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STRUCTURE OF N-(1-SILATRANYLMETHYL) AND N-(TRIMETHOXYSILYL-METHYL) DERIVATIVES OF NITROGEN-CONTAINING HETEROCYCLES ACCORDING TO DATA OF NMR, IR, AND UV SPECTROSCOPY

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The ^{1}H, ^{13}C, ^{15}N, and ^{29}Si NMR, IR, and UV spectra of N-(1-silatranylmethyl) and N-(trimethoxysilylmethyl) derivatives of nitrogen heterocycles have been studied. The dependence and interrelation of the chemical shifts of ^{29}Si and ^{15}N nuclei of the silatranyl group in the spectra of N-(1-silatranylmethyl)-substituted nitrogen heterocycles are determined by the nature of the heterocyclic system.

Keywords: N-(1-silatranylmethyl) derivatives of nitrogen-containing heterocycles, silatranes, triethanolamine, NMR spectroscopy.

Chelate compounds of pentacoordinated silicon, the silatranes, attract attention for their high biological activity, structural special features, and also as possible intermediates in nucleophilic substitution reactions at the silicon atom.

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* I and my coworkers dedicate this paper to the 70th birthday of Academician Professor Edmunds Lukevics and with all our hearts wish him good health, the further continuation of his distinguished scientific work, good fortune, and success in all deeds.

I am personally proud of the fact that for 10 years Edmunds worked in my laboratory in the Institute of Organic Synthesis of the Latvian Academy of Sciences. Together we began investigations in several new directions of organosilicon chemistry. Subsequently he brilliantly continued them independently, making an enormous contribution to the chemistry of silatranes, germatranes, and biologically active organosilicon compounds, bringing worldwide attention to it.

Together with E. Lukevics we announced the birth of a new branch of silicon chemistry, bioorganosilicon chemistry, and jointly published six monographs in Russian (five of which have been translated in USA, Germany, Roumania, and Poland).

We highly appreciate the fruitful work of E. Lukevics in the post of Editor-in-Chief of the journal Chemistry of Heterocyclic Compounds (Khimiya Geterotsiklicheskikh Soedinenii), which he has converted into the truly international periodic publication.

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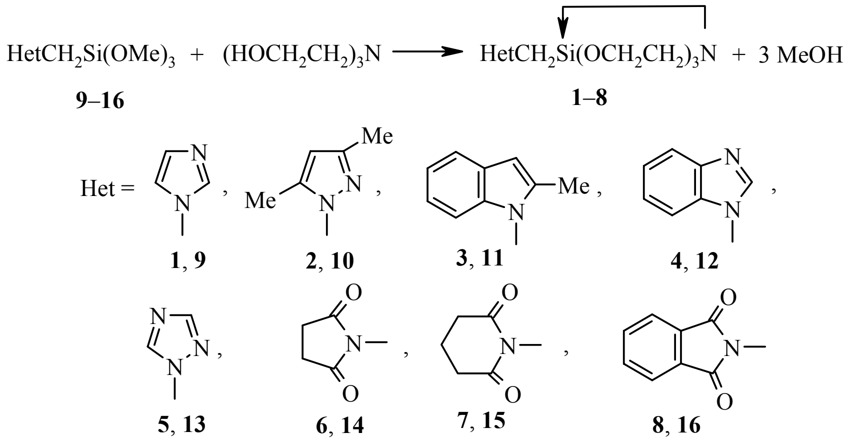
The silatranyl group $-Si(OCH_2CH_2)_3N$ (Sa) displays an extremely high electron-donating inductive effect ($\sigma_I = -0.56$, $\sigma^* = -3.40$) [1, 2]. Thanks to this the + *I*-effect of the $-CH_2Sa$ group ($\sigma_I = -0.36$, $\sigma^* = -2.24$ [1, 3] is also significant in derivatives of silatrane XCH_2Sa, where X is an inorganic or organic substituent. This is also indicated by the high basicity of compounds of the series XCH_2Sa (X = F, Cl, Br, I, –O, –S, >N, $CH_2=CH-$, etc.), determined when studying their intermolecular interaction with phenol, and their low ionization potentials [4].

Of particular interest are the N-(1-silatranylmethyl) derivatives of nitrogen-containing heterocycles $HetCH_2Si(OCH_2CH_2)_3N$ (Het is azolyl), in which the silatranyl group (Sa) is linked to an endocyclic nitrogen atom of a heteroaromatic system by an exocylic CH_2 bridge.

