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ORGANOALKOXYCYCLOSILAZANES

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Organocyclosilazanes, representing independent interest, are also convenient models of compounds for studying the structural peculiarities of organosilicon heterocycles. By varying the substituents attached to the silicon atom, with a different value of the electronegativity and a variable size, it becomes possible to vary within certain limits the molecular geometry of these compounds and the distribution of the electron density in them. The presence of alkoxy groups in the organocyclosilazanes should also facilitate the formation of hydrogen bonds between the hydrogen attached to the nitrogen and the oxygen of the alkoxy group, which in turn can exert an effect on the structure of these cycles. A convenient method for the preparation of organoalkoxycyclosilazanes is the ammonolysis of the corresponding organoalkoxydichlorosilanes, which reaction has not been studied for these compounds. As the starting organoalkoxydichlorosilanes we synthesized phenylmethoxydichlorosilane, phenylethoxydichlorosilane and methylethoxydichlorosilane. The ammonolysis of the first two compounds leads to the formation of a mixture of products, from which phenyldialkoxyaminosilanes and phenylalkoxycyclosilazanes were isolated (Table 1).

The infrared and NMR spectra were taken of the latter, which confirmed the structure of these compounds. Thus, absorption bands at 1550 cm⁻¹, corresponding to the deformation vibrations of a primary amino group, are found in the infrared spectra of the phenyldialkoxyaminosilanes. This band is absent in the case of the phenylalkoxycyclosilazanes. The absorption band, characteristic for the stretching vibrations of the NH bond in the NH₂ group, is split into two: 3380 and 3470 cm⁻¹ for phenyldimethoxyaminosilane, and 3390 and 3480 cm⁻¹ for phenyldiethoxyaminosilane. For them the band of the Si-N bond is shifted toward longer wavelengths and appears around 880-900 cm⁻¹. The ratio of the integral intensities of the protons of the different groups in the NMR spectra is in good agreement with the proposed formulas.

The formation of cyclic phenylalkoxysilazanes during the ammonolysis process apparently goes in the same manner as described earlier for the difunctional organochlorosilanes [1, 2]. However, the

	bp, °C (1 mm of Hg)	mp, °C	n ²⁰ _D	d_{4}^{20}	Found		Calculated		
Formula					mol. wt.	MR	mol. wt.	MR	Yield %
C _e H ₅ Si(OCH ₃) ₂ NH ₂	66-68		1,4980	1.0811	182	49.65	183.2	49.67	14
CeH ₅ SiNH OCH ₃],	250—252	109— 113 *	-	— [·]	443		453.6		8
$C_{\mathfrak{s}}H_{\mathfrak{s}}Si(OC_{\mathfrak{s}}H_{\mathfrak{s}})_{\mathfrak{s}}$	8789		1.4840	1.0248	200	59.07	211.3	58,93	10
$\begin{bmatrix} \mathbf{C}_{6}\mathbf{H}_{5}\mathbf{S}\mathbf{i}\mathbf{N}\mathbf{H}\\ 0\\ 0\mathbf{C}_{2}\mathbf{H}_{5}\end{bmatrix}_{3}$	256—259	77—78			481		495		17
$\begin{bmatrix} C_{6}H_{5}SiNH \\ I \\ OC_{2}H_{5} \end{bmatrix}_{4}$	289—292	-	1.5485	_	667		661		11
CH,SINH OC_H _s],	—	100—101 112—112.5	—	-	311		309.5		39 †

TABLE 1	
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*Mixture of isomers. †The total yield for the two isomers is given. UDC 542.91+546.287

