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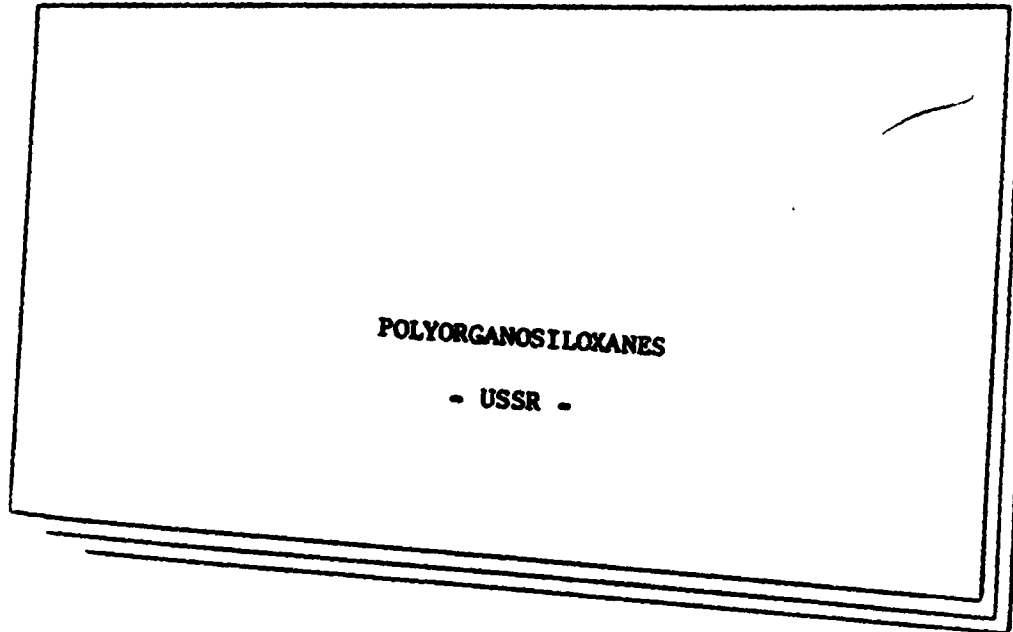
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FOREWORD

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POLYORGANOSILOXANES

-USSR-

Following is the translation of two articles in the Russian-Language publication Vysokomolekulyarnyye soyedineniya (High-molecular Compounds), Vol IV, No 2, Moscow, 1962. Additional bibliographic information accompanies each article.

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POLYORGANOTITANOSILOXANES

VI. THE COHYDROLYSIS REACTION OF BIS - (ACETYLACETONATE)DICHLOROTITANIUM WITH ALKYL(ARYL)TRICHLOROSILANES

Following is the translation of an article by K. A. Andrianov, Sh. V. Pichkadze, and I. V. Bochkareva in the Russian language publication Vysokomolekulyarnyye Soyedineniya (High-Molecular Compounds), Vol IV, No 2, Moscow, 1962, pages 256-260.⁷

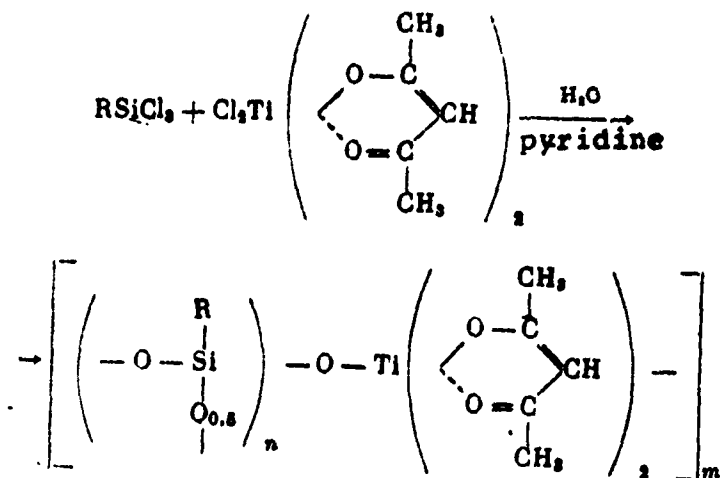
There is no data available in the literature concerning investigation of the cohydrolysis reaction of trifunctional silico-organic compounds with difunctional titano-organic compounds. In one of the papers it was shown that bis-(acetylacetonate)dichlorotitanium, upon cohydrolysis with dialkyl(aryl)dichlorosilanes, reacts like a difunctional monomer with the formation of polymers.¹

