



Analysis of Terminal Groups of Polycarbonate (PC) by Reactive Pyrolysis

Determining the structure and distribution of a polymer's terminal groups is important, because these groups have a large influence on the material properties. Pyrolysis gas chromatography (Py-GC) is a rapid and effective technique for determining terminal groups. But in the simple pyrolysis of polymers such as polyesters, the pyrolysis products from the terminal groups are masked by a large peak arising from polar material in the polymer. To analyze such samples, reactive pyrolysis with tetramethyl ammonium hydroxide, TMAH, is used.

Strongly basic TMAH selectively cleaves the ester bond, allowing the resultant products to immediately undergo methyl esterification, resulting in a simplified pyrogram. Fig. 1 figure shows a pyrogram of reactive pyrolysis of polycarbonate using TMAH. Bisphenol A is formed from the polymer main chain, and a methylester of p-tert-butylphenol is formed from terminal groups. In Table 1, the determination of the terminal group shows an extremely high reproducibility of 0.61% RSD. This analysis is best done with a pyrolyzer with a vertical furnace with rapid gravity-driven sample introduction, so that the sample temperature can be kept at ambient, then very rapidly raised to the pyrolysis temperature. This avoids sample loss or decomposition by secondary reactions during slow heating.

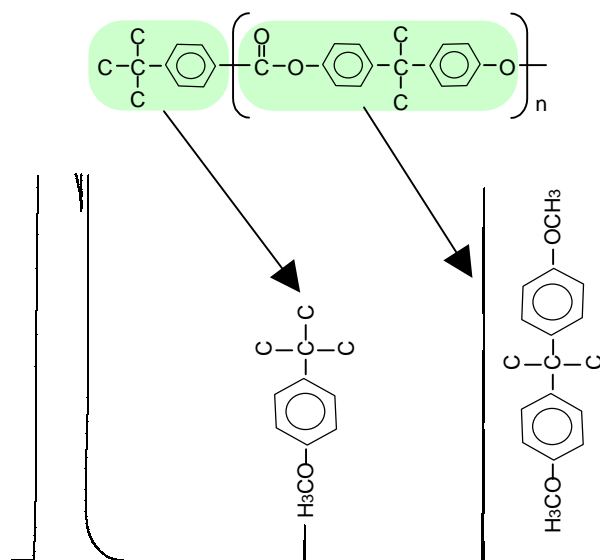


Table 1. Quantification of Terminal Group and Reproducibility

n	Peak area of methyl p-tert-butylphenol (%)
1	4.12
2	4.12
3	4.14
4	4.14
5	4.11
6	4.18
Average	4.135
RSD	0.61%

Fig. 1 Cleavage Mechanism of PC in Reactive Pyrolysis, and Pyrogram

Pyrolysis temp.: 400°C, Carrier gas : He 140kPa, 80ml/min, Column : 5% diphenyldimethylpolysiloxane, 30m, 0.25mm id.
Layer thickness 0.25µm, (Ultra ALLOY®-5, Frontier Laboratories Ltd.)
GC oven temp. : 40°C (1min) → 220°C/min → 320°C, GC injection temp.: 320°C, Sample: 20µg, TMAH : 3uL, Detector : FID

Keyword : Reactive Pyrolysis, Polycarbonate, Terminal Group, TMAH

Application : General Polymer Analysis

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