CDS olutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Evolved Gas Analysis of Polymers

Although the Pyroprobe is frequently used at high heating rates for flash Py-GC/MS, it may also be used to heat a sample slowly, in °C/minute. This may be done to a trap which is then desorbed to the GC, or, by replacing the GC column with a short piece of fused silica, directly to the mass spectrometer. In these experiments, the resulting data do not produce a chromatogram, but a plot of the compounds evolved from the sample as it is heated. The mass spectra of the compounds in the composite peaks provide information about the kinds of materials produced at that point in the heating program

Figure 1 shows the result of heating a sample of poly methyl methacrylate to 800°C at 100°C/ minute. Peak 1 and peak 2 are both essentially methyl methacrylate monomer. As the sample was heated, it first released residual (non-polymerized) monomer, producing peak 1. As the polymer was heated to higher temperatures, it underwent pyrolysis. When pyrolyzed, PMMA unzips to produce the monomer, yielding peak 2.

For the results shown in Figure 2, a sample of clear vinyl was heated rapidly to 250°C and held there for three minutes. At this temperature, the plasticizer DOP is released, making peak 1. The sample was then heated at 100°C/minute to 800°C. At a relatively low temperature, PVC releases HCl from the polymer backbone, which shows up as peak 2. The remaining, unsaturated polymer backbone then fragments at a higher temperature, forming peak 3, which contains a wide array of aromatic compounds, including benzene, toluene, xylene, indene and naphthalene.

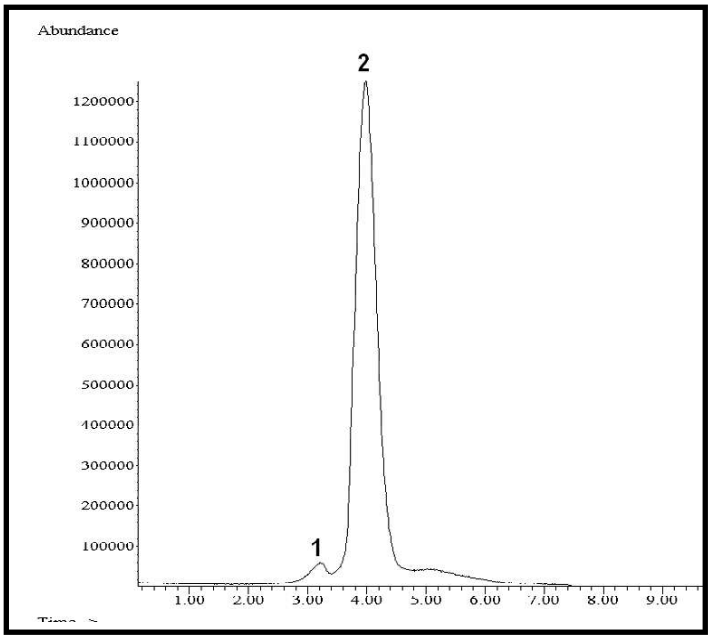

Figure 1. PMMA heated from 100° to 800°C at 100°C/minute.

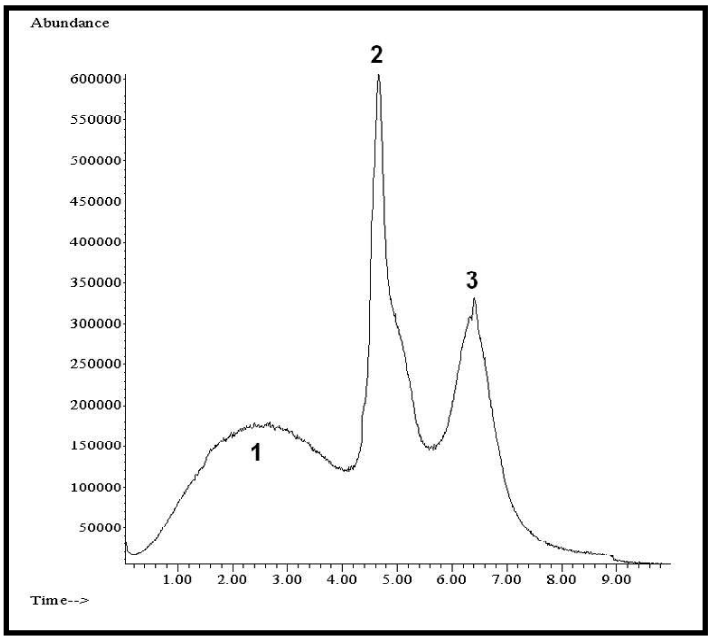

Figure 2. Clear vinyl sheet heated to 250°C for 3 minutes, then 100°C/minute to 800°C.

Equipment

Pyrolysis

CDS Analytical Pyroprobe 5200, in the Direct mode, interfaced to a GC/MS.

Pyrolysis

Initial:	100°C, 250°C
Ramp:	100°C/minute
Final:	800°C
Valve oven:	300°C
Transfer Line:	300°C

Chromatography

Column:	1 m x 0.1 mm fused silica
Oven:	250°C, isothermal 10 minutes
Injector:	300°C
Carrier:	Helium, 50:1 split
Mass range:	35 to 500 amu

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READING:

T. Wampler, Introduction to pyrolysis-capillary gas chromatography, J. Chrom. A, 842 (1999) 207-220.

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.

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