There is a definite interest to investigate the reaction of hydrolysis of bis-(acetylacetonate)dichlorotitanium with trifunctional silico-organic compounds.

This paper presents experimental data on the cohydrolysis reaction of bis(acetylacetonate)dichlorotitanium with methyl-ethyl- and phenyl-trichlorosilanes. The hydrolysis reaction was studied in an aqueous medium, using pyridine as an acceptor and toluol as a solvent.

Tests have shown that the hydrolysis reaction of bis-(acetylacetonate)dichlorotitanium with alkyl(aryl)trichlorosilanes takes place according to the equation shown on the next page, and results in the production of high-molecular compounds. However the composition of the polymers depends on the organic group of alkyl(aryl)trichlorosilanes used in the reaction.

In conducting the reaction in molar ratios of alkyl(aryl)trichlorosilanes to bis-(acetylacetonate)dichlorotitanium, the ratio of silicone atoms to titanium in the composition of the polymer did not correspond to the one used in the reaction. In every instance we observed a higher Si:Ti ratio in the polymer, and the maximum ratio was



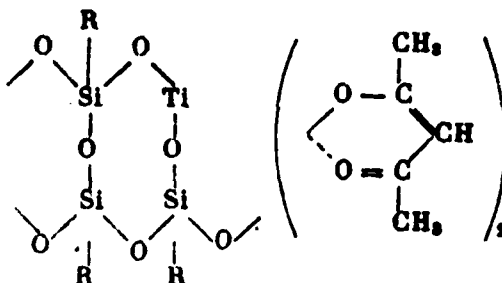
obtained with hydrolysis of bis-(acetylacetonate)dichloro-titanium with methyltrichlorosilane, while the minimum was obtained for poly-bis-(acetylacetonate)titanophenylsiloxane (see Table 1).

The data obtained from elementary analysis and analysis of the functional groups of polymers show that the composition of the polymers corresponds to a link in the polymer chain as shown in table 1. The molecular weight, as determined by the osmometric method for poly-bis-(acetylacetonate)titanophenylsiloxane, was greater and equalled 103,000 (this weight was calculated by Yu. S. Ksimantovskaya).

Investigation of the infra-red spectra of poly-bis-(acetylacetonate)titanoalkyl-(aryl)siloxanes showed that all of the polymers have an absorption band for the Ti - O bond in the Ti - O - Si group, and total absorption is observed in the region that is characteristic for Si - O bond in the Si - O - Si group (see Table 2).

The polymers' good solubility as well as their thermo-mechanical properties (Figure 1, a) are indicative of the absence of bonded [literally: stitched] or spacial molecular structure, while the data obtained by elementary analysis and functional group analysis correspond to the link in the chain whose molecule must have a bonded [stitched] or cyclo-linear structure. By comparing the experimental data from the analyses and studying the properties of poly-bis-(acetylacetonate) titanomethylsiloxane, poly-bis-(acetylacetonate)titanoethylsiloxane and poly-bis-(acetylacetonate)titanophenylsiloxane we are permitted to conclude that they have a cyclo-linear structure of such a composition that it is possible to have chains of both eight- and

six-member rings bonded in the chain with oxygen.



