

# Synthesis and Investigation of Properties of Silaoxadihydrophenanthrene-Diphenylsiloxane Fragments Containing Block Copolymers

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**ABSTRACT:** The reaction of heterofunctional condensation of 1,1-dichloro-1-sila-2-oxadihydrophenanthrene with dihydroxydiphenylsilane at various ratios of initial compounds in the presence of pyridine is investigated.  $\alpha,\omega$ -Dihydroxysilaoxadihydrophenanthrene-diphenylsiloxane oligomers with various degrees of condensation are obtained. Organosiloxane block copolymers with the regular arrangement of silaoxadihydrophenanthrene-diphenylsiloxanes fragments in the main linear dimethylsiloxane chain are produced by the reaction of heterofunctional condensation of  $\alpha,\omega$ -dihydroxysilaoxadihydrophenanthrene-diphenylsiloxanes with  $\alpha,\omega$ -dichlorodimethylsiloxanes in the presence of anhydrous pyridine, as an acceptor of hydrochloric acid. Thermogravimetry, differential scanning calorimetry, gel permeation chromatography, and wide-angle X-ray analysis are carried out on the synthesized block copolymers. The microheterogeneous structure of block copolymers is observed at definite values of the length of the flexible dimethylsiloxane chain by DSC and X-ray investigation. © 2002 John Wiley & Sons, Inc. *J Appl Polym Sci* 84: 9–16, 2002; DOI 10.1002/app.10045

**Key words:** organosilicon block copolymers; silaoxadihydrophenanthrene; heterofunctional polycondensation

## INTRODUCTION

There is some information in the literature about the card-type organic silicon copolymers cyclo-trisiloxane<sup>1</sup> and cyclocarbosiloxane<sup>2</sup> and with silaoxadihydrophenanthrene fragments in the linear dimethylsiloxane (DMS) chain.<sup>3</sup> It was shown that by the insertion of silaoxadihydrophenanthrene fragments in the DMS chain the thermal-oxidative stability of copolymers increases compared with the linear unblocked poly(DMS) (PDMS). This may be explained by the presence of

aromatic phenyl groups that are highly resistant to oxidation and have an inhibiting effect on the oxidation of methyl groups<sup>2,3</sup> and by the break of the spiral structure of the linear PDMS.<sup>4</sup> At the same time, it is known that introduction of groups distinct from  $\equiv\text{Si}-\text{O}-$  links into the PDMS backbone leads to breakdown of the destruction processes of the depolymerization mechanism, because of the impossibility of formation of a transitive complex.<sup>5</sup>

In literature there is also information about  $(\text{AB})_m\text{C}_n$  type organic silicon block copolymers (BCs) with organocyclotetra(hexa)siloxane and aromatic inclusions in the main DMS chain<sup>6,7</sup> and the side chain. In both cases the phase incompatibility of the BC is observed at the definite values

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of the lengths of the rigid and flexible DMS chains.

## EXPERIMENTAL

The 1,1-dichloro-1-silaoxaphenanthrene and  $\alpha,\omega$ -dihydroxydimethylsiloxanes starting materials with various lengths were synthesized by a well-known method.<sup>8,9</sup>

The organic solvents were purified by drying and distillation. The purity of the starting compounds was controlled by gas-liquid chromatography (LXM-8MD), phase SKTFT-100 (10%), the NAW chromosorb, helium as the carrier gas, and a 2M column.

The IR spectra of the synthesized compounds were taken on an UR-20 instrument and <sup>1</sup>H-NMR spectra were gathered with a Perkin-Elmer instrument operating at a frequency of 250 MHz in a solution of C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub> or CDCl<sub>3</sub>.

The gel permeation chromatography (GPC) investigation was carried out with a Waters model 6000A chromatograph with an R 401 differential refractometer detector. The column set comprised 10<sup>3</sup> and 10<sup>4</sup> Å Ultrastaygel columns. Sample concentrations were approximately 3% by weight in toluene, and a typical injection volume for the siloxane was 5  $\mu$ L. Standardization of the GPC was accomplished by the use of styrene or PDMS standards with a known molecular weight.

Wide-angle X-ray investigation were carried out on a DRON-2 instrument using Cu $\alpha$  radiation. The values were measured without a filter, and the angular velocity of the motor was  $\approx 2^\circ$ /min.

