Poly(1,3-disila-1,3-diphenyl-2-oxaindane)–Poly(dimethylsiloxane) Block Copolymers

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ABSTRACT: The hydrolytic condensation of 1,3-dichloro-1,3-disila-1,3-diphenyl-2-oxaindane under neutral conditions produced $\alpha'\omega$-dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane (polymerization degree ≈ 4). The homofunctional condensation of $\alpha'\omega$-dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane in a toluene solution and in the presence of activated carbon was performed, and dihydroxy-containing oligomers with various degrees of condensation were obtained. Through the heterofunctional condensation of dihydroxy-containing oligomers with $\alpha'\omega$-dichlorodimethylsiloxanes in the presence of amines, corresponding block copolymers were obtained. Gel permeation chromatography, differential scanning calorimetry, thermomechanical analysis, thermogravimetry, and wide-angle roentgenography investigations were carried out. Differential scanning calorimetry and roentgenography studies of the block copolymers showed that their properties were determined by the ratio of the lengths of the flexible and linear poly(dimethylsiloxane) and rigid poly(1,3-disila-1,3-diphenyl-2-oxaindane) fragments in the macromolecular chain. At definite values of the lengths of the flexible and rigid fragments, a microheterogeneous structure was observed in the synthesized block copolymers. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 84: 1409–1417, 2002; DOI 10.1002/app.10335

Key words: 1,3-disila-1,3-diphenyl-2-oxaindane; heterofunctional condensation

INTRODUCTION

The insertion of various heteroatoms or cyclic fragments into the linear backbone of dimethylsiloxane breaks the spiral structure of the poly(dimethylsiloxane) (PDMS) chain and changes the physicochemical indices and thermo-oxidative stability of copolymers.[1] The insertion of more rigid phenyl fragments into the main PDMS backbone hinders the cyclic depolymerization of polymeric chains[2] and imparts to copolymers thermo-oxidative stability, which may be explained by the
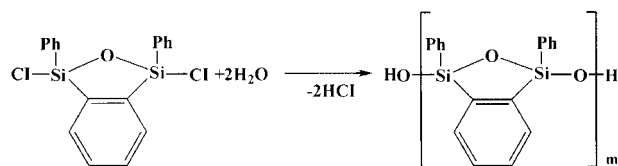
presence of phenyl groups highly resistant to oxidation and by their inhibiting effect on the oxidation of methyl groups.[3]

Organosilicon block copolymers (BCs) have recently received widespread attention,[4] and there is some information about those BCs with cyclolinear molecular structures. Known A_mB_n-type cyclolinear siliconorganic BCs include poly[organocyclotetra(hexa)siloxane dimethylsiloxane],[5] poly(organocyclocarbosiloxane dimethylsiloxane),[6,7] and ladder poly(phenylsiloxane)s with single-stranded[8,9] and double-stranded fragments in the main and side dimethylsiloxane chains;[10] one known $(AB)_mC_n$-type BC is silarylenecyclotetra(hexa)siloxane–dimethylsiloxane.[11,12]
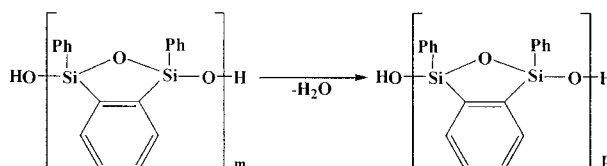
Phase incompatibility in BCs is connected to the lengths of rigid and flexible fragments. The

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Scheme 1

Scheme 2

value of the length for dimethylsiloxane blocks, from which flexible block properties are derived, must be higher than 20, whereas polycyclic (ladder) blocks, even with a polymerization degree (m) greater than or equal to 3, exhibit properties characteristic for rigid block systems.

EXPERIMENTAL

The organic solvents were purified by drying and distillation. The initial 1,3-dichloro-1,3-disila-1,3-diphenyl-2-oxaindane was received by a well-known method,[13] and the purity of the initial compounds was verified on a gas–liquid chromatograph (ЈЛХМ8Мд, Union of Soviet Socialist Republics). Phase SKTF-100 (10%) was used on an NAW chromosorb with helium as the carrier gas.

Gel permeation chromatography (GPC) was performed with a Waters model 6000A chromatograph equipped with an R 401 differential refractometer detector. The column set consisted of 10^3- and 10^4-Å Ultrastyragel columns. Sample concentrations were approximately 3 wt % in toluene, and the typical injection volume for the siloxane was 5 μL. Standardization of the GPC was accomplished with styrene or PDMS standards of known molecular weights.

