QuickProbe system, and is shipped in touchless packaging with a preinstalled O-ring. Installation of this liner into the QuickProbe inlet is easy and fast, but care must be taken. The inlet must be completely cool before changing liners for user safety and instrument safety, especially with the short distance between the inlet and mass spectrometer. It is best to complete a liner change when the system is cool, either at the end of the day or the first action of the day, when determined that a liner change is necessary.

CAUTION: Installation of the liner must be done when the inlet is completely cool. Installation of the liner when the system is hot can injure the user and cause damage to the column and mass spectrometer.

Probe holder, probes, and TSP vial

The probe holder has an O-ring in the base to grip onto the glass probes. The O-ring grip should be set during initial setup of the entire QuickProbe GC/MS system to make probe installation quick and easy throughout the lifetime of the system. This process is completed by a feel of resistance, but ensures easy probe replacements between samples. Follow these steps:

1. Unscrew the Vespel tip, and remove the plunger and spring from the probe holder.
2. Loosen the top of the plunger.
3. Insert a glass probe until it bottoms.
4. Tighten the top of the plunger until it grips the glass probe.
5. Remove and re-insert the glass probe to gauge resistance when loading a probe. The O-ring should grip to the point that the probe is held well and will not slip out, while probe insertion is still relatively easy and does not require a significant amount of force.
6. Once the O-ring on the plunger is set to the desired resistance, remove the glass probe, and re-assemble the probe holder. Insert the plunger and spring and screw on the Vespel tip.

Two types of glass probes can easily be used with the probe holder and the QuickProbe system: round-tip and pocket, which has a slightly cupped end.

The ends of the probes are fire-polished for user safety and easier insertion into the probe holder. Figure 3A shows the probe holder with the plunger depressed to position 1, and Figure 3B displays the probe holder with the plunger fully extended, and the installed probe fully retracted.

Round-tip probes (RTPs) are best for the following samples:

- Liquids
- Pills, such as tablets or caplets
- Other materials that can be scraped and will transfer material to the probe, such as vegetation and food items that will not leave a large amount of residue on the probe.

Figure 4 shows the RTP with a very small amount of tablet scrapings on the tip, as highlighted with the arrow in the figure.

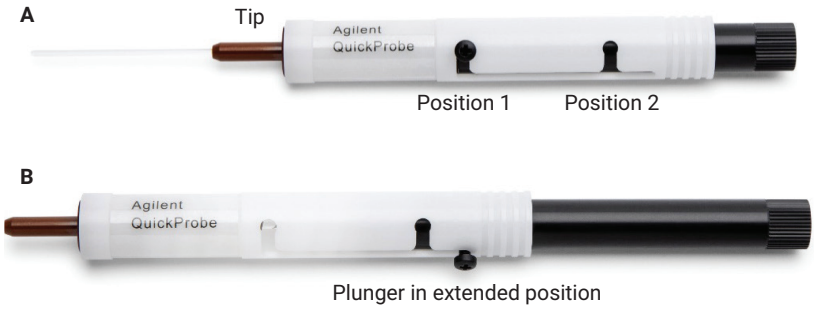

Figure 3. A) Probe holder with installed probe set at position 1 for liquid sampling; B) probe holder with probe fully retracted into the probe holder body.

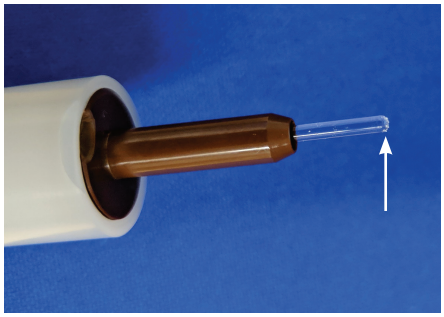

Figure 4. Round-tip probe with a very small amount of tablet scrapings on the tip, which is enough for sampling with direct-insertion GC/MS.

Liquid sampling with RTPs

To load the glass probes, depress the plunger to position 1 (Figure 3A) of the probe holder, and lock the plunger into place by rotating the plunger to trap the side screw. Then, pick up a probe, ideally with tweezers or a gloved hand, and insert it into the probe holder. There is a centering cone that should help a user find the base of the plunger. There should be a little resistance when the probe is inserted completely, where the gripping O-ring is located at the internal end of the plunger. Use a small amount of force to seat the probe into that O-ring; there should be approximately 46 mm extending past the tip of the probe holder. To check that the probe is fully seated, retract the plunger, at which point the end of the probe should be approximately flush with the tip of the probe holder.

CAUTION: Glass probes can break in the probe holder if too much force is used to insert the probe into the probe holder. Be careful when installing probes to avoid applying force with the palm of your hand. To lessen the possibility of injury, use tweezers or grip the sides of the probe (with gloved hands) to insert the probe into the holder and when applying any force.

To sample liquids with the RTP, depress the plunger to position 1, which is closest to the tip of the holder, and rotate the plunger to catch in the notch. Most of the probe will be extended, which is ideal for sampling liquids. Keep your thumb on the locking screw when holding and sampling with this holder to prevent sudden unlocking that retracts the probe quickly into the holder.

Insert the end of the RTP into the liquid sample. The probe should be inserted no more than 5 mm into a liquid (ideally 1 to 3 mm insertion depth). With every millimeter the probe is inserted into the liquid, the sample will take longer to evaporate from the probe, and could possibly overload the column or mass spectrometer. Samples with very high concentrations (1,000+ ppm) can also overload the MS. The best practice is to sample a small amount of any sample, even liquids. Alternatively, a syringe or micropipette can be used to measure a small volume (1 µL) to load at the probe tip.

Remove the probe from the liquid, and allow the solvent to evaporate while holding the probe at a downward angle to prevent liquid from moving up the length of the probe to avoid probe holder contamination. Depending on the volatility of the solvent, it may take more than 30 to 60 seconds for the solvent to evaporate. For example, polar (water, methanol) or viscous (toluene) solvents will take longer to dry, generally >30 seconds. Retracting the probe into the probe holder before the solvent is fully evaporated can cause contamination of the probe holder and sample carryover. A user sampling oils may clean the extra residue from the probe with a suitable wipe to ensure that the loading is controlled.

When the sample is fully evaporated, retract the probe inside the holder to protect the probe and the sample. When ready, align the probe holder with the inlet, and completely insert its tip to a stop. Depress the plunger completely, and press the **Start** button. Hold the plunger and probe holder in the inlet for ~5 to 8 seconds to vaporize the sample, then retract the plunger and remove the probe holder. The **Start** button light on the QuickProbe will become solid green when ready for injection. During the injection time, the light will be out (no color) and will return to blinking when the probe should be removed (end of injection time). For sampling with the glass probes, this injection time should be set to ~five seconds. All samples discussed in this White Paper had injection times of five seconds; Table 3 lists instrument conditions.

Table 3. GC and MSD instrument conditions for round-tip and pocket glass probes.

Parameter	Value
QuickProbe Inlet	250 °C; split mode
Probe Insertion Time	5 seconds
QuickProbe Column Temperature Program	50 °C (2 seconds), 7 °C/min to 310 °C (0 or 21 seconds)
Carrier Gas Pressure	Helium, 15 psi
GC Oven Temperature	280 °C
Transfer Line Temperature	280 °C
Ion Source Temperature	230 °C
Quadrupole Temperature	150 °C
Scan	*m/z* 40 to 550
Gain Factor	1
Threshold	50
A/D samples	1

Tablet, plant material, and other scrapable material sampling with RTPs

When sampling tablets or plant material, use round-tip probes. The user may want to split the tablet, caplet, or pill in half, as these samples may have a coating on the exterior. Depress the plunger to the position 1 notch, turn the screw into the notch, and hold a finger against the screw to prevent sudden movement of the plunger.

Holding the probe holder, apply some force as you scrape the pill to transfer some sample onto the probe. One to three scraping strokes should provide enough loading onto the probe. There should not be a large amount of solid on the tip of the probe, as this may overload the GC/MS system. You may or may not see a small amount of solid material on the tip of the probe, which is expected. Figure 4 shows an appropriate amount of tablet solids on the tip of an RTP. If there is a large amount of solid on the probe tip, extend the probe to the lower notch to expose more of the probe. Place a lint-free dry wipe around the probe and gently slide the wipe down the probe and past the end to remove some of the solid material, or tap the probe on a weight boat to dislodge some of the solid sample. There should be less (or possibly no) solid sample visible to the naked eye. Retraction into the probe holder and injection into the QuickProbe system is the same as liquid sampling.

Pocket probes

The pocket probes are best for powder or tablet samples (Figure 5). The cupped design will hold powder in the concave surface and provide more protection of the sample than the round tip probe, as shown in Figure 4. Similar to the RTP, only a small amount of sample in the pocket or on the end of the probe is needed to chromatographically separate and detect the compounds in the powder or tablet sample. Examples of powder and tablet samples and the related chromatograms are discussed later.

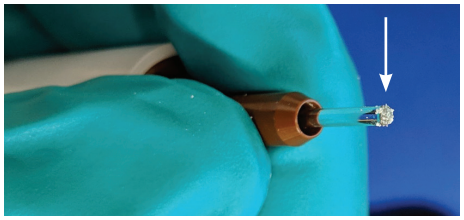

Figure 5. A pocket probe with powder sample on the tip.

Place the powder sample in a weigh boat, if possible. Extend the probe to position 1 on the probe holder, and turn the plunger into the notch. Hold the probe vertically, and gently tap the probe tip into the powder once, taking care to avoid powder transferring to the sides of the probe. Then, tap the probe on the side of the weight boat to remove loosely held powder; the goal is to avoid carryover or contamination of the probe holder and inlet. Figure 5 shows an example of how much powder should be on the end of the pocket probe to effectively detect the compounds in the powder, while also avoiding carryover or contamination issues.

If the powder must be sampled *in situ* (for example, in the bag or container in which the powder arrived), take care to avoid touching the container with the sides of the glass probe. Powder on the sides of the glass probe can cause contamination of the probe holder and sample carryover.

Tablets can be sampled in the same manner as round-tip probes, where the probe is extended to the first notch of the holder.

TSP vial and specialized TSP vial holder

The TSP vial can be used to insert solid samples with volatile or semivolatile compounds, which may be in or on the surface of these solids. Powders or crushed tablets could be tested with the TSP vial (Figure 6A). For example, a user may want to test plant material for cannabinoids, or phthalates in plastic products. In the case of plastics, a small piece of plastic cut from the product, or a ground plastic sample, can be placed in the TSP vial. The TSP vial is small and can be challenging to load with sample. For the TSP vial and holder, the user should collect holder blanks, then insert the vial and collect vial blanks. The vial must be removed from the holder to add sample. The best practice for loading the TSP vial involves using a small spatula or pair of tweezers to drop the sample into the bottom of the vial. If transferring samples from a glass or plastic container to the TSP vial, the material can gather a static charge, and may be difficult to drop into the TSP vial, and some material may cling to the upper sides or exterior of the vial. To prevent carryover or contamination, tap the TSP vial on a table top or other surface to collect the sample in the bottom of the vial, then wipe the exterior of the TSP vial thoroughly with a lint-free wipe. The TSP vial is then inserted into the vertically held TSP vial holder with the open end of the vial facing up to prevent material from falling out of the vial and holder (Figure 6B). The vial is then slid down to the bottom of the holder, shown in Figures 6B and 6C. For injection, Figure 6D illustrates how to hold the TSP vial holder for insertion into the QuickProbe inlet. The holder should be held horizontally with the concave section loaded with the vial facing up to prevent material or the vial from falling out of the holder. The holder end has an arrow to demonstrate the orientation for insertion. Arrow up means the concave part positioned up is the correct orientation.

TSP vial samples may require a longer injection time. For example, plastic samples may require more time in the hot inlet to drive off phthalates and other components in the sample. The TSP vial (with sample) should be held in the inlet for ~10 to 45 seconds during the start of the run, depending on the sample type. The samples discussed in this White Paper had injection times of 10 seconds. Table 4 presents the instrument conditions for the TSP vial experiments.

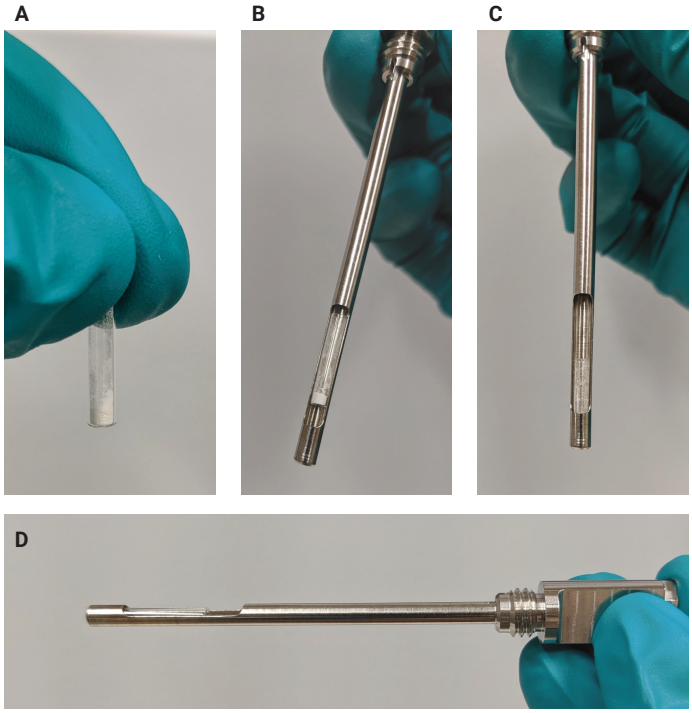

Figure 6. A) A loaded TSP vial with a powder sample that has been tapped on a surface to collect powder at the bottom of the vial; B) loading the vial into a vertically held TSP vial holder with the open end facing up; C) the TSP vial slid to the bottom of the holder and ready for injection; D) holding the TSP vial holder horizontally for insertion into the Agilent QuickProbe inlet.

