Synthesis and Investigation of the Properties of Poly(phenyl-α-naphthylsilylene)–Dimethylsilylene Copolymers

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ABSTRACT: A Wurtz-type reductive coupling reaction of dichlorophenyl-α-naphthylsilane was carried out in a mixture of toluene and *o*-xylene in the presence of sodium and a catalytic amount of mercury; α,ω-dichlorophenyl-α-naphthylsilylenes of various degrees of polymerization were obtained. Through the hydrolysis of α,ω-dichlorophenyl-α-naphthylsilylenes, corresponding dihydroxy compounds were obtained. The heterofunctional polycondensation of α,ω-dihydroxyphenyl-α-naphthylsilylenes with α,ω-dichlorodimethylsilylenes was performed both without amines and in the presence of amines. Heterofunctional polycondensation without amines did not proceed with the formation of high molecular weight compounds because the cleavage of both ≡Si—Si≡ and ≡Si—O—Si≡ bonds took place during condensation. In the presence of amines, polysilylene–silylene copolymers were obtained. The synthesized copolymers were investigated with gel permeation chromatography, differential scanning calorimetry, roentgenography, and thermogravimetry. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 85: 1047–1056, 2002

Key words: polysilanes; differential scanning calorimetry; gel permeation chromatography

INTRODUCTION

There has been considerable interest in the chemistry of organosilicon polymers, including polysilylenes and polycarbosilylenes, because they can be actively used as functional materials such as photoresists, semiconductors, hole-transporting materials, and precursors of silicon carbide and in optoelectronics.[1,2]

The usual synthesis of polysilylenes is performed through a Wurtz-type reductive coupling condensation of diorganodichlorosilanes with finely divided sodium metal in an inert diluent; in this case, both homopolymers and copolymers can be obtained.[3] In the literature,[4] there have been reported polysilylene–polysiloxane copolymers with rigid poly(phenyl-α-naphthylsilane) fragments in the main polydimethylsiloxane chain, and it has been shown that the microdomain structures of the block copolymers depend on the lengths of the rigid and flexible fragments.

In the literature,[5] there is information about the syntheses and properties of poly(oxyhexakis-dimethylsilylene) and its copolymers with dimethylsiloxanes, which are synthesized by the hydrolytic polycondensation of α,ω-dichlorohexakis-dimethylsilylene and by the cationic ring-opening polymerization of dodecamethyloxahexasilacyclo-heptane initiated with proton acid. The possibil-

ity of synthesizing alternative copolymers composed of oxyhexakisdimethylsilylene units and dimethylsiloxane or oligodimethylsiloxane units has also been explored.

In this area, the syntheses of polysilylene–silylene copolymers containing rigid poly(phenyl-α-naphthylsilylene) fragments in polydimethylsilylene backbones are interesting.

EXPERIMENTAL

The starting materials, α,ω-dichlorodimethylsilylenes with various link lengths, were synthesized by well-known methods.[6–8]

The organic solvents were purified by drying and distillation. The purity of the starting compounds was controlled with gas–liquid chromatography [ЛХМ-8МД, phase SKTF-100 (10%), NAW chromosorb, helium as the carrier gas, 2 M column].

IR spectra of the synthesized compounds were taken on a UR-20 instrument, and ^{1}H-NMR spectra were taken on a Bruker AC-250 instrument at an operating frequency of 250 MHz in a solution of $C_2D_2Cl_4$ or $CDCl_3$ as a solvent and internal standard.

Diffractograms were taken on a ДРОН-2М instrument, and A Cuα values were measured without a filter, the angular velocity of the motor being approximately 2°C/min.

Differential scanning calorimetry (DSC) was performed on a PerkinElmer instrument at heating and cooling rates of approximately 10°C/min.

Syntheses of α,ω-Dichlorophenyl-α-naphthylsilylenes

A Wurtz-type polycondensation reaction was carried out in a three-necked flask equipped with a reflux condenser, an inert-gas carrier tube, and a mechanical stirrer. A solution of 30.5 g (0.1 mol) of dichlorophenyl-α-naphthylsilane in 35 mL of dry toluene and 4.6 g of sodium was reflux until the sodium dissolved. After that, the reaction mixture was filtered, and the solution was concentrated and reprecipitated with anhydrous hexane.

The typical reactions of the syntheses of α,ω-dichlorophenyl-α-naphthylsilylenes were performed according to the aforementioned method.

Syntheses of α,ω-Dihydroxyphenyl-α-naphthylsilylenes

A hydrolysis reaction was carried out in a three-necked flask equipped with a reflux condenser, a dropping funnel, and a mechanical stirrer. To a solution of 0.44 g (0.0052 mol) of sodium hydrocarbonate, 40 mL of water, and 30 mL of benzene within a temperature range of 0 to −5°C, a solution of 10 g (0.0026 mol) of α,ω-dichlorophenyl-α-naphthylsilylene ($m \approx 15$) in 100 mL of benzene was added, after which the reaction mixture was stirred at room temperature for 2 h, washed until the reaction was neutral, and dried over anhydrous Na_2SO_4. The benzene was removed *in vacuo*, and from a concentrated solution, a dihydroxy-containing oligomer was reprecipitated with anhydrous hexane; 8.0 g (87%) of α,ω-dihydroxyphenyl-α-naphthylsilylene ($m \approx 15$) was obtained. Typical reactions of the syntheses of α,ω-dihydroxyphenyl-α-naphthylsilylenes were carried according to the aforementioned method.

