

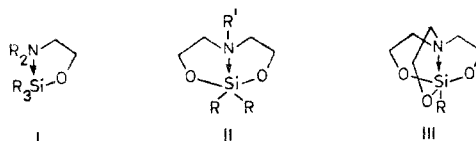
SILOXAZOLIDINES WITH A PENTACOVALENT SILICON ATOM (REVIEW)

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The literature data and the results of the authors' research on heterocyclic systems containing a pentavalent silicon atom in a siloxazolidine ring are examined. Particular attention is directed to silatranes – stable organic derivatives of pentavalent silicon that have high physiological activity.

The unshared electron pair of the nitrogen atom in organosilicon derivatives of mono-, di-, and triethanolamine may react with the vacant 3d orbitals of the silicon atom to give mono- (I), bi- (II), and tricyclic (III) siloxazolidines containing a pentavalent silicon atom:

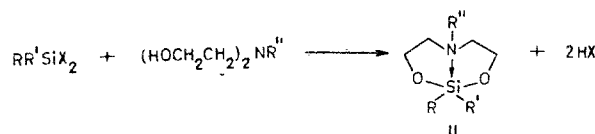


This sort of reaction has not yet been observed in the case of monoethanolamine derivatives. Depending on the nature of the substituents attached to the silicon atom, diethanolamine derivatives may exist in both bicyclic siloxazolidine form II and in the form of the isomeric monocyclic 2-sila-1,3-dioxo-6-azacyclooctanes (exo form). An equilibrium mixture of these forms is sometimes produced in solutions. On the other hand, only tricyclic siloxazolidines III (silatranes) are known for triethanolamine derivatives. The heightened interest in silatranes is due to their peculiar structure and high biological activity. Individual problems of the chemistry of silatranes have already been illuminated in a number of reviews [1-6], most of which by now have become appreciably obsolete.

The results of research on all groups of siloxazolidine derivatives of pentavalent silicon derivatives published up until July 1976 are correlated in the present review.

Bicyclic Siloxazolidines

Bicyclic siloxazolidines with a pentavalent silicon atom (II) can be obtained by reaction of diethanolamine derivatives with dialkoxysilanes [7-12] or dialkylbis(dialkylamino)silanes [13]:

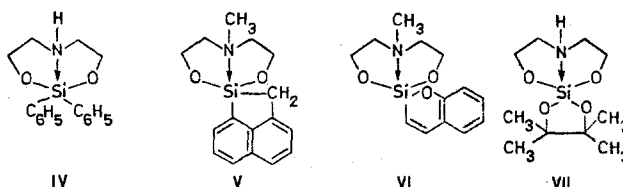


The degree of reaction between the nitrogen and silicon atoms in compounds of the II type depends on the nature of the groups attached to the silicon atom and steric factors.

The experimental values of the dipole moments of cyclic derivatives II (R, R' = alkyl) exceed the dipole moments calculated for fixed "boat" and "crown" conformations by only 0.5-1.0 D [14]. This fact, in the opinion of Mazheika and co-workers [14], indicates "the absence of a distinctly expressed donor-acceptor bond between the nitrogen and silicon atoms in these compounds."

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When electron-acceptor substituents are introduced at the silicon atom [15] or in the case of silaspirocyclic compounds [16, 17], one observes the formation of an N → Si coordinate bond, which leads to the formation of a siloxazolidine ring. It has been shown by x-ray diffraction analysis of 1,1-diphenyl-1-sila-2,8-dioxo-5-azabicyclo[3.3.0^{1,5}]octane (IV) [15] and spirocyclic derivatives of acenaphthene (V) [16] and silachromene (VI) [17] that as a result of the formation of an N → Si donor-acceptor bond (2.301 Å in IV, 2.263 Å in V, and 2.297 Å in VI) the coordination number of the silicon atom increases to five, and the coordination polyhedron is a distorted trigonal bipyramid.



The reaction of pinacol, tetraethoxysilane, and diethanolamine gave spirocyclic compound VII, to which a structure with an N → Si coordinate bond was also assigned [18].

