ISSN 1070-4280, Russian Journal of Organic Chemistry, 2016, Vol. 52, No. 10, pp. 1385–1389. © Pleiades Publishing, Ltd., 2016. Original Russian Text © B.A. Gostevskii, A.I. Albanov, A.V. Vashchenko, V.I. Smirnov, 2016, published in Zhurnal Organicheskoi Khimii, 2016, Vol. 52, No. 10, pp. 1397–1401.

## Synthesis of 1-Organyl-1-(trimethylsiloxy)-2-(dimethylamino)ethenes and New Hypervalent Silicon Compounds Based Thereon

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Received April 22, 2016

**Abstract**—1-Organyl-1-(trimethylsiloxy)-2-(dimethylamino)ethenes were synthesized for the first time, and their reactions with trichloro(methyl)silane and trifluoro(phenyl)silane afforded silicon bis-chelates, methylbis-[2-(dimethylamino)-1-(organyl)ethenolato-*N*,*O*]siliconium chlorides and bis[1-(dimethylamino)-3,3-dimethylbut-1-en-2-olato-*N*,*O*]fluoro(phenyl)silicon(IV), respectively.

**DOI:** 10.1134/S107042801610002X

As shown previously, *N*,*N*-dimethyl carbohydrazonic acid trimethylsilyl esters are convenient intermediate products for the synthesis of hypervalent silicon compounds [1–3] that are capable of undergoing various intramolecular transformations [4–7]. In continuation of studies in this line, we have developed a procedure for the synthesis of 1-organyl-1-(trimethylsiloxy)-2-(dimethylamino)ethenes and examined their reaction with polyhalosilanes with the goal of obtaining hypervalent silicon compounds.

Dimethylaminomethyl ketones 1 and 2 were synthesized by reaction of the corresponding  $\alpha$ -bromo ketones with dimethylamine in diethyl ether. The silylation of 1 and 2 with trimethylsilyl trifluoromethanesulfonate in the presence of triethylamine smoothly afforded enamines 3 and 4 (Scheme 1). The use of chloro- or bromo(trimethyl)silane for this purpose was unsuccessful even under harsh conditions (100°C, 20 h). By transsilylation [4] of 3 and 4 with trichloro(methyl)silane we obtained silicon bischelates, siliconium chlorides 5 and 6 (Scheme 2) which showed in the <sup>29</sup>Si NMR spectra a signal at  $\delta_{Si}$  –60 ppm typical of five-coordinate silicon [8].

According to the X-ray diffraction data for a single crystal of **5** (Fig. 1), the silicon atom therein has a slightly distorted trigonal–bipyramidal configuration with *trans* arranged nitrogen atoms of the donor dimethylamino groups and a Si–Cl distance of 10.705 Å (cf. 2.019 Å for a covalent Si–Cl bond).

Replacement of the chlorine atom in **5** and **6** by more silicophilic fluorine atom was expected to hamper ionization and favor formation of neutral sixcoordinate silicon compounds. In order to verify this assumption enamine **3** was subjected to transsilylation with trifluoro(phenyl)silane. <sup>1</sup>H NMR monitoring showed that this reaction was slower, and it required more severe conditions. The reason is that the Si–F bond has higher energy than Si–Cl; therefore, the transsilylation stage is more difficult and is probably reversible. Nevertheless, the reaction led to the formation of nonionic fluorosilicon bis-chelate **7** in 30% yield (Scheme 3).







In the <sup>29</sup>Si NMR spectrum of 7, the silicon nucleus resonated at  $\delta_{Si}$  –138.6 ppm, in keeping with its six-coordinate state [9]. The structure of 7 was also confirmed by X-ray analysis (Fig. 2); the coordination entity is a regular octahedron with *trans* arrangement of the nitrogen atoms.

We have found that the synthesis of 7 through sodium enolate 8 derived from dimethylamino ketone 1 is more efficient since sodium fluoride formed in this reaction is almost insoluble in most organic solvents. Enolate 8 was synthesized by metalation of 1 with sodium bis(trimethylsilyl)amide in THF, and it reacted with trifluoro(phenyl)silane in THF at room temperature (20 h) to produce compound 7 in quantitative yield (Scheme 4).



Thus, we have synthesized for the first time 1-organyl-1-(trimethylsiloxy)-2-(dimethylamino)ethenes and demonstrated the possibility of using them as precursors to new hypervalent silicon compounds which still attract persistent interest [10–12].