Heterofunctional Condensation (HFC) of α,ω-Dihydroxyphenyl-α-naphthylsilylenes ($m \approx 8$) with α,ω-Dichloropermethylsilylene ($n \approx 6$)

A polycondensation reaction was carried out in a three-necked flask equipped with a reflux condenser, a dropping funnel, and a mechanical stirrer. To a solution of 1.8214 g (0.9637 mmol) of α,ω-dihydroxyphenyl-α-naphthylsilylenes in 2.5 mL of anhydrous toluene at room temperature, a solution of 0.4037 g (0.9637 mmol) of α,ω-dichloropermethylsilylene and 0.1525 g (1.9274 mmol) of pyridine in 0.5 mL of anhydrous toluene was added. The mixture was stirred and heated to the boiling temperature of toluene for 7–8 h. Afterward, 8 mL of toluene was added and filtered, the reaction mixture was washed from the traces of the pyridine–hydrochloric acid complex and dried over anhydrous Na_2SO_4, some of the toluene was removed and reprecipitated with hexane, and 1.4 g (65%) of copolymer V was obtained. Typical reactions of the syntheses of the other copolymers were carried out according to the aforementioned method.

RESULTS AND DISCUSSION

The initial rigid α,ω-dichlorophenyl-α-naphthylsilylenes and α,ω-dichlorodimethylsilylenes with monomodal molecular mass distributions and various degrees of polymerization were obtained[4] by a Wurtz-type condensation of dichlorodiorganosilanes in the presence of various ratios (1/1.8 or 1/2) of metallic sodium in a mix-
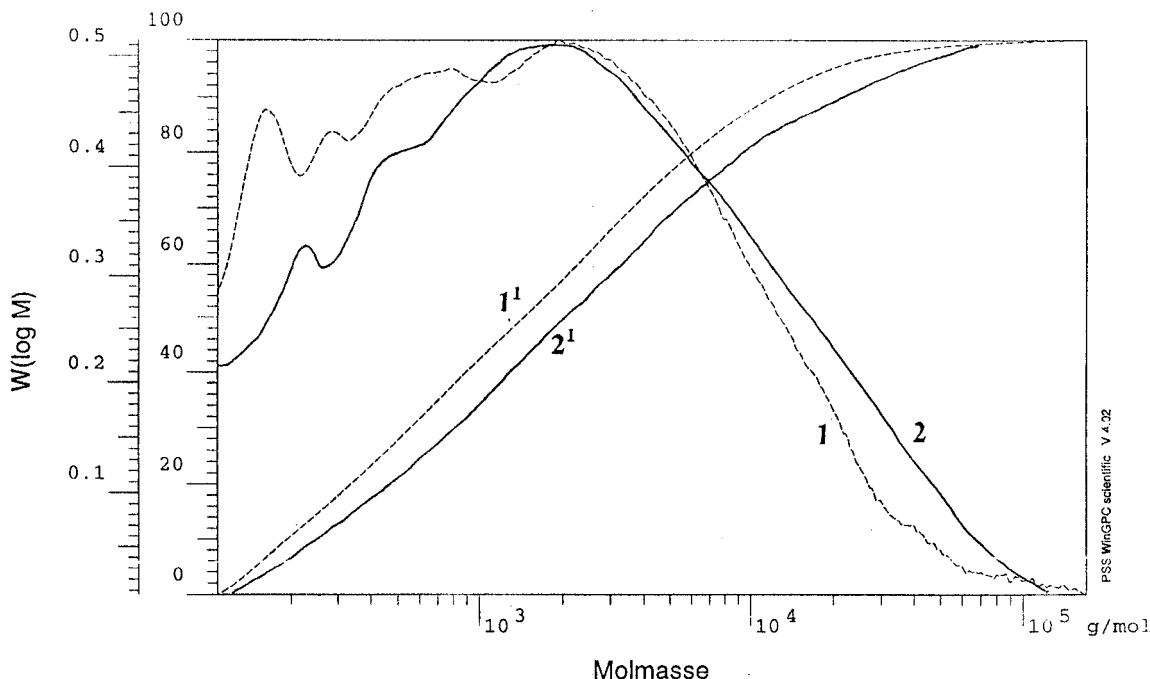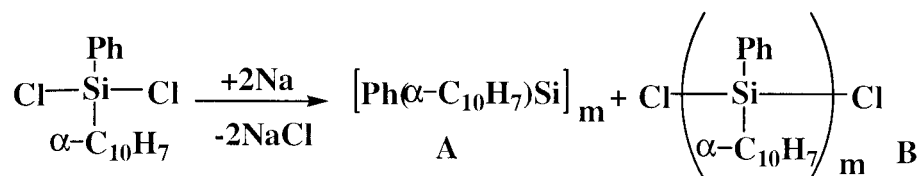
Figure 1 GPC curves of α,ω-dichlorophenyl-α-naphthylsilylenes. Curves 1 and 2 correspond to oligomers I and II, respectively.

ture of toluene and *o*-xylene (1/1 ratio) in the presence of a catalytic amount of mercury. The molar ratio of dichlorodiorganosilanes to metallic mercury was $1/10^{-2}$. The raw products pre-cipitated from a toluene solution with anhydrous hexane.

