

The Analysis of Vulcanized Rubber by Pyrolysis/GC/MS

Vulcanized rubbers are particularly difficult materials to analyze because of the opacity and insolubility of the rubber. Pyrolysis combined with gas chromatography and mass spectroscopic detection (pyrolysis/GC/MS) is an ideal approach to the study of these materials. Pyrolysis volatilizes the rubber components, which are then separated by GC. The use of MS enables the pyrolysis products to be positively identified.

In this application, a sample of rubber of unknown composition was pyrolyzed us-

ing a CDS Analytical Pyroprobe 1000 interfaced to a Varian GC with a Finnegan MAT Ion Trap Detector. The resulting total ion chromatogram (Figure 1) demonstrated the rubber to be a styrene-butadiene copolymer, about 5% styrene. Identification of the individual peaks confirmed the major components (Table 1). Because of the sensitivity and specificity of MS, the presence of trace components can be positively confirmed, which is useful in quality control, competitive analysis, and similar applications.

Figure 1 Total Ion Chromatogram

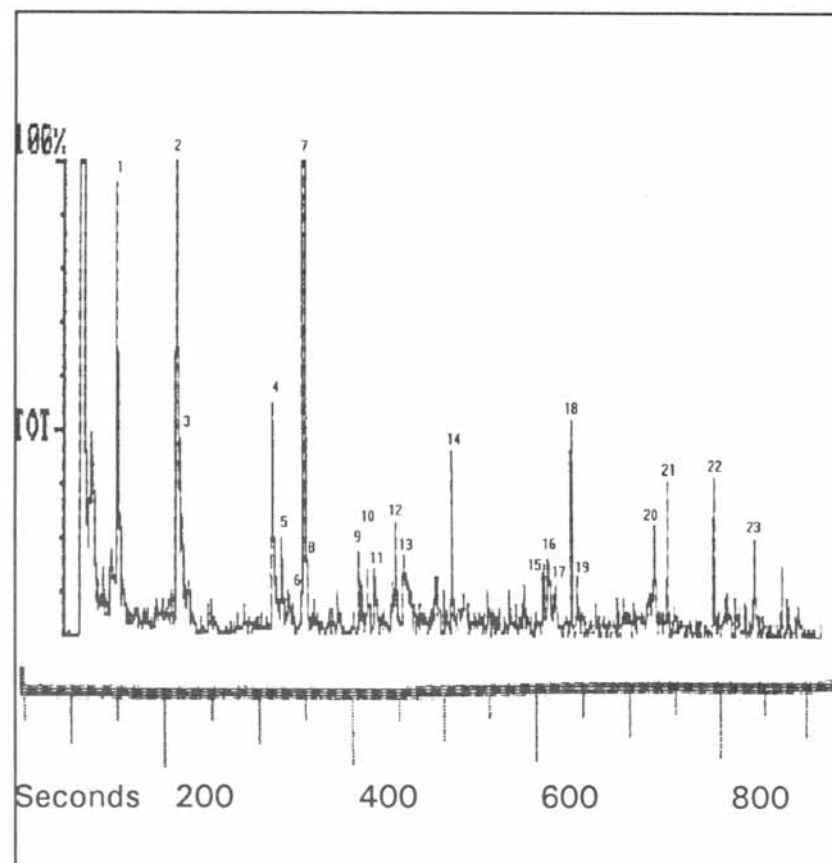


Table 1 Peak Identification

Peak	Compound
1	benzene
2	toluene
3	3-methylthiophene
4	ethylbenzene
5	2,5-dimethylthiophene
6	thiophenol or 1-ethylthiole
7	benzocyclobutane
8	m-xylene
9	2,3-diH-Indene
10	propylbenzene
11	2-(isopropyl)thiophene
12	1-ethenyl-2-methylbenzene
13	4-[methylthio]phenol
14	1-ethynyl-3-methylbenzene or 1,2-propadienylbenzene
15	1-methyl-1-H-Indene
16	[1-methylene-2-propenyl]benzene
17	1-cyclobutenylbenzene
18	azulene or naphthalene
19	benzo[b]thiophene
20	1,4-diH-1,4-methanaphthalene
21	1-ethenylindene or 1-methylnaphthalene
22	biphenyl
23	2,4-dimethylquinoline

Experimental Conditions

Pyrolysis

Instrument: CDS Analytical Model 1000 Pyroprobe

Temperature: 850 C

Ramp: 15 C/Msec

Time: 10 sec

Interface Temperature: 250 C

Gas Chromatography

Instrument: Varian Model 3400 GC

Column: 30 m DB5, 0.25 mm i.d.

Oven Temperature Program: start 40 C, hold 3 min,
then 8 C/min to 280

On-Column split Injection

Mass Spectroscopy

Instrument: Finnegan MAT Ion Trap Detector

Mass Range: 0-500 amu

Transfer Line: 325 C

Data System: Finnigan Ion Trap Data System 3.15

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

Gale, et al. Characterization of polymers by pyrolysis mass spectrometry. RCA Rev., 47, 380-397 (1986).

McGuire, J.M. and C.C. Bryden. Direct pyrolysis mass spectrometry of chlorine-containing polymers using capillary GC/MS. J. Appl. Polym. Sci., 35, 537-548 (1988).

Barkowski, et al. Comparison and identification of adhesives used in improved explosive devices by pyrolysis-capillary column gas chromatography-mass spectrometry. J. Anal. Appl. Pyrolysis, 8, 483-492 (1985).

Wampler, T.P. A selected bibliography of analytical pyrolysis applications 1980-1989. J. Anal. Appl. Pyrolysis, 16, 291-322 (1989).

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

CDS Analytical, LLC 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® 5000, 5150, 5200 and 5250 autosampler 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, including the model 8400 four-position autosampler. CDS also manufactures the Dynatherm line of thermal desorption instruments including the 9000 series for air monitoring and the 9300 TDA. 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, call us at 1 800 541 6593, or log onto www.cdsanalytical.com.