CDS solutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

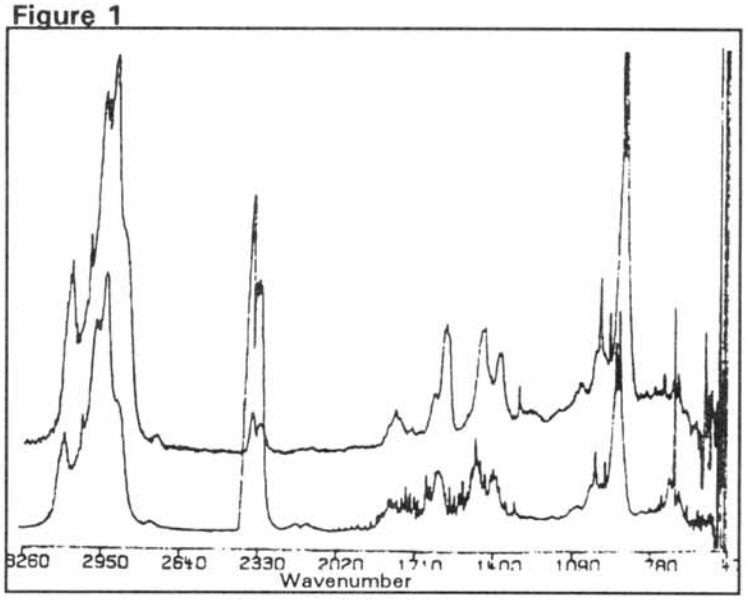
PYROLYSIS/FT-IR OF CARBON-FILLED RUBBERS

Carbon-filled rubbers are frequently difficult to analyze because they are often insoluble and are opaque. Attenuated total reflection spectroscopy (ATR) is of great value in polymer analysis, but is of limited use in the case of carbon-filled rubber analysis because of interference bands caused by the carbon black. Removing the carbon black by extraction is a cumbersome step and is not always completely successful. Direct pyrolysis/FT-IR avoids these problems because the carbon black is simply left behind as a solid when the volatiles formed by pyrolysis enter the IR light beam. In this work, the feasibility of using it to analyze carbon-filled elastomeric polymers has been demonstrated.

Eight samples identified by color and texture as being carbon-filled rubbers were randomly selected and analyzed by pyrolysis/FT-IR. The polymer compositions were determined by comparison of the FT-IR scans with 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.

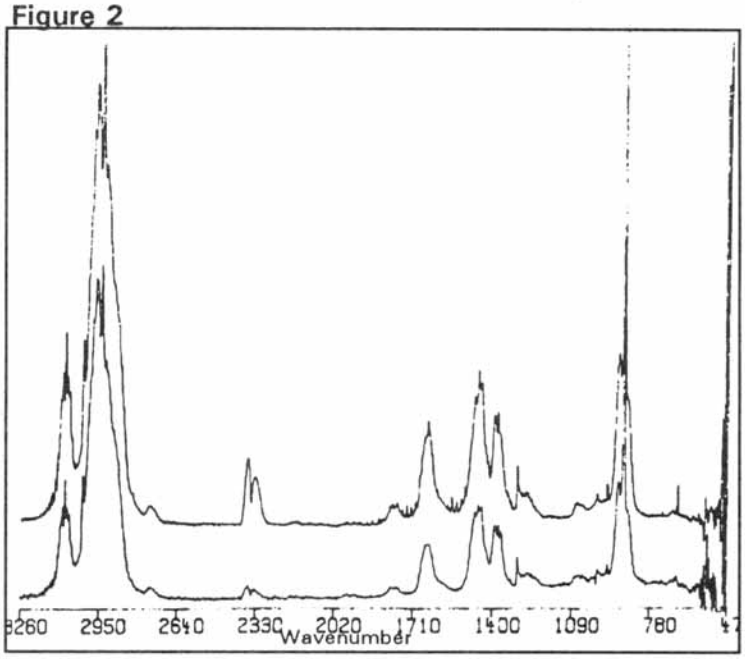
In Figure 1, the FT-IR absorbance scans of polyisoprene and a sample of rubber from a ping pong paddle are shown. The samples are almost identical except for the presence of CO_2 and water vapor, due to entrained air in the rubber. The carbon black content of this sample was estimated to be 43-53%. It is apparent from the similarities between the spectra that the presence of carbon black had little affect on the analytical results.

Figure 1



Pyrolysis/FT-IR Absorbance
Top: Polyisoprene
Bottom: Ping Pong Paddle Rubber

Figure 2



Pyrolysis/FT-IR Absorbance
Top: Polyisobutylene
Bottom: Bungee Cord

In Figure 2, a bungee cord is compared to a polyisobutylene polymer standard. Again, the only difference between the spectra is in the presence of CO_2 and water, due to entrained air in the bungee cord. The carbon black content of the bungee cord was 37-40%.

It is evident from the results of these studies that there is very little interaction between the pyrolysis products of these samples and the carbon black found in them. Direct pyrolysis/FT-IR is a rapid and effective approach to their analysis.

INSTRUMENTATION

A CDS Analytical Model 1000 Pyroprobe with a platinum coil was used for pyrolysis/FT-IR. The polymer samples were placed in a quartz boat and pyrolyzed at 850 C for 28 sec, the length of the FT-IR scan. A CDS Analytical Brill Cell, with standard 1/4" Zn/Se windows, was the interface to a Nicolet 710 FT-IR spectrometer equipped with a DTGS detector with a scan speed of 0.8 scan/sec.

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

J. W. Washall and T.P. Wampler. *Direct-Pyrolysis Fourier Transform-Infrared Spectroscopy for Polymer Analysis.* Spectroscopy 6 (4) (1989) 38-43.

P.J. Gale, et al. *Characterization of Polymers by Pyrolysis Mass Spectrometry.* RCA Rev., 47 (1986) 380-397.

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 Rubber.* PittCon 1993.

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.

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