



Dynamic Headspace Analysis of Spices

Application Note

Environmental Industry

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Headspace sampling for gas chromatography is a process by which the volatile organic compounds from a sample are collected and injected onto the GC. In static headspace analysis, the sample is placed into a sealed vessel and warmed to volatilize the organics. After the system has reached equilibrium, a small aliquot of the enclosed headspace is withdrawn and injected onto the GC. For Dynamic Headspace, the sample is placed into a tube which is flushed with carrier gas while the sample is being warmed. The carrier gas is passed through a trap (cryogenic or adsorbent packed) which collects and concentrates the organic volatiles. In this way, the volatiles from much larger headspace samples may be concentrated for injection onto the GC. After the collection is completed, the trap is backflushed with GC carrier gas, and heated to desorb the collected volatiles. For greatest sensitivity and resolution, the organics may then be cryogenically refocused onto the head of the capillary column using liquid Nitrogen.

The examples shown here were obtained using a Chemical Data Systems Sample Concentrator with a packed primary trap (Tenax at room temperature) and a cryogenic refocuser at the injection port of the GC. Single seeds were placed into the thermal desorption tube of the Concentrator and warmed to 100°C while they were flushed with helium. The released organics were collected for 10 minutes onto a Tenax trap, which was then backflushed to the GC and heated to 275°C for 10 minutes. The desorbed volatiles were recollected onto the capillary column at -100°C, then revaporized at the start of the GC program. The capillary column was attached directly to the transfer line of the Concentrator, so no split was used, permitting an increase in the sensitivity since none of the sample was lost through the split vent. The sharpness and resolution of the peaks makes it easy to compare volatile constituents between different spices, such as the black pepper and caraway shown here, and different grades of the same spice, for quality assessment upon receipt of a shipment.

Sample Concentrator Conditions:

Thermal Desorption:	100°C for 10 minutes
Trap Desorption:	275°C for 10 minutes
Cryofocusing:	-100°C for 10 minutes
Valve oven:	275°C
Transfer line:	275°C

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

Chen, E., "Analysis of Volatile Beer & Flavor Compounds by a Dynamic Headspace Entertainment Technique," ASBC Journal 41, 28-31 (1982).

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 Cryo focusing in Purge and Trap, Headspace, and Pyrolytic Analysis," American Lab., August 1985.

GC Conditions:

Varian 3700 equipped with FID

Column: 50m x 0.25mm SE-54

Program: 50°C to 275 at 8°C/minute

Carrier gas: Helium at 20psi

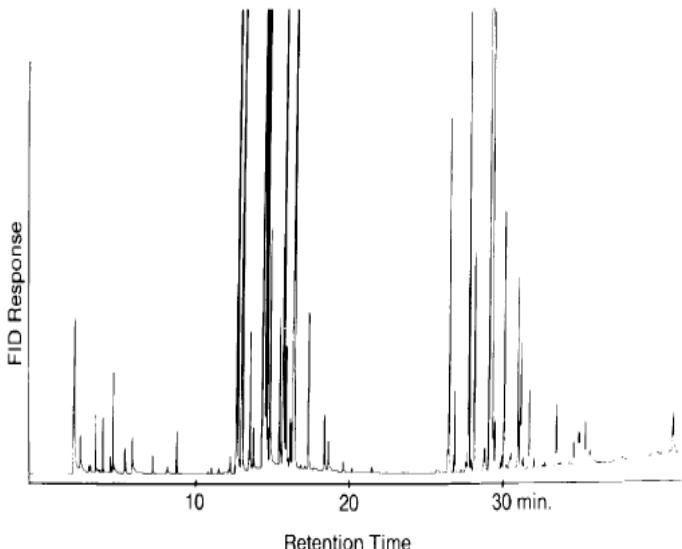


Figure 1: Dynamic Headspace of One Black Peppercorn, 100°C for 10 Minutes Direct Capillary GC with Cryofocuser

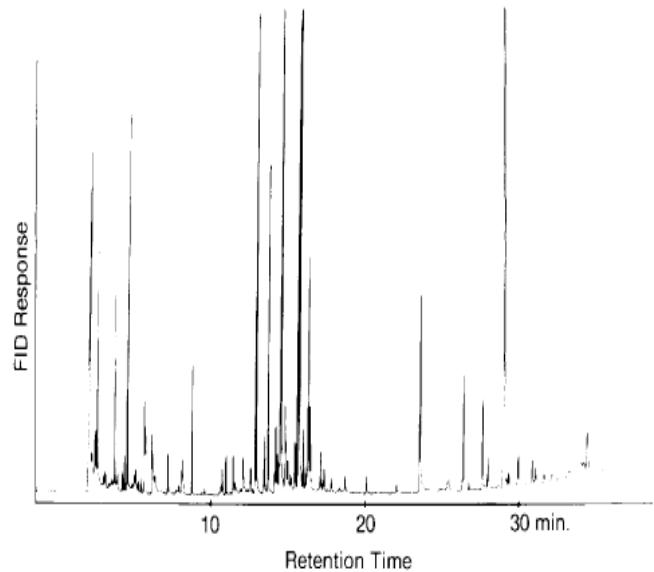


Figure 2. Dynamic Headspace of One Caraway Seed 100°C for 10 Minutes