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Transsilylation of *O*-trimethylsilyl derivatives of α-dimethylaminoketones by chloro(chloromethyl)dimethylsilane

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A R T I C L E I N F O

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Dedicated to Professor Irina P. Beletskaya on the occasion of her birthday.

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A B S T R A C T

Reaction of *O*-trimethylsilyl (*O*-TMS) derivatives of α-dimethylamino ketones with chloro(chloromethyl) dimethylsilane led to the formation of six-membered heterocyclic compounds with tetracoordinated silicon atom - 3,4-dihydro-2*H*-1,4,2-oxazasilin-4-ium chlorides. The exchange reactions of 2,2,4,4-tetramethyl-6-phenyl-3,4-dihydro-2*H*-1,4,2-oxazasilin-4-ium chloride were examined with Me_3SiI and $NaBPh_4$ and the corresponding iodide or tetraphenylborate of 2,2,4,4-tetramethyl-6-phenyl-3,4-dihydro-2*H*-1,4,2-oxazasilin-4-ium were isolated. 2,2,4,4-Tetramethyl-6-phenyl-3,4-dihydro-2*H*-1,4,2-oxazasilin-4-ium chloride easy reacts with MeOH to give *N*-((methoxydimethylsilyl)methyl)-*N*,*N*-dimethyl-2-oxo-2-phenylethanaminium chloride. The structure of synthesized compounds **10**–**15** was proved by multinuclear NMR spectroscopy, **11** was also characterised by X-ray crystallography.

1. Introduction

Silanes are important starting compounds for the production of zeolites, ceramics, silica based organic–inorganic hybrid materials obtained by using the sol–gel method and they are widely used to prepare protective silica films for electronic devices by using the chemical vapor deposition (CVD) [1–10]. Organosilicon materials have gained widespread use in the chemical and electrical engineering, electronics, aerospace industry, medicine due to their unique properties, such as high- and low-temperature resistances, weather resistance, ozone resistance, ageing resistance, electrical insulation and physiological inertia [11–14]. Silanes containing geminal N-C-Si group are promising candidates for creation of new materials as well as their application as cross-linking agents for polysiloxanes and organic polymers [15–26]. *N*-Silylmethylamine, -amides and related compounds are a matter for extensive synthetic and structural investigations. The reactions of transsilylation of *N*-TMS derivatives of lactams, ureas, and carboxamides by polyfunctional silanes lead to formation of corresponding *N*-siylmethyl derivatives. The investigation of the structure of products and mechanism of these reactions is of profound importance for both fundamental (theoretical, synthetic, physico-chemical) and applied (material science, applications) aspects of modern organosilicon chemistry [27–37].

Previously we found that the *O*-TMS- *N*′,*N*′-dimethylhydrazides **1** have been reacted with polyhalogenosilanes to give the corresponding neutral, hexacoordinate chelates **2** (Scheme 1) [36,38,39]. The reaction of compounds **1** with chloro(chloromethyl)dimethylsilane **3** was finished by the formation of the cyclic ionic compounds **4** (Scheme 2) [40,41].

The electronic distribution of molecules of compounds **4** could formally be represented by a set of canonical structures **A-D** (Scheme 3) [37] in which the negative charge can be located on the second hydrazide nitrogen atom or on the oxygen atom or the silicon atom or the chlorine atom.

The reaction of dimethylaminomethyl ketones $Me_2NCH_2C(O)R$ (R = *t*-Bu, Ph) with trimethylsilyl trifluoromethanesulfonate ($TfOSiMe_3$) in the presence of triethylamine smoothly afforded their corresponding *O*-TMS derivatives **5** and **6**. Similar to the compounds **1,** the *O*-TMS-derivatives of α-dimethylaminoketones **5, 6** interact with polyhalogenosilanes to form isomorphic ionic compounds **7**–**9** containing two N→Si chelate cycles (Scheme 4) [42].