Tricyclic Siloxazolidines (Silatranes)

Siloxazolidines III [19], which contain three condensed oxazolidine rings, are complete organosilicon chelate ethers of triethanolamine. For convenience, the shorter names organo(2,2',2''-aminotriethoxy)silanes [20, 21], organo(2,2',2''-nitrilotriethoxy)silanes [22], and silatranes [20] have been proposed in place of their full names - 2,8,9-trioxa-5-aza-1-silatricyclo[3.3.3.0^{1,5}]undecane (III, R = H). The name "silatrane" and the symbol $\text{N}(\text{CH}_2\text{CH}_2\text{O})_3\text{Si}$ have been firmly incorporated in the world literature.

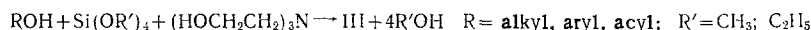
Preparative Methods

Transesterification of Alkoxysilanes with Triethanolamine. The most widely used method for the preparation of 1-organylsilatranes is the reaction of triethanolamine with alkoxysilanes [7, 8, 19, 23-53]. This method has been employed for the preparation of alkyl- [7, 8, 19, 23-28], aryl- [7, 8, 19, 34], alkoxy- [7, 8, 19, 24, 29], and heterylsilatranes [30, 32, 33, 53], as well as their carbo functional derivatives containing halogen [46, 47, 49, 50], sulfur [35-37, 43, 45, 48, 50], nitrogen [24, 26, 28, 30, 31], and phosphorus [51, 52] in the organic portion.

Tetraethoxysilane undergoes this reaction to give 1-ethoxysilatrane [29], but subsequent aminoalcoholysis to give tris(silatranxyloxyethyl)amine occurs when the reagent ratio is changed and the reaction temperature is raised to 300°C [54].

Siloxanes containing terminal silatrane groupings have been obtained by transesterification of α, ω -bis-(trialkoxy)siloxanes $(\text{RO})_3\text{SiO}[\text{R}_2\text{SiO}]_n\text{Si}(\text{OR})_3$ with triethanolamine [54].

A more convenient method has been developed for the preparation of 1-organoxy- [55-57] and 1-acyl-oxysilatranes [55]; this method involves transesterification of lower tetraalkoxysilanes with an equimolar mixture of triethanolamine and the appropriate hydroxyl-containing compound (ROH):



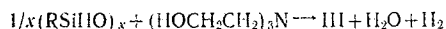
In the case of alkanols the process is accelerated by alkali metal hydroxides. Some 1-alkoxysilatranes [R = CH(CH₃)₂, C(CH₃)₃] are not formed at all in the absence of alkali. The effect of the catalyst is less substantial in the preparation of 1-alkoxysilatranes.

Reaction of Siloxanes and Siloxanols with Triethanolamine. 1-Organylsilatranes are also obtained by the reaction of polyorganylsilsesquioxanes $(\text{RSiO}_{1.5})_x$ and polyorganylsiloxanols $[\text{RSiO}_{1.5y}(\text{OH})_{2y}]_x$ with triethanolamine [1-4, 20, 55, 58, 59].

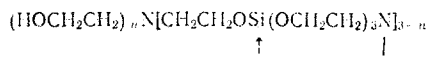
The reaction is carried out in the presence of catalytic amounts of potassium hydroxide in an inert solvent with removal of the water by azeotropic distillation. In this case the Si-O-Si bonds in the starting siloxanes are cleaved:



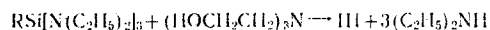
The reaction rate increases as the degree of condensation of the starting siloxanes decreases and on passing from alkyl- to aryl- and vinylpolysiloxanols. Polyorganylhydrosiloxanes of the $(\text{RSiHO})_x$ or $\text{HO}(\text{RSiHO})_x$ type can also be used in this reaction [20, 55]. In this case dehydrocondensation with hydrogen evolution takes place in the absence of a catalyst, and the addition of alkali is necessary for cleavage of the siloxane bond.



