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Electrophilic Cyclization of Dimethyl(ω-phenylaminoalkyl)alkenylsilanes

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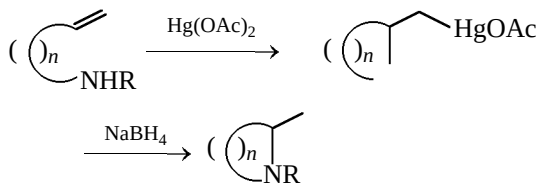
Abstract — Unsaturated organosilicon amines $[CH_2{=}CH(CH_2)_n]Me_2Si[(CH_2)_mNHPh]$ at the action of $Hg(OAc)_2$ in THF solution transform to corresponding heterocycles. Regioselectivity of this intramolecular electrophilic heterocyclization is defined by the position of double bond relatively to the silicon atom. This is caused by β-effect of the silyl group.

The capability of trialkylsilyl group to stabilize neighboring β-carbocation ("β-effect" of silicon) defines regioselectivity of addition of electrophiles to the double bond of alkenylsilanes [1–4]. Aniline is added to trimethylvinylsilane in the presence of mercury acetate (followed by reduction of the organomercury intermediate with sodium borohydride) forming adduct with phenylamino group at the terminal carbon atom. Reaction of aniline with trimethylallylsilane under the similar conditions leads to a non-terminal adduct $Me_3SiCH_2CHNHPhCH_3$ [5]. Effect of a dimethylsilyl group introduced to the alkenylamine chain on the regioselectivity of intramolecular electrophilic addition of NH group was not studied yet. The only known is transformation of dimethyl(2-phenylaminopropyl)allylsilane to a mixture of *cis*- and *trans*-1-phenyl-2,4,4,6-tetramethyl-4-silapiperidines [5].

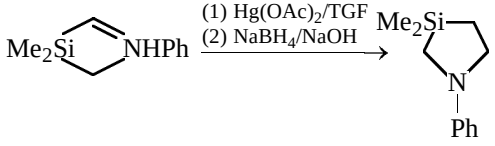
The target of this work is the study of electrophilic intramolecular cyclization of organosilicon amines $[CH_2{=}CH(CH_2)_n]Me_2Si[(CH_2)_mNHPh]$ (n = 0, 1; m = 1–3) differ by the position of amino group and double bond relatively to the silicon atom.

In a general case, addition of electrophilic agents, e.g., mercury acetate, to alkenylamine proceeds according to the Markownikoff rule and leads, after demercuration, to the corresponding C-methyl substituted heterocycles [6].



Structure of the formed heterocycle depends on the stereoelectronic and thermodynamic factors, including nature of the substituents at the double bond and extent of alkenyl chain [6, 7]. Thus, the rigity of a fourmembered ring restricts its formation at the cyclization of *N*-propyl-3-butenylamine and results in selective transformation of the latter into a fivemembered heterocycle [8].

We found that ring closure by vinyl(phenylaminomethyl)dimethylsilane (**I**) also proceeds contrary to the Markownikoff rule. At the action on it of mercury acetate in a THF solution followed by demercuration with an alkaline solution of sodium borohydride was obtained a substituted 3-silapyrrolidine (**II**) (yield 58%).



(1)

When competitive formation of isomeric five- and sixmembered heterocycles is possible, the cyclization regioselectivity is defined by the mutual positions of double bond and silicon atom. Under the conditions of aminomercuration-demercuration, allyl(phenylaminomethyl)dimethylsilane (**III**) easily forms *C*-methyl-substituted fivemembered heterocycle (**IV**) [reaction (2)]. Similarly proceeds cyclization of *N*-alkyl-4-pentenylamines, which under the action of mercury chloride or acetate followed by reduction with sodium borohydride transform into the derivatives of 2-methylpyrrolidine [8].

