CDSolutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Parameter Optimization in Pyrolysis/FT-IR

The technique of direct pyrolysis/ FT-IR makes use of a pyrolysis cell which is located in the sample compartment of the FT-IR spectrometer. A Pyroprobe, platinum filament pyrolyzer is positioned in the cell, so that the sample is only 3 mm. below the infrared beam. As the sample is pyrolyzed, the pyrolysate diffuses immediately into the beam with minimal dilution effects.

In this technique there are several variables which can effect the results. Pyrolysis temperature, cell temperature and sweep gas flow rate all contribute to ensure accurate results. Cell temperature is important in preventing condensation of the pyrolysate on the cell windows. Flow rates also need to be optimized to provide adequate ventilation of the cell without losing sensitivity.

Figure 1 shows the effect of varying cell temperature on linearity when pyrolyzing ethylene/vinyl acetate copolymers.
At temperatures greater than 100° C, linearity of this analysis is excellent. At lower temperatures, however, condensation can be a problem and this causes non-linearity.

Figure 1

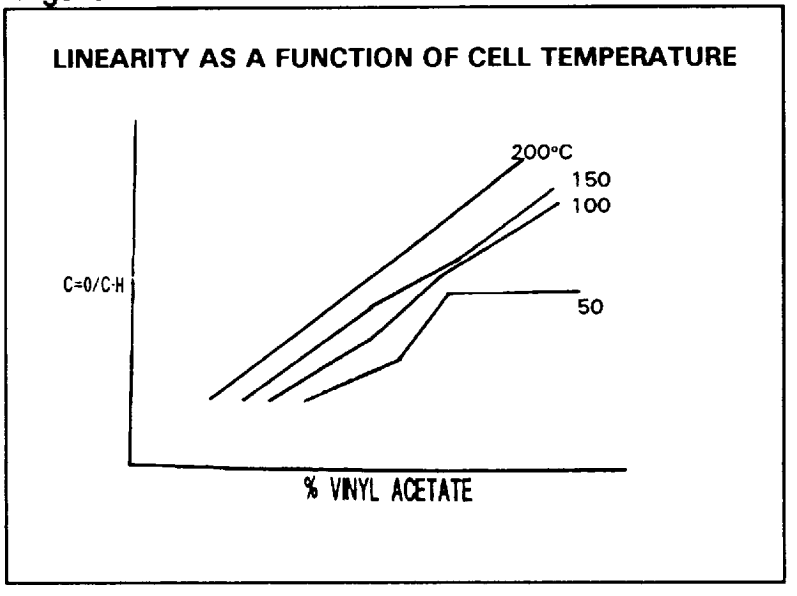

Figure 2

Cell Temperature °C	Cell Flow 0 ml/min	40 ml.min.	100 ml./min.
25	3.0%	3.6%	7.5%
100	5.0%	3.1%	1.7%
200	8.0%	0.6%	0.9%

As stated earlier, the purpose of the sweep gas is to prevent the pyrolyzed materials from condensing on the windows. Figure 2 shows the effect of varying cell flow with respect to window residue. After pyrolyzing polymer samples, a background was taken. Next a spectrum was obtained using the windows from the pyrolysis experiment. The residue spectrum was ratioed against the polymer spectrum to determine the percent of residue remaining on the windows. The optimal parameters for this type of analysis would include a cell temperature of 200° C and a cell flow rate of 40 ml/minute.

Equipment

PYROLYSIS

CDS Analytical Model 2000 Pyroprobe, Pyrolysis temperatures variable in 1° C increments to 1400° C. FT-IR interface was the CDS Analytical Brill Cell with heating capability to 250° C. Cell windows were standard 1/4" Zn/Se.

FT-IR SPECTROSCOPY

Nicolet 710 FT-IR spectrometer equipped with a DTGS detector. Scan speed 1 scan/second.

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

J. W. Washall and T. P. Wampler,"Direct pyrolysis FT-IR spectroscopy for polymer analysis", Spectroscopy, Vol 6.

J. W. Washall and T. P. Wampler, "A pyrolysis/FT-IR system for polymer quality control: A feasibility study", J. Appl. Polymer Sci, Applied Polymer Symposium, 45, (1990) 377-392.

Additional literature on this topic may be obtained by contacting your local CDS Analytical representative, or directly from CDS at the address below.

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 or 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.

CDS Analytical, Inc. 7000 Limestone Rd., P.O. Box 277, Oxford, PA USA 19363-0277 Ph: 215 932 3636