Application Note

Food Testing & Agriculture

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60-Second Screening of Foods Using the Agilent QuickProbe GC/MS System

Authors

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Abstract

The Agilent QuickProbe, a direct insertion sampling device for GC/MS, was evaluated for the screening of nonextracted food samples. Foods analysis benefits from fast screening because it quickly identifies samples that are suspect and require further investigation.

Introduction

Typical GC/MS screening of foods and botanicals requires sample preparation such as QuEChERS or other liquid extraction methods. Using the QuickProbe system enables a simple and fast screening analysis that requires no sample preparation. The QuickProbe unit contains a short GC column, and is mounted on the top of the oven of either an Agilent 5975 or 5977 GC/MSD instrument. Sampling is accomplished by touching the sample with a glass probe and inserting the probe into an open atmosphere heated inlet. Ultra fast heating of the column in the presence of helium flow accomplishes the separation of sample components. Data acquisition and analysis is performed using Agilent MassHunter Workstation Acquisition and Unknowns Analysis software, and spectra are identified by searching against user or commercial libraries. Many food sample types have been studied including various oils, spice mixes, beverages, plant material, and flavorings. Samples may consist of either unprepped samples before extraction, as described here, or extracts resulting from the existing laboratory workflow.

Experimental

An Agilent 5977B single quadrupole mass spectrometer was coupled to an Agilent 7890B GC instrument equipped with a separate QuickProbe control unit (Figure 1). The QuickProbe system (G3971A) had an open inlet containing a specialty liner with frit (5190-5104), as shown in Figure 2, a 1.5 m × 0.25 mm, 0.1 µm DB-1HT column, and a 0.7 m × 0.18 mm, 0.18 µm DB1-MS column used as a restrictor into the mass spectrometer.

Helium was used as the carrier gas. The GC/MS system was autotuned. Round tip, glass sample probes (5190-5118) were obtained in touchless packaging (Figure 3), and were held using the QuickProbe holder (G3971-60200) shown in Figure 4, that works as the sample insertion device. Pocket tip probes (5190-5113) contain an indentation or "pocket" at the tip, and are useful for powders. Table 1 lists instrument conditions. Some variations in column temperature hold time and ramp rate were also used.

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

Figure 2. Specialty liner with frit (5190-5104).

Figure 3. Sample probes in touchless packaging (round tip, 5190-5118; pocket tip, 5190-5113).

Figure 4. Probe holder shown in loading position with inserted probe on left side (G3971-60200).

Sampling was performed by first inserting a glass probe into the probe holder then, while in loading position (Figure 4), scraping the probe along the solid food or plant material. In a liquid sample, the tip of the probe was dipped into the liquid. Powdered or granular samples were loaded by rubbing the glass probe with sample or tapping the pocket tip probe into the sample. Sample introduction was performed by first retracting the 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 position the probe into the hottest part of the inlet. Insertion time was generally five seconds, but this could be varied as required. MassHunter Workstation Acquisition and Unknown Analysis software were used for data acquisition and processing. A minimum match factor of 60 was used for NIST library matches.

Results and discussion

Various food components were easily differentiated using the QuickProbe GC/MS system. The chromatograms resulted from analysis of nonextracted food samples. They demonstrate the power of chromatographic separation coupled with mass spectral deconvolution to screen highly complex samples and identify targets (Figures 5 to 9). NIST library match scores for each component are in parentheses. As a demonstration, several types of oils such as fish, sesame seed, and vegetable were differentiated using the GC/MS QuickProbe system.

Table 1. Instrument conditions.

