

Effect of Coexisting Inorganic Impurities on Reactive Py-GC in the Presence of Organic Alkali

[Background] The reactive Py-GC technique in the presence of an organic alkali, such as tetramethylammonium hydroxide [TMAH, (CH₃)₄NOH] has been successfully applied to precise compositional analysis of intractable condensation polymers. However, it has been suspected that the presence of inorganic salt impurities might hinder the quantitative reaction between the condensation polymer sample and organic alkali. In this report, the effect of KOH as a possible coexisting impurity on the reactive pyrolysis was described for the sake of the improvement of reproducibility and quantitiveness of the technique.

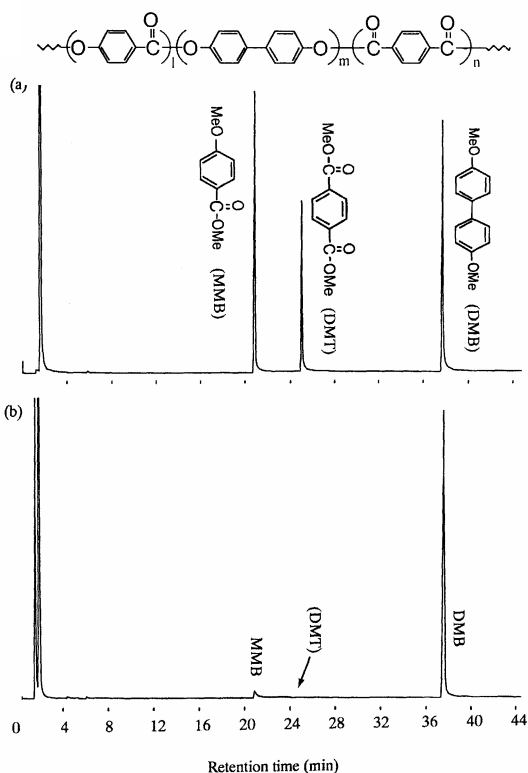


Figure 1. Typical pyrograms of aromatic polyester sample obtained at 400°C, (a) in the presence of 25% TMAH aqueous solution and (b) TMAH-KOH (10 wt%) aqueous solution.

*Contents excerpted from Y. Ishida, H. Ohtani, S. Tsuge, *J. Anal. Appl. Pyrolysis* **1995**, 33, 167.

[Experimental] An aromatic polyester sample, prepared with *p*-hydroxybenzoic acid (PHB), terephthalic acid (TA) and 4,4'-biphenol (BP) (PHB:TA:BP = 2:1:1), was used. About 100 µg of the cryo-milled polyester sample was subjected to reactive Py-GC at 400°C in the presence of 25% TMAH aqueous solution, or the TMAH solution containing KOH (10 wt%).

[Results] Figure 1(a) and (b) show typical pyrograms of the polyester sample obtained in the presence of pure TMAH and TMAH-KOH solution in water, respectively. In this figure, the peak intensity of DMB remained almost unchanged with or without addition of KOH while those of MMB and DMT nearly disappeared from the pyrogram measured in the presence of KOH. This result suggests that the presence of K⁺ ions significantly hinder the quantitative methylation of the carboxyl sites contained in the polyester sample presumably due to the formation of their corresponding potassium salts. Therefore, contamination by the inorganic salt impurities should be avoided when polyester samples are to be subjected to quantitative Py-GC analysis in the presence of TMAH.

Keyword : Aromatic polyester, Reactive Pyrolysis, Py-GC, TMAH, Inorganic alkali, impurities

Applications : Condensation polymer analysis

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