## **EXPERIMENTAL**

The <sup>1</sup>H, <sup>13</sup>C, <sup>29</sup>Si, and <sup>15</sup>N NMR spectra were recorded on Bruker DPX-400 and Bruker AV-400

spectrometers at 400.13, 100.61, 161.98, and 40.56 MHz, respectively. The <sup>1</sup>H, <sup>13</sup>C, and <sup>29</sup>Si chemical shifts were measured relative to tetramethyl-silane, and the <sup>15</sup>N chemical shifts, relative to nitromethane. The elemental analyses were obtained on a Thermo Scientific Flash 2000 automated CHNS analyzer; the silicon content was determined by the dry combustion method.

**Dimethylaminomethyl ketones 1 and 2** (general procedure). A 100-mL glass ampule was charged with 0.025 mol of the corresponding bromomethyl ketone and 50 mL of anhydrous diethyl ether. The mixture was degassed and frozen with liquid nitrogen, 0.050 mol of anhydrous dimethylamine was condensed thereinto, and the ampule was sealed. The ampule was allowed to warm up to room temperature with intermittent shaking and kept for 24 h at room temperature. The precipitate was filtered off, the filtrate was evaporated under reduced pressure, and the residue was purified by vacuum distillation.

**1-(Dimethylamino)-3,3-dimethylbutan-2-one (1).** Yield 2.88 g (80%, 0.020 mol), colorless liquid, bp 72–74°C (28 mm),  $n_D^{20} = 1.4296$ . <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 1.16 s (9H, *t*-Bu), 2.29 s (6H, NMe<sub>2</sub>), 3.33 s (2H, CH<sub>2</sub>). <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>),  $\delta_C$ , ppm: 26.40 [C(CH<sub>3</sub>)<sub>3</sub>], 43.44 [C(CH<sub>3</sub>)<sub>3</sub>], 45.63 (NCH<sub>3</sub>), 63.28 (CH<sub>2</sub>), 152.86 (C=O). Found, %: C 67.21; H 12.03; N 9.54. C<sub>8</sub>H<sub>17</sub>NO. Calculated, %: C 67.09; H 11.96; N 9.78.

**2-(Dimethylamino)-1-phenylethan-1-one (2).** Yield 5.31 g (90%, 0.023 mol), colorless liquid, bp 57°C (0.08 mm),  $n_D^{20} = 1.5085$ . <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 2.38 s (6H, NMe<sub>2</sub>), 3.76 s (2H, CH<sub>2</sub>), 7.43–7.47 m (2H, *m*-H), 7.54–7.58 m (1H, *p*-H), 7.98– 8.00 m (2H, *o*-H). <sup>13</sup>C NMR spectrum,  $\delta_C$ , ppm: 45.88 (NCH<sub>3</sub>), 65.66 (CH<sub>2</sub>), 128.10 (C<sup>*m*</sup>), 128.55 (C<sup>*o*</sup>), 133.18 (C<sup>*p*</sup>), 135.99 (C<sup>*i*</sup>), 196.86 (C=O). Found, %: C 73.67; H 8.11; N 8.43. C<sub>10</sub>H<sub>13</sub>NO. Calculated, %: C 73.59; H 8.03; N 8.58.

**O-Trimethylsilyl derivatives 3 and 4** (general procedure). Trimethylsilyl trifluoromethanesulfonate was added dropwise with stirring in a stream of argon to a solution of equimolar amounts of triethylamine and amino ketone 1 or 2 in anhydrous diethyl ether. The mixture was refluxed for 3 h, the solution was separated by decanting and evaporated under reduced pressure, and the residue was distilled in a vacuum.

