Quantitative reactive pyrolysis of liquid crystal polyesters using the "sandwich" prep method

[Background] Technical note PYA2-025E describes the use of carbon-powder-coated filter papers and carbon-powder-containing TMAH reagent when doing reactive pyrolysis. This technical note describes the reactive pyrolysis of a three-dimensional copolymer liquid crystal polyester (LCP, Fig. 1) prepared from p-hydroxy benzoic acid (PHB), terephthalic acid (TA), and biphenol (BP). The composition ratios are reported.

[Experimental] Six different LCP samples (60 mesh powder) were analyzed (Table 1) using the so-called "sandwich" method. About 50 µg of each LCP sample was placed between two carbon-coated filters (diameter 4 mm, thickness 0.1 mm) in a sample cup (Eco-cup). 4 µL of the TMAH/carbon mixture were added directly to the LCP sample and to the top filter (8 µL total). The sample was analyzed using reactive pyrolysis GC at 400ºC. The Auto-shot sampler was used throughout the study.

[Results] The calibration curves for the three LCP constituents are shown in Fig. 2; the composition ratios are normalized using PHB. The molar composition ratios experimentally obtained (Table 2) are in a good agreement of those of the starting molar ratios (Table 1). The relative standard deviation (RSD, n=5) is less than 5%. This example demonstrates that reactive pyrolysis using the "sandwich" method to prepare the sample for reactive pyrolysis provides quantitative results with high precision. The method is compatible with the use of an auto-sampler which greatly increases laboratory productivity.

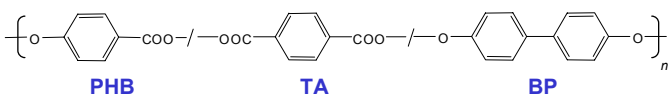

Fig. 1. Chemical structure of LCP

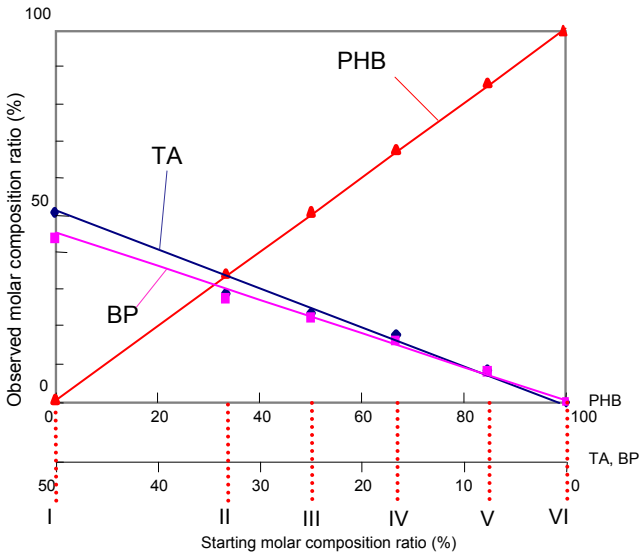

Fig. 2. Calibration curves for the six different LCP samples

Table 1. Molar composition ratios of the LCP samples

Sample No.	PHB	TA	BP
I	0	1	1
II	1	1	1
III	2	1	1
IV	4	1	1
V	11	1	1
VI	1	0	0

Table 2. Molar composition ratios actually obtained

Sample No.	PHB	TA	BP
I	0.0	1.0	0.89
II	1.0	0.86	0.82
III	2.0	0.94	0.89
IV	4.0	1.05	0.94
V	11.0	1.04	0.97
VI	1.0	0.0	0.0

RSD* (n= 5)

PHB: 1.1%, TA: 4.7%, BP: 2.6%

* Average results of 6 samples

Keyword : Quartz filter paper, Reactive pyrolysis, TMAH, Sample preparation, LCP, Quantification, Sandwich method, Auto-Shot Sampler

Applications : Sample preparation, Polymer analysis, Reactive pyrolysis

Related technical notes : PYA2-025E

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