Generally, the condensation reactions proceeds as follows:

$$Cl-\underset{\underset{\alpha\text{-}C_{10}H_7}{|}}{\overset{\overset{Ph}{|}}{Si}}-Cl \xrightarrow[\text{-2NaCl}]{\text{+2Na}} \underset{A}{\left[Ph(\alpha\text{-}C_{10}H_7)Si\right]_m} + \underset{B}{Cl\left(\underset{\underset{\alpha\text{-}C_{10}H_7}{|}}{\overset{\overset{Ph}{|}}{Si}}\right)_m Cl}$$

where *m* is approximately 8 (I) or 15 (II) for structure B.

Oligomers I and II were partially reprecipitated from a toluene solution with anhydrous hexane; the yield was about 62%. During this reaction, in addition to intermolecular condensation, intramolecular cyclization with the formation of cyclic phenyl-α-naphthylsilylenes took place. Dividing cyclosilylenes ($m \approx 4-6$) by distillation *in vacuo* ($\approx 1-2 \times 10^{-2}$ mmHg) was unsuccessful because during distillation the polymerization reaction of phenyl-α-naphthylcyclosilanes took place. Gel permeation chromatography (GPC) showed a monomodal character for the molecular mass distribution (Fig. 1). In Table I, some phys-icochemical properties of the synthesized oligomers are presented.

The synthesized oligomers were coffee-colored hard-type products that were quite soluble in aromatic solvents. The structures and compositions of the synthesized oligomers were determined with functional and elemental analyses via molecular masses and IR spectra. In the IR and UV spectra of the compounds, there were absorption bands for valence oscillation characteristic of $\equiv$Si—Si$\equiv$ (290 nm), $\equiv$Si–C$_{aryl}$ (1000 and 1430 cm^{-1}), $\equiv$Si–Cl (490 cm^{-1}), C—H (3000–3080 cm^{-1}), and CH$=$CH (3050 cm^{-1}) and bands for monosubstituted benzole rings (700 and 1600 cm^{-1}).

Table I   Physicochemical Properties of α,ω-Dichlorophenyl-α-naphthylsilylenes

No.	Ratio RR′SiCl$_2$/Na	Yield (%)	T_{melt}°C	M^a	Cl(%)
I	1/1.8	62	194–199	2005	3.54
				1927	3.68
II	1/1.8	61	206–212	3504	2.02
				3551	1.99

Where R = Ph, R′ = α-C$_{10}$H$_7$.
[a] In the denominator, there are calculated values, in the numerator, there are experimental values. Average molecular weights were determined by the ebuliometric method by GPC (II) and by Cl% end-group analysis (I).

A hydrolysis reaction of α,ω-dichlorophenyl-α-naphthylsilylenes in an alkaline solution in the presence of sodium hydrocarbonate within a temperature range of −5 to −10°C was carried out, and corresponding α,ω-dihydroxyphenyl-α-naphthylsilylenes were obtained. The reaction proceeds as follows:
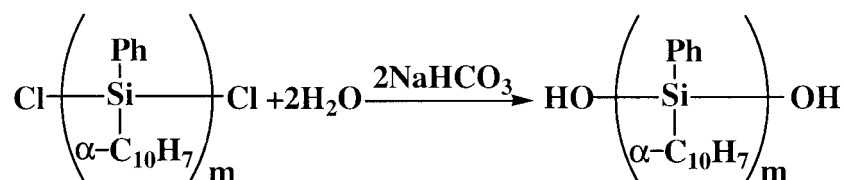

where m is approximately 8 (III) or 15 (IV).

The oligomers were reprecipitated from a toluene solution with anhydrous hexane. They were white solids soluble in aromatic solvents. In the IR spectra of dihydroxy compounds, absorption bands characteristic of ≡Si–Cl bonds disappeared, and absorption bands characteristic of associated ≡Si–OH bonds appeared at 3200–3600 cm^{-1}. Some physicochemical properties and the yields of α,ω-dihydroxyphenyl-α-naphthylsilylenes are presented in Table II.

Polysilylene–silylene copolymers interrupted with oxygen and regularly structured were obtained by HFC of α,ω-dihydroxyphenyl-α-naphthylsilylenes with α,ω-dichlorodimethylsilylenes in diluted solutions of anhydrous toluene in a 1/1 ratio of initial compounds within a temperature range of 20–25°C.

