

Organometallic Chemistry

Investigation of the structure of polyphenylironsiloxane

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The main features of the polyphenylironsiloxane (PPIS) structure were established by a combination of preparative fractionation, elemental analysis, gel-permeation chromatography, ^1H NMR and UV spectroscopy, and destructive trimethylsilylation. Specimens of PPIS contain a large amount of oligophenylsiloxane, which is apparently closed polycyclic oligomers. PPIS macromolecules consist of oligomeric siloxane fragments linked *via* iron oxide fragments. PPIS is characterized by the high heterogeneity of macromolecules and their fragments.

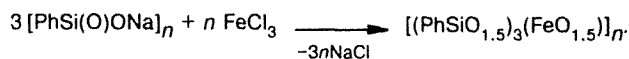
Key words: polyorganometallosiloxanes, polyphenylironsiloxane, gel-permeation chromatography.

Much attention has been given to synthesis of polyorganometallosiloxanes (POMS) beginning from the end of the 1950s, when the first representatives of compounds of this class were prepared.¹ However, the problem on the structure of POMS remained unsolved up to the beginning of the 1990s, when cage polyphenyl-metallosiloxanes (PPMS) containing bivalent transition metal atoms (nickel, manganese, cadmium, and copper) were synthesized for the first time and structurally characterized.^{2–5} These compounds were isolated in the crystalline state by the exchange reactions of sodium phenylsiloxanolate (SPS) and chlorides of the corresponding metals. Cage PPMS have sandwich-like structures, and their main structural unit is a stereoregular siloxane cycle linked to metal atoms *via* oxygen bridges.

The structure of POMS based on trivalent transition metals, which have been obtained presently as amorphous materials soluble in many organic solvents, remains unstudied. From the practical viewpoint, poly-

phenylironsiloxane (PPIS) is one of the most interesting representatives of this class. The presence of the constituent paramagnetic iron atoms is the reason for the specific interest in the magnetic properties of PPIS and makes it possible to consider it as a promising precursor for the preparation of artificial ferromagnetic materials. Therefore, considerable attention was given to the study of its magnetic parameters as a function of thermal treatment.^{6–8}

The purpose of the present work is to study the composition and structure of PPIS obtained by the exchange reaction of SPS and ferric chloride in butanol at the boiling point of the solvent by a procedure similar to the synthesis of cage PPMS:



Recent success in the study of the structure of cage POMS has been achieved^{9–12} due to the combination of

X-ray diffraction analysis, destructive trimethylsilylation, and gel-permeation chromatography (GPC), while a different approach should be developed for the study of the composition and structure of PPIS, because PPIS and the products of its destructive trimethylsilylation are unable to crystallize, which precludes their study by X-ray diffraction analysis.

To solve this problem we used a complex approach including such methods as preparative fractionation, elemental analysis, GPC, UV spectroscopy, destructive trimethylsilylation, and subsequent analysis of the decomposition products by ^1H NMR spectroscopy and GPC. This approach is necessary due to the high heterogeneity, which, as will be shown below, is inherent in the objects under study.

Two laboratory PPIS specimens and a specimen obtained on a pilot setup were studied. Specimen 1 was analyzed in detail according to the scheme presented above, and specimens 2 and 3 and the products of their destructive trimethylsilylation were analyzed by GPC. The study performed made it possible to discuss the structure of PPIS and chemical reactions resulting in the formation of these structures.

Experimental

PPIS specimen 1 was synthesized by the following procedure. Sodium hydroxide (2.40 g, 0.06 mol) was added to a solution of polyphenylsiloxane (15.0 g, 0.12 mol) in freshly distilled butanol (300 mL). The reaction mass was heated to boiling and boiled for 1 h with vigorous stirring until complete dissolution. Then metallic sodium (1.38 g, 0.06 g-at.) was introduced to the solution, which was boiled for an additional 2 h. A solution of anhydrous FeCl_3 (6.49 g, 0.04 mol) in 80 mL of butanol was added dropwise to the reaction mixture, and stirring was continued at the boiling point of butanol for 1 h. Then the solution was separated from the precipitated NaCl, and the solvent was distilled off. The yield of PPIS thus obtained was 17.80 g (95.2 %). Found (%): C, 47.50; H, 4.27; Fe, 11.11; Si, 15.50. $(\text{C}_6\text{H}_5\text{SiO}_{1.5})_{12}(\text{FeO}_{1.5})_4(\text{C}_4\text{H}_{10}\text{O})_3(\text{OH})_1$. Calculated (%): C, 47.81; H, 4.39; Fe, 10.59; Si, 15.98.

