

Quantitative analysis of a cross-linking agent in polycarbonate using reactive pyrolysis - GC/MS

K.Odagiri^{a)}, S.Takeda^{a)}, C.Watanabe^{a)}, S.Tsuge^{a),b)} and R.R. Freeman^{a),c)}

a) Frontier Laboratories, b) Nagoya University, c) Quantum Analytics

E-mail: kiri@frontier-lab.com

Polycarbonate (PC) is often the polymer of choice in many commercial and household products, due to its durability, clarity and temperature stability. Cross-linking agents such as 1,1,1-Tris(*p*-hydroxyphenyl)ethane (THPE) are often used to increase the melt viscosity and average molecular weight of the polymer. Because of its impact on the characteristics of the polymer, it is important to know the final concentration of THPE incorporated into the polymer. This analysis normally involves hydrolysis of the PC followed by solvent extraction. The extract is then analyzed chromatographically. This methodology is cumbersome and often results in poor data quality.

The work presents a new approach for analyzing THPE in cross-linked PC. It is based upon reactive pyrolysis – GC⁽¹⁾. Preparation of the sample is simple and straightforward – a known amount of PC is dissolved in a known volume of a solvent, such as methylene chloride to obtain 20 mg (PC) /ml(CH₂Cl₂) solution. Five microliters of this solution containing ca. 0.1mg (PC) along with 10μL of tetramethyl ammonium hydroxide (TMAH) [25% in methanol] are placed in a small sample cup. The sample is ready for analysis.

Analysis is performed using a multi-functional pyrolyzer (model 2020iD, Frontier-Lab, Japan) interfaced to a GC/MS. The sample cup is dropped into the furnace, which is at 400°C. The heat initiates the hydrolysis of the cross-linked PC followed by methylation of the hydrolyzates.

During the development of this method, both evolved gas analysis (EGA) and flash pyrolysis (Py) were evaluated. The results from these studies will be presented; however, both methods proved to be inadequate for the quantitative determination of “bound” THPE in polycarbonate. The ester linkages in cross-linked PC should be amenable to thermal hydrolysis followed by in-situ derivatization, that is, reactive pyrolysis precision and accuracy data obtained by the developed method clearly illustrate that this method is practically applicable to make quantitative analysis of the cross-linking agent in PC.

(1) Y. Ishida, S. Kawaguchi, Y. Ito, S. Tsuge, H. Ohtani, *J. Anal. Appl. Pyrolysis*, **40-41**, 321-329, 1997