

Polymer Characterization by 94-0485 Fourier Transform Mass Spectrometry

❏ Laser desorption Fourier Transform Mass Spectrometry (FT/MS®)
for molecular weight and structural polymer information in a single shot

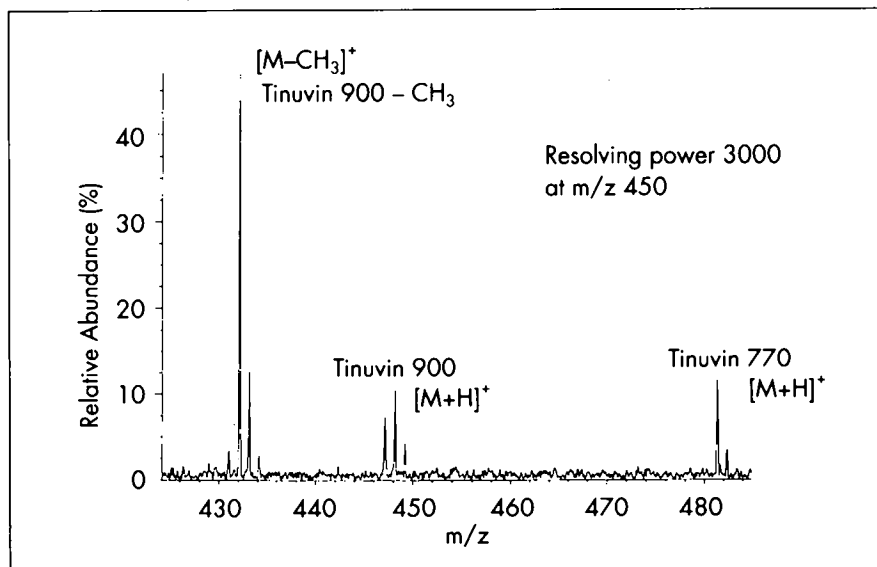
Millipore together with Extrel FTMS is setting a course into the future of polymer research by applying laser technology and mass spectrometry to polymer analysis. The Extrel Laser Probe FT/MS instrument can easily and quickly analyze any kind of sample, providing weight average (M_w) and number average molecular weight (M_n) for low molecular weight polymers ($M_w < 10,000$) as well as information about the structure, degree of branching, and chemical composition of a variety of polymers and polymer additives. High-resolution, accurate mass spectra can be obtained from materials, surfaces, and micro samples that cannot be analyzed by other techniques, even conventional mass

spectrometry. The following examples illustrate the use of the Extrel Laser Probe FT/MS instrument in polymer characterization.

Identification of additives in polymer based paints

Additives play an important role in improving the properties of polymeric materials. Here, the Extrel Laser Probe FT/MS quickly identified two photostabilizers in a cross-linked polymeric paint by yielding a mass spectrum that reveals the unique molecular weight of each additive. A paint chip was inserted directly into the instrument, and the additives were identified in seconds.

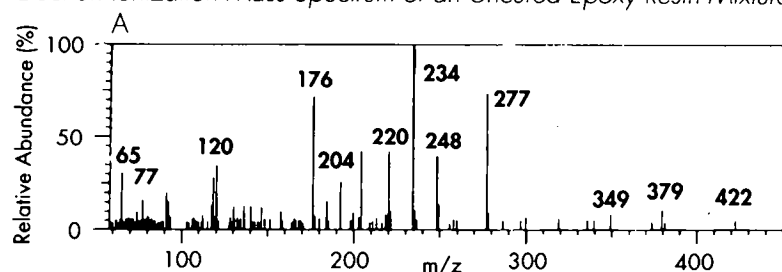
CO₂ laser desorption/electron ionization of a polymer-based paint.



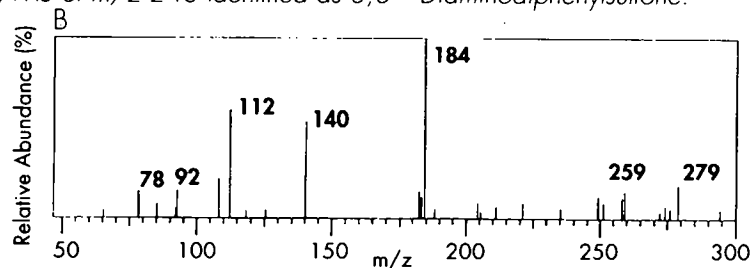
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Identification of a hardening agent in an epoxy resin by MS/MS.

Electron Ionization Mass Spectrum of an Uncured Epoxy Resin Mixture.