In contrast, intramolecular ring closure by vinyl(2-phenylaminoethyl)dimethylsilane (**V**) results in formation of substituted 4-silapiperidine (**VI**). However, it is formed with 3% yield only. {It is noteworthy that *N*-alkyl-4-silapiperidines are also

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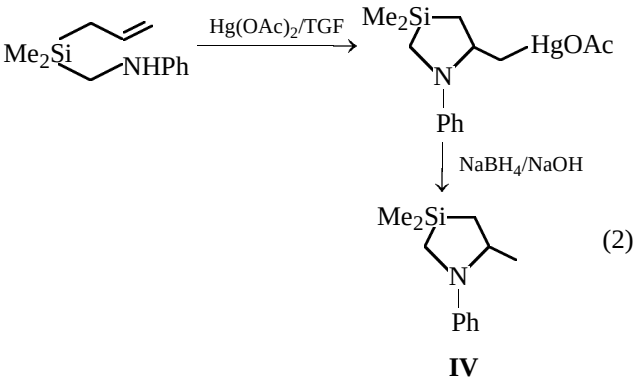
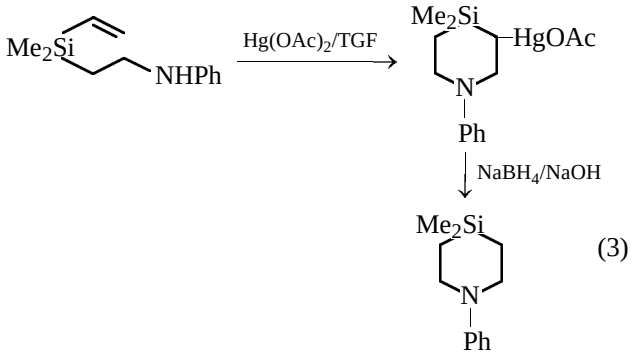


(2)

IV

formed in such a low yield (2–5%) at the addition of alkylamine lithium derivatives to diphenyldivinylsilane [9].} The predominate reaction product is 1,1,3,3-tetramethyl-1,3-bis(2-phenylaminoethyl)disiloxane (yield 26%), formed at the Si–C_{vinyl} bond splitting in te parent silane (**V**).



(3)

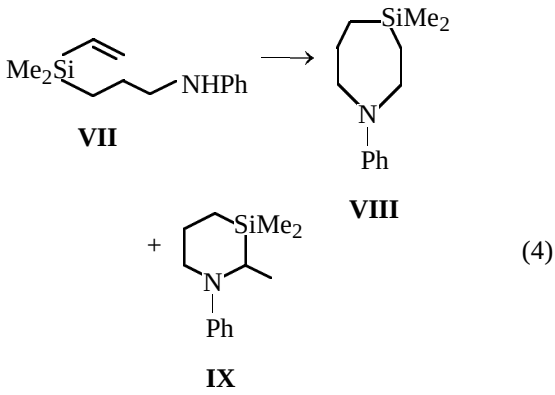
VI

Formation of a sixmembered heterocycle **VI** in the reaction (3) gives rise to an assumption that in the initial step the electrophilic mercury acetate ion is added at the double bond to non-terminal carbon atom.

This is probably results from the stabilizing effect of dimethylsilyl group on the neighboring β-carbocation, changing the cyclization regioselectivity of compound **V** compared to its carbon analoag. In reaction (2), a high yield of heterocycle **IV** (above 90%) also can be assigned to the appearance of activating β-effect of dimethylsilyl group.

In this respect, one could expect formation of a sevenmembered heterocycle **VIII** at the intramolecular cyclization of vinyl(3-phenylaminopropyl)dimethylsilane (**VII**). Actually, after aminomercuration–demercuration of amine **VII** the reaction mixture contains predominantly the silaazepan derivative **VIII**. The **VIII**:**IX** ratio achieves 2.7:1, with the total yield 49%.