Table 4. GC and MSD instrument conditions for TSP vial samples.

Parameter	Value
QuickProbe Inlet	250 °C; split mode
Probe Insertion Time	10 seconds
QuickProbe Column Temperature Program	35 °C (10 seconds), 10 °C/min to 340 °C (10 seconds)
Carrier Gas Pressure	Helium, 15 psi
GC Oven Temperature	280 °C
Transfer Line Temperature	280 °C
Ion Source Temperature	230 °C
Quadrupole Temperature	150 °C
Scan	*m/z* 40 to 550
Gain Factor	1
Threshold	50
A/D samples	1

Instrument conditions

Two sets of instrument conditions were used in testing the QuickProbe GC/MS system and consumables. Table 3 summarizes the method parameters when using glass probes, round-tip or pocket, with samples. Table 4 summarizes the TSP vial method parameters. Based on the TSP vial design and its use with testing plastics, the injection time is longer to allow volatilization of compounds of interest.

Suggested workflow for sampling

1. Run a system blank.
2. Run a probe blank.
3. Run the sample.
4. Run a blank.
5. Run a standard, if necessary.

The following are notes for each step:

1. System blanks are completed at the beginning of the sequence or day, like a normal GC/MS blank.
2. A probe is installed into the probe holder, then inserted into the inlet. The glass (round-tip or pocket) probes are held in the inlet for ~five seconds, the typical injection time for this system, then removed.
3. When probe blanks are completed, collect the sample with the probe in the probe holder. Round-tip probes work best with liquid and tablets, and pocket probes are used for powders and scraping tablets. TSP vials can be used for solid samples, such as plant material or plastics. Complete a run with the sample on the probe with the same procedure as 2. Depending on the sample in the TSP vial and probe, the probe insertion ("injection") time may be increased to 10 or up to 45 seconds for effective heating and transfer of the sample into the GC/MS system. For example, a plastic sample being tested for phthalates would require longer insertion time in the inlet.
4. Run a system blank to verify that there is no carryover or inlet contamination. A user may run a probe holder blank to verify that there is no contamination on the probe holder tip.

This workflow was used to test the efficacy of round-tip and pocket probes with various sample types such as liquids, tablets, and powders, and the TSP vial with plant material, thin plastics, and powders. Agilent MassHunter Data Acquisition software was used to collect data. The data were analyzed with Agilent MassHunter Unknowns Analysis, using deconvolution and library matching features. Mass spectra of the deconvoluted peaks were compared to the NIST14 library in the data analysis software with a library match cutoff of 70. Agilent ChemStation Enhanced Data Analysis can also be used with the NIST libraries to match MS peaks to their respective compounds.

Results and discussion: Using the sampling workflow and probes for liquid, powder, and tablet sampling

System blanks

System blanks should be run at the beginning of a data series, such as the beginning of the day, like most GC/MS systems. This system blank can be repeated two to five times to verify a flat baseline. Typically, the baseline is flat by the second run. Figure 7 shows the total ion chromatograms (TICs) for the first three system blanks of a day. To verify that the system was settled, the system data collection method was loaded and allowed to sit for approximately 30 minutes at these method parameters with elevated temperatures. Figure 7A has an overlay of the three system blanks (blank 1 is black, blank 2 is blue, and blank 3 is red), where the first run has a large *bis*(2-ethylhexyl)phthalate peak. Blanks 2 and 3 are near the baseline. Figure 7B is the zoom-in of the baseline region, which better highlights these blank runs. After the initial system blank, the baseline flattens out quickly with only a small *bis*(2-ethylhexyl) phthalate peak.

Probe blanks

After system blanks are completed, a probe should be installed into the holder and inserted into the QuickProbe system to blank the probes. Probe blanks can be repeated two to three times to verify a flat or very low baseline. Typically, contamination is removed within one to three runs. Figure 8 shows a representative set of probe blank runs.

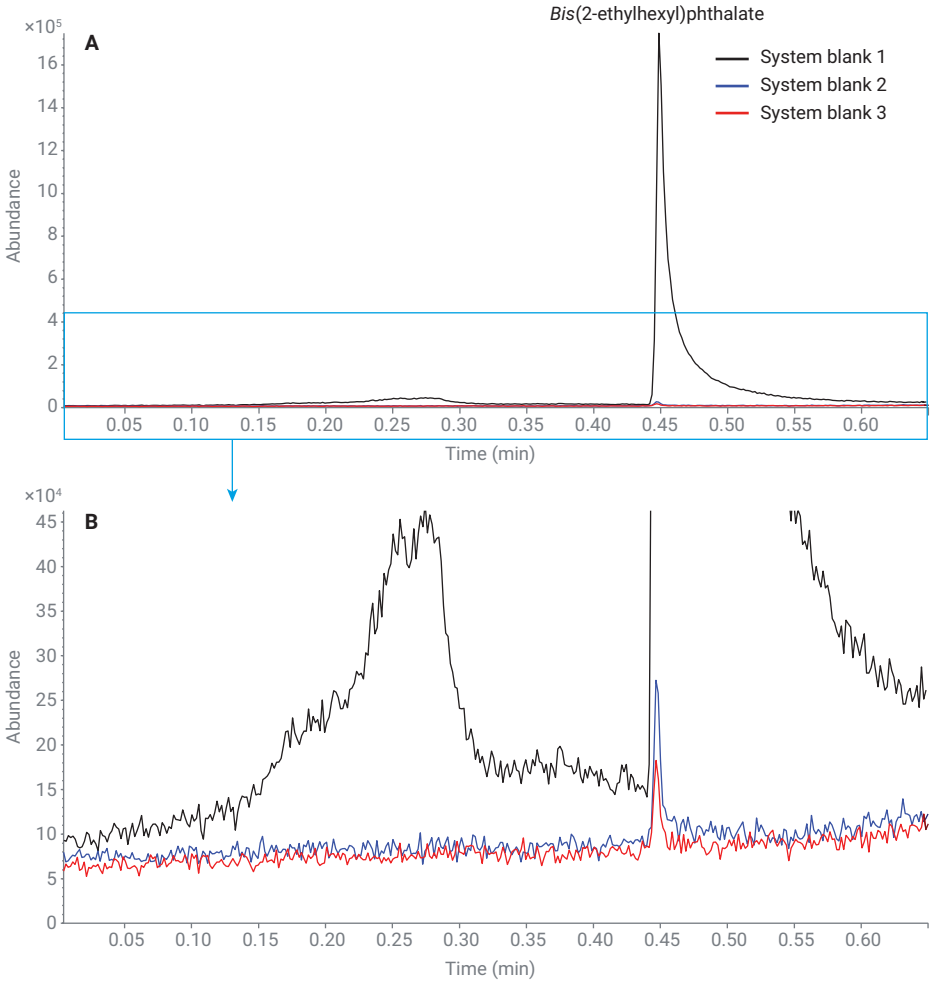

Figure 7. A) System blanks 1 (black), 2 (blue), and 3 (red) overlaid to show the blanks after initial startup of the system; B) zoom-in of the baseline region where blanks 2 and 3 can be better observed.

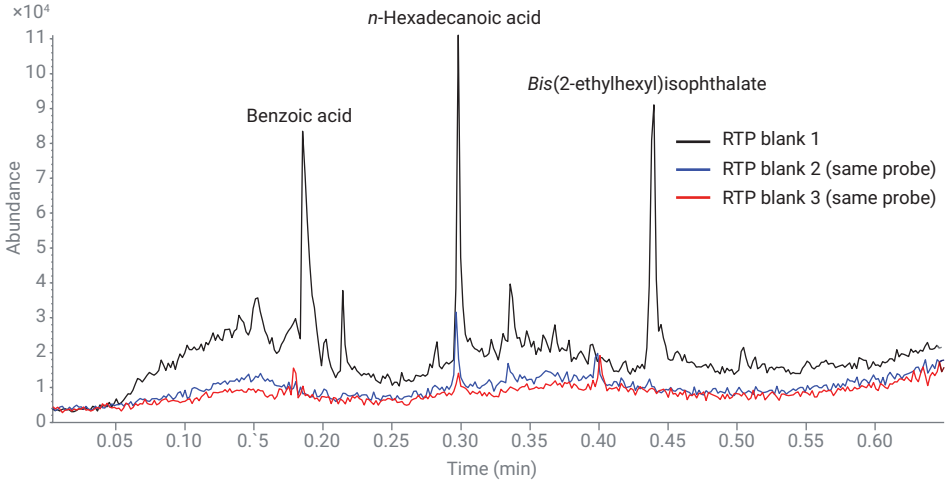

Figure 8. Repeated blanks of a round tip probe (RTP) to show drop in background with each subsequent blank run: blank run 1 in black, blank run 2 in blue, and blank run 3 in red.

It has been common to observe palmitic acid (*n*-hexadecanoic acid), stearic acid (octadecanoic acid), and some phthalates, such as *bis*(2-ethylhexyl) isophthalate, in the first probe blank, as shown in Figure 8. An RTP was inserted into the probe holder, and a probe blank run was completed on the QuickProbe instrument followed by two more blank runs. The first run has measurable levels of palmitic acid, benzoic acid, and *bis*(2-ethylhexyl)isophthalate. From the first blank to the second blank (blue trace), there is a dramatic decrease in the background profile, nearly a factor of 10. In the third blank run of the same probe (red trace), the peaks are smaller yet. After approximately three to five blank runs of the same probe, the background profile is stable and tends not to lower significantly. After three blank runs, generally the fatty acid and other background peaks are within the baseline level and are less noticeable or hidden by compounds in the real samples. PTEG packaging helps to lower the background contamination, but minor background contamination can still occur as the probes are exposed to the plastic packaging and the environment.

If concerned about repetitive probe blanks, the probes can be cleaned before use by rinsing with a polar solvent, followed by a nonpolar solvent, then placed in a drying oven for five hours at 500 °C.

Liquid sampling with RTPs

Liquid mixtures that contained compounds of the same class type, such as opioids, were used to test the ability of the QuickProbe system to separate and identify closely eluting compounds.

An amines mixture in methanol containing amphetamine, phentermine, methamphetamine, MDA, MDMA, and MDEA at 250 µg/mL was sampled with an RTP. Since methanol is a polar solvent, it took more time to dry on the end of the RTP. In typical GC/MS analysis, these peaks tend to elute early and can be sensitive to inlet and column parameters. Figure 9 illustrates the ability of the system to separate these early eluting compounds. All six amine class compounds were identified with library match scores greater than 88. The peaks were not fully baseline resolved, but the data analysis software was still able to identify each compound correctly with high match scores.

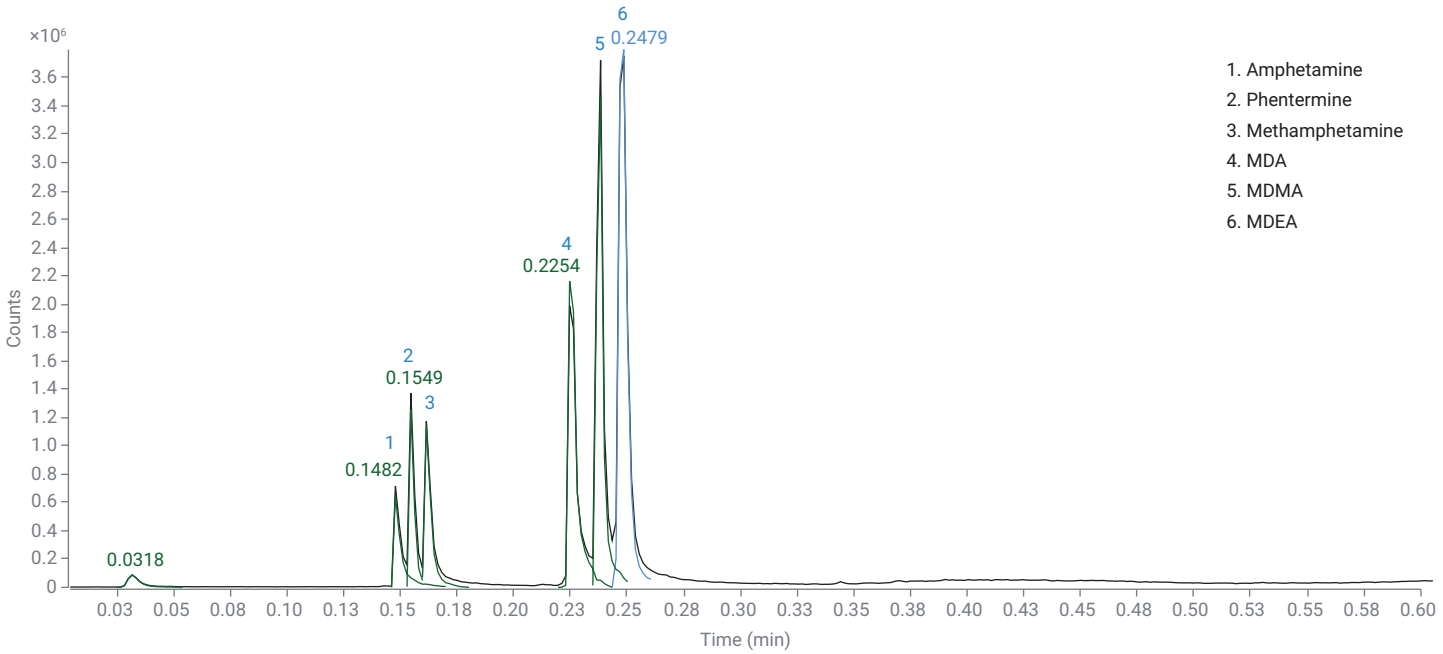

Figure 9. TIC of an amines mixture at 250 µg/mL in methanol, sampled with an RTP.

