EGA and the Identification of a Clear Polymer

Application Note

Polymer

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Pyrolysis-GCMS has commonly been thought of as an approach for polymer identification. But with some basic modifications, it is possible to use a traditional programmable pyrolyzer and GC/MS similar to a TGA-MS system. This Evolved Gas Analysis (EGA), performed with a properly programmed pyrolyzer, can offer some of the same information as an expensive Thermal Gravimetric Analyzers (TGA), such as thermal degradation profiles and compound identification. And, without the wait for a long GC run.

By replacing the analytical column of the GC/MS with a short piece of uncoated fused silica, it is possible to process a sample thermally with immediate transfer of the resulting volatiles to the mass spectrometer. The splitter of the injection port is still used to limit the amount of sample entering the mass spec, and one meter of 0.10mm fused silica provides enough restriction to permit the mass spec to maintain vacuum. The latest pyrolyzer from CDS, the model 6000, is programmable in degrees per millisecond, second or minute, providing an extremely wide range of heating profiles. In this example, a piece of clear plastic was heated to 250°C for 3 minutes to desorb volatiles, then at 100°C/min for polymer identification. The GC was held at 275°C for a 10 minute isothermal run, keeping the fused silica hot for transfer of the analytical products from the injection port to the mass spec. The resulting analytical run is shown in Figure 1.

Even though there is no chromatographic separation, all the analytical products are transferred to the mass spectrometer. At any point in time a spectrum is actually a composite of multiple compounds entering the mass spec at that time. Averaging the spectra for the run (or individual peaks in a run) provides a single spectrum containing information on all the products formed, as shown in Figure 2. The CDS Polymer Library can then be used to search for the best match among averaged spectra for known polymers. In this case, the best match is for poly ethylene terephthalate, as shown in Figure 2.

CDS Pyrolyzer 6200 Conditions:

Pyroprobe	
Initial:	250°C for 3 minutes
Ramp:	100°C/minute
Final:	700°C for 1 minute
Interface	
Rest:	300°C
Initial:	300°C for 0 minutes
Ramp:	0°C/minute
Final:	300°C for 10 minutes
Iso Zones	
Valve oven:	300°C
Transfer line:	315°C

GC Conditions:

Column:	1 m X 0.10 mm fused silica
Carrier:	Helium
Split:	100:1

Oven Program
275°C for 10 minutes isothermal

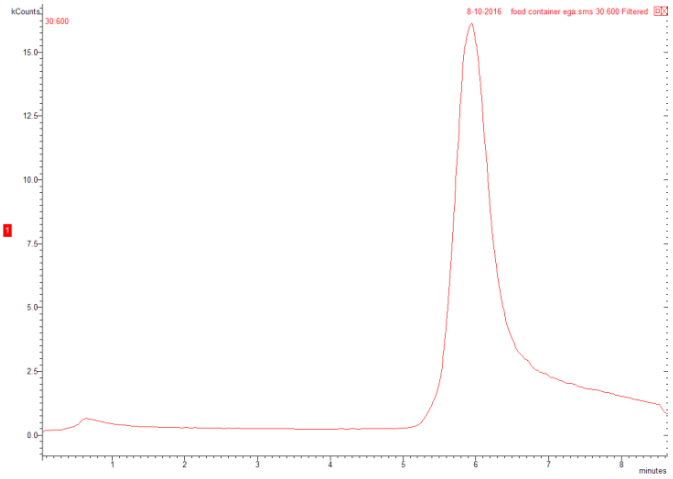

Figure 1. Clear polymer sampled at 100°C/min to mass spec

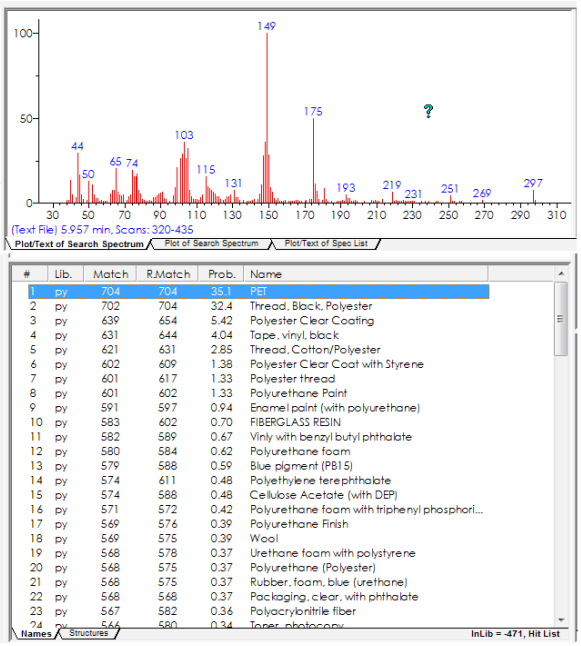

Figure 2. Averaged spectrum for run, searched using NIST and the CDS Polymer Library