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APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

# Analysis of Polyolefins by Pyrolysis GC

Pyrolysis GC is a quick and straightforward way of analyzing solid materials by gas chromatography. Since most solids do not have an appreciable volatility at GC temperatures, they must be treated in some way for this technique to be of use. Pyrolysis fragments large molecules, even very large ones such as natural and synthetic polymers, and produces smaller, volatile compounds which may be analyzed by gas chromatography. This fragmentation is reproducible, and it produces a chromatogram that is characteristic for the macromolecule which was pyrolyzed.

The polyolefins are essentially hydrocarbons of very high molecular weight. When pyrolyzed, they produce smaller hydrocarbons which retain the inherent differences among the polymers. Polyethylene, for example, produces mainly straight chain alkanes and alkenes, while in polypropylene the polymer chain is highly substituted with methyl groups, which causes the production of more branched fragments, and a more complex chromatogram. In polyisobutylene, alternate carbons in the polymer chain are disubstituted with methyl groups, generating a different distribution of peaks in the pyrogram.

This can readily be appreciated by comparing the accompanying chromatograms. Figure 1 shows the fragments produced when

Figure 1

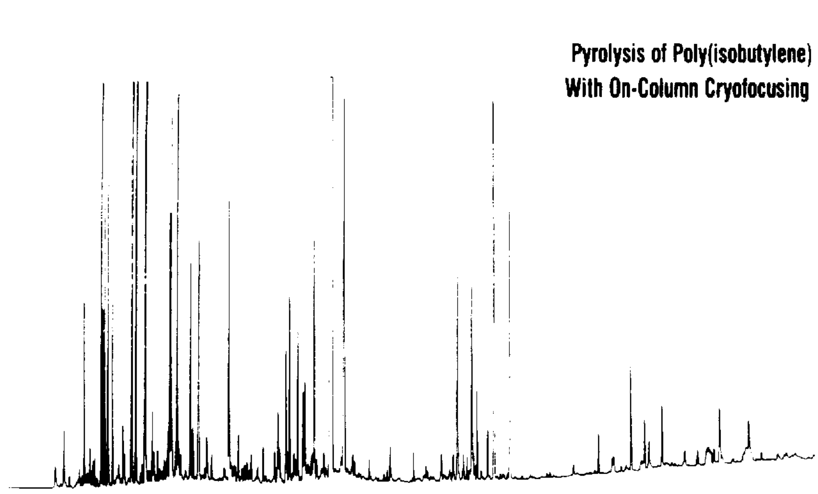
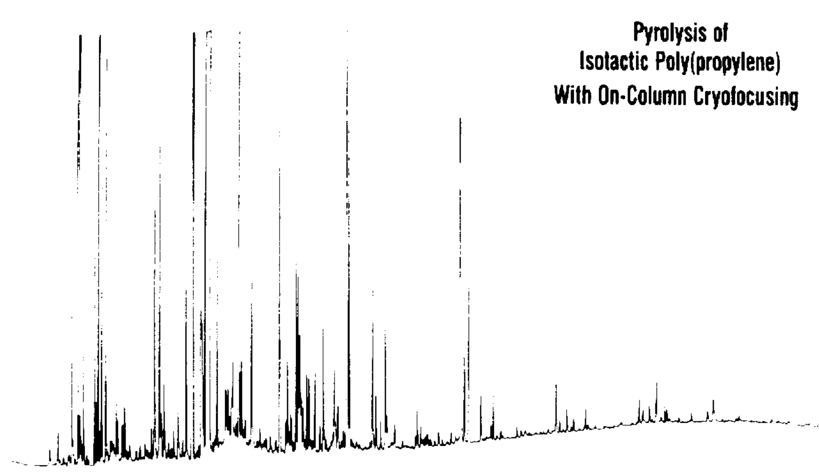



Figure 2

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polyisobutylene is pyrolyzed at 700°C. Although the high degree of methyl substitution produces a fairly complex chromatogram, it may be easily distinguished from the pyrogram of polypropylene shown in Figure 2. Collection of a library of pyrograms makes it easy to distinguish one from the other, and permits the more detailed study of microstructural defects, degradation mechanisms and related topics. In these examples, the resolution has been enhanced by the use of on-column cryogenic focusing, which permits the splitless capillary analysis of very small samples by freezing the analytes onto the head of the GC column prior to analysis.

## Equipment

PYROLYSIS

Pyroprobe 124, equipped with automatic cryogenic focuser

Pyrolysis temperature: 700°C for 10 seconds

Interface temperature: 275°C

Cryogenic collection: -100°C for 10 minutes

Revaporization: 275°C for 10 minutes

GAS CHROMATOGRAPHY

Varian 3700 equipped with flame ionization detector

Column: 50m x 0.25mm SE-54 Capillary

Initial temperature: 50°C for 2 minutes

Program rate: 7°C/minute to 290°C

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

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, and E. Levy, "Effects of Slow Heating Rates on Products of Polyethylene Pyrolysis," Analyst, 111, (1986), 1065-1067.

Y. Sugimura, T. Nagaya, S. Tsuge, and T. Murata, "Microstructural Characterization of Polypropylenes by High-Resolution Pyrolysis-Hydrogenation Glass Capillary Chromatography," Macromol., 1980, 13, 928.

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



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