Specimen 2 was synthesized by a similar procedure but using freshly sublimed ferric chloride. Found (%): C, 48.34; H, 4.85; Fe, 8.68; Si, 14.78. $(\text{C}_6\text{H}_5\text{SiO}_{1.5})_{12}(\text{FeO}_{1.5})_4(\text{C}_4\text{H}_{10}\text{O})_5$. Calculated (%): C, 47.62; H, 4.78; Fe, 9.63; Si, 14.53.

Specimen 3 was obtained on the pilot setup by the procedure described previously.¹³ Found (%): C, 47.13; H, 4.04; Fe, 9.35; Si, 16.07. $(\text{C}_6\text{H}_5\text{SiO}_{1.5})_{10}(\text{FeO}_{1.5})_3(\text{C}_6\text{H}_6)_{1.5}(\text{H}_2\text{O})_5$. Calculated (%): C, 47.66; H, 4.00; Fe, 9.63; Si, 16.16.

Trimethylsilylation of specimens and fractions of PPIS was performed in the following way. A calculated amount of a 20 % toluene solution of Me_3SiCl corresponding to the content of iron, butanol, and water in the specimen and 10 % excess was added dropwise to a mixture of a 10 % solution of PPIS and pyridine in toluene with stirring. The amount of pyridine was equivalent to the excess of Me_3SiCl and sufficient for binding hydrogen chloride evolved. The solution was boiled for 0.5 h. After the completion of the reaction, the solution was separated from the precipitate, washed from chloride ions, dried with Na_2SO_4 , and evacuated for 1 h (100 °C/1 Torr).

Fractional precipitation was carried out in the following way. A solution of PPIS (7.5 g) in 375 mL of benzene was placed in a cylindrical vessel with a stirrer and a water jacket to maintain constant temperature. Hexane was added until a bright brown turbidity appeared, and the solution was let to stand overnight. The solution was siphoned off from the precipitate, concentrated to dryness, and redissolved for the isolation of the next fraction. The precipitate formed (fraction) was transferred with a solvent into a round-bottomed flask and concentrated on a rotary evaporator. Fraction 1 (18.6 %) was isolated at 30 °C, and fractions 2–4 (14.3, 13.5, and 13.7 %, respectively) were isolated with a gradual decrease in temperature and increase in concentration (fraction 4, 15 °C and 5 g mL⁻¹). Fractions 5 and 6 (12.8 and 27.1 %, respectively) were obtained by thermal precipitation from a solution in 2-propanol: the precipitate after isolation of fraction 4 (3 g) was dissolved upon heating in 150 mL of 2-propanol, the precipitate formed upon slow cooling to 15 °C was isolated as fraction 5, and the concentrated solution was fraction 6.

Since the compounds studied are highly soluble in benzene–hexane mixtures, the initial specimen was fractionated by extraction with hexane: hexane (30 mL) was poured on PPIS powder (1 g), and the mixture was heated to boiling. The solution obtained was filtered and concentrated *in vacuo*. The yield of the extract was 0.16 g.

Electronic absorption spectra were obtained on an HP-8452 instrument in chloroform at a concentration of 0.015 g/100 mL (for the extract, 0.02 g mL⁻¹).

GPC was performed on a Waters instrument with an M-600 pump and an M-484 UV-detector with a variable wave length on columns U-Styragel 500, 1000 Å or two columns U-Styragel Linear, using a Maxima information processing computer system.

^1H NMR spectra of 20 % solutions of specimens in CCl_4 were recorded on a Bruker R-200 SU spectrometer (200.13 MHz, SiMe_4 as internal standard).

Results and Discussion

The most general notion about the composition and structure of PPIS specimens is provided by the molecular-mass distribution (MMD) curves in Fig. 1 obtained by GPC on two U-Styragel Linear columns in THF, the values of average molecular weight in Table 1, and the elemental composition of specimens presented in the Experimental section. These data show that the maximum of MMD of laboratory specimens 1 and 2 falls in the region of oligomers; however, the specimens give long tails, which is reflected by the high values of \bar{M}_z and \bar{M}_z/\bar{M}_w . Specimen 3 obtained on a pilot setup consists of high-polymeric and oligomeric portions and is characterized by extremely high polydispersity. Particular parameters of the polymeric and oligomeric portions of the specimen are also presented in Table 1.

