TELEDYNE INSTRUMENTS
Tekmar
A Teledyne Technologies Company

Profiling of Volatile Organic Compounds in Milk and Orange Juice Using Headspace Analysis

HT3 Application Note

Introduction ▼

Volatile organic compounds, VOCs, present in liquid food products play a very important role in flavor and aroma characteristics. The monitoring of these VOCs are extremely important when controlling a manufacturing process, investigating adulteration, and determining product quality. In the past, a static headspace technique wouldn't yield enough data to make testing practical. Therefore a purge and trap technique was employed, but the main problem faced is having to deal with the moisture.

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. Until now, typical headspace samplers could not achieve the needed low-end detection required by the testing laboratory.

A new headspace sampler has been developed for the testing of VOCs and is capable of unprecedented low-end detection. By incorporating EPA approved trapping techniques, the HT3 Headspace sampler is now capable of offering purge & trap analytic performance.

Experimental ▼

The milk and orange juice were analyzed via the Dynamic Headspace option from the HT3, which provides significantly lower detection limits. The Dynamic option continually sweeps the headspace of the vial, depositing and concentrating the volatile organic compounds onto an analytical trap. Commercially available samples were analyzed. The samples were kept refrigerated prior to being pulled for sampling. A 5ml sample was sealed in a 22ml vial. Several variables were experimented with while determining a suitable method; those being sample pretreatment temperature, hold time before analysis, sweep time and flow rate, and GC profile. A vocarb trap was utilized for the dynamic trapping.

Table 1: HT3 Dynamic Headspace Parameters

Variable	Value
Headspace Vials	22 mL, PTFE Silicone Septa
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

Conclusion ▼

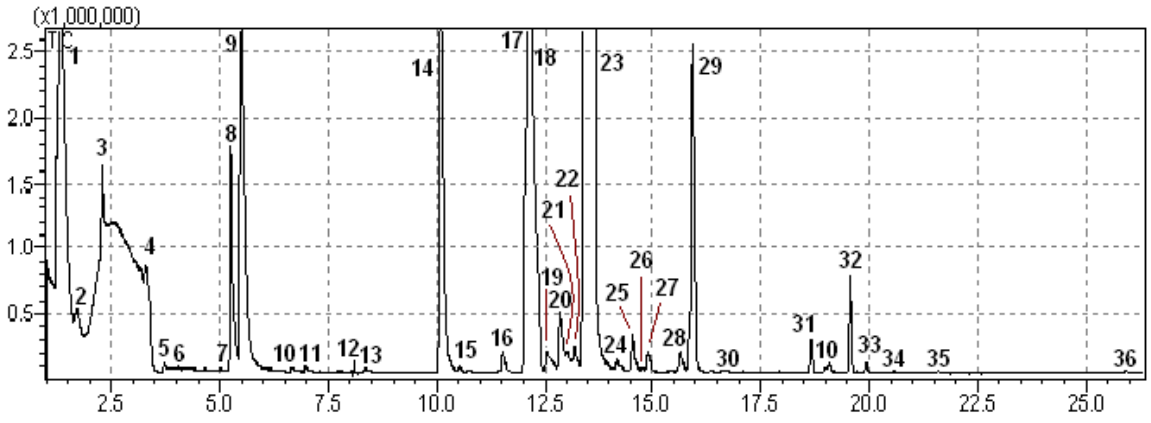
In regards to dairy products and orange juice, the volatiles present are in a complex mixture. By monitoring the absolute amount and proportional levels of several volatile compounds present, conclusions can be made as to final product acceptability, distinguish product adulteration or impurity contamination, and flagging any off-odor characteristics.

The critical conditions that must be controlled when performing headspace analysis are pressure, flow and temperature. All three of these criteria are controlled, regulated, and modified with extreme precision with the HT3. The next step to help increase sensitivity is to incorporate the trapping technology once only used by classic purge and trap techniques. With all of these improvements, the method developed on the HT3 achieved unprecedented headspace analysis.

The Dynamic Headspace sampling technique offers several unique advantages over Static Headspace including greater sensitivity and the detection of a wider range of volatile organics. It is also up to 100 times more sensitive as compared to the static headspace technique.

