

CDSolutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Py-GC HR-TOF of Circuit Board Polymer

Considering pyrolysis creates fragments which are typically not seen in traditional GC/MS(solvent) injections, interpretation may be tricky; some compounds are not found in traditional mass spectral databases. Py-GC HR-TOF can help in these situations. Below is an example of a Py-GC HR-TOF of a circuit board to illustrate this point.

When 100 μg of the circuit board was pyrolyzed at 750°C using nominal mass TOF (Figure 1a), bisphenol A along with isopropyl phenol in the pyrogram indicate that it is a polycarbonate material. However, there are also halogenated peaks such as bromomethane, bromophenols, and tetrabromobisphenol A (TBBPA), which are not common in polycarbonates; as well as, several unknown peaks with mass spectra indicating additional halogenated compounds.

When the same sample was run on LECO's Pegasus® GC-HRT (High Resolution Time of Flight Mass Spectrometer), accurate mass measurements offered by the HRT allowed molecular formulae to be determined for the previously unidentified peaks, which were determined to be mono, di, and tri bromobisphenol A (Figure 1b). From this added information, we can be reasonably sure the circuit board is a polycarbonate that uses tetrabromobisphenol A as an "in-polymer" flame retardant. That is, this flame retardant is part of the polymer as a finished product, and not added to the polymer afterwards.

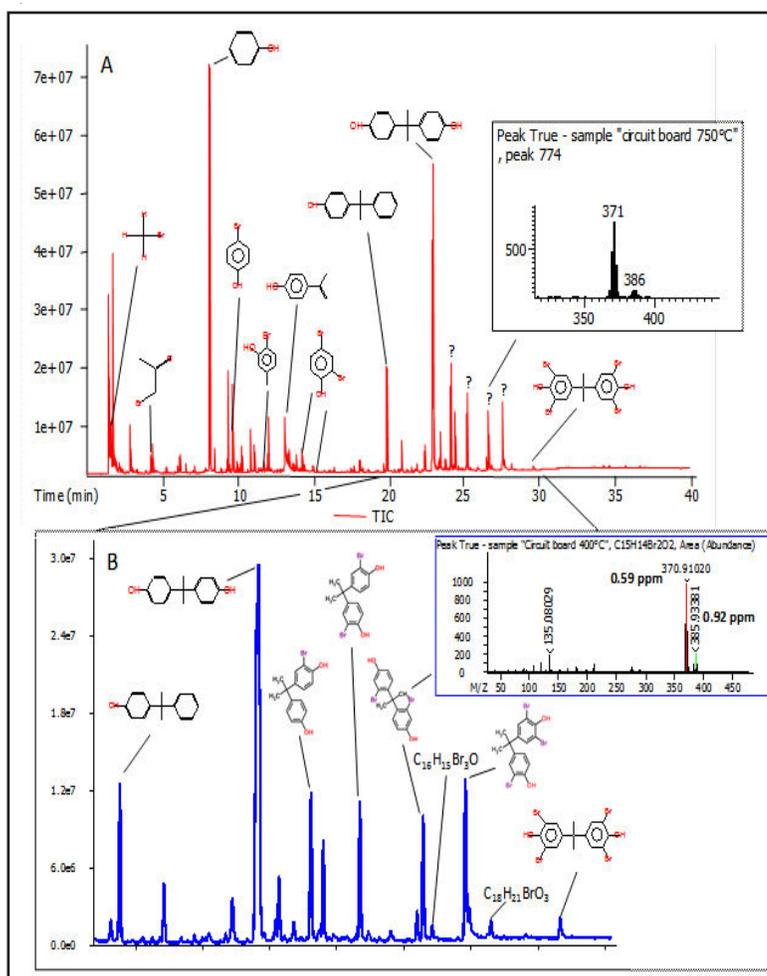


Figure 1: (A) Pyrogram of a circuit board at 750°C by TOFMS. (B) Pyrogram of the same circuit board at 400°C by high resolution MS (Pegasus GC-HRT), zooming in on the region with unknown halogenated compounds. The accurate mass measurements aided in identifying the unknowns which were not present in commercially available mass spectral databases.

Equipment

These samples were analyzed using a CDS Model 5200 Pyroprobe, interfaced to a LECO Pegasus GC-HRT

Model 5200 Conditions

Valve Oven: 325°C
Transfer Line: 325°C
Temperature: 750°
Time: 30 seconds
Interface Final: 325°C for 3 minutes

GC Conditions

Carrier: Helium
Injector: 325°C
Split: 50:1
Column: 5% phenyl(30m X 0.25mm)
Detector: HRT
Range: 35 - 550amu

GC Program:

Initial: 40°C for 2 minutes
Ramp: 10°C/min.
Final: 300°C for 5 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, LLC 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. 5000, 5150, 5200 and 5250 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 as well as purge & trap instruments for drinking and waste water. CDS also manufactures the Dynatherm line of thermal desorption instruments including the 9300 series for air monitoring and the 7500 autosampler. 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.