CDS solutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

PYROLYSIS/GC OF CARBON-FILLED RUBBERS

Pyrolysis/GC is a well-established technique for qualitative and quantitative analysis of polymers, but has seldom been applied to carbon-filled rubbers. These samples are particularly difficult to analyze because, in addition to being insoluble and nonlabile like most polymers, the carbon black filler is difficult to remove and often interferes with analytical results. To demonstrate the feasibility of using pyrolysis/GC to analyze these samples, eight materials identified by color and texture as being carbon-filled rubbers were randomly selected and analyzed. The resulting chromatograms (pyrograms) were compared to those of standard (non-carbon-filled) polymers. Five of these selections were found to be polyisoprene, and the other three were polybutadiene, polyethylene, and polyisobutylene. The carbon black content of the samples was estimated by the amount of material left over after pyrolysis of the samples, compared to the amount left after pyrolysis of the standards. Two examples are given here.

In Figure 1, the pyrograms of polyisobutylene and a piece of a bungee cord are shown. The carbon black content of the bungee cord was 37-40%. The fingerprints of the two polymers are so similar that it is apparent that there is no interference from the carbon black in the pyrolysis products.

Figure 1

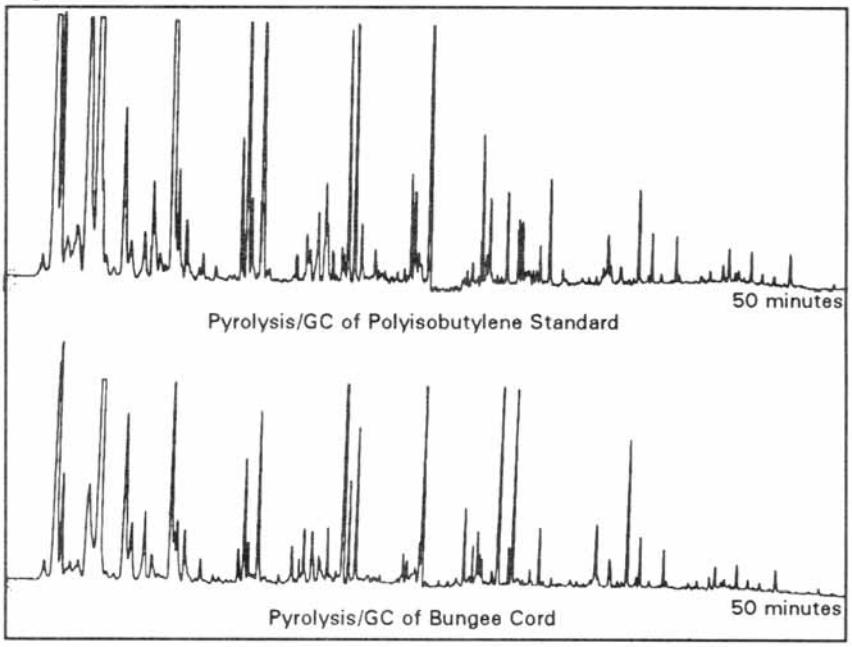
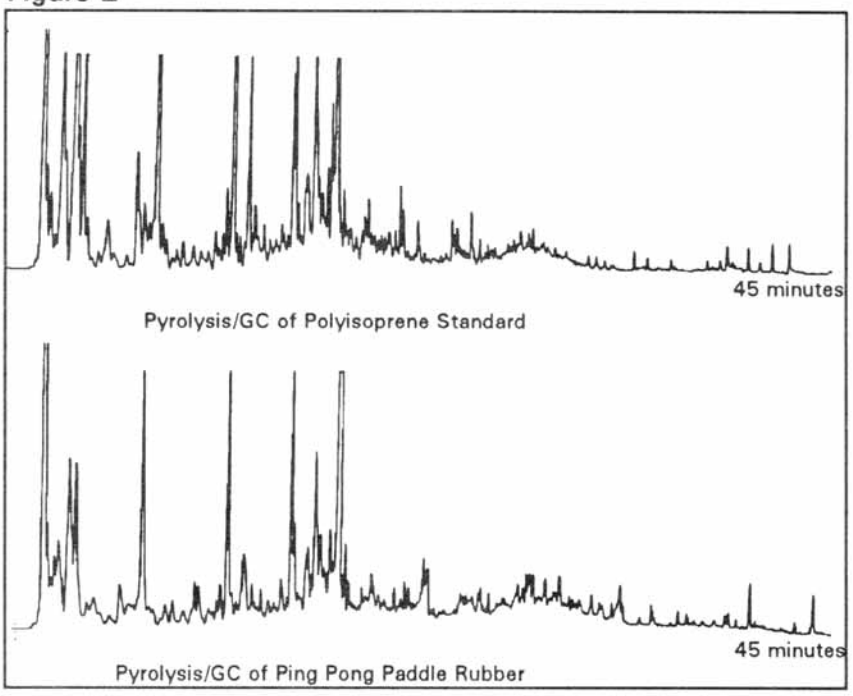


Figure 2



In Figure 2, the pyrograms of polyisoprene and a sample of rubber from a ping pong paddle are shown. The carbon black content of the ping pong paddle rubber sample was 43-53%. Again, there is no interference in the pyrogram from the presence of the large amount of carbon black.

Pyrolysis/GC can be used to analyze carbon-filled rubbers with the same degree of confidence as it is used with polymers without a carbon filling. The carbon black present in the sample, even at high concentrations, does not interfere with the pyrolysis products.

Instrumentation

For pyrolysis/GC, a CDS Analytical Model 2000 Pyroprobe with a platinum coil and a CDS Analytical PeakMaster interfaced to a Hewlett-Packard 5890 GC were used. The GC column was a 30 m by 0.53 mm SE54. Pyrolysis occurred in the thermal desorber of the PeakMaster, and the resulting gases were swept onto a Tenax trap, then desorbed onto the head of the GC column. The PeakMaster program was as follows: valve oven and transfer line, 300 C; desorber temperature, 300 C for 10 min at 40 ml/min He flow; trap A rest, 35 C; trap A desorb, 300 C for 4 min; trap A bake, 300 C for 5 min. The GC temperature program was: start 35 C, hold 6 min, then ramp at 6 C/min to 300 C.

The carbon black content of the samples was determined by weighing the quartz boats before and after pyrolyzing the samples and standards in a helium atmosphere. The difference between the amount of char formed by the standard and the sample is presumed to be primarily carbon black. The average of three samples, plus and minus the standard deviation, is given as the range.

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

L.S. Bark and Allen, N.S. *Analysis of Polymer Systems.* Applied Science Publishers LTD., Essex, England, 1982.

Tsuge, et al. *Structural Characterization of Polyolefins by Pyrolysis-Hydrogenation Glass Capillary Gas Chromatography.* J. Anal. App. Pyrolysis, 1 (1980) 221-229.

K. V. Alekseeva. *Gas Chromatographic Identification of Polymers Using Individual Pyrolysis Products.* J. Anal. App. Pyrolysis, 2 (1980) 19-34.

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

K.G. Hausler, et al. *The Effect of Carbon Black Filler on the Pyrolysis Behavior of Vulcanized cis-Polybutadiene.* J. Anal. App. Pyrolysis, 13 (1988) 287-304.

Available from CDS Analytical:

T.P. Wampler, J.W. Washall, and M.J. Matheson. *Analytical Pyrolysis in the Analysis of Carbon-Filled Rubbers.* PittCon 1993.

CDS Analytical, LLC 465 Limestone Road, P.O. Box 277, Oxford, PA USA 19363-0277 PH: 610 932 3636 FX: 610 932 4158