TELEDYNE INSTRUMENTS
Tekmar
A Teledyne Technologies Company

The Analysis of Commercial Beers by Dynamic Headspace

HT3 Application Note

Introduction ▼

In the analysis of alcoholic beverages such as beer, there are three main categories of volatile analysis: alcohols / esters, aldehydes, and sulfur compounds. The sulfur compounds are an impurity by-product of the fermentation process that typically requires an additional detector to the GC or at least a sulfur specific column. 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. The focus of this paper is on alcohols and esters. Largely, the chemical makeup of beer comes from its raw materials of production (i.e. malted barley, hops, and water) or during its alcoholic fermentation process; therefore it is important to be able to analyze the product pre-fermentation and post-fermentation. In that same breath, the malt and yeast are significant characters in beer, but equally significant is the quality of both the water and hops. Regulating the alcohol levels is essential, not just because there are alcoholic content requirements, but also because they are the immediate precursors to the more flavor-active esters. Similarly, regulating acetaldehyde levels is critical as consistently high levels could indicate poor yeast quality.

Headspace analysis of volatile compounds offers many benefits, such as simpler sample preparation and a cleaner injection technique for the GC which reduces 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 alcoholic beverage.

Experimental ▼

The beers 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 beers were analyzed. Each sample contained 2 mL of beer. No further sample preparation techniques were 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 dynamic trapping purposes.

Table 1: HT3 Dynamic Headspace Parameters

Variable	Value
Headspace Vials	22 mL, PTFE Silicone Septa
GC Cycle Time	35.00 min.
Valve Oven Temp.	120 °C
Transfer Line Temp.	120 °C
Standby Flow Rate	50 mL/min.
Trap Standby Temp.	30 °C.
Platen/Sample	30 °C for 10.00 min.
Preheat Mixing	Off.
Preheat Mixer Stabilize Time	--
Sweep Flow	50 mL/min. for 8.00 min.
Trap Sweep Temp.	Ambient
Dry Purge	50 mL/min. for 1.00 min.
Dry Purge Temp.	25 °C
Desorb Preheat	200 °C
Desorb	220 °C for 2.00 min.
Trap Bake Temp.	250 °C
Trap Bake	400 mL/min. for 10.00 min.

Table 2: GC/MS Parameters

Variable	Value
Column Type	Rtx-1301 60 m x 0.32 mm x 1.5 µm film
Column Oven and Injection Temp.	40 °C; 260 °C
Pressure and Split Ratio	5.30 psi; 60:1
Total Flow of Carrier Gas	He @ 75.6 mL/min.
Oven Temperature Program	40 °C (2.0 min.) → 180 °C @ 12 °C/min. → 260 °C @ 20 °C/min. (3.0 min.)
Ion Source Temperature	220 °C
Scan Time and Speed	0.50 – 20.67 min.; 1000
Detector Gain and Mass Range	1.0 kV; 35 - 350

Results ▼

To preserve the volatile integrity of the analysis, the samples had to be transferred to a sealed vial immediately after opening the container. The chromatographs show the ability of the dynamic option to further resolve food volatiles and semi-volatiles. The typical alcohols and fatty acid methyl esters were easily observed. Heating the sample at 30 °C for 10 minutes yielded the highest sensitivity and stability.

