SYNTHESIS AND STRUCTURE OF METHYL-SUBSTITUTED 1,3-DIOXA-2-SILACYLOHEXANES

R. S. Musavirov, E. P. Nedogrei,
L. F. Lapuka, E. A. Kantor,
R. S. Sarmanaev, and D. L. Rakhmankulov

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The reaction of dimethyl(diethylthio)silane with 1,3-diols gave methyl-substituted 1,3-dioxa-2-silacyclohexanes. ^{1}H and ^{13}C NMR spectroscopy were used to study the configuration and predominant conformations of the compounds studied.

The cyclization of diols with various bifunctional compounds is the most common method for the synthesis of 1,3-dioxa-2-silacyclohexanes [1-16].

The use of dihalosilanes [1-3] at temperatures not exceeding 50°C permits the preparation of the silicon analogs of acetals in 50-80% yield. An advantage of this synthetic method lies in the availability of the starting halosilanes. However, the need to remove the HCl formed by the usual method of converting it to an amine hydrochloride somewhat reduces the preparative value of this reaction. Interest is found in a method involving the substitution of the arsenic atom by a silicon atom in 1,3-dioxa-2-arsocyclopentane by reaction with dimethyldichlorosilane [4] which permitted the synthesis of previously unreported 1,2-dimethyl-1,3-dioxa-2-silacyclopentane.

The removal of the alcohol or acetic acid formed from the reaction mixture and the shift in the equilibrium upon using dialkoxysilanes [1, 5-9] and diacetoxysilanes [10, 11] raises the yield of cyclic ethers of dialkylsilanediols to 80-90%. The reactions are usually carried out in the presence of a solvent and acid or basic catalysts. The reaction temperature is dictated by the need to remove volatile reaction products.

The high reactivity of silazanes permits carrying out the reaction most often without solvent by the simple mixing of the reagents [12-16]. The mild reaction conditions permit the synthesis of 1,3-dioxa-2-silacycloalkanes containing not only alkyl substituents but also halogen atoms and alkoxy groups at the silicon atom.

The cleavage of linear or cyclic polydialkylsiloxanes [1] or silazanes [13] in the presence of basic catalysts leads to cyclic esters of orthosilicic acid in 60-80% yield. This method requires the continuous azeotropic distillation of the water formed from the reaction mixture.

Diols, especially secondary and tertiary diols, are not readily available compounds and, thus, methods which permit the synthesis of 1,3-dioxa-2-silacycloalkanes without the use of diols hold definite interest. The use of cyclic acetals instead of diols in the reaction with dialkoxy- or diacetoxysilanes leads to cyclic esters of dialkylsilanediols [17, 18]. Diethylsilanes react with α, β, γ-hydroxyacids in the presence of colloidal nickel or palladium to form 1,3-dioxa-2-silacycloalkanones in 60-70% yield [19-23]. The reaction of 1,2-dicarbonyl compounds with silylphosphine permits the synthesis of 1,3-dioxa-2-silacyclanes containing phosphorus atoms [24]. Alcohols readily displace mercaptans in alkylthiosilanes [25]. On the other hand, the reactions of dialkylthiosilanes with diols have not been described.

In the present work, we studied the reaction of dimethyl(diethylthio)silane (I) with 1,3-diols II-VI which permits the synthesis of methyl-substituted 2,2-dimethyl-1,3-dioxa-2-silacyclohexanes VII-XI (Table 1).

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TABLE 1. Physicochemical Constants of Compounds Synthesized VII-XI

Compound	M*	bp, °C (mm Hg)	n_D^{20}	d_4^{20}	Found, %			Chemical formula	Calculated, %		
					C	H	Si		C	H	Si
VII†	132	123 (752)	1,4122	0,9701	45,40	9,20	21,20	$C_5H_{12}O_2Si$	45,42	9,15	21,24
VIII	146	132 (754)	1,4168	0,9532	49,30	9,60	19,20	$C_6H_{14}O_2Si$	49,27	9,64	19,22
IX	160	139 (751)	1,4213	0,9421	52,50	10,10	17,50	$C_7H_{16}O_2Si$	52,45	10,06	17,05
X‡	160	141 (753)	1,4169	0,9759	52,40	10,00	17,50	$C_7H_{16}O_2Si$	52,45	10,06	17,54
XI	174	146 (755)	1,4263	0,9362	54,60	11,10	16,10	$C_8H_{18}O_2Si$	54,62	11,13	16,10

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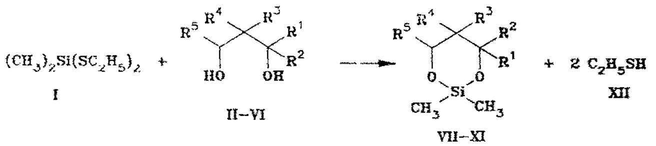
*Determined by mass spectrometry.

