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1,1-Dimethyl-1-(trialkoxysilylmethyl)hydrazinium and 1,1-Dimethyl-1-(silatranylmethyl)hydrazinium Halides

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Abstract — Formerly unknown 1,1-dimethyl-1-(trialkoxysilylmethyl)- and 1,1-dimethyl-1-(silatranylmethyl)-hydrazinium halides were prepared by reaction of 1,1-dimethylhydrazine with (halomethyl)trialkoxysilanes $XCH_2Si(OR)_3$ (X = Cl, I; R=Me, Et) and 1-(halomethyl)silatranes $XCH_2Si(OCH_2CH_2)_3N$ (X = Cl, Br). 1,1-Dimethyl-1-(silatranylmethyl)hydrazinium chloride and iodide were also obtained by transetherification of corresponding 1,1-dimethyl-1-(trimethoxysilylmethyl)hydrazinium halides with tris(2-hydroxyethyl)amine.

We recently prepared organosilicon compounds simultaneously containing a five-coordinate silicon and an onium nonmetal atoms (nitrogen, phosphorus, sulfur, silicon, and tellurium) intervened by a methylene bridge. They all had the general formula $N(CH_2CH_2O)_3SiCH_2Y^+I^-$, where $Y^+ = N^+Me_3$, N^+HEt_2, $N^+H_2C_6H_5$, $N=C_5H_5$, $N^+C_9H_7$, etc [1]; P^+Ph_3 [2]; S^+Me_2, S^+MeEt, S^+MePh, $S^+Me(CH_2Ph)$, $S^+Me\cdot(CH_2Ph)$, $S^+Me(CH_2COOH)$, $S^+Me(CH_2CH_2OH)$, $S^+Me[CH_2Si(OCH_2CH_2)_3N]$, $S^+[CH_2Si(OCH_2\cdot CH_2)_3N]_2$, etc.[3]; Se^+Me_2, Se^+MeEt, Se^+MePh [4]; Te^+Me_2, Te^+Vin_2 [5]. Along with these compounds, we also prepared their analogs containing a four-coordinate silicon and an onium nitrogen, sulfur, or selenium atom of the general formula $R_3SiCH_2Y^+I^-$ where R = Me, $Y^+ = N^+Me_3$ [6, 7], R = MeO, Y = S^+MeEt; R = EtO, Y = S^+MeEt; R = EtO, Y = S^+Me_2[3], R = EtO, Y = Se^+Me_2 [8]. The crystal and molecular structure of trimethyl(silatranylmethyl)ammonium [9], triphenyl(silatranylmethyl)phosphonium [2], dimethyl(silatranylmethyl)sulfonium [10], and trimethyl(trimethylsilylmethyl)ammonium iodides [7] was established by means of X-ray diffraction.

Developing this aspect of our research, we have studied reactions of 1,1-dimethylhydrazine with (halomethyl)trialkoxysilanes $XCH_2Si(OR)_3$ **I**–**IV** in the absence of solvent and in hexane, as well as reactions of 1,1-dimethylhydrazine with 1-(halomethyl)silatranes $XCH_2Si(OCH_2CH_2)_3N$ **IX, X** in methanol, acetonitrile, and DMF. It was found that anhydrous dimethylhydrazine reacts with (halomethyl)trialkoxysilanes **I**–**IV** in the absence of solvent already at room temperature. Independent of the reagent molar ratio (1 : 1 or 1 : 2), the major reaction product is 1,1-dimethyl-1-(trialkoxysilylmethyl)hydrazinium halide **V**–**VIII** [Scheme (1)]. In the case of (iodomethyl)trialkoxysilanes **II** and **IV**, the reactions are complete within 8 h, and the yields of the iodides come close to quantitative. The reactions of dimethylhydrazine with the less active (chloromethyl)trialkoxysilanes **I** and **III** are complete within 14 h. The yields of chlorides **V** and **VII** reach 85%.

$$\underset{\mathbf{I{-}IV}}{(RO)_3SiCH_2X} + Me_2NNH_2 \longrightarrow \underset{\mathbf{V{-}VIII}}{(RO)_3SiCH_2Me_2\overset{+}{N}NH_2\ X^-}, \quad (1)$$

R = Me, X = Cl (**I, V**), I (**II, VI**); R = Et, X = Cl (**III, VII**), I (**IV, VIII**).