Herein, we report on the preparation, characterization and reactivity of novel ionic heterocyclic organosilicon compounds obtained by the interaction of chloro(chloromethyl)dimethylsilane

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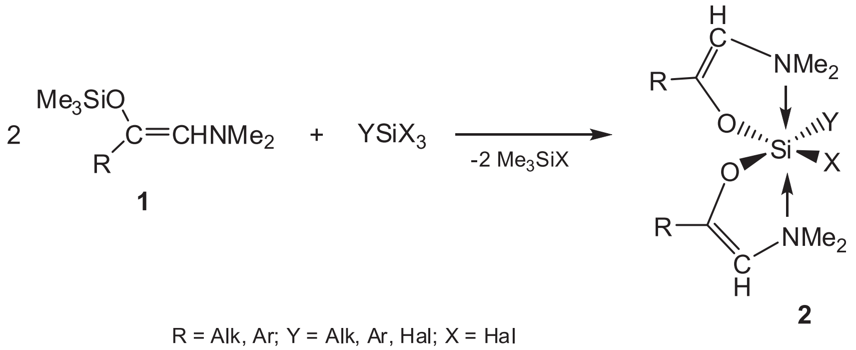
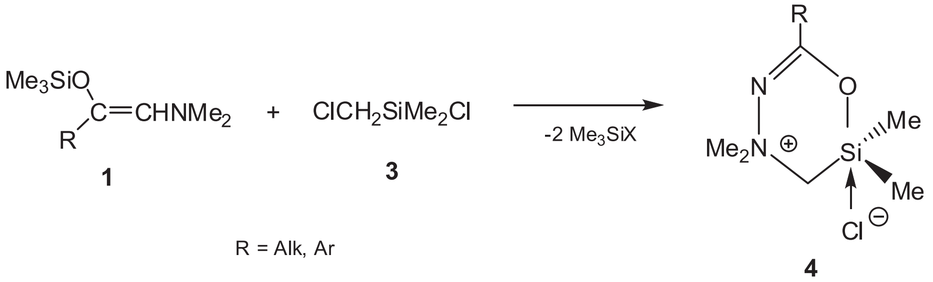
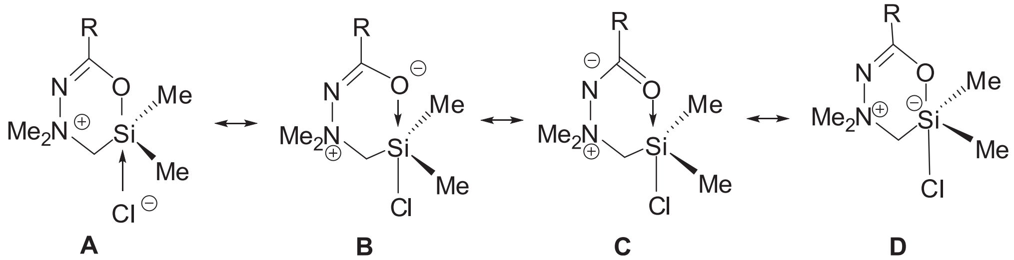


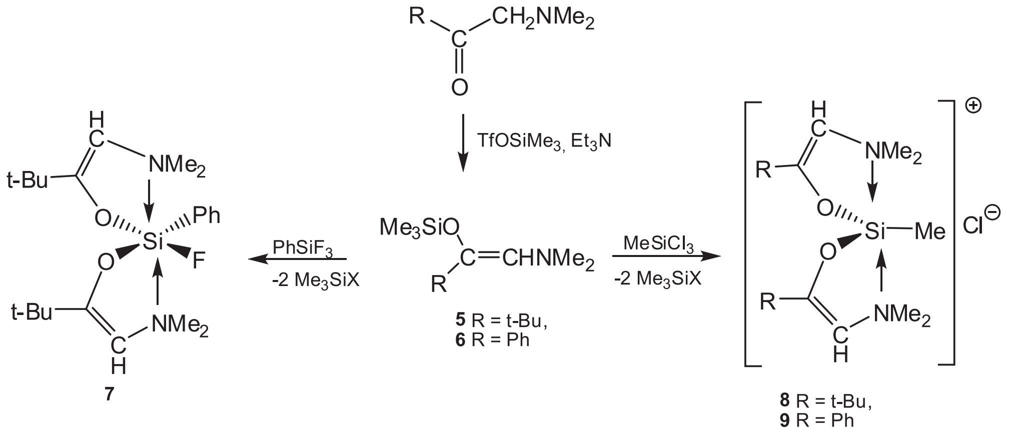
Scheme 1. Reaction of *O*-TMS derivatives of *N*′,*N*′-dimethylhydrazides of carboxylic acids **1** with polyhalogenosilanes.



