CDS olutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

On-Column Cryofocusing for Improved Resolution in Pyrograms

Pyrolysis has been used for years as a means to permit the analysis of natural and synthetic polymers by gas chromatography. Inherent in the system is a chamber in the GC carrier gas stream where the pyrolysis is to take place. Unfortunately, this adds dead volume to the GC system upstream from the column, and may produce broad or poorly resolved peaks in the pyrogram. One solution to this problem is the use of a split capillary injection system with a high enough split ratio to permit rapid sweeping of the pyrolysis chamber. This solution, however, necessitates the use of larger samples, since most of the pyrolysates are consequently swept out the splitter vent, and large samples are not generally the best for reproducible pyrolysis results.

Another solution is to refocus the pyrolysates directly onto the capillary column using a cryogenic trap. The example shown here used the Chemical Data Systems Model 335 Cryogenic Focuser, which mounts onto the gas chromatograph at the injection port, and uses liquid nitrogen to focus the organic volatiles onto the GC column.

For the pyrolysis of polypropylene shown in Figure 1, the fused silica capillary column was brought up through the injection port of the GC and passed through the cryofocuser.

Figure 1

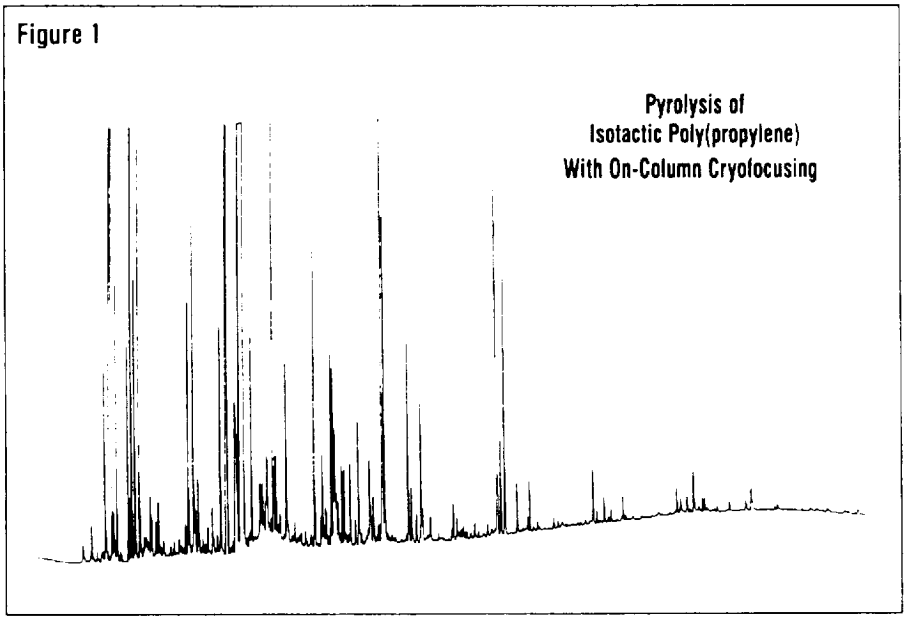
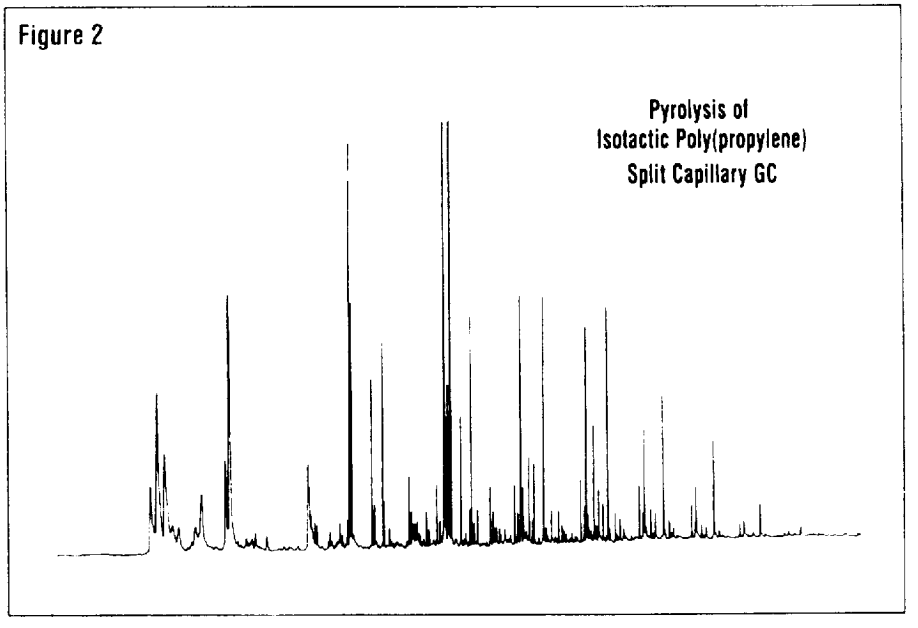


Figure 2



(cont'd on back)

(cont'd from front page)

It was then inserted into the interface of the Pyroprobe and sealed with a graphite ferrule. Now all of the flow through the interface went directly into the column, with no splitting. Prior to pyrolysis, the cryofocuser was cooled to -100°C, so that the pyrolysates would be collected as they left the interface. Once the pyrolysates were collected, the cryofocuser was heated to 280°C and the GC program was started.

For comparison, Figure 2 shows a pyrogram of polypropylene using a split capillary system, with a 60:1 split ratio. Not only could a much smaller sample be used with the cryofocusing system, but a comparison of the first 10 minutes of the two chromatograms shows how much more information is revealed by the improved resolution gained with the cryofocuser. Now many well resolved peaks may be seen which were formerly hidden in the broad, unresolved peaks seen in the split capillary chromatogram.

Equipment

PYROLYSIS

CDS Model 120 Pyroprobe, coil probe with quartz tube

Temperature: 750°C for 10 seconds

Interface temperature: 280°C

CRYOFOCUSER

CDS Model 335 Free-standing Cryogenic Focuser

Collection Temperature: -100°C for 10 minutes

Desorption temperature: 280°C

GAS CHROMATOGRAPHY

Varian 3700 equipped with a flame ionization detector

Column: 20m x 0.53mm SE-54 Capillary

Initial temperature: 50°C for 2 minutes

Program: 7°C/min. to 290°C

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

E. Levy, and T. Wampler, "Identification and Differentiation of Synthetic Polymers by Pyrolysis Gas Chromatography," J. Chem. Ed., 63, (1986), 64-68.

T. Wampler and E. Levy, "Cryogenic Focusing of Pyrolysis Products for Direct (Splitless) Capillary Gas Chromatography," J.A.A.P., 8, (1985), 65-72.

T. Wampler, W. Bowe, J. Higgins, and E. Levy, "Systems Approach to Automatic Cryofocusing in Purge and Trap, Headspace, and Pyrolytic Analysis," American Lab., 1986, (August).

Additional literature may be obtained by contacting your Chemical Data Systems representative, or by writing to the CDS Applications Lab.

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.

CDS Analytical, Inc. 7000 Limestone Rd., P.O Box 277, Oxford, PA USA 19363-0277 Ph: 215 932 3636