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TABLE 2

Formula	ν _s (SiNSi), cm ⁻¹	v _{as} (SiNSi), cm ⁻¹	φ, deg	f, Mdyn per A	v(multipli- city of bond)
$[(C_2H_5)_2SiNH]_3 \\ [CH_3C_2H_5SiNH]_3 \\ [(CH_3)_2SiNH]_3 \\ [CH_3C_6H_5SiNH]_3 \\ [CH_3C_8H_5SiNH]_3 \\ [CH_3C_8H_3C]_2SiNH]_3 \\ [CH_3C_8H_3C]_2SiNH]_3 \\ [CH_3C_2H_3CSiNH]_3 \\ [C_4H_5C_4H_5C]_SiNH]_3 \\ [C_6H_5C_4H_5CSiNH]_3 \\ [C_6H_5CH_3OSiNH]_3 \\ [C_8H_5CH_3OSiNH]_3 \\ [C_8H_5CH_3OSiN$	597	928	123.1	3.75	1.10
	608	925	121.9	3.80	1.11
	618	928	120.9	3.85	1.12
	650	930	117.2	3.99	1.15
	680	945	115.0	4.20	1.19
	655	944	117.7	4.10	1.17
	652	960	119.4	4.17	1.21
	680	960	116.2	4.29	1.21
	680	980	114.8	4.41	1.24

phenyldialkoxyaminosilanes, obtained in comparatively high yield (see Table 1), are the result of the following possible reaction



After the fractional distillation of the ammonolysis products there remains in the still pot a brittle glassy polymer, which is soluble in organic solvents and, based on the elemental composition, approximates the structure of polyphenylsilsesquiazanes. To verify the theoretical possibility of the above given exchange reaction we subjected the phenyldialkoxyaminosilanes to heating, and it actually proved that, together with condensation of the aminosilanes, there occurs further exchange of the amino group by alkoxy with the formation of phenyltrialkoxysilanes. The high yield of the latter indicates the predominant character of the exchange reaction.

The ammonolysis of methylethoxydichlorosilane leads to obtaining cyclic and linear products. Here methyldiethoxyaminosilane was not isolated, while the yield of the cyclic products proved to be higher than in the first two cases. This can be explained by the fact that the ratio in the rates of the condensation reaction of the free amino groups and the exchange reaction, which is also not excluded in the given case, is much higher for the methylalkoxyaminosilanes than for the phenylalkoxyaminosilanes. As a result, the amount of cyclic products in the reaction mixture increases, while the products of the exchange reaction undergo condensation to methylalkoxydisilazanes and trisilazanes.

The structural features and the reactivity of the synthesized organoalkoxycyclosilazanes should be determined to a large degree by the influence of the alkoxy group on the Si-N linkage. This influence is observed in the shift of the frequencies of the stretching vibrations of this bond. The limiting shift of the frequencies of the symmetric stretching vibrations of the Si-N bond when compared with the ordinary dialkyl- and alkylarylcyclosilazanes testifies to the strong inductive effect of electronegative groups on the distribution of the electron density in the silazane ring (Table 2). The values given in Table 2 for the force constants f and the multiplicities of the silazane bonds, obtained using equations that link these values with the frequencies of the symmetric and antisymmetric stretching vibrations [3], indicate a general tendency for increase in the force constants in the investigated series with increase in the chemical properties of the substituent attached to the silicon atom. This tendency is in agreement with the chemical properties of the investigated compounds [2]. An especially strong inductive effect is manifested in the case of the phenylalkoxycyclosilazanes.

Still another interesting feature of the infrared spectra of the organoalkoxycyclosilazanes is the shift and splitting of the absorption bands, characteristic for the stretching vibrations of the N-H bond, and this is apparently associated with the formation of both intra- and intermolecular hydrogen bonding between the protons attached to nitrogen and the oxygen of the alkoxy group, and also with the conformational peculiarities in the structure of these cycles. Thus, if for the dialkyl- and alkylarylcyclosilazanes there is only one band, corresponding to the N-H bond and appearing in the 3380-4000 cm⁻¹ region, then for triphenyltrimethoxycyclotrisilazane it is split into 3 bands: 3265, 3290 and 3335 cm⁻¹, and for triphenyltriethoxycyclotrisilazane it is split into 2 bands: 3310 and 3340 cm⁻¹ (the spectra were taken as a KBr tablet). When the spectra of triphenyltriethoxycyclotrisilazane, taken under different conditions, are





Fig. 2. NMR spectrum of [CH₃C₂H₅OSiNH]₃ in benzene.

compared, it can be seen that in CCl_4 a new band at 3380 cm⁻¹ appears for this compound, while the other two bands have values of 3300 and 3350 cm⁻¹, i.e. only a part of the bands is disturbed in solution. If it is taken into account that the organocyclosilazanes can exist in the form of different stereoisomers with a fixed conformation of the silazane ring, then the splitting of the bands of the N-H bond in the spectra of these compounds becomes understandable, since in this case, depending on the geometric arrangement of the different NH and OR groups, the ability to form hydrogen bonds will be different for them.

