APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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Coffee Characterization Using Clarus SQ 8 GC/MS, TurboMatrix HS Trap and GC SNFR Olfactory Port

Introduction

Coffee is a very popular drink in most parts of the world and is one of the most traded agricultural commodities on the planet. The drinking of coffee, however, is a fairly recent activity. Although its origin may be attributed to Ethiopia a thousand years ago, its popularity as a beverage really started in the Middle East around the start of the 17th century.

Part of its popularity is due to the stimulating effect of its caffeine content (a cup of coffee may contain as much as 150 mg) and part is due to its rich complex taste. The taste of a cup of coffee depends on many factors – the coffee bean variety and horticulture and the way the beans are stored, roasted, ground and brewed. Even the water used to make the coffee can have an effect on its flavor.

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For such a commercially significant product, it is important that there should be some means to characterize and control its taste at the various stages of production. This may achieved organoleptically (i.e. by smelling and tasting) or by using powerful analytical tools like gas chromatography mass spectrometry (GC/MS) to determine chemical composition.

Aroma plays a very important part in the taste of coffee. This application note presents a system for characterizing finished coffee aroma while simultaneously performing a chemical analysis on a mass spectrometer. Further data may be acquired using a flame ionization detector (FID) for chemometric processing to provide further insight into the individual character of each coffee sample. The results provide a powerful insight into both the chemical composition and the sensory perception of coffee aroma. Such a system can be used for quality control purposes, process and product development, storage studies, trouble-shooting and evaluating competitive products.

Instrumentation

In this analysis, a headspace trap system may be utilized for sample introduction to characterize the flavor of roasted coffee beans. This technique ensures that non-volatile material in the beans does not enter the analytical system, which can cause interference in the chromatography and potential system contamination. The headspace trap extracts the volatile components from a large sample and focuses them onto an inline adsorbent trap. It also facilitates very easy sample preparation – a weight of ground beans is dispensed into a vial and sealed. The subsequent analysis is then fully automated.

A PerkinElmer TurboMatrix™ Headspace Trap connected to a PerkinElmer Clarus® SQ 8 GC/MS with a flame ionization detector is used for these experiments. The MS provides the ability to identify each separated component and the FID is used to provide the quantitative data used in the chemometrics analysis. A schematic diagram of the GC system is given in Figure 1.

Using a headspace trap instead of the classical headspace technique enables up to 100 times improved detection limits over classical static headspace methods.

A polar 60 m x 0.25 mm x 1.0 µm Elite Wax column is used. This thick-film column provides sufficient chromatographic retention to separate the early-eluting most volatile components and provided the dynamic range necessary to chromatograph both high level and low level components in the coffee.

The column effluent is split between a PerkinElmer SNFR™ GC olfactory port, the MS detector and the FID. This splitting is performed using an S-Swafer™ in a standard active splitting configuration.

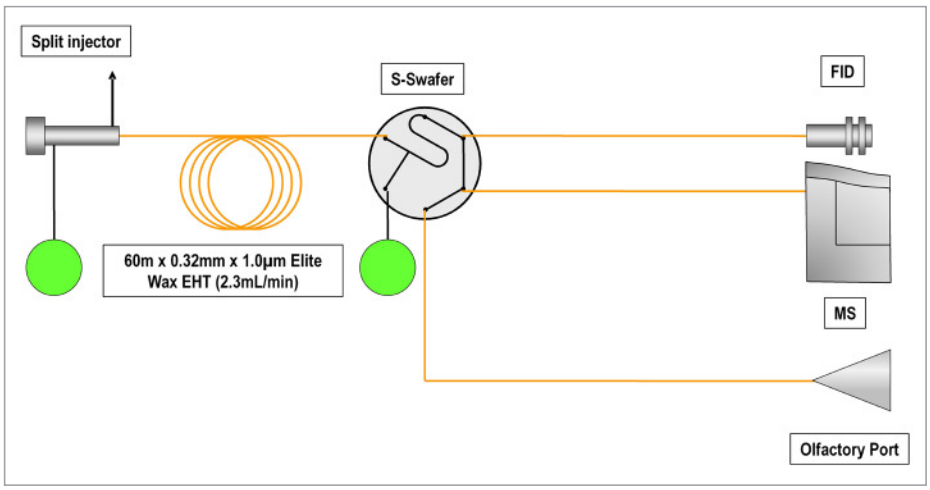

Figure 1. Schematic diagram of the GC system.

