CDSolutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Dynamic Headspace Analysis of Pharmaceutical Samples

Headspace sampling is generally divided into two categories—static and dynamic (Figure 1). In static headspace sampling, the test material is placed in a sealed vessel and warmed, permitting volatile compounds to enter the atmosphere around the sample. After an equilibrium period, a portion of the headspace is removed with a syringe and injected into the gas chromatograph.

Dynamic headspace sampling concentrates the evolved compounds from the sample by removing them from the heated chamber with a stream of carrier gas and collecting them onto a suitable sorbent or cryogenic trap. This trap is subsequently backflushed with GC carrier gas and pulse heated to desorb the collected organics, transferring them to the GC for analysis.

Volatile organic compounds may be purged from pharmaceutical materials for analysis of residual solvents, contaminants, flavor and aroma constituents, or to evaluate product quality and stability. Figure 2 compares 2 samples of aspirin tablets heated to 60°C and purged with helium. Both acetic acid from product degradation and salicylic acid may be determined by this analysis. Packaging materials may be evaluated for residual monomer, solvents and contaminants using the same techniques. An example is the residual styrene mono-

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Figure 1

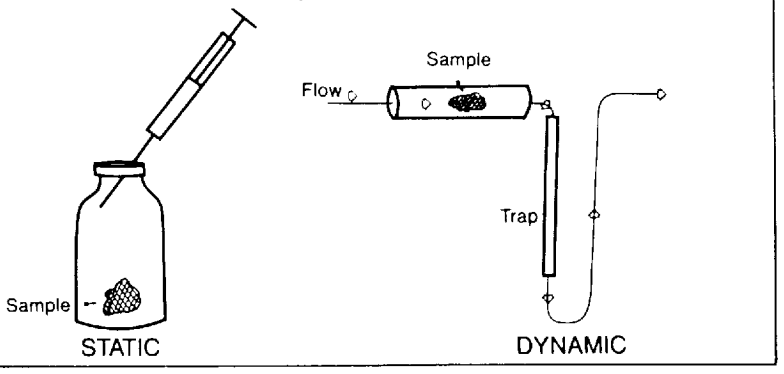


Figure 2

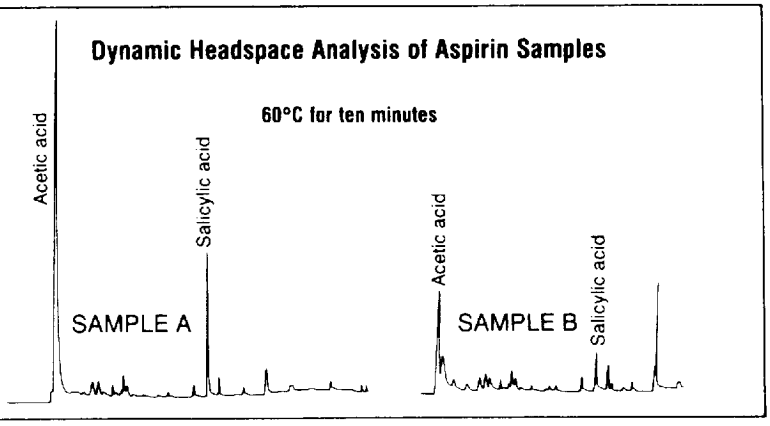
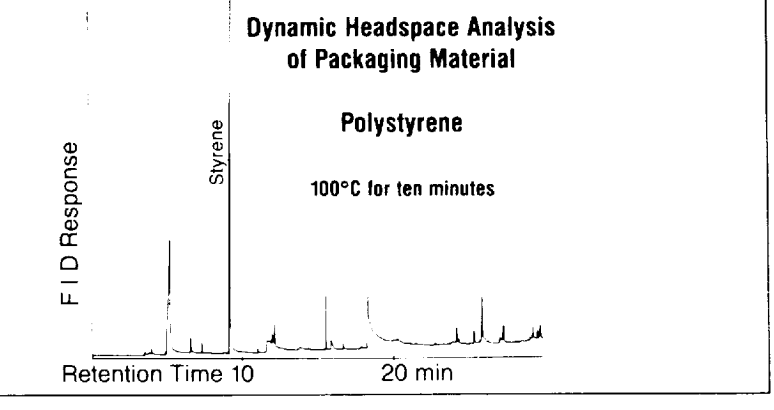


Figure 3



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mer in a polystyrene tablet bottle. A 100mg sample was heated to 100°C for 10 minutes and purged with helium which carried the volatiles to a Tenax trap. The trap was then backflushed to the GC for analysis, producing the chromatogram shown in Figure 3.

EQUIPMENT

CDS Model 330 Sample Concentrator equipped with a Tenax TA trap
Carrier gas: Helium
Purge time: 10 minutes
Trap desorption: 250°C

GAS CHROMATOGRAPHY
Varian 3700 equipped with an FID
Column: 50m x 0.25mm SE-54 Fused silica capillary
Carrier: Helium
Program: 50°C for 2 minutes, then 10°C/min to 200°C

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

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

Wampler, T., Bowe, W., Higgins, J., and Levy, E., "Systems Approach to Automatic Cryofocusing in Purge and Trap, Headspace and Pyrolytic Analyses," *American Lab,* 17, 8 (1985) 82-87

Wampler, T., "Automatic Analysis of Volatiles in Pharmaceutical Environments and Products," *Pharm. Manufact.,* 1, 5, (1984) 20-25

Additional literature may be obtained from CDS by calling 1-800-541-6593 or in Pennsylvania 215-932-3636.

ABOUT CDS

CDS Analytical, Inc. is a leader in the design and manufacture of laboratory instruments for sample preparation and analysis. With 20 years experience in the field, CDS is dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of analytical pyrolysis, CDS manufactures the Pyroprobe 1000 and 2000 for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of purge and trap instruments for the analysis of volatile organic compounds in the environmental, food and pharmaceutical areas, as well as custom systems for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet their needs, we offer a wide range of analytical information and the services of our applications laboratory. If you would like additional information, please contact us at the address below, or call us at 1 800 541 6593.

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