CDS solutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Pyrolysis Autosampling Reproducibility

To be analytically useful any technique must be shown to be reproducible. In the analysis of polymers by pyrolysis-gas chromatography, analysts are interested in reproducible retention times and peak areas for the pyrolysis products, and how they may be affected by the pyrolysis instrumentation used.

Automating sample introduction eliminates many of the operator associated variables which can adversely impact reproducibility. To demonstrate pyrolysis-GC reproducibility in the analysis of polyethylene, seven samples were loaded into the CDS Model 2500 autosampling Pyroprobe and pyrolyzed at 850°C to a capillary GC equipped with an FID. Figure 1 shows four consecutive runs, with the distribution of hydrocarbons - straight chain alkanes, alkenes and dienes - characteristic of polyethylene.

Retention times were averaged for decene (9.4 min) and undecene (12.5 min), and then the peak area ratio for these peaks was calculated for each run. The retention times averaged 9.424 ± 0.008 and 12.514 ± 0.008 respectively, and the peak area ratio averaged 1.284 ± 0.04, for a relative standard deviation of 3.08%.

In a separate evaluation, ten samples of a styrene/butadiene copolymer were pyrolyzed at 750°, again using the Pyroprobe 2500 autosampler. The average peak area ratio for the butadiene dimer peak to the styrene monomer in this case showed an RSD of 2.6%.

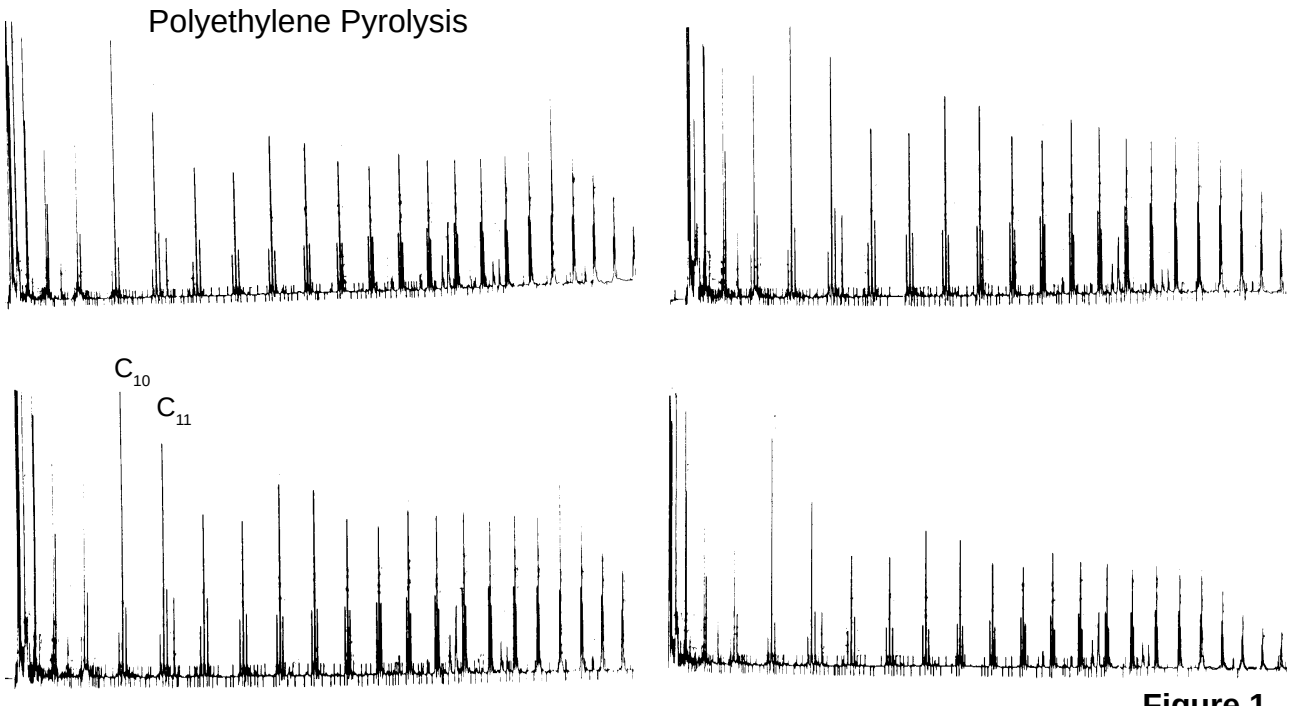

Figure 1.

REPRODUCIBILITY DATA

Item	Average	± SD	RSD
C_{10} Retention time	9.424 min	0.008	0.08%
C_{11} Retention time	12.514 min	0.008	0.06%
C_{10}/C_{11} Area ratio	1.284	0.04	3.08%
Butadiene dimer/ Styrene area ratio	0.114	0.003	2.63%

Equipment

All samples were pyrolyzed using a CDS Model 2500 Pyrolysis Autosampler interfaced to a Hewlett-Packard 5890 gas chromatograph with a flame ionization detector.

Pyrolysis

Interface oven:	300°C
Ramp:	10°C/ms
Temperature:	850°C
Time:	20 seconds
Clean:	1000°C for 10 seconds

Chromatography

Carrier:	He
Column:	HP-5
	30 m x 0.25 mm
Split:	75:1
Initial temperature:	40°C for 1 minutes
Ramp:	5°C/minute
Final temperature:	300°C for 12 minutes

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

Reproducibility in Pyrolysis, Recent Developments, T. Wampler and E. Levy, J.A.A.P., 12 (1987) 75-82.

Thermometric Behavior of Polyolefins, T. Wampler, J.A.A.P, 15 (1989) 187-195.

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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