QuickProbe and GC Conditions	
Inlet Temperature	250 °C (isothermal only)
Injection Mode	Split (the split is fixed at ~1:10)
Column Temperature	35 °C, hold for 6 seconds 4 °C/sec to 325 °C, hold for 0 seconds (or extended hold)
Run Time	Generally 40 to 60 seconds
Transfer Line Temperature	280 °C
MS Conditions	
Ion Source Temperature	280 °C
Quadrupole Temperature	150 °C
Ionization	EI mode
EMV Mode	Gain factor
Gain Factor	10 (should be lowest value required to detect peaks of interest; minimum is 0.05)
Solvent Delay	0 minutes
Scan Type	Scan (38 to 550 μ, 6,250 μ/sec)
Scans Per Second	9.7

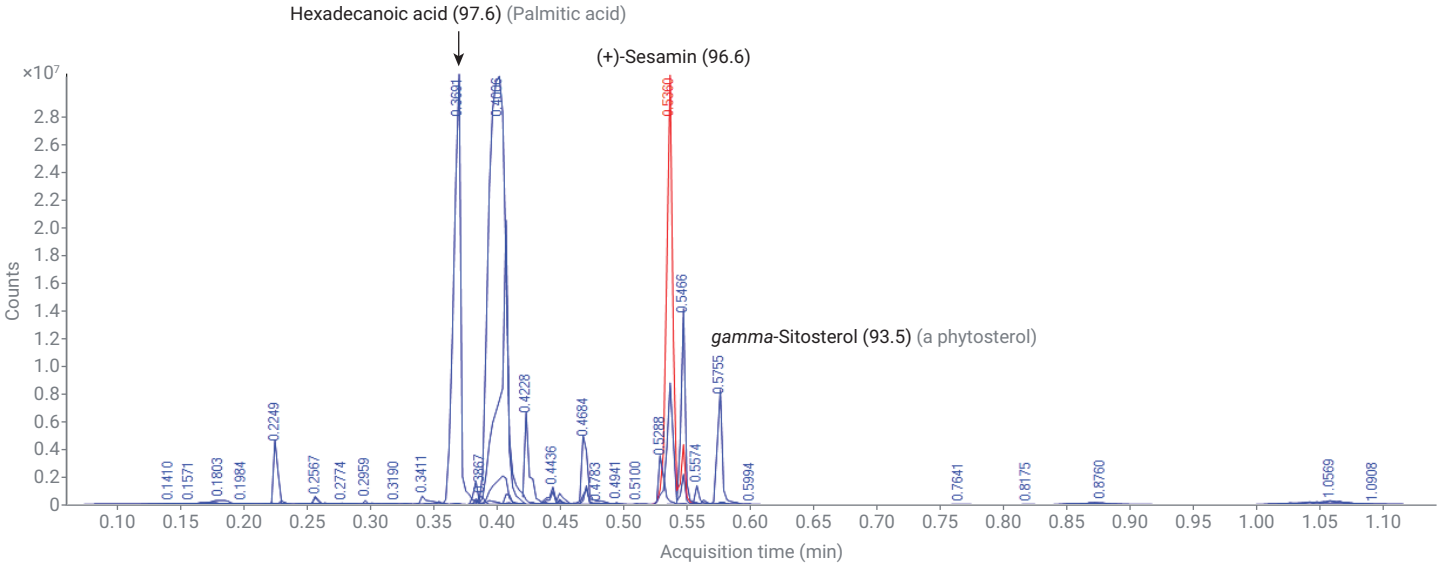

Figure 5. Sesame seed oil. The characteristic component sesamin is identified with a high library match score of 96.6.

This was due to the presence of characteristic components such as sesamin, in sesame seed oil (Figure 5), and cholesterol in fish oil (Figure 6). The profile for vegetable oil shown in Figure 7 shows a peak for 2,4-decadienal, which is formed upon oxidation and contributes to the characteristic aroma of fried foods.

Plant material was able to be screened for components by manually crushing a leaf around the glass probe. The characteristic compound umbellunone was found in California Bay Laurel leaf (Figure 8); this compound differentiates this species from the true bay leaf, or *Laurus nobilis*.[1] Native Americans used the California Bay Laurel leaves for various medicinal purposes due to their curative properties. This species is sometimes known as the "headache tree" because umbellunone can cause headaches in some sensitive individuals. Methyl eugenol was also determined to be a major constituent of the sample.