The differential scanning calorimetry (DSC) analyses were carried out on a Perkin-Elmer DSC-7 with a cooling accessory for the determination of thermal transitions in the polymers. A temperature increase or scanning rate decrease of  $\sim 10^\circ\text{C}/\text{min}$  was used. The glass-transition temperatures ( $T_g$ ) were read from endothermic DSC traces, which were approximated to be midpoints between the extrapolated tangents to the baselines above and below the glass-transition region.

Thermogravimetric investigations were carried out on a Paulic-Paulic Erdey derivatograph (model MOM-102). The test conditions were a temperature rise rate of  $\approx 5^\circ\text{C}/\text{min}$  in an open area.

### Heterofunctional Condensation of 1:0.9 1,1-Dichloro-2-Oxadihydrophenanthrene with Dihydroxydiphenylsilane

The polycondensation reaction was carried out in a three-necked flask equipped with a reflux condenser, a dropping funnel, and a mechanical stirrer. To the solution of 7.00 g (26.2172 mmol) of 1,1-dichloro-1-sila-2-oxaphenanthrene in 7 mL of anhydrous toluene at room temperature was added the solution of 5.0966 g (23.5955 mmol) dihydroxydiphenylsilane and 4.1476 g (26.2172 mmol) anhydrous pyridine. The mixture was stirred and heated to the boiling temperature of toluene for 5 h. Then 8 mL of toluene was added, and the reaction mixture was filtered and washed before neutral ambience and to remove the traces of the pyridine hydrochloric acid complex. The mixture was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, part of the toluene was removed and reprecipitated by using methyl alcohol, and 9.9 g of 92%  $\alpha,\omega$ -dihydroxydihydrosilaoxaphenanthrene-diphenylsiloxane oligomer I was received.

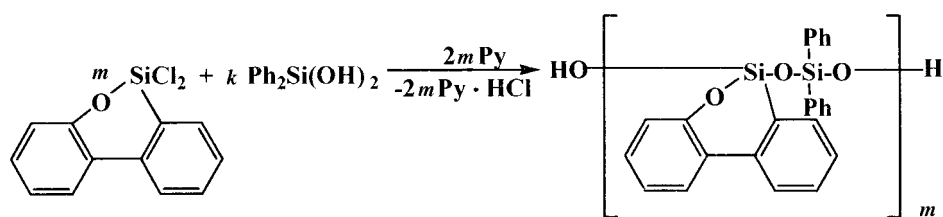
### Heterofunctional Condensation of 1:0.95 1,1-Dichloro-2-Oxadihydrophenanthrene with Dihydroxydiphenylsilane

The polycondensation reaction was carried out in a three-necked flask equipped with a reflux condenser, dropping funnel, and mechanical stirrer. To the solution of 5.00 g (18.7265 mmol) of 1,1-dichloro-1-sila-2-oxaphenanthrene in 5 mL of anhydrous toluene at room temperature was added the solution of 3.8426 g (17.7902 mmol) of dihydroxydiphenylsilane and 2.9625 g (18.7265 mmol) of anhydrous pyridine. The mixture was stirred and heated to the boiling temperature of toluene for 5 h; after that, 8 mL of toluene was added. Then the reaction mixture was filtered and washed before neutral ambience and to remove the traces of pyridine hydrochloric acid complex, it was dried above anhydrous Na<sub>2</sub>SO<sub>4</sub>, part of the toluene was removed and reprecipitated by using methyl alcohol, and 7.1 g of 93%  $\alpha,\omega$ -dihydroxydihydrosilaoxaphenanthrene-diphenylsiloxane oligomer II was received.

### Heterofunctional Polycondensation of $\alpha,\omega$ -Dihydroxysilaoxadihydrophenanthrene-Diphenylsiloxane ( $m \approx 10$ ) with 1,3-Dichlorotetramethyldisiloxane

The polycondensation reaction was carried out in a three-necked flask equipped with a reflux con-

denser, dropping funnel, and mechanical stirrer. The solution of 2.3227 g (0.564 mmol) of  $\alpha,\omega$ -dihydroxydihydrosilaoxaphenanthrene-diphenylsiloxane and 0.08922 g (1.128 mmol) of dry pyridine in 2 mL of anhydrous toluene at room temperature was added to the solution of 0.1145 g (0.564 mmol) of 1,3-dichlorotetramethyldisiloxane in 0.4 mL of anhydrous toluene. The mixture was stirred and heated to the boiling temperature of toluene for 5 h, and after that 8 mL of toluene was added. The reaction mixture was filtered and washed before neutral ambience to remove the traces of pyridine hydrochloric acid complex; it was dried above anhydrous  $\text{Na}_2\text{SO}_4$ , part of the toluene was removed and reprecipitated by using methyl alcohol, and 2.2 g of 89% copolymer III was attained.



where  $m \approx 10$  (I) and 14 (II).