The opioid compound class was also tested on the QuickProbe system as later eluting, but difficult, compounds, because certain opioids are known to tail in normal GC/MS systems with longer runs times. In the QuickProbe GC/MS system and an analysis time of <one minute, all five opioids were separated and identified (Figure 10). Like the amines class, not all opioids are baseline resolved under the oven parameters; however, all compounds were successfully identified with library match scores above 95. Additionally, the fast analysis method retained excellent peak shape for the opioid compounds, including hydrocodone and oxycodone. If these compounds were required to have baseline resolution, the QuickProbe parameters could be altered for greater separation.

Aerosol (liquids in spray bottles) sampling

Aerosol spray bottle contents can be tested with probes, whether the probe is dipped into the liquid, the contents sprayed onto the probe, or the contents sprayed onto a surface, such as a plastic weight boat, then touched with a probe. A spray bottle containing nitroglycerin was discharged onto the round tip probe, allowed to dry, and tested in the system (Figure 11). Spraying an aerosol can leave many droplets on and along a long section of the probe, and can take a long time to dry. The nitroglycerin spray had an oily consistency and required >one minute to dry on the probe. Caution must be taken with aerosols to avoid carryover in the probe holder and the overall QuickProbe GC/MS system, such as the liner or column.

This nitroglycerin spray sample had a very complex chromatogram compared to some of the previously discussed mixtures. Unknowns Analysis data analysis software was used to deconvolute the data and compare mass spectra to the NIST14 library mass spectra with a match score cut-off of 70. The identified compounds included eucalyptol, levomenthol, menthol, 1,3-dicaprin, 1-dioctanoin, glycerols, and organic ethyl ester acidic compounds such as ethyl ester octanoic acid and *n*-caprylic acid isobutyl ester.

Carryover can occur in liquid samples such as aerosols, depending on the concentration of the compounds in the liquid sample or how the user samples the aerosol. When spraying the aerosol directly onto the probe, it can be difficult to control the area the spray reaches. To mitigate this, the user can spray the aerosol into a plastic weigh boat or a similar surface, then touch the probe to that surface. This action could also lower the time of evaporation for viscous samples on the probe, since less sample will be on the tip and sides of the probe. Ideally, there would be no sample up the sides of the probe, if the aerosol was first sprayed onto a weigh boat or another sampling medium.

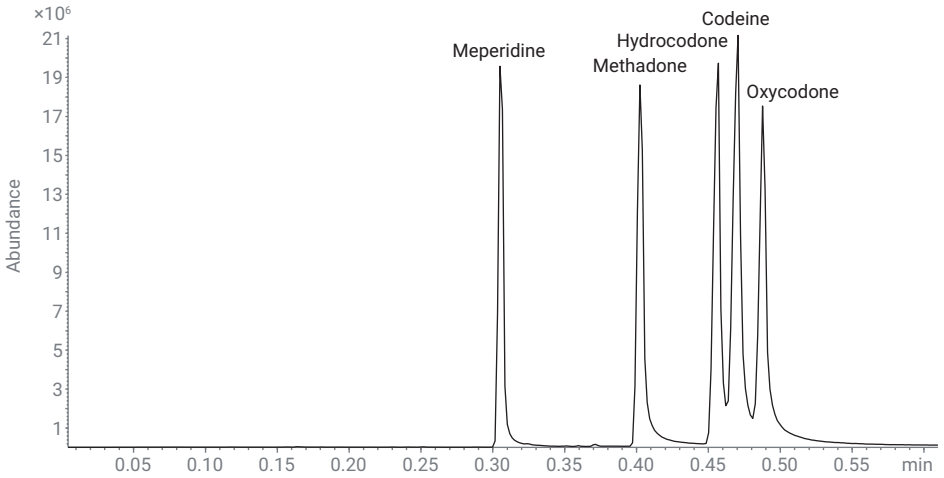

Figure 10. Agilent QuickProbe GC/MS TIC showing fast separation of opioid compounds (250 µg/mL) from a liquid sample (methanol) using an RTP.

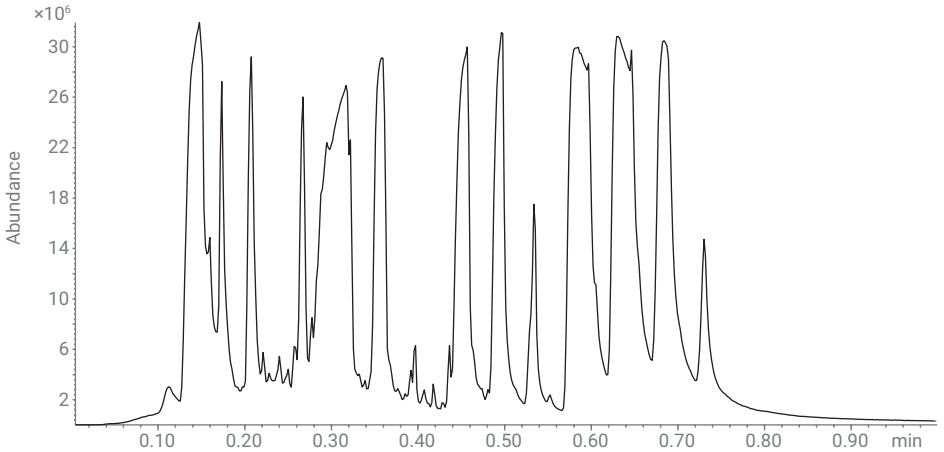

Figure 11. TIC of a nitroglycerin aerosol spray onto an RTP, where most peaks correspond to esters and alcohols.

Figure 12 illustrates the results of spraying the aerosol onto the extended probe (while in the probe holder). The black trace represents the probe holder blank after the nitroglycerin spray in which several compounds, such as glycerols, have high responses, indicating that the probe holder or system was contaminated. The next run was a system blank, shown in red, which has lower abundances, but still retains some of the glycerol contaminant peaks; this result indicates that the system, probably the liner, was contaminated. This result does not rule out the possibility that the probe holder tip was also contaminated; therefore, the probe holder tip was replaced with a new tip as the system cooled. If the probe holder tip has become contaminated, the tip can be removed, rinsed with methanol or acetone and, when dry, placed in an oven at ~80 °C for 15 to 30 minutes to remove the contamination. When the system was at ambient temperatures for user safety, a new liner was installed. The system was held for ~15 minutes at the lower temperatures to flush out any air from the inlet area; then, temperatures were increased to test for any additional contamination. The blue trace in Figure 12 illustrates the results of a system blank after a new liner was installed, where there are no remaining glycerol or other contaminant peaks.

Less viscous liquid samples can also exhibit carryover, specifically if the compounds are in high concentrations or solvated in polar liquids such as water. Water will take a significant amount of time to dry; if the probe is not dry before retracting into the holder, the probe holder tip can become contaminated. Also, when concerned about carryover, the best practice is to analyze the sample runs and blanks immediately to test for carryover. A liquid diphenhydramine sample (50 mg/mL in saline solution) was procured to test a high concentration sample solvated in water. When the diphenhydramine liquid was sampled with an RTP, the solvent took a long amount of time to evaporate (~60+ seconds). Figure 13A contains the TIC of the diphenhydramine sample, where diphenhydramine is the very large, overloaded peak from 0.35 to 0.44 minutes. After the RTP was removed from the probe holder and discarded, a probe holder blank was completed because the sample was high in concentration and solvated in saline solution (Figure 13B). At the time, the samples were not immediately reviewed and the carryover of diphenhydramine at 0.35 minutes was not identified, as shown in Figure 13B. Carryover continued into the next blank runs for a newly installed RTP (Figures 13C and 13D). At this point, the carryover of diphenhydramine was decreasing with more blank injections, but the probe holder tip was removed and a new one was installed to save time and number of repetitive blank injections. The contaminated probe holder tip was rinsed with methanol and dried in an oven at 80 °C for approximately one hour. If the carryover was noticed immediately during the probe holder blank (Figure 13B), additional time could

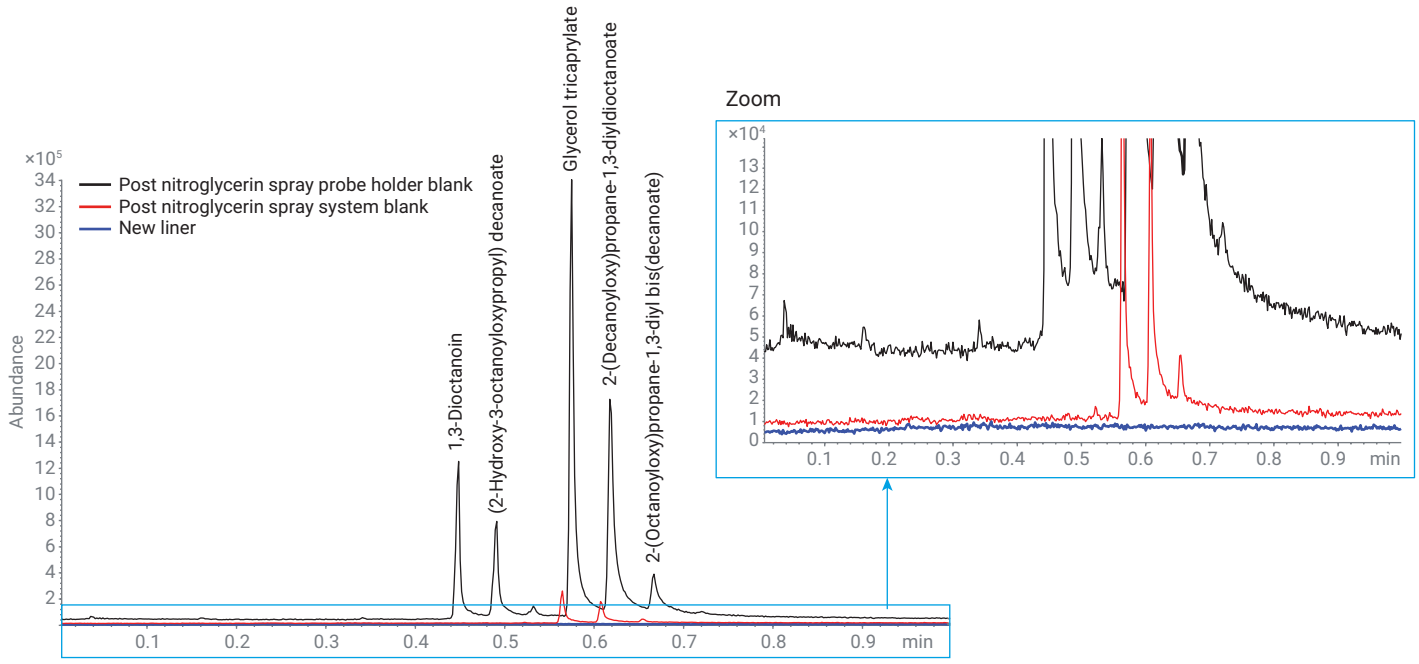

Figure 12. Carryover from nitroglycerin spray in blank chromatograms and clean system blank with new liner; inset: zoom-in of baselines.