^{1}H-NMR spectra was taken on a PerkinElmer R-32 instrument at an operating frequency of 90 MHz with the internal standard HMDS or tetramethylsilane. The IR spectra of all the samples were taken on a UR-20 spectrophotometer.

The thermomechanical curves were taken with a custom-made installation. The test conditions were $v \approx 5°$/min (where v is the temperature-rise rate) and $\sigma = 0.5$ kg/cm^2.

Thermogravimetric investigations were carried out on a Paulic–Paulic–Erday model MOM-102 derivatograph. The test condition was $v = 5°$/min. The differential scanning calorimetry (DSC) analysis was carried out on a PerkinElmer instrument with $v \approx 10°$/min (where v is the temperature-rise or -decrease rate).

Diffractograms, taken on a дРОЍ-2 instrument (A Cuα), were measured without a filter, with an angular velocity of the motor of $\omega \approx 2°$/min.

### Hydrolysis of 1,3-Dichloro-1,3-disila-1,3-diphenyl-2-oxaindane under the Soft Condition

Hydrolysis was carried out in a three-necked flask equipped with a reflux condenser, a dropping funnel, and a mechanical stirrer. To a solution of 3.72 g (0.04 mol) of aniline and 3 mL of water in 15 mL of diethyl ether at −5 to −10°C, a solution of 7.46 g (0.02 mol) of 1,3-dichloro-1,3-

Table I   Some Physicochemical Properties of Oligomeric α,ω-Dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane

No.	Solvent	C_{act} (%)	p	η_{sp}[a]	T_g (°C)	d_1 (Å)	M[b]	OH[b] (%)	Yield (%)
II	Toluene	—	17	0.04	135–141	11.50	$\frac{5600}{5424}$	$\frac{0.60}{0.62}$	97
III	m-Xylene	—	23	0.04	137–145	—	$\frac{7550}{7322}$	$\frac{0.45}{0.46}$	97
IV	Toluene	7	29	0.04	142–147	11.50	$\frac{9000}{9240}$	$\frac{0.38}{0.37}$	97

[a] In 1% solution of toluene at 25°C.
[b] In the denominator, there are calculated values; in numerator, there are experimental values.
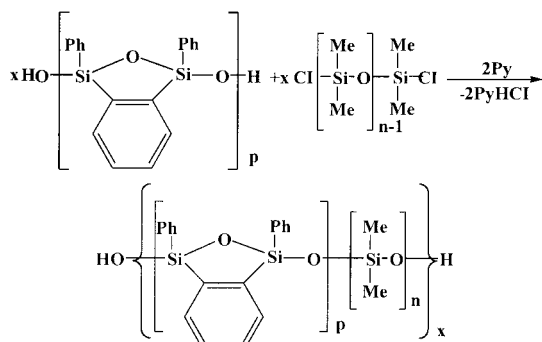
Scheme 3

diphenyl-2-oxaindane in 100 mL of diethyl ether was added over 2 h. The residue was filtered off, the solvent was evaporated *in vacuo*, and 6.1 g of a solid product was obtained. After reprecipitation from a toluene solution by hexane, 5.6 g of a white, solid product, $\alpha'\omega$-dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane I, was obtained.

IR: 540   [γ_s ($\equiv$Si—O—Si$\equiv$)],   470   [γ_s ($\equiv$Si—Ph)], 1000, 1440 ($\equiv$Si—Ph), 940, 1020 cm^{-1} [γ_{as} ($\equiv$Si—O—Si$\equiv$)]. ELEM. ANAL. Calcd. for $C_8H_{58}Si_8O_9$: C, 66.98%; H, 4.34%; Si, 17.36%; OH = 2.63; M = 1290. Found: C, 66.35%; H, 4.12%; Si, 17.23%; OH $\approx$ 2.55; M $\approx$ 1340.

Homofunctional Condensation of $\alpha'\omega$-Dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane I on Activated Carbon

Homofunctional condensation was carried out in a flask equipped with Dean–Stark packing and a reflux condenser. A solution of 10.0 g (7.7519 mmol) of compound I in 20 mL of anhydrous toluene and 0.5 g (0.04167) of activated carbon was refluxed for 24 h. The reaction product was filtered off, and the solvent was evaporated *in vacuo* at 60–70°C ($\approx$1–2 mmHg) until a constant mass was obtained and 9.4 g of compound II was obtained.

