Multi-Step Analysis of Automobile Tire Rubber

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Application Note

Automobile

Automobile tires are complex products which may include natural and synthetic rubbers, carbon black, hydrocarbon oil, and a variety of additives to promote vulcanization and to protect the final product from oxidation. Pyrolysis-GC has long been used to analyze for the polymers used in manufacturing the tire rubber. But with recent advancements in pyrolyzer capabilities, a multi-stepped temperature program approach can be used. This advanced method can help facilitate the analysis of the total organic composition by permitting selective vaporization of various volatiles & semi-volatiles, as well as pyrolysis of the polymer for identification of the monomers.

A first run at a relatively low temperature, as in Figure 1, can eliminate volatile contaminants from “real world” samples, so that these peaks do not show up in analytical runs.

Heating the sample again to 300°C (Figure 2) reveals several additives intrinsic to the rubber formulation. There is a broad peak from about 20 to 30 minutes that is due to the hydrocarbon oil that is added to tire rubber, usually in the low percent range. At about 27 minutes there is a peak for the antioxidant 6-PPD.

The pyrolysis run at 700° shown in Figure 3 is now relatively simple, since the additives and contaminants have been removed. The pyrogram shows that the tire sample is a butadiene-isoprene rubber clearly displaying the polymer monomers and dimers formed from pyrolysis.

CDS Pyrolyzer 6200 Conditions:

Pyroprobe	
Initial:	150°C for 20 sec., 300°C for 15 sec., and 700°C for 10 sec
Interface	
Rest:	300°C
Initial:	300°C for 2 minutes
Iso Zones	
Valve oven:	300°C
Transfer line:	315°C

GC Conditions:

Column:	30 m x 0.25 mm 5% phenyl MS
Carrier:	Helium
Split:	100:1

Oven Program	
Initial Temp:	40°C
Initial Hold:	2 minutes
Ramp:	10°C/minute to 325°C

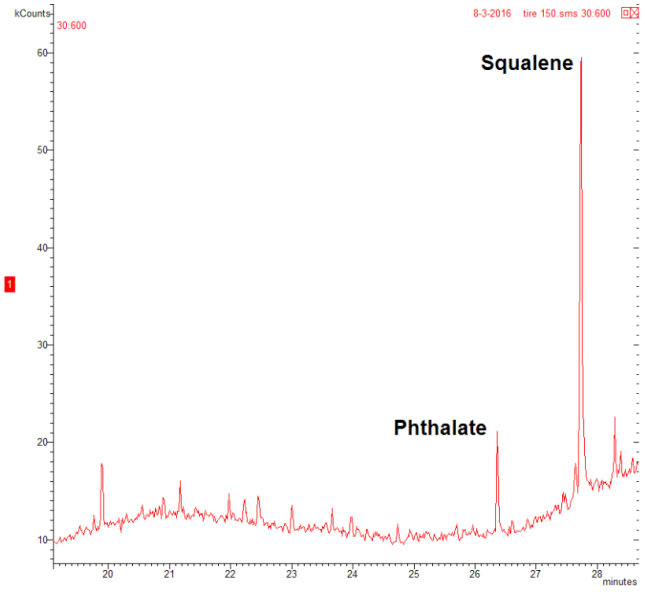

Figure 1. Automobile tire sample heated to 150°C

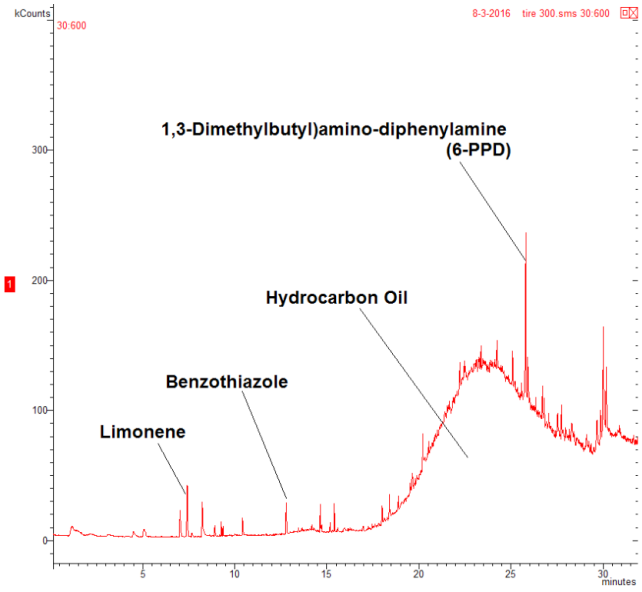

Figure 2. Rubber heated to 300°C after the run at 150°C

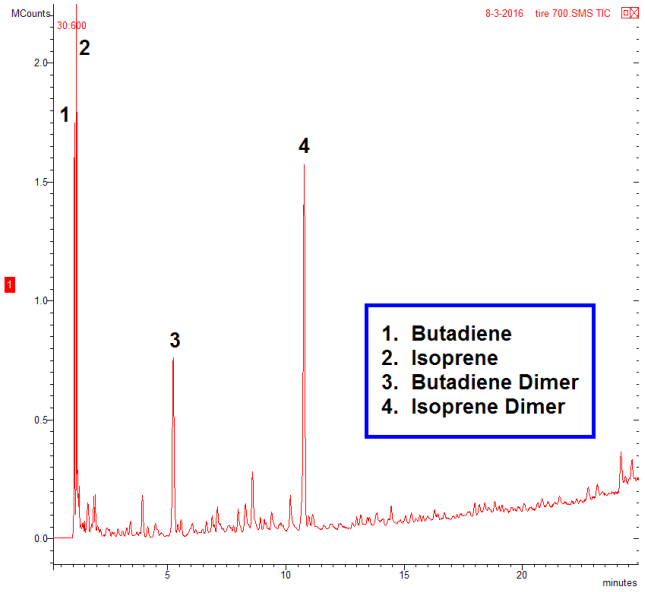

Figure 3. Pyrolysis of the rubber at 700°C