Characterization of Polysiloxanes by (Py-GC/MS)

[Background] Polysiloxanes have been widely used in industry. The physical and chemical properties depend on the structure and composition of pendent and end groups. It is therefore important to establish the structure-property relationship of polysiloxanes. High resolution Py-GC/MS, one of the most powerful technique for structure and thermal degradation studies, was used for this study.

[Experimental] Several polysiloxane samples were used (some are listed in Table 4.). A vertical micro-furnace-type pyrolyzer was directly attached to a GC with an FID detector. 0.2mg each of polymer samples was pyrolyzed under nitrogen at 600˚C. A fused-silica capillary column coated with immobilized poly(5%-phenyl)-methylsiloxane was used. The oven temperature was programmed from 0°C to 320°C at 5°C/min The peak identification was made using a directly coupled GC/MS with EI and CI sources.

[Results]. Figure 1 shows the pyrograms of a polysiloxane sample utilized. Table 1 summarizes the compositions of various siloxane copolymers determined from the pyrograms along with the reference nominal ones. The estimated compositions are in fairly good agreement with the nominal values. These results suggest that the composition of a siloxane copolymer can be accurately determined by high-resolution Py-GC under the given experimental conditions.
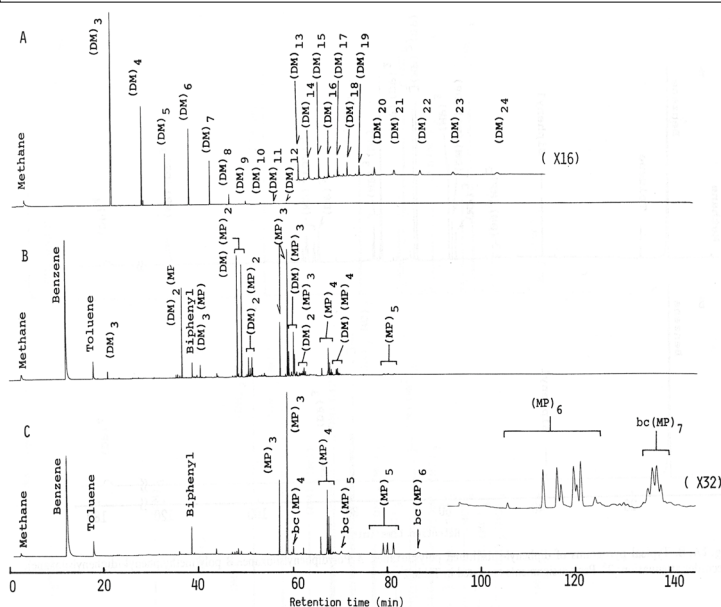

Table 1 Nominal and estimated composition of siloxane copolymer

Sample	Nominal Composition mol%	Estimated Composition mol%
OV-3	DM:MP = 80:20	DM:MP = 82.5:17.5
OV-7	DM:MP = 60:40	DM:MP = 61.8:38.2
OV-11	DM:MP = 30:70	DM:MP = 31.1:68.9
OV-25	MP:DP = 50:50	MP:DP = 46.5:52.5
SE-52	DM:DP = 95: 5	DM:DP = 94.9: 5.1
XE-60	DM:MC = 50:50	DM:MC = 45.3:54.7

* DM Dimethylsiloxane, MP Methylphenylsiloxane, DP Diphenylsiloxane, MC 2-Cyanoethylmethylsiloxane

Figure 1. Typical pyrograms of dimethyl- and/or methylphenyl-substituted polysiloxanes.

A Polydimethylsiloxane; **B** Poly(dimethyl-methylphenylsiloxane);
C Poly(methylphenylsiloxane).

Keyword : Polysiloxane, Py-GC/MS, Characterization

Applications : General polymer analysis

Related technical notes :

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