CDS solutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Quantitative Determinations of Poly(propylene/1-butene)

Quantitative pyrolysis/GC is relatively simple when the polymer degrades by a depolymerization mechanism. For instance, poly(methyl methacrylate/ethyl acrylate) degrades to give only two major peaks. From the area ratios, it's easy to obtain quantitative information about the polymer. When a polymer system is more complex, quantitative studies are more difficult.

Poly(propylene/1-butene) is a copolymer system which yields a very complex pyrogram. The degradation process is primarily governed by random scission. Figure 1 shows the pyrogram of a poly(propylene/1-butene sample at 750°C. Several copolymers were analyzed, with 1-butene concentrations ranging from 11.5% to 46.5%. Quantitative data can be obtained by identifying pyrolysis fragments which are indicative of the respective monomers. In this particular example, we chose a theoretical model of the polymer. It was proposed that at various points in the polymer chains there are segments that contain two propylene groups and segments which contain adjacent 1-butene groups. Based on bond strenghts and the mechanism of random scission, it is then possible to determine the identity of some of the major pyrolysis products. In this case these products include 2,4-di-

Figure 1

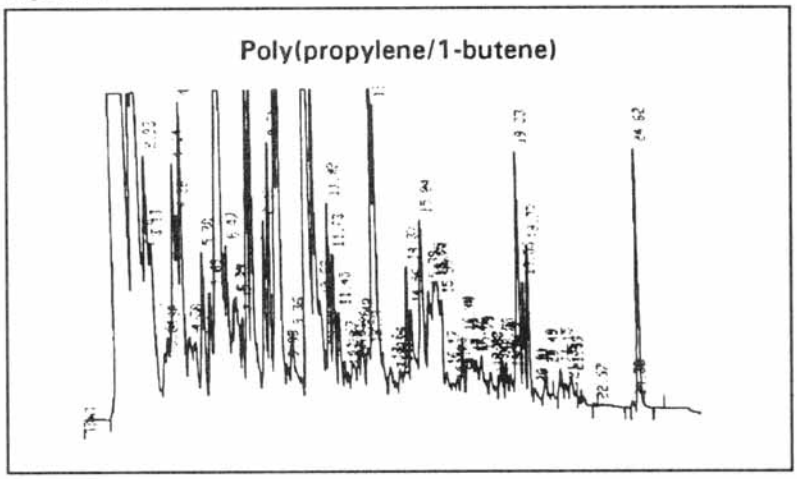
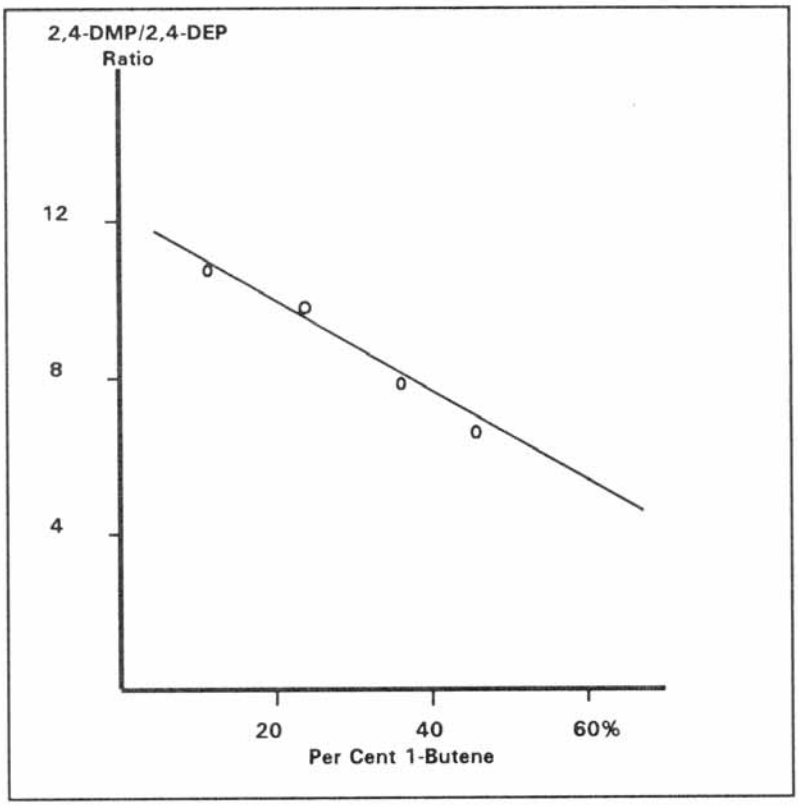


Figure 2



methyl,1,4-pentadiene (2,4-DMP), which indicated propylene monomer, and 2,4-diethyl,1,4-pentadiene (2,4-DEP), indicative of butene monomer. The ratio of the peak areas of these two pyrolysis fragments were found to be directly related to the original monomer composition.

Figure 2 shows the calibration curve obtained from pyrolyzing copolymer samples with 1-butene concentrations ranging from 11.5% to 46.5%. The area ratios of 2,4-DMP and 2,4-DEP were calculated and plotted versus 1-butene concentration. This gave a very linear calibration curve even without the presence of an internal standard.

EQUIPMENT:

PYROLYSIS

CDS Analytical model 1000 Pyroprobe

CHROMATOGRAPHY

Hewlett-Packard 5890 Gas chromatograph equipped with a flame ionization detector.
Column: 30m. X 0.53mm. SE-54 Capillary column with a 0.5um. film.

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READING:

Analytical Pyrolysis of Complex Multicomponent Samples.
J. W. Washall and T. P. Wampler, J. Chromatogr. Sci., 27, 144-148, (1989).

Reproducibility in Pyrolysis - Recent Developments
T.P. Wampler and E. J. Levy, J. Anal. Appl. Pyrol., 12, 75-82, (1987).

Additional literature on this topic may be obtained by contacting your local CDS Analytical representative, or directly from CDS at the address below.