CDS Solutions

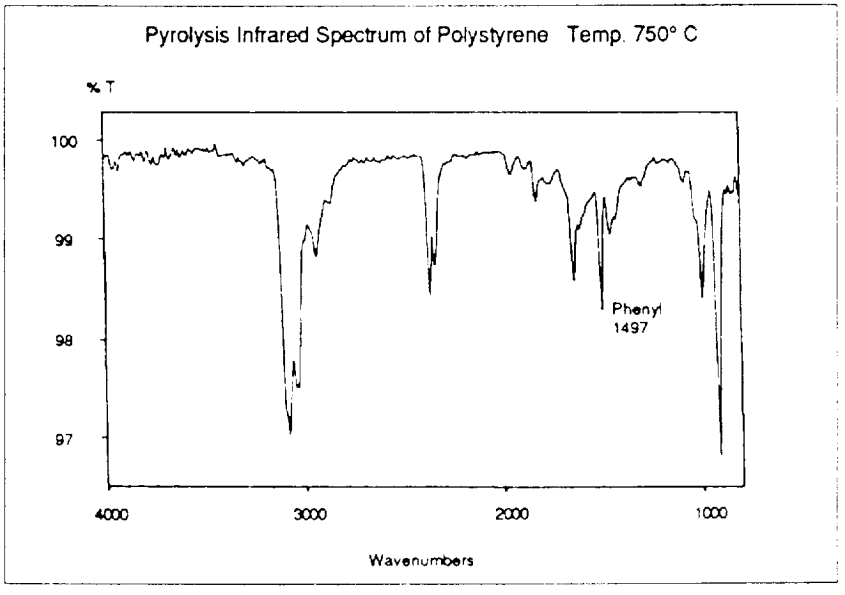
APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

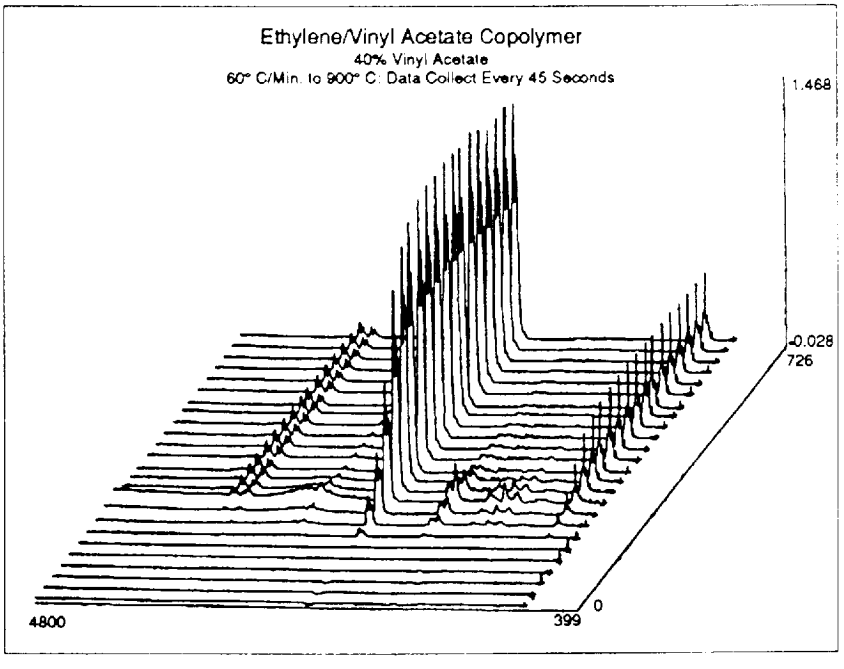
DIRECT PYROLYSIS/FT-IR: AN ALTERNATIVE SAMPLING SYSTEM

In polymer analysis, FT-IR has become a routine method for identification and characterization. For this purpose there are numerous sample handling techniques. Some of the more popular of these techniques are solvent casting and film pressing. For solid samples this can often be time consuming and sometimes ineffective.

Pyrolysis of polymer samples in the infrared beam can be used as a quick means of identification with very little sample preparation. Past efforts in pyrolysis/FT-IR did not feature direct pyrolysis in the infrared beam. Instead, samples were heated, usually with little temperature control, and the pyrolysate was condensed on an infrared transmitting plate. Allowing the products to condense can have a deleterious effect on the results due to side reactions. Rapid pyrolysis in the beam eliminates this possibility.

Direct Py/FT-IR is accomplished with a platinum filament pyrolyzer and a specially designed interface. The interface, a cylindrical cell with windows on each end, houses the Pyroprobe. Positioning of the Pyroprobe is crucial for direct IR measurements. With this interface the filament is located a few millimeters below the path of the beam. Upon pyrolysis, the volatile pyrolysate diffuses immediately into the beam where it is detected. Samples may also be heated at rates as slow as 0.01° C/ minute in order to do time-evolved studies and simulated distillations.





An example of direct flash pyrolysis/FT-IR is shown in Figure 1. Polystyrene has been pyrolyzed at 750° C at the maximum rate of 20,000° C/second. The rapid heating causes the polymer to depolymerize forming styrene vapor. The resulting spectrum is that of vaporphase styrene monomer. Particular bands to notice are the phenyl ring stretch and the vinyl stretch at 970 cm^{-1}.

In order to demonstrate the versatility of this system, Figure 2 displays ethylene/vinyl acetate copolymer which was heated at 60° C/minute to a final temperature of 900° C. IR scans were taken every 45 seconds and plotted versus time. From this data it can be determined at which temperature the sample begins to degrade and what the initial degradation products are.

Direct Pyrolysis/FT-IR provides a quick and easy way to obtain qualitative identifications of polymers, quantitative data on monomer ratios of copolymers, determination of degradation mechanisms, kinetic studies and combustion analysis. With such a wide range of information that can be obtained, this system can have a wide range of applications in polymers, geochemistry and composite research.

EQUIPMENT PYROLYSIS

Pyroprobe model 1000 filament pyrolyzer, with temperatures continuously variable to 1400° C.

INTERFACE

Brill Cell for Direct Pyrolysis/FT-IR, containing ZnSe windows and sweep gas inlet ports.

DATA ACQUISITION

IBM PS2-70 with a Hewlett-Packard plotter.

For more information on this and related applications, we recommend the following readings:

J. T. Cronin and T. B. Brill, "Thermal Decomposition of Energetic Materials 26. Simultaneous Temperature Measurements of the Condensed Phase and Rapid-Scan FT-IR Spectroscopy of the Gas Phase at High Heating Rates." Appl. Spectr. 41, 1147, (1987).

J. W. Washall and T. P. Wampler, "Analytical Pyrolysis of Complex Multicomponent Samples." J. Chromatogr. Sci. 27, 144, (1989).

T. P. Wampler, "Thermometric Behavior of Polyolefins." J.A.A.P., 15, 187, (1989).

Additional literature may be obtained by contacting your CDS Instruments Representative, or by writing to the CDS Applications Lab.

ABOUT CDS

CDS Analytical, Inc. is a leader in the design and manufacture of laboratory instruments for sample preparation and analysis. With 20 years experience in the field, CDS is dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of analytical pyrolysis, CDS manufactures the Pyroprobe 1000 and 2000 for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of purge and trap instruments for the analysis of volatile organic compounds in the environmental, food and pharmaceutical areas, as well as custom systems for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet their 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, or call us at 1 800 541 6593.

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