HFC was studied both without an acceptor and in the presence of pyridine. During HFC without amines, the conversion of hydrogen chloride changed from 32 to 38%; as a result of isolated hydrogen chloride, concurrent reactions take place:

$$\equiv Si–O–Si\equiv + H–Cl \rightarrow \equiv Si–O–H + Cl–Si\equiv \quad (I)$$
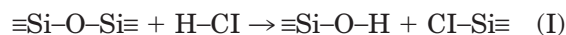
The first reaction (I) is the cleavage of the ≡Si—O—Si≡ bond, which is known from the literature[9] to proceed with hydrogen chloride:

Table II   Some Physicochemical Properties of α,ω-Dihydroxyphenyl-α-naphthylsilylenes

|	Yield (%)	T_{melt} (°C)	η_{sp} (1% solution, in toluene, at 25°C)	d_1 (Å)	M^a		OH%	
					Calcu	Found	Calcu	Found
III	87	115–120	0.04	11.05	1890	1900	1.80	1.79
IV	89	127–131	0.04	11.05	3514	3550	0.97	0.95

[a] Average molecular weights were determined by the ebuliometric methods and by OH end-group analysis.
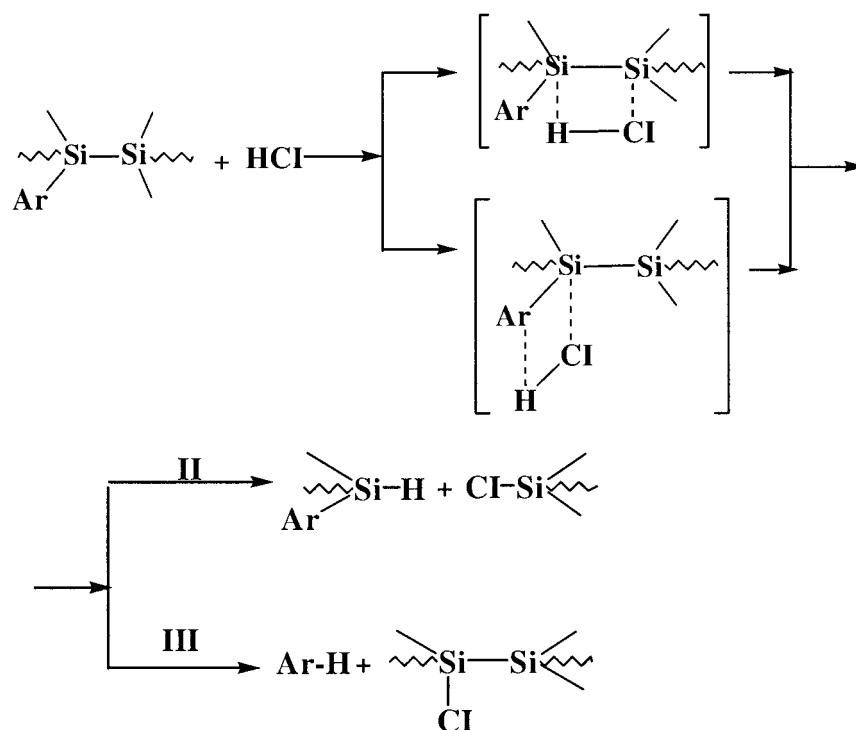
The other reactions are the cleavage of the $\equiv$Si—Si$\equiv$ bond (reaction II) and the selective cleavage of aryl groups (reaction III) with the formation of functional chloro groups, which can then be used in the further synthesis of macrochains. As seen in the schemes, we think that both reactions proceed with the formation of a four-member transition complex.

In our case, however, during HFC without amines, a mixture of low molecular weight compounds ($\overline{M}_\omega \approx 2.3 \times 10^3$ to 3.7×10^3) soluble in organic solvents was obtained. This proved that during HFC without amines, the cleavage of the main $\equiv$Si—Si$\equiv$ bonds took place as well as the cleavage of the $\equiv$Si—O—Si$\equiv$ bonds.[9,10] For the synthesis of silylene–silylene copolymers with high molecular weights, HFC was performed in the presence of pyridine. In the presence of amines, HFC of α,ω-dihydroxyphenyl-α-naphthylsilylene with α,ω-dichlorodimethylsilylenes, as known from the literature[11] may proceed with two concurrent reactions:
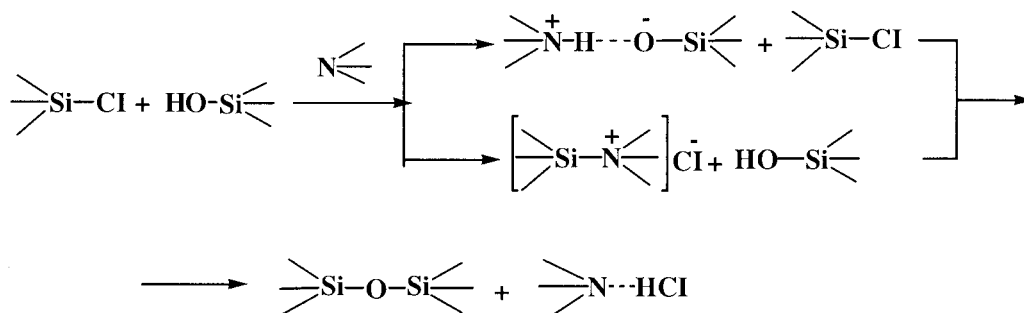
During HFC, Si—O—Si bonds are obtained in both cases.

