Novel Aroma Profiling of Coffee by Thermal Desorption with GC/MS

Application Note
Food and Beverage

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Abstract
This application note presents a novel study of coffee aroma profiling by analyzing volatile organic compounds (VOCs) emitted at a series of progressively higher temperatures.

Introduction
Coffee has surpassed Tea in popularity during the past decade and become the most popular non alcoholic beverage in the world. The earliest credible evidence of coffee drinking could be traced back to the late 15th century in Ethiopia. Throughout the spread of coffee drinking in the last 5 hundred years, varieties of commercial coffee products emerged on the market. The preference of a specific coffee product by the consumer is determined by the aroma and taste, where the former factor could be categorized as flowery, nutty, smoky and herby, while the latter factor includes acidity, bitterness, sweetness, saltiness and sourness. Previous study[1] has correlated 28 volatile organic compounds (VOCs) from coffee to the first determining factor - aroma. The traditional method[2] to map the aroma profiling is using the headspace sampling technique to count the integrated peak areas for the target 28 VOCs at a fixed temperature. The drawbacks of this method are (1) due to the complex matrices in coffee, target VOCs may possess low concentration and overlap with many high concentration compounds without sufficient separation; (2) the aroma profile built at a single temperature point may not dynamically represent the principal components shift along with the coffee temperature change, since the kinetic of thermal extraction on a specific VOC varies by temperature. In this application note, a novel aroma profiling analysis is performed by studying volatile organic compounds (VOCs) emitted from a coffee sample at progressively higher temperatures instead of a fixed temperature.

Experimental Setup
A 10 mg coffee sample was loaded into an empty Camsco quartz tube and packed with quartz wool. The sample was then inserted into the sample rack system on a CDS 7550S Thermal Desorption autosampler with a Shimadzu GC/MS QP-2010 for analysis. The sample was thermally extracted in a series of temperature points that were programmed by the 7550S, which provided a precise temperature control algorithm to desorb the sample from 50 °C to 130 °C with a temperature increment as small as 5 °C. The desorption temperatures were 50, 55, 60, 65, 70, 75, 80, 85, 90, 95, 110 and 130 °C. The emitted VOCs from the sample were collected on the 2nd analytical trap, which was desorbed to the GC/MS for analysis to improve focusing.coupled to a GC/MS system.

7550S Thermal Desorber:

Valve oven:	220 °C	Desorb temp.:	50, 55, 60, 65, 70, 75, 80, 85, 90, 95, 110 and 130 °C
GC transfer line:	250 °C		
Tube purge flow:	30 mL/min	Desorb time:	10 min
Pre-heat time:	15 s	Trap:	
Tube desorber:		Rest temp.:	30 °C
Rest temp.:	35 °C	Desorb temp.:	300 °C
Dry purge temp.:	35 °C	Desorb time:	3 min
Dry purge time:	2 min for first run only		

GC:
Oven Temp.: 40.0 °C
Injection Temp.: 230.00 °C
Injection Mode: Split
Carrier: Helium, 1.01mL/min
Split Ratio: 10.0
Oven: 40°C for 1 minutes
40°C/min to 120°C
16°C/min to 320°C
Hold 3.5 min

MS:
Ion Source Temp: 200.00 °C
Interface Temp: 250.00 °C
Solvent Cut Time: 2.00 min
ACQ Mode: Scan
Event Time: 0.30sec
Scan Speed: 1666
Start m/z: 29.00
End m/z: 450.00

Results and Discussion

Total of twelve chromatograms were generated at progressively higher temperatures. Six chromatograms were picked and shown in Figure 1. The aroma compounds detected were listed in the order of retention time as Acetic acid, Acetic anhydride, Pyrazine, Pyridine, Methylpyrazine, Furfural, 2-Furanmethanol, Acetoxyacetone, Butyrolactone, 2,3-Pentanedione, 5-Methylfurfural, 2-Vinylfuran, Furfuryl acetate, N,N-Dimethyl-4-aminopyridine, 1H-Pyrrole-2-carboxaldehyde, 2-Acetylpyrrole, 2-Furanglyoxylonitrile and Varamol. The odorless and tasteless caffeine was also analyzed as a control.