Typical homofunctional condensations in the presence of activated carbon were carried out by the aforementioned method.

Heterofunctional Condensation of $\alpha'\omega$-Dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane II with 1,3-Dichlorotetramethyldisiloxane

Polycondensation was carried out in a three-necked flask equipped with a reflux condenser, a dropping funnel, and a mechanical stirrer. To a solution of 0.0638 g (3.646 mmol) of 1,3-dichlorotetramethyldisiloxane in 0.5 mL of anhydrous toluene at room temperature, a solution of 2.0 g (3.646 mmol) of product II in 0.0577 g (7.292 mmol) of pyridine and 1.2 mL of anhydrous toluene was added. The reaction mixture was stirred and heated to the boiling temperature of toluene for 5 h. After that, 6 mL of toluene was added; the filtered reaction mixture was washed from traces of pyridine hydrochloric acid, dried over anhydrous Na_2SO_4, and reprecipitated from methanol; and copolymer V was received. The syntheses of other copolymers were carried out with the same method.

RESULTS AND DISCUSSIONS

Mass losses of the copolymers with 1,3-diorgano-1,3-disila-2-oxaindane fragments in dimethylsiloxane chains were observed from 280 to 310°C. The main destruction process proceeded within the temperature range 420–550°C.[14]

In ref. 15, hydrolyses were investigated, but the corresponding dihydroxy-containing oligomers were obtained. The high reactivity of the $\equiv$Si—CI bonds was explained by the change in the valence angle in silacyclanes.

For the synthesis of 1,3-dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane, the hydrolysis of 1,3-dichloro-1,3-disila-1,3-diphenyl-2-oxaindane under the soft condition was investigated in the presence of natrium bicarbonate and aniline within the temperature range −5 to −10°C, but in both cases, oligomeric $\alpha'\omega$-dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane was obtained. The reaction proceeded as shown in Scheme 1 [where m $\approx$ 4 (I)]. To increase the length of oligomeric $\alpha'\omega$-dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane, we carried out homofunctional condensation in the medium of a 50% solution of different solvents and in the presence of activated carbon, which led to changes in the values of m for the polycondensation products.

Despite the large variety of catalysts accelerating the condensation process of silanes, the catalysts can be divided into two groups: equilibrating and nonequilibrating.[16–18] In the first group are those catalysts in the presence of which the decomposition of $\equiv$Si—O—Si$\equiv$ bonds occurs under condensation conditions; in the second group are those catalysts that do not set off the decomposition of $\equiv$Si—O—Si$\equiv$ bonds. Activated carbon is a nonequilibrating catalyst.[5]

Table II Some Physicochemical Properties of Disilaoxaindane–Dimethylsiloxane BCs

No.	Yield (%)	$[\eta]$[a] (dL/g)	d_1 (Å)	T_g (°C)	T_{melt} (°C) T_{soft} (°C)	5% Mass Losses	Residual Mass (%)	Elemental Composition[b] (%) C	H	Si
V	93	0.12	11.05	—	88–90	420	51	66.78 66.32	4.49 4.08	18.09 17.84
VI	94	0.14	10.70	—	75–78	—	—	65.89 66.07	4.58 4.11	18.61 18.32
VII	95	0.21	10.35	—	84–87	390	46	62.75 62.40	4.91 4.54	20.41 19.89
VIII	96	0.26	—	−123	−48 to 73	—	—	56.99 56.54	5.52 5.67	23.72 23.45
IX	96	0.31	9.41 7.63	—	—	—	—	50.99 50.64	6.15 5.98	27.17 26.87
X	92	0.11	11.10	—	89–91	420	53	67.08 66.78	4.47 4.30	17.97 17.53
XI	93	0.17	—	—	84–88	—	—	66.46 66.21	4.54 4.14	18.36 18.67
XII	94	0.22	10.40	—	87–91	380	42	63.96 63.47	4.76 4.52	19.61 19.43
XIII	96	0.29	9.49 7.59	−123	−48 78	—	—	59.15 59.68	5.30 5.49	22.52 22.30
XIII[1]	19	0.37	—	—	—	—	—	59.10 59.68	5.09 5.49	22.41 22.30
XIII[2]	22	0.32	—	—	—	—	—	59.05 59.68	5.19 5.49	22.22 22.30
XIII[3]	34	0.24	—	—	—	—	—	59.37 59.68	5.24 5.49	22.52 22.30
XIII[4]	24	0.17	—	—	—	—	—	59.41 59.68	5.62 5.49	22.11 22.30
XIV	95	0.35	—	−123	−45 75	360	30	53.64 53.23	5.88 5.49	25.67 25.89
XV	92	0.12	11.11	—	87–89	—	—	67.25 67.56	4.55 4.87	17.89 17.60
XVI	93	0.21	—	—	79–82	—	—	66.71 66.54	4.51 4.33	18.21 17.93
XVII	93	0.23	—	—	77–79	380	36	64.71 65.21	4.72 4.40	19.36 18.90
XVIII	94	0.34	10.79 7.68	—	—	—	—	60.59 61.01	5.15 4.99	21.71 21.51
XIX	96	0.37	10.72 7.70	−123	−40 81	330	32	55.57 55.98	5.68 5.47	24.58 24.02

