

Multi-Step Pyrolysis-GC/MS Analysis of PVC Copolymer

Pyrolysis-gas chromatography/mass spectrometry is a convenient and sensitive way to analyze complex polymeric materials. Frequently, the pertinent information may be obtained from a single run, for example, at 700°C. Other times, a thermal separation may be desired, by analyzing for volatiles at a low temperature and then pyrolyzing the remaining polymer.

For a better understanding of the behavior of a specific material, however, it may be advantageous to analyze the sample using a series of increasing temperatures. The Model 2500+ Autosampler, used in these analyses, is capable of performing an unlimited number of runs on each sample, complete with automatic start of the GC-MS for each run.

The plastic material shown here was a copolymer of poly vinylchloride and poly methylmethacrylate. When heated, PVC releases HCl, then produces aromatics, including benzene and toluene. Figure 1 shows the benzene peak for a series of runs on the same sample, heating sequentially to 200°, 300°, 400°, 500°, 600°, 700° and then 1000°C. Although the greatest production of benzene is seen at 700°, some is still released when the sample is heated to 1000°C.

Figure 2 compares the production of benzene, from the PVC to the formation of methyl methacrylate from the PMMA, at 600° and 1000°C. Although the PVC continues to generate benzene at higher temperatures, the PMMA is essentially unzipped well before that temperature.

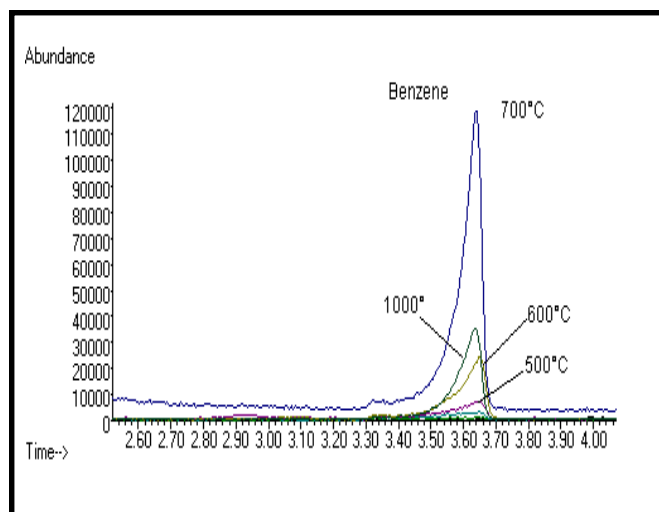


Figure 1.

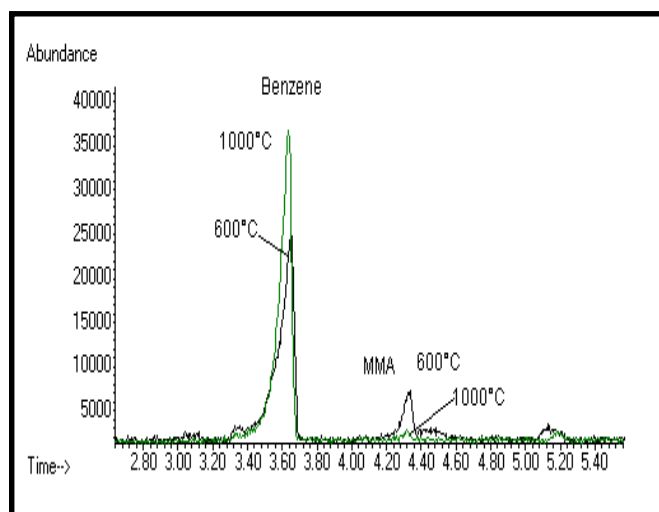


Figure 2.

Equipment

These samples were analyzed using a CDS Model 2500+ Pyrolysis Autosampler, interfaced to an HP6890 gas chromatograph which was equipped with an HP5973 MSD as the detector. The 2000+ Software of the Pyroprobe permitted multiple runs per sample tube, automatically loading the pyrolysis parameters and starting the gas chromatograph.

Model 2500+ Conditions

Valve Oven: 300°C
Pyrolysis Setpoint: 200° - 1000°C
Time: 15 seconds

GC Conditions

Carrier: Helium
Split: 50:1
Column: HP-5 (30 m X 0.25 mm)
Detector: MSD

GC Program:

Initial: 40°C for 2 minutes
Ramp: 8°C/min.
Final: 300°C

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

S. A. Liebman, et al., *Thermal degradation Studies of PVC with Time-Resolved Pyrolysis GC and Derivative TGA*, J. Polymer Sci., 16, (1978) 3139.

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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