Investigation of the viscosity of the solutions in benzol has shown that the viscosity of poly-bis-(acetylacetonate)titanophenylsiloxane constituted 0.073, that of poly-bis-(acetylacetonate)titanoethylsiloxane -- 0.0670, and that of poly-bis-(acetylacetonate)titanomethylsiloxane -- 0.069. These data are not typical for filamented molecules, which speaks in favor of the above-mentioned molecular structure of the polymer. The polymers obtained were submitted to structurized heating at 100, 160 and 200°. It was established that as a result of such heating all of the polymers lost their solubility (table 3).

As can be seen, only poly-bis-(acetylacetonate)titanophenylsiloxane retained partial solubility even after heating at 200° for a period of four hours, while poly-bis-(acetylacetonate)titanomethylsiloxane and poly-bis-(acetylacetonate)titanoethylsiloxane lose their solubility entirely.

At the same time, after heating the thermomechanical properties of the polymers are sharply altered (see Figure 1, b). After heating, the polymers possess the typical properties of structurized polymers.

Roentgenostructural examination of these polymers indicates (figure 2) that poly-bis-(acetylacetonate)titanomethylsiloxane and poly-bis-(acetylacetonate)titanoethylsiloxane have a structure that, by the degree of regularity in the arrangement of macromolecules, is transitional, between crystalline and amorphous structures, and only poly-bis-(acetylacetonate)titanophenylsiloxane has an amorphous structure.

All of the synthesized polymers are in the form of a yellow substance which, in a soluble state, forms hard films on the surface after evaporation of the solvent.

Experimental Section

Bis-(acetylacetonate)dichlorotitanium was synthesized

by the Rosenheim² method.

Cohydrolysis of methyltrichlorosilane with bis-(acetylacetonate)dichlorotitanium. The reaction was done in a four-neck flask equipped with a mechanical stirrer, a reverse condenser and two dropping funnels. Into the flask were placed 100 milliliters of toluol, 1416 grams (0.0947 mols) of methyltrichlorosilane, and 30 grams (0.0947 mols) of bis-(acetylacetonate)dichlorotitanium. While stirring, pyridine and a ten-fold excess of required water were introduced through the dropping funnels. Upon termination of the hydrolytic reaction the toluol layer was separated from the aqueous layer and it was washed several times with distilled water. The toluol was distilled under reduced pressure, and the polymer obtained was vacuum-dried until it reached a constant weight. We obtained 12.55 grams of polymer (yield: 40.3% of the theoretical).

Cohydrolysis of ethyltrichlorosilane with bis-(acetylacetonate)dichlorotitanium. The synthesis was conducted with the equipment described above; from 15.48 grams (0.0947 mols) of ethyltrichlorosilane and 30 grams (0.0947 mols) of bis-(acetylacetonate)dichlorotitanium we obtained 18 grams of polymer (yield: 48% of the theoretical).

Cohydrolysis of phenyltrichlorosilane with bis-(acetylacetonate)dichlorotitanium. Poly-bis-(acetylacetonate)titanophenylsiloxane was synthesized from 30 grams (0.0949 mols) of bis-(acetylacetonate)dichlorotitanium and 20.03 grams (0.0949 mols) of phenyltrichlorosilane. We obtained 23.19 grams of polymer (yield: 62.6% of the theoretical).

The solubility of the polymers was determined by the method described in the literature. The hydroxyl groups were determined by the Terent'yev method. The thermomechanical examination was conducted according to the method described⁴.

The infra-red spectra were obtained in the INBOS optical laboratory, under the management of I. V. Obreimov; the thermomechanical measurements were conducted in the laboratory of polymer physics managed by G. L. Slonimskiy. The roentgenograms were taken in the laboratory of roentgenostructural analysis directed by A. I. Kitaygorodskiy. The authors wish to express their appreciation for the assistance rendered by the workers in these laboratories.

CONCLUSIONS

1. We investigated the cohydrolysis reaction of alkyl(aryl)trichlorosilanes with bis-(acetylacetonate)dichlorotitanium and showed that it leads to a synthesis of polyorganotitanosiloxanes.

