CDS solutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Quantitative Pyrolysis/GC in the Analysis of Copolymers

Analytical pyrolysis has been used for the qualitative identification of polymers for many years. This type of analysis usually took the form of comparing pyrolysis fingerprints. It is, however, possible to attain quantitative results from this technique. One of the most simple quantitative methods involves comparing peak area ratios of monomers or primary pyrolysis products.

Figure 1

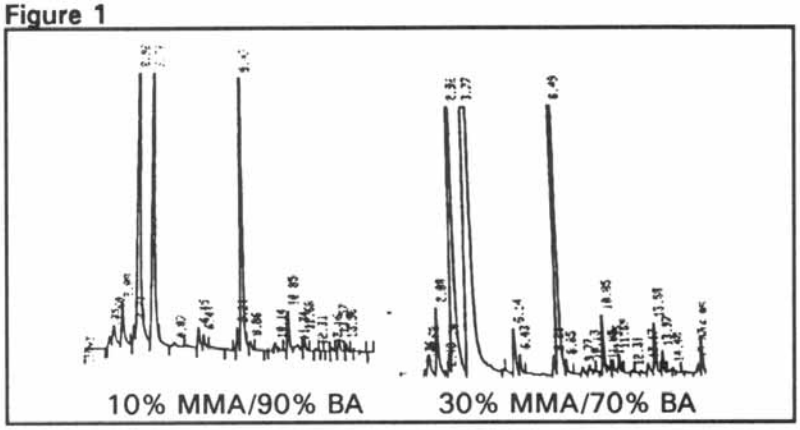
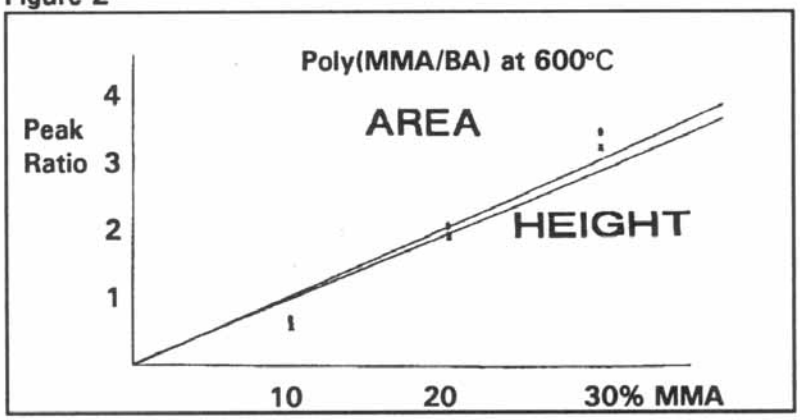


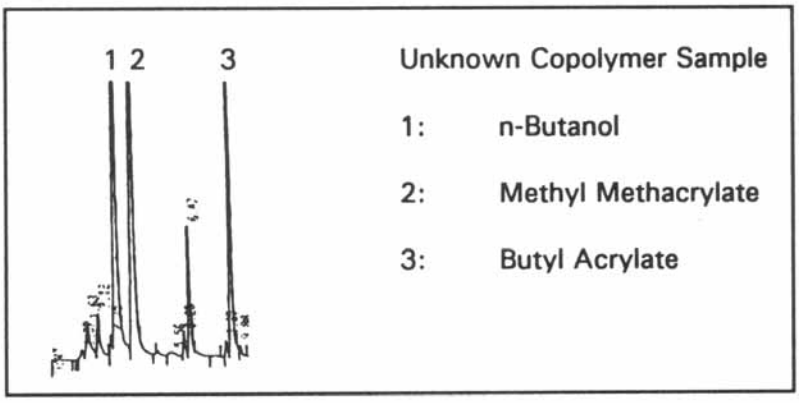
Figure 1 shows pyrograms obtained from samples of poly(methyl methacrylate/butyl acrylate) copolymer. The samples contained 10% and 30% methyl methacrylate. As you can see the chromatogram is very simple, with only three major pyrolysis products present. These products were identified as methyl methacrylate monomer, n-butanol and butyl acrylate monomer. The n-butanol was formed via a 1,5-hydrogen transfer mechanism which seemed to be preferred over production of butyl acrylate monomer. As a result of this finding, the peak area ratios of methyl methacrylate monomer and n-butanol were used for quantitative analysis.

Figure 2



Samples containing methyl methacrylate, (MMA), concentrations ranging from 10-30% MMA were all pyrolyzed at 600° C and the ratios of MMA to n-butanol determined. Figure 2

Figure 3



shows the calibration curve which was obtained from this data. The peak area ratios of MMA/n-butanol was quite linear indicating that unknown copolymers could be quantitatively analyzed.

Figure 3 shows the pyrogram of an unknown copolymer sample. By comparing the area ratios of MMA and n-butanol in relation to the calibration curve, it was determined that this sample contained 12.6% MMA and 87.4% butyl acrylate. The technique of comparing area ratios of copolymers is not restrictive to materials which degrade by simple processes; it can be used even with copolymers of poly-olefins which generally yield very complex pyrograms.

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

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

Pyrolysis Gas Chromatographic Analysis (PGC) of Methyl Methacrylate (MMA)-Ethyl Acrylate (EA) Copolymers.
S. Paul, and W. Becker, J. Coatings Technol. 52, 47-55, (1980).

EQUIPMENT

PYROLYSIS:
CDS Analytical Model 1000 Pyroprobe

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

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