With the aim of studying the influence of the high electron-donating inductive effect of the silatranylmethyl group (CH_2Sa) on the stereoelectronic structure and reactivity of nitrogen-containing heterocycles by NMR, IR, and UV spectroscopic methods we have investigated N-(1-silatranylmethyl)-substituted imidazole (**1**), 3,5-dimethylpyrazole (**2**), 2-methylindole (**3**), benzimidazole (**4**), 1,2,4-triazole (**5**), succinimide (**6**), glutarimide (**7**), and phthalimide (**8**). They were all synthesized in high yield (89-97%) by the transesterification of the appropriate (N-heterylmethyl)trimethoxysilanes **9-16** with tris(2-hydroxyethyl)amine in the absence of catalyst [5-7].



$$HetCH_2Si(OMe)_3 + (HOCH_2CH_2)_3N \longrightarrow HetCH_2Si(OCH_2CH_2)_3N + 3\,MeOH$$

Compounds **1-8** were colorless crystalline substances with mp from 127 (**4**) to 267°C (**6**), soluble in halogenated hydrocarbons, MeCN, DMSO, and DMF.

We have compared the UV absorption spectra of N-(1-silatranylmethyl) (**2-4**, **8**) and of N-(trimethoxysilylmethyl) derivatives (**10-12**, **16**) of nitrogen-containing heterocycles in acetonitrile solution. A general tendency was observed in the UV spectra of solutions of compounds **2-4**, **8** and **10-12**, **16** for a long wave displacement of bands on replacing the trimethoxysilyl group by silatranyl (Table 1). The size of this displacement varied from 3 to 10 nm. In the spectrum of compound **6** the long wave band was a doublet (213 and 230 nm), while in the spectrum of compound **16** it was observed at 222 nm. Moreover, in the spectrum of compound **8** the long wave band was observed at 322 nm, which is probably caused by a $n \rightarrow \pi^*$ transition of a lone pair of the carbonyl oxygen from a nonbonding orbital to vacant π-orbitals of the heterocycle. Until now this band has only been observed in the spectra of α,β-unsaturated ketones in the region of 300-330 nm. The bathochromic shift in the UV absorption spectra of 1-(N-heterylmethyl)silatranes **2-4**, **8** compared with the UV spectra of the corresponding N-(trimethoxysilylmethyl) derivatives **10-12**, **16** is caused by the fact that it is possible to consider the silatranyl group as an intramolecular donor-acceptor complex of the *n*,σ-type. The mesomeric effect of the silatranyl group on the π-electron system of the heterocycles may be attributed to the overlap of the axial C–Si←N fragment of the silatranyl group with the lone pair of the nitrogen atom of the heterocycle.

TABLE 1. UV Absorption Spectra of Solutions of $HetCH_2Si(OCH_2CH_2)_3N$ (**2-4**, **8**) and $HetCH_2Si(OMe)_3$ (**10-12**, **16**) in MeCN

Compound	λ_1, nm (ε)	λ_2, nm (ε)
2	224 (7800)	—
3	290 (6300)	229 (41 000)
4	259 (6200), 269 (5400) 276 (5100) 283 (3700)	211 (38 300)
8	283 (1050), 291 (1250) 299 (1120), 322 (600)	213 (30 800) 230 (28 500)
10	221 (5900)	—
11	284 (8050)	227 (47 200)
12	255 (7100), 269 (5300) 277 (5050), 285 (4050)	208 (25 000)
16	293 (1800), 299 (1750)	222 (51 200)
3,5-Dimethylpyrazole	210 (3650)	—
2-Methylindole	270 (6600), 288 (4700)	220 (30 300)
Benzimidazole	244 (5500), 250 (5300) 268 (3900), 273(5100 280 (5800)	203 (30 500)

TABLE 2. Characteristic Group Frequencies in the Spectra of $HetCH_2Si(OCH_2CH_2)_3N$ (**2-5**, **8**) and $HetCH_2Si(OMe)_3$ (**10-13**, **16**)