† n_D^{20}: 1,4130 [1], 1,4118 [17], 1,4120 [2]; d_4^{20} 0,9701 [17], 0,9703 [2]; T_{bp}: 122 (763) [1], 123 (752) [17], 121,7° [2].

‡ n_D^{20}: 1,4272 [1], 1,4170 [43]; T_{bp} 144—145 (762) [1], 24—25 (5)° [43].



II, VII $R^1=R^2=R^3=R^4=R^5=H$; III, VIII $R^1=CH_3$, $R^2=R^3=R^4=R^5=H$; IV, IX $R^1=$ $=R^2=CH_3$, $R^3=R^4=R^5=H$; V, X $R^1=R^2=R^5=H$, $R^3=R^4=CH_3$; VI, XI $R^1=R^2=R^5=CH_3$, $R^3=R^4=H$

This reaction is not an equilibrium process and proceeds both with and without acid catalysts such as H_2SO_4, $SnCl_4$, and p-toluenesulfonic acid (Table 2). The use of $SnCl_4$ and p-toluenesulfonic acid at concentrations less than 0.01 mole/liter has virtually no effect on the yield of dioxasilacyclohexane IX (Table 2). Increasing the concentration of $SnCl_4$ or p-toluenesulfonic acid to 0.01 mole/liter or the use of H_2SO_4 markedly reduces the yield of IX. The reaction occurs instantaneously upon mixing of the reagents. An increase in the temperature increases the yield of 1,3-dioxa-2-silacyclohexanes such as XI having gem-dimethyl substituents at the quaternary carbon atom in the ring. An increase in the temperature from 20 to 60°C in the reaction of 1,3-propanediol with silane I leads to a reduction in the yield of VII from 58 to 10%. The temperature effect on the yield of the desired product is much less pronounced upon using methyl-substituted propanediols III-V (Table 2).

Thus this reaction, in comparison with the reaction of diols with chlorosilanes [1-3], alkoxysilanes [1, 5-9], and acetoxysilanes [10, 11] or the cleavage of polyorganylsiloxanes [1], proceeds under milder conditions similar to the reaction of diols with silazanes [12-16] and gives 1,3-dioxa-2-silacyclohexanes in yields up to 76% (Table 2).

There have been only a few conformational studies of 1,3-dioxa-2-silacyclohexanes, and their results have been contradictory [26-29]. Thus, for example, Albriktsen [28] and Voronkov [29] concluded chair—chair conformational transitions on the basis of the averaged ^1H NMR spectral parameters of 2,2-dimethyl- and 2,2,5,5-tetramethyl-1,3-dioxa-2-silacyclohexanes, while Hellier [27] indicated a significant contribution of flexible structures to the conformational equilibrium. Low-temperature measurement of ^1H NMR spectra of these compounds [28] showed a lower inversion barrier than that for 1,3-dioxanes. For cyclic system having a silicon atom with tetrahedral bond configuration [30], we should expect a stereochemical analogy with the corresponding 1,3-dioxanes. 2-Alkoxy-2-sila-1,3-dioxacyclohexanes may be taken as an example. These compounds, similar to 2-alkoxy-1,3-dioxanes, are found predominantly in their conformation with axial orientation of the alkoxyl substituent [9].

We should note that the energy barriers in cyclohexanes and 1,3-dioxanes for chair—chair interconversion are 1.2-2.0 times higher than in chair—twist interconversions [32, 33, 42]. The Si—O (1.66 Å [31]) and Si—C (1.85 Å [31]) bond lengths in organosilicon compounds are greater than the lengths of the analogous C—O (1.44 Å [12]) and C—C bonds (1.50 Å [32]) in their carbon analogs. Therefore, some levelling out of the rotational barriers is possible in organosilicon compounds due to a diminution of steric hindrance. Thus, for example, the energy barriers for chair—chair and chair—twist interconversions in 1,1-dimethyl-1-silacyclohexane are virtually identical (5.5 and 5.4 kcal/mole, respectively [33]).

