

Application Note

Food and Flavor

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Headspace sampling of volatiles from food samples is a convenient way of introducing aroma constituents into a gas chromatograph. Traditional or “static” headspace techniques involve placing the food into a sealed container, warming it, and then withdrawing a small portion of the headspace in the vial with a syringe for injection onto the GC. “Dynamic” headspace is an automated process in which the food sample is swept with a stream of inert gas while it is warmed, so that a larger sample of the volatiles may be used. The carrier gas passes through a trap which may be packed with adsorbent material and used at ambient temperatures, or cooled for cryogenic collection. Here the aroma volatiles are trapped while the headspace stream is vented. After collection, the trap is heated and backflushed to the gas chromatograph for analysis of the trapped compounds.

The chromatograms shown here were obtained using a Pyroprobe with automatic cryogenic refocusing onto the GC column. Pieces of citrus peel weighing 10mg were placed into the thermal desorber of the Pyroprobe and heated to 75° C for 10 minutes, while they were swept with helium at a flow of 30ml/min. The resulting volatiles were collected onto a trap filled with Tenax, which was then heated to 250° C and backflushed to the gas chromatograph. Here, the organics were refocused onto the GC column at -100° C by the cryofocuser of the 330. When all of the volatiles had been recollected, the GC program was started automatically, and the cryofocuser heated to 250° C to volatilize the collected compounds. The gas chromatograph was a Varian 3700 equipped with a 50m SE-54 capillary column.

The examples shown compare orange peel to grapefruit peel. Although the two peels contain many of the same compounds - especially the large limonene peak at about 12 minutes, there are some important differences which are easy to see. The relative amounts of the peaks marked 2,3,5, and 8 in the grapefruit, for example, are far greater than those seen in the orange. On the other hand, the amounts of peaks marked 1,6, and 7 are considerably greater in the orange peel. Similar differences may also be seen for lemon and lime peels, as well as for a wide variety of other foods, including coffee, tea, nuts, herbs, meats, vegetables and baked goods.

Equipment: **Pyroprobe with Cryogenic Refocusing**

| | | | |
|------------------|-----------------------|----------------|--------------------|
| Desorption: | 10 min at 75° C | Cryogenic | |
| Sample carrier: | Helium at 30ml/min. | refocusing: | -100° C for 15 min |
| Initial Trap: | Tenax at ambient temp | Valve oven: | 250° C |
| Trap desorption: | 250° C for 15 minutes | Transfer line: | 250° C |

GC Conditions:
Varian 3700 equipped with FID
Column: 50m x 0.25mm SE-54
Program: 50°C for 2 minutes, then 5° C/min to 225° C
Carrier: Helium

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

T. Wampler, W. Bowe and E. Levy, "Splitless Capillary GC Analysis of Herbs and Spices Using Cryofocusing," American Lab., October, 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.

T. Wampler, W. Bowe, and E. Levy, "Dynamic Headspace Analysis of Residual Volatiles in Pharmaceuticals," J. Chrom. Sci., 23, 64-67 (1985).

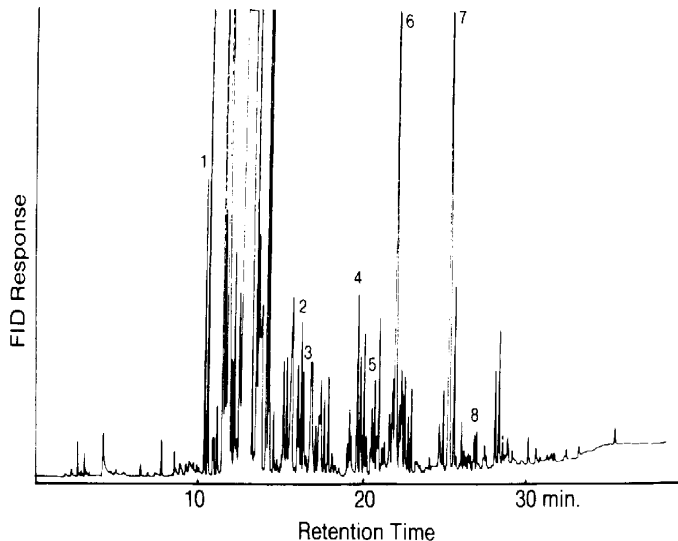


Figure 1: Dynamic Headspace of Orange Peel, 75°C for 10 minutes.

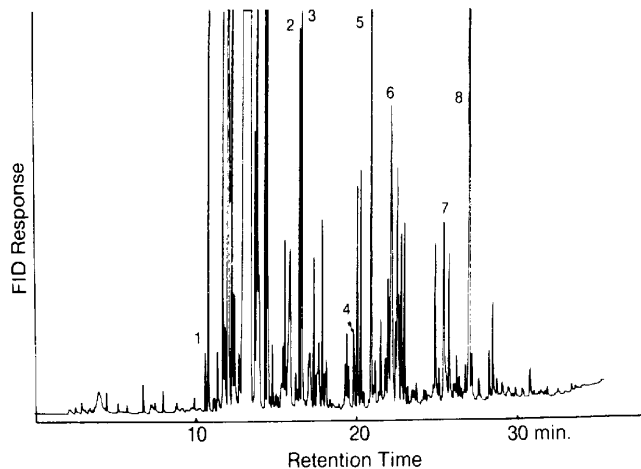


Figure 2: Dynamic Headspace of Grapefruit Peel, 75°C for 10 minutes.