Experimental difficulties did not let us separate the isomers of the phenylalkoxycyclosilazanes. In the case of trimethyltriethoxycyclotrisilazane, as the result of fractional crystallization from petroleum ether, we isolated two substances with different melting points, but with the same molecular weight and elemental composition. A study of their infrared spectra under various conditions revealed that the frequencies of the stretching vibrations of the N-H bond in the crystalline state have a value of 3300 cm⁻¹. They behave differently in CCl₄ solution, and specifically the substance with mp 112-112.5° has a frequency of 3380 cm⁻¹ for the stretching vibrations of the N-H bond, while the substance with mp 100-101° has values of 3330 and 3380 cm⁻¹. A shift of the frequencies in solution when compared with the frequencies manifested in the crystalline state, indicates the intermolecular character of the hydrogen bond in the first compound. A splitting of the corresponding frequency in the second compound testifies to the fact that the hydrogen bond in the crystalline state bears both an intermolecular and an intramolecular character. The intermolecular bond does not appear in the spectrum of the second compound in solution, while the intramolecular bond is manifested as a weak band with a value of 3330 cm⁻¹. On the basis of these data, taking into account the molecular geometry of the organocyclosilazanes, the latter compound can be assigned a structure of the "bathtub" type.

To ascertain the peculiarities in the geometric structure of the organoalkoxycyclosilazanes when compared with the ordinary dialkyl- and alkylarylcyclosilazanes, we studied the NMR spectra of the following compounds: $[CH_3C_2H_5SiNH]_3$ (I), $[CH_3C_6H_1_3SiNH]_3$ (II), $[CH_3C_6H_1_7SiNH]_3$ (III), $[CH_3C_6H_5SiNH]_3$ (IV), $[CH_3C_2H_5OSiNH]_3$ (V), $[C_6H_5CH_3OSiNH]_3$ (VI), and $[C_6H_5C_2H_5OSiNH]_3$ (VII). The signals of the protons of the methyl groups in the spectra of compounds (I)-(III) represent singlet signals with chemical shifts $\delta = 0.01$ to $0.02 \cdot 10^{-6}$ p.p.m., in which connection a complication of the spectra of the methyl protons does not occur as a result of the nonequivalent orientation of the methyl groups toward the bonds of the silazane ring and other substituents. This fact is explained by an averaging of the local magnetic fields due to internal rotation in the molecule (conversion of the ring). We encountered a similar phenomenon when studying the spectra of the (IV) isomers. The chemical shifts of the signals of the protons of the methyl groups for the trans-isomer are $\delta^1 = 0.36 \cdot 10^{-6}$ and $\delta^m = 0.39 \cdot 10^{-6}$ (the ratio of the intensities is 1:2), and for the cis-isomer, $\delta = 0.44 \cdot 10^{-6}$. This result was obtained by us by calculation, with an accuracy of $0.01-0.02 \cdot 10^{-6}$, using the data for the magnetic anisotropy of the phenyl group, and the distances and angles of the (IV) molecule for the case of a rapid conversion of the silazane ring.

Going on further to compound (VI), we were justified in assuming that, in view of an increase in the corresponding distances between the methyl and phenyl groups, the difference between the chemical shifts

of the protons of the different methyl groups in the trans-isomer, caused by the magnetic anisotropy of the benzene ring, should decrease. However, the opposite phenomenon is observed: the chemical shifts of the methyl protons of the trans-isomer of (VI) have the values $\delta^{i} = 3.50$ and $\delta^{m} = 3.62 \cdot 10^{-6}$ (the ratio of the intensities is 1:2), while the difference $\delta^{m} - \delta^{i} = 0.12 \cdot 10^{-6}$ [for the trans-isomer of (IV) the difference $\delta^{m} - \delta^{i} = 0.33 \cdot 10^{-6}$]. From this it follows that we are dealing with a fixed conformation of the silazane ring in compound (VI), since an increase in the Me-Ph distance can be masked by the absence of an averaging of the local magnetic fields from the bonds Si-N, Si-O, N-H, C-H and the C₆H₅ group. The same conclusions may be reached when the spectra of compound (V) are examined, having in mind the non-equivalence of the methyl and ethyl groups, which can be associated only with inhibited internal rotation in the molecule (Figs. 1 and 2).