*N*,*N*,**3**,**3**-Tetramethyl-2-[(trimethylsilyl)oxy]but-1-en-1-amine (3) was synthesized from 2.25 g (0.016 mol) of **1** and 3.73 g (0.017 mol) of CF<sub>3</sub>SO<sub>3</sub>SiMe<sub>3</sub> using 1.70 g (0.017 mol) of Et<sub>3</sub>N. Yield 3.00 g (91%, 0.015 mol), colorless liquid, bp 70–72°C (15 mm),  $n_D^{20} = 1.4314$ . <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 0.18 s (9H, Me<sub>3</sub>Si), 1.00 s (9H, *t*-Bu), 2.24 s (6H, NMe<sub>2</sub>), 4.78 s (1H, CH=). <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>),  $\delta_C$ , ppm: 0.55 (SiMe<sub>3</sub>), 28.15 [C(CH<sub>3</sub>)<sub>3</sub>], 34.88 [C(CH<sub>3</sub>)<sub>3</sub>], 45.31 (NCH<sub>3</sub>), 119.24 (=CH), 152.59 (=CO). <sup>29</sup>Si NMR spectrum (CDCl<sub>3</sub>):  $\delta_{Si}$  15.2 ppm. <sup>15</sup>N NMR spectrum (CDCl<sub>3</sub>):  $\delta_N$  –355.1 ppm. Found, %: C 61.45; H 11.84; N 6.91; Si 13.25. C<sub>11</sub>H<sub>25</sub>NOSi. Calculated, %: C 61.33; H 11.70; N 6.50; Si 13.04.

*N*,*N*-Dimethyl-2-phenyl-2-[(trimethylsilyl)oxy]ethen-1-amine (4) was synthesized from 1.40 g (0.009 mol) of 2 and 2 g (0.009 mol) of CF<sub>3</sub>SO<sub>3</sub>SiMe<sub>3</sub> using 0.91 g (0.009 mol) of Et<sub>3</sub>N. Yield 1.50 g (75%, 0.006 mol), colorless liquid, bp 65°C (0.08 mm),  $n_D^{20}$  = 1.5089. <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 0.18 s (9H, SiMe<sub>3</sub>), 2.65 s (6H, NMe<sub>2</sub>), 5.72 s (1H, =CH), 7.08–7.11 m (1H, *p*-H), 7.20–7.24 m (2H, *m*-H), 7.36– 7.38 m (2H, *o*-H). <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>),  $\delta_C$ , ppm: 0.53 (SiMe<sub>3</sub>), 43.66 (NCH<sub>3</sub>), 123.70 (C<sup>o</sup>), 124.71 (HC=), 125.64 (C<sup>p</sup>), 127.98 (C<sup>m</sup>), 134.58 (PhC=), 139.16 (C<sup>i</sup>). <sup>29</sup>Si NMR spectrum (CDCl<sub>3</sub>):  $\delta_{Si}$  19.6 ppm. <sup>15</sup>N NMR spectrum (CDCl<sub>3</sub>):  $\delta_N$  –342.4 ppm. Found, %: C 66.85; H 8.73; N 6.31; Si 11.82. C<sub>13</sub>H<sub>21</sub>NOSi. Calculated, %: C 66.33; H 8.99; N 5.95; Si 11.93.

**Compounds 5 and 6** (general procedure). A Schlenk flask was evacuated, 5 mL of chloroform and trichloro(methyl)silane were condensed thereinto through a glass adapter equipped with a rubber tube, and enamine **3** or **4** was then added in one portion from a preliminarily evacuated ampule. The mixture was stirred under the conditions indicated below, and the



**Fig. 1.** Structure of the molecule of bis[1-(dimethylamino)-3,3-dimethylbut-1-en-2-olato-N,O]methylsiliconium chloride (**5**) according to the X-ray diffraction data. Hydrogen atoms are not shown; non-hydrogen atoms are shown as thermal vibration ellipsoids with a probability of 50%.



**Fig. 2.** Structure of the molecule of bis[1-(dimethylamino)-3,3-dimethylbut-1-en-2-olato-*N*,*O*]fluoro(phenyl)silicon(IV) (7) according to the X-ray diffraction data. Hydrogen atoms are not shown; non-hydrogen atoms are shown as thermal vibration ellipsoids with a probability of 50%.

product was isolated by evaporation under reduced pressure, followed by recrystallization of the residue from hexane. Compounds **5** and **6** were formed quantitatively, but the preparative yields were fairly low due to their very high solubility even in hexane.