2. In the initial phase the polymers obtained have properties that are typical for cyclo-linear structured polymers, and after thermal treatment they are transformed into structurized polymers.

From the Institute of Elemento-Organic Compounds, AN SSSR (Academy of Sciences, USSR)

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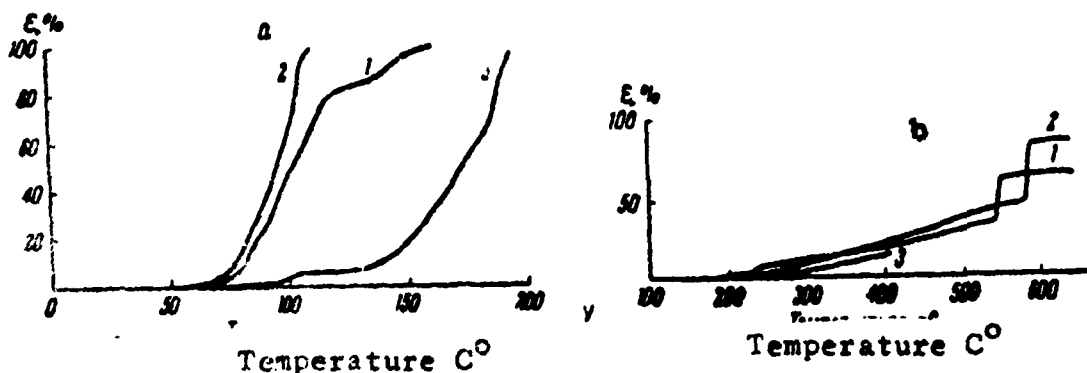


Fig. 1. Thermomechanical curves of polymers; a -- before heating; b -- after heating at 200°

- 1 = poly-bis-(acetylacetonate)titanomethylsiloxane;
- 2 = poly-bis-(acetylacetonate)titanoethylsiloxane;
- 3 = poly-bis-(acetylacetonate)titanophenylsiloxane.

С. Структурный состав полимеров

b) Реагенты	c) Структурное звено цепи полимера	d) Элементный состав, %						e) Элементарный состав, %				f) Молекулярный вес			
		C	H	N	TI	OH	C	H	N	TI					
$C_2H_4O_2 + C_2H_4TI$		31,07	5,46				7,46	5,51			30,15	4,85	23,45	8,04	
$C_2H_4O_2 + C_2H_4TI$		31,35	5,60	24,80			7,39	5,46			36,85	5,80	19,11	8,19	
$C_2H_4O_2 + C_2H_4TI$		52,89	4,74				7,44	2,01			51,77	4,46	12,94	7,39	103,000

Table 1. Legend: a) Elementary composition of polymers

b) Reagents

c) Elementary link of the polymer chain

d) % found

e) % calculated

f) Molecular weight

Frequencies for groups, cm^{-1}

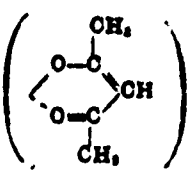
Polymers	$\text{CH}_3 - \text{Si}$	$\text{C}_6\text{H}_5 - \text{Si}$	$\text{C}_6\text{H}_5 - \text{Si} - \text{O} - \text{Si} - \text{Si}$	$\text{Ti} - \text{O} - \text{Si}$	Ti	
Poly-bis-(acetylacetonate)titanomethylsiloxane	1260	—	—	915	1370 1520 1570	
Poly-bis-(acetylacetonate)titanoethylsiloxane	—	1248	—	921	1370 1520 1570	
Poly-bis-(acetylacetonate)titanophenylsiloxane	—	—	1130	921	1370 1520 1570	

Table 2. Infra-red spectra of polymers.

a) Растворитель	b) Поли-бис-(ацетилацетонат)-титанофенилсилоксан				c) Поли-бис-(ацетилацетонат)-титаноэтилсилоксан				d) Поли-бис-(ацетилацетонат)-титанометилсилоксан			
	—	100°	160°	200°	—	100°	160°	200°	—	100°	160°	200°
	e) Продолжительность нагревания, часы											
	0	5	2	4	0	5	2	4	0	5	2	4
f) Толуол	100	100	100	79	100	100	h) Нерастворим	100	100	h) Нерастворим		
g) Ацетон	100	100	63	33	100	100	i) То же	100	100	i) То же		

Table 3. Solubility of poly-bis-(acetylacetonate)titano-organosiloxanes.

