

Pyrolysis-MSD for Rapid Polymer Analysis

Application Note

Plastics

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Although most analytical pyrolysis performed on polymers involves gas chromatography, a quick analysis may be achieved by connecting the Pyroprobe directly to the MSD using a short length of fused silica. The injection port is operated in the split mode to limit the amount of material entering the mass spec, just as in chromatography. The fused silica is housed in the GC oven, acting as a transfer line, and the oven must be kept hot to facilitate transfer of the pyrolysis products to the mass spec. Since most contemporary mass spectrometers are designed to be used only as GC detectors, this is a simple way to simulate analyses performed using the direct insertion probes which were common on earlier mass spectrometers.

The pyrolysate enters the mass spectrometer almost immediately, where spectra may be taken in the same way they would if a column were present. For polymers which unzip, the resulting peak would consist mostly of monomer, so a general library could identify it. For most polymers, however, this composite peak is a combination of multiple compounds.

Figure 1 shows the total pyrolysate peak for a piece of Nylon. Since the pyrolysate contains many compounds, including cyclopentanone from adipic acid, nitriles, etc., the spectra do not represent pure compounds, but the whole mixture. In this case, a library of averaged spectra is used to identify the polymer. Figure 2 shows the best match for the pyrolysate peak from the sample compared to the pyrolysis library, which correctly identifies the sample as Nylon 6/6.

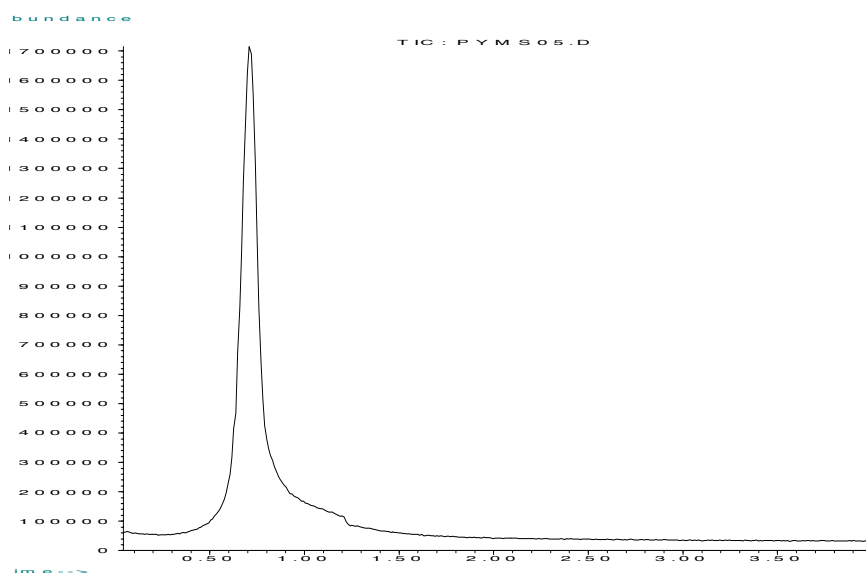


Figure 1. Pyrolysis-MS composite peak.

Instrument Conditions

Pyroprobe

Valve Oven: 300°C
Temperature: 750°
Time: 20 seconds
Rate: 10°C/second

GC/MS

Column: 1 m x 0.1 mm, uncoated
Carrier: Helium, 75:1 split
Injector: 300°C
Oven: 250°C isothermal

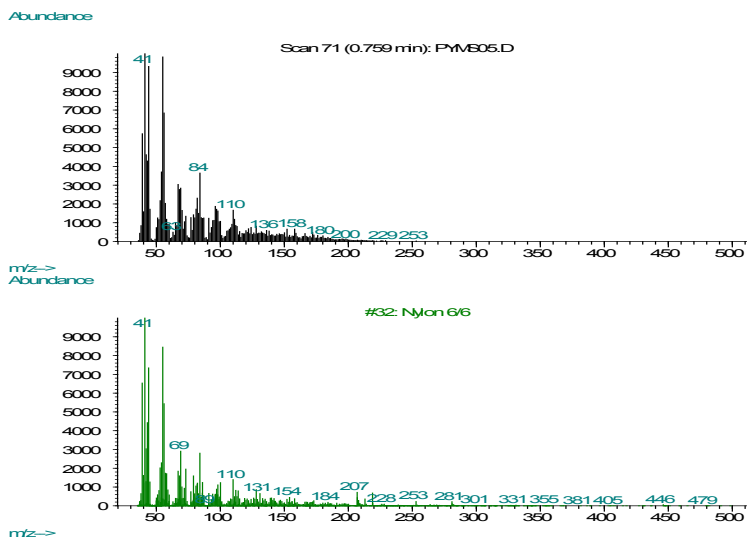


Figure 2. Comparison to Nylon 6/6 in Pyrolysis Library.

The screenshot shows a window titled 'Pyrolysis Library Search Results: C:\DATA\MASG\Tpyro1'. It contains a table with the following data:

Name	Ref. No.	MW	Qual
1. Nylon 6/6	002	9999	64
2. Polyurethane	005	9999	25
3. Yellow azo dye artist color	002	9999	25
4. Blue (Hid, solid)	0113	9999	25

Below the table are buttons for 'Difference', 'Statistics', 'Test', 'Print', 'Done', and 'Help'.

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

K. Qian, et al., Rapid Polymer Identification by In-Source Direct Pyrolysis Mass Spectrometry and Library Searching Techniques,
Anal. Chem., 68 (1996) 1019.