Compound	ν, $Si(OR)_3$*, cm^{-1}	ν, Het, cm^{-1}
2	580, 647, 787, 798, 807, 918, 948, 1022, 1091, 1144	1552, 3122
3	574, 630, 640, 767, 791, 801, 908, 914, 941, 1018, 1088, 1121	737, 1542, 1572, 1609, 3022, 3038
4	590, 650, 774, 784, 794, 814, 921, 948, 958, 1031, 1088, 1128	754, 761, 1010, 1499, 1502, 1626, 3045, 3060, 3095
5	594, 644, 771, 801, 824, 924, 950, 968, 1018, 1091, 1121	1519, 3128
8	574, 607, 634, 781, 794, 804, 914, 938, 944, 1018, 1088, 1115	717, 724, 1482, 1489, 1499, 1616, 1696, 3035, 3048
10	844, 1095, 1200, 2848	1559, 3121
11	821, 1085, 1188, 2841	747, 1550, 1572, 1610, 3050, 3075
12	831, 1075, 1188, 2841	741, 1002, 1492, 1585, 1612, 3052, 3075
13	845, 1090, 1200, 2852	1559, 3121
16	832, 1081, 1185, 2841	717, 1469, 1616, 1709, 3025, 3050

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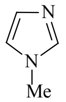
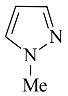
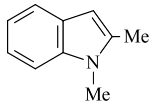
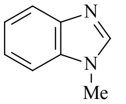
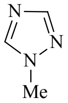
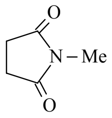
* **2-5**, **8** $Si(OR)_3$ = Sa; **10-13**, **16** $Si(OR)_3 = Si(OMe)_3$

To assess the electron-donating effect of the silatranylmethyl group we studied the IR spectra of 1-(N-heterylmethyl)silatranes **2-5**, **8** and (N-heterylmethyl)trimethoxysilanes **10-13**, **16** in MeCN. In the IR spectra of silatrane derivatives **2**, **5**, and **8** the frequencies of the unsaturated fragments C=N and C=O of the heterocycle were reduced significantly in comparison with the corresponding trimethoxysilyl-substituted derivatives **10**, **13**, and **16**. The group characteristic frequencies in the IR spectra of silatrane derivatives **2-5** do not depend in practice on the nature of the heterocycle with the exception of compound **8**. In its spectrum the bands of the SiO_3 grouping at

TABLE 3. ^{1}H, ^{13}S, ^{15}N, and ^{29}Si NMR Spectra of $HetCH_2Si(OCH_2CH_2)_3N$ (**1-8**) and $HetCH_2Si(OMe)_3$ (**9-16**)