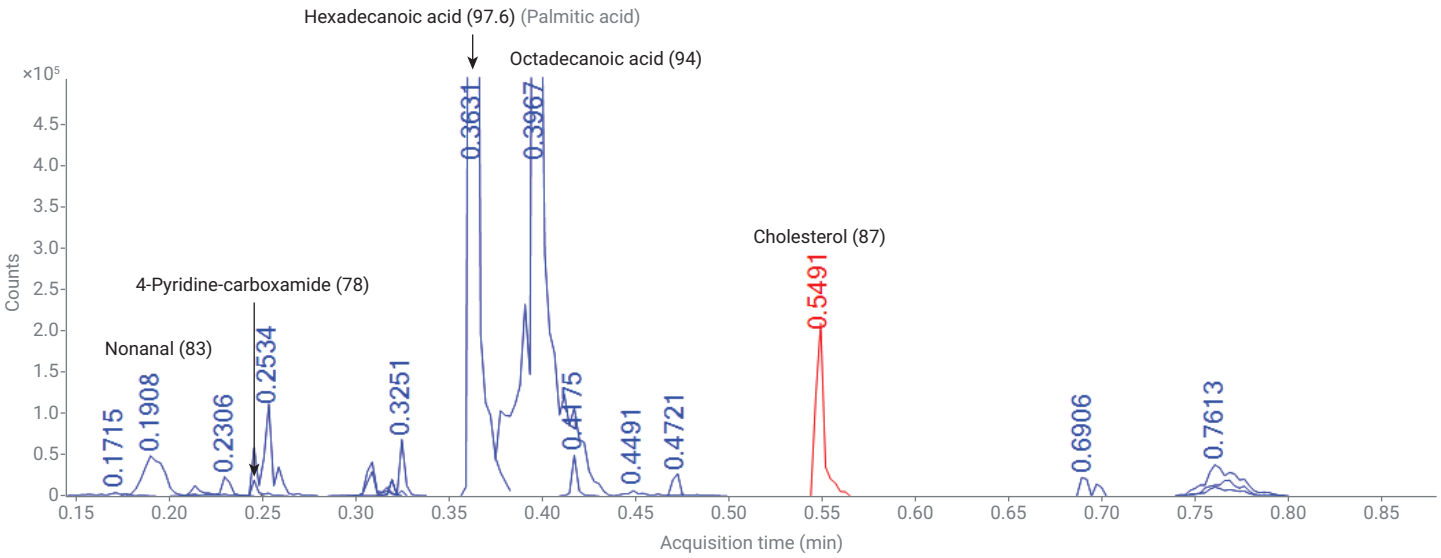

Figure 6. Commercial fish oil showing a peak for cholesterol.