[a] In toluene at 25°C.
[b] In the numerator, there are experimental values; in the denominator, there are calculated values.

The homofunctional condensation proceeded according to Scheme 2 [where the polycondensation degree (p) was approximately 17 (II), 23 (III), or 29 (IV)]. The synthesized oligomers were white, solid products that were quite soluble in ordinary organic solvents. Some physi-
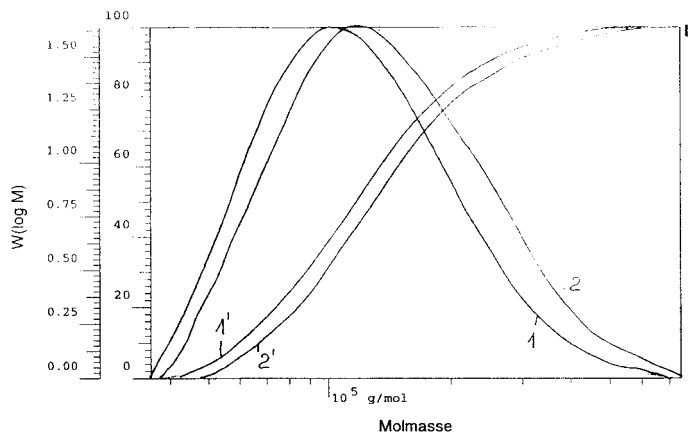
Figure 1 GPC curves of disilaoxaindane–dimethylsiloxane BCs. Curves 1 and 1′ correspond to BC XIX, and curves 2 and 2′ correspond to BC XVII.

cochemical properties of the oligomers are given in Table I.

For this homofunctional polycondensation, solvents with different boiling points were used; this led to changes in the p values of the products. As
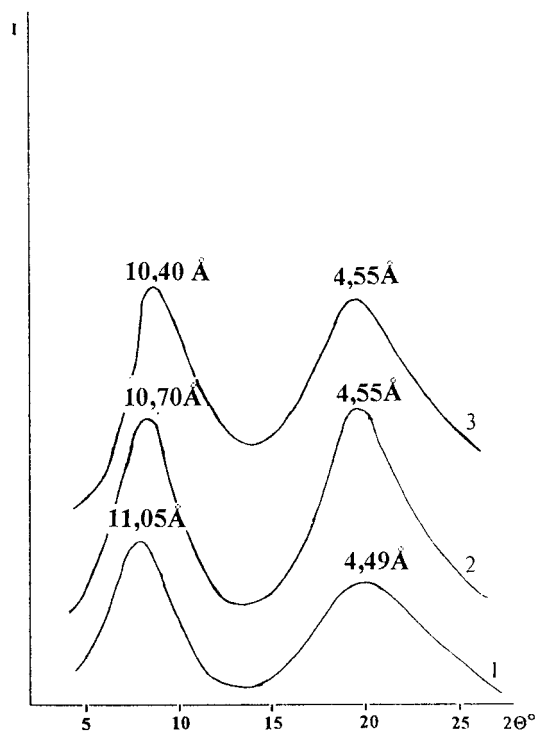

Figure 3 Wide-angle roentgenography curves of disilaoxaindane–dimethylsiloxane BCs. Curve 1 corresponds to BC XIX, and curve 2 corresponds to BC IX.