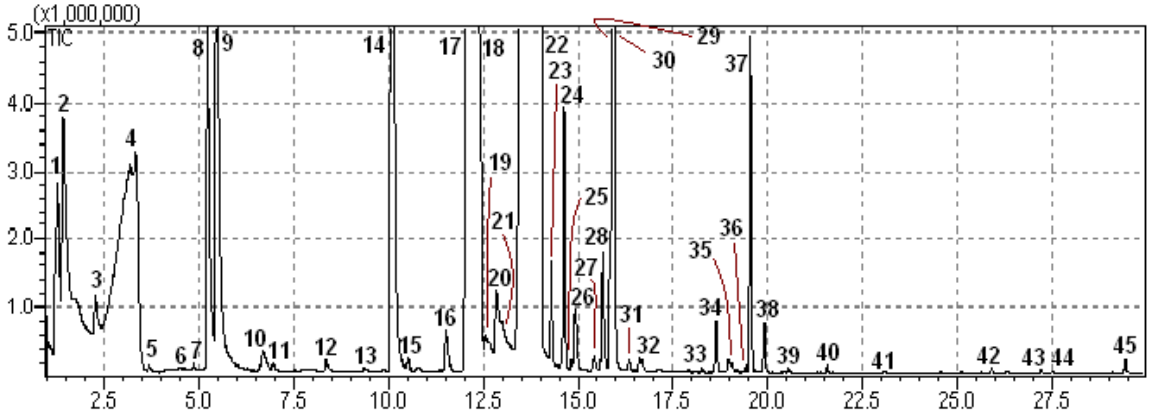
The HT3 Headspace sampler has brought headspace sampling to a new level. By combining a Mass Flow Controller, Pressure Transducer, Temperature Control and Trapping System, the HT3 is capable of analyzing very difficult samples with ease.

Figure 1: Orange Juice from Concentrate



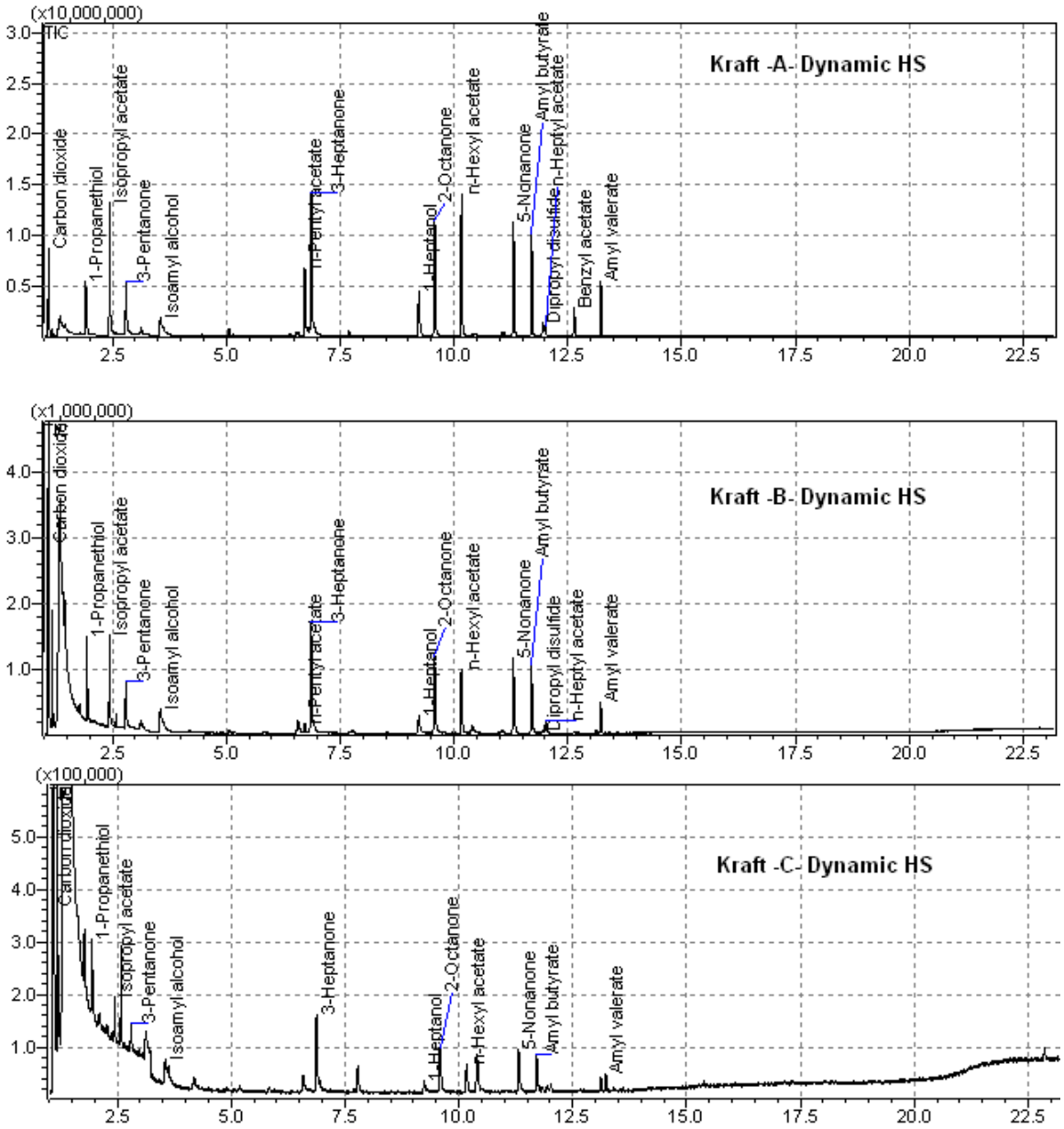
1. Acetaldehyde
2. Acetone
3. Chloroform
4. Ethanol
5. Methyl butanoate
6. Dimethyl disulfide
7. Dibromochloro-methane
8. Hexanal
9. Ethyl butanoate
10. -column-
11. Ethyl-2-methyl-butanoate
12. Argon
13. 3-Methylbutanal
14. a -Pinene
15. Camphene
16. b -Pinene
17. Octanal
18. Myrcene
19. a -Phellandrene
20. 3-Carene
21. 4-carene
22. -column-
23. Limonene
24. b -Ocimene
25. g -Terpinene
26. Isopulegol
27. 1-Octanol
28. 2-Carene
29. Linalool
30. Farnesol
31. *trans*-Sabinene-hydrate
32. Decanal
33. Octyl acetate
34. Carvone
35. Perillal
36. Copaene