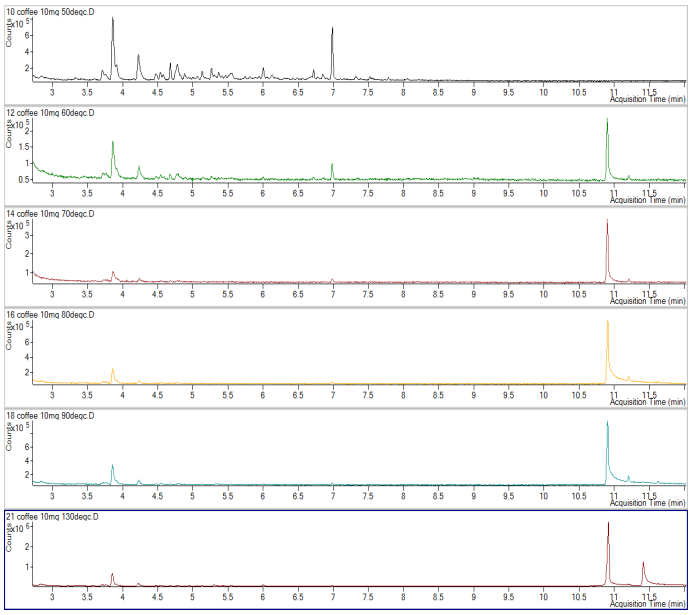

Figure 1: Selected chromatograms from the multiple desorption of a coffee sample

By further analyzing the twelve chromatograms, a desorption curve was plotted for each VOC as showing the integrated peak areas of each run as well as the accumulated integrated peak areas from pervious runs against the sampling temperature. Figure 2 is the desorption curve of 2-furanmethanol, an important aroma in the furan family. The shape of the accumulated integrated peaks suggested a two-phase desorption process, where this compound would establish a stable thermal equilibrium between 50 °C and 70 °C. Once the thermal desorption temperature went beyond 70 °C, the equilibrium was broken with much more compound released. This implied a potential aroma profiling shift while the coffee cools down.

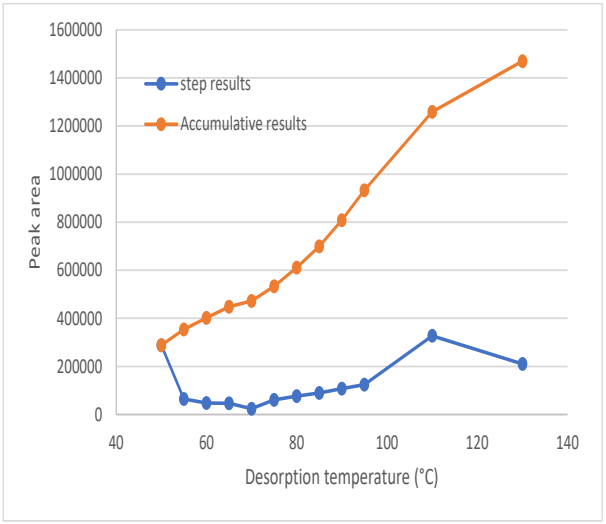

Figure 2: Desorption curve of 2-furanmethanol (RT = 3.857 min) from the coffee sample

By listing the accumulated integrated peak areas for each aroma compound, the aroma profile was drawn in Figure 3. The caffeine was shown in the data as a control point. The significance of the comparison with caffeine was that most of the aroma compounds had much lower content comparing to caffeine. If a single thermal slice was measured at a high temperature point without completing the multiple-step desorption, the capillary GC column would be easily overloaded without yielding meaningful data.

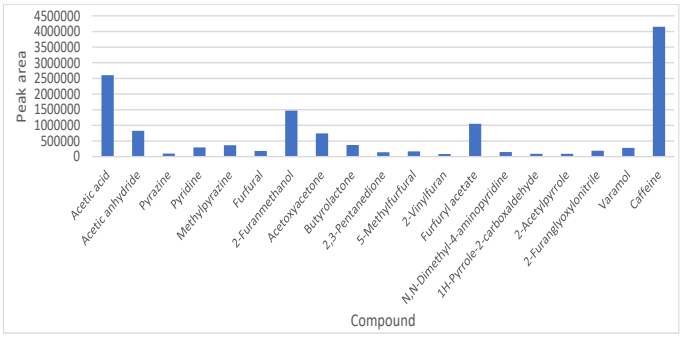

Figure 4: Aroma profile with caffeine as the comparison compound

Conclusions:

A series of thermal extractions on a 10 mg coffee sample were studies from 50 °C to 130 °C using a CDS 7550S Thermal Desorption system. The precise temperature control feature allowed the increment on sampling temperature to be as small as 5 °C, which leads to a novel method of mapping the aroma profile to address the issues from traditional studies.

References:

1. Blank, I.; Sen, A.; Grosch, W. Aroma impact compounds of Arabica and Robusta coffee. Qualitative and Quantitative investigations. ASIC, 14th Colloque, San Francisco, CA, 1991; pp 117-129.

2. Laura Maeztu, Cristina Sanz, Susana Andueza, M. Paz De Peña, José Bello, and Concepción Cid. Characterization of Espresso Coffee Aroma by Static Headspace GC–MS and Sensory Flavor Profile. Article in Journal of Agricultural and Food Chemistry 49(11):5437-44 · December 2001