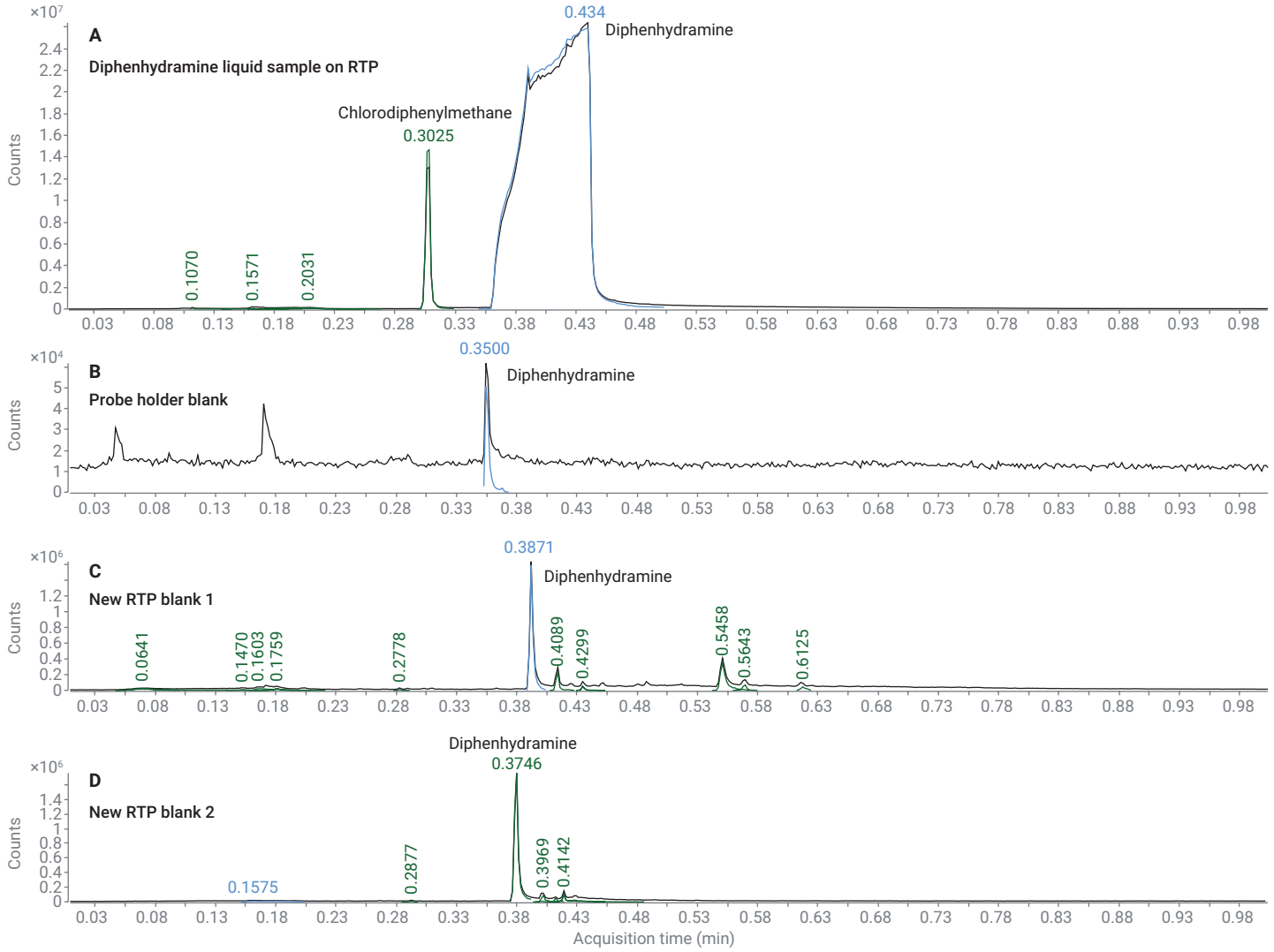

Figure 13. A) TIC of a diphenhydramine liquid sample (50 mg/mL in saline solution) on an RTP; B) probe holder blank TIC after removal of the diphenhydramine RTP; C) TIC of the first blank run for a new RTP installed into the probe holder; D) TIC of the second blank run for the same RTP installed into the probe holder.

have been saved by running a system blank immediately after the probe holder blank to determine if the system or probe holder were contaminated. If the system blank showed no carryover, the probe holder tip could have been replaced earlier; however, with a run time of one minute, only four minutes were lost, when accounting for runtime and the time between runs. The best practice is to run a set of probe holder and system blanks after a sample to verify a clean system and probe holder.

Tablet scraping with an RTP

Sampling both the interior and exterior of a tablet can provide information about tablet components, handling, and surrounding environment. Some tablets have a sugar or an inactive ingredient exterior coating, which requires the tablet to be cut to expose the interior for accurate analysis of the active components. In some cases, scraping the exterior surface with some applied force on the glass probe can expose the interior ingredients.

One such example of exterior coated tablets is ibuprofen. The exterior of an ibuprofen tablet was gently scraped with a round tip probe (three to five light scrapes with tip of the probe) and tested on the QuickProbe instrument. Next, a new RTP was installed, blanked, and the exterior of the same tablet, in a different spot, was scraped with applied force (~five scrapes), which tore away the red-colored exterior coating, and exposed the interior in a small section, as evidenced by the observation of white substance. Third, the tablet was cut in half; a new

RTP was installed and blanked, then the exposed interior of the ibuprofen tablet was scraped with the probe. The respective TICs were overlaid to compare the results of different sampling forces and locations (Figure 14). A significant peak was identified in the interior and forceful exterior scrapings TIC as ibuprofen, with match scores of 98 for both chromatograms. The gentle exterior scrape did not show a peak corresponding to ibuprofen.

Sampling the exterior of a tablet can provide information about the environment to which the tablet was exposed. A lorazepam tablet was procured. The exterior of the tablet, like the ibuprofen tablet, was scraped once with a new RTP, and tested. Figure 15 illustrates what was found on the exterior of the lorazepam tablet. Lorazepam was identified at 0.45 minutes with a match score of 84, as were aspirin, ibuprofen, and acetaminophen with match scores of 84, 83, and 97, respectively, indicating that the lorazepam was stored in close proximity to these compounds. The palmitic acid, stearic acid, and oleamide are common fatty acids that likely came from human handling of the tablets.

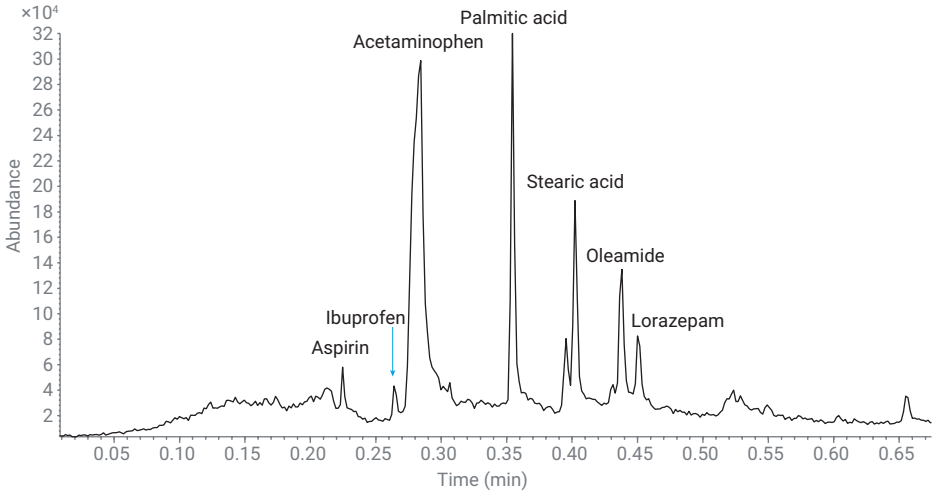

Figure 15. TIC of scrape from the exterior of a lorazepam tablet with an RTP.

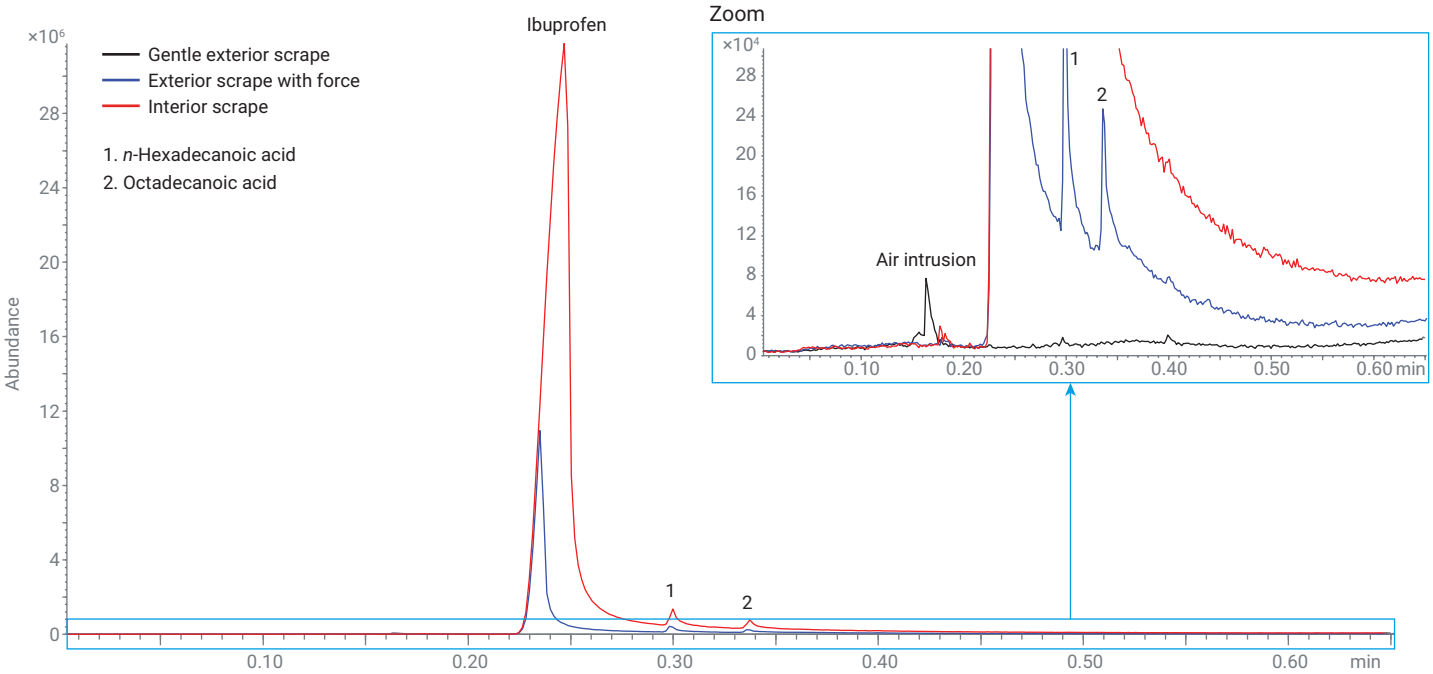

Figure 14. Comparison of TICs for a gently scraped exterior of ibuprofen tablet (black), an ibuprofen scraped with enough force to remove the exterior coating (blue), and scrape of the ibuprofen tablet interior after being cut in half. The zoom-in region (inset) shows the scale of the TIC for the gently scraped exterior compared to the other two TICs. All three samples used different round tip probes.

The lorazepam tablet was cut in half, and the interior was scraped with a new RTP. Figure 16A illustrates what was found on the interior of the lorazepam tablet. Lorazepam was again identified at 0.45 minutes and confirmed by MS (Figure 16B), which matched with a score of 98 to the NIST14 spectrum. For the interior scrape, there are small peaks attributed to ibuprofen, acetaminophen, stearic acid, and palmitic acid, which were likely transferred into the cut from the cutting tool or when handling the tablet to scrape the interior. The peaks are unlikely due to carryover, since the system blank and probe blank were clear of these compounds. If the observation of these compounds in a TIC for an interior tablet scrape is concerning to an analysis, the user could hold the tablet in a lint-free wipe to avoid transfer onto gloves.

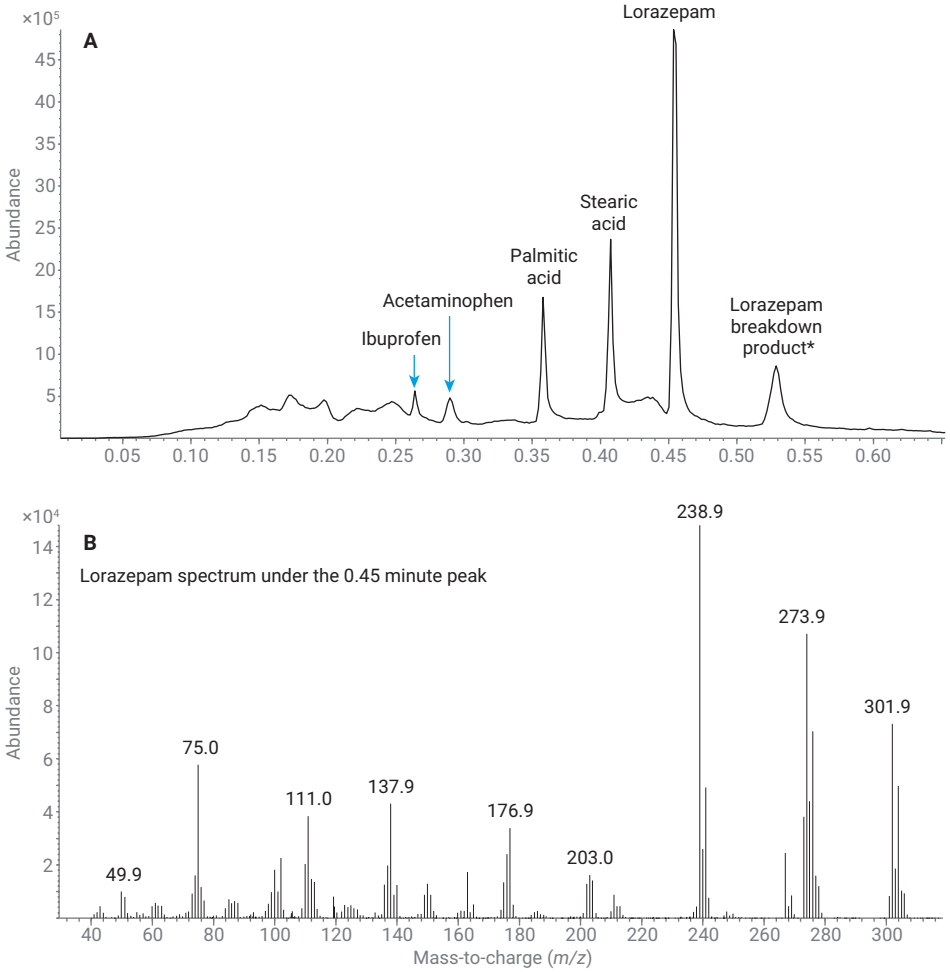

Figure 16. TIC (A) and mass spectrum (B) of lorazepam tablet cut in half, then scraped with an RTP.