- Legend: a) Solvent
 b) Poly-bis-(acetylacetonate)titanophenylsiloxane
 c) Poly-bis-(acetylacetonate)titanoethylsiloxane
 d) Poly-bis-(acetylacetonate)titanomethylsiloxane
 e) Duration of thermal treatment in hours
 f) Toluol
 g) Acetone
 h) Insoluble
 i) The same

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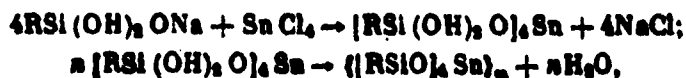
POLY TINORGANOSILOXANES

Following is the translation of an article by B. Z. Asnovich and K. A. Andrianov in the Russian language publication Vysokomolekulyarnyye Soyedineniya (High-Molecular Compounds), Vol IV, No 2, Moscow, 1962, pages 216-220/

Elemento-organic polymers with inorganic molar chains are known under the name of polymetallo-organosiloxanes and at the present time are drawing much attention. In this paper we present a study of new polymers --polytinorganosiloxanes--whose main molar chains are made up of atoms of silicone, oxygen and tin, and the silicone atoms are surrounded by methyl, ethyl and phenyl groups.

The first samples of polytinorganosiloxanes were obtained in 1956¹ by the concurrent hydrolysis of diethyl-dichlorotin with dialkyldichlorosilanes as well as with a mixture of diethyldichlorosilane and phenyltrichlorosilane with subsequent condensation of the products of cohydrolysis.

The polytinorganosiloxanes studied in this paper were obtained from a reaction of double decomposition of sodium salts of alkyl-(aryl)-trioxosilane tin tetrachloride according to the equation:



$\begin{array}{c} \text{O} \\ | \\ \text{where } \text{R} = \text{CH}_3, \text{C}_2\text{H}_5, \text{C}_6\text{H}_5. \end{array}$

By changing the ratios of the reagents, it was possible to regulate the ratios between the number of silicone and tin in the end product over a wide range and consequently to obtain polymers having different properties. By using the above-indicated reaction we obtained polytin-methylsiloxanes with a ratio between the silicone atoms

and tin atoms ranging from 4:1 to 17:1, polytinethylsiloxanes with an Si:Sn ratio from 1.25:1 to 19.5:1, and polytinphenylsiloxanes with an Si:Sn ratio from 1.17:1 to 17.4:1. Table 1 presents the results of chemical analysis of the new polytinorganosiloxanes.

Polytinmethylsiloxanes, polytinphenylsiloxanes and polytinethylsiloxanes with a ratio of silicone atoms to tin atoms in the polymer equal to 1.25, 3.98 had the appearance of colorless, glass-like, transparent, solid and brittle substances. Polytinethylsiloxanes with Si:Sn ratio equal to 7.74, 15.5 and 19.5 at room temperature look like viscous tars, light yellow in color.

Polytinmethylsiloxanes are stable in solution, however when they are distilled from the solution, even at 20-22°, they are no longer soluble in organic solvents and do not melt upon heating. Polytinethylsiloxanes and polytinphenylsiloxanes differ from polytinmethylsiloxanes in that they can be distilled from the solution with preservation of polymer solubility in organic solvents. Table 2 shows the solubility in organic solvents of polytinethylsiloxanes and polytinphenylsiloxanes of different chemical compositions.