Figure 2 Wide-angle roentgenography curves of disilaoxaindane–dimethylsiloxane copolymers. Curve 1 corresponds to copolymer V, curve 2 corresponds to copolymer VI, and curve 3 corresponds to copolymer XII.
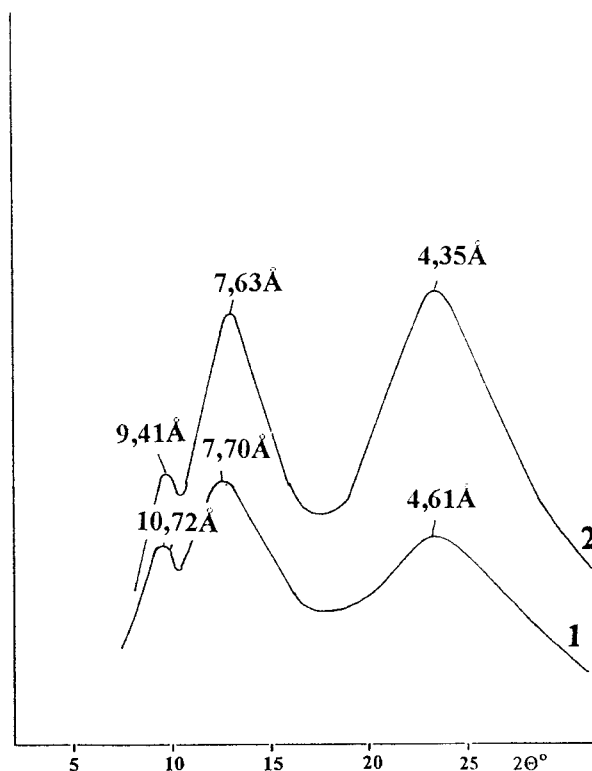
seen in Table I, the degree of homofunctional condensation increased with the boiling point of the solvent.

In IR spectra of the oligomers, there are absorption bands at 540 cm^{-1} and 940 and 1020 cm^{-1} that are characteristic of γ_s and γ_{as} $\equiv$Si—O—Si$\equiv$ bonds, respectively, in 1,3-disila-2-oxaindane fragments. There are also absorption bands at 3200–3600 cm^{-1} that prove the presence of $\equiv$Si—OH bonds. With an increase in p, the absorption band of the $\equiv$Si—OH bond became less intensive. Therefore, the homofunctional polycondensation of oligomeric $\alpha'\omega$-dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindane in the presence of activated carbon took place without the cleavage of $\equiv$Si—O—C$\equiv$ bonds of silaoxaindane rings and led to the formation of oligomeric siloxanes with high values of p. The synthesized oligomers were amorphous compounds, and the interchain distances of the oligomers were $d_1 \approx 11.50$ Å. Thermogravimetric investigations of the oligomers showed that for oligomer IV there was a 5% mass loss at 450°C. The main destruction process proceeded within the temperature range 560–720°C, and above 730°C, mass losses did not occur.
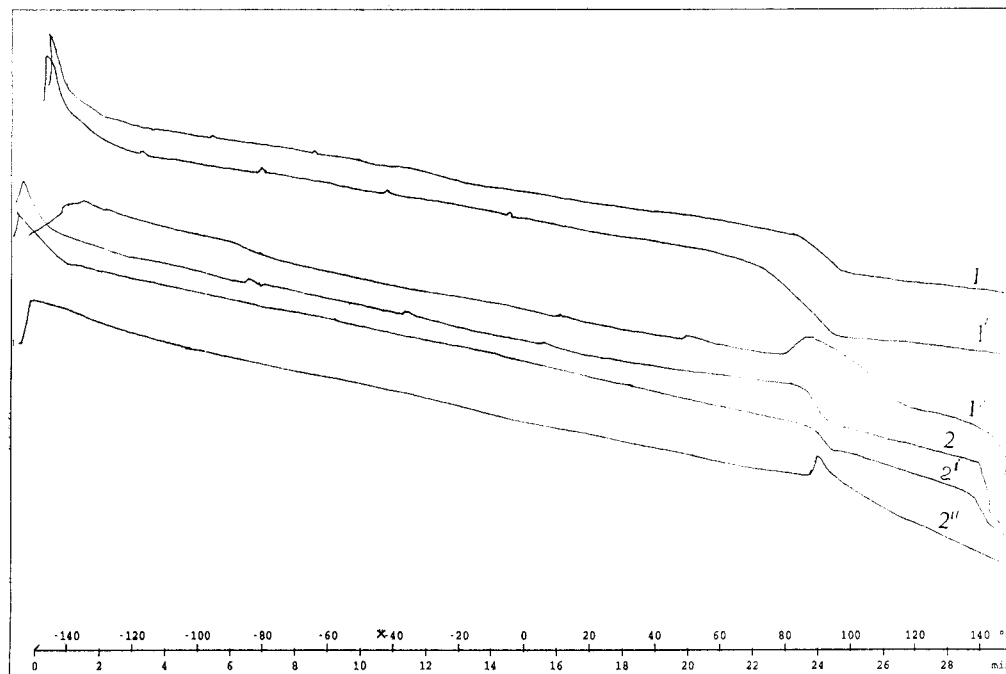
Figure 4 DSC thermograms of disilaoxaindane–dimethylsiloxane BCs. Curves 1 and 1′ correspond to the first and second heating scans, respectively, for copolymer VII, and curve 1″ corresponds to cooling scans for copolymer VII. Curves 2 and 2′ correspond to the first and second heating scans, respectively, for copolymer XII, and curve 2″ corresponds to cooling scans for copolymer XII.