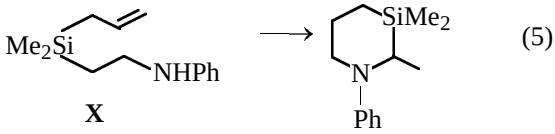
Formation of thermodynamically disadvantageous sevenmembered ring was not observed at all in the



VII **VIII** **IX**

(4)

electrophilic cyclization of N-substituted 5-hexenylamines: this reaction led only to methyl-substituted sixmembered heterocycles [8, 10, 11]. In correspondence with Markownikoff rule proceeds also the ring closure in the case of allyl(2-phenylaminoethyl)dimethylsilane **X**, an isomer of compound **VII**.



X **XI**

(5)

Yield of heterocycle **XI** is 30%.

Formation of a sixmembered ring **IX** in reaction (4) can be a result of isomerization process in the step of reduction of the sevenmembered mercury-contained intermediate [7, 12].

Thus, the stabilizing effect of silyl group defines high regioselectivity in the electrophilic cyclization of unsaturated organosilicon amines bearing a vinyl or an allyl group at the silicon atom.

EXPERIMENTAL

Analysis of reaction products and testing for their individuality were conducted by the GLC method on a LXM-8MD chromatograph (stainless steel column 200 × 3 mm, liquid phase Lucopren G-1000, 10% on Chromaton N-AW-HMDS) as well as by TLC on the Silufol UV-254 plates, development with iodine vapor. For column adsorption chromatography we used silica gel-60 (35–70 mesh ASTM, Merck). ^{1}H, ^{13}C, and ^{29}Si NMR spectra were recorded on a Bruker DPX-400 spectrometer from the solutions of compounds in $CDCl_3$. Mass spectra were taken up at ionizing voltage 70 eV on a chromato-mass spectrometer Hewlett–Packard HP5971A (the mass-selective detection) with HP-5890 chromatograph.

Vinyl(chloromethyl)dimethylsilane was prepared according to procedure [13], allyl(chloromethyl)dimethylsilane, allyl(2-hydoxyethyl)dimethylsilane and allyl(2-chloroethyl)dimethylsilane accrding to [14].

The initial amines **I**, **III**, and **VII** were synthesized according to a published procedure and purified by column chromatography (see below). Their constants and ^{1}H NMR data obey to the published ones [15].

Vinyl(2-phenylaminoethyl)dimethylsilane (V). *a*. **Vinyl(2-hydoxyethyl)dimethylsilane (XII).** To a Grignard reagent prepared from 16.00 g of vinyl(chloromethyl)dimethylsilane and 3.15 g of magnesium in 60 ml of ether was added in portions powdered paraform (Merck). The reaction mixture was refluxed for 10 h. The thick mass formed was poured into a glass on saturated NH_4Cl solution with ice. After separation of organic layer, the water solution was extracted with ether and methylene chloride. The collected organic phase was dried over $MgSO_4$. After removing of solvents in a vacuum rotor evaporator by vacuum distillation was isolated 7.36 g of crude product containing 95% of compound **XII**, yield 45%. Compound **XII** was purified by column chromatography on silica gel, consecutive eluing with hexane, methylene chloride and methanol. R_f 0.87 (CH_3OH), bp 45°C (8 mm Hg), n_D^{20} 1.4450. ^{1}H MMR spectrum, δ, ppm: 0.10 s (6H, Me_2Si), 1.01 m (*A* part of *AA*'*XX*' spin system, 2H, $SiCH_2$, *J* 7.55, 13.4, 3.1 Hz), 3.73 m (*X* part of *AA*'*XX*' spin system, 2H, CH_2O, *J* 4.25, 13.4, 3.1 Hz), 5.69 d.d (1H, =CH-*trans*, J_{gem} 3.9, J_{trans} 20.2 Hz), 5.96 d.d (1H, =CH-*cis*, J_{cis} 14.7 Hz), 6.13 d.d (1H, CH=). Found, %: C 55.15; H 11.77; Si 21.98. $C_6H_{14}OSi$. Calculated, %: C 55.32; H 10.83; Si 21.56.

