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

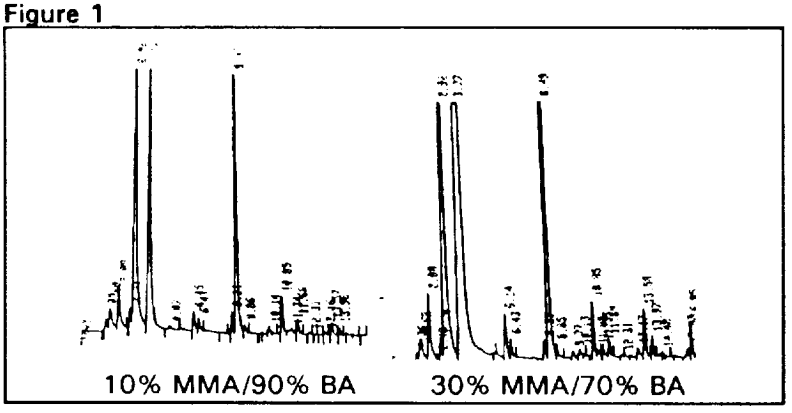
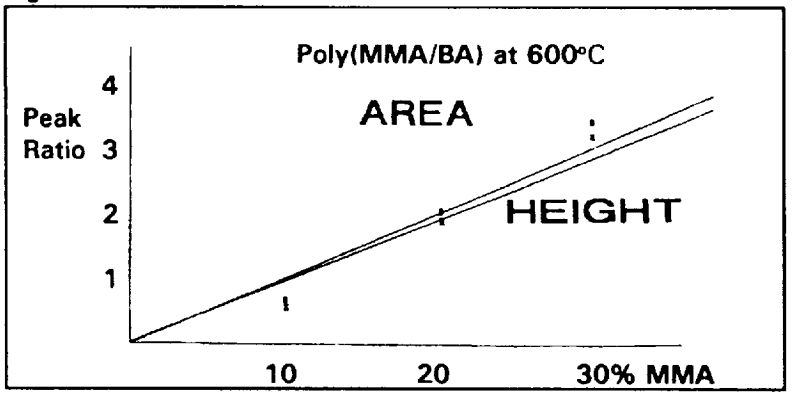


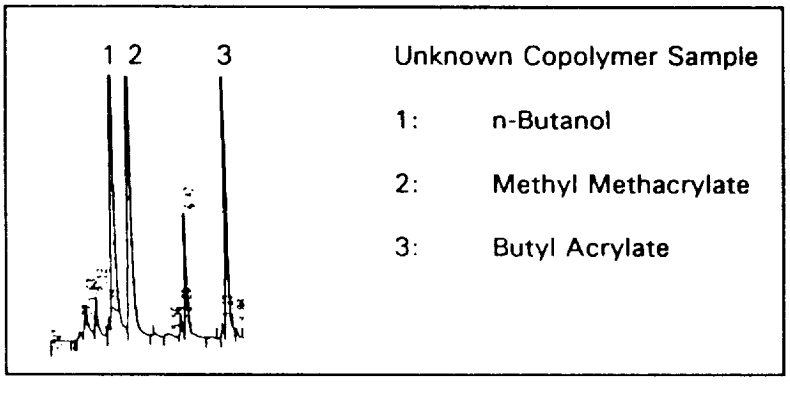
Figure 1 shows pyrograms obtained from samples of poly(methyl methacrylate/butyl acrylate) copolymer. The samples contained 10% and 30% methyl methacrylate. As you can see the chromatogram is very simple, with only three major pyrolysis products present. These products were identified as methyl methacrylate 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.

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.

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).

ABOUT CDS

CDS Analytical, Inc. is a leader in the design and manufacture of laboratory instruments for sample preparation and analysis. With 25 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 or 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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