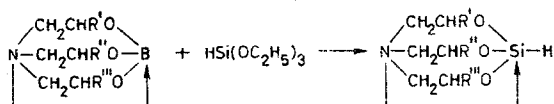
A product containing a silatrane grouping has been obtained by reaction of SiO_2 with excess $\text{N}(\text{CH}_2\text{CH}_2\text{OH})_3$ [60]:



Reaction of Aminosilanes with Triethanolamine. Silatranes are also obtained from organyltris(diethylamino)silanes and triethanolamine with simultaneous removal of the resulting diethylamine by distillation [13]:



Other Methods. 1-Hydrosilatranes, which are extremely difficult to obtain on the basis of the reaction of triethoxysilane with trialkanolamines (because of cleavage of the Si-H bond), have been synthesized by transesterification of triethoxysilane with the appropriate easily accessible boratranes [55, 61, 62]:



1-(3'-Nitrophenyl)silatrane was obtained by reaction of tris[2-(trimethylsiloxy)ethyl]amine with 3-nitrophenyltrifluorosilane [60].

Physical Properties

Dipole Moments. The most convincing confirmations of the existence of a donor-acceptor transannular bond in silatranes have been obtained by measurement of their dipole moments [55, 63-67]. The formation of a coordinate bond is characterized by deviations of the dipole moments from additivity on the order of several Debye units. A comparison of the experimental and vectorially calculated dipole moments therefore serves as a criterion for the presence of bonds of this type. According to recently published data [67], the dipole moment of the $\text{Si}(\text{OCH}_2\text{CH}_2)_3\text{N}$ heterocyclic framework is ~ 3 D (directed from N to Si). The dipole moment of the $\text{N} \rightarrow \text{Si}$ bond has been estimated to be equal to 2.2 D, which is in agreement with charge transfer from the nitrogen atom to the silicon atom (0.2 e). These results compel us to review the concept of the exceptionally high polarity of the $\text{N} \rightarrow \text{Si}$ bond in silatranes.

IR Spectra. Analysis of the IR spectra of hydro-, organyl-, and organoxysilatranes [19, 55, 68-71] has shown that the presence of an $\text{N} \rightarrow \text{Si}$ bond in them does not have a substantial effect on the vibrational frequencies of the bonds of the central silicon atom and the frequencies of the internal vibrations of the substituents bonded to this atom. The frequency of the Si-H stretching vibrations is not found in the region characteristic for ordinary organosilicon compounds with an $\text{HSi}(\text{O})_3$ grouping but is shifted to the lower-frequency region only in the case of 1-hydrosilatrane [19]. The band at $568\text{-}590\text{ cm}^{-1}$ has been tentatively assigned to the stretching vibration of the $\text{N} \rightarrow \text{Si}$ coordinate bond [55, 68-70]. However, according to recent data [71], it is most likely related to the skeletal vibration of the silatrane skeleton.

UV Spectra [72, 73]. The formation of an $\text{N} \rightarrow \text{Si}$ donor-acceptor bond in silatranes III increases the energy of the unshared electron pair of the nitrogen atom. This leads to a shift of the band of the amino group to the short-wave region as compared with the spectrum of triethylamine, which contains a nitrogen atom with a free unshared electron pair.

NMR and Nuclear Quadrupole Resonance (NQR) Spectra. Valuable information regarding the electronic and three-dimensional structures of silatranes has been obtained by means of PMR spectroscopy [55, 74-81]. According to the PMR data, the Si-O-C-C-N rings in the III molecules are nonplanar and extremely labile.

The electronic effects of the substituent attached to the silicon atom are transmitted to the nitrogen atom mainly through the transannular N → Si bond, since a change in the nature of R gives rise to approximately identical changes in the τ_{OCH_2} and τ_{NCH_2} values. The difference in the chemical shifts of the CH₂N protons in silatranes and some of their analogs (linear and cyclic organosilicon ethers of diethanolamine), in the molecules of which the N → Si bond is absent, is small. This is probably explained by incomplete transfer of the unshared electron pair of the nitrogen atom to the vacant 3d orbital of the silicon atom, in connection with which the latter has hybridization intermediate between sp³ and sp³d in silatranes.