The synthesized oligomeric $\alpha'\omega$-dihydroxy-1,3-disila-1,3-diphenyl-2-oxaindanes were used for heterofunctional polycondensation (HFC) with $\alpha'\omega$-dichlorodimethylsiloxanes. HFC was carried out with a 1:1 ratio of the initial compounds in a 60–70% anhydrous toluene solution in the presence of pyridine. HFC proceeded according to Scheme 3 [where $p \approx 17$ and n (length of the dimethylsiloxane chain) = 2 (V), 4 (VI), 12 (VII), 32 (VIII), or 66 (IX); $p \approx 23$ and n = 2 (X), 4 (XI), 12 (XII), 32 (XIII), or 66 (XIV); or $p \approx 29$ and n = 2 (XV), 4 (XVI), 12 (XVII), 32 (XVIII), or 66 (XIX)].

The obtained BCs were light yellow or white, viscous or solid products, depending on the length of the dimethylsiloxane chains, that were quite soluble in ordinary organic solvents, with an intrinsic viscosity ($[\eta]$) of approximately 0.12–0.37 dL/g. Some physicochemical properties, elemental analyses, and copolymer yields are presented in Table II. The partial fractionation of copolymer XIII from a toluene solution by methanol was performed, and four fractions (XIII[1] to XIII[4]) were obtained. As seen in Table II, the elemental analyses of these fraction were distinguished from one another a little. This demonstrated that the poly-

condensation mainly proceeded heterofunctionally.

In the IR spectra of synthesized BCs, there are absorption bands at 540 cm^{-1} and 940 and 1020 cm^{-1} that are characteristic of γ_s and γ_{as} $\equiv$Si—O—Si$\equiv$ bonds in 1,3-disila-2-oxaindane fragments and linear dimethylsiloxane chains, respectively. The absorption bands at 1275 and 1435 cm^{-1} prove the presence of $\equiv$Si—Me and $\equiv$Si—Ph bonds, and those bands at 3400–3600 cm^{-1} prove the presence of $\equiv$Si—OH bonds. In the ^{1}H-NMR spectra of block copolymer XVII from 0 to 0.2 ppm, there is overlapping of various resonance bands of different $\equiv$Si—Me groups; moreover, one can see the signal for methyl protons with the center $\delta \approx 0.1$ ppm and complicated multiplets for phenyl protons within the range 6.5–7.8 ppm. The ratio of phenyl and methyl protons corresponds to the proposed elemental link.