b. **Vinyl(2-chloroethyl)dimethylsilane (XIII).** A mixture of 7.56 g of silane **XII** and 20.46 g of triphenylphosphine in 55 ml of CCl_4 was heated for 2 h. After adding 60 ml of hexane the precipitate formed was filtered off and the solvent was removed in a rotor evaporator. From the residue by vacuum distillation 1.50 g (17%) of compound **XIII** was isolated, bp 78°C (91 mm), n_D^{20} 1.4490. ^{1}H MMR spectrum, δ, ppm: 0.14 s (6H, Me_2Si), 1.27 m (*A*-part of *AA*'*XX*' spin system, 2H, $SiCH_2$), 3.64 m (*X* part of *AA*'*XX*'spin system, 2H, CH_2Cl), 5.70 d.d (1H, =CH–*trans*, J_{gem} 4.0, J_{trans} 19.9 Hz), 5.99 d.d (1H, =CH–*cis*, J_{cis} 14.7 Hz), 6.11 d.d (1H, CH=). Found, %: C 48.54; H 9.31; Cl 23.02; Si 18.95. $C_6H_{13}ClSi$. Calculated, %: C 48.46; H 8.81; Cl 23.84; Si 18.89.

c. **Vinyl(2-phenylaminoethyl)dimethylsilane (V).** A mixture of 1.50 g of chloroethylsilane **XIII** and 7.00 g of aniline was heated in a sealed ampoule for 22 h at 80–85°C. To the reaction mixture was added methylene chloride and then water for dissolving aniline hydrochloride precipitate. Organic layer was separated and washed with 5% KOH solution, then dried over Na_2SO_4. The residue (8.13 g) after removing of solvents in a rotor evaporator was passed through a column with silica gel, eluent hexane–ether, 9 : 1. After removing of the solvents in a vacuum, 0.60 g (29%) of amine was obtained, R_f 0.38, n_D^{22} 1.5160. ^{1}H MMR spectrum, δ, ppm: 0.14 s (6H, Me_2Si), 0.97 m (*A* part of *AA*'*XX*' spin system, 2H, $SiCH_2$), 3.15 m (*X* part *AA*'*XX*' spin system, 2H, CH_2N), 3.54 br.s (1H, NH), 5.72 d.d (1H, =CH-*trans*, J_{gem} 3.9, J_{trans} 20.2 Hz), 6.00 d.d (1H, =CH-*cis*, J_{cis} 14.7 Hz), 6.16 d.d (1H, CH=), 6.58 m ($2H_o$, C_6H_5, ^3J 7.6 Hz), 6.68 t ($1H_p$, C_6H_5, ^3J 6.3 Hz), 7.16 d.d ($2H_m$, C_6H_5, ^3J 7.6, ^3J 6.3 Hz). ^{13}C NMR spectrum, δ_C, ppm: –3.20 (MeSi), 16.64 ($SiCH_2$), 40.03 (CH_2N), 112.89 (C_o, C_6H_5), 117.29 (C_p, C_6H_5), 129.29 (C_m, C_6H_5), 132.36 (=CH_2), 138.50 (CH=), 148.47 (C_i, C_6H_5). ^{29}Si NMR spectrum, δ_{Si}, ppm: –6.6. Mass spectrum, *m/z* (I_{rel}, %): 205 (30) $[M]^+$, 176 (15), 162 (56), 150 (26), 134 (9), 120 (11), 106 (100), 91 (7), 85 (41), 65 (9), 59 (23). Found, %: N 6.12; Si 13.20. $C_{12}H_{19}NSi$. Calculated, %: N 6.82; Si 13.68.