Therefore, HFC in the presence of pyridine proceeds according to the following scheme:

Table III   Some Physicochemical Properties and Yields of Polyphenyl-α-Naphthylsilylene–Polydimethylsilylene Copolymers

No.	Yield (%)	$[\eta]$ (dL/g)	d_1, (Å)	T_{soft} (°C)	$\overline{M}_\omega \times 10^4$	10% Mass Loss	Residual Mass (%)	Elemental Composition[a] (%)		
								C	H	Si
V	65	0.10	11.00	+138	5.95	370	34	74.93 / 75.68	5.61 / 5.95	17.34 / 17.65
VI	72	0.21	10.72	+128	—	360	28	67.71 / 68.01	6.52 / 6.93	24.01 / 24.49
VII	64	0.26	10.72	+105	—	320	26	60.33 / 60.89	7.34 / 7.84	30.98 / 30.86
VII[1]	12	0.34	—	—	—	—	—	60.37 / 60.89	7.30 / 7.84	31.21 / 30.86
VII[2]	23	0.25	—	—	—	—	—	60.23 / 60.89	7.30 / 7.84	30.88 / 30.86
VII[3]	31	0.14	—	—	—	—	—	60.42 / 60.89	7.65 / 7.84	30.60 / 30.86
VII[4]	33	0.10	—	—	—	—	—	60.45 / 60.89	7.43 / 7.84	30.11 / 30.86
VIII	63	0.12	11.40	+140	—	400	41	78.40 / 78.67	5.32 / 5.62	15.08 / 15.29
IX	70	0.21	10.72	+125	—	370	38	72.95 / 73.36	5.85 / 6.29	19.23 / 19.99
X	72	0.32	9.82	+100	6.86	350	27	66.78 / 67.32	6.73 / 7.06	25.01 / 25.33

[a] In the denominator, there are calculated values; in the numerator there are experimental values. Molecular weights were determined by the GPC method
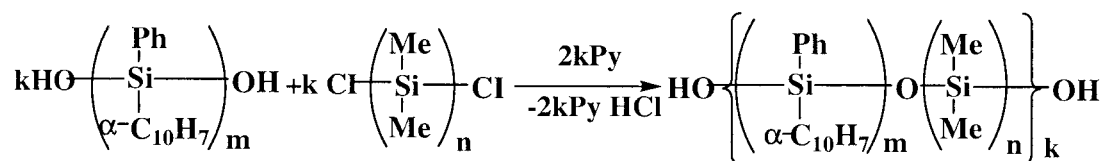
where m is approximately 8 and n is approximately 6 (V), 17 (VI), or 35 (VII) or m is approximately 15 and n is approximately 6 (VIII), 17 (IX), or 35 (X). The synthesized copolymers were reprecipitated from a toluene solution with anhydrous hexane. They were cream-colored solids that were soluble in organic solvents ($[\eta] \approx 0.11–0.32$).

Copolymer VII was fractionated by partial reprecipitation from a toluene solution with anhydrous hexane; four fractions were obtained. An elemental analysis of these fractions showed that they were somewhat different from one another. This showed that during HFC in the presence of pyridine, secondary reactions, homofunctional condensation or the breaking of $\equiv$Si—Si$\equiv$ bonds, did not take place noticeably (or appreciably), and copolymers, mainly with a regular arrangement of rigid poly(phenyl-α-naphthylsilylene) fragments in the main dimethylsilylene chain, were obtained.

Some physicochemical properties of synthesized copolymers are presented in Table III. In
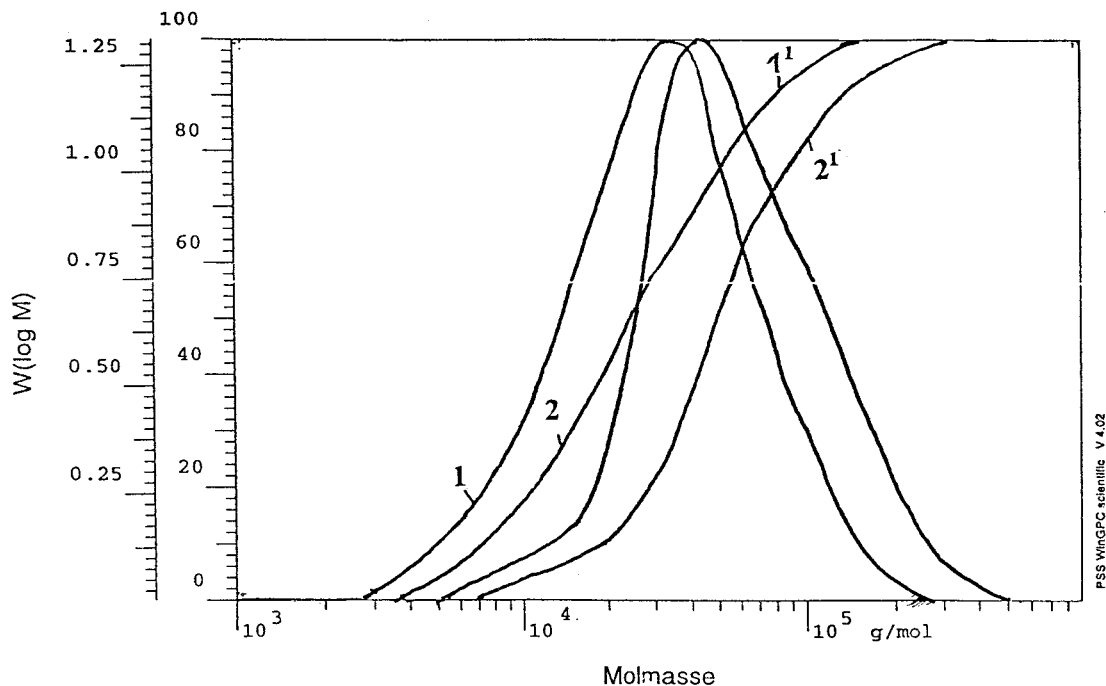
Figure 2 GPC curves of poly(phenyl-α-naphthylsilylene)–polydimethylsilylene copolymers. Curves 1 and 1′ and curves 2 and 2′ correspond to copolymers I and VI, respectively.