The study of the absorption spectra of PPIS specimens shows that all of them have a form similar to that presented in Fig. 2, *a* with weakly pronounced maxima in the range of 260–270 nm. The spectrum of polyphenylsiloxane (PPS), the product of hydrolysis of phenyltrichlorosilane, with maxima at 260, 264, and 270 nm, is presented in Fig. 2, *b* for comparison. The

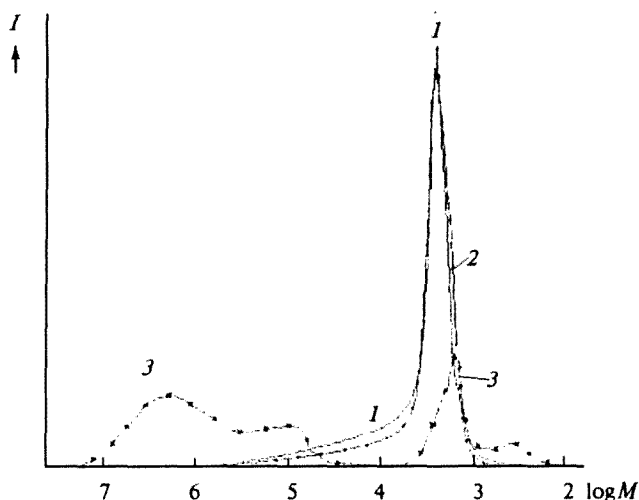


Fig. 1. MMD curves of specimens 1 (1), 2 (2), and 3 (3) obtained by GPC on U-Styragel Linear columns in THF, detection at 260 nm.

spectra presented and the MMD curves of PPIS specimens allow one to suppose that they are highly heterogeneous, which was confirmed by GPC on columns U-Styragel 1000 and 500 Å in chloroform with detection at 260 and 320 nm (Fig. 3, *a*). Specimen 1 contains a low-molecular portion (peak with retention time 16.65 min), which does not absorb at 320 nm, and the higher-molecular and more polydisperse portion of the specimen absorbs at 260 and 320 nm. It is noteworthy that in chloroform, unlike THF, the chromatograms of PPIS do not return to the base line after elution with a total volume of the column, which testifies to certain adsorption activity of the specimens in this solvent.

The results of elemental analysis and GPC of fractions of specimen 1 indicate that the fractionation occurs predominantly according to composition (see Table 2). The histogram plotted by the data of fractionation by fractional precipitation (Fig. 4) testifies to the high heterogeneity of specimen composition. Elemental analysis of the extract showed the absence of iron (limit of sensitivity of the method 1 %). The chromatogram of the extract is presented in Fig. 3, *b*. The main peak (detected at 260 nm and undetectable at 320 nm) almost coincides with the peak with a retention time of