Allyl(2-phenylaminoethyl)dimethylsilane (X) was prepared similarly. Yield 27%, R_f 0.36 (hexane–ether, 9:1), n_D^{21} 1.5180. ^{1}H MMR spectrum, δ, ppm: 0.05 s (6H, Me_2Si), 0.92 m (*A* part of *AA*'*XX*' spin system, 2H, $SiCH_2$), 1.56 d.t (2H, $SiCH_2C$=, ^3J 8.1, ^4J 1.1 Hz), 3.15 m (*X* part of *AA*'*XX*' spin system, 2H, CH_2N), 3.58 br.s (1H, NH), 4.86 m (1H, =CH-*trans*), 4.85 m (1H, =CH-*cis*), 5.78 q.t (1H, CH=, J_{cis} 10.2, J_{trans} 16.9, ^3J 8.1 Hz), 6.56 d.d ($2H_o$, C_6H_5, ^3J 7.7, ^4J 1.0 Hz), 6.67 t.t ($1H_p$, C_6H_5, ^3J 7.3, ^4J 1.0 Hz), 7.15 d.d ($2H_m$, C_6H_5, ^3J 7.3, ^3J 7.7 Hz). Found, %: N 5.54; Si 12.11. $C_{13}H_{21}NSi$. Calculated, %: N 6.38; Si 12.80.

General procedure for mercurycyclization. To a solution of 1.90 g of vinyl(phenylaminomethyl)dimethylsilane (**I**) in 50 ml of THF was added with stirring 3.19 g of mercury acetate. After 6-h stirring was added consecutively 50 ml of 0.5 M sodium hydroxide and 0.38 g of sodium borohydride in 10 ml of 2.5 M of sodium hydroxide. After 48 h the reaction mixture was decanted from the dropped mercury, organic layer was separated, water phase was extracted with ether. Collected orgaic phase was dried over anhydrous $MgSO_4$. After distilling off the solvents in a rotor evaporator, the residue was distilled in a vacuum and a fraction bp 106—110°C (1.5 mm) 1.26 g was separated. According to GLC, it contained parent silane **I** and **1-phenyl-3,3-dimethyl-3-silapyrrolidine (II)** in 1 : 7.5 ratio.

To a 0.68-g portion of this fraction dissolved in 2 ml of ether a solution of 0.4 ml of acetylchloride in 1.5 ml of ether was added and after 15-min stirring the mixture was hydrolyzed with water. To the water layer was added 10% solution of NaOH to alkaline reaction and it was extracted with ether. Organic phase was dried over Na_2SO_4. After removing of ether in a rotor evaporator 0.15 g of compound **II** was isolated by column chromatography (hexane–ether, 9 : 1–1 : 1), R_f 0.73 (hexane–ether, 9 :1), n_D^{24} 1.5174. ^{1}H NMR spectrum, δ, ppm: 0.25 s (6H, Me_2Si), 1.03 t (2H, $SiCH_2C$), 2.52 s (2H, $SiCH_2N$), 3.44 t (2H, CCH_2N), 6.68 t.t (1H$_p$, C_6H_5, ^3J 7.4, ^4J 1.0 Hz), 6.80 d (2H$_o$, C_6H_5, J 8.0 Hz), 7.23 d.d (2H$_m$, C_6H_5, ^3J 8.0, ^3J 7.4 Hz). These data are identical with the earlier published ones [15]. ^{13}C NMR spectrum, δ_C, ppm: –2.67 (CH_3Si), 12.54 (SiCC); 37.16 (SiCN), 48.06 (C*C*N), 113.06 (C_o), 116.148 (C_p), 128.947 (C_m), 151.19 (C_i). ^{29}Si NMR spectrum, δ_{Si}, ppm: 16.54.

Similarly, from amines **III**, **V**, **VII**, and **X** were prepared heterocyclic compounds **IV**, **VI**, **VIII**, **IX**, and **XI**, respectively.

1-Phenyl-3,3,5-trimethyl-3-silapyrrolidine (IV). Yield 92%, R_f 0.73 (hexane–ether, 9 : 1), n_D^{20} 1.5170. ^{1}H NMR spectrum, δ, ppm: 0.22 s (3H, MeSi), 0.33 s (3H, MeSi), 0.81 d (1H, $SiCH_2C$, J_{AB} 14.5 Hz), 0.99 d (3H, CH_3, ^3J 6.4 Hz), 1.19 d.d (1H, $SiCH_2C$, J_{AB} 14.5, ^3J 8.5 Hz), 2.31 d (1H, $SiCH_2N$, J_{AB} 13.3 Hz), 2.66 d (1H, $SiCH_2N$, J_{AB} 13.3 Hz), 4.51 d.q (1H, CH, ^3J 8.5, ^3J 6.4 Hz,), 6.64 t (1H$_p$, C_6H_5, ^3J 5.2 Hz), 6.79 d (2H$_o$, C_6H_5, ^3J 8.2 Hz), 7.20 d.d (2H$_m$, C_6H_5, ^3J 8.2, ^3J 5.2 Hz). This corresponds to the published data [15].