As seen from the structure of 1,1-dichloro-1-sila-2-oxadihydrophenanthrene, it contains a six-member heterocyclic fragment with a Si—O—C bond. From the literature it is known that the ordinary Si—O—C bond is easily cleaved by basic and acid reagents, and the Si—C<sub>aryl</sub> bond is especially sensitive to the action of acids. However, it was shown that the cyclic fragment in 1,1-dichloro-1-sila-2-oxadihydrophenanthrene is not destroyed during boiling with water, alkali-alcohol solution, and concentrated hydrogen chloride.<sup>8</sup> It was demonstrated that alkylation and arylation with Grignard reagents, restoration with  $\text{LiAlH}_4$ , and processing with hydrogen fluoride do not break the Si—O—C bond. The high chemical stability of silaoxadihydrophenanthrene fragments indicates a definite degree of aromaticity of these compounds.<sup>10</sup>

After reprecipitation of the reaction products from toluene solution by methanol, white solid-type  $\alpha,\omega$ -dihydroxysilaoxadihydrophenanthrene-diphenylsiloxane oligomers were obtained that were soluble in ordinary organic solvents.

The composition and structure of the synthesized oligomers were determined by means of

The typical reactions of the synthesis of other BCs were carried out by the above-mentioned method.

## RESULTS AND DISCUSSION

Heterofunctional condensation reactions of 1,1-dichloro-2-oxadihydrophenanthrene with dihydroxydiphenylsilane at 1:0.9 and 1:0.95 ratios of the initial compounds in the presence of dry pyridine in a 50–60% solution of anhydrous toluene were carried out to synthesize silaoxadihydrophenanthrene-diphenylsiloxane containing dihydroxy end group oligomers. The reaction proceeds according to the following:

elementary and functional analysis, ascertaining the molecular masses, and IR spectra data. Some physical-chemical properties of the synthesized oligomers are presented in Table I.

In the IR spectra of the synthesized oligomers one can observe absorption bands characteristic for asymmetric valence oscillation of  $\equiv\text{Si—O—Si}\equiv$  and  $\equiv\text{Si—O—C}\equiv$  bonds in the 1020 and 1075  $\text{cm}^{-1}$  region. Accordingly, one can also see absorption bands of CH—bonds characteristic for monosubstituted phenyl rings in the 3030–3080 and 1590  $\text{cm}^{-1}$  region and for  $\equiv\text{Si—Ph}$  bonds at 1435  $\text{cm}^{-1}$ . There are also absorption bands for associated and nonassociated  $\equiv\text{Si—OH}$  bonds in the 3200–3600 and 920  $\text{cm}^{-1}$  regions, respectively.

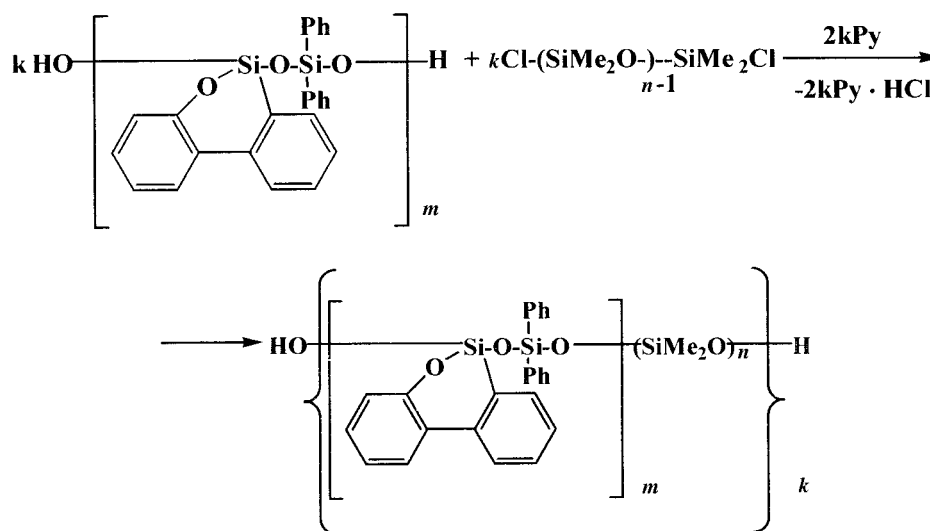
The synthesized oligomeric  $\alpha,\omega$ -dihydroxysilaoxadihydrophenanthrene-diphenylsiloxanes were used in the heterofunctional reaction with  $\alpha,\omega$ -dichlorodimethylsiloxanes. The reaction of heterofunctional condensation was carried out at a 1:1 ratio of initial compounds in a 50–60% anhydrous solution of toluene in the presence of anhydrous pyridine in a temperature range of 20–25°C; at the completion stage, the reaction mixture was heated to the boiling point of toluene for 6–7 h. The reaction proceeds according to the following:

