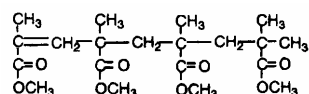
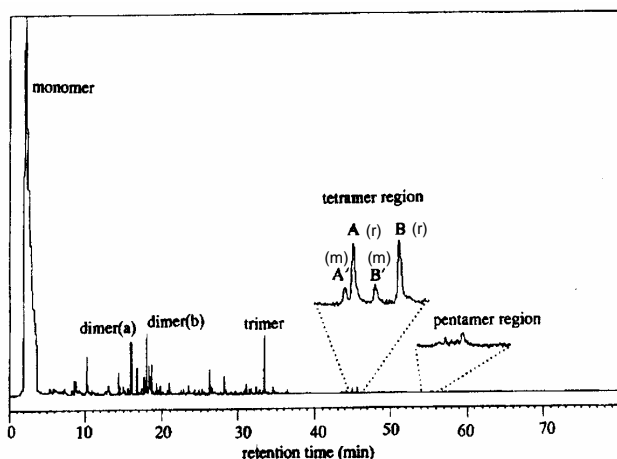


Stereoregularity of Poly(methyl methacrylate)s Studied by Pyrolysis-Gas Chromatography/Mass Spectroscopy

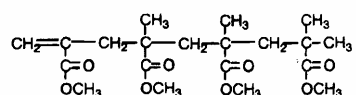
[Background] Py-GC has been successfully applied to the analysis of the stereoregularity of various vinyl polymers. Stereoregularity is a function of the diastereoisomers which are larger than the tetramers, which have at least two asymmetric centers. Moreover, Py-GC results are used to characterize the tacticity of vinyl polymers consisting of relatively larger monomer units, such as polystyrenes (PSs) and poly(methyl methacrylate)s (PMMA). This note presents the study of the tacticity of PMMA samples.

[Experimental] A series of PMMA samples with known and tacticity values, determined by ^1H NMR, are analyzed. About 400 μg of the sample is subjected to Py-GC at 500 $^\circ\text{C}$. A deactivated metal capillary column is temperature programmed from 50 to 340 $^\circ\text{C}$ which facilitates the separation of the diastereoisomers.

[Results] Figure 1 shows a typical pyrogram of the PMMA sample ($m : r = 24.0 : 76.0$) obtained when pyrolyzed at 500 $^\circ\text{C}$. This pyrogram clearly contains minor peaks for MMA dimers, trimers, tetramers (about 0.1%) and even pentamers (about 0.03%) in addition to the main monomer peak. The chemical structures of the two tetramers, A and B, are estimated using their EI and CI mass spectra. Additionally, the small peaks (A' and B') appearing a little earlier than those of the main tetramers (A and B) are identified as the corresponding diastereoisomers. Thus, estimated tacticity values, calculated using the peak intensities of either the tetramer pair, A and A', or B and B', agree with those obtained by ^1H -NMR.



tetramer A (racemo; r)



tetramer B (racemo; r)

Figure 1. Typical pyrogram of PMMA sample ($m : r = 24.0 : 76.0$).

Desorption temp. : 500 $^\circ\text{C}$, GC oven temp. : 50 $^\circ\text{C}$ -(4 $^\circ\text{C}/\text{min}$)-340 $^\circ\text{C}$
 Separation column : Poly(dimethylsiloxane), Length 50 m, 0.25 mm i.d., Film thickness 0.15 μm
 Carrier gas flow : 50 mL/min, Column flow : 1.0 mL/min, Detector : FID

*Contents excerpted from T. Nonobe, S. Tsuge, H. Ohtani, T. Kitayama, K. Hatada, *Macromolecules* **1997**, 30, 4891-4896.

Keyword : Stereoregularity, Poly(methyl methacrylate)s, Deactivated metal capillary column

Applications : Vinyl polymer analysis

Related technical notes :

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