Figure 1: Canned Domestic Beer

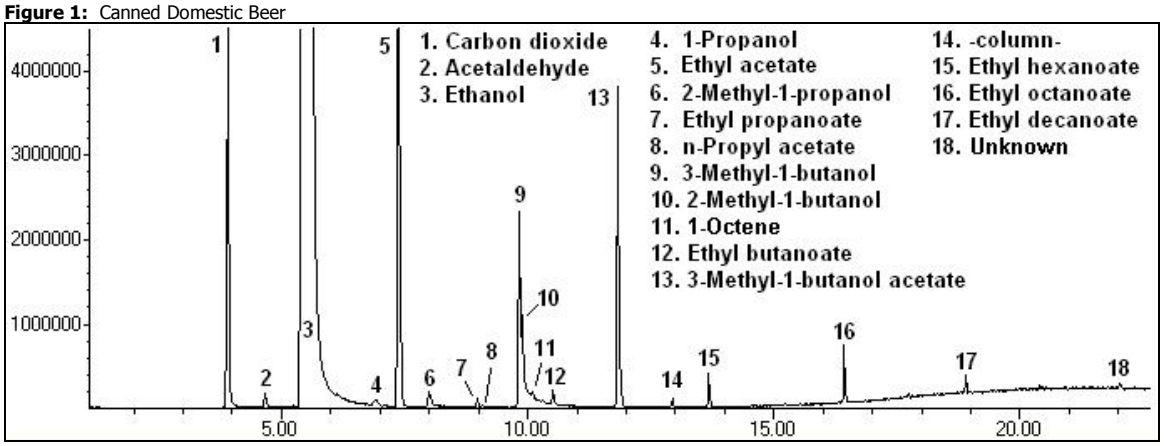


Figure 2: Canned Import Beer

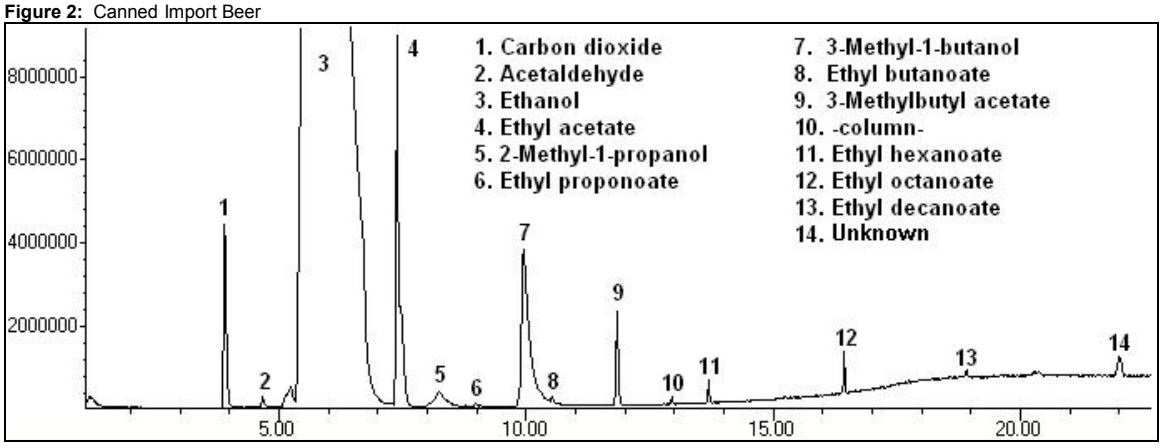


Figure 3: Canned Domestic Light Beer

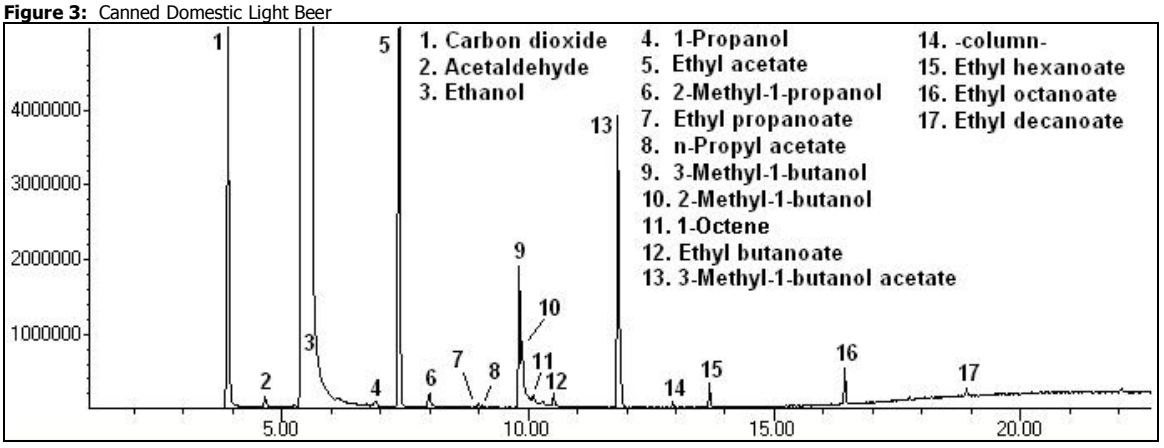


Figure 4: Canned Stout

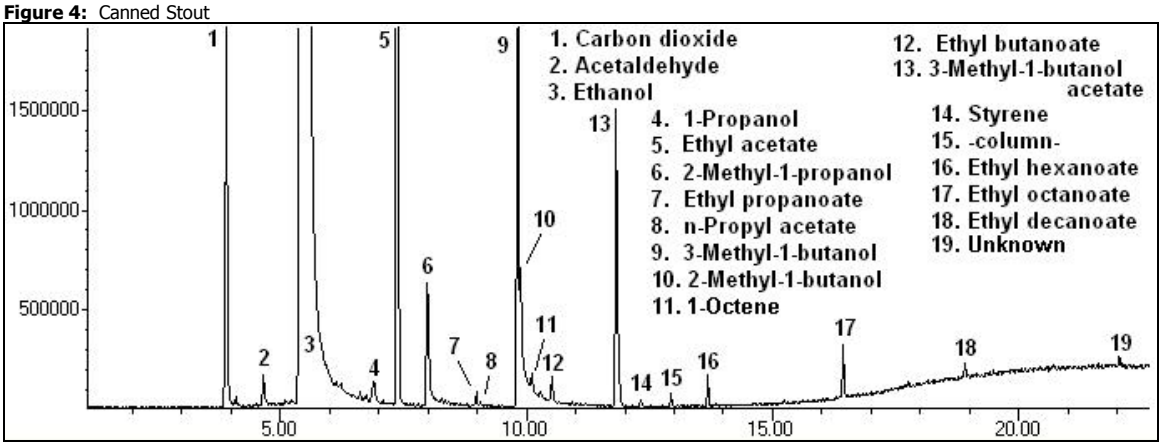
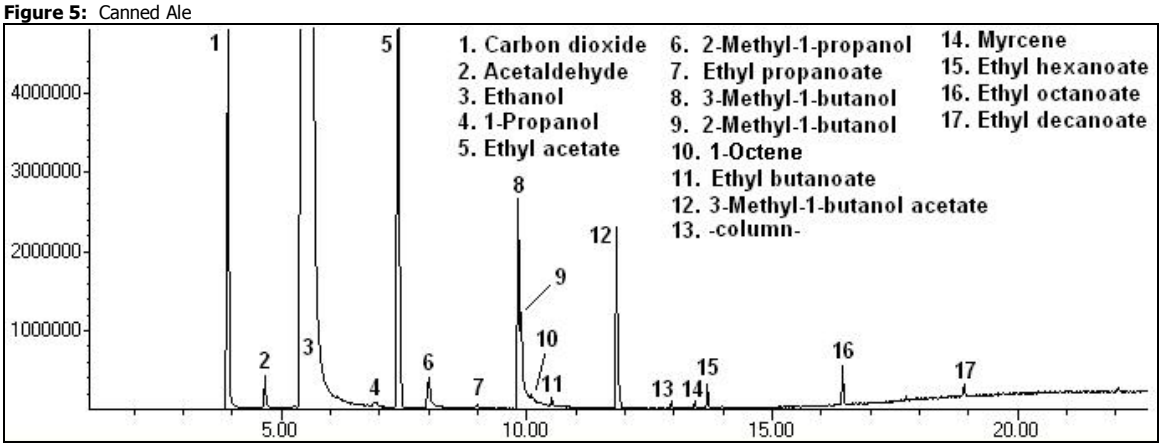
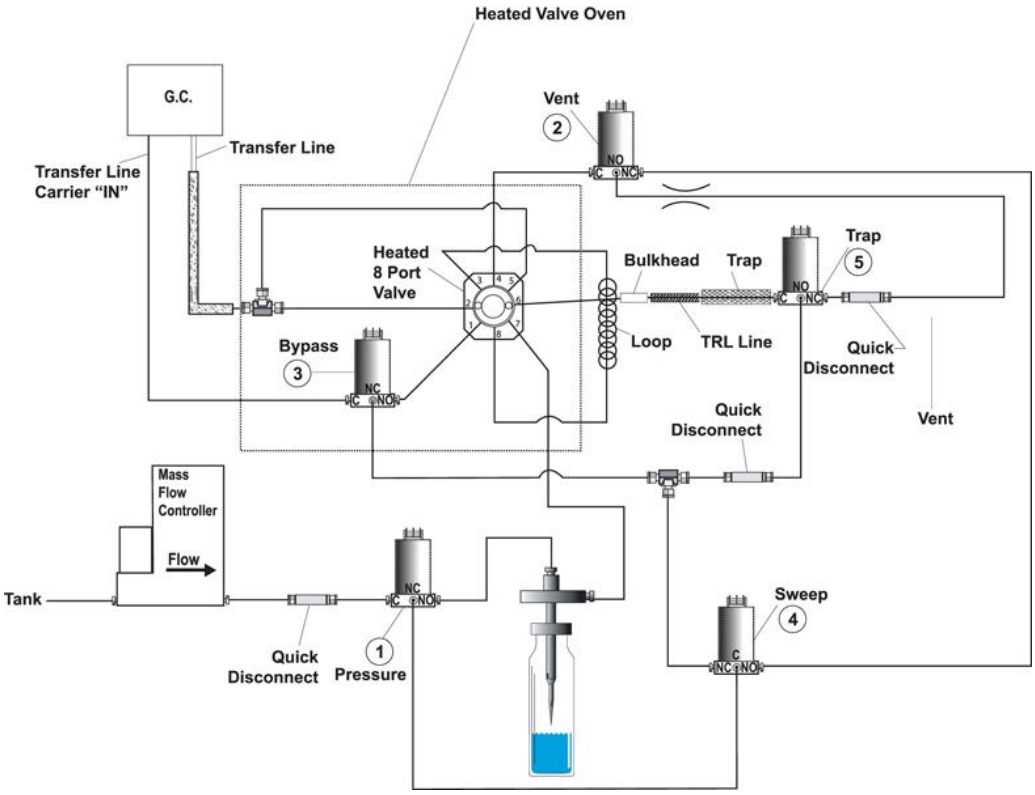


Figure 5: Canned Ale





Conclusion ▼

The increased sensitivity of the headspace trapping technique allows the analyst to achieve detection limits previously only achievable by classic purge and trap instrumentation. Not only is sample preparation simple, but also the Dynamic Headspace sampling allows one the freedom of choosing smaller sample volumes that provides detailed results. 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.