the ^{1}H-NMR spectra of copolymer VI, one can observe a singlet signal for methyl protons with the chemical shift $\delta \approx 0.1$ ppm and a complicated multiplet for phenyl protons with the chemical shift $\delta \approx 6.3$–8.0 ppm. The found ratio of phenyl and methyl protons was in conformity with its chemical structure.

In the IR and UV spectra of the synthesized copolymers, one can observe absorption bands at 290 nm and 1020 cm^{-1} that are characteristic of $\equiv$Si—Si$\equiv$ and $\equiv$Si—O—Si$\equiv$ bonds, respectively, and absorption bands at 1450 cm^{-1} and 700 and 1590 cm^{-1} that are characteristic of $\equiv$Si–C$_{aryl}$ bonds and monosubstituted benzene rings, respectively. One can also see a broadened signal within the range 3200–3600 cm^{-1} that is characteristic of associated $\equiv$Si–OH groups. By GPC, molecular masses and molecular mass distributions of some copolymers were determined. As seen in Figure 2 (curves 1 and 2), copolymers V and X had monomodal molecular mass distributions, and their average molecular masses changed as follows: number-average molecular weight $\approx 2.13 \times 10^4$ to 2.77×10^4, $\overline{M}_\omega \approx 5.95 \times 10^4$ to 6.86×10^4, and z-average molecular weight $\approx 9.86 \times 10^4$ to 1.26×10^5 (polydispersity ≈ 2.47–2.79).
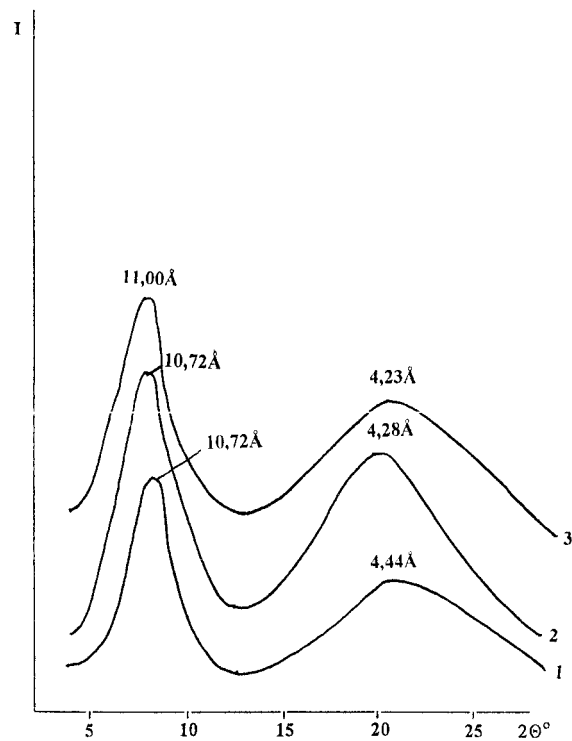

Figure 3 Wide-angle X-ray diffraction patterns of poly(phenyl-α-naphthylsilylene)–polydimethylsilylene copolymers. Curves 1, 2, and 3 correspond to copolymers VII, VI, and V, respectively.
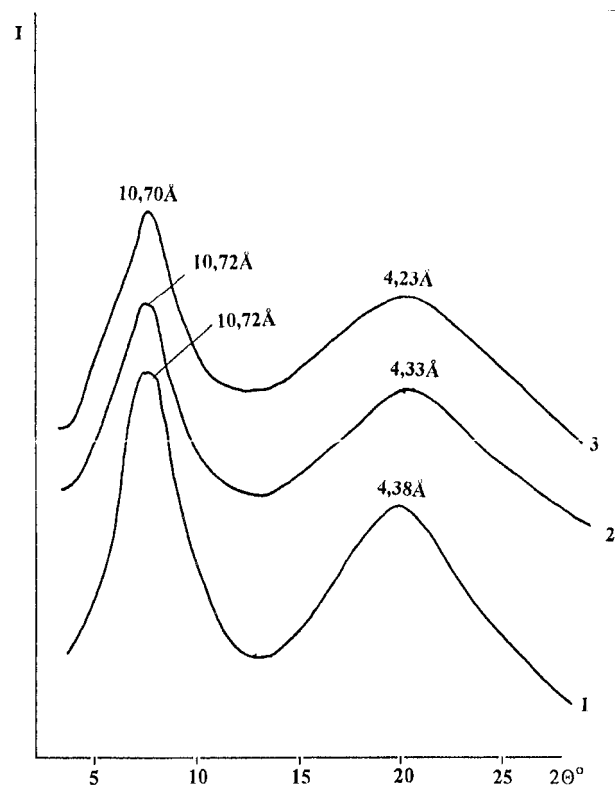
Figure 4 Wide-angle X-ray diffraction patterns of poly(phenyl-α-naphthylsilylene)–polydimethylsilylene copolymers. Curves 1, 2, and 3 correspond to copolymers X, IX, and VIII, respectively.

