



Application Note 

Analysis of Thermoset Resin Using Pyrolysis-GC/MS

Introduction

Thermoset polymers or resins are materials that strengthen when heated. A Thermoset cannot be remolded or heated after initial forming. Manufacturers in various industries utilize thermosets due to their resistance against high temperature. Thermosets are hard, rigid, and have high mechanical properties as they cannot be remolded or reshaped after initial shaping.

The chemical composition analysis of thermosets is critical in the supply chain. In this technical note, a thermoset resin's chemical composition is analyzed using Pyrolysis-GC/MS. Using this technique, the thermoset sample can be analyzed in its solid form. There is no solvent or sample preparation needed; the Pyrolyzer expands the capability of the GCMS system by broadening the range of organic analysis, including the insoluble solids.

Experimental

First, Evolved Gas Analysis (EGA) was performed from 100 to 600 °C (20°C/min). EGA is one of the modes of operation using the multi-functional micro-furnace pyrolyzer. In this mode, no column is used; a short, small diameter (2.5m, 0.15 mm id.) deactivated tube connects the GC injection port to the detector. The sample is dropped into the furnace, which is at relatively low temperature (in this example 100 °C). The furnace is then programmed to a much higher temperature (in this example 600 °C). Compounds “evolve” continuously from the sample as the temperature increases. A plot of detector response versus furnace temperature is obtained, which provides a clear picture of the thermal profile of the sample.

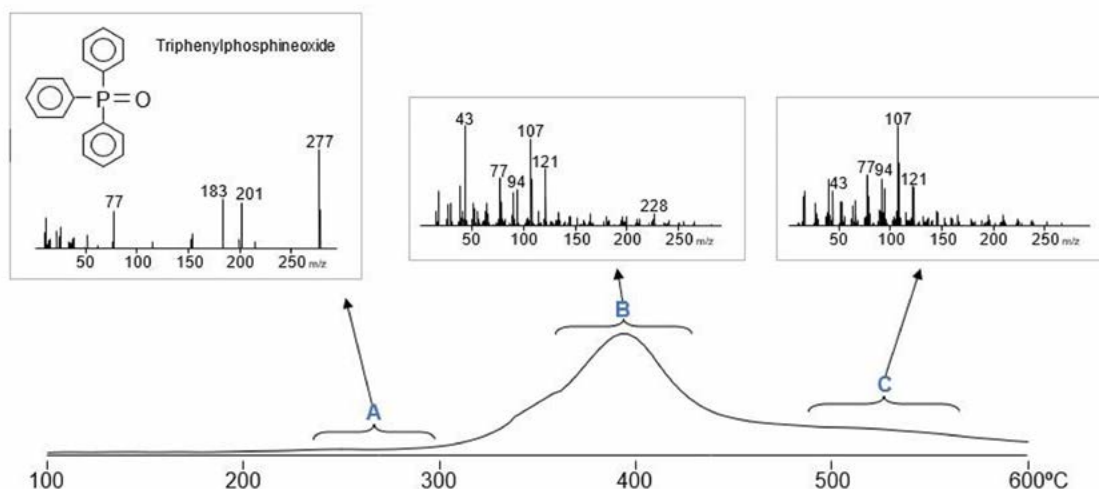


FIGURE 1. EGA CURVE OF A FLAME-RETARDANT RESIN (FRONTIER LABORATORIES LTD.)

Results

Figure 1 shows the EGA of the thermoset resin and averaged spectra obtained from regions A, B, and C. Cresol and phenol resins were found in region B, a cresol resin was detected in region C, and Triphenylphosphineoxide, a reaction catalyst, was found in region A.

To further analyze the sample, the Heart-Cutting mode of operation of the Pyrolyzer was used. The Heart-cutting is sequential thermal slicing of the obtained EGA thermogram for chemical composition analysis of each region, A, B, and C. Figure 2

shows the chromatograms of three temperature regions, A, B, and C observed in the EGA curve of a thermoset resin.

Triphenylphosphine oxide, a reaction catalyst, was found in region A, while various phenols, thermal decomposition products of phenol resin, and styrene monomer, thermal decomposition product from polystyrene, were found in regions B and C.

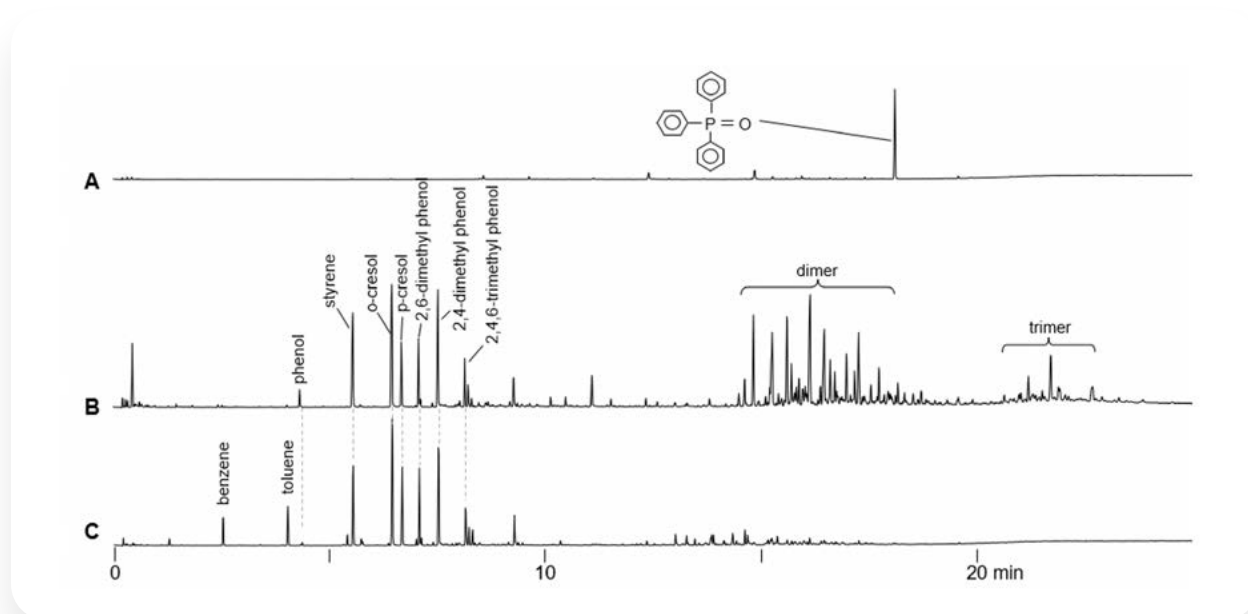


FIGURE 2. CHROMATOGRAM OF EACH TEMPERATURE REGION OF EGA CURVE

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Summary

The Frontier Multi-Functional Micro-Furnace Pyrolyzer (EGA/PY-3030D) allows multiple analyses on a sample while performing a full range analysis; volatiles, additives, polar, heavy, and polymeric identification. The pyrolyzer directly interfaces with the GC injection port. There is no transfer line, no focus trap, and no switching valves. As a result, there is no degradation before the analysis, no cross-contamination, no active sites, and all the pyrolyzates directly and continuously transfer on-column.

Reference

1. This technical note was developed by Frontier Laboratories Ltd. 4-16-20 Saikon, Koriyama, Fukushima, 963-8862 JAPAN



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