Scheme 2. The formation of the cyclic ionic compounds **4**.



Scheme 3. The canonical structures of compounds **4**.



Scheme 4. The reaction of *O*-TMS-derivatives of α-dimethylaminoketones **5, 6** with polyhalogenosilanes.

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3 with *O*-TMS-derivatives of α-dimethylaminoketones **5**, **6**.

2. Results and discussion

Compounds **5**, **6** react with chloro(chloromethyl)dimethylsilane **3** in chloroform or acetonitrile solutions to form 6-*tert*-butyl-2,2,4,4-tetramethyl-3,4-dihydro-2*H*-1,4,2-oxazasilin-4-ium chloride **10** and 2,2,4,4-tetramethyl-6-phenyl-3,4-dihydro-2*H*-1,4,2-oxazasilin-4-ium chloride **11**, respectively (Scheme 5). This reaction proceeds only upon heating to 80–90 °C in contrast to the transsilylation reaction of *O*-TMS derivatives of *N′*,*N′*-dimethylhydrazides of carboxylic acids **1** by silane **3** taking place at room temperature [40].

The structure of compounds **10** and **11** was confirmed by ^{1}H, ^{13}C and ^{29}Si NMR spectroscopy. The ^{29}Si NMR spectra of compounds **10** and **11** in $CDCl_3$ showed the single resonance (18.33 ppm and 18.78 ppm, respectively). Their pronounced displacement in region of down field shifts in comparison with ^{29}Si chemical shift of compound **4** (δ $^{29}Si = -17.4$ ppm.) indicates that the silicon atom is tetracoordinated.

The single crystal of compound **11** suitable for X-ray analysis was obtained by its crystallization from MeCN. The summary of crystallographic data is provided in experimental section. The bond distances and bond angles are presented in Tables 1S and 2S (Supporting information). A comparison of the molecular structures of compound **11** (Fig. 1 and Table 1) and the isostructural pentacoordinated silicon compound **4** ($R = 4\text{-MeO-}C_6H_4$) [41] (Table 1) shows the substantial differences in their geometric parameters. The Si-Cl bond length in compound **11** is substantially larger than in compound **4** (3.108 Å and 2.624 Å, respectively) and is closely approximating the sum of the Van der Waals radii of the Si and Cl atoms. The Si-O bond length in compound **11** is significantly shorter than in compound **4** (1.713 Å and 1.788 Å, respectively) and this value is only 0.03 Å longer than the value of Si-O covalent bond length in compounds of tetrahedral silicon atom. The values of O-Si-Cl angles in compounds **4** and **11** are closely (173.8° and 173.6°, respectively). The sum of bond angles C-Si-C in **11** differ significantly from the sum of the equatorial bond angles C-Si-C in **4** (348.44° and 357.29°, respectively). The displacement of the silicon atom relative to the equatorial plane defined by three carbon atoms towards the O atom substituent approximated 0.178 Å and 0.728 Å for compounds **4** [41] and **11**, respectively (see Figs. 1 and 2 in Supporting information). The silicon atom in compound **11** has a tetrahedral geometry common to tetracoordinated silicon compounds in contrast to distorted trigonal-bipyramidal geometry of silicon atom in compound **4**. Based on these results, it may be concluded that compound **11** exists as ionic compound with tetracoordinated silicon atom.