Compound	Chemical shifts ($CDCl_3$), δ, ppm. (*J*, Hz)			
	^{1}H	^{13}C	^{15}N	^{29}Si
1	2.86 (t, $^{3}J = 5.9$, NCH_2); 3.23 (s, CH_2); 3.77 (t, $^{3}J = 5.9$, OCH_2); 6.91 (d, $^{3}J = 1.1$, H-4,); 6.94 (d, $^{3}J = 1.1$, H-5); 7.49 (s, H-2)	38.12 ($^{1}J_{C\text{-}Si} = 108.7$, CH_2); 50.97 (NCH_2); 57.07 (OCH_2); 121.31 (C-4); 127.06 (C-5); 138.41 (C-2)	-121.8 (N-3); -218.9 (N-1); -351.6 (N-Si)	-80.3
2	2.20 (s); 2.21 (s, CH_3); 2.82 (t, $^{3}J = 5.9$, NCH_2); 3.42 (s, CH_2); 3.77 (t, $^{3}J = 5.9$, OCH_2); 5.67 (s, H-4)	11.46 (CH_3, (C-5)); 13.71 (CH_3, (C-3)); 41.23 ($^{1}J_{C\text{-}Si} = 111.7$, CH_2); 50.77 (NCH_2); 57.09 (OCH_2); 103.22 (C-4); 138.28 (C-5); 144.23 (C-3)	-77.8 (N-2); -172.9 (N-1); -351.0 (N-Si)	-79.7
3	2.45 (s, CH_3); 2.77 (t, $^{3}J = 5.8$, NCH_2); 3.40 (s, CH_2); 3.72 (t, $^{3}J = 5.8$, OCH_2); 6.14 (s, H-3); 6.96 (dd, $^{3}J = 8.2$, $^{3}J = 7.1$, H-6); 7.01 (dd, $^{3}J = 8.7$, $^{3}J = 7.1$, H-5); 7.42 (d, $^{3}J = 8.7$, H-4); 7.46 (d, $^{3}J = 8.2$, H-7)	13.24 (CH_3); 34.85 (CH_2); 50.88 (NCH_2); 57.09 (OCH_2); 97.46 (C-3); 111.32 (C-7); 117.71 (C-6); 118.55 (C-5); 118.74 (C-4); 127.59 (C-9); 137.45 (C-8); 138.28 (C-2)	-351.9 (N-Si)	-77.6
4	2.87 (t, $^{3}J = 5.8$, NCH_2); 3.37 (s, CH_2); 3.79 (t, $^{3}J = 5.8$, OCH_2); 7.17 (ddd, $^{3}J = 8.1$, $^{4}J = 7.1$, $^{4}J = 1.3$, H-5); 7.21 (ddd, $^{3}J = 8.1$, $^{4}J = 7.1$, $^{4}J = 1.3$, H-6); 7.43 (ddd, $^{3}J = 8.1$, $^{4}J = 1.3$, $^{5}J = 0.7$, H-4); 7.71 (ddd, $^{3}J = 8.1$, $^{4}J = 1.3$, $^{5}J = 0.7$, H-7); 8.10 (s, H-2)	35.05 ($^{1}J_{C\text{-}Si} = 110.6$, CH_2); 50.71 (NCH_2); 56.94 (OCH_2); 110.64 (C-7); 119.22 (C-4); 120.76 (C-5); 121.54 (C-6); 135.88 (C-8); 143.23 (C-9); 145.02 (C-2)	-142.9 (N-3); -255.5 (N-1); -350.6 (N-Si)	-80.1
5	2.87 (t, $^{3}J = 5.9$, NCH_2); 3.49 (s, CH_2); 3.78 (t, $^{3}J = 5.9$, OCH_2); 7.77 (s, H-3); 8.16 (s, H-5)	40.86 ($^{1}J_{C\text{-}Si} = 105.8$, CH_2); 50.77 ($^{1}J_{C\text{-}C} = 35.0$, NCH_2); 56.86 ($^{1}J_{C\text{-}C} = 35.0$, OCH_2); 143.37 (C-5); 149.69 (C-3)	-79.1 (N-2); -132.1 (N-4); -160.1 (N-1); -350.6 (N-Si)	-81.2
6	2.60 (s, H-3,4); 2.80 (t, $^{3}J = 5.9$, NCH_2); 2.90 (s, CH_2); 3.74 (t, $^{3}J = 5.9$, OCH_2)	28.31 (C-3,4); 30.34 (CH_2); 51.23 (NCH_2); 57.36 ($^{1}J_{C\text{-}C} = 35.0$, OCH_2); 177.47 (C=O)	-200.1 (N-1); -354.9 (N-Si)	-78.7
7	1.83 (m, H-4); 2.57 (t, $^{3}J = 6.6$, H-3,5); 2.76 (t, $^{3}J = 5.9$, NCH_2); 3.24 (s, CH_2); 3.72 (t, $^{3}J = 5.9$, OCH_2)	17.43 (C-4); 31.05 (CH_2); 33.25 (C-3,5); 51.35 (NCH_2) 57.57 (OCH_2); 172.41 (C=O)	-201.9 (N-1); -356.4 (N-Si)	-77.0
8	2.81 (t, $^{3}J = 6.0$, NCH_2); 3.07 (s, CH_2); 3.77 (t, $^{3}J = 6.0$, OCH_2); 7.59 (dd, $^{3}J = 5.3$, $^{3}J = 3.0$, H-5,6); 7.77 (dd, $^{3}J = 5.3$, $^{3}J = 3.0$, H-4,7)	29.23 ($^{1}J_{C\text{-}Si} = 111.7$, CH_2); 51.22 (NCH_2); 57.34 (OCH_2); 122.46 (C-4,7); 132.87 (C-5,6); 133.07 (C-8,9); 168.68 (C=O)	-221.0 (N-1); -351.6 (N-Si)	-78.4
9	3.54 (OCH_3); 4.14 (CH_2); 7.26 (d, $^{3}J = 2.0$, H-4); 7.38 (d, $^{3}J = 2.0$, H-5)	32.31 (CH_2); 50.30 (OCH_3); 140.79 (C-2); 114.49 (C-4); 123.32 (C-5)	-112.0 (N-3); -201.1 (N-1)	-55.6
10	2.10 (s, CH_3 (C-3)); 2.12 (s, CH_3 (C-5)); 3.49 (s, OCH_3); 3.50 (s, CH_2); 5.66 (s, H-4)	10.93 (CH_3 (C-5)); 13.27 (CH_3 (C-3)); 34.18 (CH_2); 50.80 (OCH_3); 104.40 (C-4); 138.78 (C-5); 143.18 (C-3)	-80.6 (N-2); -187.3 (N-1)	-54.1
12	3.36 (s, OCH_3); 3.47 (s, CH_2); 7.11 (m, H-5,6); 7.24 (d, $^{3}J = 8.1$, H-4); 7.65 (d, $^{3}J = 8.1$, H-7); 7.81 (s, H-2)	28.37 (CH_2); 50.37 (OCH_3); 109.21 (C-7); 119.46 (C-4); 121.24 (C-5); 122.10 (C-6); 134.38 (C-8); 142.95 (C-9); 143.07 (C-2)	-140.5 (N-3); -238.3 (N-1)	-54.5
13	3.39 (s, OCH_3); 3.65 (s, CH_2); 7.72 (s, H-3); 7.95 (s, H-5)	34.23 (CH_2); 50.66 (OCH_3); 143.29 (C-5); 150.81 (C-3)	-82.8 (N-2); -132.7 (N-4); -173.1 (N-1)	-55.7
14	2.53 (s, H-3,4); 2.88 (s, CH_2); 3.40 (s, OCH_3)	22.76 (CH_2); 27.89 (C-3,4); 50.52 (OCH_3); 176.54 (C=O)	-208.3 (N-1)	-53.5
15	1.82 (m, H-4); 2.54 (m, H-3,5); 3.25 (s, CH_2); 3.45 (s, OCH_3)	17.05 (C-4); 24.09 (CH_2); 32.50 (C-3,5); 50.65 (OCH_3); 172.05 (C=O)	-208.9 (N-1)	-52.3
16	3.24 (CH_2); 3.52 (OCH_3); 7.65 (dd, $^{3}J = 5.5$, $^{3}J = 3.1$, H-5,6); 7.77 (dd, $^{3}J = 5.5$, $^{3}J = 3.1$, H-4,7)	22.90 ($^{1}J_{C\text{-}Si} = 111.7$, ($CH_2$)); 50.75 ($OCH_3$); 122.91 (C-4,7); 133.72 (C-5,6); 132.22 (C-8,9); 168.07 (C=O)	-225.0	-61.3