The ³⁵Cl NQR frequencies of 1-(chloromethyl)-, 1-(dichloromethyl)-, and 1-(α -chlorovinyl)silatranes, which are lowered considerably as compared with the ³⁵Cl NQR frequencies of the corresponding haloalkyl-triethoxysilanes [82], are in agreement with the presence of N → Si coordination in their molecules. The increase in the $\Delta\delta_{\text{F}}$ values (the differences in the ¹⁹F chemical shifts in the spectra of fluorosilanes and fluorobenzene) in the case of 4-fluorophenyl- [83] and 3- and 4-fluorobenzylsilatranes [84] as compared with the starting alkoxysilanes has served as another piece of evidence for the tricyclic structure of silatranes.

X-Ray Spectra. Silatranes have been subjected to x-ray spectral studies [85–89]. The proposed shift in the SiK α line in the spectra disregarding the N → Si bond is 0.47 eV. An effective charge of 0.81 atomic unit (au) on the Si atom corresponds to this shift. However, the experimentally determined chemical shifts range from 0.27 to 0.37 eV, and the charge on Si ranges from 0.46 to 0.65 au [87]. This result serves as another confirmation of the presence of an N → Si bond in silatranes III.

X-Ray Diffraction Analysis. The direct study of the molecular and crystal structures of silatranes by x-ray diffraction analysis is of particular interest [90–100]. The determination of the molecular structures of 1-phenyl- [22, 91, 92], 1-(*m*-nitrophenyl)- [93], and 1-chloromethylsilatrane [94] has shown that the atrane framework is composed of three condensed siloxazolidine heterorings with a common transannular nitrogen–silicon donor–acceptor bond. The coordination polyhedron about the silicon atom is a distorted trigonal bipyramid due to the sp³d hybridization of the valence orbitals of the central silicon atom that arises as a result of partial transfer of the unshared pair of electrons of the nitrogen atom to the 3d orbitals of the silicon atom. Three oxygen atoms are situated in the equatorial plane of the trigonal bipyramid, and the nitrogen atom and the carbon atom of substituent R are found at its apexes. 1-Phenylsilatrane has several crystal modifications (α , β , and γ), the existence of which is explained by a change in the conformation of the siloxazolidine rings. The nitrogen–silicon interatomic distance in the α , β , and γ forms of 1-phenylsilatrane are 2.193 Å [90, 91], 2.156 Å [91], and 2.132 Å [92], respectively, as compared with 2.116 Å in the *m*-nitrophenyl derivative [93] and 2.12 Å in the chloromethyl derivative [94].

Other Methods. The mass spectra of some 1-organylsilatranes are presented in [101, 102]. It has been noted [102] that the character of the fragmentation of the ethyl derivatives of silatrane, germatrane, and stannatrane is similar. However, the mass spectra of silatranes have not been studied systematically.

After the realization of the conformational analysis of the 1-methylsilatrane molecule [103, 104], it was concluded that there is a considerable (~ 8.5 kcal/mole) gain in energy when the hybridization of the silicon atom changes from sp³ to sp³d in the endo form (the unshared pair of the nitrogen atom is directed toward the heteroring in the endo form).

Chemical Properties

Complexing. Complexes of silatranes with TiCl₄ and AlBr₃ have been obtained [105]. On the basis of an analysis of their IR spectra it was assumed that the TiCl₄ and AlBr₃ are coordinated with the oxygen atoms. A study of the isotherms of the dielectric losses of the C₆H₆–CHCl₃–RSi(OCH₂CH₂)₂N and C₆H₆–CHCl₃–CH₃Si(OC₂H₅)₃ systems showed that chloroform forms with silatranes III an intermolecular hydrogen bond, in the formation of which the nitrogen atom of silatranes does not participate [106]. Charge transfer complexes of 1-organylsilatranes with tetracyanoethylene have been obtained [84, 107].

Reactions of Functional Groups Bonded to the Silicon Atom. The hydrosilylation of vinylsilatrane with polyhydrosiloxanes [108] and furylhydrosilanes [109] has been realized in benzene solution in the presence of the H₂PtCl₆·H₂O catalyst. The Si–H bond in 1-hydrosilatrane is readily halogenated with free halogens [60, 110], hydrogen halides [60], and N-halosuccinimides [60]. 1-Hydrosilatrane undergoes catalytic dehydrocondensation with organic compounds that contain a labile hydrogen (alcohols [111], phenols [111], carboxylic acids [112], and monosaccharides [39]).