From table 2 it can be seen that polytinethylsiloxanes and polytinphenylsiloxanes with an Si:Sn ratio of 4 or more dissolves well in most organic solvents. Polytinethylsiloxane and polytinphenylsiloxane with an Si:Sn ratio equal to 1 dissolves only in acetone. When they are heated polytinethylsiloxanes quickly lose their solubility (table 3).

From table 3 it can be seen that polytinethylsiloxane with an Si:Sn ratio equal to 4 can no longer be dissolved in acetone, benzol and carbon tetrachloride after it has been heated at 200° for a period of two hours. When this same polymer is heated at 150° for ten hours there is significant loss of solubility.

In contrast to polytinethylsiloxanes, the polytinphenylsiloxanes lose their solubility upon heating more slowly. Table 4 shows the changes in solubility of polytinphenylsiloxanes with an Si:Sn ratio equal to 4 and 17 in benzol, acetone and carbon tetrachloride depending on the different temperatures at which the polymers were heated.

Table 4 shows that polytinphenylsiloxane with an Si:Sn ratio equal to 4 loses its solubility in organic solvents after being heated at 200°, whereas polytinphenylsiloxane with an Si:Sn ratio equal to 17 loses its solubility more slowly. Thus, for instance, after being heated at 200°

for ten hours the polymer with an Si:Sn ratio of 4 is completely insoluble in benzol and acetone, and the polymer with an Si:Sn ratio of 17, on the contrary, retains its solubility in full, in the above-mentioned solvents. From table 4 it can be seen also that acetone is the best solvent for polytinphenylsiloxanes. The solubility of polytinphenylsiloxanes and polytinethylsiloxanes containing various quantities of tin in their composition decreases with increased tin content in the polymers.

Investigation of the kinematic viscosity of 5, 10 and 30% solutions of polytinethylsiloxanes and polytinphenylsiloxanes in toluol with different Si:Sn ratios showed very close results. The polymerization time for polytinethylsiloxanes and polytinphenylsiloxanes depends on their tin content. Table 5 shows the change in polymerization time of the polymers depending upon the Si:Sn ratio in the polymer.

It can be seen from Table 5 that, as the tin content in the polymers increases, the polymerization time decreases. When the Si:Sn ratio of polytinethylsiloxanes is changed from 4 to 8, the polymerization time is about 30 times greater, while a change in Si:Sn ratio from 1.25 to 4 does not change the polymerization time of the same polymers. The polymerization time of polytinphenylsiloxanes is considerably lower than that of polytinethylsiloxanes having the same Si:Sn ratio in the polymers. The polymerization time for polytinphenylsiloxanes increases sharply for polymers with an Si:Sn ratio of 13.7 or more. While polytinphenylsiloxanes with an Si:Sn ratio of 8.5 are polymerized within 35 seconds, those with an Si:Sn ratio equal to 17.4 are polymerized only in 10 minutes. The thermomechanical properties of polytinethylsiloxanes and polytinphenylsiloxanes depend on the tin content in the polymers. Thus, polytinphenylsiloxane with an Si:Sn ratio of 4 did not demonstrate flow up to 400°, but when it was plastified [?] with pentachlordiphenyl (20%) it demonstrated flow at 80-90° (figure 1, a). Polytinphenylsiloxanes with an Si:Sn ratio of 14 demonstrated flow at 130-140°. The thermomechanical properties of polytinphenylsiloxanes with various tin content show that an increase in tin content leads to an increase in hardness of polymer molecules.

The flow temperature of polytinethylsiloxanes with an Si:Sn ratio of 4 is about 80° (figure 1, b) while that of the same polymer with an Si:Sn ratio of 7.75 is 30°. For polytinethylsiloxanes with an Si:Sn ratio of 1.25 no flow is detected up to 400°. The thermomechanical properties of polytinethylsiloxanes and polytinphenylsiloxanes

with an Si:Sn ratio of 4 vary: the polytinphenylsiloxanes did not flow up to 400°, while the polytinethylsiloxanes melted at 80°. Polytinethylsiloxanes with an Si:Sn ratio of 15.5 melt at 30°, and polytinphenylsiloxanes with an Si:Sn ratio of 14 melt at 135°. These data show that the molecular structure of polytinphenylsiloxanes is harder as compared to that of polytinethylsiloxanes.