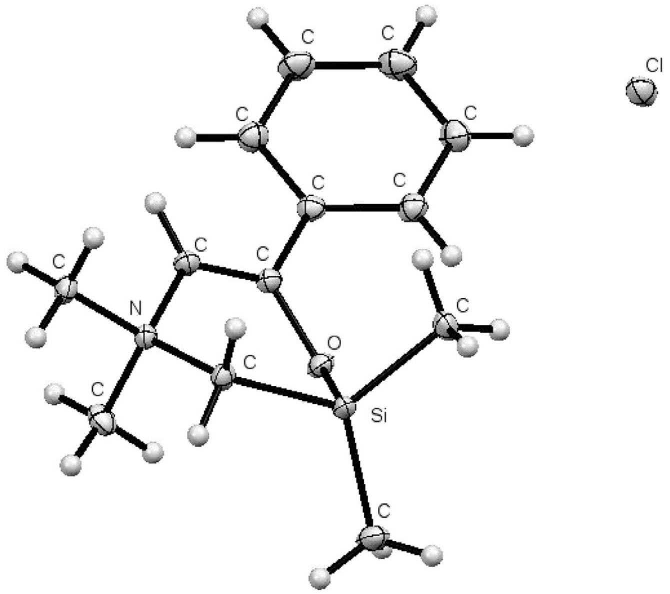
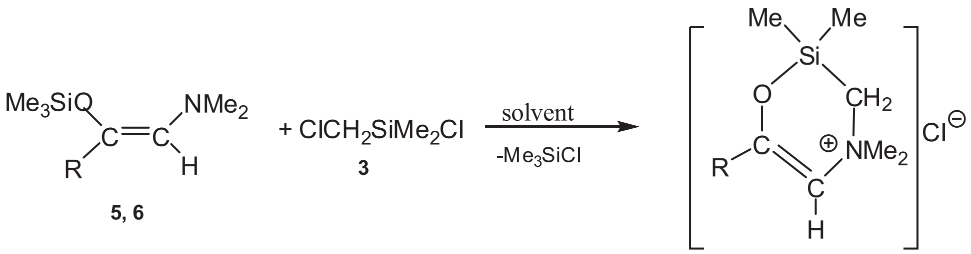


Fig. 1. Molecular structure of **11** in the crystal. Anisotropic displacement parameters are depicted at the 50% probability level, and hydrogen atoms are omitted for clarity.

Table 1
The selected geometrical parameters of compounds **4** and **11**.

Compound	l, Å				$\Sigma_{C\text{-}Si\text{-}C,}$ °	Ref.
	Si-Cl	Si-O	Si-CH_2	Si-Me		
4	2.624(8)	1.788(1)	1.885(2)	1.849(2) 1.885(2)	357.29	[41]
114	3.108(2)	1.713(9)	1.900(1)	1.856(1) 1.865(1)	348.44	this work

The compounds **10**, **11** contain simultaneously silyl enol ether group and a fragment of a vinyl ammonium salt. Such unique structure opens up new avenues for the future development of syntetic organic chemistry. Considering the high reactivity of silyl enol ethers and their wide application in chemistry [43–51] it was interesting to study the reactivity of these new compounds. This part of our investigation was performed using compound **11**. Its interaction with trimethyl iodosilane, much like the interaction of Me_3SiI with *N*-[chloro(dimethylsilyl)methyl]lactams [27], is a typical exchange reaction and leads to the quantitative formation of the corresponding 2,2,4,4-tetramethyl-6-phenyl-3,4-dihydro-2*H*-1,4,2-oxazasilin-4-yl iodide **12** (Scheme 6). It has been known that



5, 6

3

10, R = t-Bu ($CHCl_3$, 40 h, 47%);
11, R = Ph (CH_3CN, 3 h, 82%)

Scheme 5. The transsilylation reaction of *O*-TMS-derivatives of α-dimethylaminoketones **5**, **6** by with chloro(chloromethyl)dimethylsilane.

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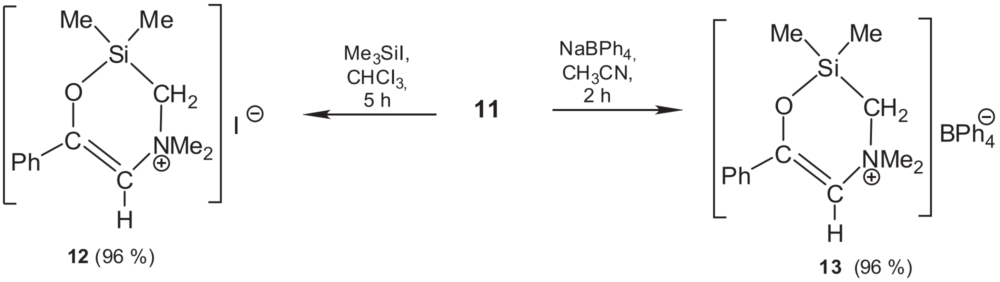