A wide-angle X-ray analysis of the copolymers was carried out. As seen in Figure 3, the copolymers were amorphous systems. Maximum values of the interchain distances were achieved at small values of the dimethylsilylene chain ($n \approx 6$). On diffractogram curves, one can see two diffraction maxima, $2\theta \approx 7.75$–$8.00°$ and $2\theta \approx 20.5$–$21.0°$. The first value corresponds to the interchain distance $d_1 \approx 11.0$–11.4 Å in copolymers V and VIII accordingly. This value of the interchain distance is near the interchain distances characteristic of poly(phenyl-α-naphthylsilylene)–polydimethylsiloxane block copolymers.[4]

The second maximum, $2\theta \approx 20.5$–$21.0°$, corresponds to $d_2 \approx 4.23$–4.33 Å, which characterizes both intrachain and interchain atomic interactions.[12] With an increase in the length of rigid poly(phenyl-α-naphthylsilylene), the value of the interchain distances slightly increased.

As seen in Figure 4, at high values of the dimethylsilylene chain ($n \approx 35$), the copolymers are also shown as one-phase systems with one value of d_1. With an increase in the length of the dimethylsilylene chain, the interchain distances slightly decreased up to $d_1 \approx 9.80$–10.72 Å. The interchain distances in linear polydimethylsilylene were equal to $d_1 \approx 7.70$ Å.

Unlike for poly(phenyl-α-naphthylsilylene)–polydimethylsiloxane block copolymers, in poly(phenyl-α-naphthylsilylene)–polydimethylsilylene copolymers there were no segregation processes leading to the formation of a microdomain struc-
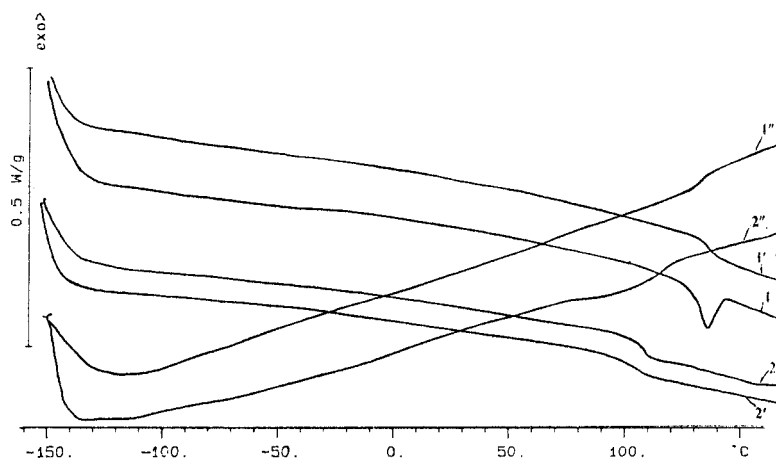

Figure 5 DSC thermograms of poly(phenyl-α-naphthylsilylene)–polydimethylsilylene copolymers V and VII. Curves 1, 1′, and 1″ correspond to the first heating scans, second heating scans, and cooling scans for copolymer VII, respectively. Curves 2, 2′, and 2″ correspond to the first heating scans, second heating scans, and cooling scans for copolymer V, respectively. The scanning rate was approximately 10°C/min.

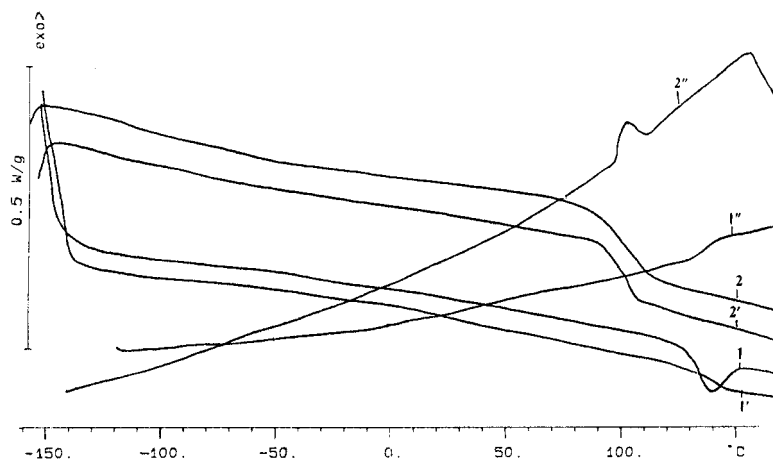**Figure 6** DSC thermograms of poly(phenyl-α-naphthylsilylene)–polydimethylsilylene copolymers VIII and X. Curves 1, 1′, and 1″ correspond to the first heating scans, second heating scans, and cooling scans for copolymer VIII, respectively. Curves 2, 2′, and 2″ correspond to the first heating scans, second heating scans, and cooling scans for copolymer X, respectively. The scanning rate was approximately 10°C/min.

ture. The copolymers are shown as a common and continuous rigid system.

In Figures 5 and 6, DSC curves of synthesized copolymers are shown. As shown in the indicated figures and the table, the copolymers were characterized by a higher softening temperature (T_{soft}).

