

Application Note

Multistep

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Most polymer based products also contain non-polymeric constituents, either retained small molecules such as residual monomers and solvents or additives such as plasticisers. Analyzing these volatiles and semi-volatiles traditionally requires long extraction times using solvents, which dilute the sample and present a disposal problem after the assay.

The analysis of these products may be simplified and automated using thermal sampling, which permits the separation of volatile and non-volatile components and eliminates solvents from the method. A first step, at a relatively low temperature, releases the smaller molecules from the polymer matrix for analysis. A subsequent, high temperature step pyrolyzes the polymer and permits identification and microstructural analysis.

The sample shown here was a clear packaging plastic, which included poly vinylchloride, poly methylmethacrylate and poly styrene, plasticized with dioctyl phthalate (DOP). Figure 1 is the first chromatogram, produced by heating the sample to 300°C for 15 seconds directly to the GC. This process released all of the DOP from the sample, without degrading the polymer.

When the GC was ready, the CDS Pyroprobe automatically produced the second run, this time at 750°C for polymer analysis. This pyrogram shows HCl, benzene, toluene and naphthalene, all PVC products, methyl methacrylate monomer from the PMMA, and styrene from the polystyrene.

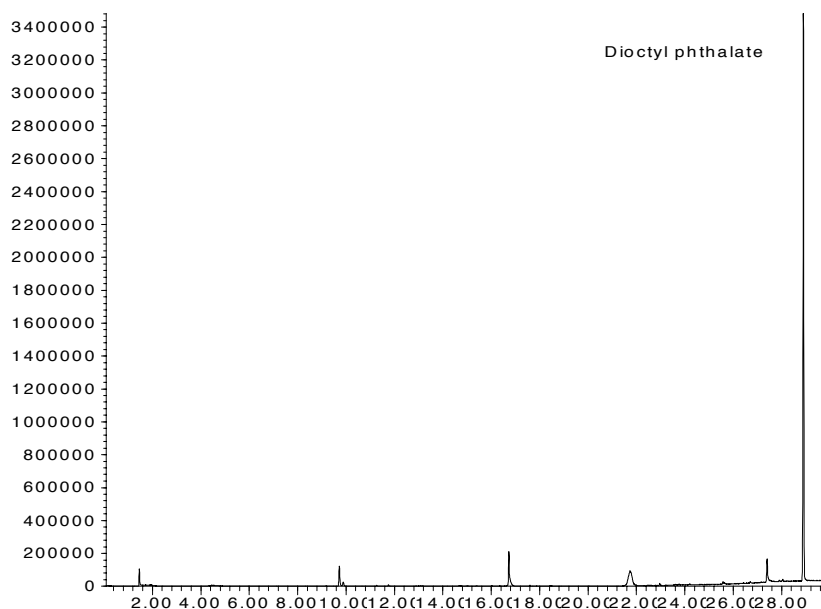


Figure 1. Dioctyl phthalate released at 300°C.

Instrument Conditions

Pyroprobe Autosampler

Valve Oven: 300°C
Temperature: 300°, 700°C
Time: 15 seconds

GC/MS

Column: 5% phenyl (30m x 0.25mm x .25µm)
Carrier: Helium, 50:1 split
Injector: 300°C
Oven: 40°C for 2 minutes
8°C/min to 300°C hold 12 minutes

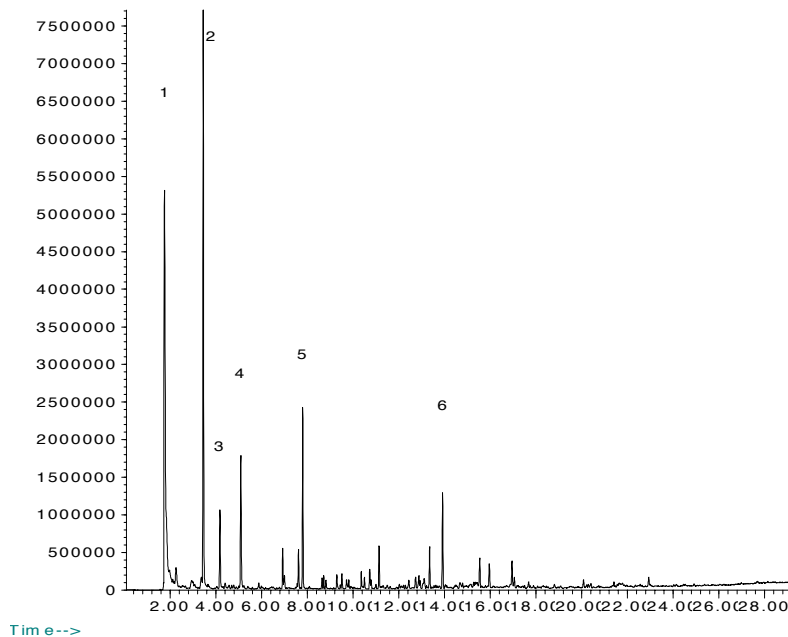


Figure 2. Pyrolysis at 750°C.

1) HCl, 2) Benzene, 3) MMA, 4) Toluene, 5) Styrene, 6) Naphthalene

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

T. P. Wampler, Introduction to Pyrolysis-Capillary Gas Chromatography, J. Chromatography A, 842 (1999) 207.