CDS Analytical, Inc.

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Quantitation of Ethylene/Propylene Copolymers

Under pyrolysis conditions, polyolefins degrade by random scission to produce a chromatogram with a characteristic repeating pattern of oligomeric fragments. Thus polyethylene (Figure 1, bottom) shows a pattern of normal hydrocarbons, while polypropylene (Figure 1, top) generates hydrocarbons with many methyl groups along the chain, and consequently a different pattern. This makes it easy to differentiate polyethylene from polypropylene, and in fact, from the other polyolefins as well.

When a material is a block copolymer of ethylene and propylene, the pyrogram will contain oligomers from each polymer. The center pyrogram in Figure 1 is of a copolymer containing 25% ethylene and 75% propylene. As expected, the oligomers from polypropylene are easy to identify, since this is the major constituent, but the normal hydrocarbons from polyethylene may be seen as well. In the figure, several peaks have been marked with the letter "e" to show the contribution from the ethylene portion of the polymer, and it is clear that these peaks are present in the pyrogram of polyethylene and absent in the pyrogram of polypropylene.

If a series of copolymers of known monomer content is pyrolyzed, a graph may be prepared showing the relationship between peaks which come from the respective monomers and the relative concentration of those monomers. In this example, the first peak labeled "e" in Figure 1 was selected for the ethylene content. This peak is pentadecene and elutes at 20.5 minutes, a retention time when little is eluting from polypropy-

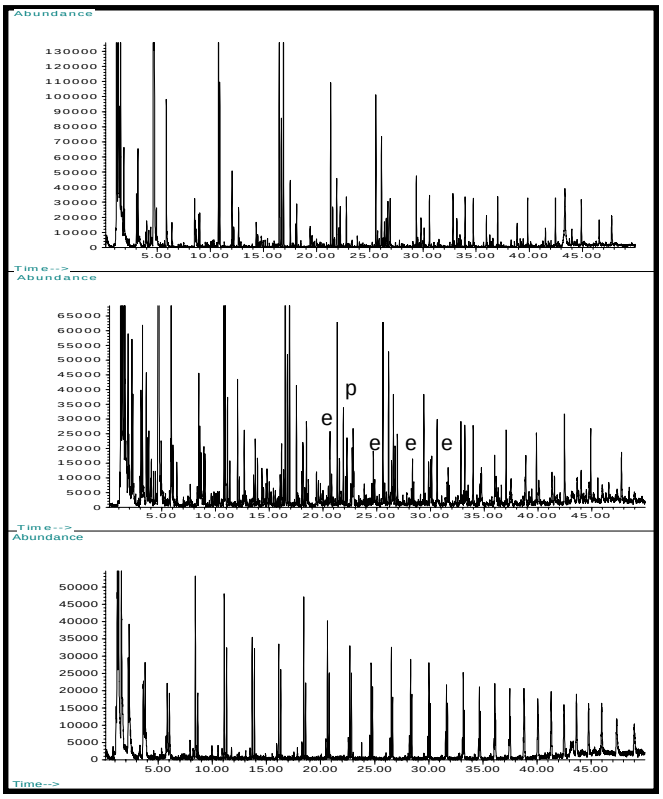

Figure 1

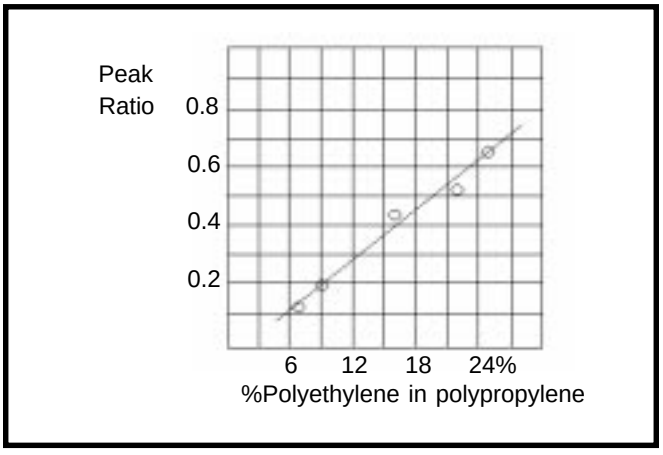

Figure 2

lene, so it is well resolved even in the presence of the other oligomers. Likewise the peak marked “p”, a hexamer of polypropylene, is well resolved, even in the presence of polyethylene oligomers. When the ratio of “e” to “p” for a series of polymers from 7 to 25% ethylene is plotted against the concentration of ethylene for these polymers, the straight line graph shown in Figure 2 results. This graph can then be used to calculate the relative amounts of the two monomers in copolymers of unknown composition.

Equipment

All samples were pyrolyzed using a CDS Model 2500 Pyrolysis Autosampler interfaced to a Hewlett-Packard 6890 gas chromatograph with a mass selective detector.

Pyrolysis

Interface oven:	300°C
Ramp:	10°C/ms
Temperature:	750°C
Time:	15 seconds
Clean:	1000°C for 10 seconds

Chromatography

Carrier:	He
Column:	HP-5
	30 m x 0.25 mm
Split:	75:1
Initial temperature:	40°C for 2 minutes
Ramp:	8°C/minute
Final temperature:	290°C for 10 minutes

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

Microstructures of Polyolefins, S. Tsuge and H. Ohtani in *Applied Pyrolysis Handbook*, T. Wampler, Editor, Marcel Dekker, New York (1995).

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

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

CDS Analytical, Inc.

CDS Analytical, Inc. has been a leader in the design and manufacture of laboratory instruments for sample preparation and analysis since 1969. We are dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of 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 dynamic headspace instruments for the analysis of volatile organic compounds in environmental, pharmaceutical and food applications, as well as custom systems for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet your 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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