TABLE 4. ^{13}C and ^{15}N NMR Spectra of Compounds **17-22**

Compound	Formula	Chemical shifts, δ, ppm *	
		^{13}C [9]	^{15}N [8]
17		119.7 (C-5); 129.3 (C-4); 137.6 (C-2)	-219.5 (N-1); -124.3 (N-3)
18		105.1 (C-4); 129.5 (C-5); 138.7 (C-3)	-73.7 (N-2); -179.3 (N-1)
19		100.0 (C-3); 109.2 (C-7); 119.6 (C-6); 120.7 (C-5); 120.0 (C-4); 128.9 (C-9); 137.0 (C-8); 138.1 (C-2)	-253.6
20		108.9 (C-7); 119.7 (C-4); 121.5 (C-5); 122.4 (C-6); 134.1 (C-8); 143.1 (C-2); 143.2 (C-9)	-136.3 (N-3); -236.4 (N-1)
21		142.7 (C-5); 150.7 (C-3)	-82.2 (N-2); -129.1 (N-4); -171.3 (N-1)
22		28.7 (C-3,4); 177.7 (C=O)	-199.3 (N-1)

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*^{13}C NMR spectra were taken in $CDCl_3$, ^{15}N NMR spectra in $CDCl_3$ (compounds **17** and **18**) and DMSO-d_6 (compounds **19-22**).

600-650 cm^{-1} are a doublet (607, 634 cm^{-1}) (Table 2). This corresponds to the data of UV spectroscopy, indicating the presence of two structures for the molecule of compound **8**. The group frequencies in the IR spectra of (N-heterylmethyl)trimethoxysilanes **10-13**, **16** did not depend in practice on the structure of the heterocycle.

The ^{1}H, ^{13}C, ^{15}N, and ^{29}Si NMR spectra of the N-(1-silatranylmethyl) derivatives compounds **1-8** are given in Table 3.

