

# CDSolutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

## 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.

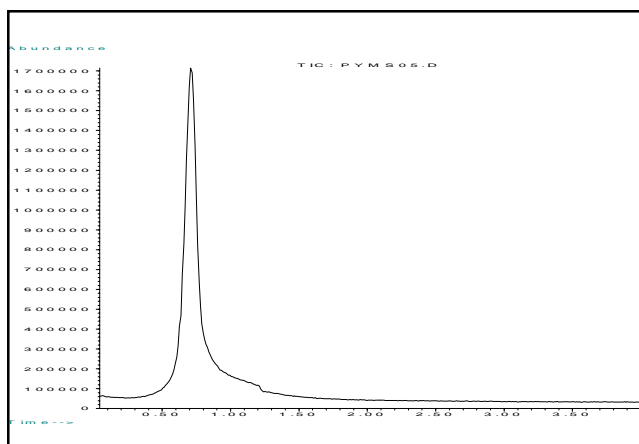


Figure 1. Pyrolysis-MS composite peak.

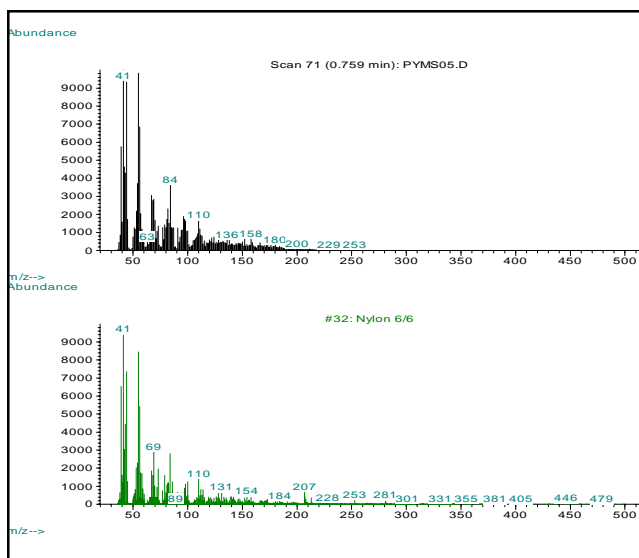


Figure 2. Comparison to Nylon 6/6.

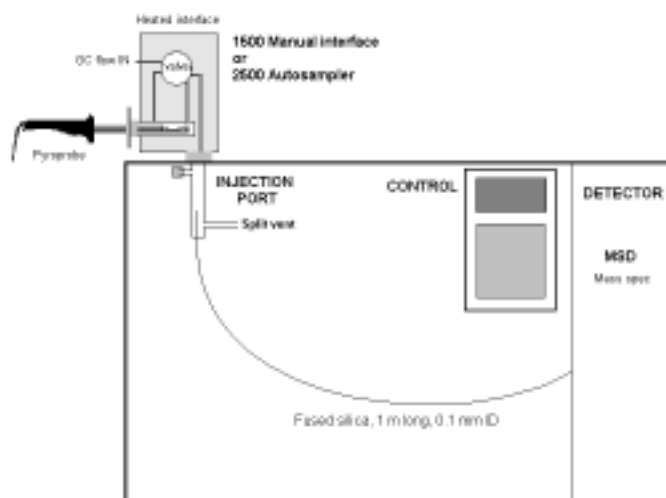
PM Search Results: C:\DATABASE\ppol1

Name	Ref No.	MW	Qual
1. Nylon 6/6	820	9999	64
2. Polyurethane	885	9999	25
3. Yellow acrylic artist color	852	9999	25
4. Glas (Hidc, solid)	8113	9999	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.



CDS Analytical, Inc. 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® 1000, 2000 and 2500 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 6500 16 position autosampler for complex, multicomponent materials investigation. 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](http://www.cdsanalytical.com).