**Table I** Physical–Chemical Properties of  $\alpha,\omega$ -Dihydroxysilaoxadihydrophenanthrene-Diphenylsiloxanes

No.	$\eta_{sp}^a$	$d_1$ (Å)	$T_{melt}$ (°C)	OH (%)	$\bar{M}^b$	Yield (%)
I	0.04	11.05	67–72	0.83	4111	92
				0.81	4200	
II	0.05	11.05	74–77	0.59	5758	93
				0.56	6070	

<sup>a</sup> The specific viscosity in 1% toluene solution at 25°C.

<sup>b</sup> The calculated values are in the numerator, and the experimental values are in the denominator.



where  $m \approx 10$ ,  $n = 2$  (III), 10 (IV), 21 (V), 27 (VI), and 39 (VII);  $m \approx 14$ ,  $n = 2$  (VIII), 10 (IX), 21 (X), 27 (XI), and 39 (XII).

The synthesized copolymers after reprecipitation from the toluene solution, depending on the length of the linear DMS chain, are solid or liquid systems with a specific viscosity of 0.10–0.34 and are soluble in ordinary organic solvents. Some physical–chemical properties of the synthesized copolymers are presented in Table II. Four fractions were obtained by partial reprecipitation of copolymer V from the toluene solution by methanol. As seen from Table II the elementary analyses of these fractions V<sup>1</sup>–V<sup>4</sup> are slightly distinguished from each other, which shows that, during heterofunctional condensation, the homofunctional polycondensation is improbable and copolymers with a regular arrangement of the silaoxadihydrophenanthrene-diphenylsiloxanes fragments in the DMS chain are obtained. In the

IR spectra of copolymers one can observe absorption bands characteristic for asymmetric valence oscillation of  $\equiv\text{Si}-\text{O}-\text{Si}\equiv$  and  $\equiv\text{Si}-\text{O}-\text{C}\equiv$  bonds in the 1020 and 1075  $\text{cm}^{-1}$  region; accordingly, one can also see absorption bands of  $\text{CH}-$  bonds characteristic for monosubstituted phenyl rings in the 3030–3080 and 1590  $\text{cm}^{-1}$  region and for  $\equiv\text{Si}-\text{Ph}$  bonds at 1435  $\text{cm}^{-1}$ . One can also see absorption bands characteristic for  $\equiv\text{Si}-\text{Me}$  bonds at 1275 and 1410–1412  $\text{cm}^{-1}$ . Because the initial compound I in the oligomeric structure contained only phenyl groups, by a comparison of the IR spectra of polymers III–VII one could trace the tendency for alteration of the correlation between valence asymmetric oscillations of methyl groups at 2900  $\text{cm}^{-1}$  and composite valence oscillations of  $\text{CH}-$  bonds of monosubstituted phenyl rings at 3030–3070  $\text{cm}^{-1}$ . The changes in the correlations of the intensities in the samples were observed at 1410–1412  $\text{cm}^{-1}$ , which is character-

**Table II Physical-Chemical Properties of Silaoxadihydrophenanthrene-Diphenylsiloxane-Dimethylsiloxane Block Copolymers**