TABLE 2. Conditions and Results of the Reaction of Dimethyldiethylthiosilane (I) with 1,3-Diols (II-VI) in 1,4-Dioxane

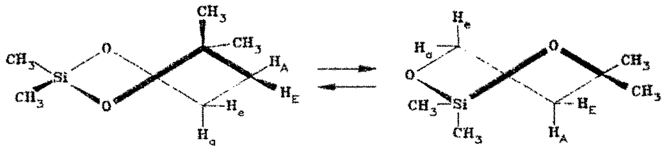
Diol*	Catalyst, mole/liter	Dioxasilacyclohexane†	Yield, % of diol taken at T, °C			Diol*	Catalyst, mole/liter	Dioxasilacyclohexane†	Yield, % of diol taken at T, °C		
			20	40	60				20	40	60
II		VII	58	34	10	IV	p-TSA 0,01	IX			52
III		VIII	36		32	IV	p-TSA 0,04	IX			32
IV		IX	66	70	76	V		X	42	42	42
IV	H_2SO_4 0,01	IX			6	VI		XI	52		66
IV	$SnCl_4$ 0,01	IX			48						

*1,3-Propanediol (II), 1,3-butanediol (III), 3-methyl-1,3-butanediol (IV), 2,2-dimethyl-1,3-propanediol (V), and 4-methyl-2,4-pentanediol VI.

†2,2-Dimethyl-1,3-dioxa-2-silacyclohexane (VII), 2,2,4-trimethyl-1,3-dioxa-2-silacyclohexane (VIII), 2,2,4,4-tetramethyl-1,3-dioxa-2-silacyclohexane (IX), 2,2,5,5-tetramethyl-1,3-dioxa-2-silacyclohexane (X), 2,2,4,4,6-pentamethyl-1,3-dioxa-2-silacyclohexane (XI).

The ^1H and ^{13}C NMR spectra of 2,2-dimethyl- (VII), 2,2,5,5- (X) and 2,2,4,4-tetramethyl-1,3-dioxa-2-silacyclohexanes (IX) taken at room temperature indicate the stereochemical equivalence of the geminal substituents in all the ring positions (Tables 3 and 4), which indicates rapid ring inversion for these molecules on the NMR time scale. Thus, for example, the protons of the methyl groups at Si in VII give a ^1H NMR signal at δ 0.06 ppm. The protons at $C_{(4)}$ and $C_{(6)}$ give rise to a triplet at δ3.98, while the protons at $C_{(5)}$ give a quintet at δ1.78. The averaged vicinal spin–spin coupling constant for the protons of the carbon ring segment is 6.2 Hz. As shown for several cis-1,4-substituted cyclohexanes [34], this behavior is possible for molecules with rapidly inverting twist conformations.

An analogous conclusion was drawn by Hellier [27] in a conformational study of a series of 2,2-dimethyl-, 2,2-diphenyl-, 2-methyl-2-phenyl-1,3-dioxa-2-silacyclohexanes and their 5,5-dimethyl derivatives. However, chair–chair inversion was indicated by other workers in describing the spectra of similar structures [28, 29]. The methyl protons of the gem-dimethyl groups at Si and $C_{(4)}$ in the ring of IX give singlets at δ0.02 and 1.20 ppm, respectively. The methylene protons at $C_{(5)}$ and $C_{(6)}$ give distorted triplets at δ1.66 and 3.96 ppm, respectively; the middle line of these triplets is the superposition of five lines and its amplitude is comparable to the two side lines. Analysis of the multiples according to second order rules [35] gives the absolute sum of the averaged vicinal coupling constants of the methylene protons (11 Hz) and the absolute difference of the geminal coupling constants of the methylene protons at $C_{(5)}$ and $C_{(6)}$ (2.9 Hz). Assuming that the vicinal coupling constants have the same signs, we expect that $^3J_{6E5e} + {}^3J_{6A5a} = 15.1$ Hz. In accord with Pihlaja and Auras [36], such a value is characteristic for non-chairlike structures, while the analogous sum of the coupling constants for chairlike 1,3-dioxanes does not exceed 14 Hz [32, 36-38] (for the ideal 1,3-dioxane chair, $^3J_{6E5e} = 1.2$ and $^3J_{6A5a} = 12.4$ Hz). Thus, the NMR spectra of IX indicate rapid conversions of the following type:



Analogous structures were established for alkyl-substituted 1,3-dioxanes, in which 2,4-syndiaxial nonbonding interactions are possible in the chair conformation between the two alkyl groups [32, 37, 38].