The effect of structural factors on the chemical properties of the Si-N bond was studied on the example of the reaction of organoalkoxycyclosilazanes with catalytic amounts of alkali. As is known, this reaction leads to cleavage of the substituent attached to silicon and its stabilization by the proton attached to nitrogen, in which connection the ion of the alkali metal goes to the nitrogen, while the hydroxyl adds to the silicon atom. Further, the obtained intermediate compound attacks the neutral molecule of the cycle with the liberation of a new molecule of the hydrocarbon and the formation of a new Si-N bond, etc. [2, 4, 5]. Since here, as a rule, it is the more electronegative radicals that substitute, then in the case of the organoalkoxycyclosilazanes a cleavage of the alkoxy group could be expected, with the formation of alcohols or alcoholates as the low-molecular products, but these compounds are extremely reactive reagents toward organosilazanes and break the Si-N bond with the liberation, in the final analysis, of ammonia [6].

In the case of the phenylalkoxycyclosilazanes the liberation of ammonia occurs at 160°. The liberation of small amounts of benzene is observed when the temperature is raised above 200°. Further heating of the reaction mixture up to 270° ends in the formation of phenyltrialkoxysilazanes and soluble brittle polymers.

It is interesting to mention that cleavage of phenyl radicals in the case of the phenylalkoxycyclosilazanes occurs at a higher temperature (~230°), than, for example, in the case of trimethyltriphenylcyclotrisilazane (~170°). This fact is found to be in agreement with the data of the NMR spectra and the made calculations of the chemical shifts of the protons of the methyl groups. The difference in the chemical shifts of the protons of the methyl groups in the trans-isomers of trimethyltriphenylcyclotrisilazane and trimethoxytriphenylcyclotrisilazane is found to be in agreement with the fact that the energy barriers of the conformational transitions are somewhat higher for the latter. And since the formation of the transition complex and its decomposition, leading to rupture of the Si-R bond, are associated with a geometric rebuilding of the starting molecule, i.e., with a change in the conformation of the cyclic system, then for rupture of the Si-C₆H₅ bond more drastic conditions are needed in the case of the phenylalkoxycyclosilazanes than in the case of the phenylmethylcyclosilazanes.

The reaction of trimethyltriethoxycyclotrisilazane goes in the same manner as in the case of the phenylalkoxy derivative, but the temperature range is shifted toward lower temperatures, which testifies to the lower energetic stability of the conformers when compared with the phenylalkoxycyclosilazanes.

EXPERIMENTAL

<u>A m m on olysis of Phenyl methoxydichlorosilane</u>. 24.31 g of $C_6H_5CH_3OSiCl_2$, with bp 210.5–211.5° and analyzing 34.57% Cl, was dissolved in 200 ml of absolute benzene and then, with stirring, gaseous ammonia was passed over the solution until the phenylmethoxydichlorosilane was completely neutralized. The temperature during the reaction process remained below 30°. The NH₄Cl was filtered and the benzene was distilled from the solution under reduced pressure. Here a turbid liquid was obtained as residue, which was fractionally distilled in vacuo. From 15.8 g of product we isolated the fractions: I – bp 66–68° (1 mm), 2.25 g; II – bp 68–250° (1 mm), 2.34 g; III – bp 250–252° (1 mm), 1.29 g; IV – bp 252 to 289° (1 mm), 2.35 g. For fraction I: Found %: C 52.80, 52.91; H 7.01, 7.10; Si 15.19, 15.28; N 6.17, 6.00. $C_6H_5(CH_3O)_2SiNH_2$. Calculated %: C 52.43; H 7.15; Si 15.31; N 7.64. For fraction III: Found %: C 55.75, 55.74; H 6.06, 5.97; Si 18.37, 18.58; N 8.82, 8.69. $[C_6H_5CH_3OSiNH]_3$. Calculated %: C 55.60; H 6.00; Si 18.56; N 9.25. The still residue (5.84 g) was a brittle glassy polymer. Found %: C 57.52, 57.64; H 4.95, 4.90; Si 22.49, 22.33; N 11.05, 11.00. $[C_6H_5Si(NH)_{1.5}]_n$. Calculated %: C 56.55; H 5.15; Si 22.08; N 16.51.