No.	Yield (%)	$[\eta]^a$ (dL/g)	$d_1$ (Å)	$T_g$ (°C)	$T_{\text{melt}}, T_{\text{soft}}$ (°C)	5% Mass Losses	Residual Mass (%)	$\bar{M}_w \times 10^{-5}$ (Da)	Elementary Composition <sup>b</sup>		
									C	H	Si
III	89	0.08	10.35	—	+61 to +65	—	—	—	68.63	4.54	14.43
									68.21	4.30	14.50
IV	91	0.12	10.4	-115	+27	260	10.4	—	64.22	4.98	17.27
									63.87	4.72	16.95
V	93	0.21	7.70	-123	+27	385	9.6	—	59.66	5.43	20.24
			10.15						59.34	5.09	20.37
V <sup>1</sup>	10	0.35	—	—	—	—	—	—	59.66	5.43	20.24
V <sup>2</sup>	12	0.27	—	—	—	—	—	—	59.11	5.00	20.11
									59.66	5.43	20.24
V <sup>3</sup>	42	0.24	—	—	—	—	—	—	59.24	5.21	20.04
									59.66	5.43	20.24
V <sup>4</sup>	36	0.17	—	—	—	—	—	—	59.38	5.37	20.33
									59.66	5.43	20.24
VI	92	0.26	7.70	-123	-40 to -60	—	—	1.27 (1.41)	57.68	5.62	21.52
			10.1						+41 to +48	57.45	5.30
VII	94	0.31	7.53	-123	-35 to -54	—	—	—	54.48	5.94	23.59
			9.83						+39 to +45	54.07	5.43
VIII	92	0.11	10.1	—	+64 to +69	—	—	—	67.33	4.67	15.35
			—						—	66.78	4.30
IX	91	0.14	10.1	—	+45	375	14.4	—	65.74	4.83	15.37
			—						—	65.32	4.42
X	94	0.28	7.50	-122	+35	3.95	8.8	—	62.03	5.20	18.76
			9.83						—	61.69	4.87
XI	93	0.30	7.87	-123	-39 to -60	—	—	1.98 (1.53)	60.34	5.36	19.85
			10.1						+40 to +53	59.86	5.03
XII	94	0.34	—	-123	-36 to -52	—	—	—	57.47	5.65	21.70
			—						+35 to +42	56.98	5.31

<sup>a</sup> In 1% toluene solution at 25°C. Average weight molecular masses were determined by gel permeation chromatography.

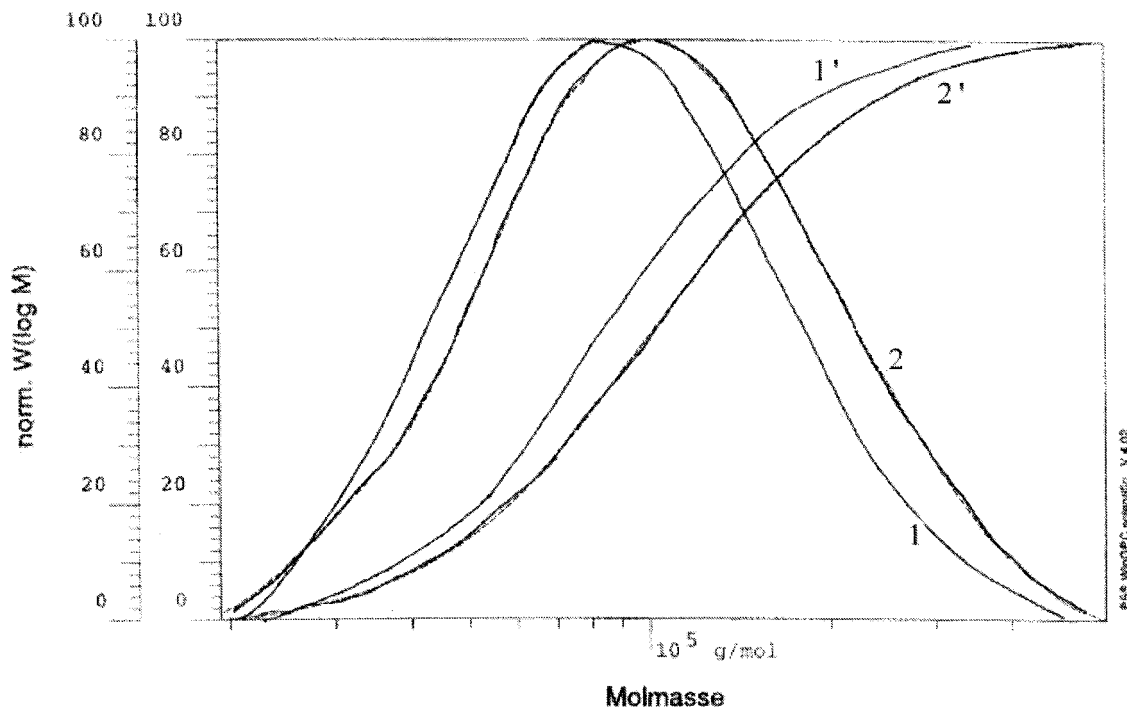
<sup>b</sup> The calculated values are in the numerator, and the experimental values are in the denominator.

istic for  $\equiv\text{Si}-\text{CH}_3$  bonds, and at  $1435\text{ cm}^{-1}$ , which is characteristic for  $\equiv\text{Si}-\text{Ph}$  bonds. Crossing from DMS fragments at  $n = 2$  to  $\approx 39$  leads to the increase of the intensity of signals characteristic of  $\equiv\text{Si}-\text{CH}_3$  bonds.

