

Effect of elapsed time after the addition of tetramethylammonium hydroxide in reactive pyrolysis

[Background] Reactive Py-GC, which is performed in the presence of a strong alkali such as tetramethyl ammonium hydroxide [TMAH, (CH₃)₄NOH], is widely used for the precise characterization of condensation polymers and fatty acids.(1) The results obtained when using this analytical method are affected by factors such as the hydrolysis (i.e. furnace) temperature, the reagent used, the sample form, impurities, etc. This note reports the effect that the time interval between adding the reagent and the actual hydrolysis and methylation has on the analytical results.

[Experimental] Nine micrograms of polycarbonate (PC) was placed in a sample cup, followed by the addition of 3 μL of a methanolic solution of TMAH (25wt%). Reactive Py-GC/MS was performed (at 400°C) at different intervals (3 minutes to 5 hours).

[Results] Fig. 1 shows pyrograms obtained at elapsed times ranging from 3 minutes to 5 hours after the addition of TMAH. Pyrogram A with an elapsed time of 3 min, has a peak for the dimethyl ester of bisphenol A and the methyl ester of 4-*t*-butylphenol, which originates from the from the terminal group. Methanol and trimethylamine from the TMAH are also observed. As the time interval increases, the peak intensities and their ratio for the two peaks originating from PC remained unchanged; however, peaks for amines and methyl esters not observed in pyrogram A and not derived from PC were observed, and these peaks became larger as the sample sets after the addition of TMAH. These results show that when using TMAH, the elapsed time between the addition of the reagent and the thermal hydrolysis does not have much affect much on the analytical result; however, as a general rule, it is best if the hydrolysis occurs as soon as possible once the reagent is added.

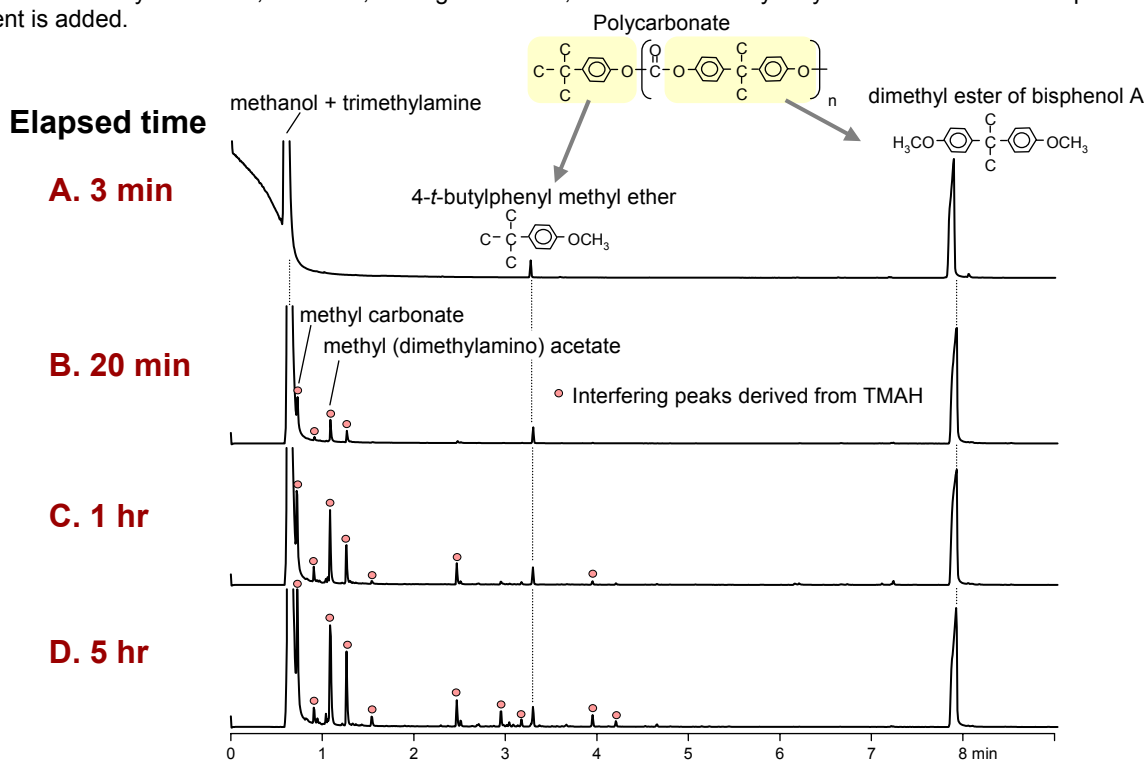


Fig. 1 Pyrograms of polycarbonate after addition of reagent, arranged in chronological order

Pyrolysis temp.: 400°C, GC oven temp.: 60 – 260°C (20°C/min), separation column: Ultra ALLOY+ -1 (dimethylpolysiloxane) L=30 m, i.d.=0.25 mm, df=0.25 μm, Column flow rate: 1 mL/min He, split ratio: 1/50, sample: 9 μg (solvent casting)

1) Y. Ito, et al., *Polym. J.*, 28(1996), 1090

Keyword : Reactive pyrolysis, TMAH, Polycarbonate

Applications : General polymer analysis

Related technical notes : PYA2-001, PYA2-015, PYA2-019

Please forward your inquiries via our web page or send us a fax message.

R&D and manufactured by :
Frontier Laboratories Ltd.

1-8-14 Saikon, Koriyama,
Fukushima 963-8862 JAPAN
Phone: (81)24-935-5100 Fax: (81)24-935-5102
<http://www.frontier-lab.com/>