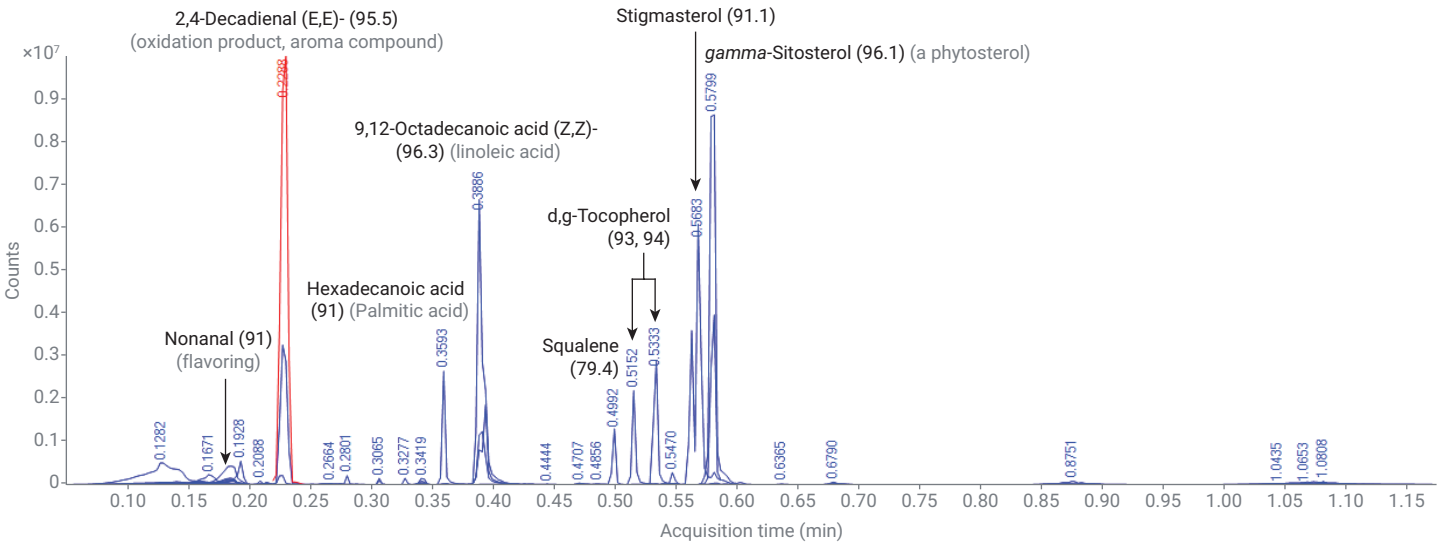

Figure 7. Vegetable oil profile (cottonseed).

Figure 9 shows a chromatogram for a peppery spice rub mix. The compound piperine, from black pepper, was determined in this sample along with *n*-isobutyl-2,4-decadienamide, which is found in herbs and spices. Vitamin E was also detected, and had a library match score of 81.

The QuickProbe GC/MS system successfully characterized several food samples in under one minute without the need for sample preparation. Diverse sample types such as liquids (oils), granular or whole food, and plant material were sampled using a round tip or pocket tip glass probe. Other means of sampling solid plant material (i.e., cannabis), using a thermal desorption technique, have been used with success, and are described elsewhere (Agilent publication 5994-1357EN).

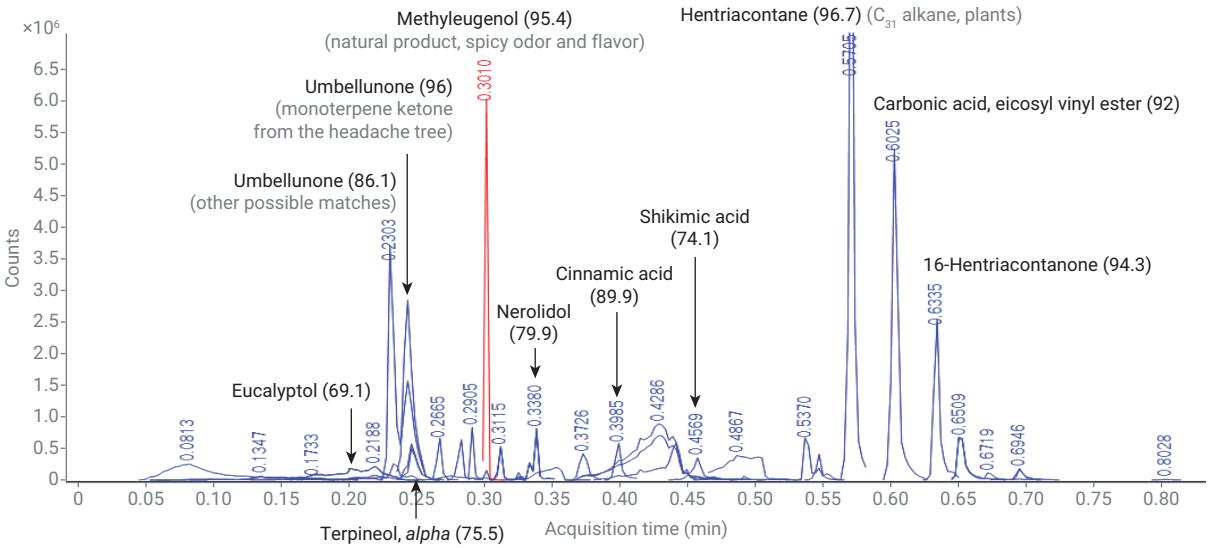

Figure 8. Leaf from the California Bay Laurel (headache tree).