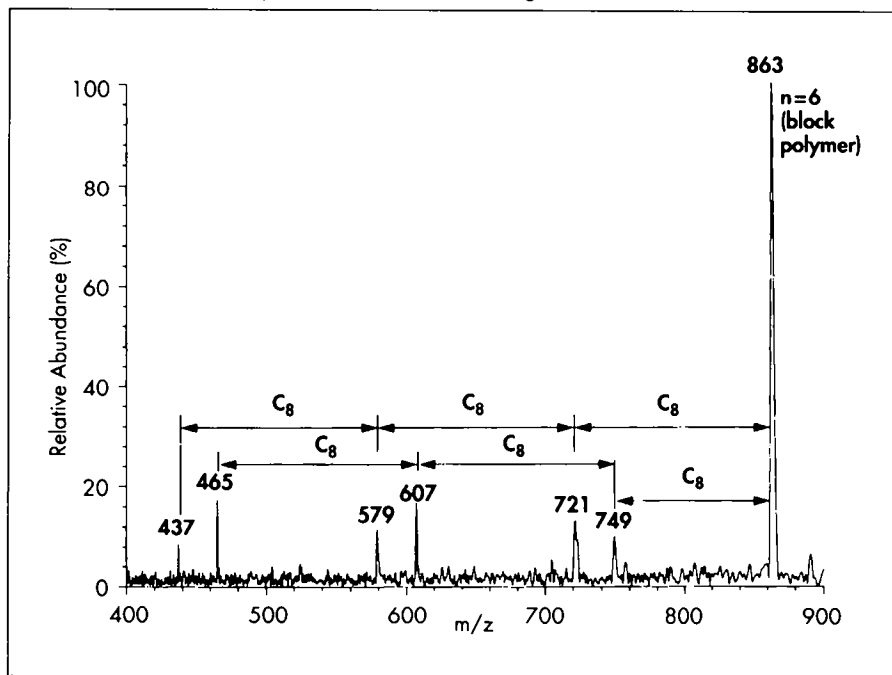
MS/MS of m/z 248 Identified as 3,3'-Diaminodiphenylsulfone.



Rapid characterization of industrial resins

By using the Extrel Laser Probe FT/MS method, the components of an uncured epoxy resin can be quickly identified. The identity of the starting material used in this epoxy resin mixture can be obtained from the mass spectrum of one of the ions (B) contained in the electron ionization mass spectrum of the resin mixture (A). A comparison of the mass spectrum of this ion to the mass spectrum of the same ion found in the spectral profile of 3,3'-Diaminodiphenylsulfone standard reveals similarities which identify it as one of the materials for the resin.

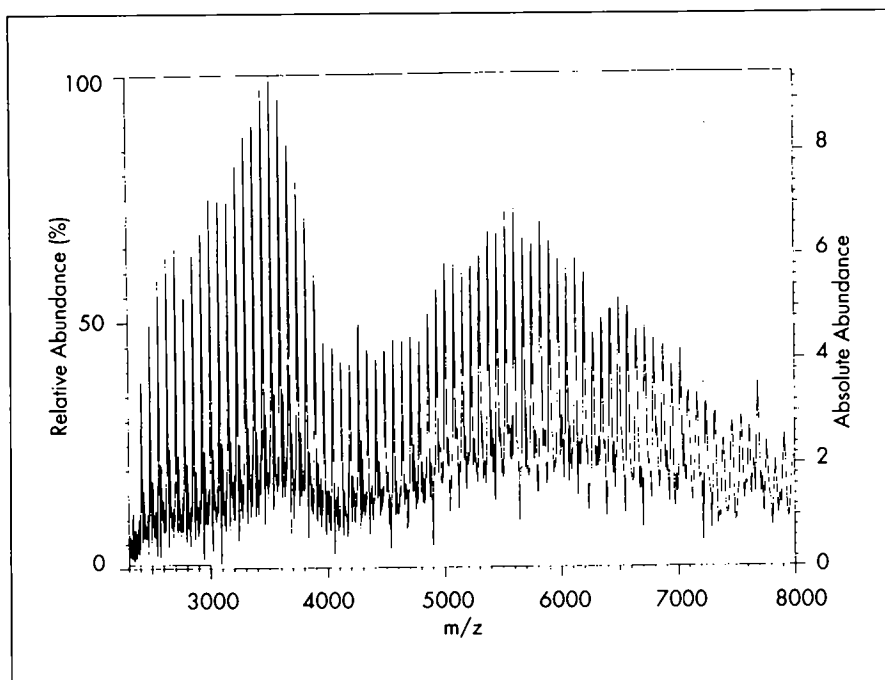
Mass spectra of a polymer and one of its oligomers.



Structure determination of all polymers

Determining polymer structures is a key component of today's polymer research. The structure of any polymer can be determined by the structural analysis of its comprising oligomers. By using the Extrel SWIFT™ method, it was determined that a bio-synthetic polyhydroxyalkanone used to manufacture a biodegradable plastic was composed of repeating C_8 units with one, two, and three C_6 units incorporated into the chain. The technique also indicated that a C_6 unit was located at the terminus of the oligomer.

Laser desorption FT/MS spectrum of polydimethylsiloxane.