The PMR spectra of XI indicate the realization of a predominant conformation. The proton at $C_{(6)}$ gives a multiplet at δ4.14 ppm with coupling constants 6.2, 4.8, and 8.8 Hz. The splitting by 6.2 Hz is due to coupling of this proton with the protons of the methyl group at $C_{(6)}$, while the two other coupling constants are due to the protons at $C_{(5)}$. The value $^3J_{HH} = 8.8$ Hz is characteristic for trans orientation of the coupled protons [32, 38] and corresponds to a dihedral angle θ between the C–H bonds at $C_{(5)}$ and $C_{(6)}$ close to 180° on the Karplus curve. This permits assignment of pseudoaxial orientation for the proton at $C_{(6)}$ and,

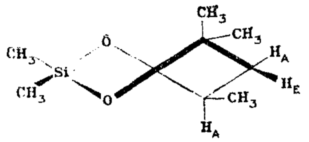
TABLE 3. Chemical Shifts and Coupling Constants in the ^1H NMR Spectra of 1,3-Dioxa-2-silacylohexanes (VII-XI)

Compound	δ, ppm, signal type, J, Hz						
	ring protons				substituent protons		
	$C_{(4)}$	$C_{(5)}$	$C_{(6)}$	2-CH_3	4-CH_3	5-CH_3	6-CH_3
VII	3,98 t $^3J_{HH}=6,2$	1,78q	3,98t	0,06 s	—	—	—
	3,97 t [26]	1,76 q [26]	3,97 t [26]	0,09 s [26]	—	—	—
	3,9 t [28]	1,7 q [28]	3,9 t [28]	0,08 s [28]	—	—	—
	3,98 t [27] $^3J_{HH}=5,4$ [27, 28] $^3J_{HH}=5,6$ [37]	1,81 q [27]	3,98t [27]	−0,2 s [27]	—	—	—
IX	—	1,66t	3,96 t $^3J_{6E5e}+^3J_{6A5a}=15,1$	0,02 s	1,20 s	—	—
X	3,44 s	—	3,44 s	0,06 s	—	0,78	—
	3,56 s [26]	—	3,56 s [26]	0,09 s [26]	—	0,95 s [26]	—
	3,6 s [27]	—	3,6 s [27]	0,2 s [27]	—	0,95 s [27]	—
XI	—	1,48 Ha 1,41 He $^2J_{5a5e}=-14,0$ $^3J_{5a6A}=8,8$ $^3J_{5e6A}=4,8$	4,14 HA $^3J_{6A5e}=4,8$ $^3J_{6A5a}=8,8$ $J_{6ACH_3}=6,2$	0,03 s	1,13 s 1,19 s	—	1,08 d $^3J_{CH_36A}=6,2$
VIII	3,90 HA $^3J_{4A5a}=9,6$ $^3J_{4A5e}=5,8$	1,51 Ha 1,22 He $^2J_{5a5e}=-14,0$ $^3J_{5a4A}=9,6$ $^3J_{5a6E}={}^3J_{5e4A}=5,8$ $^3J_{5e6E}=2,8$	3,84 HA 3,79 HE $^2J_{6A6E}=-10,2$ $^3J_{6A5a}=9,6$ $^3J_{6A5e}={}^3J_{6E5a}=5,8$ $^3J_{6E5e}=2,8$	0,03 s 0,05 s	1,05 d $^3J_{CH_34A}=6,5$	— —	— —

TABLE 4. Chemical Shifts and $^1J_{C-H}$ Coupling Constants in the ^{13}C NMR Spectra of 1,3-Dioxa-2-silacyclohexanes (VII-XI)

Compound	δ, ppm, signal type, J, Hz						
	ring atoms			substituent atoms			
	$C_{(4)}$	$C_{(5)}$	$C_{(6)}$	2-CH_3	4-CH_3	5-CH_3	6-CH_3
VII	58,6 t $J=142$	35,1 t $J=125$		−3,6 q $J=118$			
VIII	68,7 d $J=139$	38,2 t $J=126$	61,9 t $J=144$	−0,90 q $J=118$ −2,8 q $J=118$	23,9 q $J=125,5$		
IX	74,7 s	41,5 t $J=125$	59,1t $J=144$	0,3q $J=117$	29,7 q $J=124$		
X	72,7 t $J=145$	34,6 s	72,7 t $J=145$	−3,0 q $J=118$		21,4 q $J=125$	
XI	71,6 s	48,9 t $J=125$	64,7 d $J=125$	0,2 q $J=118$ 1,0 q $J=118$	27,7 q $J=123$ 32,7 q $J=123$		24,4 q $J=126$

correspondingly, a pseudoequatorial orientation for the methyl substituent at $C_{(6)}$ (δ 1.06 ppm). The magnetic inequivalence of the protons at $C_{(5)}$ is 0.07 ppm.* The protons of the methyl groups at Si give rise to a broadened singlet. The methyl groups at $C_{(4)}$ give singlets with a chemical shift difference of 0.04 ppm. These spectral indices indicate that in solutions of XI, the conformational equilibrium is shifted toward the predominant formation of the 2,5-twist conformer:



