

## Dynamic Headspace Analysis of Fruit-Flavored Cereal

### Application Note

Food and Flavor

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Dynamic headspace is a simple and sensitive technique for the evaluation of volatile compounds from a wide variety of samples. It differs from static headspace in that the atmosphere around the sample is purged to a trap, so that the volatiles from a very large headspace sample may be injected for a single assay, increasing the sensitivity of the analysis. Foods in particular may be gently warmed in a chamber which is purged to remove the volatiles and collect them onto the sorbent trap, which is then thermally desorbed to the GC.

Figure 1 shows the chromatogram of volatiles collected from a fruit-flavored breakfast cereal. For the analysis, a 250 ml beaker was filled with the cereal, and placed into an 800 ml purging chamber. The sample was heated to 50°C, allowed to equilibrate, then the vacuum pump of the Dynatherm 9350 transferred the volatiles at 50 ml/min to the collection trap. After sampling for ten minutes, the volatiles were desorbed to the focusing trap, which was in turn desorbed to the gas chromatograph. No solvents were used, and sample-prep involved only filling the beaker with the cereal and placing it into the sampling chamber. The entire process, including sampling time, flow rate, trap desorption and starting the gas chromatograph, is automated.

Table 1 lists the most prominent aroma compounds collected from the cereal. Compounds include aldehydes like hexanal and decanal, terpenes like pinene and limonene, alcohols, acetic acid and butylated hydroxytoluene (BHT), an antioxidant.

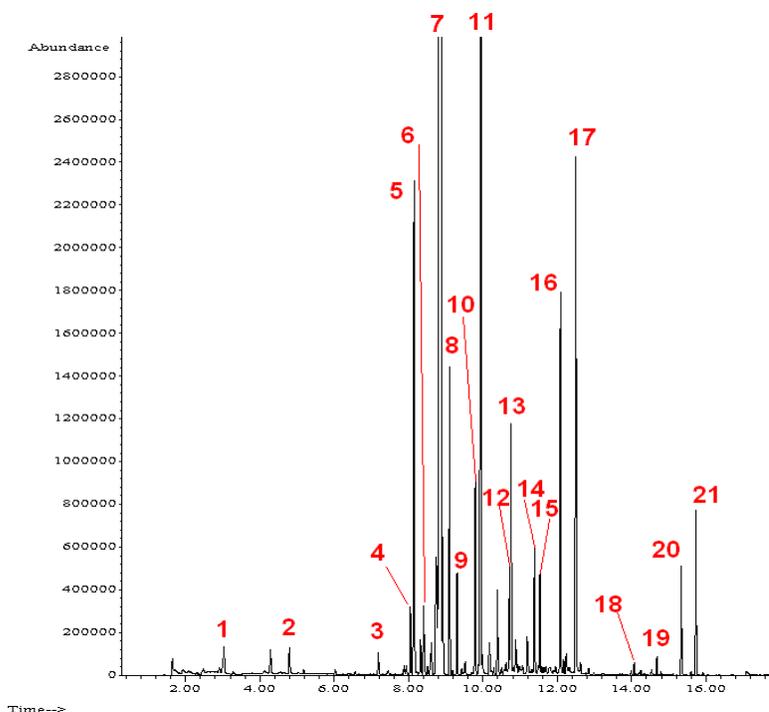


Figure 1. Volatiles from cereal at 50°C.

## Instrument Conditions

### Dynatherm 9350

Sampler Method: Vac 10 Min  
Sample Tube: AO062731 (20:35 Tenax-TA/Carboxen 1000/Carbosieve SIII)  
Focus Trap: MX062234 (60:80 mesh Tenax-TA/Carboxen 1000/Carbosieve SIII)

Operating Mode: Inject on A  
Valve Oven: 300°C  
GC Xfer Line: 300°C  
Sampling Line: 300°C  
Tube Maximum: 350°C  
Tube Idle: 45°C  
Sampling: 10.0 Minutes @ 0.05 Liters/Minute  
Dry Tube: 0.0 Minutes  
Tube Heat: 300°C 3.0 Minutes  
Trap Maximum: 350°C  
Trap Idle: 40°C  
Trap Heat: 300°C 4.0 Minutes

### Gas Chromatograph

#### OVEN

Initial temp: 40 °C  
Initial time: 2.00 min  
Equilibration time: 0.50 min

#### Ramps:

#	Rate	Final temp	Final time
1	10.00	300	5.50

Run time: 35.00 min

#### FRONT INLET

Mode: Split  
Initial temp: 300 °C (On)  
Pressure: 7.00 psi (On)  
Split ratio: 50:1  
Split flow: 49.7 mL/min  
Total flow: 53.6 mL/min  
Gas saver: Off  
Gas type: Helium

#### COLUMN

Capillary Column: HP-5MS 5% Phenyl Methyl Siloxane  
Nominal length: 30.0 m  
Nominal diameter: 250.00 um  
Nominal film thickness: 0.25 um  
Constant pressure: 7.00 psi

### TABLE 1

1. Acetic Acid
2. Hexanal
3. alpha Pinene
4. 6-Methyl-5-heptene-2-one
5. beta-Myrcene
6. p-Cymene
7. Limonene
8. beta-cis-Ocimene
9. Terpinene
10. Terpinolen
11. beta-Linalool
12. Isopregol
13. Citronellal
14. alpha-Terpieol
15. Decanal
16. beta-Citral
17. Citral
18. Copaene
19. Caryophyllene
20. beta-Gurjunene
21. Butylated Hydroxytoluene