The reaction of dimethylhydrazine with (chloromethyl)trimethoxysilane (**I**) under the conditions of the synthesis of (1,1-dimethylhydrazinomethyl)dimethylmethoxysilane $Me_2NNHCH_2SiMe_2(OMe)$ from dimethylhydrazine and (chloromethyl)methoxydimethylsilane [11] leads to 1,1-dimethyl-1-(trimethoxysilylmethyl)hydrazinium chloride (**V**) in 45% yield. Along with this product, other products were formed, among which (1,1-dimethylhydrazinomethyl)trimethoxysilane $Me_2NNHCH_2Si(OMe)_2$ was identified by GC–MS ($M^{+\cdot}$ 194), but its fraction was no more than 10%.

1,1-Dimethyl-1-(trialkoxysilylmethyl)hydrazinium halides **V**–**VIII** are easily cleaved with lower alcohols (methanol, ethanol) by the Si–C bond to form corresponding 1,1,1-trimethylhydrazinium halides **IX** and **X** and tetraalkoxysilane.

$$\mathbf{V{-}VIII} + ROH \longrightarrow (RO)_4Si + \underset{\mathbf{IX,\ X}}{Me_3\overset{+}{N}NH_2\ X^-}, \quad (2)$$

X = Cl (**IX**), I (**X**).

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Similar Si–C bond cleavage we observed in the reaction of lower alcohols with methylorganyl(trialkoxysilylmethyl)sulfonium iodides [3].

Unlike (halomethyl)trialkoxysilanes **I**–**IV**, 1-(chloromethyl)silatrane (**XI**) and 1-(iodomethyl)silatrane (**XII**) in the absence of solvent fail to react even with boiling dimethylhydrazine. At the same time, boiling of 1-(iodomethyl)silatrane with dimethylhydrazine in methanol or acetonitrile for 6 h leads to 1,1-dimethyl-1-(silatranylmethyl)hydrazinium iodide (**XIV**) though in moderate yield (about 30%). However, the yield of iodide **XIV** upon heating of a mixture of 1-(iodomethyl)silatrane (**XII**) with dimethylhydrazine in DMF at 100–110°C for 2 h reaches 82%. The reaction of 1-(chloromethyl)silatrane ((26)XI) with dimethylhydrazine under analogous conditions for 6 h leads to 1,1-dimethyl-1-(silatranylmethyl)hydrazinium chloride (**XIII**) in ca. 70% yield.

$$\underset{\mathbf{XI,\ XII}}{N(CH_2CH_2O)_3SiCH_2X} + Me_2NNH_2 \longrightarrow \underset{\mathbf{XIII,\ XIV}}{N(CH_2CH_2O)_3SiCH_2Me_2\overset{+}{N}NH_2\ X^-}, \tag{3}$$

X = Cl (**XI, XIII**), I (**XII, XIV**).

1,1-Dimethyl-1-(silatranylmethyl)hydrazinium halides **XIII** and **XIV** were also prepared by transetherification of 1,1-dimethyl-1-(trimethoxysilylmethyl)hydrazinium halides (**V** and **VII**) with tris(2-hydroxyethyl)amine.

$$N(CH_2CH_2OH)_3 + \mathbf{V,\ VII} \longrightarrow \mathbf{XIII,\ XIV} + 3ROH. \tag{4}$$

In chloroform, the reactions proceed almost immediately in the absence of any catalyst. The yields of 1,1-dimethyl-1-(silatranylmethyl)hydrazinium halides **XIII** and **XIV** are 75 and 85%, respectively. The by-product of these reactions is 1,1,1-trimethylhydrazinium halide **XI** or **XII** formed by Si–C bond cleavage in starting 1,1-dimethyl-1-(trimethoxysilylmethyl)hydrazinium halides **V** and **VII** by the evolving methanol.

1,1-Dimethyl-1-(trialkoxysilylmethyl)hydrazinium halides **V**–**VIII** and 1,1-dimethyl-1(silatranylmethyl)hydrazinium halides **XIII** and **XIV** are colorless fine crystals. Compounds **V**–**VIII** are readily soluble in chloroform, acetonitrile, and DMF and insoluble in ether and alkanes (pentane, hexane). Compared to trialkoxysilyl derivatives **V**–**VIII**, 1,1-dimethyl-1-(silatranylmethyl)hydrazinium halides **XIII** and **XIV** have considerably higher melting points and are worse soluble in chloroform. At the same time, 1,1-dimethyl-1-(silatranylmethyl)hydrazinium halides **XIII** and **XIV**, like 1,1-dimethyl-1-(trialkoxysilylmethyl)hydrazinium halides **V**–**VIII**, are readily soluble in lower alcohols but without Si–C bond cleavage.