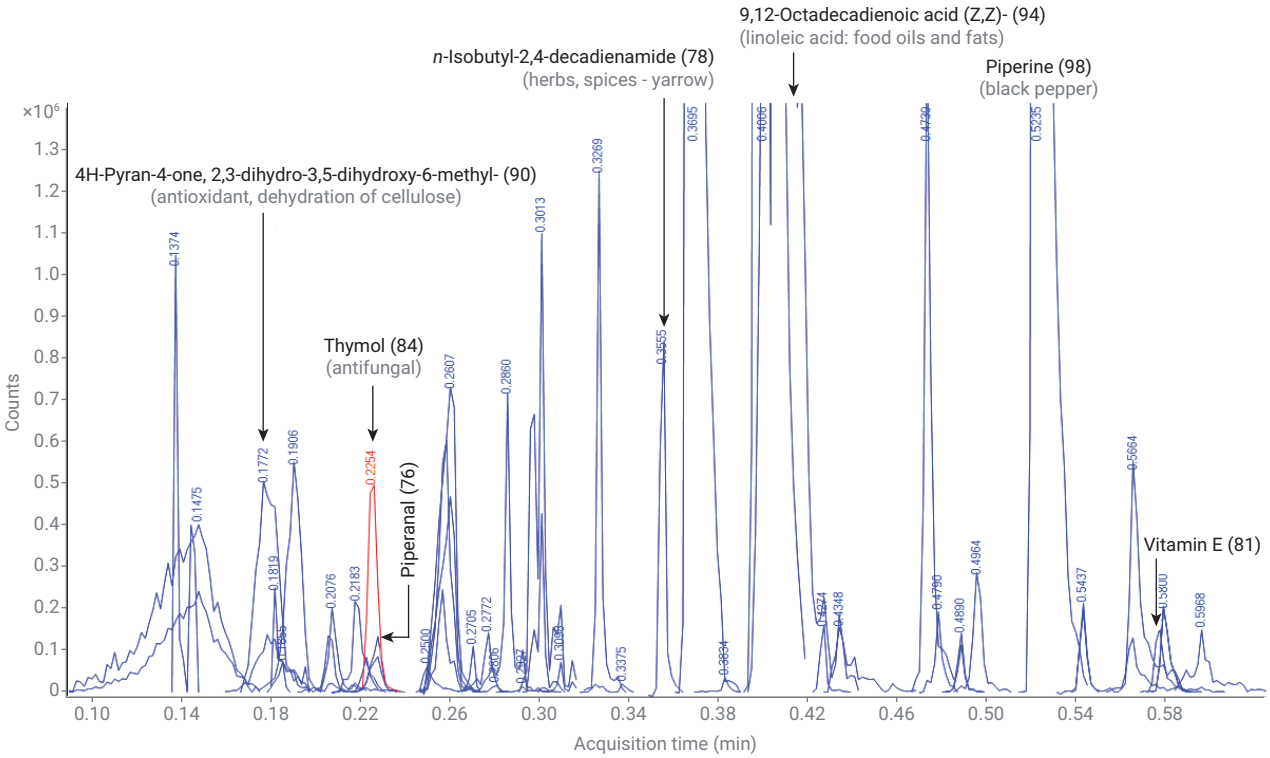

Figure 9. Spice rub mix with black pepper.

Conclusion

The power of the Agilent QuickProbe GC/MS system lies in its ability to quickly chromatograph complex foods and plants, without prior extraction, using a short GC column coupled to a mass spectrometer. Characteristic sample components were identified using Agilent Unknowns Analysis software with spectral match against the NIST library. Thus, a 60-second food screen is made possible using the QuickProbe GC/MS system.

Reference

1. Wang, M. *et al.* Application of GC/Q-TOFQ Combined with Advanced Data Mining and Chemometric Tools in the Characterization and Quality Control of Bay Leaves. *Planta Med* **2018** Sep, *84(14)*, 1045–1054. doi: 10.1055/a-0585-5987. Epub 2018 Mar 14.

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