Experimental

Overview

Twenty seven varieties of pre-roasted and freshly roasted coffee beans from throughout the world were procured and examined in this work. These are listed in Table 1.

Table 1. Coffee samples examined.

1	Kona Cloud® Hawaiian coffee beans
2	Green Mountain® ground coffee (15 g packets)
3	Green Mountain® ground decaffeinated coffee (15 g packets)
4	Harar Longberry® Ethiopian coffee beans
5	Moka Harar® CP Select Ethiopian coffee beans
6	Kona Cloud® Hawaiian coffee beans medium roast
7	Kona Cloud® Hawaiian coffee beans dark roast
8	Other Kona coffee beans from Hawaii
9	Coffee beans from El Salvador
10	Coffee beans from Yemen
11	Coffee beans from Sidamo
12	Ground coffee from Trinidad
13	Ethiopian decaffeinated coffee beans
14	Guji Sueq'to Ethiopian coffee beans roasted before first crack
15	Guji Sueq'to Ethiopian coffee beans roasted just after first crack
16	Guji Sueq'to Ethiopian coffee beans roasted just before second crack
17	Guji Sueq'to Ethiopian coffee beans roasted just after second crack
18	Guji Sueq'to Ethiopian coffee beans roasted long after second crack
19	Guji Sueq'to Ethiopian coffee beans carbonized
20	Folgers® 5 g ground coffee bag
21	Folgers® 5 g ground decaffeinated coffee bag
22	Kona Cloud® freshly roasted beans
23	Trader Joe's® Cafe Pajoro beans (old)
24	Costa Rican El Trapiche beans bought at plantation
25	Costa Rican Britt® medium roasted beans
26	Costa Rican Britt® dark roasted beans
27	Barista® French roast ground coffee machine cartridge

Analytical Method

The experimental conditions for this analysis are given in Tables 2 to 8.

Table 2. HS trap conditions.

Headspace System	TurboMatrix 110 HS Trap
Vial Equilibration	80 °C for 20 minutes
Needle	120 °C
Transfer Line	140 °C, long, 0.25 mm i.d. deactivated fused silica
Carrier Gas	Helium at 25 psig
Dry Purge	7 min
Trap	Air Toxics, 25 °C to 260 °C, hold for 7 min
Extraction Cycles	1 with 40 psig extraction pressure

Table 3. GC conditions.

Gas Chromatograph/ Mass Spectrometer	Clarus 580 SQ 8
Column	60 m x 0.32 mm x 1.0 µm Elite-5MS connected directly to the HS Trap
Oven	40 °C for 1 min, then 5 °C/min to 200 °C for 5 min
Carrier Gas	Helium at 25 psig at injector and 13 psig at Swafer
Flame Ionization Detector	275 °C, range x1, attenuation x8

Table 4. MS conditions.

Scan Range	m/z 35 to 350
Scan Time	0.1 s
Interscan Delay	0.06 s
Source Temp	250 °C
Inlet Line temp	250 °C
Multiplier	1700V

Table 5. Olfactory port conditions.

Olfactory Port	PerkinElmer SNFR
Transfer Line	225 cm x 0.250 mm at 240 °C
Humidified Air	500 mL/min with jar set to 37 °C

Table 6. Chemometric.

Software	InfoMetrix Pirouette Version 4.0
Data	Collected using the flame ionization detector

Table 7. Swafer conditions.

Swafer	PerkinElmer S-Swafer in the S1 configuration
Settings	Developed using the Swafer Utility Software – see Figure 2.

Table 8. Sample details.

Sample Preparation	Beans were freshly ground and 1 g was weighed into a sample vial and sealed
Vial	Standard 22 mL vial with aluminum crimped cap with PTFE lined silicone septum

