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Pyrolysis of Rubber with Antioxidant 6-PPD

Rubber products are complex polymeric materials which generally include copolymers, fillers, antioxidants, plasticisers and other additives. Automobile tire rubbers are good examples, since they usually contain significant amounts of carbon black, hydrocarbon oil and antioxidants in a matrix that may include styrene, butadiene and isoprene. Nevertheless, a single pyrolysis run can provide significant information about such a complex material.

Figure 1 shows the pyrolysis at 700°C of a sample of tire rubber. The matrix is an isoprene/butadiene copolymer, and early in the pyrogram the monomers and dimers from the copolymer may be seen. Later in the chromatogram is a peak for the antioxidant 6-PPD [N-(1,3-dimethyl butyl)-N'-phenyl-p-phenylenediamine]. 6-PPD has a molecular weight of 268, and produces the mass spectrum seen in Figure 2.

Other antioxidants, especially hindered phenolics, are also frequently seen in the pyrolysis of rubber samples. Some of these, like BHT, are present as the parent molecule. Other antioxidant molecules are too large to permit GC analysis intact, and are seen as fragments, just as for the polymer itself.

At the end of the pyrogram is a region of unresolved peaks (marked 6 in Figure 1) which results from the oil added to the rubber in making the tire. Consequently, from just a single pyrolysis run, the monomer composition of the rubber, the antioxidant used and the oil added may be determined.

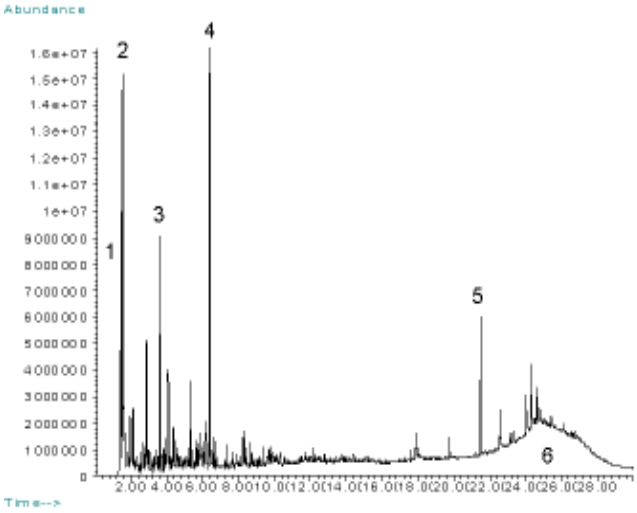

Figure 1. Pyrolysis of tire rubber at 700°C. Peak 1=butadiene, 2=isoprene, 3=butadiene dimer, 4=isoprene dimer, 5= 6-PPD, 6=Hydro-carbon oil.

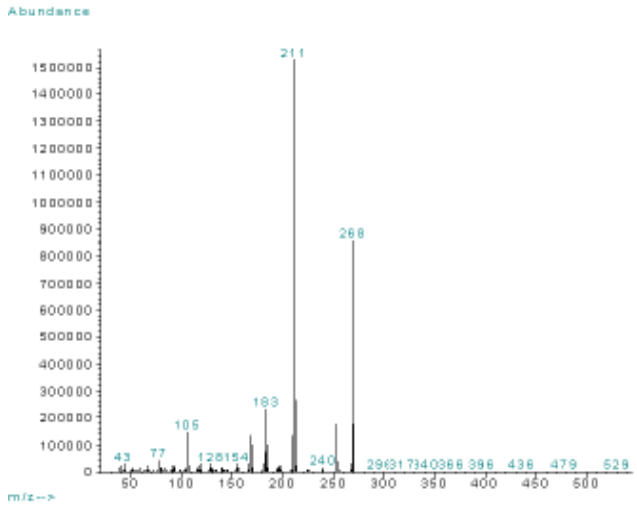

Figure 2. Mass spectrum of peak at 21.5 min.

Equipment

The rubber sample was analyzed using a CDS Pyroprobe Model 2000, interfaced to an Agilent 6890 gas chromatograph which was equipped with an Model 5973 MSD as the detector. A 100µG piece of the rubber was pleced into a qaurtz tube, which was inserted into the coil filament of the Pyroprobe.

Pyroprobe 2000 Conditions

Interface:	Model 1500 valved
Interface temp:	300°C isothermal 700°
Temperature:	C for 10 seconds 10°
Rate:	C/ms
Probe:	Platinum coil

GC Conditions

Carrier:	Helium
Split:	50:1
Column:	HP-5 (30m X 0.25)
Detector:	MSD
GC Program:	
Initial:	40°C for 2 minutes
Ramp:	10°C/min.
Final:	300°C

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

Temperature as a Sample Preparation Tool in the Analysis of Materials by GC-MS, T. P. Wampler, LC.GC, 17 (1999) S14.

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