Scheme 6. Synthesis of compounds **12** and **13**.

trimethyliodisilane causes the cleavage of the Si-*O*-C bond to form siloxanes [52–54]. However, under the conditions of our experiment the products of the cleavage of Si-*O*-C bond not were observed. Apparently this is due that the rate of the halogen exchange reaction is substantially higher than the rate of the bond cleavage reaction. Compound **11** reacts with $NaBPh_4$ with forming the corresponding 2,2,4,4-tetramethyl-6-phenyl-3,4-dihydro-2*H*-1,4,2-oxazasilin-4-yl tetraphenylborate **13** (Scheme 6).

The addition of methanol to compound **11** runs at 80 °C without use of any catalyst and finish by the formation of *N*-((methoxydimethylsilyl)methyl)-*N*,*N*-dimethyl-2-oxo-2-phenylethanaminium chloride **14** (Scheme 7). Notice that signal in ^{29}Si NMR spectrum of **14** is shifted highfield compared to the compound **11** (12.53 ppm and 18.78 ppm, respectively). Similar changes in ^{29}Si NMR chemical shifts are observed in acyclic silyl enol ethers at the replacement of a alkenyloxy substituent by an alkoxy group. For example the highfield chemical shifts in ^{29}Si NMR spectra of $Me_2CCH_2CH_2OSiMe_3$ and $PhCH_2OSiMe_3$ (15.6 ppm and 17.4 ppm, respectively) [55] are observed compared to $Me_2C=CHOSiMe_3$ and $Me_2C=CPhOSiMe_3$ (18.7 ppm and 22.4 ppm, respectively) [56].

Interaction of compound **14** with boron trifluoride etherate in THF gave rise to *N*-((fluorodimethylsilyl)methyl)-*N*,*N*-dimethyl-2-oxo-2-phenylethanaminium chloride **15** with quantitative yield (Scheme 7).

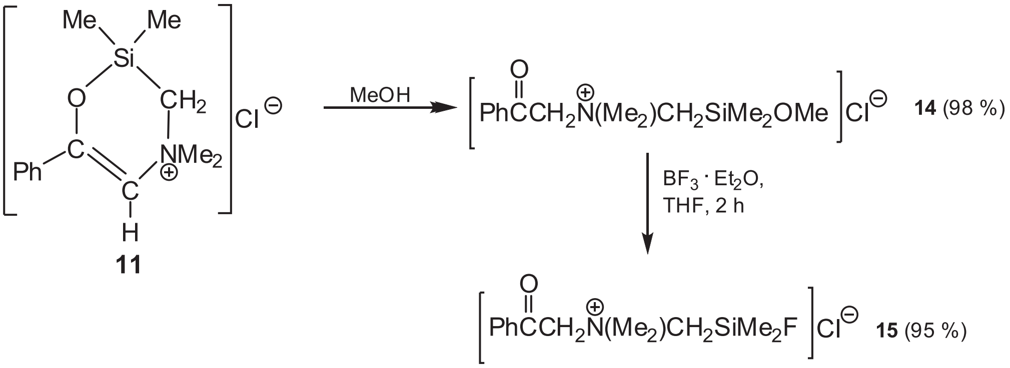
In conclusion, we have demonstrated that the transsilylation reaction of *O*-TMS α-dimethyamino ketones by $ClCH_2SiMe_2Cl$ is efficient approach for the synthesis of novel Si-containing heterocyclic compounds. Structure of these silanes is unique: simultaneously they are *N*-silylmethylamines, silyl enol ethers and vinyl ammonium salts. Further application of this strategy for the synthesis of novel Si-containing compounds with geminal *N*-C-Si group is currently in progress and we hope that results will be useful for the development of synthetic organic chemistry and the chemistry of materials.

3. Experimental section

3.1. General procedures

All experiments were carried out under a dry argon atmosphere using standard Schlenk techniques. Solvents were dried and freshly distilled under argon prior to use [57].

