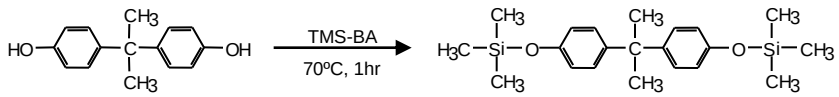
Determination of Residual Bisphenol A in Polycarbonate by Thermal Desorption (TD)-GC/MS with TMS-Derivatization

[Background] A trace amount of unreacted bisphenol A (Bis A), the starting material for polycarbonate (PC), is known to remain in the PC matrix as a residue despite efforts to remove it in the manufacturing process. Bis A is an alleged endocrine disruptor, therefore, the determination of residual Bis A in PC is extremely important. When thermal desorption (TD)-GC/MS is used as an analytical tool, a small amount of Bis A is also generated as a decomposition product of PC making the determination of residual Bis A originally present difficult. This report describes a new method for the determination of residual Bis A in PC. Residual Bis A is converted to a thermally stable trimethylsilyl derivative, prior to TD-GC/MS analysis. The method is simple and provides an accurate measurement of residual Bis A.

[Experimental] About 10 mg of the PC sample is placed in a vial along with 700 µL of dichloromethane. 300 µL of N,O-bis(trimethylsilyl)acetamide (TMS-BA) is then added to the vial. The vial is heated at 70ºC for 1 hour. A small amount of n-$C_{19}H_{40}$ is added in order to monitor the system performance. 10 µL of the reaction mixture (equivalent to ca. 0.1 mg of PC) was placed in a sample cup, the solvent was allowed to evaporate prior to TD-GC/MS analysis.

[Results] Scheme 1 shows the reaction products from the TMS-derivatization of Bis A. Fig. 1 shows the chromatogram obtained by TD-GC/MS of the reaction products. As shown in Scheme 1, hydroxyl groups at both ends of Bis A were TMS-derivatized to give 2,2-bis(4'-trimethylsiloxyphenyl)propane (TMS-Bis A). Because TMS-Bis A is less polar than Bis A, there is less degradation due to active sites in the injection port and separation column. This improves the over all peak shape and enhances reproducibility – see Fig. 1. Using an absolute calibration curve obtained using standard Bis A reagent, the concentration of Bis A in PC was found to be 1,170 ppm, with very good reproducibility (RSD=4.7%, n=5). When the thermal desorption temperature is less than 300ºC, the base polymer of PC remains in the sample cup unchanged; thus, the analytical system is not contaminated by the thermal decomposition products of PC.



Scheme 1 TMS-derivatization of Bisphenol A

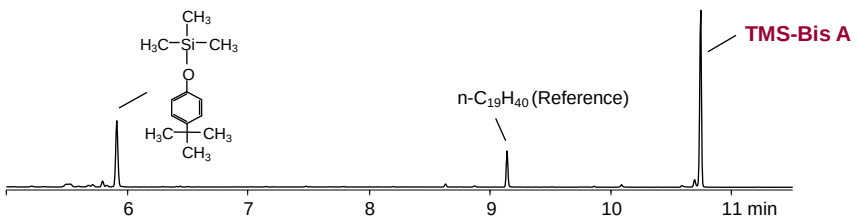


Fig. 1 Chromatogram of TMS-derivatized PC sample obtained by TD-GC/MS

Pyrolyzer furnace temp.: 100-300ºC (100ºC/min), GC oven temp.: 100-300ºC (20ºC/min, 5 min hold)
Separation column: Ultra ALLOY-5 (5% diphenyl 95% dimethylpolysiloxane, L=30 m, id.=0.25 mm, df=0.25 µm)
Carrier gas flow rate: 1.0 mL/min, split ratio: 1/50

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Keywords : Polycarbonate, Bisphenol A, Trimethylsilanation (TMS), Trimethylsilyl derivatization, Thermal desorption-GC/MS

Products used : Multi-functional pyrolyzer, Vent-free GC/MS adapter, UA-5

Applications : General polymer analysis

Related technical notes :

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