TELEDYNE INSTRUMENTS
Tekmar
A Teledyne Technologies Company

Dynamic Headspace Analysis of Coffees and Teas

HT3 Application Note

Introduction ▼

Coffee and tea are more than just caffeine, as both multi-billion dollar businesses will attest. The unique flavors and aromas associated with foods and beverages are due to the presence of complex combinations of volatile organic compounds. In this analysis, there is no single compound that gives a food product it's distinctive flavor and smell, hence, the desire to have an even more comprehensive flavor profile for each product.

Although coffee is more than caffeine, it is important to be able to analyze the semi-volatile caffeine in beans, as EC regulations require the amount of caffeine in a decaffeinated beverage to be less than 0.1% (by dry weight). The decaffeination process may involve the use of a class-2 solvent and then that should be analyzed as well. It is also important to know the levels of the volatile aroma compounds because of the potential of coffee fraud. The adulteration of high quality coffee beans mixed with poorer ones can lead to an inferior product, yet the buyer could not be sure without analyzing the volatile aromas.

Headspace analysis of volatile compounds offers many benefits. Some of those being are a cleaner analysis, potentially quicker run times, and elimination of possible system contamination from high concentrated samples. By incorporating EPA approved trapping techniques, the HT3 Headspace sampler is now capable of offering purge & trap analytic performance. This application note will describe the methodology and present a comprehensive compound list for each caffeinated beverage.

Experimental ▼

The coffees and teas were analyzed via the Dynamic Headspace option from the HT3, which provides significantly lower detection limits than traditional Static Headspace analysis. The Dynamic option continually sweeps the headspace of the vial, depositing and concentrating the volatile compounds onto an analytical trap. Several commercially available coffees and teas were analyzed. Each sample weighed 150 mg and was then placed into vials with or without 5 mL of D.I. water. No further method was used to extract the volatile compounds. These compounds were analyzed via the Teledyne Tekmar HT3 coupled to a GC/MS unit and the parameters of both are in the following tables. A vocarb trap was used for the Dynamic Headspace trapping.

Table 1: HT3 Dynamic Headspace Parameters

Variable	Value
Headspace Vials	22 mL, PTFE Silicone Septa
GC Cycle Time	35.00 min.
Valve Oven Temp.	155 °C
Transfer Line Temp.	155 °C
Standby Flow Rate	50 mL/min.
Trap Standby Temp.	30 °C
Platen/Sample	85* or 150 °C for 10.00 min.
Preheat Mixing	Level 5, 2.00 min.
Preheat Mixer Stabilize Time	0.50 min.
Sweep Flow	40 mL/min. for 10.00 min.
Trap Sweep Temp.	0 °C
Dry Purge	50 mL/min. for 1.00 min.
Dry Purge Temp.	25 °C
Desorb Preheat	220 °C
Desorb	250 °C for 2.00 min.
Trap Bake Temp.	300 °C
Trap Bake	400 mL/min. for 5.00 min.

* Water Matrix Conditions (vs. Dry)

Table 2: GC/MS Parameters

Variable	Value
Column Type	J&W DB-VRX 60 m x 0.25 mm x 1.4 µm film
Column Oven and Injection Temp.	40 °C; 260 °C
Pressure and Split Ratio	19.6 psi; 60:1
Total Flow of Carrier Gas	He @ 75.6 mL/min.
Oven Temperature Program	40 °C (2.0 min.) → 259 °C @ 15 °C/min. → 260 °C @ 1 °C/min. (12.4 min.)
Ion Source Temperature	220 °C
Scan Time and Speed	0.50 - 30.00 min.; 1000
Detector Gain and Mass Range	1.0 kV; 35 - 350

Results ▼

The chromatograms show the ability of the dynamic option to further resolve food volatiles and semi-volatiles. The typical aldehydes, ketones, carboxylic acids, and furans were easily observed. Interestingly, caffeine repeatedly did not show significant levels as a solid matrix for caffeinated coffee while all of the other samples did show significant levels, but when in a water matrix, the caffeinated coffee produces the largest (significantly) amount of caffeine. The changing from a dry to a wet matrix corresponded with an average decrease of nearly 50% in volatile peaks among all 8 samples.