Compounds with two silatrane groupings – α,ω -bis(1'-silatranyloxy)alkanes – have been obtained by reaction of alkanediols with 1-hydro- and 1-ethoxysilatrane [113, 114].

The corresponding 1-halosilatrane have been obtained by reaction of alkoxy-silatrane with HF [60], as well as POX_3 (X = Cl, Br, I), SOCl_2 , and CH_3POF_2 [115].

1-Haloalkylsilatrane react with complete esters of phosphorous acid to give 1-(O,O-dialkylphosphono-alkyl)silatrane [51, 52, 116].

It has been shown that 1-hydroxysilatrane (III, R = OH), which is obtained by hydrolysis of tris(silatranyloxyethyl)amine, undergoes the reactions that are characteristic for silanols [60].

2-Chloro-3-[γ -(1'-silatranyl)propylamino]benzo[b]thiophene 1,1-dioxide has been obtained by reaction of 1-(γ -aminopropyl)silatrane with 2,3-dichlorobenzo[b]thiophene 1,1-dioxide [117].

Aminoalkylsilatrane form methiodides [30].

Reactions with Disruption of the Atrane Structure. The kinetics of the hydrolysis of silatrane in neutral [118] and acidic [119, 120] aqueous media have been investigated. The hydrolysis of silatrane in neutral [118] aqueous media is a first-order reaction, whereas a second-order reaction is observed in acidic media [119]. The hydrolysis of benzyl- and arylsilatrane with an aqueous methanol solution of HCl is described by a first-order equation (with respect to both the silatrane and HCl) [120]. The rate-determining step includes protonation of the nitrogen atom and is accompanied by cleavage of the N → Si bond.

The reaction of 1-organylsilatrane with heavy metal salts in aqueous media in the presence of the F⁻ anion leads to disruption of the silatrane structure and the formation of an organometallic compound [101, 121-124].

The reaction of 1-organylsilatrane with alkanols leads to the corresponding organyltrialkoxysilane [125].

Biological Properties

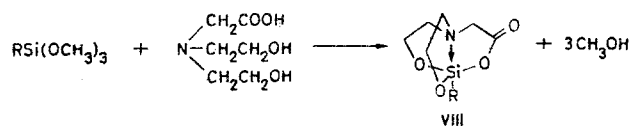
Silatrane have high and specific physiological activity [1-5, 55, 126-161]. Thus 1-phenylsilatrane is usually poisonous to warm-blooded animals: in the case of white mice its median lethal dose (LD_{50}) is 0.33 mg/kg.

On the whole, the toxicity of silatrane varies over extremely wide limits and is determined primarily by the nature of the substituent bonded to the silicon atom. Some silatrane (III, R = CH_3 , $\text{CH}_2 = \text{CH}$) have an anesthetizing and tranquilizing effect, lower the blood pressure, and have an effect on hexobarbital-induced narcosis (in experiments with rate) [136]. 1-(Carboalkoxyphenoxy)silatrane have bactericidal action [151].

A number of carbofunctional aliphatic derivatives of silatrane intensify the biosynthesis of protein by facilitating the growth and regeneration of connective and epithelial tissues, wool, and hair and prevent the development of atherosclerosis in animals [152-157]. Individual silatrane have a tranquilizing effect [158]. Fluoroalkylsilatrane have a pronounced sedative effect [159]. However, an increase in the number of fluorine atoms in substituent R sharply increases the toxicity [159, 160]. The introduction of a silatrane grouping in the indolyacetic acid molecule prolongs its auxinic effect [161]. Some silatrane have been suggested for application in agriculture as agents for raising the productivity of chickens and silkworms [127, 128, 149].

Oxosilatrane

Derivatives of a new heterocyclic system of pentavalent silicon – 2,8,9,-trioxa-5-aza-1-silatricyclo-[3.3.3.0^{1,5}]undecan-3-ones (silatranones) – have been obtained by reaction of organyltrimethoxysilane with N,N-bis(2-hydroxyethyl)aminoacetic acid [162, 163].