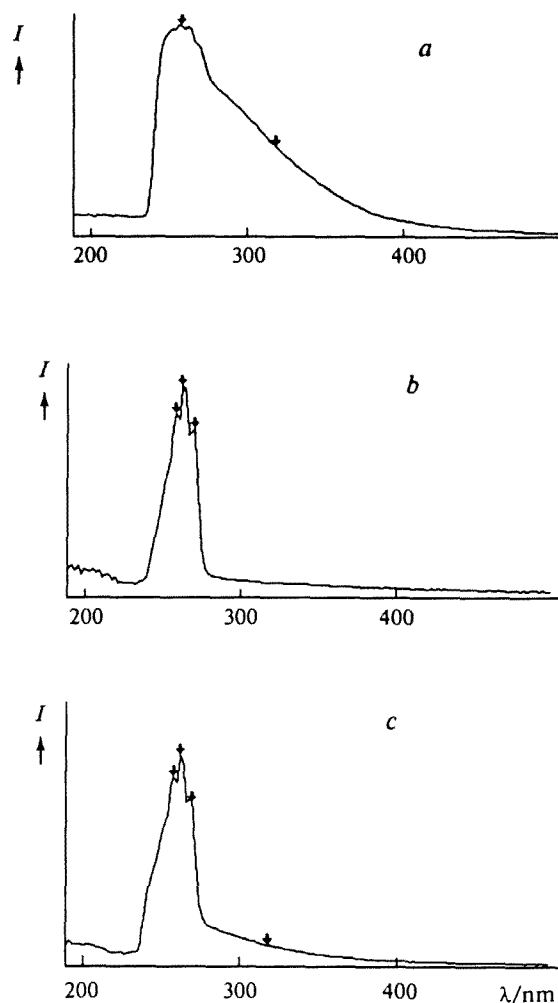


Fig. 2. UV spectra of initial specimen 1 of polyphenylironsiloxane (*a*), polyphenylsiloxane (*b*), and extract (*c*). Arrows indicate the positions of absorption maxima.

16.65 min on the chromatogram of the initial specimen. The absorption spectrum of this fraction (Fig. 2, *c*) is similar to that of polyphenylsiloxane. Thus, one may conclude that the extract and, hence, a portion of the initial PPIS corresponding to the peak of 16.65 min, is mainly oligophenylsiloxane, whose structure is similar to those of the known products of hydrolysis of phenyl-

Table 1. Molecular-mass parameters of specimens of polyphenylironsiloxane from GPC data*

Specimen	\bar{M}_n	\bar{M}_w	\bar{M}_z	\bar{M}_w/\bar{M}_n	\bar{M}_z/\bar{M}_w
1**	2670	13030	138200	4.9	10.6
2**	2220	9540	145700	4.3	15.3
3**	2440	1252800	4852800	513	3.9
3 (polymeric portion)	247380	1814600	4827500	7.3	3.9
3 (oligomeric portion)	760	1450	2210	1.9	1.5

* The data were obtained on U-Styragel Linear columns in THF, detection at 260 nm, calibration by polystyrene.