A third example of comparing exterior and interior tablet scrapings with an RTP was completed using a hydrocodone/ibuprofen tablet. As with the previous examples, an RTP was scraped across this tablet one to three times to gather material onto the tip, then inserted into the QuickProbe GC/MS system. Then, a new probe was installed into the probe holder and probe blanks were completed while the hydrocodone/ibuprofen tablet was cut in half. The new RTP was then scraped along the open interior of the tablet, and a run was completed for the sample. Figure 17 contains TIC overlays of the exterior scrape (blue) and the interior scrape of the same tablet (black). The black trace (tablet interior) has a very strong ibuprofen peak and smaller, but easily identifiable, hydrocodone peak. The ibuprofen peak is close to overloading the detector with an abundance of >10^6 abundance units, and exists at a significantly higher concentration than hydrocodone. The zoom-in region box best highlights the tablet exterior components of acetaminophen, caffeine, fatty acids, and a very small ibuprofen peak at 0.264 minutes (just before acetaminophen), confirming that the hydrocodone/ibuprofen tablet has a coating on the exterior and requires scoring or a cut to determine the tablet components. The compounds identified on the exterior reflect the environment in which the tablet was kept.

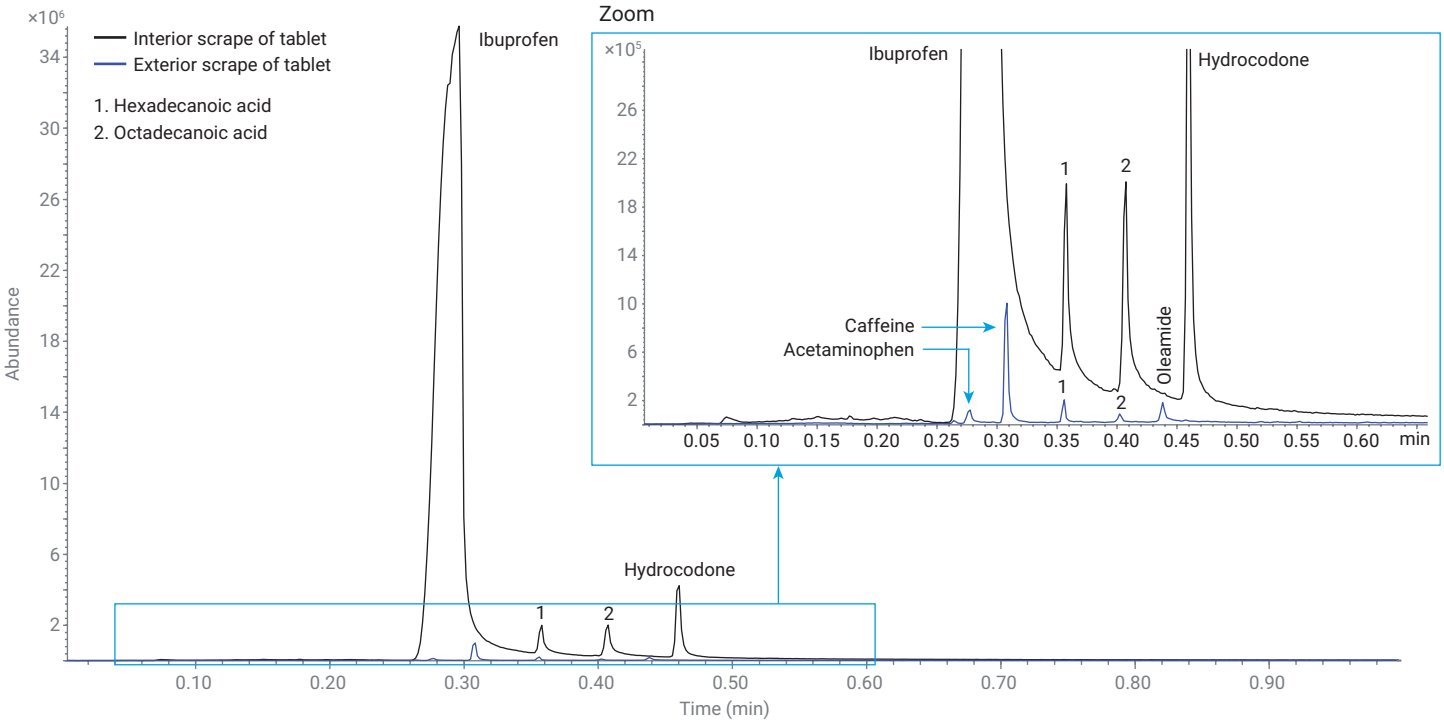

Figure 17. Comparison of TICs for an exterior scrape (blue trace) of a hydrocodone/ibuprofen tablet, and scrape of the tablet interior (black trace) when cut in half.

Ointments/creams

Ointments or creams, such as an anti-itch cream, can be tested with the QuickProbe system. A small amount of diphenhydramine cream was dispensed into a weight boat. An RTP tip was extended to position 1 and gently dipped into the ointment. The glass probe was then wiped down and around the bottom of the tip with a lint-free wipe to remove excess ointment. The TIC in Figure 18 illustrates inactive ingredients of propylene glycol, methyl paraben, 1-hexadecanol, and 1-octadectanol. The active ingredient of diphenhydramine is on the shoulder of 1-hexadecanol, and can be identified with deconvolution. The blue trace in Figure 18 is the extracted ion chromatogram (EIC) of *m/z* 165, and is overlaid onto the TIC to highlight the diphenhydramine peak. Because of the potential for coelution with other compounds when using the fast separation, it is critical to use mass spectral deconvolution software (such as Unknown Analysis) and a large mass spectral library to make data analysis and peak identification easier and faster for the user.

Plant material

Plant materials, such as cannabis leaves, can be sampled by scraping or rubbing the herbaceous material with the tip of the RTP. A ground cannabis sample was rubbed with an RTP (extended to position 1 in the holder). THC was the most intense peak in the chromatogram (Figure 19) with a corresponding high library match score of 99. Also identified were small peaks of terpene compounds and cannabinol with match scores higher than 75.

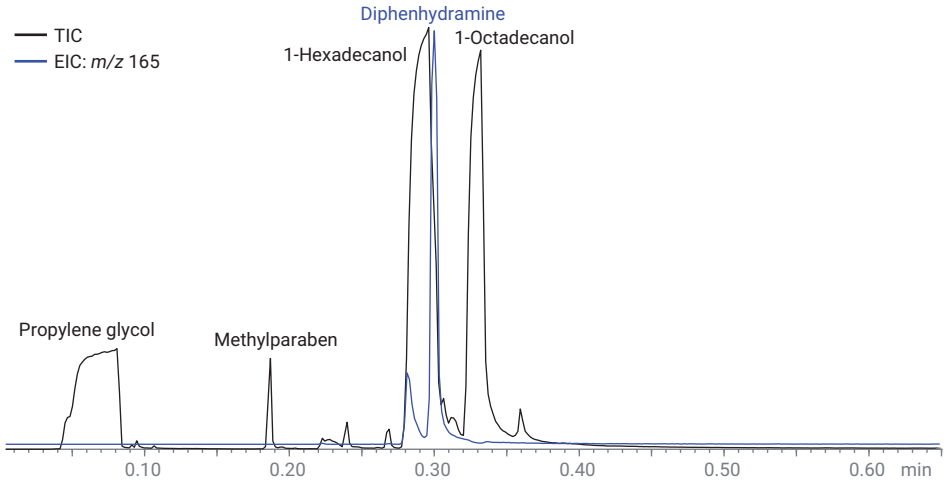

Figure 18. TIC (black trace) and EIC *m/z* 165 (blue trace) of diphenhydramine cream sampled with an RTP.

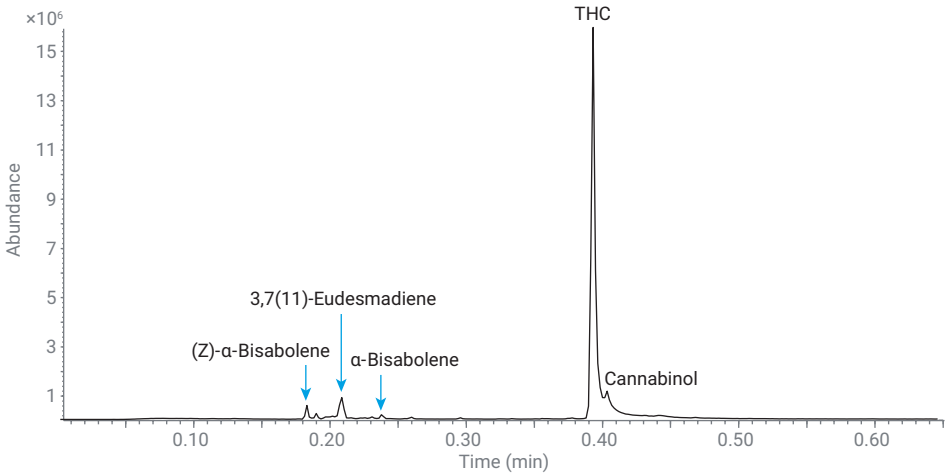

Figure 19. TIC of ground cannabis sample transferred onto an RTP.

Sampling powder and tablets with a pocket probe

A pocket probe can be used with powder and tablet samples. Foot powder was procured to test for ingredients and mimic forensic testing of the powder samples. The powder was placed in a weight boat and tapped gently with the pocket probe, which was already installed in the probe holder and with which a series of blanks had been performed. The pocket probe tip was tapped against the side of the weight boat, to dislodge loose powder and the sides of the probe were wiped with a lint-free wipe (from the probe holder tip down to the pocket tip) to remove any excess. Figure 20 displays the peaks identified from the foot powder sample deconvoluted with Unknowns Analysis software. Eucalyptol, levomenthol, and thymol were identified with library matches scores of 98, 93, and 96, respectively; methyl salicylate was identified with a match score of 81.

To mimic powder samples, individual tablets were crushed in weigh boats to sample with the pocket probe. A tablet containing aspirin, acetaminophen, and caffeine was sampled with a pocket probe. A pocket probe was installed into the probe holder, and blank runs were performed. The crushed tablet powder was gently tapped with the pocket probe, then tapped against the side of the weight boat to dislodge any excess material. There should be a very small amount of material on the pocket probe, similar to what is shown in Figures 5 and 20 (inset). Figure 21 displays the TIC result of a tablet containing acetaminophen, aspirin (acetyl salicylic acid), and caffeine. Many of the peaks in the TIC are overloaded and overlapping, as evidenced by the peak width and non-Gaussian shapes, but an interesting set of compounds and peaks are identified. Salicylic acid, a breakdown product of acetyl salicylic acid, is identified in the TIC, as are aspirin, caffeine, and acetaminophen. Also

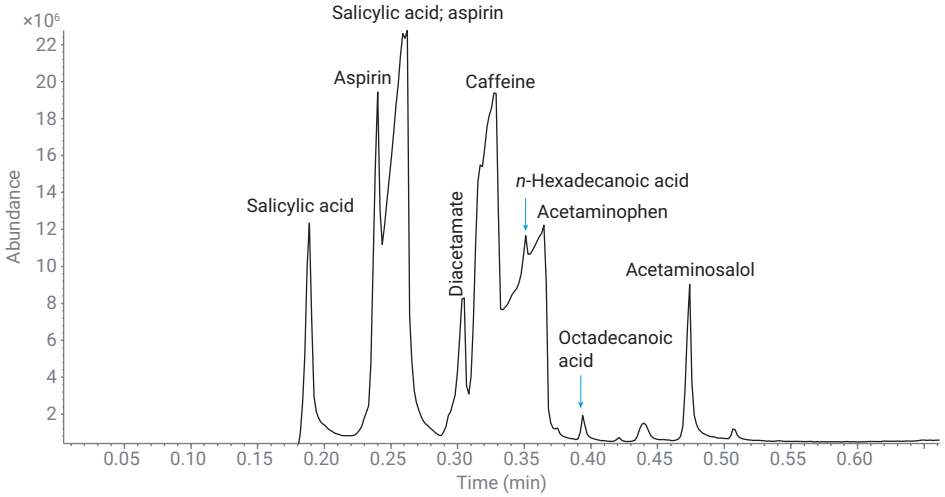

Figure 21. TIC of crushed tablet with main ingredients of acetaminophen, aspirin, and caffeine.