Figure 1: Dry Caffeinated Coffee

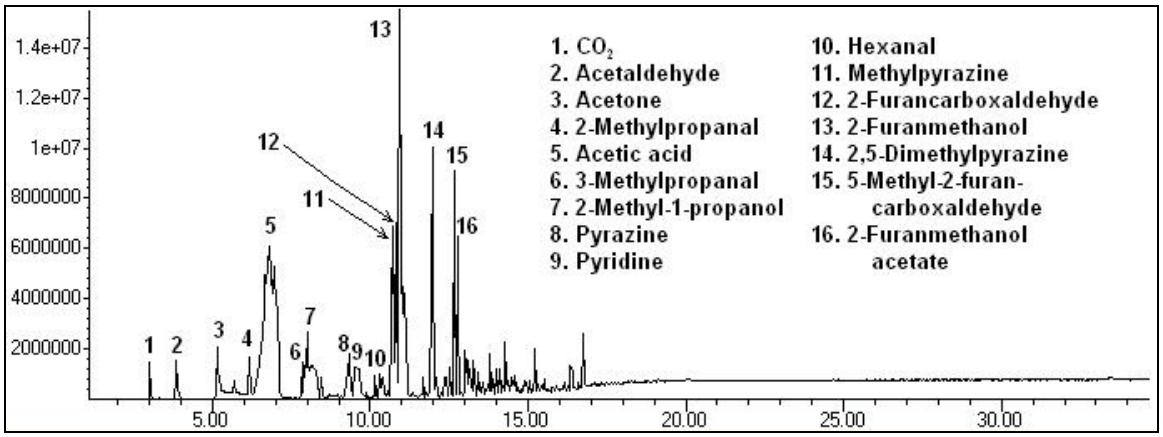


Figure 2: Wet Caffeinated Coffee

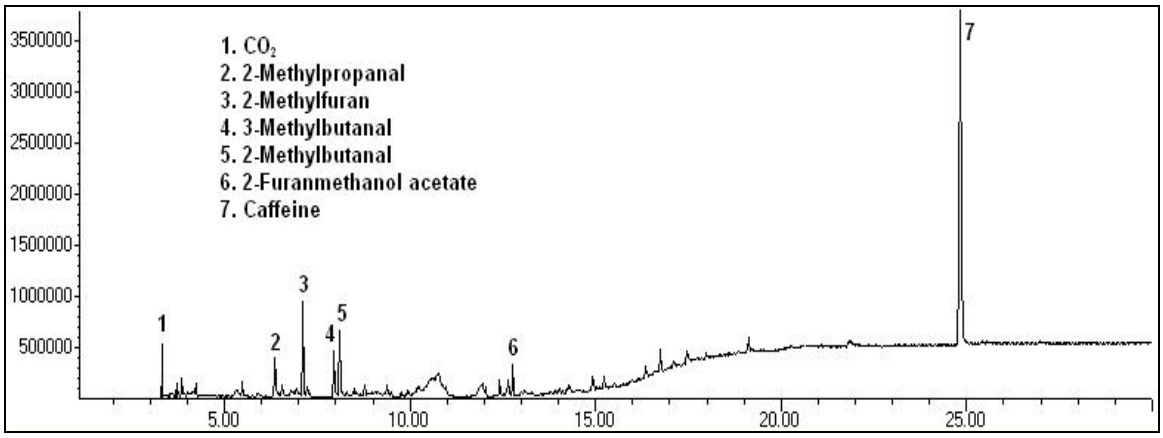


Figure 3: Dry Tea

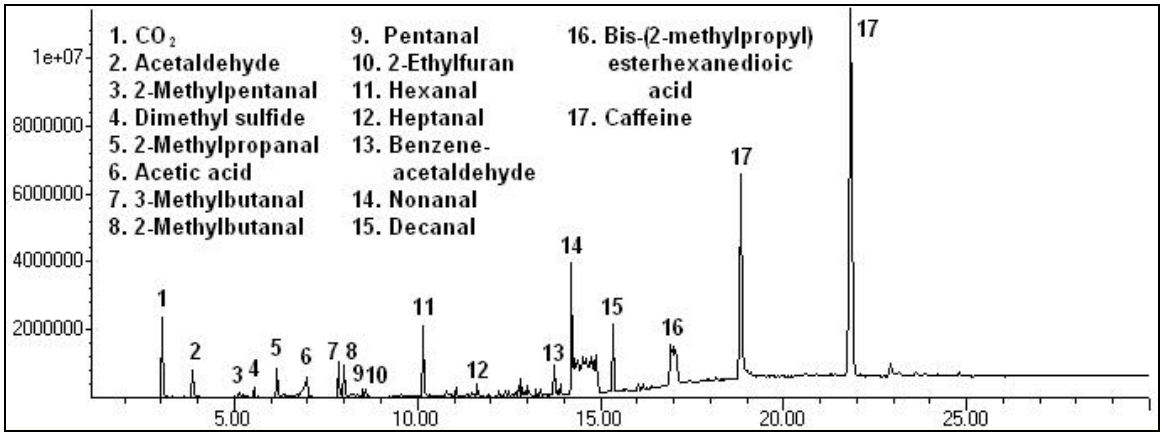


Figure 4: Wet Tea

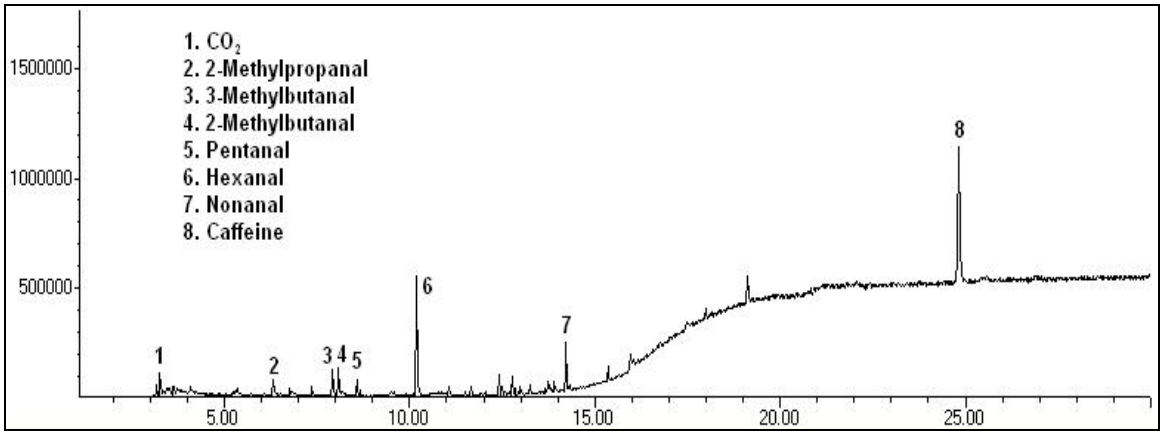


Figure 5: Dry Vanilla Tea

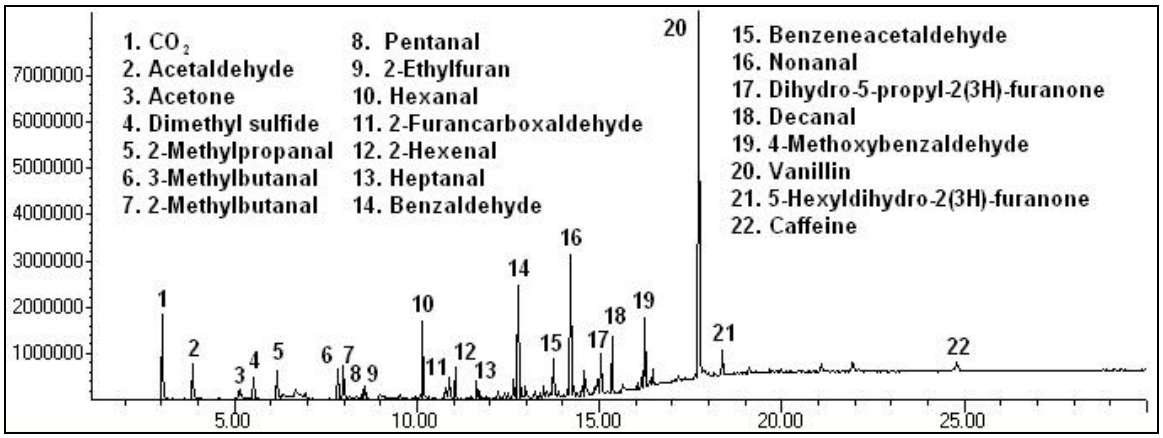
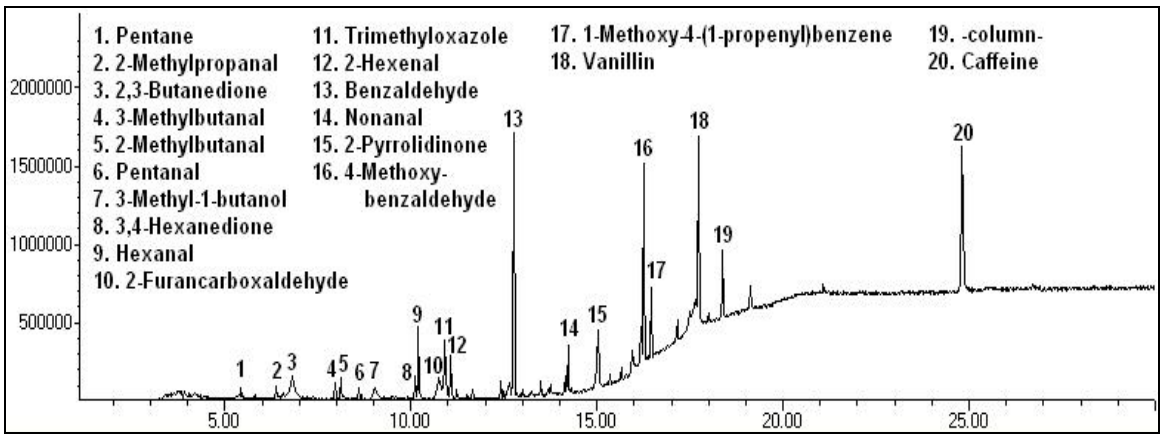
