Dynamic Headspace Analysis of Food Volatiles Coffee and Tea

Application Note
Food & Flavor

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Because the aroma and flavor of foods are due largely to volatile organic compounds, headspace analysis is very popular in food research laboratories. In static headspace analysis, a sample of the food is placed into a sealed vessel and warmed to promote the volatilization of the aroma constituents. A sample of the headspace is then removed with a syringe and injected onto a gas chromatograph for analysis.

Improved sensitivity may be realized using a dynamic head space approach, in which the food sample is constantly swept with a stream of inert gas while it is heated. This permits the collection of a much larger headspace sample than could be injected onto the GC with a syringe, while enhancing the production of volatiles by removing them from the sample chamber as they are evolved. The carrier gas passes through a collecting trap filled with an adsorbent material before it is vented. After collection, the trapped volatiles are backflushed from the trap to the gas chromatograph while the trap is heated. This thermal desorption of the volatiles from the adsorbent eliminates the need for a solvent, so there is no solvent peak in the chromatogram, and no need to analyze only a small portion of the material collected.

The accompanying chromatograms show the volatile compounds which were collected from coffee and tea samples. The instrument used to collect the volatiles was a sample concentrator, equipped with a cryogenic focuser at the injection port of the gas chromatograph. The coffee and tea samples were heated to 75°C and purged with helium for 10 minutes. The evolved organics were collected onto a Tenax trap, which was then dried of moisture before being back flushed to the gas chromatograph. At the GC, the fused silica capillary column was brought up through the injection port and passed through a cryogenic focuser, then attached to the transfer line from the sample concentrator. In this way, the organics for analysis could be refocused onto the capillary column itself prior to starting the GC run, which produced sharp peaks without the use of a splitter at the injection port. In addition to improved peak resolution, this results in greater sensitivity, since all of the sample goes onto the column instead of venting most of it out the splitter vent.

A wide variety of foods have been analyzed using dynamic headspace techniques, including nuts, grains, meats, cereals, cheeses, and spices. Prepared foods such as breads, cookies and potato chips have also been studied. Using a liquid sampling attachment, the same technique may also be applied to beverages, including fruit juices, soft drinks, beer and wine.

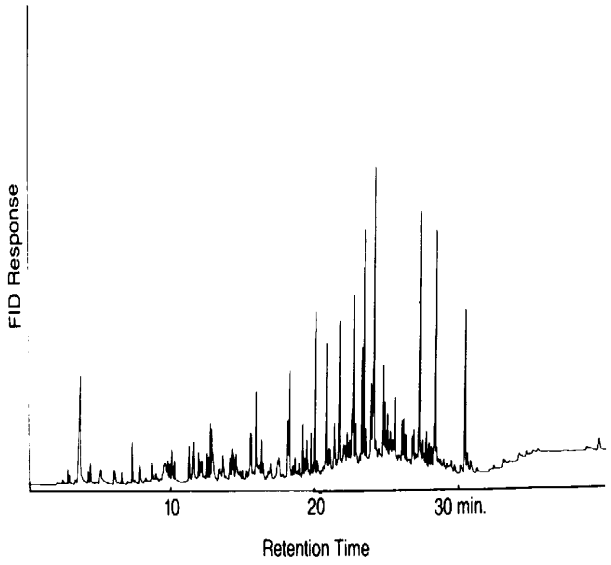

Figure 1: Dynamic Headspace of Tea Leaves, 75°C for 10 minutes.

Equipment:
DYNAMIC HEADSPACE
Sample Concentrator equipped with Tenax internal trap and cryogenic focuser at the gas chromatograph.

Thermal Desorption:	75°C and 100°C
Sample Flow:	Helium at 30ml/min
Trap Dry:	3 minutes at 35°C
Trap desorption:	250°C for 10 minutes
Cryogenic focuser:	-100°C for 10 minutes, then 250°C for 5 minutes

GC Conditions:
Varian 3700 equipped with FID
Column: 50m x 0.25mm SE-54 fused silica capillary
Program: 50°C for 2 minutes, then 6°C/min to 275° C

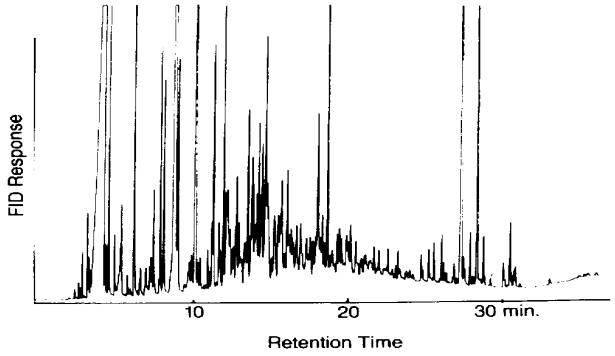

Figure 2: Dynamic Headspace of Roast Ground Coffee, 75°C for 10 minutes.

For more information on this and related applications, we recommend the following readings:

A. Zlatkis, S. Weisner, and L. Ghaoui, “Recent Develop ments in Gas Chromatographic Trace Analysis Using New Concentrator Techniques,” Texas Journal of Science, Vol. XXXVII, No. 4, Dec., 1985.

T. Wampler, W. Bowe, and E. Levy, “Splitless Capillary GC Analysis of Herbs and Spices using Cryofocusing,” American Lab., Oct., 1985.

T. Wampler, W. Bowe, J. Higgins, and E. Levy, “Systems Approach to Automatic Cryofocusing in Purge and Trap, Headspace and Pyrolytic Analysis,” American Lab., August, 1985.

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