Unequivocal Identification of Pyrolysis Products by Retention Index Data

[Background] The concept of Kovat's retention index (RI), is used widely in the field of gas chromatography. The indices assist in: (1) the unequivocal identification of "unknowns" and 2) the prediction of retention times for pyrolysis products. This note presents the unambiguous identification of a characteristic product, related to the xanthone structure, one of the abnormal structures in thermally-treated polycarbonate (PC) samples. This compound is commonly founded in pyrograms obtained by reactive Py-GC in the presence of tetramethylammonium hydroxide (TMAH).

[Experimental] An commercial PC sample was thermally treated to cause abnormal structures in the presence of atmospheric oxygen at 400ºC for 1 hr using an electric furnace. The thermally-treated PC sample was ground into fine powder using a freezer mill. Approximately 100 µg of the sample was subjected to reactive Py-GC at 400ºC in the presence of 2 µL of 25% TMAH methanol solution. The RI data for the main pyrolysis products were determined by comparing the retention data for n-alkanes obtained using the same separation column. The alkanes were formed during the pyrolysis (at 600ºC) of polyethylene.

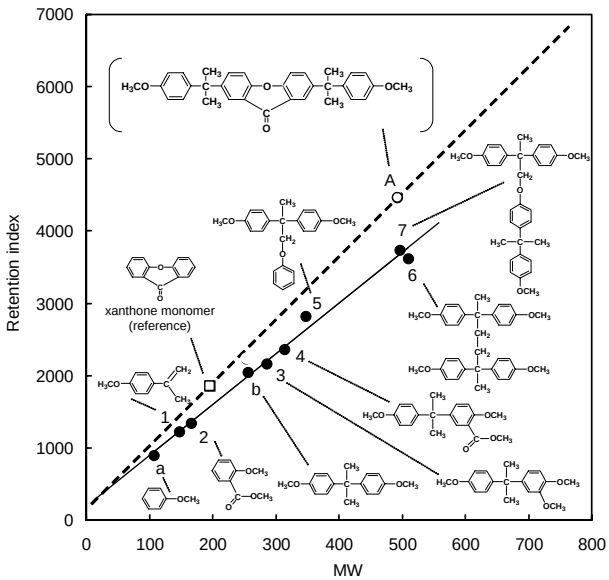

Figure 1. Relationship between molecular weight and retention index for pyrolysis products observed on pyrogram of thermally-treated PC sample.

[Results] The pyrogram of the PC sample, exhibited the characteristic product (peak A), presumed to be derived from the xanthone structure. In addition to peak A, the degradation products from the main chain (b), end groups (a) and other types of abnormal structures (peaks 1-7) are observed. Figure 1 shows the relationship between molecular weight and RI for the main degradation products (a, b and peaks 1-7; solid circle) and peak A (open circle) along with the reference data obtained for the xanthone monomer (open square). The solid line was obtained by the method of least squares applied to peaks a, b and 1–7. Although the data for the products designated by solid circles show an almost linear relationship, the data for peak A deviated to some extent at higher RIs. Furthermore, the line connecting the plots of the xanthone monomer and peak A almost pass through the origin of the coordinate axes. These results strongly suggest that these two products contain the xanthone structure but belong to a different class of homologue.

*Contents excerpted from K. Oba, H. Ohtani, S. Tsuge, *Polymer Degradation Stab.* **2001**, 74, 171-176.

Keyword : Retention index, Identification, Reactive Py-GC, TMAH, Polycarbonate, Xanthone structure

Applications : Condensed polymer analysis, General GC analysis

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