Dynamic Headspace- Glass vs Metal

Application Note

The advent of adsorptive sample concentrating techniques has greatly simplified many chromatographic analyses. Originally applied to the analysis of volatile pollutants in drinking water, the concept has been extended to a variety of fields including soil and sludge analysis, determination of residual solvents in pharmaceuticals, volatiles analysis of polymers, and the assay of aroma compounds in the food industry.

Early instruments used metal tubing for the flow path from the sample chamber to the trap, and from the trap to the gas chromatograph, which was fine for those applications in which the analytes were stable, non-polar molecules. For many analyses, however, the presence of hot metal surfaces must be avoided, especially for polar and heat labile compounds which may be adsorbed or destroyed by these components.

Our instruments use glass lined stainless steel, quartz, or fused silica tubing throughout the sample path. Volatiles may be purged from liquids or desorbed from solids, collected either cryogenically or onto an ambient packed trap, then backflushed to the gas chromatograph where they are cryogenically refocused onto the capillary column for analysis. Trace amounts in the parts per trillion level may be assayed, since there is no split, and all of the collected organ ics are transferred to the GC.

Figure 1 shows a comparison of polar components in a test solution processed through glass and metal systems. The recovery of most of the com pounds is greatly enhanced, especially for the butane dials and dimethyl aniline, which were not detected on the metal system. An application of this technique may be seen in Figure 2. Here a 2mg piece of raw garlic was warmed to 70° C and purged with helium for 10 minutes. The evolved organics were collected onto a Tenax trap, which was dried of water vapor before backflushing to the gas chromatograph. The analytes were refocused cryogenically onto the capillary column at -100° C for 10 minutes, then revaporized when the GC program was begun.

1. 1,3 Butane Diol
2. Methyl Hexanoate
3. 1,4 Butane Diol
4. Decane
5. Octanol
6. 2,6 Dimethyl Phenol
7. 2,4 Dimethyl Aniline
8 2 Decanone
9. Hexadecane

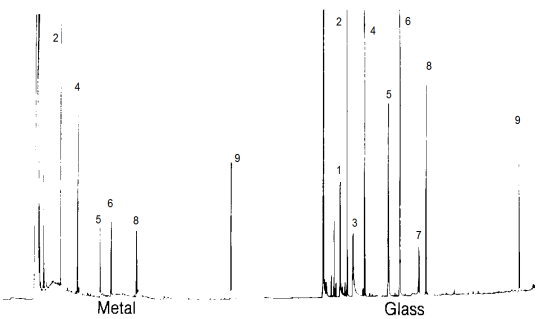

Figure 1. Polar Test Solution on Glass and Metal Systems

Sample Concentrator

Valve Oven: 250°C
Transfer Line: 250°C
Vessel Heat: 700°C 10 minutes
Sample Flow: 30ml/min
Trap Heat: 250°C 10 minutes
Cryofocus: -100°C 10 minutes

GC-FID
Column: 0.53mm x 20m SE- 54
Carrier: He at 2psi
Program: 50° C for 2 minutes,7°C/min to 250°C

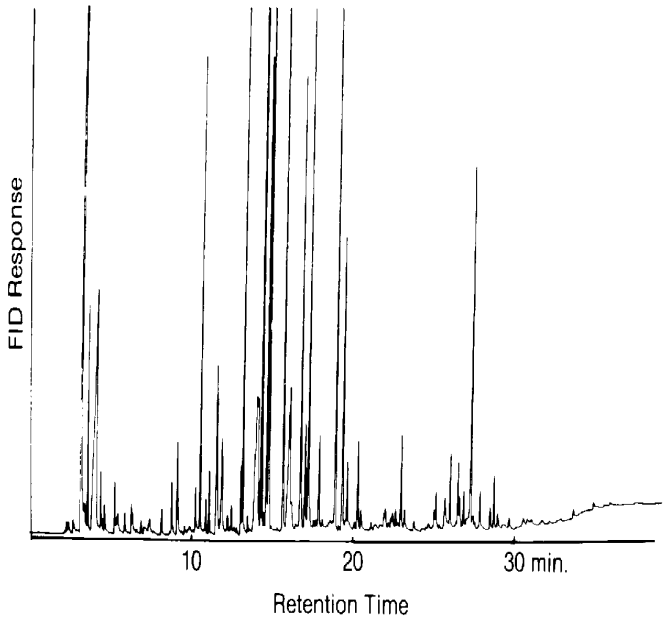

Figure 2. Dynamic Headspace of Garlic, Glass Lined System

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.

Jacobsson, S., “Analysis of Volatile Organic Compounds in Polymers by Dynamic Headspace and Gas Chromatography/Mass Spec trometry,” HRC & CC7,
185-190(1984).