Experimental Section

The solubility of the polymers was determined by a method described earlier (2).

The polymerization time (3) of polytinethylsiloxanes and polytinphenylsiloxanes was determined on a polymerization plate at 160°.

Investigation of thermomechanical properties (4) of the polymers was conducted in accordance with the method indicated, with a specific load of 1 kilogram per centimeter square and a rate of temperature elevation of 70° per hour. As samples we used tablets made up of fragmented polymers compressed at room temperature.

Plastification of polytinphenylsiloxanes with penta-chlordiphenyl was performed according to an earlier description (5).

CONCLUSIONS

The solubility, thermomechanical properties and polymerization time of polytinmethylsiloxanes, polytinethylsiloxanes and polytinphenylsiloxanes, obtained by the double decomposition reaction, were investigated.

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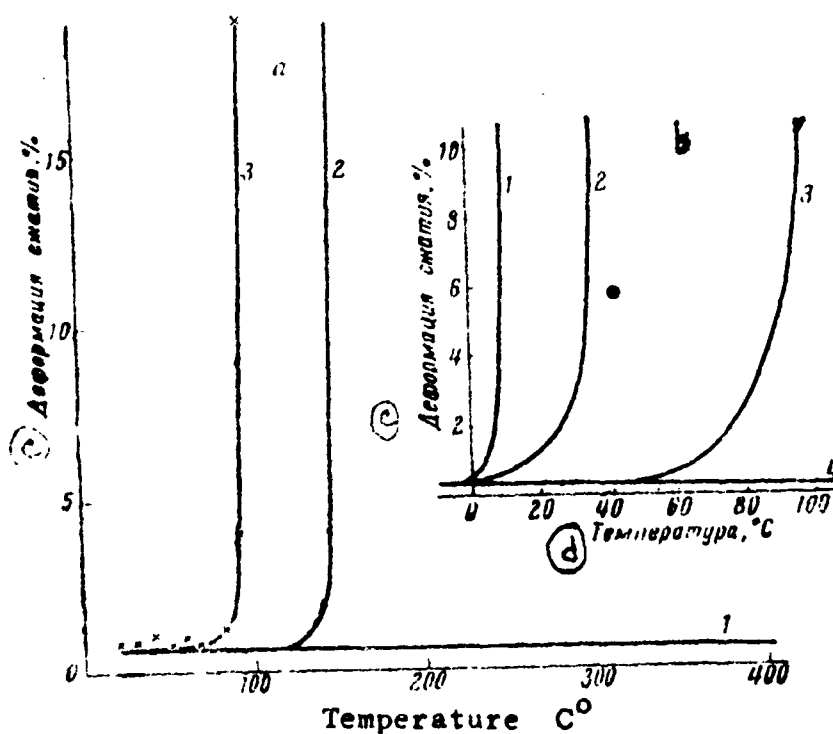


Figure 1. Thermomechanical curves.

Legend: a) Polytinphenylsiloxane, Si:Sn ratio =
 1 -- 4; 2 -- 14; 3 -- 4, plastified
 with 20% pentachlordiphenyl;
 b) Polytinethylsiloxane, Si:Sn ratio =
 1 -- 15.5; 2 -- 7.75; 3 -- 3.98;
 4 -- 1.25
 c) Compression deformation, %
 d) Temperature, C°