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*The chemical shift difference for H_a and H_e at $C_{(5)}$ is 0.25-0.60 ppm in the chair conformation of the 1,3-dioxane ring [32, 40, 41].

2,2,4-Trimethyl-1,3-dioxa-2-silacyclohexane VIII gives a complex spectrum due to the presence of two stereoisomers in 55:45 ratio.

Enrichment of the mixture with the more volatile component of VIII permitted interpretation of the spectrum of this stereoisomer. The methylene protons H_A and H_E at $C_{(6)}$ are characterized by inversion of the nuclear magnetic shielding constants σ. The axial proton has a greater chemical shift, probably as a result of the redistribution of electron density in VIII upon the interaction of the unshared electron pairs of the oxygen atoms with the d orbitals of the silicon atom. However, the geminal and vicinal coupling constants in VIII are close to the typical values in 1,3-dioxanes [32, 38] with chair conformation. The proton adjacent to the methyl group at $C_{(4)}$ gives rise to a signal at δ3.9 ppm with characteristic vicinal constants 9.6 and 5.8 Hz due to coupling of the axial proton with the methylene protons at $C_{(5)}$. Thus, the methyl group occupies the equatorial position at $C_{(4)}$. The protons at $C_{(5)}$ have inversion to the σ_a and σ_e constants. Inversion of the shielding constants at $C_{(5)}$ is typical for 1,3-heterocycles containing oxygen or nitrogen only in the chair conformation [39]. Thus, these results indicate that the more volatile stereoisomer is predominantly in the chair conformation.

The chemical and three-dimensional structures of these compounds were also supported by their ^{13}C NMR spectra. In the spectra of 2,2-dimethyl-2,2,4,4-, and 2,2,5,5-tetramethyl-1,3-dioxa-2-silacyclohexanes with total proton decoupling, the signals of the methyl carbons of the gem-dimethyl groups are singlets (Table 4), which, as in the PMR spectra, indicates rapid ring inversion. In the case of XI, in which the equilibrium is enanchomeric, the carbon atoms of each of the gem-dimethyl groups resonate at different fields. The chemical shift difference of the carbon atoms of the methyl groups at $C_{(4)}$ ($\Delta\delta$ = 5.0 ppm) is significantly greater than that for the carbon atoms of the methyl groups at the ring silicon atom ($\Delta\delta$ = 0.8 ppm).

EXPERIMENTAL

The ^1H NMR spectra of VII, IX-XI in CCl_4 or in CCl_4 with 5% benzene or $CHCl_3$ for VIII were taken at 24°C on a Tesla BS-497 NMR spectrometer at 100 MHz. The ^{13}C NMR spectra were taken on a Bruker WH-90 spectrometer with and without suppression of C–H coupling with spectral width 6 kHz. Hexamethyldisiloxane was used as the internal standard.

The syntheses were carried out in a jacketed 6 cm^3 glass reactor with a device for the removal of samples for chromatographic analysis. The temperature was maintained with an ultrathermostat to ±0.1°C. The calculated amounts of the reagents and solvent (1,4-dioxane) were added to the reactor. The catalyst was added after the solution reached the required temperature and the reaction mass was maintained at this temperature for 10 min. The reaction was stirred with a magnetic stirrer in order to obtain rapid homogenization of the catalyst.

The analysis of the starting and final products was carried out by gas-liquid chromatography on an LKhM-8MD model 5 chromatograph using a katharometer detector and a 3 m × 3 mm column packed with SE-30 on Chromatone N-AB (0.15-0.20 mm fraction). The column temperature was 50-250°C. The programming was accomplished at 6°/min. The helium gas flow rate was 2 liter/h.

The reaction products were fractionated by distillation and on a PAKhV-08 preparative chromatograph using a 2 m × 9 mm column packed with 10% SE-30 on Chromatone N-AB (0.3-0.6 mm fraction) and helium as the gas carrier.

The physicochemical indices of VII-XI are given in Table 1.

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