

Determination of Average Molecular Weight of Polycarbonate by Reactive Py-GC in the Presence of Organic Alkali

[Background] Analytical pyrolysis techniques such as Py-GC and reactive Py-GC have been extensively used to characterize the end groups of various synthetic polymers. The average molecular weights (MW) of various polymers can often be precisely determined using quantitative information of the end group concentrations. This note describes the estimation of the average MW of an commercially available polycarbonate (PC) by means of reactive Py-GC in the presence of tetramethylammonium hydroxide (TMAH).

[Experimental] A polycarbonate (PC) sample synthesized using the solvent method (SM) was used. About 50 µg of a freezer-milled PC sample was subjected to reactive Py-GC at 400°C in the presence of 1 µL of 25wt% TMAH methanol solution.

[Results] Figure 1 shows a typical pyrogram of the PC sample obtained at 400°C in the presence of TMAH. In this pyrogram, *p*-*tert*-butylanisole (peak A) and the dimethylether of bisphenol-A (peak B), derived from the end groups and the main chain of the PC sample, respectively, are clearly observed. Because both terminals in the SM-PC molecules are completely end-capped with *p*-*tert*-butylphenoxy groups, the average molecular weight of the sample (\overline{M}_n) can be estimated from the two peak intensities:

$$DP = \frac{I_B / 15.4}{(I_A / 10.2) / 2} \quad (1)$$

$$\overline{M}_n = DP \times 254 + 326 \quad (2)$$

where *DP* is the degree of polymerization, I_A and I_B are the intensities of peaks A and B, respectively, and the divisors 10.2 and 15.4 are empirically-determined effective carbon numbers (ECN) of the respective compounds for a flame ionization detector. The values of 254 and 326 in equation 2 are the MWs of the monomer unit and the two end groups, respectively.

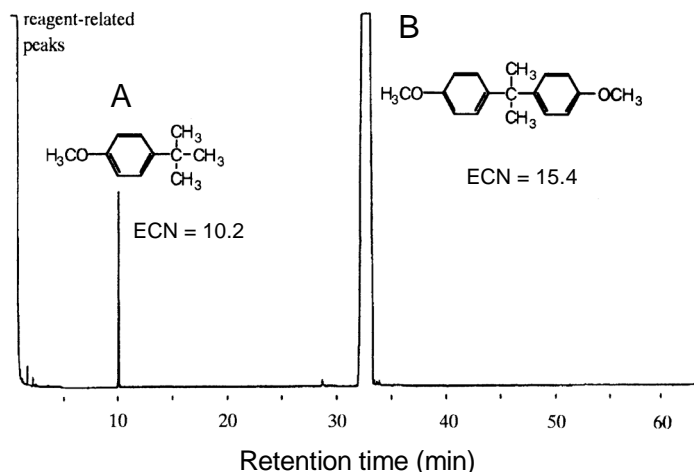


Figure 1. Pyrograms of PC sample obtained at 400°C in the presence of TMAH.

Pyrolysis temp. : 400°C, GC oven temp. : 50°C- (4 °C/min)-300°C
 Separation column : Poly(dimethylsiloxane), Length 25mm, 0.25mm i.d., Film thickness 0.25µm
 Carrier gas flow : 50 ml/min, Column flow : 1.3 ml/min, Detector: FID

*Contents excerpted from Y. Ito, H. Ogasawara, Y. Ishida, H. Ohtani, S. Tsuge, *Polymer J.*, **1996**, 28, 1090-1095.

Keyword : Polycarbonate, Reactive Py-GC, TMAH, End group, Number average molecular weight

Applications : Condensed polymer analysis

Related technical notes :

Please direct your inquiries via our web site at :

<http://www.frontier-lab.com/>

R&D and manufactured by :

Frontier Laboratories Ltd.

1-8-14 Saikon, Koriyama

Fukushima-ken 963-8862 JAPAN

Phone: (81)24-935-5100 Fax: (81)24-935-5102

© : Registered trademark of Frontier Laboratories Ltd.

Dealer