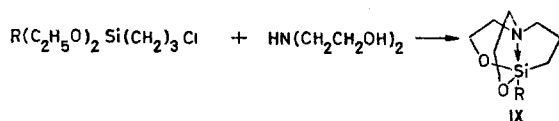


The structure of the latter was proved by IR and PMR spectra and by potentiometric titration.

It has been reported in a dissertation [164] that the corresponding silatran-3,7,10-trione can be obtained by reaction of alkyltriacetoxysilane with nitrilotriacetic acid.

Carbasilatrane s.

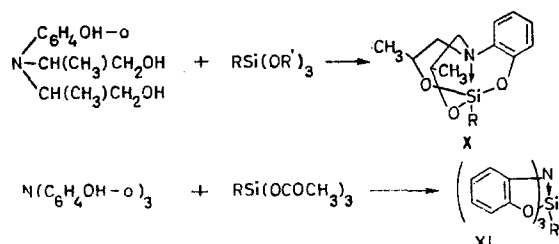
Silicoalkylation of the amino group and intramolecular transesterification to give 1-organyl-2-carbasilatrane s occur in the reaction of organyl(3-chloropropyl)diethoxysilane with diethanolamine [165-167]:



The latter are also obtained from alkyl(3-aminopropyl)diethoxysilanes and oxiranes [167, 168]. Studies of the dipole moments [14, 167] and PMR spectra [76, 77] of carbasilatrane s IX have shown that the N → Si bond in them is weaker than in silatrane s III. This conclusion is also confirmed by an x-ray diffraction study of 1-methyl-2-carbasilatrane (IX, R = CH₃), the N → Si distance in which is 2.336 Å [169].

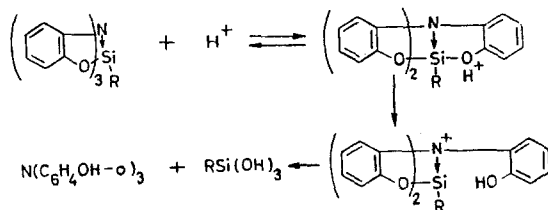
Benzosilatrane s

Aromatic analogs of silatrane s III – mono- (X) [170] and tribenzosilatrane s (XI) [171] – have been synthesized via the scheme



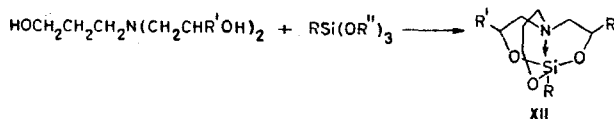
According to the results of x-ray diffraction analysis, phenylbenzosilatrane (XI, R = C₆H₅) has the configuration of a distorted trigonal bipyramid [172] in which the C–Si–O angle of 100° lies approximately halfway between 90° (in the ideal trigonal bipyramid) and 109.5° (in the tetrahedron). The N → Si distance is 2.344 Å, i.e., 0.5 Å greater than the length of the normal nitrogen–silicon bond.

A study of the hydrolysis of some tribenzosilatrane s with a mixture of dioxane and water in the presence of HCl showed that the rate-determining step is cleavage of the Si–O bond [173], which leads to a cation:



Analog s of Silatrane s with an Expanded Ring

Representatives of a heterocyclic system that contains one six-membered perhydrosiloxazine ring and two five-membered siloxazolidine rings are known [174-177]. We first obtained compounds of this type – 2,9,10-trioxa-6-aza-1-silatricyclo[4.3.3.0^{1,6}]dodecane s (3-homosilatrane s) – by the reaction of organyltrialkoxysilane s with 3-hydroxypropylbis(2-hydroxyethyl)amine or 3-hydroxypropylbis(2-hydroxypropyl)amine [174, 176, 177]:



1-Aroxy derivatives were obtained by transesterification of tetramethoxy- or tetraethoxysilane with an equimolar mixture of tris(hydroxyalkyl)amine and the corresponding phenol (naphthol) [174].

A study of the dipole moments [175] and PMR spectra [176] of XII led to the conclusion that the strengths of the intramolecular nitrogen–silicon bonds in them and silatrane s III are approximately identical.

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