

Pyrolysis-MSD for Rapid Polymer Analysis

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.

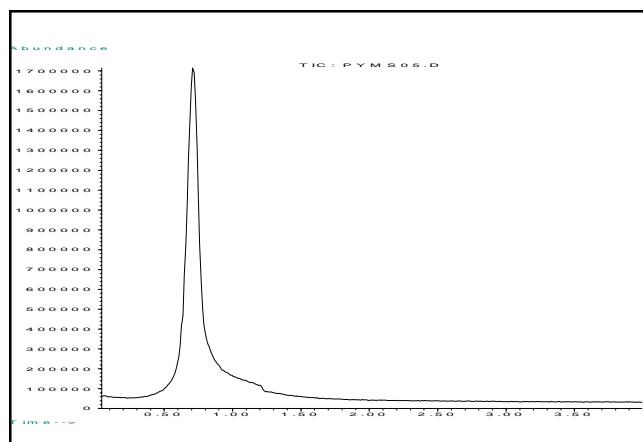


Figure 1. Pyrolysis-MS composite peak.

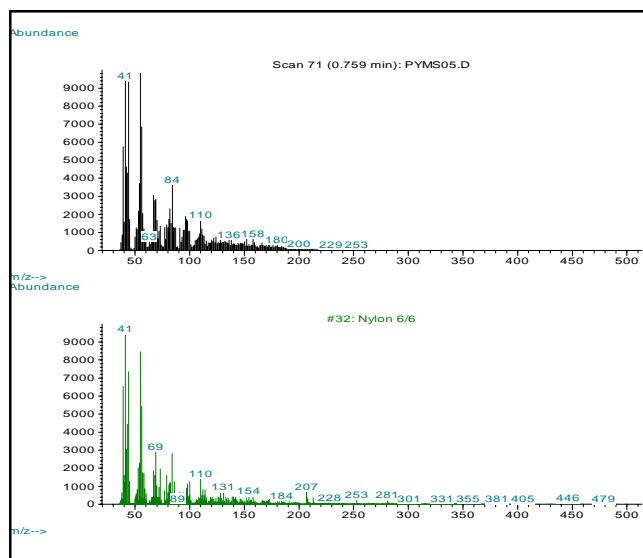


Figure 2. Comparison to Nylon 6/6.

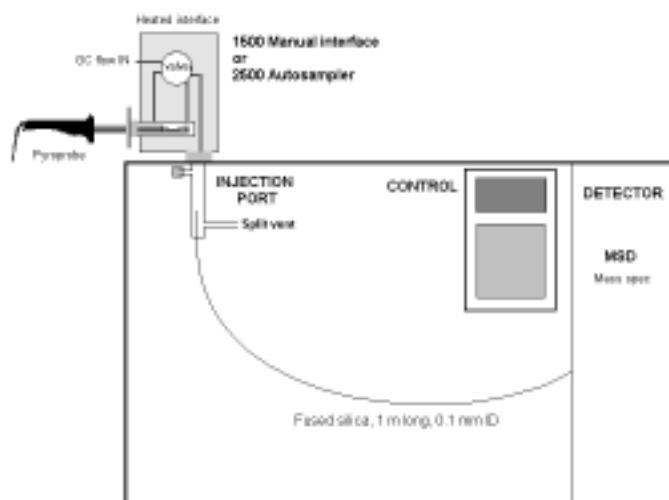
Pyro Search Results: C:\DATABASE\pyro1

Name	Ref No.	MW	Qual
1. Nylon 6/6	822	999	64
2. Polycarbonate	885	999	25
3. Yellow acrylic artist color	852	999	25
4. Glass (fibre, solid)	8112	999	25

Buttons: Difference, Statistics, Test, Print, Done, Help

Equipment

The Pyroprobe was interfaced to an Agilent 6890 GC with a 5973 MSD. The column was removed and replaced with a 1 m x 0.1 mm piece of fused silica directly from the injection port to the mass spec inlet.



Pyrolysis Conditions

Valve Oven: 30°C
Temperature: 750°
Time: 20 seconds
Rate: 10°C/second

GC Conditions

Carrier: Helium
Split: 75:1
Column: 1 m x 0.1 mm, uncoated
Detector: 5973 MSD
Injector: 300°C
Oven: 250°C isothermal

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.

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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