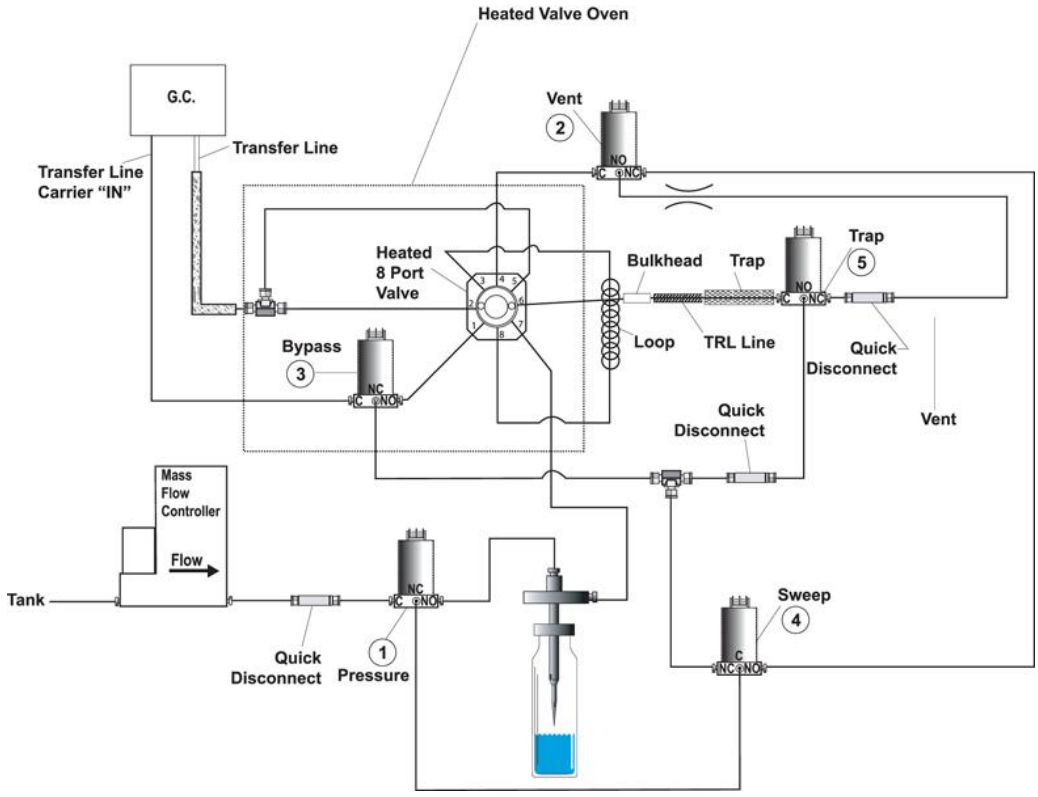


Figure 6: Wet Vanilla Tea





Conclusion ▼

The increased sensitivity of the trapping technology once only used by classic purge and trap techniques, allows for a broader analysis and increased resolution of the volatiles in food and beverages. Not only is sample preparation simple, the elimination of any extraction procedures makes this a cleaner sample introduction technique which reduces the need for injection port maintenance and column replacement. The HT3 Headspace autosampler has brought headspace sampling to a new level. By combining a Mass Flow Controller, Pressure Transducer, Temperature Control and Trapping System, the HT3 is capable of analyzing very difficult samples with ease.