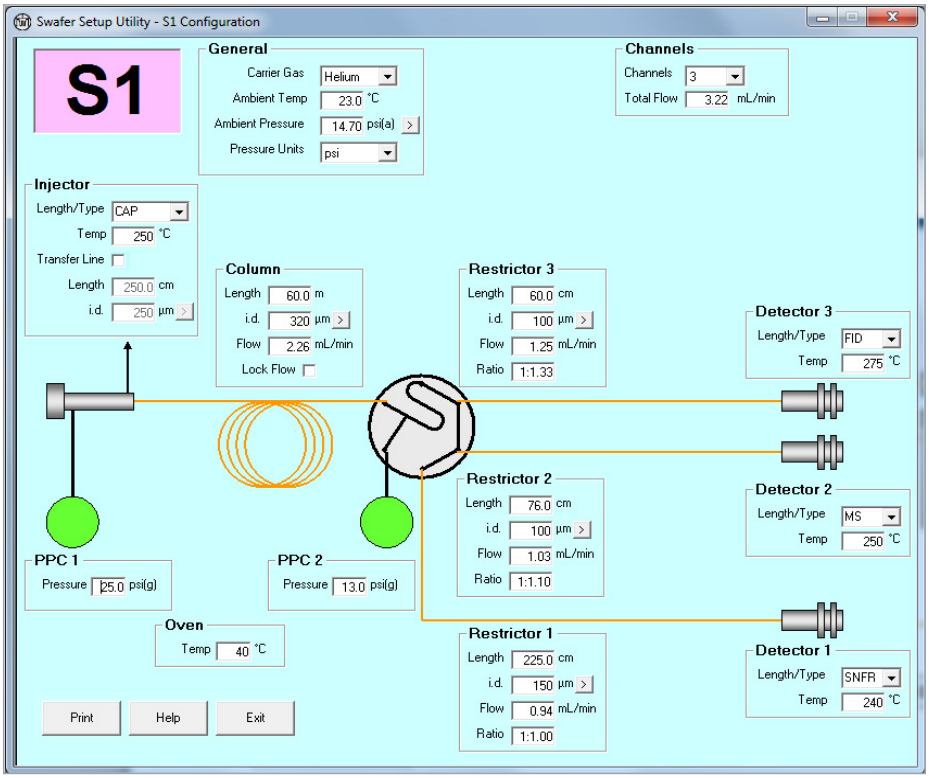

Figure 2. The S-Swafer in the S1 configuration for MS, FID and olfactory work.

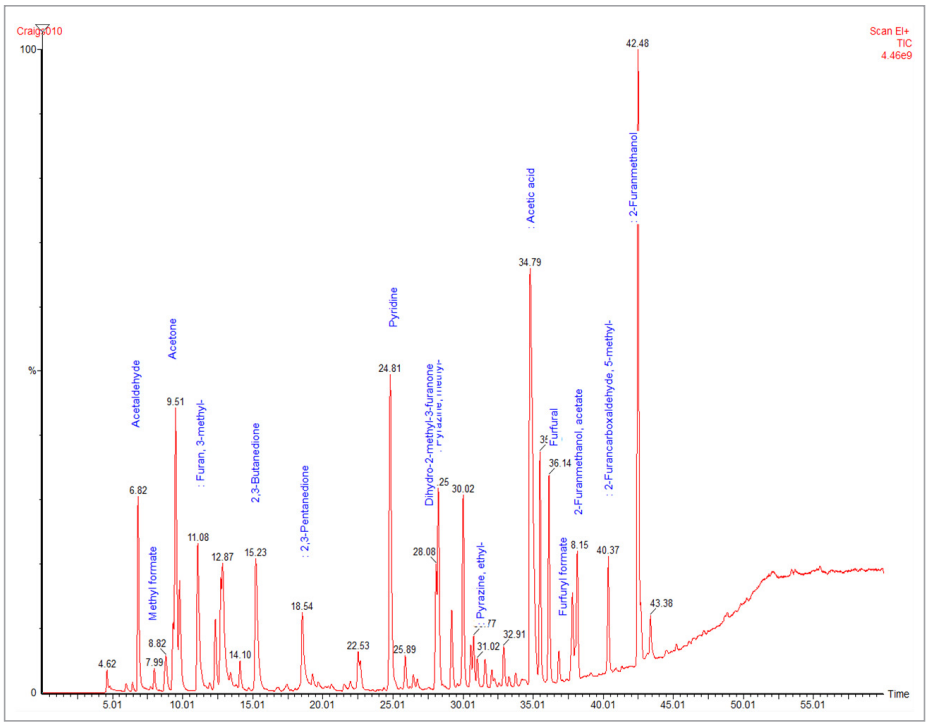

Figure 3. Typical chromatogram from 1 g coffee grains.

Results

Chromatography on the MS

Slow chromatographic times are preferred to enable the analyst to fully elucidate his or her sensory experience as the peaks elute. Faster chromatography is possible but then there is a risk that odors from adjacent peaks may start to overlap. Slower chromatography also gives the user more time to fully narrate and record their sensory perceptions.

Figure 3 shows a section from a chromatogram of coffee sample #3. The key components were identified using the library search capabilities of the TurboMass™ software supplied with the Clarus SQ 8 GC/MS.

