

Determination of the Cross-linking Agent in Cross-linked Polycarbonate by Reactive Pyrolysis GC/MS

[Background] Increasing the melt viscosity of polymeric materials is often required in order to improve their molding properties, or to increase the molecular weight of recycled polymeric materials. For example, the melt viscosity of polycarbonate (PC) can be fine-tuned by introducing small amount of cross-linking agent into the polymer backbone chain of PC using 1,1,1-tris(parahydroxyphenyl)ethane (THPE) as a cross-linking agent. The concentration of the cross-linking agent incorporated into the PC will greatly influence the polymer's properties; therefore, it is important to determine the concentration of THPE incorporated into the PC. The conventional method requires cumbersome pre-treatments such as hydrolysis followed by solvent extractions, which are time consuming and adversely affect data quality. This technical note describes the development of a simple, rapid and quantitative method for the determination of THPE in PC. The method is based on reactive pyrolysis utilizing tetramethyl ammonium hydroxide (TMAH) as the derivatizing agent.

[Experimental] A Frontier double-shot pyrolyzer (Model 2020iD) was directly interfaced to the split/splitless injection port of a GC-FID. PC material that had been cross-linked with THPE was dissolved in dichloromethane (20 mg/mL). Five µL of the solution (equivalent to 100µg of PC) was placed in a deactivated sample cup. The solvent was evaporated, and 10µL of a 25% methanol solution of TMAH was added to the cup. Reactive pyrolysis was performed using a furnace temperature of 400°C.

[Results] Figure 1 shows that two reaction products are expected from the reactive pyrolysis of cross-linked PC in the presence of TMAH. The pyrogram is shown in Figure 2. As expected, a dimethyl derivative of bisphenol A (Me-BisA) originating from the major component of cross-linked PC and a trimethyl derivative of THPE (Me-THPE) originating from the cross-linking reagent are observed. The amount of cross-linking agent can be determined using the peak areas. The amount of THPE in the cross-linked PC was found to be ca. 0.4 wt%. The reproducibility of this method (RSD of 1.2 % (n=5)) is much better than that obtained using conventional methodology.

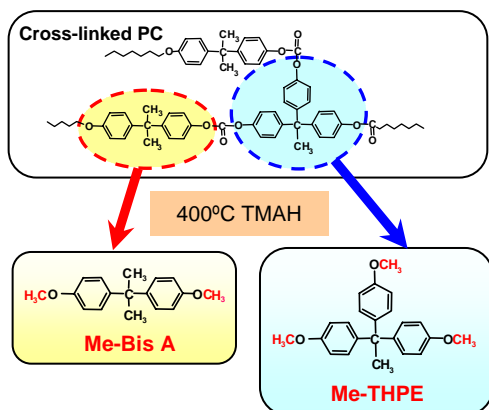


Fig. 1 Expected products from reactive pyrolysis of cross-linked PC

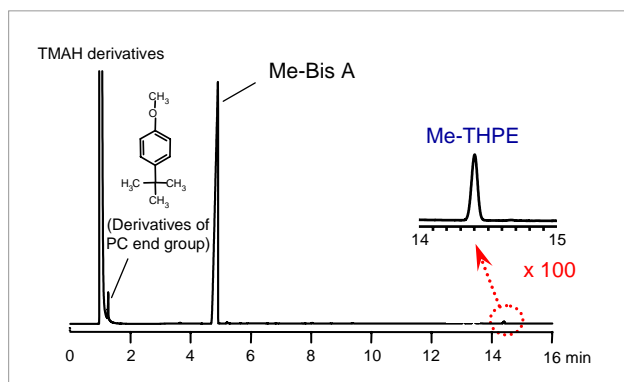


Fig. 2 Pyrogram obtained by reactive pyrolysis of cross-linked PC

Detector: FID, Separation column: Ultra ALLOY®-5 (5% diphenyldimethyl polysiloxane) (length 30 m, id 0.25 mm, film thickness 0.25 µm), Reactive pyrolysis temp.: 400°C, GC oven temp.: 200-320°C(5°C/min), GC injection port temp.: 300°C, Carrier gas: He, Split ratio: 1/200, Flow rate: 1.0 mL / min

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