CDS solutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Detecting Differences in PEEK Preparation

Pyrolysis, a good technique for competitive analysis, quickly yields information on subtle differences between polymers. In this application note, we spot small differences in two poly ether ether ketone (PEEK) samples.

PEEK is high-strength, radiation-resistant plastic. Thermally stable and highly resistant to chemicals, it is used in machine parts, nuclear power-plant equipment, automobile parts, aerospace components, cable insulation, and medical implants.

Figures 1 and 2 are pyrograms of 2 different PEEK samples. Although largely similar, (they both contain typical pyrolysis products of PEEK such as benzene, phenol, biphenyl, fluorene, and diphenyl methanone), we can see tiny differences between the them. The first PEEK sample has siloxanes (peak #s 2 and 4).

The second sample instead has diflurobenzophenone, dichlorobenzophenone, and diphenyl sulfone (DPS). These products could be residual starting components or solvent. One way of making PEEK involves reacting benzenediol's di-potassium salt with difluorobenzophenone using DPS as a solvent.

Using pyrolysis can make work in the lab easier by providing a quick, simple way for competitive analysis. With almost no sample preparation, you get a complete profile of the unknown polymer, and clues as to how it was made.

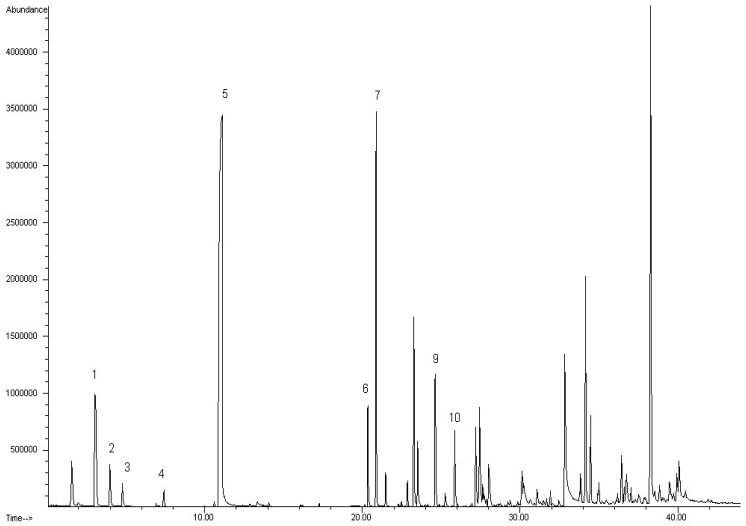

Figure 1

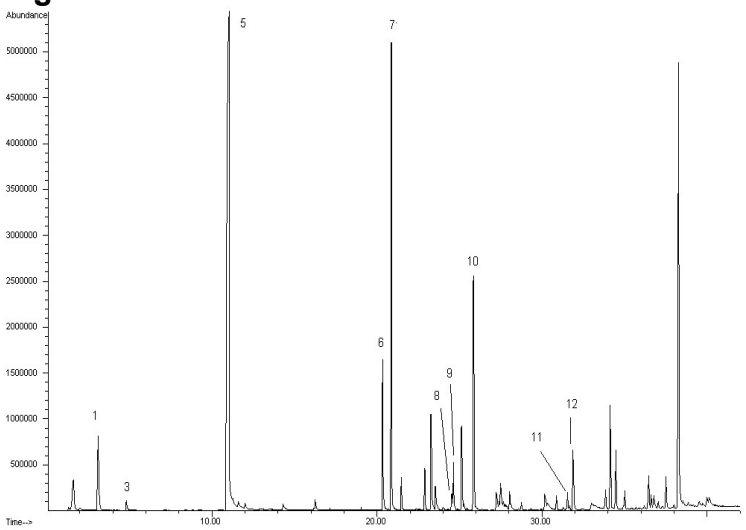

Figure 2

Peak Identification

1. Benzene
2. Hexamethyl Cyclotriloxane
3. Toluene
4. Octamethyl Cyclotetrasiloxane
5. Phenol
6. 1,1'-Biphenyl
7. Diphenyl ether
8. Difluorobenzophenone
9. Fluorene
10. Diphenyl Methanone
11. Dichlorobenzophenone
12. Diphenyl Sulfone

Both polymers were pyrolyzed at 750°C for 10 seconds using the CDS Model 5200 interfaced via a transfer line to an Agilent 6890/5973 GC/MS.

Model 5200 Conditions

Pyrolysis to GC mode (no trapping)
Interface zone: 300°C
Transfer Line: 290°C
Pyrolysis probe: Coil
Pyrolysis time: 10 seconds
Valve oven: 250°C

Gas Chromatograph Conditions

Carrier: Helium
Column: 35% Phenyl, methyl silicone
Detector: MSD

GC Program:
Initial: 40°C for 2 minutes
Ramp: 7°C/min.
Final: 300°C for 10 minutes

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.

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CDS Analytical, Inc. 465 Limestone Road, P.O. Box 277, Oxford, PA USA 19363-0277 PH: 610 932 3636 FX: 610 932 4158