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**Bis**[1-(dimethylamino)-3,3-dimethylbut-1-en-2-olato-*N*,*O*]methylsiliconium chloride (5) was synthesized from 1.87 g (8.68 mmol) of **3** and 0.65 g (4.35 mmol) of MeSiCl<sub>3</sub>; the reaction mixture was heated for 40 h at 90°C. Yield 0.79 g (50%, 2.19 mmol), white powder, mp 166–169°C. <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>), δ, ppm: 0.33 s (3H, MeSi), 1.21 s (18H, *t*-Bu), 2.91 s and 2.94 s (12H, NMe<sub>2</sub>), 5.86 s (2H, HC=). <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>), δ<sub>C</sub>, ppm: -9.98 (H<sub>3</sub>CSi), 26.56 [(H<sub>3</sub>C)<sub>3</sub>C], 33.51 (CMe<sub>3</sub>), 46.43, 49.29 (NCH<sub>3</sub>), 114.34 (=CHN), 159.98 (OC=). <sup>29</sup>Si NMR spectrum (CDCl<sub>3</sub>):  $\delta_{Si}$  –61.1 ppm. <sup>15</sup>N NMR spectrum (CDCl<sub>3</sub>):  $\delta_N$  –331.9 ppm. Found, %: C 56.31; H 9.84; N 7.33; Si 7.84. C<sub>17</sub>H<sub>35</sub>ClN<sub>2</sub>O<sub>2</sub>Si. Calculated, %: C 56.25; H 9.72; N 7.72; Si 7.74.

**Bis**[2-(dimethylamino)-1-(phenyl)ethenolato-*N*,*O*]methylsiliconium chloride (6) was synthesized from 0.97 g (4.12 mmol) of 4 and 0.28 g (1.88 mmol) of MeSiCl<sub>3</sub>; the reaction mixture was stirred for 14 h at 25°C. Yield 0.24 g (31%, 0.59 mmol), white powder, mp 172–174°C. <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>), δ, ppm: 0.50 s (3H, SiCH<sub>3</sub>), 3.18 s (12H, NMe<sub>2</sub>), 6.98 s (2H, HC=), 7.41–7.43 m (6H, *m*-H, *p*-H), 7.62–7.64 m (4H, *o*-H). <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>), δ<sub>C</sub>, ppm: –9.11 (SiCH<sub>3</sub>), 46.85 and 49.43 (NCH<sub>3</sub>), 116.23 (HC=), 124.87 and 128.87 (C<sup>o</sup>, C<sup>m</sup>), 129.39 (C<sup>i</sup>), 130.28 (C<sup>p</sup>), 148.83 (PhC=). <sup>29</sup>Si NMR spectrum (CDCl<sub>3</sub>): δ<sub>Si</sub> –60.6 ppm. Found, %: C 62.71; H 6.66; N 6.74; Si 7.05. C<sub>21</sub>H<sub>27</sub>ClN<sub>2</sub>O<sub>2</sub>Si. Calculated, %: C 62.59; H 6.75; N 6.95; Si 6.97.

Bis[1-(dimethylamino)-3,3-dimethylbut-1-en-2-olato-N,O|fluoro(phenyl)silicon(IV) (7). A Schlenk flask was evacuated and charged with 1.29 g (9.00 mmol) of 1, 1.65 g (9.00 mmol) of NaN(SiMe<sub>3</sub>)<sub>2</sub>, and 10 mL of THF, and the mixture was stirred for 2 h at 25°C. Volatile compounds were removed under reduced pressure to leave 1.50 g of sodium salt 8 as a yellowish powder. Tetrahydrofuran, 10 mL, and trifluoro(phenyl)silane, 0.74 g (4.54 mmol), were condensed into the flask, and the mixture was stirred for 20 h at 25°C. The mixture was filtered and evaporated under reduced pressure. The crude product, 1.86 g (100%) was purified by recrystallization from hexane where it is soluble very readily. Yield of 7 0.70 g (37%, 1.71 mmol), colorless crystals, mp 81°C. <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 1.08 s (18H, *t*-Bu), 2.33 s [12H, N(CH<sub>3</sub>)<sub>2</sub>], 4.68 s (2H, HC=,  ${}^{4}J_{\text{HF}}$  = 1.8 Hz), 7.17-7.22 m (3H, m-H, p-H), 7.83-7.86 m (2H, o-H). <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>),  $\delta_{C}$ , ppm: 27.85 [(H<sub>3</sub>C)<sub>3</sub>C], 33.82 [C(CH<sub>3</sub>)<sub>3</sub>], 50.14 [N(CH<sub>3</sub>)<sub>2</sub>], 109.50 (=CHN), 126.68 (C<sup>m</sup>), 126.91 (C<sup>p</sup>), 137.88 d

(C<sup>o</sup>,  ${}^{3}J_{CF} = 7.3 \text{ Hz}$ ), 146.58 d (C<sup>i</sup>,  ${}^{2}J_{CF} = 48.3 \text{ Hz}$ ). <sup>29</sup>Si NMR spectrum (CDCl<sub>3</sub>):  $\delta_{Si}$ -138.6 ppm.  ${}^{15}$ N NMR spectrum (CDCl<sub>3</sub>):  $\delta_{N}$ -334.5 ppm. Found, %: C 64.79; H 9.01; N 6.71; Si 6.69. C<sub>22</sub>H<sub>37</sub>FN<sub>2</sub>O<sub>2</sub>Si. Calculated, %: C 64.66; H 9.13; N 6.86; Si 6.87.