The GPC analysis of some of the synthesized copolymers was carried out. As is seen from Figure 1 the copolymers have a monomodal molecu-

lar weight distribution of  $(\approx 1.27-1.98) \times 10^5$  and polydispersity changes of  $\approx 1.41-1.53$ .

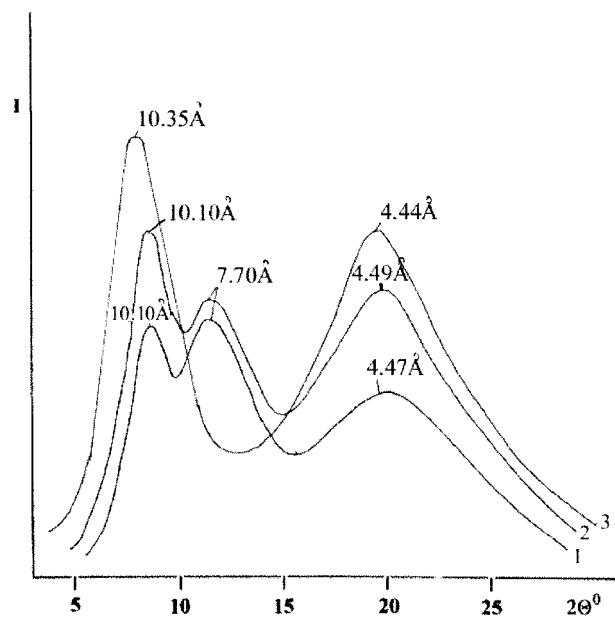
A wide-angle X-ray investigation was carried out for the synthesized copolymers. Figures 2 and 3 show that, depending on the lengths of the rigid silaoxadihydrophenanthrene-diphenylsiloxanes fragments ( $m$ ) and flexible PDMS fragments ( $n$ ) in the copolymers, a block structure and phase



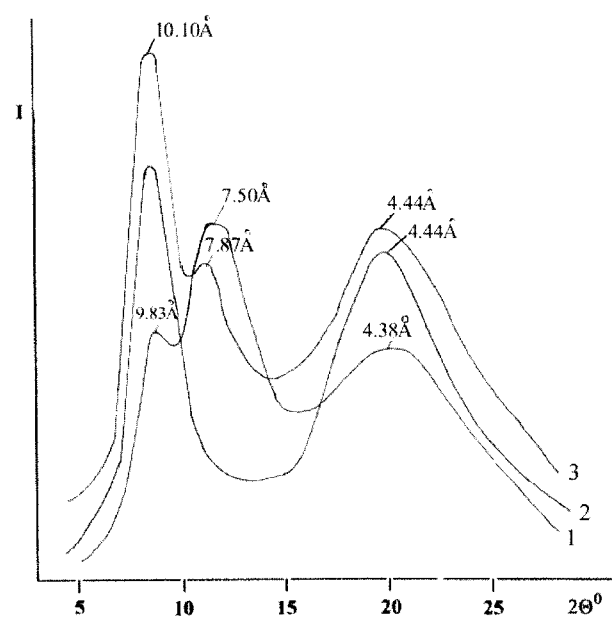
**Figure 1** Gel permeation chromatographic curves of silaoxidihydrophenanthrene-diphenylsiloxane-dimethylsiloxane block copolymers XI (curves 1 and 1<sup>1</sup>) and VI (curves 2 and 2<sup>1</sup>).

incompatibility are realized. At small values of the DMS chain ( $n \geq 10$ ) the copolymers were a one phase system and the maximum value of the