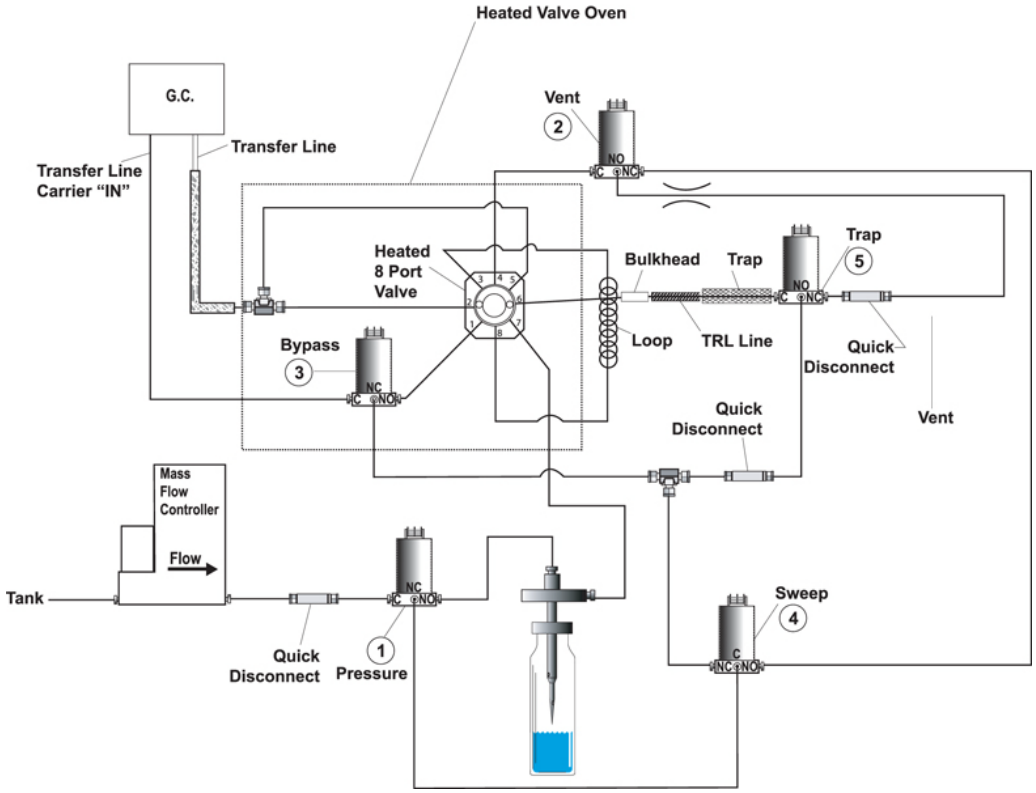
Figure 2: Orange Juice not from Concentrate



1. Dimethylamine
2. Acetaldehyde
3. Ethyl acetate
4. Ethanol
5. Methyl butanoate
6. Toluene
7. 1-Methylcyclohexene
8. Hexanal
9. Ethyl butanoate
10. 2-Hexenal
11. Ethyl-2-methylbutanoate
12. Heptanal
13. Methyl hexanoate
14. a -Pinene
15. Camphene
16. b -Pinene
17. Octanal
18. b -Myrcene
19. a -Phellandrene
20. 3-Carene
21. 4-Carene
22. Limonene
23. b -Ocimene
24. g -Terpinene
25. 2-Nonen-1-ol
26. 1-Octanol
27. Isopropyltoluene
28. Terpinolene
29. Nonanal
30. Linalool
31. Farnesol
32. 4-Acetyl-1-methyl-cyclohexene
33. Isopulegol
34. *trans*-Sabinene-hydrate
35. -column-
36. Ethyl octanoate
37. Decanal
38. Octyl acetate
39. Carvone
40. Perillal
41. Undecanal
42. Copaene
43. Caryophyllene
44. a -Cubebene
45. Guaia-3,9-diene

Figure 3: Dairy Products




Heated Valve Oven
G.C.
Transfer Line
Transfer Line
Carrier "IN"
Vent
2
NO
C
NC
Heated
8 Port
Valve
Bulkhead
Trap
Trap
5
Bypass
3
NC
Loop
TRL Line
Quick
Disconnect
Vent
Quick
Disconnect
Mass
Flow
Controller
Flow
Tank
Sweep
4
Quick
Disconnect
1
Pressure