A mmonolysis of Phenylethoxydichlorosilane. A solution of 65.15 g of $C_{6}H_{5}C_{2}H_{5}OSiCl_{2}$, with bp 222-225° and analyzing 31.73% Cl, in 450 ml of absolute benzene was aminated as described above.

From 43.02 g of obtained product we isolated the fractions: $I = bp 87-89^{\circ} (1 \text{ mm}), 10.29 \text{ g}; \Pi = bp 256 \text{ to} 259^{\circ} (1 \text{ mm}), 7.28 \text{ g}; \Pi = bp 289-292^{\circ} (1 \text{ mm}), 4.74 \text{ g}.$ The still residue represented 15.8 g of brittle glassy product, soluble in organic solvents. For fraction I: Found %: C 56.84, 56.98; H 8.20, 8.28; Si 13.15, 12.80; N 6.68, 6.87. $C_{6}H_{5}(C_{2}H_{5}O)_{2}SiNH_{2}$. Calculated %: C 56.83; H 8.11; Si 13.29; N 6.63. For fraction II: Found %: C 58.31, 58.46; H 6.64, 6.68; Si 16.98, 16.87; N 8.51, 8.30. $[C_{6}H_{5}C_{2}H_{5}OSiNH]_{3}$. Calculated %: C 58.13; H 6.71; Si 16.99; N 8.47. For fraction III: Found %: C 58.34, 58.22; H 6.64, 6.56; Si 17.04, 17.00; N 7.15, 7.41. $[C_{6}H_{5}C_{2}H_{5}OSiNH]_{4}$. Calculated %: C 58.13; H 6.71; Si 16.99; N 8.47.

<u>A m m o nolysis of Methylethoxydichlorosilane</u>. A solution of 58.53 g of $CH_3C_2H_5OSiCl_2$, with bp 101.5-102.5° and analyzing 45.1% Cl, in 250 ml of benzene was aminated in the same manner as described above. After removal of the NH₄Cl and benzene a viscous turbid liquid remained in the flask, which was dissolved in a small amount of petroleum ether and the insoluble crystalline portion was filtered and washed on the filter with petroleum ether. The yield of crystalline product was 15 g (39%). Found %: C 34.81, 35.00; H 8.81, 8.85; Si 26.51, 26.82; N 13.02, 13.30. [CH₃C₂H₅OSiNH]₃. Calculated %: C 34.92; H 8.75; Si 27.20; N 13.58. The petroleum ether was distilled from the solution. 21.24 g of the obtained clear viscous liquid was fractionally distilled into fractions. We obtained: I - bp 140-160°, 2.46 g; II - bp 160-170°, 3.06 g; III - bp 210-291°, 9.72 g. 5.4 g of solid product remained in the still pot. For fraction II: Found %: C 35.68, 35.57; H 9.02, 8.72; Si 25.74, 25.50; N 12.71, 12.66. C₂H₅O[CH₃C₂H₅OSiNH]₃H. Calculated %: C 33.10; H 9.26; Si 25.78; N 17.16.

<u>Heating of Phenyldimethoxyaminosilane</u>. 4.13 g of $C_6H_5(CH_3O)_2SiNH_2$, with bp 66-68° (1 mm) and n_D^{20} 1.4980, was heated in a flask under reflux for 8 h at 180-190°. The evolved NH₃ was absorbed in water and titrated. 0.081 g of NH₃ was evolved during this time. The rate of NH₃ evolution was insignificant on further heating: thus in 9.5 h another 0.007 g of NH₃ was evolved. The reaction products were fractionally distilled in vacuo. The following fractions were isolated: I - bp 65-70° (1 mm), 1.82 g, n_D^{20} 1.4745; II - bp 161-183°, 0.62 g, n_D^{20} 1.5120; III - bp 265-275° (1 mm), 0.6 g. 2.01 g of viscous product remained in the still pot. Literature data for $C_6H_5Si(OCH_3)_3$: bp 103° (20 mm); n_D^{20} 1.4734.