The NMR spectra ^{1}H, ^{13}C and ^{29}Si were recorded on a Bruker DPX-400 и Bruker AV-400 (400.13, 100.61 и 161.98 MHz, respectively). Chemical shifts (δ, ppm) are referenced to TMS = tetramethylsilane (δ = 0.0). For the assignment of the multiplicities the following abbreviations were used: s = singlet, d = doublet, m = multiplet. Carbon, hydrogen and nitrogen elemental analysis was performed on a Thermo Scientific Flash 2000 CHNS analyzer. The content of silicon, bor and halogene was determined thermogravimetrically. Crystal data were collected on a Bruker D8 Venture diffractometer with a Photon 100 detector using ω-2θ scanning (Table 2). The intensity of the reflections was integrated using the Bruker SAINT software. The absorption of X-ray by a crystal is taken into account from the analysis of the intensities of equivalent reflections. Only independent reflections were used after averaging the intensities of equivalent reflections. The structures were solved and refined by direct methods using the SHELX programs set [58]. Nonhydrogen atoms were refined anisotropically using SHELX [58]. **CCDC 1549966** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



Scheme 7. Synthesis of compounds **14** and **15**.

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Table 2
Experimental data for compound **11**.

CCDC № 1549966	
Formula	$C_{13}H_{20}NOSi$, Cl
Mr, g/mol	269.84
Temperature, K	100(2)
Crystal system	triclinic
Space group	P-1
a, Å	6.6889(5)
b, Å	8.8221(7)
c, Å	12.9551(11)
α, deg	77.662(3)
β, deg	76.194(3)
γ, deg	88.492(3)
V, Å^3	725.00(10)
Z	2
P_{calc}, Mg/m^3	1.236
F(000)	288.0
θ_{range}, deg	4.728 to 60.554
Ref. collected	44171
Independ. ref	4326
R_{int}	0.0305
Max. And min. broadcast	4326
Num. of refinement parameters	159
Goodness-of-fit on F2	1.047
R1 wR2 [I > 2σI]	$R_1 = 0.0278$, $wR_2 = 0.0738$
R1 wR2 [all data]	$R_1 = 0.0332$, $wR_2 = 0.0766$
(Δρ)max and (Δρ)min, e/Å3	0.41/-0.32

3.2. Experimental procedure

3.2.1. Synthesis of 6-tert-butyl-2,2,4,4-tetramethyl-3,4-dihydro-2H-1,4,2-oxazasilin-4-ium chloride (**10**)

The chloro(chloromethyl)dimethylsilane **3** (0.93 g, 6.40 mmol), 10 mL $CHCl_3$ were condensed into a Schlenk flask using a glass adapter and then the *O*-TMS α-aminoketone **5** (1.04 g, 4.80 mmol) was added in one portion. The mixture was stirred at 90 °C for 40 h. The solid residue obtained after removal of the volatile substances was washed with hexane (2 × 20 mL). The white powder was dried in vacuo. Yield 0.57 g (47%, 2.29 mmol). M. p. 173 °C (In a capillary under vacuum). ^1H ($CDCl_3$): δ 0.54 (s, 6 H, $SiMe_2$), 1.11 (s, 9 H, CMe_3), 3.73 (s, 6 H, NMe_2), 3.75 (s, 2 H, NCH_2), 5.90 (s, 1 H, =CH). ^{13}C ($CDCl_3$): δ −0.23 ($SiCH_3$), 26.99 [$C(\underline{C}H_3)_3$], 36.16 (CMe_3), 56.00 ($SiCH_2$), 58.92 (NCH_3), 113.68 (=CH). Anal. Calc. for $C_{11}H_{24}NOSiCl$: C 52.89; H 9.68; N 5.61; Si 11.24; Cl 14.19%. Found C 52.93; H 9.73; N 5.48; Si 11.32; Cl 13.97%.