A GPC investigation of some BCs was carried out. In Figure 1, the molecular weight distributions (MWDs) of the copolymers are presented. The MWDs of the copolymers were monomodal, the molecular weights (M_ω) of the polymers ranged from 1.37×10^5 to 1.52×10^5, and the
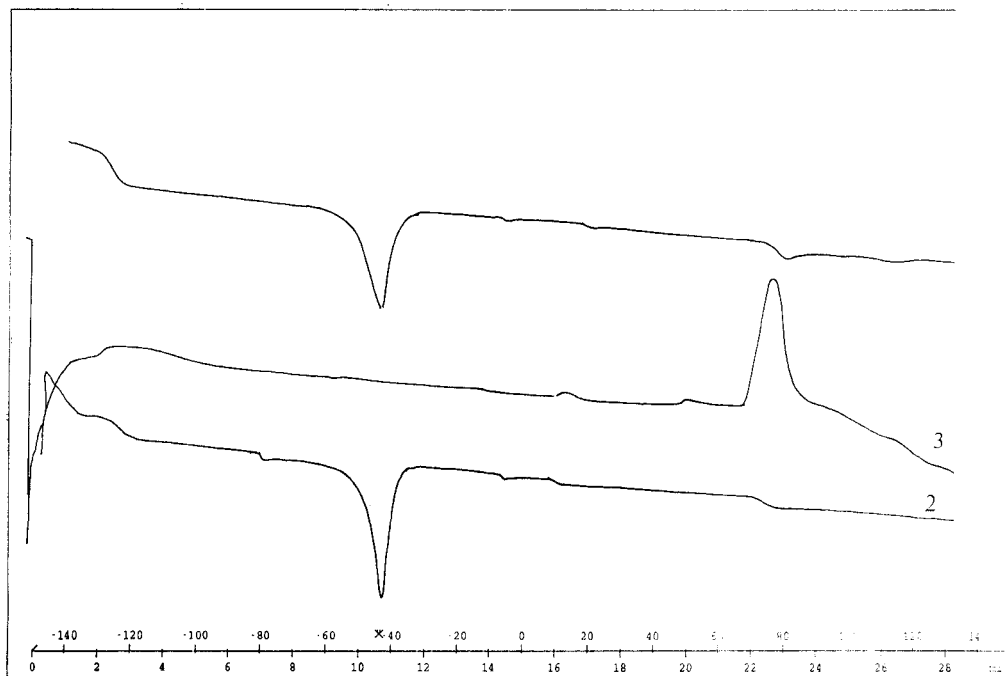
Figure 5 DSC thermograms of disilaoxaindane–dimethylsiloxane BCs. Curves 1 and 2 correspond to the first and second heating scans, respectively, for BC XIV, and curve 3 corresponds to cooling scans for BC XIV.

polydispersities of the polymers ranged from 1.32 to 1.73.The values of the molecular masses and polydispersities of some BCs are presented in Table II.

A roentgenographic analysis of synthesized BCs was carried out. As seen in Figures 2 and 3, for small values of n (2–12), the copolymers were one-phase systems. In the diffractograms, one can observe two maximums, $2\theta \approx 8.0-8.5°$ and $2\theta \approx 19.25-19.50°$. The main maximum $2\theta \approx 8.0-8.5°$ corresponds to the maximum value of the interchain distance, $d_1 \approx 11.05-10.40$ Å, and $2\theta \approx 19.25-19.50°$ corresponds to $d_2 \approx 4.49-4.55$ Å, which characterized both intrachain and inter-
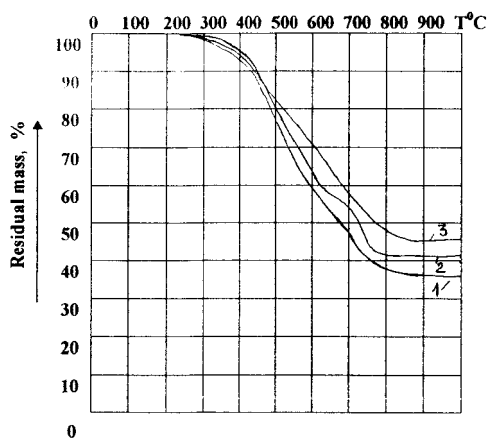

Figure 6 Thermogravimetric curves of disilaoxaindane–dimethylsiloxane BCs. Curve 1 corresponds to copolymer XVII, curve 2 corresponds to copolymer XII, and curve 3 corresponds to copolymer VII.
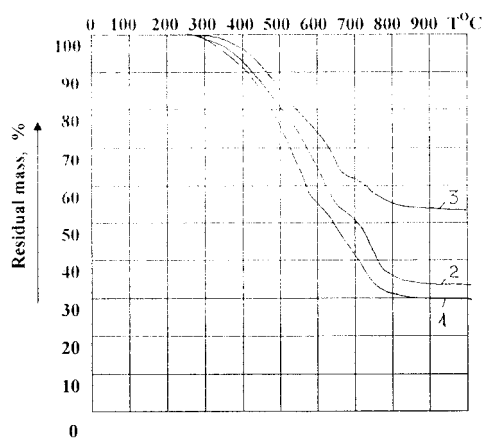

Figure 7 Thermogravimetric curves of disilaoxaindane–dimethylsiloxane BCs. Curve 1 corresponds to BC XIV, curve 2 corresponds to BC XIII, and curve 3 corresponds to BC X.