** Complete MMD.

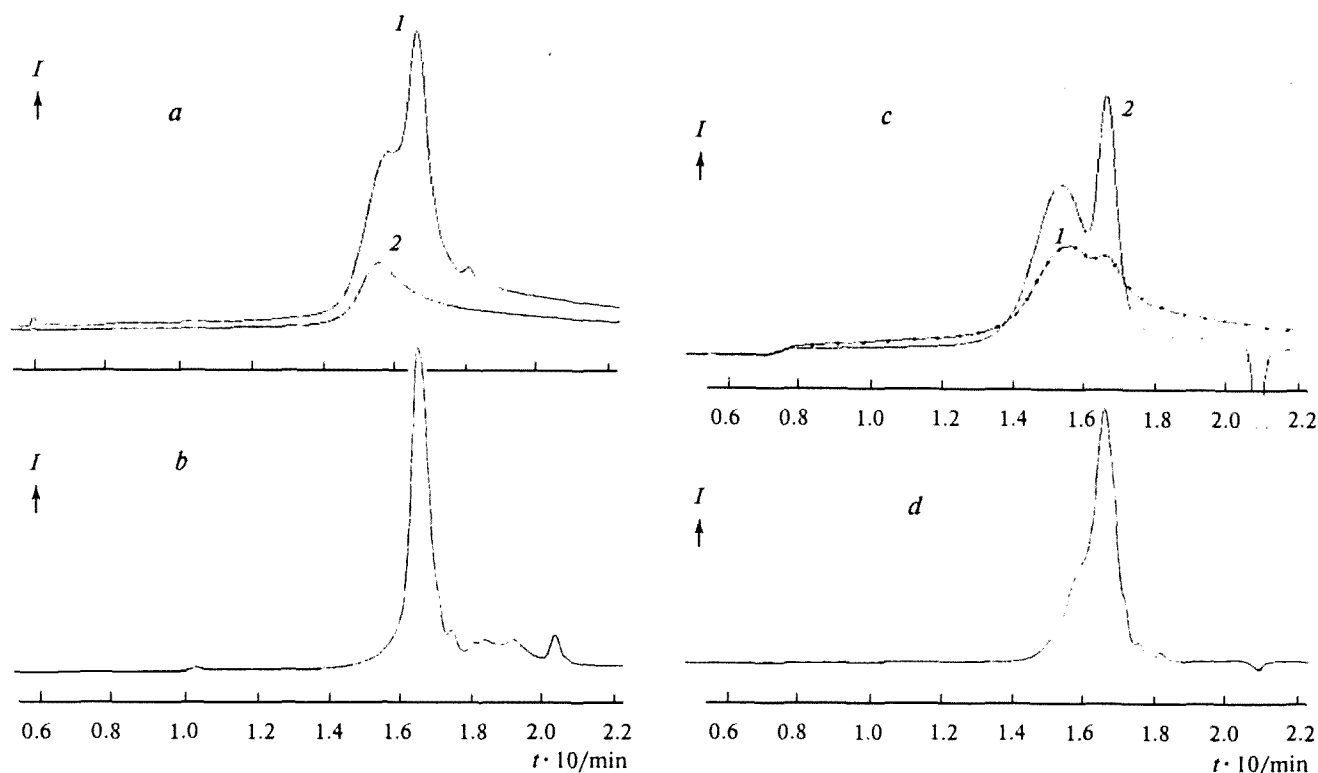


Fig. 3. *a.* GPC of specimen 1 in CHCl_3 on columns U-Styragel 1000 and 500 Å with detection at 260 (1) and 320 (2) nm. *b.* GPC of the extract, detection at 260 nm. *c.* GPC of fractions 1 (1) and 6 (2), detection at 260 nm. *d.* GPC of the trimethylsilylation product of specimen 1, detection at 260 nm.

trichlorosilane. As is seen from the chromatogram of the extract, it is not adsorbed on the column under the GPC conditions chosen, unlike iron-containing fractions.

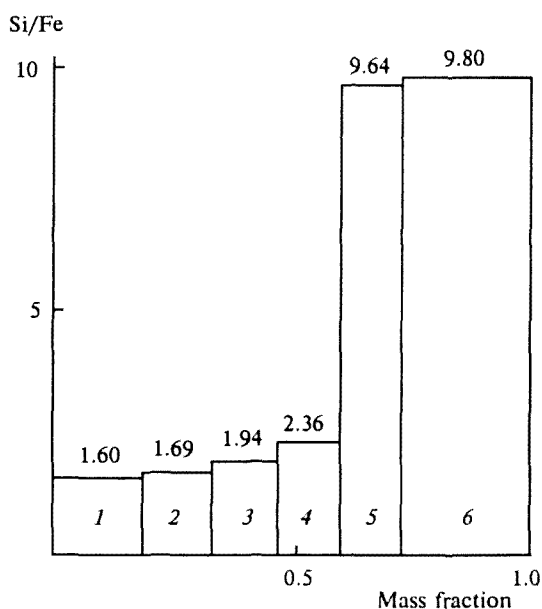


Fig. 4. Distribution histogram of specimen 1 from the data of fractionation by fractional precipitation and elemental analysis.

The GPC analysis of all isolated fractions shows that two main components, oligophenylsiloxane and polyphenylironsiloxane, are redistributed upon fractional precipitation. As can be seen from Fig. 3, *c*, fraction 1 consists predominantly of polyphenylironsiloxane (a broad peak with retention time ~ 15.80 min) with a small admixture of oligophenylsiloxane. A quantitative estimate of the content of the latter is difficult due to the strong overlap of peaks and differences in the absorption coefficients. Both of the components are pronounced on the chromatogram of fraction 6: overlapping peaks of

Table 2. Element composition of specimen 1 of polyphenylironsiloxane and its fractions and ^1H NMR data of products of their trimethylsilylation

Specimen	Fe (%)	Si (at./at.)	Si/Fe (at./at.)	$\text{SiMe}_3/\text{SiPh}$ (^1H NMR)
Initial specimen 1	11.11	15.50	2.78	0.36
Fraction 1	16.88	13.55	1.60	0.56
Fraction 2	16.63	14.10	1.69	—
Fraction 3	14.64	14.27	1.94	0.56
Extract	0.0	17.56	—	—
"Ideal" specimen $(\text{C}_6\text{H}_5\text{SiO}_{1.5})_3(\text{FeO}_{1.5})$	11.96	18.04	3.0	1.0

polyphenylironsiloxane and oligophenylsiloxane (extreme estimates for different methods of peak separation range from 18 to 42 % of oligophenylsiloxane).