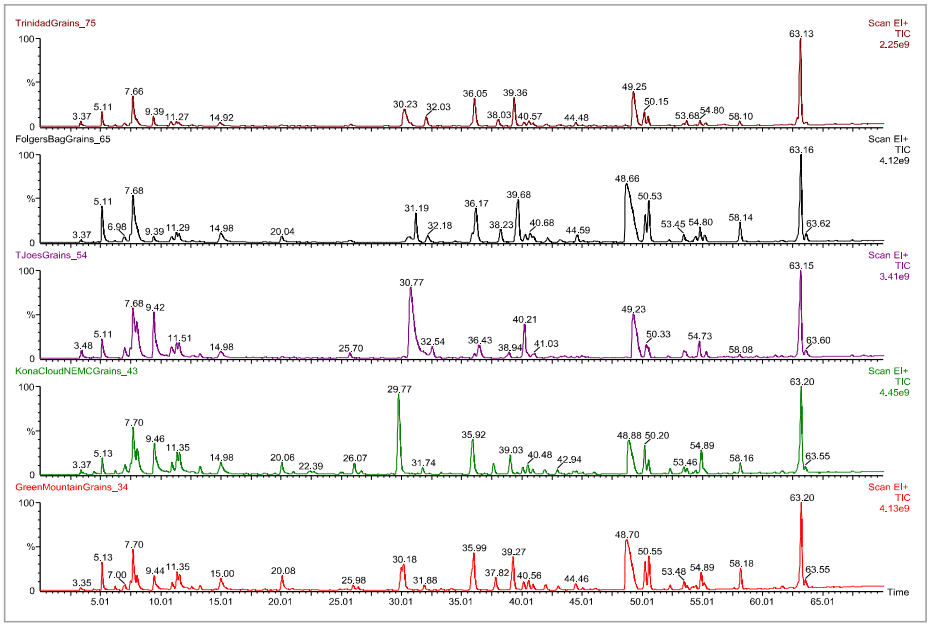

Figure 4. Chromatogram from five different types of ground coffee.

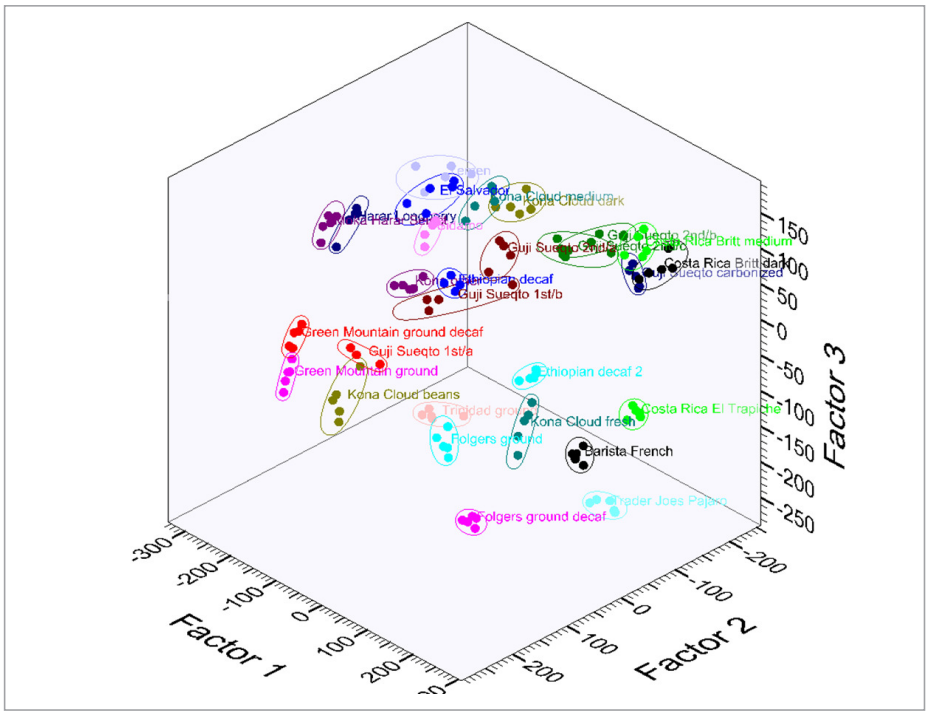

Figure 5. Principal component analysis loadings of first three factors for the 27 coffee samples examined.