The chemical shifts of the ^{1}H and ^{13}C nuclei of the heterocyclic fragment (Het) of compounds **1-8** (Table 3) differed little from the corresponding N-methyl derivatives of the azoles (Table 4) [8, 9]. In the ^{13}C NMR spectra of compounds **1-8** the direct ^{13}C coupling constant in the exocyclic NCH_2Si fragment with ^{29}Si ($^{1}J_{C\text{-}Si}$ ~106-112 Hz), was characteristic for silatranes (Table 3).

Among the ^{15}N NMR signals the most sensitive to the influence of the silatranylmethyl substituent was the signal of the ^{15}N nitrogen atom at position 1 (N-1), which was changed by 0.6-20.0 ppm (Table 3) in comparison with the analogous signal in the spectra of the 1-methylazoles (Table 4) [8]. Other ^{15}N signals for N-2, N-3, and N-4 differed little from the signals in the spectra of the corresponding 1-methylazole derivatives. With an increase in the electron-withdrawing effect of the heterocyclic substituent (depending on the number, disposition, and character of the bonding of the endocyclic nitrogen atoms) the shielding of the ^{29}Si nucleus and the deshielding of the ^{15}N atom in the silatrane framework were increased. Such a dependence of δ ^{15}N and δ ^{29}Si is characteristic of the XCH_2Sa silatrane series [10]. In the series of compounds studied the chemical shifts of ^{29}Si and ^{15}N nuclei of the silatrane fragment changed in an interlinked manner, depending on the substituent Het (Table 3).

The increase in the shielding of the ^{29}Si nuclei in compounds **7**, **3**, **8**, **4**, and **5** is accompanied by a proportional reduction in the shielding of the ^{15}N nuclei (Table 3). The close values of the chemical shifts of the

^{29}Si (-77 to -81 ppm) and ^{15}N nuclei (-350 to -356 ppm) correspond to the insignificant differences in the length of the transannular N→Si bond in the silatrane framework in compounds **1**, **2**, **4**, and **5** [11-13]. This indicates that in these molecules the electron-withdrawing inductive effects of the Het and HetCH$_2$ substituents are extremely close. The latter observation must be reflected in the complex-forming ability of 1-(N-heterylmethyl)silatranes.

EXPERIMENTAL

The ^{1}H, ^{13}C, ^{15}N, and ^{29}Si NMR spectra of the synthesized compounds were obtained on a Bruker DPX-400 spectrometer (resonance frequencies 400, 101, 41, and 80 MHz, respectively) in CDCl$_3$ at room temperature, internal standard was TMS for ^{1}H, ^{13}C, and ^{29}Si nuclei and Me^{15}NO$_2$ for ^{15}N nuclei. The impulse sequence INEPT was used to obtain ^{29}Si NMR spectra. The ^{15}N NMR chemical shifts were measured from inverse two-dimensional ^{1}H-^{15}N spectra by the HMBC-GP procedure. The precision of measuring chemical shifts of ^{1}H and ^{13}C nuclei was 0.01 and 0.02 ppm, respectively, and 0.1 ppm for ^{15}N and ^{29}Si nuclei. The UV spectra of solutions of compounds were recorded on a Specord UV-vis spectrophotometer, and the IR spectra on a Specord IR 75 spectrometer in microfilms or in KBr disks.*

The N-(trimethoxysilylmethyl) and N-(silatranylmethyl) derivatives of imidazole (**1**, **9**), 3,5-dimethylpyrazole (**2**, **10**), 2-methylindole (**3**, **11**), benzimidazole (**4**, **12**), 1,2,4-triazole (**5**, **13**), succinimide (**14**), glutarimide (**15**), and phthalimide (**8**, **16**) were obtained by methods described previously [5-7].

N-(1-Silatranylmethyl)succinimide (6). A mixture of compound **14** (4.31 g, 17 mmol) and triethanolamine (2.54 g, 17 mmol) was stirred at 25°C for 10 min. The precipitated white solid was recrystallized from chloroform-hexane, 1 : 1. Compound **6** (5.01 g, 89%) was obtained having mp 267°C. Found, %: C 46.02; H 6.08; N 9.93; Si 9.63. $C_{11}H_{18}N_2O_5Si$. Calculated, %: C 46.14; H 6.34; N 9.78; Si 9.81.

N-(1-Silatranylmethyl)glutarimide (7) was obtained analogously. Mp 238°C, yield 90%. Found, %: C 47.86; H 6.62; N 9.39; Si 9.46. $C_{12}H_{20}N_2O_5Si$. Calculated, %: C 47.98; H 6.71; N 9.32; Si 9.35.

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