

## Two - Step Polymer Analysis

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 2500+ 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.

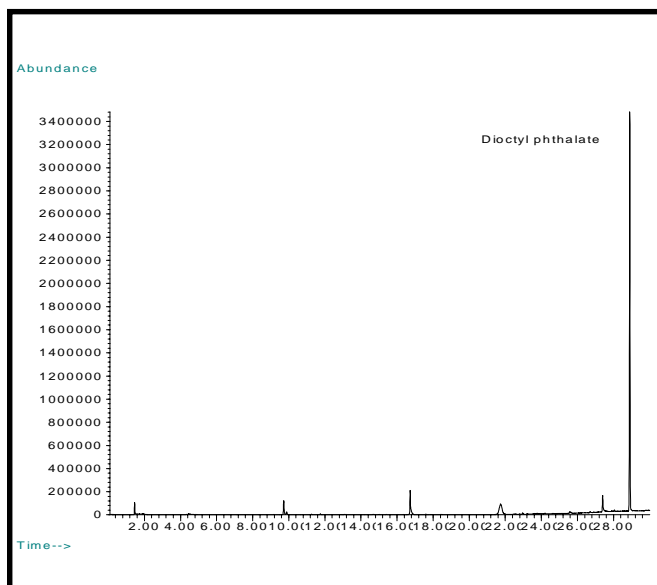


Figure 1. Dioctyl phthalate released at 300°C.

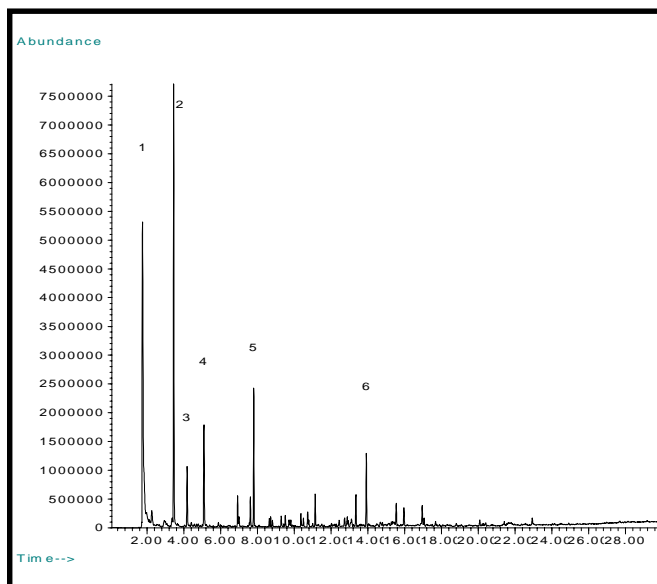


Figure 2. Pyrolysis at 750°C.  
1) HCl, 2) Benzene, 3) MMA, 4) Toluene, 5) Styrene, 6) Naphthalene

## Equipment

These analytical runs were produced using a CDS Model 2500+ Pyrolysis Autosampler, interfaced to an HP6890 gas chromatograph which was equipped with an HP5973 Mass Selective Detector.

### Model 2500 Conditions

Valve Oven: 300°C  
Temperature: 300°, 750°C  
Time: 15 seconds

### GC Conditions

Carrier: Helium  
Split: 50:1  
Column: HP-5 (30 m X 0.25 mm)  
Detector: MSD

### GC Program:

Initial: 40°C for 2 minutes  
Ramp: 8°C/min.  
Final: 300°C

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.

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, Inc. 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® 1000, 2000 and 2500 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, including the model 6500 16 position autosampler for complex, multicomponent materials investigation. 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](http://www.cdsanalytical.com).