Chemometrics

Visual analysis of the five chromatograms in Figure 4 shows the subtle differences between the different coffees. While it is possible to identify one or two peaks that differ between one or two chromatograms this is unfeasible in a production setting and a better, faster and more objective solution is required.

The InfoMetrix® Pirouette® software was used to perform a principal components analysis (PCA) on chromatographic data from replicate analysis of all 27 of the coffee bean samples collected using the flame ionization detector. The PerkinElmer FID data files were able to be read and processed by the Pirouette® software.

Figure 5 shows plots of the loadings for each coffee for the first three PCA factors. These factors can be regarded as a 'building blocks' that are common to each chromatogram but are present at different levels (or loadings). As can be seen in Figure 5 the three loadings for the replicate chromatograms for each sample are very similar giving rise to tight clustering in the plots. Each sample cluster is separated from the other sample clusters. In this way, subtle differences or patterns in the chromatography can be used to discriminate between different coffee types. This discrimination may be correlated with sensory perception; in which case, the PCA may be used to guide the user to specific chemical compounds or groups of compounds that are responsible for the aroma character of that coffee.

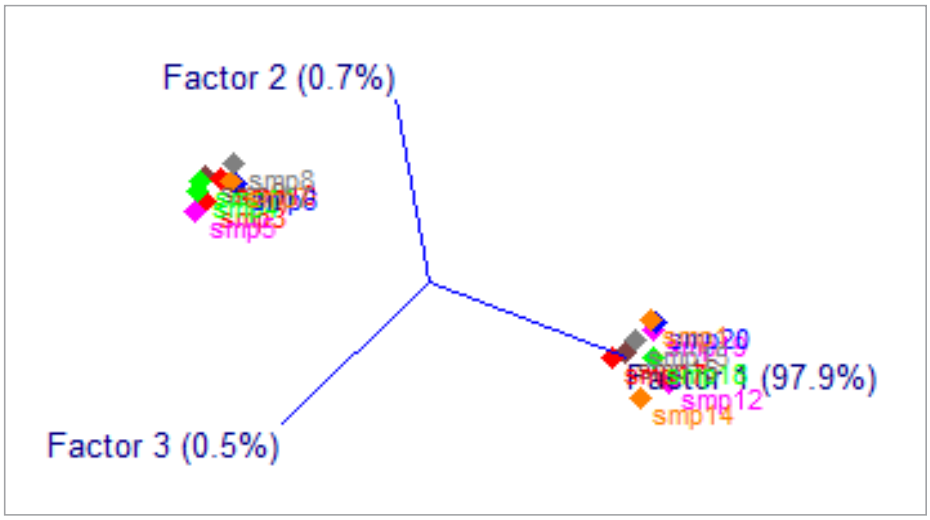

Figure 6. Detail from PCA map of two coffee sample chromatograms.