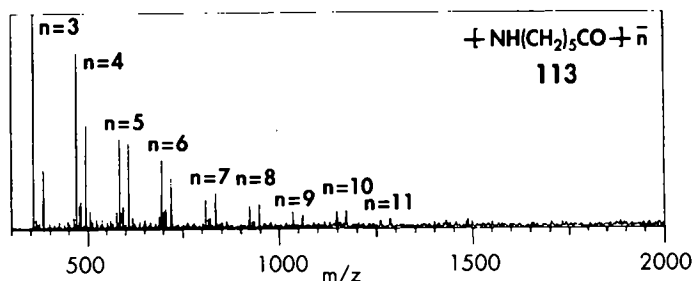


Fast and accurate molecular weight distributions of low molecular weight polymers

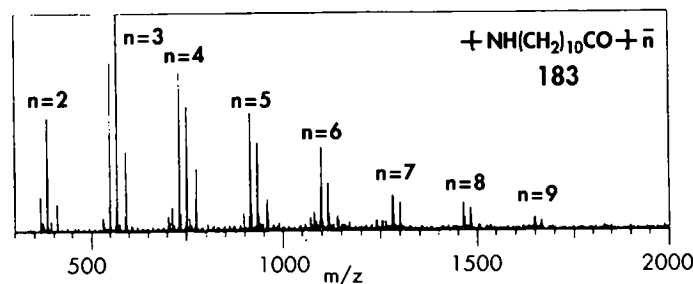
Determination of molecular weight distributions is important in predicting the physical properties of a polymer for a formulation or process. A lot of poly(dimethylsiloxane) was specified by the manufacturer to have a weight-average molecular weight of about 2,000. Direct analysis of a sample by the Extrel Laser Probe FT/MS revealed a bi-modal distribution starting at 2,400 and continuing to 8,000 giving a weight average of 5,300, well above that specified.

Laser desorption mass spectral fingerprint of nylon.

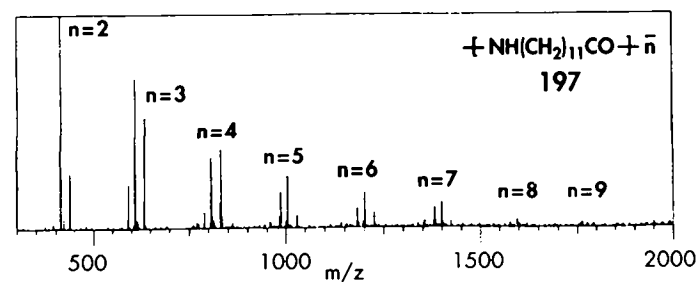
Nylon 6



Nylon 11



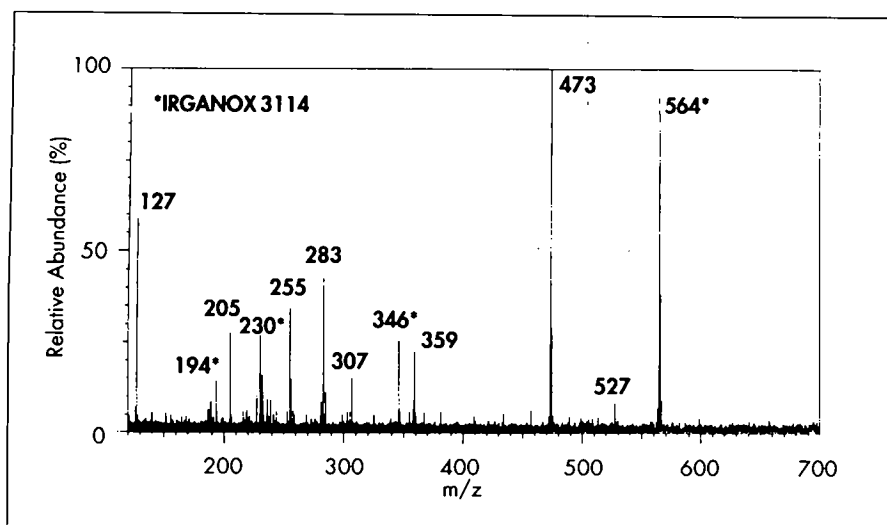
Nylon 12



Easy fingerprinting of difficult-to-analyze polymers

Obtaining a chemically-meaningful molecular fingerprint of intractable polymers is difficult with other analytical methods. For example, the Extrel Laser Probe FT/MS easily fingerprints Nylon 6, 11, and 12. The molecular weight of the repeating unit of each nylon can be obtained from the spacing of the peaks in the mass spectra.

Negative ion laser desorption spectrum of a polyethylene can extract.



Identification of extractables in polymer packaging and medical implants

The identification of extractable materials from polymers used to make medical implants, as well as food and pharmaceutical packaging, is becoming increasingly important. The Extrel Laser Probe FT/MS can quickly and easily identify the presence of an antioxidant (Irganox 3114) in the extract of a polyethylene can.

Worldwide service and support

Millipore Corporation, the manufacturer of Extrel products for MS and FT/MS, is the only company to provide you with everything you need — instrumentation, chemistries, and support — for HPLC, IC, CE, CIA, GPC, FPLC®, LC/MS, MS, and FT/MS, preparative chromatography, and amino acid analysis and synthesis. Our world-class service and support organization — staffed by highly-trained technical representatives, field engineers, and laboratory applications specialists — ensures your success.

For more applications information about Extrel products, call our FT/MS product specialists at (608) 273-8262 for further information.

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