A quantitative estimate of the ratio of these two components in the initial PPIS cannot be obtained directly from chromatograms or from the ratio of fractions, because none of the fractionation procedures resulted in complete separation of these components. Nevertheless, almost pure oligosiloxane was isolated by extraction, although this isolation was not complete: the residue after extraction contained a great amount of oligosiloxane. Therefore, we performed this estimate using the results of analysis of the content of Fe and Si in fraction 1 or 2 (Fe_f and Si_f), considering that these fractions consist completely of polyphenylsiloxane, which is a sufficiently good approximation [fractions 3–6 contain a considerable amount of the oligosiloxane component (peak of 16.65 min) along with polyphenylironsiloxane], in the extract ($\text{Fe}_e = 0$ and Si_e), considering it to be pure oligophenylsiloxane, and in the initial specimen (Fe_i and Si_i). The necessary data are presented in Table 2. We obtained the following equations:

$$\text{Si}_i = \text{Si}_f x + \text{Si}_e(1-x),$$

$$\text{Fe}_i = \text{Fe}_f y,$$

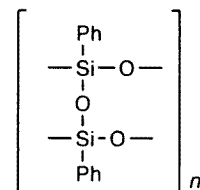
where x and y are the mass fractions of polyphenylironsiloxane in the initial specimen. Let us consider the arithmetic mean of x and y to be the desired value. Then, for fraction 1 we obtain $(x + y)/2 = 58.5 \pm 7.5$ % and for fraction 2, $(x + y)/2 = 63.5 \pm 3.5$ %.

To solve the question about the structure of each of the components of the initial PPIS, we used the destructive trimethylsilylation of the initial specimen and its fractions followed by the ^1H NMR analysis. It has been shown previously^{9,12} that the treatment of polyorganometallosiloxane with trimethylchlorosilane in the presence of pyridine allows one to isolate its cyclic organosiloxane fragments retaining their size and configuration in which a metal atom in the $\text{Si}-\text{O}-\text{M}$ group is substituted for the trimethylsilyl group. Therefore, the products of destructive trimethylsilylation of PPIS can be considered as siloxane fragments that were present in the initial specimen and bound to iron atoms by oxygen bridges, in which the latter bond is replaced by the trimethylsilyloxy group.

The chromatogram of the trimethylsilylation product of specimen 1 is presented in Fig. 3, *d*. As has been shown previously,¹² the chromatogram of the trimethylsilylation product of high-molecular specimen 3 has a similar form. The characteristic feature of these chromatograms is the shift of their polymeric portions to the region of lower molecular weights as compared to the initial PPIS. This shift is rather considerable in the case of specimen 3, and the peak of oligophenylsiloxane is almost unaltered. This change in the profile of the chromatograms after trimethylsilylation indicates that

PPIS macromolecules consist of oligomeric siloxane fragments linked *via* metal oxide bridges. The sizes of the siloxane fragments are almost independent of the overall size of PPIS macromolecules.

A quantitative idea of the size and polydispersity of the oligosiloxane component of the specimens is provided by the results of calculation of molecular-mass parameters of the extract, using calibration by phenyl(trimethylsilyloxy)cyclosiloxanes (PTC): $\bar{M}_n = 1225$, $\bar{M}_w = 2100$, $\bar{M}_w/\bar{M}_n = 1.22$, $\bar{M}_z/\bar{M}_w = 1.41$, $\bar{M}_p = 1295$ (for comparison, the molecular mass of six-membered PTC is 1260). Therefore, it can be supposed that the phenylsiloxane studied is a set of cyclized ladder fragments of the silsesquioxane type



with the most probable number of structural units $n \approx 6$. (It has preliminarily been established that the retention times of four-membered PTC and the ladder structure with the same number of silicon atoms [$\text{PhSiO}_{1.5}$]₈ are alike).

Additional data on the structure of ironsiloxane molecules can be obtained from a comparison of the elemental analysis and ^1H NMR data for the trimethylsilylation products (see Table 2). The $\text{SiMe}_3/\text{SiPh}$ ratio shows a fraction of silicon atoms in ironsiloxane linked *via* the oxygen bridge to iron. In hypothetical ideal ironsiloxane, each silicon atom should have such a bond, and this ratio should be equal to 1. For fraction 1, which, as has been mentioned above, consists mainly of ironsiloxane, this ratio is a little higher than 1/2. This can mean that in ironsiloxane itself, about 1/2–2/3 of silicon atoms are linked to iron and not less than 1/3 of them are not linked to iron.