chain atomic interactions.[19] With an increase in n, the interchain distances decreased, and for copolymer XII ($n = 12$), $d_1 \approx 10.4$ Å. For higher values of the length of the dimethylsiloxane links ($n \geq 32$), a two-phase system formed. The formation of two-phase systems for such BCs can be explained by the high degree of incompatibility of the flexible dimethylsiloxane fragments with the rigid oligomeric 1,3-disila-1,3-diphenyl-2-oxaindane blocks. As seen in Figure 3, one can observe three maximums: $2\theta \approx 9.25$–$9.40°$, $2\theta \approx 11.50$–$11.60°$, and $2\theta \approx 19.25$–$20.40°$. The maximum $2\theta \approx 9.25$–$9.40°$ corresponds to the maximum values of the interchain distances between rigid oligomeric 1,3-disila-1,3-diphenyl-2-oxaindane blocks, $d_1 \approx 9.41$–10.72 Å. The third diffraction maximum at $2\theta \approx 11.50$–$11.60°$ corresponds to the interchain distance in the linear and flexible dimethylsiloxane blocks, $d_1 \approx 7.63$–7.70 Å. $2\theta \approx 19.25$–$20.40°$ corresponds to $d_2 \approx 4.23$–4.61 Å (see Fig. 3).

The synthesized BCs were studied with a DSC method. At small values of the linear dimethylsiloxane chain ($n \leq 12$), the copolymers were represented as one-phase systems with one transition temperature, the melting temperature ($T_{\text{melt}} \approx 84$–$91°C$; see Figure 4). An increase in the length of the rigid disilaoxaindane fragments did not noticeably influence the melting point of the copolymers. Curves 1 and 2 and curves 1′ and 2′ showed temperature transitions during the first and second heating scans, and curves I″ and I″ showed temperature transitions during the cooling scans (Fig. 4). This one temperature transition of the copolymers also was proven with thermomechanical investigations, and those values were near the DSC values. With the increase in n (≥ 32) shown in Figure 5, on the DSC curves one can observe the glass-transition temperature (T_g) from approximately -120 to $-123°C$ and T_{melt} at approximately $-45°C$; this value of T_{melt} is characteristic of flexible and linear PDMS chains and conforms with literature data.[5] $T_{\text{melt}} \approx +75°C$ is characteristic for rigid poly(disilaoxaindane) fragments (Fig. 5). In Figure 5, curves 1 and 1′ and curves 2 and 2′ show phase transitions during the first and second heating scans, and curves 3 and 3′ show phase transitions during the cooling scans. In this case, we clearly see the effects produced by the vitrification and melting of the dimethylsiloxane links and by the softening and melting of polycyclic disilaoxaindane blocks. The formation of a two-phase system for such BCs demonstrated the microdomain structures of these BCs. In thermomechanical curves, no melting transition characteristic of PDMS links was observed. Therefore, when the content of rigid polycyclic disilaoxaindane blocks was high, the polymer represented a continuous phase in which discrete aggregates of dimethylsiloxane fragments were distributed. The formation of the microdomain structure was observed only when p and n had definite values.

Thermogravimetric investigations of the synthesized BCs were carried out. As seen in Figure 6 and 7, the BCs were characterized by higher thermo-oxidative stability with respect to the linear PDMS. This could be explained by the introduction of more rigid fragments into the PDMS chains, which hindered cyclic depolymerization,[2] and by the high resistance of the phenyl groups to oxidation and their inhibiting effect on the oxidation of the methyl groups.[3] As seen in Figure 6, an increase in the length of the rigid poly(disilaoxaindane) blocks did not noticeably influence the thermo-oxidative stability of the BCs; 10% mass losses can be observed within the 400–$450°C$ temperature range. With respect to the disilaoxaindane–dimethylsiloxane copolymers,[14] for synthesized BCs the main destruction process proceeded at higher temperatures, 450–$800°C$, and above $800°C$, curves of mass losses did not occur. Therefore, with the insertion of rigid poly(disilaoxaindane) blocks into the linear PDMS chains, the main destruction process proceeded about 100–$120°C$ higher than for unblocked PDMS. For both, with an increase in the length of the dimethylsiloxane links, the mass losses increased (Fig. 7).

The synthesized BCs are interesting products because they contain reactive cyclosiloxane fragments; in the presence of an anionic catalyst, it is possible to cause the opening of cyclosiloxane links without the selection of volatile gaseous products. Therefore, it is possible to regulate the frequency of the reticle.

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