Pyrolysis-gas chromatography for end group analysis of polystyrene macromonomers using stepwise pyrolysis combined with on-line methylation

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Abstract:

The end group functionalities of polystyrene macromonomers with methacryloyl (ML) end groups were determined by stepwise pyrolysis-gas chromatography (Py･GC) at different temperatures, using a two stage pyrolyser consisting of two independent furnaces, combined with an on-line methylation technique. The macromonomer sample in THF solution was mixed with tetramethylammonium hydroxide in methanol solution, as the methylation reagent, in a sample cup; this was introduced into the first furnace (250ºC) of the pyrolyser attached to a gas chromatograph to decompose selectively the ML end group moieties in the macromonomer to methyl methacrylate (MMA). After GC analysis of the formed MMA, the sample cup was dropped down into the second furnace (650ºC) to pyrolyse the residual polystyrene main chain thoroughly. The concentration of the ML end groups was determined by comparing the peak intensity of MMA observed in the former pyrogram with those of the characteristic products formed from the polystyrene main chain in the latter pyrogram. The end group functionalities of several macromonomer samples calculated from the concentrations of the end groups, determined by Py･GC, were in fairly good agreement with those determined by 1H NMR, and with those calculated from the data of the maximum conversion of the macromonomers.

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