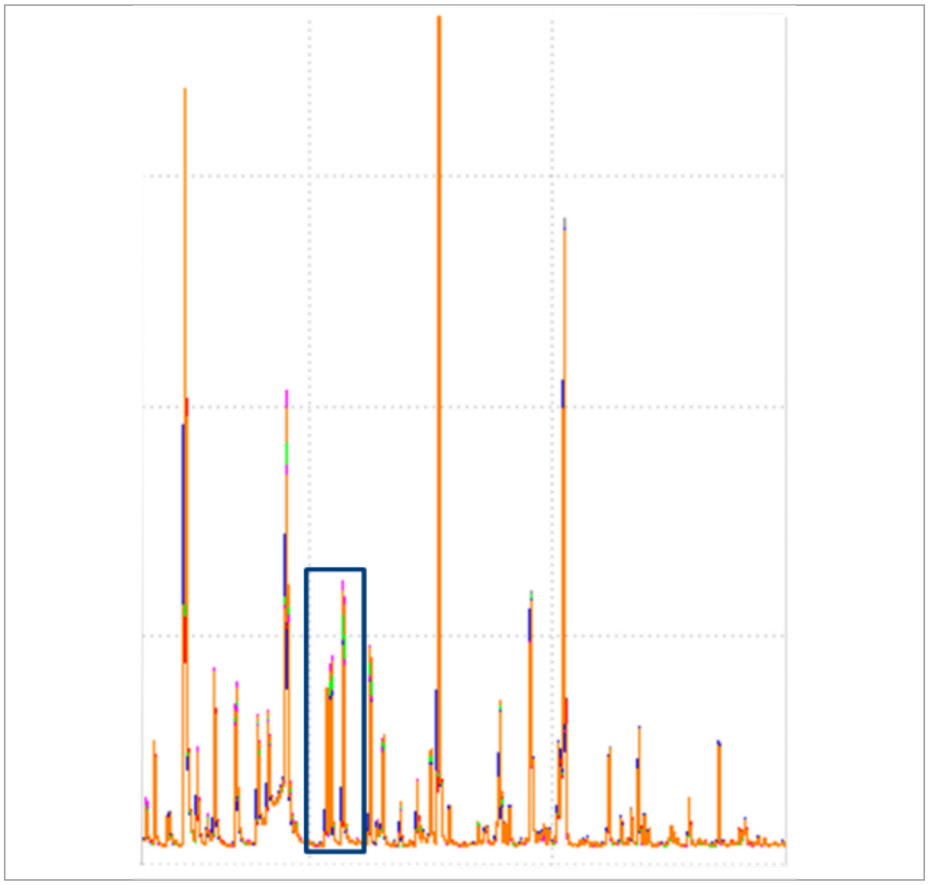

Figure 7. Chromatography of samples #2 and #22 overlaid.

For example, Figure 6 shows the PCA loadings for just two of the coffee samples. The replicate PCA results are tightly clustered for each sample type but well separated from the other sample type. Clearly there are differences in the chromatography between these two samples. Inspection of the PCA factors highlights an area in the chromatography where significant differentiation is apparent. This area is shown in Figures 6 and 7. In this instance, the difference is clear but there may be areas in the chromatography where the difference may be more subtle or may be because of a combination of peaks (patterns). This is where PCA would be a powerful tool to highlight such areas.

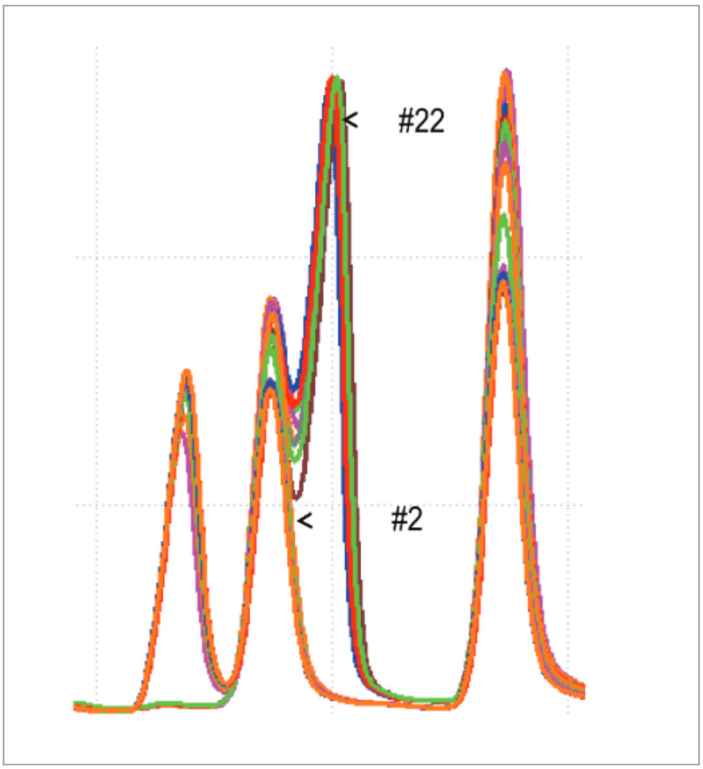

Figure 8. Detail from figure 7.

Olfactory Monitoring

Figure 9 shows an image of the SNFR system used for the olfactory monitoring. Figure 10 shows a photograph of Mr. Snow, a coffee expert, using the device to monitor the aroma of individual compounds. While the coffee aroma components are being monitored, the user is able to record his or her sensory perceptions by voice into the supplied microphone and by positioning a joystick to indicate the intensity of the aroma. This information may be accessed and reviewed when displaying the chromatogram after the run is complete.

Figure 9. The GC SNFR system.

Figure 10. Photograph of coffee expert monitoring coffee aroma compounds.

Conclusion

The combination of chromatographic, mass spectral, chemometric and olfactory data from a single analysis provides a very powerful insight into the aroma and taste of complex samples such as coffee. Users can quickly identify which compounds are largely responsible for the aroma of a given coffee and what are the key differences and similarities between different coffees. The system that produces all this data would be at home in both a development laboratory or in a QC environment.

Acknowledgements

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