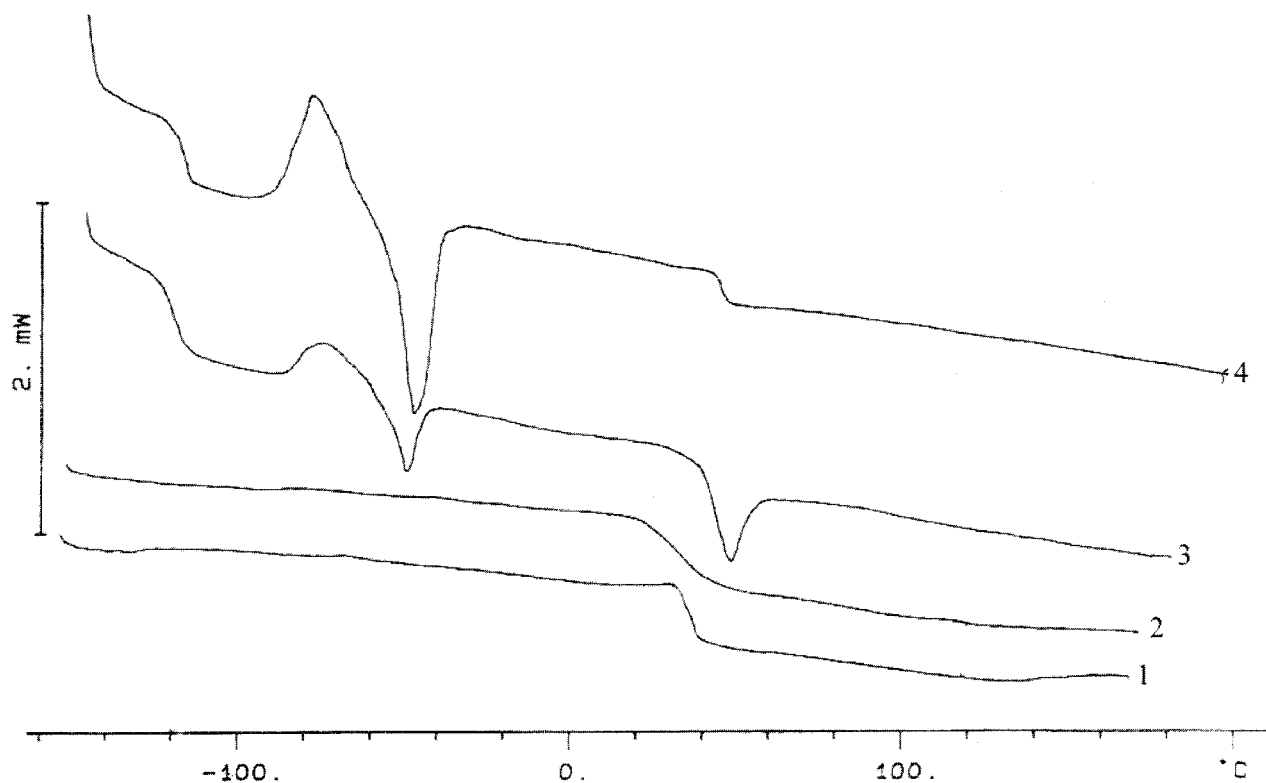
interchain distances was achieved at  $n = 2$ . On the diffractograms one can see two diffraction maximums  $2\theta \approx 8.45^\circ$  and  $2\theta \approx 20-20.25^\circ$ . The



**Figure 2** Wide-angle roentgenographic curves of silaoxidihydrophenanthrene-diphenylsiloxane-dimethylsiloxane block copolymers VI (curve 1), V (curve 2), and III (curve 3).



**Figure 3** Wide-angle roentgenographic curves of silaoxidihydrophenanthrene-diphenylsiloxane-dimethylsiloxane block copolymers XI (curve 1), X (curve 2), and IX (curve 3).



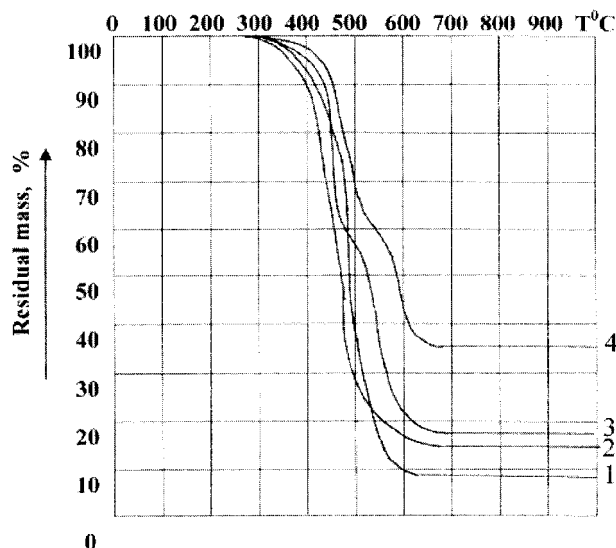
**Figure 4** Differential scanning calorimetric curves of siloxadihydrophenanthrene-diphenylsiloxane-dimethylsiloxane block copolymers IX (curve 1), IV (curve 2), VI (curve 3), and XI (curve 4).