1-Phenyl-4,4-dimethyl-4-silapiperidine (VI). Yield 3%, R_f 0.79 (hexane–ether, 9 : 1). ^{1}H MMR spectrum, δ, ppm: 0.09 s (6H, Me_2Si), 0.81 m (4H, $SiCH_2C$), 3.86 m (4H, CH_2N), 6.70 t (1H$_p$, C_6H_5, ^3J 5.2 Hz), 6.88 d (2H$_o$, C_6H_5, ^3J 8.1 Hz), 7.24 d.d (2H$_m$, C_6H_5, ^3J 8.1, ^3J 5.2 Hz). Mass spectrum, *m/z* (I_{rel}, %): 205 (60) $[M]^+$, 176 (33), 162 (12), 149 (100) $[Me_2SiNC_6H_5]^+$, 134 (9), 119 (12), 104 (12), 91 (5), 77 (23), 59 (9). Found, %: N 6.41; Si 13.39. $C_{12}H_{19}NSi$. Calculated, %: N 6.82; Si 13.68.

1-Phenyl-4,4-dimethyl-4-silaazepane (VIII) was isolated from ethereal layer after treatment of reaction mixture with acetyl chloride. Yield 36%, R_f 0.73 (hexane–ether, 9 : 1), n_D^{23} 1.5180. ^{1}H NMR spectrum, δ, ppm: –0.11 s (6H, Me_2Si), 0.76 (*A* part of *AA*'*XX*' spectrum, 2H, $SiCH_2CN$), 1.13 t (2H, $SiCH_2CC$, ^3J 6.9 Hz), 1.84 m (2H, CCH_2C), 3.46 (*X* part *AA*'*XX*' spectrum, 2H, $SiCCH_2N$), 3.60 t (2H, FCCCH2N, ^3J 6.9 Hz), 6.64 t (1H$_p$, C_6H_5, ^3J 7.2 Hz), 6.67 d (2H$_o$, C_6H_5, ^3J 8.0 Hz), 7.23 d.d (2H$_m$, C_6H_5, ^3J 8.0, ^3J 7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: –1.83 (CH_3Si), 15.36 (SiCCN), 15.56 (Si*C*CC), 23.37 (*C*CC), 47.93 (SiC*C*N), 54.15 (CC*C*N), 110.85 (C_o), 114.98 (C_p), 129.30 (C_m), 147.61 (C_i). ^{29}Si NMR spectrum, δ_{Si}, ppm: 5.4. Found, %: N 6.07; Si 12.84. $C_{13}H_{21}NSi$. Calculated, %: N 6.38; Si 12.80.