The term *softening temperature* is represented by us more correctly in this case. As for roentgenographic investigations, by DSC analyses we proved that even at higher values of the length of the more flexible dimethylsilylene chain (n), the formation of two-phase systems did not take place. On DSC curves separately, the effects stipulated by the devitrification, crystallization, and melting of polydimethylsilylene fragments and the softening of poly(phenyl-α-naphthylsilylene) are not shown, which may be explained by the increased rigidity of polydimethylsilylene fragments. At one and the same values of dimethylsilylene fragments, the increasing values of rigid poly(phenyl-α-naphthylsilylene) fragments did not influence T_{soft} values of the copolymers, but at one and the same values of rigid blocks, with an increase in the length of the dimethylsilylene chain, T_{soft} values of the copolymers decreased (see Table III). A comparative estimation of T_{soft} values of the copolymers with the melting temperature (T_{melt}) and T_{soft} values of poly(phenyl-α-naphthylsilylene)–polydimethylsiloxane block copolymers[13] showed that at one and the same values of the length of poly(phenyl-α-naphthylsilylene),

dimethylsilylene, and dimethylsiloxane fragments, the synthesized copolymers were characterized by higher values of T_{soft}.

Thermogravimetric investigations of silylene–silylene copolymers were carried out. As seen in Figures 7 and 8, the copolymers were characterized by higher thermooxidative stability. One can observe 10% mass losses at 300–410°C. The high thermooxidative stability of the copolymers may
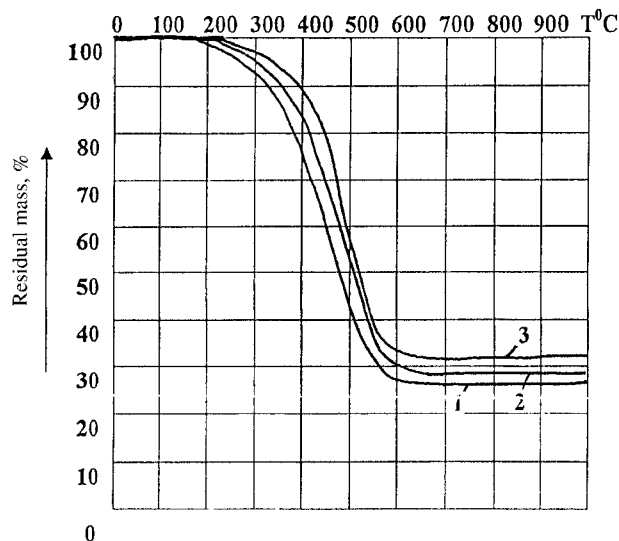

Figure 7 Thermogravimetric curves of poly(phenyl-α-naphthylsilylene)–polydimethylsilylene copolymers. Curves 1, 2, and 3 correspond to copolymers VII, VI, and V, respectively.
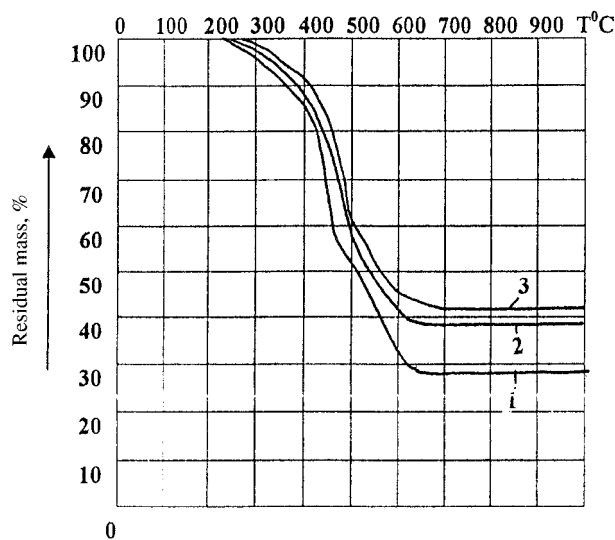
Figure 8 Thermogravimetric curves of poly(phenyl-α-naphthylsilylene)–polydimethylsilylene copolymers. Curves 1, 2, and 3 correspond to copolymers X, IX, and VIII, respectively.

be explained by the high resistance of phenyl groups to oxidation and their inhibiting effect on the oxidation of methyl groups;[14] moreover, it is in agreement with the conclusion that with the insertion of more rigid fragments in polydimethylsiloxane chains, cyclic depolymerization,[15] which proceeds with the release of D_n-type dimethylcyclosiloxanes, is hindered. In our case, rigid poly-(phenyl-α-naphthylsilylene) fragments hindered cyclic depolymerization with the release of $[Me_2Si]_n$-type dimethylcyclosilylenes. At one and the same values of dimethylsilylene fragments and with an increase in the length of poly(phenyl-α-naphthylsilylene) fragments, the thermooxidative stability of the copolymers rose. The main destruction process proceeded at 400–650°C. Above 680°C, the curves of the mass losses of the copolymers did not change, and residual masses were 41–26%. The poly(phenyl-α-naphthylsilylene)–poly-

dimethylsilylene copolymers, compared with poly-(phenyl-α-naphthylsilylene)–polydimethylsiloxane block copolymers,[4,13] were characterized by approximately the same thermooxidative stability.

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