Полиоловометилсилосаны			Полиоловоэтилсилосаны			Полиоловофенилсилосаны		
содержание, %		Si/Sn	содержание, %		Si/Sn	содержание, %		Si/Sn
Si	Sn		Si	Sn		Si	Sn	
23,23	24,98	3,95	12,39	42,0	1,25	10,85	39,28	1,17
27,02	18,48	6,20	21,22	22,64	3,98	17,14	18,08	4,0
29,45	15,41	8,10	24,47	13,42	7,75	20,23	10,11	8,5
30,15	11,98	10,7	27,38	7,48	15,5	20,43	6,34	13,7
33,54	8,38	17,0	29,27	6,38	19,5	20,92	5,13	17,4

Table 1. Silicone and tin content in polytinorganosiloxanes

Legend: a) Polytinmethylsiloxanes
 b) Polytinethylsiloxanes
 c) Polytinphenylsiloxanes
 d) Content, %

Растворитель	Полиоловоэтилсилосаны с отноше- нием Si/Sn			Полиоловофенил- силосаны с отно- шением Si/Sn		
	1,25	3,98	19,5	1,17	4,0	17,4
Бензол	-	+	+	-	+	+
Хлорбензол	-	+	+	-	+	+
Ацетон	+	+	+	+	+	+
Эфир	-	+	+	-	+	+
Петролейный эфир	-	+	+	-	-	-
Четыреххлористый углерод	-	+	+	-	+	+

Table 2. Solubility of polytinethylsiloxanes and polytinphenylsiloxanes at 20°

Legend: a) Solvent
 b) Si:Sn ratio of polytinethylsiloxanes
 c) Si:Sn ratio of polytinphenylsiloxanes
 d) Benzol
 e) Chlorobenzol
 f) Acetone
 g) Ether
 h) Petroleum ether
 i) Carbon tetrachloride

Note: + = soluble
 - = insoluble

a) Условия нагревания полуполовинхлорсана, имеющего Si:Sn = 4		b) Растворители		
c) температура, °C	d) продолжительность, часы	e) ацетон	f) бензол	g) CCl ₄
150	10	9	6	5
200	2	3	1	1

Table 3. Solubility of polytinethylsiloxanes at 20°, %

- Legend: a) Heating conditions for polytinethylsiloxane with an Si:Sn ratio = 4
 b) Solvents
 c) Temperature, C°
 d) Duration, hours
 e) Acetone
 f) Benzol
 g) CCl₄

a) Условия нагревания полиоловофенилсилоксанов		b) Растворители					
f) температура, °C	g) продолжительность, часы	c) бензол		d) ацетон		e) четыреххлористый углерод	
		h) отношение Si:Sn в полимерах					
		4	17	4	17	4	17
150	10	97	100	100	100	44	100
200	2	2	100	14	100	0	100
200	4	0	100	5	100	0	100
200	10	0	100	0	100	0	35
300	0,25	3,5	100	25	100	1,5	32
400	0,25	0	8	0	12,5	0	5

Table 4. Solubility of polytinphenylsiloxanes at 20°, %

- Legend: a) Heating conditions for polytinphenylsiloxanes
 b) Solvents
 c) Benzol
 d) Acetone
 e) Carbon tetrachloride
 f) Temperature, C°
 g) Duration, hours
 h) Si:Sn ratio of the polymers

a) Полиметокси- силосаны с от- ношением Si : Sn	b) Время полимеризации до стадии, мин.		c) Полиметоксифенил- силосаны с от- ношением Si : Sn	d) Время полимериза- ции до стадии, мин.	
	В	С		В	С
1,25	—	1,1	1,17	—	0,17
3,98	—	1,5	4,0	—	0,27
7,75	8,33	34,0	8,5	0,42	0,58
15,50	72,25	181,25	13,7 17,4	1,0 3,0	4,33 10,0

Table 5. Polymerization time for polytinethylsiloxanes and polytinphenylsiloxanes at 160° C.

- Legend:
- a) Si:Sn ratio of polytinethylsiloxanes
 - b) Polymerization time to stage, in minutes.
 - c) Si:Sn ratio of polytinphenylsiloxanes
 - d) Polymerization time to stage, in minutes

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