1-Phenyl 2,3,3-trimethyl-3-silapiperidine (IX) was isolated from water layer after treatment of the reaction mixture with acetyl chloride. Yield 13%, R_f 0.71, n_D^{20} 1.5162. ^{1}H NMR spectrum, δ, ppm: 0.07 s (3H, MeSi), 0.15 s (3H, MeSi), 0.73 m (2H, $SiCH_2C$), 1.17 d (3H, CH_3, ^3J 7.5 Hz), 1.80 m (1H$_{ax}$, CCH_2C), 1.90 m (1H$_{eq}$, CCH_2C), 3.06 q.d (1H$_{ax}$, CCH_2N, J_{AB} 14.1, $^3J_{H_{a_x}-H_{ax}}$ 11.2, $^3J_{H_{ax}-H_{eq}}$ 2.2 Hz), 3.30 q (1H, CH, J 7.5 Hz), 3.42 d.t (1H$_{eq}$, CCH_2N, $^2J_{AB}$ 14.1, $^3J_{H_{eq}-H_{ax}}$ 2.2, $^3J_{H_{eq}-H_{eq}}$ 2.2 Hz), 6.65 t (1H$_p$, C_6H_5, ^3J 7.2 Hz), 6.84 d (2H$_o$, C_6H_5, ^3J 8.5 Hz), 7.18 d.d (2H$_m$, C_6H_5, ^3J 7.2, ^3J 8.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: –3.66 (CH_3Si), –3.84 (CH_3Si), 10.08 (Si*C*C), 11.39 (CH_3), 23.72 (*C*CC), 43.03 (CH), 45.76 (CH_2N), 114.75 (C_o), 116.58 (C_p), 129.04 (C_m), 147.64 (C_i). Found, %: N 6.67; Si 12.62. $C_{13}H_{21}NSi$. Calculated, %: N 6.38; Si 12.80.

1-Phenyl 2,4,4-trimethyl-4-silapiperidine (XI). Yield 30%, R_f 0.69. ^{1}H MMR spectrum, δ, ppm: 0.07 s (3H, MeSi), 0.17 s (3H, MeSi), 0.69 d.t (1H$_{ax}$, $SiCH_2C$, ^2J 14.4, $^3J_{H_{eq}-H_{ax}}$ 3.6, $^3J_{H_{ax}-H_{ax}}$ 3.2 Hz), 0.77 d.d (1H$_{ax}$, $SiCH_2C$, ^2J 14.7, $^3J_{H_{eq}-H_{ax}}$ 7.0 Hz), 1.11–1.19 m (2H$_{eq}$, $SiCH_2CN$ and $SiCH_2C$), 1.32 d (3H, CH_3, ^3J 6.5 Hz), 3.43 q.d (1H$_{ax}$, CH_2N, ^2J 14.9, $^3J_{H_{ax}-H_{ax}}$ 12.9, $^3J_{H_{ax}-H_{eq}}$ 3.6 Hz), 3.74 q.d (1H$_{eq}$, CH_2N, ^2J 14.9, $^3J_{H_{ax}-H_{eq}}$ 6.5, $^3J_{H_{eq}-H_{eq}}$ 2.7 Hz), 4.15 q (1H, CH, J 6.5 Hz), 6.73 t.t (1H$_p$, C_6H_5, ^3J 8.3, ^4J 1.0 Hz), 6.82 d (2H$_o$, C_6H_5, ^3J 8.1 Hz), 7.23 d.d (2H$_m$, C_6H_5, ^3J 8.3, ^3J 8.1 Hz). Found, %: N 6.99; Si 12.80. $C_{13}H_{21}NSi$. Calculated, %: N 6.38; Si 12.80.

1,2-Bis(2-phenylaminoethyl)-1,1,2,2-tetramethyldisiloxane (XIV) was isolated in 26% yield, R_f 0 (hexane–ether, 9 : 1). ^{1}H NMR spectrum, δ, ppm: 0.14 s (6H, Me_2Si), 0.96 m (2H, $SiCH_2$), 3.18 m (2H, CH_2N), 6.60 m (2H$_o$, C_6H_5), 6.71 t (1H$_p$, C_6H_5), 7.15 m (2H$_m$, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 0.76 (MeSi), 19.37 ($SiCH_2$), 39.78 (CH_2N), 113.09 (C_o, C_6H_5), 117.58 (C_p, C_6H_5), 129.32 (C_m, C_6H_5), 148.22 (C_i, C_6H_5). ^{29}Si NMR spectrum, δ_{Si}, ppm: 6.87. Mass spectrum, *m/z* (I_{rel}, %): 372 (20) $[M]^+$, 253 (15), 224 (55), 208 (100), 177 (15), 147 (22), 131 (26), 106 (86), 77 (22), 65 (11). Found, %: N 7.44; Si 15.36. $C_{20}H_{32}N_2OSi_2$. Calculated, %: N 7.52; Si 15.07.

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