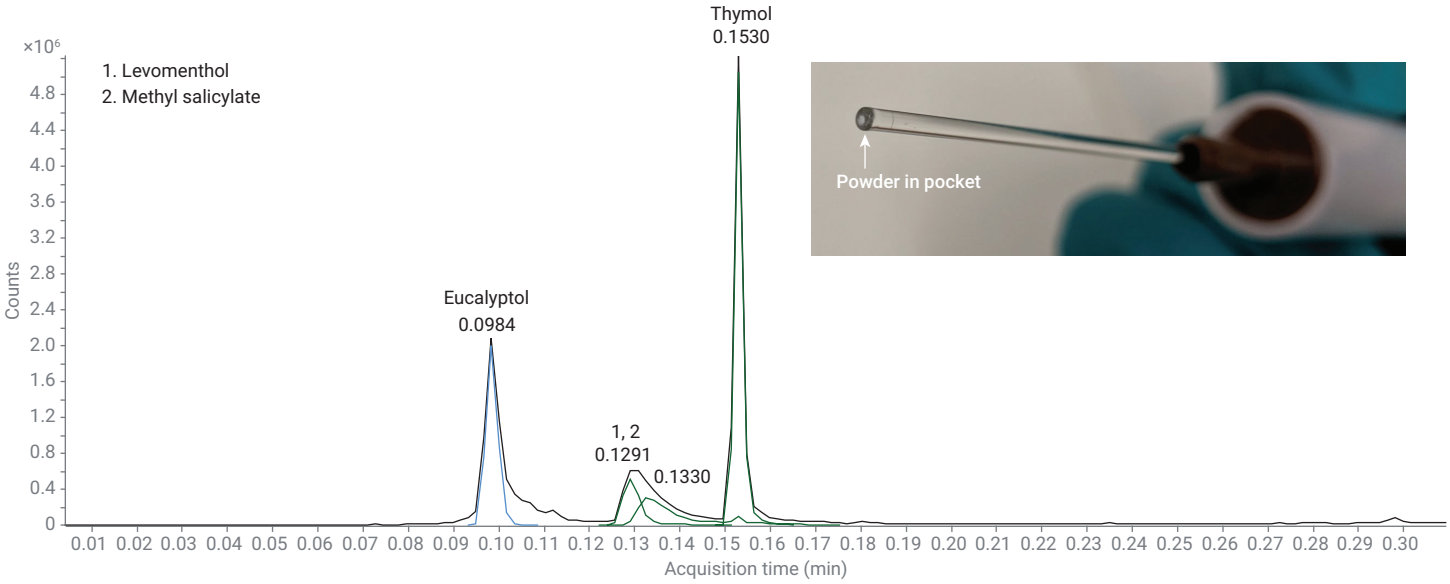

Figure 20. TIC of foot powder with deconvoluted peak shapes and compounds highlighted per identified compound. Inset: picture of the pocket filled with foot powder for a pocket probe installed in a probe holder.

identified are diacetamate, which is an impurity in acetaminophen production, and acetaminosalol, a product from the esterification of paracetamol (acetaminophen) and salicylic acid. The multiple peaks of salicylic acid and aspirin may be related to the different sizes of the crushed tablet powder, or the binding agents were breaking down and releasing these compounds at slightly different times during the injection.

Pocket probes can be used for tablet scraping. For this example, a hydrocodone/acetaminophen tablet was cut in half, then the interior was scraped with the pocket probe. The same process was used for tablet scraping with the pocket probe as with the round-tip probe, where the tablet interior was scraped once, and the pocket end was examined to check for excessive sample amounts. Figure 22 shows the results of a tablet scrape with the pocket probe; there is a very large, overloaded peak corresponding to acetaminophen, and a smaller peak of hydrocodone. The inset takes a closer view of the hydrocodone peak, which is nicely shaped and has an easily distinguishable signal from the baseline. The hydrocodone peak was verified using the mass spectrum (inset) with a library match score of 95.

Part of the acetaminophen/hydrocodone tablet was also crushed to examine the difference in results when scraping the interior of the tablet compared to a powder/crushed sample. The pocket probe was used in both cases. Figure 23 overlays the TICs from these experiments, where the black trace is the powder (crushed tablet) and the blue trace is the scrape from the interior of the tablet. Both acetaminophen and hydrocodone are easily identifiable in both chromatograms. The acetaminophen and hydrocodone peaks are larger in the scraped sample than in the powder sample peaks, but this also means that the acetaminophen is less overloaded in the powder sample, which is better for the QuickProbe column and MS detector. It is probable that there was more material on the pocket probe from the tablet scrape than the probe used to tap the powder, which would account for the peak difference. In both cases, there was no carryover in the system or probe holder blanks, indicating that there was enough material on each probe to identify the components, but not contaminate the system.

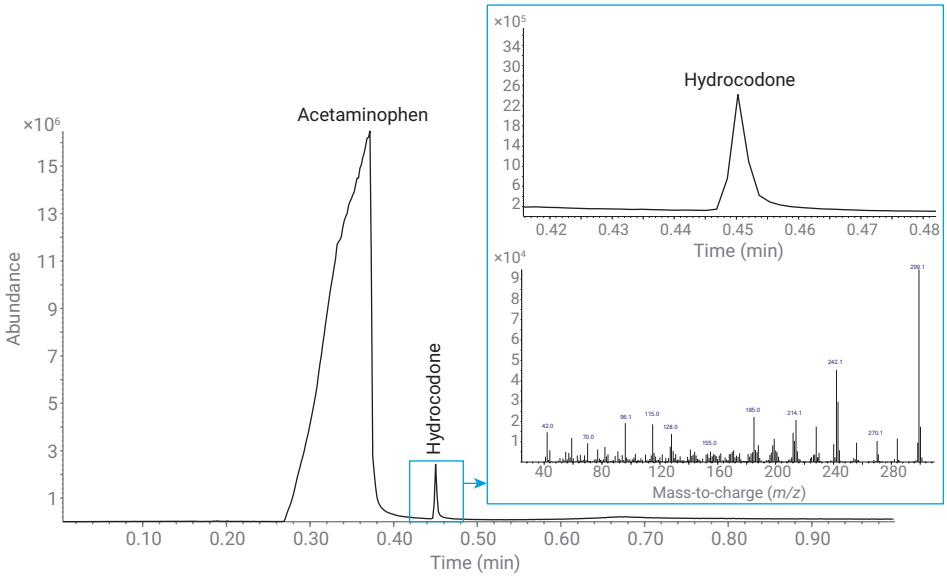

Figure 22. TIC of acetaminophen/hydrocodone tablet (interior) scraping; inset: zoom-in of hydrocodone peak and the corresponding mass spectrum under the peak.

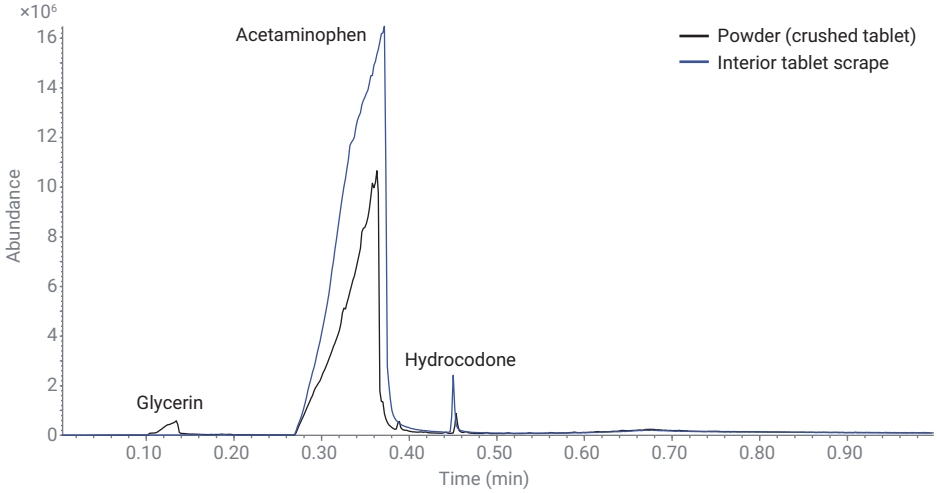

Figure 23. Comparison of the acetaminophen/hydrocodone tablet sampled with pocket probes by scraping the tablet interior (after cutting in half, blue) and crushing part of the tablet into a powder (black).

TSP vials and probe holder

The TSP vials were designed to hold materials that may not be easily sampled with the glass probes, such as plant materials or plastics. TSP vials can also be used for crushed or broken tablets or powders. Based on the design of the TSP vial and holder, the TSP vial method parameters outlined in Table 4 were used with a longer injection time and slower oven rate.

TSP vial holder and vial blanks

Since more of the TSP vial holder is inserted into the inlet than the glass probe holder, it is best to complete a series of TSP holder blanks (no vial) before running a sample or vial blank. As observed in the glass probe work, hexadecanoic acid and octadecanoic acid are the prominent peaks in the TIC for the TSP vial holder (not shown). Once the TSP holder has a low background, install a TSP vial and run a series of blanks. Like the TSP holder, hexadecanoic acid and octadecanoic acid are the prominent peaks in the TIC (Figure 24). After the first blank, the background drops significantly in intensity and slowly settles over four blank runs; Figure 24 compares the vial blank runs to the final blank run (holder blank 5 in gold) to show the cleanliness of the vial and holder after a set of holder and vial blanks.

Plastics

Plastics should be cut, chopped, or ground into small pieces to insert into the TSP vial. A nitrile glove was cut into small strips, and a piece was inserted into the TSP vial with tweezers (after a series of system, holder, and vial blanks were completed). The TSP method parameters were used with a 10 second injection time. Injections times may be increased up to 45 seconds, depending on the consistency of the plastic material, as larger chunks of material may require more time to heat and volatilize compounds of interest, such as phthalates. If injection time is increased, the QuickProbe column hold at the initial temperature should be increased to the same time.

Figure 25 shows that 10 seconds was enough time to volatilize several compounds from the nitrile glove. There is a wide baseline increase that is attributed to dimethylamine followed by a very large peak of 2-methyl-2-undecanethiol. As expected, hexadecanoic acid and octadecanoic acid are identified, along with diethyl phthalate and squalene. A longer injection hold could alter the types of compounds observed, but care should be taken to avoid melting, burning, or otherwise compromising the plastic material, especially if any of the material is contacting the TSP vial holder.

The TSP vial should not be packed with a large amount of material. Less than 10 mg of material can be enough to identify compounds of interest.

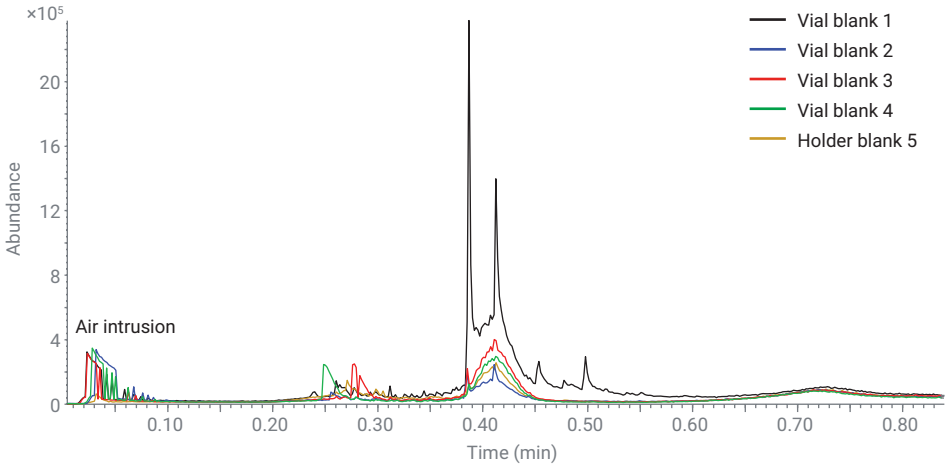

Figure 24. TIC overlays of four blank runs for the TSP vial holder with an installed vial. The gold overlay TIC is from the TSP holder blank run 5.

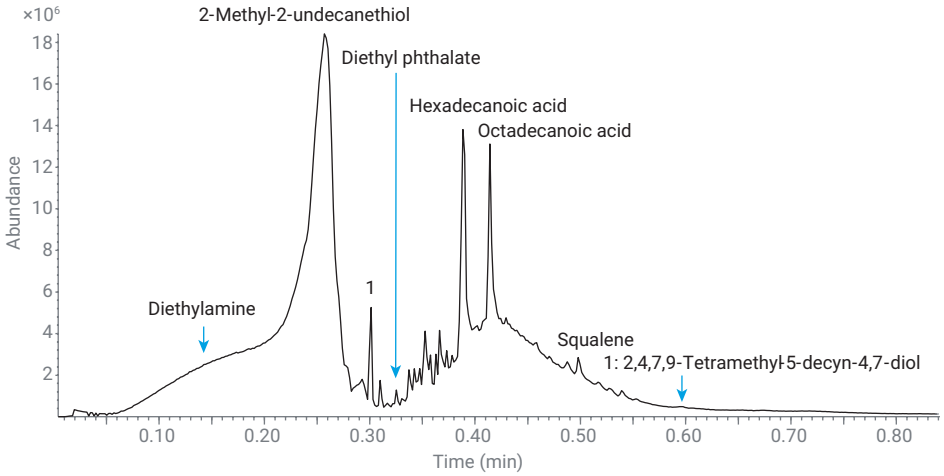

Figure 25. TIC of a slice of nitrile glove sampled with the TSP vial and holder. The sample was held in the inlet for 10 seconds injection time.

Powder

Powders can be tested with a TSP vial or a pocket probe, depending on user preference. Foot powder was procured and placed in a weight boat. Approximately 7 mg of foot powder was transferred into a TSP vial with a spatula. When filling with powder or another sample, it is best to tap the vial on a tabletop to collect the sample at the base of the vial. Approximately 7 mg of fine powder, after collection at the bottom of the vial, has a height of ~2 mm in the TSP vial. There may be some powder on the exterior of the TSP vial from transferring the sample. If allowable by operating procedures, wipe the exterior of the TSP vial with lint-free wipes (dry or slightly wet) to remove any sample and prevent holder contamination. The user may also need to change gloves.