3.2.2. Synthesis of 2,2,4,4-tetramethyl-6-phenyl-3,4-dihydro-2H-1,4,2-oxazasilin-4-ium chloride (**11**)

The chloro(chloromethyl)dimethylsilane **3** (0.60 g, 4.16 mmol), 5 mL CH_3CN were condensed into a Schlenk flask using a glass adapter and then the *O*-TMS α-aminoketone **6** (0.87 g, 3.70 mmol) was added in one portion. The mixture was stirred at 80 °C for 3 h. The solid residue obtained after removal of the volatile substances was washed with Et_2O (2 × 10 mL). The white powder was dried in vacuo. Yield 0.82 g (82%, 3.03 mmol). M. p. 160–162 °C (in a capillary under vacuum). ^1H ($CDCl_3$): δ 0.62 (s, 6 H, $SiMe_2$), 3.85 (s, 8 H, NMe_2, NCH_2), 6.86 (s, 1 H, =CH), 7.38 (m, 3H, $2H^m$, $1H^p$), 7.67 (m 2 H, H^o). ^{13}C ($CDCl_3$): δ - 0.10 ($SiCH_3$), 56.04 ($SiCH_2$), 59.17 (NCH_3), 115.05 (=CH), 125.65, 128.42 (C^o, C^m), 130.64 (C^p), 132.40 (C^i), 149.58 (PhCO). ^{29}Si ($CDCl_3$): δ 18.78. Anal. Calc. for $C_{13}H_{20}NOSiCl$: C 57.86; H 7.47; N 5.19; Si 10.41; Cl 13.14%. Found C 57.91; H 7.43; N 5.21; Si 10.53; Cl 13.21%.

3.2.3. Synthesis of 2,2,4,4-tetramethyl-6-phenyl-3,4-dihydro-2H-1,4,2-oxazasilin-4-ium iodide (**12**)

A mixture of compound **11** (1.22 g, 4.52 mmol), Me_3SiI (1.13 g, 5.65 mmol) and 10 mL $CHCl_3$ was stirred at 20 °C for 5 h. The solid residue obtained after removal of the highly volatile substances was washed with Et_2O (3 × 20 mL). The slightly cream-colored powder was dried in vacuo. Yield 1.58 g (96%, 4.37 mmol). M. p. 148 °C (in a capillary under vacuum). ^1H ($CDCl_3$): δ 0.65 (s, 6 H, $SiMe_2$), 3.83 (s, 8 H, NMe_2, NCH_2), 6.79 (s, 1 H, =CH), 7.37–7.41 (m, 3 H, 2 H^o, H^p), 7.69–7.71 (m, 2 H, H^m). ^{13}C ($CDCl_3$): δ −0.11 ($SiCH_3$), 56.53 (CH_2), 59.37 (NCH_3), 115.00 (=CH), 125.64, 128.66 (C^o, C^m), 130.69 (C^p), 132.02 (C^i), 149.69 (=CO). ^{29}Si ($CDCl_3$): δ 19.09. Anal. Calc. for $C_{13}H_{20}NOSiI$: C 43.22; H 5.58; N 3.88; Si 7.77%. Found C 43.31; H 5.46; N 3.67; Si 7.85%.

3.2.4. Synthesis of 2,2,4,4-tetramethyl-6-phenyl-3,4-dihydro-2H-1,4,2-oxazasilin-4-ium tetraphenylborate (**13**)

A mixture of compound **11** (0.91 g, 4.63 mmol), $NaBPh_4$ (1.44 g, 5.43 mmol) and 20 mL CH_3CN was stirred at 90 °C for 2 h. The solution was decanted carefully and residue was washed by CH_3CN (20 mL). The combined solution was evaporated under reduced pressure, the solid residue was washed with Et_2O (2 × 30 mL). The white powder was dried in vacuo. Yield 1.793 g (96%, 3.24 mmol). M. p. 175–176 °C with decomposition. ^1H (CD_3CN): δ 0.48 (s, 6 H, $SiMe_2$), 3.02 (s, 2 H, CH_2), 3.05 (s, 6 H, NMe_2), 6.17 (s, 1 H, =CH), 6.82–6.86 (m, 4 H, BPh_4, H^p), 6.98–7.01 (m, 8 H, BPh_4, H^m), 7.30 (br, 9 H, 1 H^p; BPh_4, 8 H^o), 7.39–7.43 (m, 2 H, H^m), 7.56–7.58 (m, 1 H, H^o). ^{13}C (CD_3CN): δ −0.61 (SiC), 56.65 (CH_2), 59.43 (NCH_3), 115.38 (=CH), 122.75 (BPh_4, C^p), 126.56 (C^m), 126.58 (BPh_4, C^m) 129.68 (C^o), 131.55 (C^p), 133.59 (C^i), 136.70 (BPh_4, C^o), 150.58 (PhCO), 164.76 (q, J_{CB} = 49.3 Hz, BPh_4, C^i). ^{29}Si (CD_3CN): δ 19.46. Anal. Calc. for $C_{37}H_{40}NOSiB$: C 80.27; H 7.28; N 2.53; Si 5.07; B 1.95%. Found C 80.31; H 7.16; N 2.41; Si 5.26; B 1.65%.