Single crystals of **5** and **7** for X-ray analysis were obtained by crystallization from diethyl ether and hexane, respectively. The X-ray diffraction data were acquired at 100.0 K on a Bruker D8 Venture diffractometer equipped with a Photon 100 detector (Mo $K_{\alpha}$  radiation,  $\lambda$  0.71073 Å;  $\omega$ -2 $\theta$  scanning,  $2\Theta_{\text{max}} = 52.76^{\circ}$ ). The data were processed using Bruker SAINT software. Absorption by the crystal was taken into account by analysis of equivalent reflection intensities. The structures were solved by the direct method using SHELXS [13] and were refined by the least-squares method using SHELXL [13].

Compound **5**.  $C_{17}H_{34}N_2O_2SiCl$ , *M* 363.01; monoclinic crystal system, space group *Cc*; unit cell parameters: a = 13.5869(5), b = 13.5818(5), c = 11.7494(4) Å;  $\beta = 104.3070(10)^\circ$ ; V = 2099.14(13) Å<sup>3</sup>; Z = 4;  $\mu(MoK_{\alpha}) = 0.25 \text{ mm}^{-1}$ ;  $d_{calc} = 1.149 \text{ g/cm}^3$ . Total of 22019 reflection intensities were measured, including 4078 independent reflections ( $R_{int} = 0.027$ ) which were used in the structure solution and refinement. Final divergence factor R = 0.071. Interatomic distances: Si–N<sup>1</sup> 2.017, Si–N<sup>2</sup> 1.992, Si–O<sup>2</sup> 1.670, Si–O<sup>0.4.4</sup> 1.673, Si–C<sup>15</sup> 1.858 Å; bond angles: O<sup>0.4.4</sup>SiN<sup>1</sup> 85.17, O<sup>0.4.4</sup>SiN<sup>2</sup> 85.25, O<sup>2</sup>SiC<sup>15</sup> 109.98, O<sup>0.4.4</sup>SiO<sup>2</sup> 137.99, N<sup>1</sup>SiN<sup>2</sup> 156.20°. The complete set of X-ray diffraction data for compound **5** was deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 1471762).

Compound 7. C<sub>22</sub>H<sub>37</sub>N<sub>2</sub>O<sub>2</sub>SiF, M 406.61; triclinic crystal system, space group P-1; unit cell parameters: a = 8.5268(9), b = 10.5515(11), c = 13.7342(9) Å;  $\alpha =$ 77.703(4),  $\beta = 73.365(4)$ ,  $\gamma = 83.992(5)^{\circ}$ ; V =1155.53(19) Å<sup>3</sup>; Z = 2;  $\mu(MoK_{\alpha}) = 0.13 \text{ mm}^{-1}$ ;  $d_{calc} =$ 1.169 g/cm<sup>3</sup>. Total of 24946 reflection intensities were measured, including 4431 independent reflections  $(R_{int} = 0.022)$  which were used in the structure solution and refinement. Final divergence factor R = 0.029. Interatomic distances: Si–N<sup>8</sup> 2.068, Si–N<sup>1</sup> 2.046, Si–O<sup>1</sup> 1.768, Si–O<sup>0.4.4</sup> 1.780, Si–F 1.664 Å; bond angles: O<sup>1</sup>SiN<sup>1</sup> 86.36, O<sup>0.4.4</sup>SiN<sup>1</sup> 86.48, O<sup>1</sup>SiN<sup>8</sup> 91.33, O<sup>0AA</sup>SiN<sup>8</sup> 85.43, N<sup>1</sup>SiF 89.54, N<sup>8</sup>SiF 92.20, O<sup>1</sup>SiF 174.38, O<sup>0.AA</sup>SiF 88.18°. The complete set of X-ray diffraction data for compound 7 was deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 1473286).

The main results of this study were obtained using the facilities of the Baikal Joint Analytical Center, Siberian Branch, Russian Academy of Sciences.

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