The TSP method parameters were used for testing foot powder with the TSP vial. Several compounds were deconvoluted from the TIC in Unknowns Analysis (Figure 26). A handful of terpene compounds were identified along with menthol, methyl salicylate, and thymol, which are summarized in Table 5. No carryover was observed in the TSP holder blanks after the vial was removed, even though powder had spilled outside of the vial when transferring the powder into the TSP vial. The exterior of the vial was wiped with several lint-free wipes to prevent carryover.

Table 5. Compounds identified in the foot powder sample (shown in Figure 25) with retention times (RT), library match scores (LMS), chemical formulae, and CAS numbers.

RT (min)	Compound	LMS	Formula	CAS Number
0.2161	γ-Terpineol	83.8	$C_{10}H_{18}O$	586-81-2
0.2307	α-Phellandrene	85.3	$C_{10}H_{16}$	99-83-2
0.2382	Terpinolene	87.9	$C_{10}H_{16}$	586-62-9
0.2563	Menthol	95.2	$C_{10}H_{20}O$	1490-04-6
0.2593	Methyl salicylate	97.0	$C_8H_8O_3$	119-36-8
0.2826	Thymol	96.9	$C_{10}H_{14}O$	89-83-8
0.3002	6-Tetradecene	90.8	$C_{14}H_{28}$	41446-64-4
0.3715	(−)-Abietadiene	74.0	$C_{20}H_{32}$	35241-40-8
0.4128	Octadecanoic acid	82.9	$C_{18}H_{36}O_2$	57-11-4
0.4865	4-Methylhept-3-yl octyl ester phthalic acid	79.9	$C_{24}H_{38}O_4$	1000377-94-3
0.4986	Squalene	83.4	$C_{30}H_{50}$	111-02-4

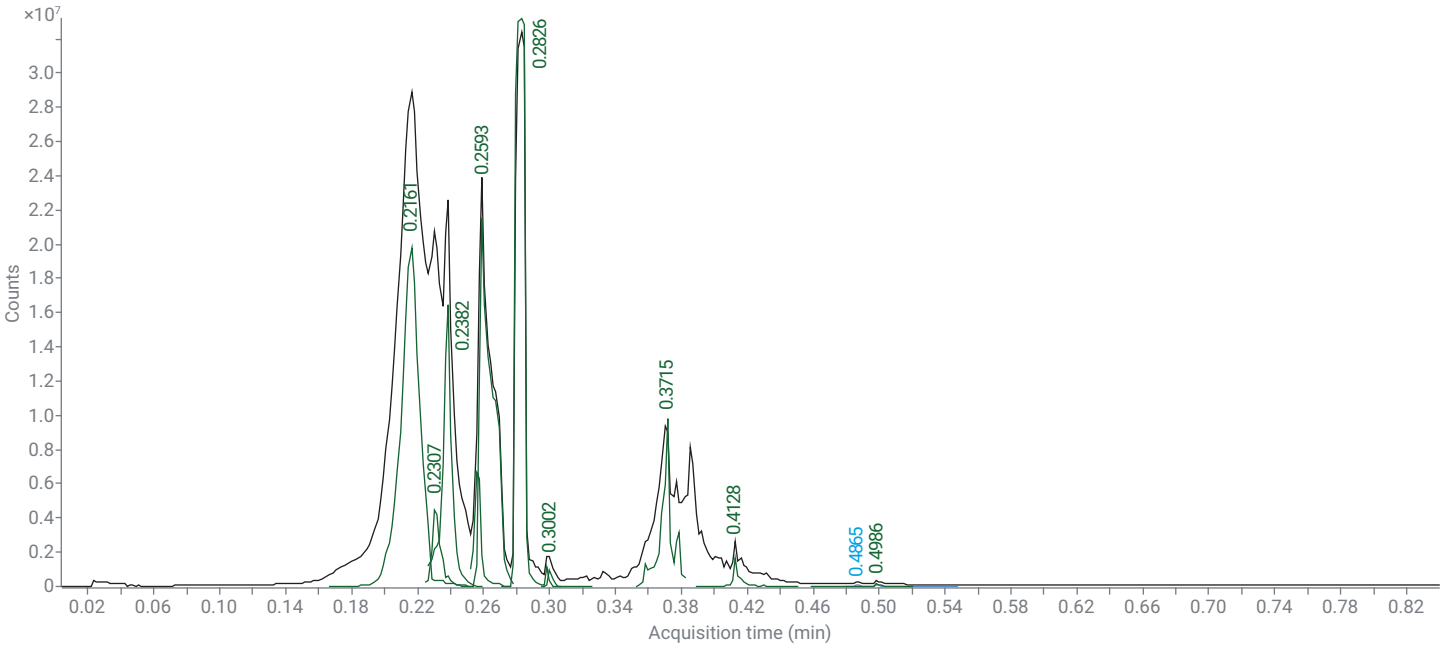

Figure 26. TIC of foot powder sampled with a TSP vial held in the inlet for 10 seconds with EICs per identified peak shown in green.

Plant materials

Plant material testing was discussed in the context of using a round-tip glass probe to scrape the material. Plant material can also be tested with the TSP vial. Before any sample loading, holder and TSP vial blanks should be completed to check for contamination. A small amount of cannabis was dropped into the TSP vial with a small spatula. Transferring a material from a glass or plastic container to the TSP vial can create a static charge and cause material to cling to the exterior of the TSP vial. This also can occur for powder or plastic sampling. The exterior of the TSP vial should be wiped with a lint-free wipe to remove excess material and avoid carryover. The exterior could also be rinsed with a small amount of solvent, such as acetone, to remove the material and allowed to dry before installing the TSP vial into the holder. The TSP method parameters (Table 4) were used for the cannabis testing. Two cannabis samples were tested with the TSP vials.

Cannabis sample 1 was obtained as a coarsely ground sample, which was challenging to load into the TSP vial since the plant material would cling to the exterior of the TSP vial due to a static charge. Approximately 10 mg of cannabis sample was loaded into the TSP vial. The TSP vial was tapped against the tabletop to settle the plant material to the bottom of the vial, shown in Figure 27 (inset), and the exterior was wiped with lint-free wipes to remove any sample before installation into the TSP holder. Cannabis sample 1 has a complex chromatogram (Figure 27) that was deconvoluted in Unknowns Analysis to reveal various terpene compounds, cannabinoids, and THC. Using the TSP vial with the longer injection time and lower starting column temperature allows more volatilization of the terpene compounds from the material, along with the cannabinoid compounds. Comparatively in the round-tip probe work, a very small amount of sample is transferred onto the probe with a shorter injection time and higher starting column temperature, which accounts for the very small terpene peaks and large THC peak. The identified compounds for cannabis sample 1 can be found in Table 6 with their respective RTs, library match scores (LMS), and CAS numbers.

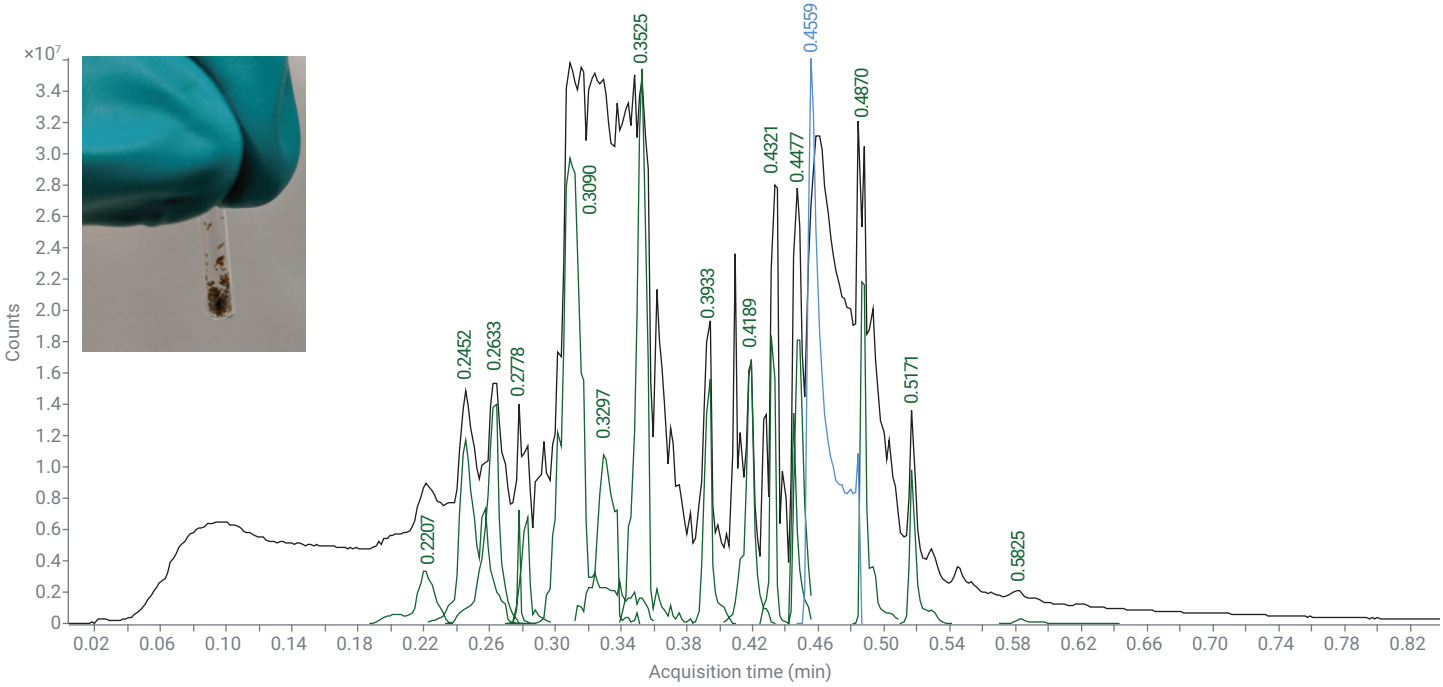

Figure 27. TIC of cannabis sample 1 with EICs corresponding to the deconvoluted peaks shown in green; the inset shows the cannabis sample in the TSP vial.

Cannabis sample 2 was a finely ground material that clumped together in small balls; approximately 10 mg was transferred into the TSP vial with a spatula. The TIC for cannabis sample 2 (Figure 28) was also complex, with a different profile of terpenes and cannabinoids. In cannabis sample 1, Δ-9-tetrahydrocannabivarin, cannabichromene, THC, and cannabinol were identified; comparatively, cannabis sample 2 contained cannabinol, cannabidiol, and THC (Table 7). For the terpenes, fenchol, 4-methyl-benzaldehyde, α-bisabolol, and β-amyrin were identified in both samples. A TSP vial can be used when interested in a fast, qualitative overview of the terpene and cannabinoid profiles of different cannabis samples.

Table 6. Compounds identified in cannabis sample 1, shown in Figure 26, with RTs, LMS, chemical formulae, and CAS numbers.

RT (min)	Compound	LMS	Formula	CAS Number
0.2207	D-Limonene	95.1	$C_{10}H_{16}$	5989-27-5
0.2452	Fenchol	89.4	$C_{10}H_{18}O$	1632-73-1
0.2633	α.-Terpineol	75.8	$C_{10}H_{18}O$	98-55-5
0.2778	4-Methyl-benzaldehyde	79.5	$C_{8}H_{8}O$	104-87-0
0.2823	Benzoic acid	89.0	$C_{7}H_{6}O_{2}$	65-85-0
0.3090	(−)-Aristolene	80.1	$C_{15}H_{24}$	6831-16-9
0.3525	α-Bisabolol	81.4	$C_{15}H_{26}O$	515-69-5
0.3933	*n*-Hexadecanoic acid	94.9	$C_{16}H_{32}O_{2}$	57-10-3
0.4189	Octadecanoic acid	88.5	$C_{18}H_{36}O_{2}$	57-11-4
0.4321	Δ-9-Tetrahydrocannabivarin	70.8	$C_{19}H_{26}O_{2}$	31262-37-0
0.4452	Sugiol	73.4	$C_{20}H_{28}O_{2}$	511-05-7
0.4477	Cannabichromene	88.3	$C_{21}H_{30}O_{2}$	20675-51-8
0.4559	THC	82.3	$C_{21}H_{30}O_{2}$	1972-08-3
0.4870	Cannabinol	90.6	$C_{21}H_{26}O_{2}$	521-35-7
0.5171	Nonacosane	93.0	$C_{29}H_{60}$	630-03-5
0.5825	β-Amyrin	73.1	$C_{30}H_{50}O$	559-70-6