3.2.5. Synthesis of N-((Methoxydimethylsilyl)methyl)-N,N-dimethyl-2-oxo-2-phenylethanaminium chloride (**14**)

A mixture of compound **11** (0.42 g, 1.56 mmol) and 5 mL MeOH (excess) was stirred at 80 °C for 1 h. The volatile matter was removed under reduced pressure. The obtained residue was washed with THF (20 mL), than Et_2O (20 mL) and colorless resin was dried in vacuo. Yield 0.46 g (98%, 1.52 mmol). ^1H ($CDCl_3$): δ 0.32 (s, 6 H, $SiMe_2$), 3.43 (s, 3 H, OCH_3), 3.73 (s, 2 H, $SiCH_2$), 3.75 (s, 6 H, NCH_3), 5.96 (s, 2 H, CH_2CO), 7.46–7.50 (m, 2 H, H^m), 7.58–7.61 (m, 1 H, H^p), 8.01–8.09 (m, 2 H, H^o). ^{13}C ($CDCl_3$): δ −1.25 ($SiCH_3$), 50.84 (OCH_3), 55.49 (NCH_3), 56.76 ($SiCH_2$), 69.58 ($\underline{C}H_2CO$), 126.66 (C^i),128.40 (C^o), 129.11 (C^m), 134.67 (C^p), 192.19 (C = O). ^{29}Si ($CDCl_3$): δ 12.53. Anal. Calc. for $C_{14}H_{24}NO_2SiCl$: C 55.70; H 8.01; N 4.64; Si 9.30; Cl 11.74%. Found C 55.38; H 8.27; N 4.38; Si 8.97; Cl 11.43%.

3.2.6. Synthesis of N-((fluorodimethylsilyl)methyl)-N,N-dimethyl-2-oxo-2-phenylethanaminium chloride (**15**)

A mixture of compound **14** (1.29 g, 4.26 mmol), $BF_3 \cdot Et_2O$ (0.94 g, 6.62 mmol) and 20 mL THF was stirred at 80 °C four 2 h. The volatile matter was removed under reduced pressure and obtained residue was washed with mixture of Et_2O and THF (~2: 1, 2 × 20 mL). The colorless resin was dried in vacuo. Yield 1.18 g (95%, 4.07 mmol). ^1H ($CDCl_3$): δ 0.41 (d, $^3J_{HF}$ = 7.15 Hz, 6 H, $SiMe_2$), 3.43 (s, 6 H, NMe_2), 3.60 (br s, 2 H, CH_2Si), 5.08 (s, 2 H, $CH_2C = O$), 7.42–7.45 (m, 2 H, H^m), 7.58–7.62 (m, 1 H, H^p), 7.91–7.93 (m, 2 H, H^o). ^{13}C ($CDCl_3$): δ −0.75 (d, $^2J_{CF}$ = 13.38 Hz, $SiCH_3$), 55.33 (NCH_3), 56.98 (d, $^2J_{CF}$ = 14.15 Hz, CH_2Si), 69.12 ($\underline{C}H_2C = O$), 128.13 (C^o), 129.13 (C^m), 133.94 (C^i), 135.04 (C^p), 190.86 (C = O). ^{29}Si ($CDCl_3$): δ 26.27 (d, $J_{Si\text{-}F}$ 285.79 Hz). Anal. Calc. for $C_{13}H_{21}NOSiFCl$: C 53.87; H 7.30; N 4.83; Si 9.69%. Found C 53.80; H 7.46; N 4.56; Si 9.34%.

These results were obtained using analytical equipment of the Baikal Analytical Center for collective use of the SB RAS.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.jorganchem.2018.01.014.

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