maximum value of the interchain distances for copolymers III and VIII is  $d_1 \approx 10.35 \text{ \AA}$ . The second maximum at  $2\theta \approx 20\text{--}20.25^\circ$  corresponds to  $d_2 \approx 4.38\text{--}4.49 \text{ \AA}$ ; this value of  $d_2$  corresponds to intra- and interchain atomic distances.<sup>11</sup> With an increase of the length of PDMS chain ( $n < 21$ ) the phase incompatibility begins to be revealed and two phase systems were obtained. As seen from Figures 3 and 4, in the diffractograms the third diffraction maximum in the range  $d_1 \approx 7.50\text{--}7.87 \text{ \AA}$  appeared, which corresponds to interchain distances of the linear flexible PDMS. The increasing value of the length of  $m$  does not lead to the appreciable rise of the  $d_1$ , but with the increase of the length of PDMS fragments the intensity of the third maximum rises. Thus, when  $n \geq 21$  the segregation processes take place separately for rigid siloxadihydrophenanthrene-diphenylsiloxanes fragments as well as for flexible linear PDMS chains with separate formation of domains for rigid and flexible blocks. These domains are divided into separate phases, each of which are realized as an independent packing.

The synthesized BCs were studied by the DSC method. At small values of the length of the flex-

ible PDMS chain ( $n < 21$ ) the copolymers were represented as one phase systems with one softening temperature. With an increase of the length of the chains ( $n \geq 21$ ) on the DSC curves (Fig. 4, curves 1, 2) one can observe two glass-transition temperatures in the range of  $\approx -110^\circ$  to  $\approx -123^\circ\text{C}$ , which is characteristic for linear PDMS chains, and  $\approx +40$  to  $+50^\circ\text{C}$ , which is characteristic for rigid siloxadihydrophenanthrene-diphenylsiloxane fragments. Crystallization temperatures ( $T_{\text{cryst}}$ ) of  $\approx -100$  to  $-60^\circ\text{C}$  and melting temperatures ( $T_{\text{melt}}$ ) of  $\approx -60$  to  $-40^\circ\text{C}$  are characteristic for linear PDMS blocks (Fig. 4, curves 3, 4). This melting temperature value characteristic for linear PDMS blocks changes a little.

The thermogravimetric investigation of the synthesized BCs was carried out. Figure 5 shows that the BCs are characterized by higher thermal-oxidative stability than linear PDMS. There are 10% mass losses for BCs that one can observe in the  $390\text{--}420^\circ\text{C}$  range. The main destruction process proceeds in the  $450\text{--}540^\circ\text{C}$  range, and above  $600^\circ\text{C}$  the mass losses do not occur. With the increase of the length of linear PDMS chain the losses of BC rises (Fig. 5, curves 1, 4). With



**Figure 5** Thermogravimetric curves of silaoxadihydrophenanthrene-diphenylsiloxane-dimethylsiloxane block copolymers XI (curve 1), IV (curve 2), IX (curve 3), and VIII (curve 4).

the increase of the length of rigid silaoxadihydrophenanthrene-diphenylsiloxanes fragments the thermal-oxidative stability of the BC rises slightly (Fig. 5, curves 2, 3).

## CONCLUSIONS

Organosiloxane BCs with a regular arrangement of silaoxadihydrophenanthrene-diphenylsiloxanes fragments in the main linear DMS chain were synthesized by us for the first time by a heterofunctional condensation reaction of  $\alpha,\omega$ -dihydroxysilaoxadihydrophenanthrene-diphenylsiloxanes with  $\alpha,\omega$ -dichlorodimethylsiloxanes in the pres-

ence of anhydrous amines. We showed that inserting the silaoxadihydrophenanthrene-diphenylsiloxanes fragments in the DMS chain caused the thermal-oxidative stability of the copolymers to rise and the main destruction process proceeds at 80–100°C higher than the destruction of unblocked PDMSs.<sup>12,13</sup> The microheterogeneous structure of the BCs is observed by DSC and X-ray investigation at definite values of the length of the flexible DMS chain.

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