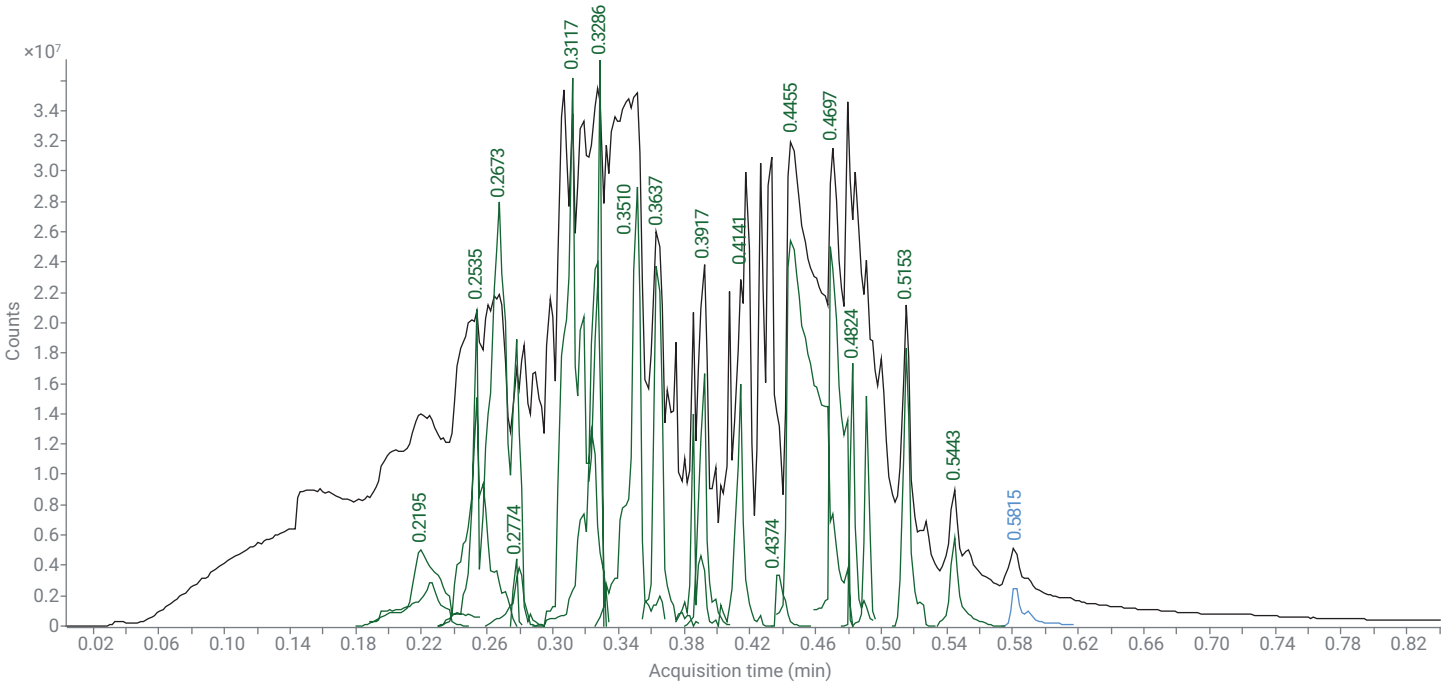

Figure 28. TIC of cannabis sample 2 with EICs corresponding to the deconvoluted peaks shown in green.

Table 7. Compounds identified in cannabis sample 2, shown in Figure 27, with RTs, LMS, chemical formulae, and CAS numbers.

RT (min)	Compound	LMS	Formula	CAS Number
0.2195	β-Thujene	87.4	$C_{10}H_{16}$	28634-89-1
0.2254	Acetic acid	95.7	$C_2H_4O_2$	64-19-7
0.2535	Fenchol	73.6	$C_{10}H_{18}O$	1632-73-1
0.2673	Isobornyl acetate	78.2	$C_{12}H_{20}O_2$	125-12-2
0.2774	4-Methyl-benzaldehyde	79.0	C_8H_8O	104-87-0
0.2796	Glycerin	77.4	$C_3H_8O_3$	56-81-5
0.3117	2-Methylene-4,8,8-trimethyl-4-vinyl-bicyclo[5.2.0]nonane	83.5	$C_{15}H_{24}$	242794-76-9
0.3265	γ-Cadinene	74.7	$C_{15}H_{24}$	39029-41-9
0.3286	Selina-3,7(11)-diene	74.9	$C_{15}H_{24}$	6813-21-4
0.3510	α.-Bisabolol	81.7	$C_{15}H_{26}O$	515-69-5
0.3637	Drimenol	75.4	$C_{15}H_{26}O$	468-68-8
0.3852	m-Camphorene	81.2	$C_{20}H_{32}$	20016-73-3
0.3917	*n*-Hexadecanoic acid	88.8	$C_{16}H_{32}O_2$	57-10-3
0.4141	9,12-Octadecadienoic acid	75.2	$C_{18}H_{32}O_2$	60-33-3
0.4374	Cannabinol	78.0	$C_{21}H_{26}O_2$	521-35-7
0.4455	Cannabidiol	94.1	$C_{21}H_{30}O_2$	13956-29-1
0.4697	THC	74.2	$C_{21}H_{30}O_2$	1972-08-3
0.4824	Cannabinol	88.7	$C_{21}H_{26}O_2$	521-35-7
0.4912	Docosane	82.1	$C_{22}H_{46}$	629-97-0
0.5153	Tetratriacontane	93.2	$C_{34}H_{70}$	14167-59-0
0.5443	Hentriacontane	82.3	$C_{31}H_{64}$	630-04-6
0.5815	β-Amyrin	91.8	$C_{30}H_{50}O$	559-70-6

For both cannabis samples, carryover was a concern. There was no contamination of the system; however, the TSP holder did have carryover of the cannabinoids for both cannabis samples. It can be difficult to remove the TSP vial from the holder, and it is possible that some material may fall out of the vial and contact the holder. Alternatively, if any material was clinging to the exterior of the vial during loading, the material could transfer onto the holder. Shown in Figure 29A are overlays of the seven holder blanks after the cannabis 2 sample was run. The first blank run (black trace of Figure 29) shows significant carryover of cannabidiol, THC, and a small peak of cannabinol. Looking closer at the repetitive blank runs in Figure 29B shows a significant decrease in all cannabinoid compounds. By blank run 5 (gold trace), cannabidiol is no longer identified with a match score above 70, but we can still observe a small peak, and if we sample the mass spectra under the peak (and subtract the nearby baseline), the mass spectra for cannabidiol is still identifiable. By blank 7, there is a very small peak in the TIC and a spectral ion of *m/z* 231 is still observable, but the rest of the fragmentation pattern for cannabidiol is obscured by the background and not identifiable. Rinse the TSP holder with solvent after removing the sample vial to avoid this type of holder or sample handling carryover.

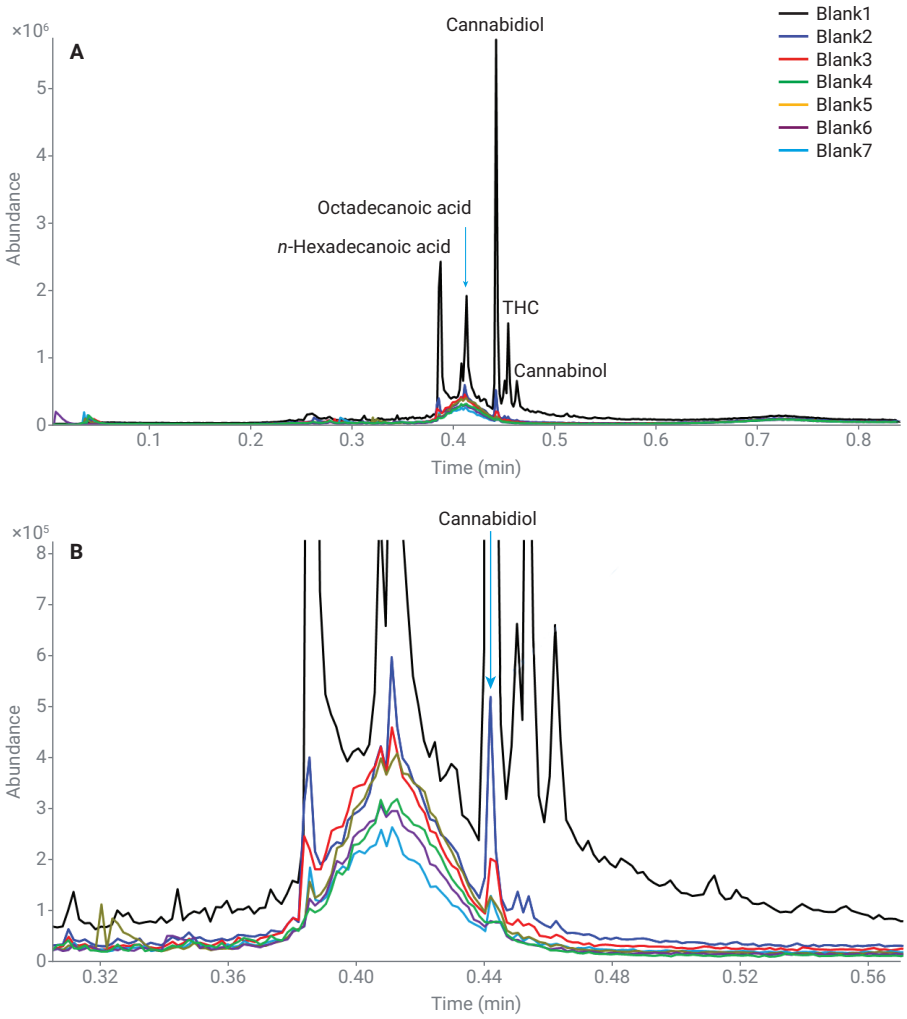

Figure 29. A) TIC overlays of TSP holder blank runs after cannabis sample 2 was completed and the vial was removed; B) zoom-in of overlays from 0.3 to 0.56 minutes to better observe the decrease in cannabinoid carryover.

Mitigating carryover

Carryover was observed in select plant material with TSP vial holder experiments, powder experiments, liquid experiments, and the nitroglycerin spray with the glass probes. There are several ways to mitigate carryover when preparing the samples. Attention to sampling techniques, probe blanks, probe holder blanks, and system blanks help the user avoid carryover issues or rectify the issue quickly. Familiarization with the following manual techniques is critical for the successful operation of the QuickProbe GC/MS system.

Always use the probe holder (or TSP vial holder) for sample collection and injection. After performing system blanks and probe holder blanks, it is best to install the glass probe into the probe holder. Collect the probe blanks, and leave the probe in the probe holder to collect the sample.

For the TSP vial and holder, the vial must be removed from the holder to add sample and avoid holder contamination. A user should collect the holder blanks, insert the vial and collect vial blanks. Then, remove the vial to add sample, wipe the vial with a lint-free wipe to remove sample from the exterior, and reinstall it into the vial holder.

Wipe the glass rod (or TSP vial) with lint-free wipes to remove excess sample, especially in the cases of viscous liquids, powders, or tablet scrapings, where sample may exist on the sides of the glass probe (Figure 30). This may not be feasible in all laboratories, but any sample on the sides of the glass probe can contaminate the probe holder, especially the tip. For liquids, care should be taken to ensure that the probe is dipped <5 mm into a liquid and the probe holder is held at a downward angle to avoid sample migrating up the probe towards the probe holder while drying.

Powders or plant material, especially when stored in vials or plastic bags, can gather a static charge and cling to the sides of the glass probe (or TSP vial).

Place powder or plant material in a weight boat for sampling, touch the material gently with a probe, then tap it against the side of the weigh boat to remove excess. If feasible, placing the material in a weight boat can help lower the possibility of carryover, since the sample can be spread across the weight boat and easily tapped with a lower probability of material being deposited up the sides of the probe.

Rinse the probe with solvent to remove excess. If feasible, the user can use a pipette to lightly rinse the probe with ~1 mL or less of solvent to remove some of the sample, especially in the case of powders that may be deposited on the sides of the glass probe (Figure 31). This may not be feasible in all laboratories.

Dilute the sample with solvent. If the sample is a powder, a known quantity of powder (for example, 500 µg) could be placed in 1 mL of solvent and dissolved to lower the concentration of the compounds and the probability of carryover. In this case, an RTP would be dipped into the liquid sample, removed to evaporate the solvent, then inserted into the inlet of the QuickProbe GC/MS system. For this technique, compounds at low concentrations would be further lowered with the dilution technique, which may affect detection.

If concerned about carryover after running a TSP vial sample, a user can rinse the TSP holder with a polar solvent and nonpolar solvent, such as methanol and acetone. After the run of interest has completed and the solvents have dried, insert the TSP holder into the inlet for 20 to 30 seconds to bake the metal holder. Wait two to three minutes, and run a TSP holder blank to check for carryover.

Figure 30. Using a lint-free wipe to clean the sides of a glass probe after adding sample to lower the possibility of carryover.

Figure 31. Rinsing the glass probe after adding sample with a solvent such as acetone can lower the possibility of carryover.

Conclusion

Various forensic sample types can be tested with the QuickProbe GC/MS system for fast, qualitative analysis. Liquid, tablets, ointments, and plant material can be sampled with round-tip probes by dipping the probe into the sample or scraping along the sample. The pocket probe has been designed to hold a small amount of powder material in the concave tip and can also be used to scrape tablets. TSP vials can be used with, ideally small, ground, or chopped plastic pieces, powder, or plant material. For powders and plant materials, a longer injection time can provide more time for terpene or terpene-like compounds to volatilize and profile those compounds per sample. Carryover can be mitigated with careful sampling techniques and frequent probe holder and system blanks. With the potential for coelution and overlapping peaks in the total ion chromatogram, mass spectral deconvolution software and a large mass spectral library make data analysis and peak identification easier and faster for the user.

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

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