On the other hand, it is seen from the results of elemental analysis (see Table 2) that the amount of silicon atoms in fraction 1 is insufficient to completely occupy the valences of iron (the ideal ratio is 3). Almost half of the iron valences should not be involved in $\text{Fe}-\text{O}-\text{Si}$ bonds even in the case where all silicon atoms are linked to iron. Since at least 1/3 of these atoms are not linked to iron, approximately 1/3 of the iron valences is involved in the formation of $\text{Fe}-\text{O}-\text{Si}$ bonds. The remaining 2/3 of the iron valences likely form $\text{Fe}-\text{O}-\text{Fe}$ bonds. The tendency of iron hydroxide or ferric chloride hydrolyzate to form bonds of this type is well known.¹⁴

Thus, in our opinion, the main features of the PPIS are the following: PPIS macromolecules are built of alternating polycyclic phenylsiloxane fragments similar to the oligosiloxane described above and oligomeric

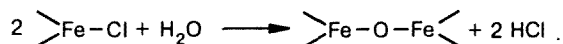
cyclic or polycyclic fragments of iron oxide that are linked by Si—O—Fe bridges. On the whole, the macromolecules can be both linear and branched with branching centers in both the siloxane and iron oxide fragments.

It should be mentioned that the polyphenylironsiloxane in the specimens studied is characterized by considerable heterogeneity in both the size of the macromolecule (which can be seen from Table 1) and the size of their fragments. The latter follows from the differences in the shapes of chromatograms of different fractions of PPIS and their SiMe₃-derivatives.

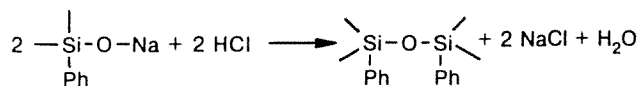
The data obtained on the structure of the specimens make it possible to suggest the type of chemical processes occurring upon the reaction of SPS with ferric chloride. If this process is considered as polycondensation, the following can be noted: the first monomer, FeCl₃, is trifunctional; and the second monomer, SPS, which most likely exists in an equilibrium between cyclic oligomeric and monomeric forms,¹⁵ can also be considered trifunctional. Formally, heavily cross-linked polymeric networks can be expected in this case at a high degree of conversion. However, if we turn to the synthesis of cage organometallosiloxanes involving bivalent transition metal chlorides,^{2–5} where the average functionality is also greater than 2 and, from the formal viewpoint the formation of polymeric networks should be expected, cyclic sandwich-like molecules are in fact mainly formed.

This specific feature results from two tendencies: (1) the high tendency of the siloxane chain to form cycles, which increases as the geometric size of the substituents at the silicon atom increases and (2) high coordination ability of transition metal ions, due to which they insert between siloxane rings as metal oxide layers, controlling the sizes of these rings and stabilizing the cage structure due to additional coordination bonds with the oxygen atoms.

In the case of the reaction of SPS with ferric chloride, the first tendency is undoubtedly manifested. The second tendency should also be observed, but under real conditions the third tendency to the formation of Fe—O—Fe chains competes with the second tendency and disables a portion of the iron valences, disturbing the regularity of its binding with siloxane fragments. This reaction is possible under the action of air or reaction medium moisture:



Hydrogen chloride evolved in this reaction, in its turn, causes hydrolysis and condensation of silanolate groups.



Therefore, silicon atoms nonlinked with iron appear, and the degree of involvement of iron valences in binding with silicon decreases. Purely siloxane fractions and fragments of polymeric iron oxide are formed in PPIS with considerable development of processes of separate condensation. The differences in molecular weights and molecular heterogeneities of the specimens studied confirm the following conclusion: the most heterogeneous specimen 3 with the highest molecular weight was obtained under conditions favorable for the hydrolytic decomposition of iron chloride by air and reaction medium moisture.

Data indicating the existence of bridge Fe—O—Fe bonds in PPIS have been obtained previously in the study of their magnetic properties.⁶

This work was financially supported by the Russian Foundation for Basic Research (Project No. 93-03-18121), the International Science Foundation (Grant JK 5100), and the International Science Technical Center (Project No. 015-94).

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Received March 13, 1995;
in revised form November 28, 1995