<u>Heating of Phenyldiethoxyaminosilane</u>. 7.05 g of $C_6H_5(C_2H_5O)_2SiNH_2$, with bp 87-89° (1 mm) and n_D^{20} 1.4840, was heated at 180-190° for 22 h. 0.082 g of NH₃ was evolved during this time. The following fractions were isolated by fractional distillation: I = bp 230-234° (760 mm), 2.61 g, n_D^{20} 1.4632; II = bp 160-165° (1 mm), 0.72 g, n_D^{20} 1.4891; III = bp 230-240° (1 mm), 0.77 g; IV = bp 260-262° (1 mm), 0.38 g. 1.39 g of substance remained in the still pot. For fraction II: Found %: C 58.37, 58.45; H 6.84, 6.98; Si 14.30, 14.59. $C_2H_5O[C_6H_5C_2H_5OSINH_2H$. Calculated %: C 57.41; H 7.40; Si 14.90. Literature data for $C_6H_5Si(OC_2H_5)_3$; bp 233-234° (760 mm), and n_D^{20} 1.4620.

<u>Reaction of Triphenyltrimethoxycyclotrisilazane with KOH</u>. A mixture of 6.09g of $[C_6H_5CH_3OSiNH]_3$ and 0.05 g of KOH was heated for 40 min at 120°. The evolution of NH₃ began at 170°. Further heating led to the liberation of small amounts of benzene (two drops), while the liberation of phenyltrimethoxysilane was observed at 270°, the total amount of which was 1.81 g, bp 202° (760 mm), n_D^{20} 1.4714. NH₃ liberated during reaction was 0.07 g.

Reaction of Triphenyltriethoxycyclotrisilazane with KOH. A mixture of 7.28 g of $[C_6H_5C_2H_5OSiNH]_3$ and 0.07 g of KOH was heated at 120° for 1 h. Then the temperature was raised gradually and the liberation of NH₃ was observed at 160°, and of benzene at 230-240°, the total amount of which was 0.3 g. Further increase in the temperature up to 280° led to the liberation of phenyltriethoxysilane, with bp 233-236° and n_D^{20} 1.4589, in an amount of 2.43 g. The amount of evolved NH₃ was 0.086 g. The still residue represented a brittle glassy mass, soluble in organic solvents.

<u>Reaction of Trimethyltriethoxycyclotrisilazane with KOH</u>. A mixture of 3.45 g of $[CH_3C_2H_5OSiNH]_3$ and 0.04 g of KOH was heated, gradually raising the temperature up to 135°. NH₃ began to be evolved at this temperature. Further heating at 145-160° led to the liberation of methane in an amount of 0.036 g. The liberation of methyltriethoxysilane began at 210°, bp 142°, and n_D^{20} 1.3860) (literature data: bp 143°, and n_D^{20} 1.3860). Its amount was 1.02 g. A solid soluble polymer remained in the still pot.

CONCLUSIONS

1. A study was made of the ammonolysis of some organoalkoxydichlorosilanes and it was established that, together with the formation of cyclic products, exchange reactions take place, which lead to the formation of organoalkoxyaminosilanes and linear products.

2. A study was made of the inductive effect of electronegative substituents in organoalkoxycyclosilazanes on the spectroscopic characteristics of the Si-N bond in these compounds, and also in some other organocyclosilazanes.

3. On the basis of the infrared and NMR spectra it was established that the conformation of the silazane ring in organoalkoxycyclosilazanes is fixed, in contrast to the ordinary organocyclosilazanes, which is apparently due to the formation of hydrogen bonds between the oxygen of the alkoxy group and the hydrogen of the NH group.

4. A study was made of some of the peculiarities of the reaction of organoalkoxycyclosilazanes with nucleophilic reagents.

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