The structure of compounds **V**–**VIII, XIII,** and **XIV** is confirmed by their ^1H, ^{13}C, and ^{29}Si NMR (see table) and IR spectra. In the ^1H NMR spectra of 1,1-dimethyl-1-(trialkoxysilylmethyl)hydrazinium halides **V**–**VIII**, the proton signal of the CH_2Si bound with the onium nitrogen atom is near δ 3.60 ppm. The signal of the same protons in the ^1H NMR spectra of silatranyl derivatives **XIII** and **XIV** is shifted upfield (~2.80 ppm). The Me_2N proton signals of halides **V**–**VIII** and **XIII, XIV** are observed near δ 3.70 and 3.50 ppm, respectively. The proton signals of the $Si(OMe)_2$ and $Si(OEt)_3$ bands in compounds **V**–**VIII** and endocyclic OCH_2 groups of the silatrane skeleton in compounds **XIII, XIV** appear at almost the same δ values as the respective signals of starting (halomethyl)trimethoxysilanes **I** and **II**, (halomethyl)triethoxysilanes **III** and **V**, 1-(halomethyl)silatranes **XI** and **XII**, respectively (see table). The chemical shifts (δ ~3.0–3.1 ppm) of signals of the endocyclic NCH_2 group of the silatrane skeleton in compounds **XIII** and **XIV** provides evidence for their hydrazinium structure with the positive charge on the nitrogen atom of the $H_2NN^+Me_2CH_2$ fragment. The same chemical shifts of endocyclic NCH_2 proton signals are observed in the ^1H NMR spectra of dialkyl(silatranylmethyl)ammonium [1, 12], -sulfonium [3, 13, 14], -selenonium [4], and -telluronium iodides [5]. The chemical shifts of the Me_2N carbon signals in the ^{13}C NMR spectra of these compounds vary only slightly and lie in the range 58–60 ppm. At the same time, the CH_2Si carbon signals of compounds **V**–**VIII, XIII,** and **XIV** are appreciably downfield from those of the CH_2N group in starting (halomethyl)trialkoxysilanes **I**–**IV** and 1-(halomethyl)silatranes **XI** and **XII**. The δ_C increase with increasing bulk of substituent R^3 on the silicon atom and is equal to ~51 ppm for **V**, ~58 ppm for **VII, VIII**, and ~64 ppm for **XIII, XIV**. The chemical shifts in the ^{29}Si NMR spectra of halides **V**–**VIII, XIII,** and **XIV** are determined by the character of substitution (R_3) and the coordination number of silicon, equaling –58 ppm for **V** and **VI**, –62 ppm for **VII** and **VII**, and –85 ppm for **XIII** and **XIV**, respectively.

The IR spectra of 1,1-dimethyl-1-(trialkoxysilylmethyl)hydrazinium halides **V**–**VIII** and 1,1-dimethyl-1-(silatranylmethyl)hydrazinium halides **XIII** and **XIV** contain bands of stretching and deformation vibrations of the NH_2 group (~3200, 3100, and ~1600 cm^{-1}, respectively) and a set of characteristic Si–O–C stretching vibration bands. The absence of the broad strong band of stretching vibrations of the N^+H_2 group at ~2700 cm^{-1} and bands of stretching vibrations of

^1H, ^{13}C, and ^{29}Si NMR chemical shifts of (halomethyl)trialkoxysilanes **I–IV**, 1-(halomethyl)silatranes **XI** and **XII**, 1,1-dimethyl-1-trialkoxysilylmethyl)hydrazinium halides **V–VIII**, and 1,1-dimethyl-1-(silatranylmethyl)hydrazinium halides **XIII** and **XIV** in $CDCl_3$

Comp. no.	δ, ppm				δ_C, ppm			δ_{Si}, ppm
	Me_2N^+	CH_2Si	SiR^3	NH_2	Me_2N^+	CH_2Si	SiR^3	
I	–	2.81 s	3.62 s (9H, 3MeO)	–	–	22.6	51.0 (MeO)	–54.9
II	–	1.95 s	3.62 s (9H, 3MeO)	–	–	–26.0	51.5 (MeO)	–53.4
III	–	2.79 s	1.24 t (9H, 3Me), 3.90 q (6H, $3CH_2O$)	–	–	23.7	18.1 (CH_3 in Et), 59.2 (CH_2O)	–58.2
IV	–	1.87 s	1.20 t (9H, 3Me), 3.84 q (6H, $3CH_2O$)	–	–	–25.8	18.1 (CH_3 in Et), 59.1 (CH_2O)	–56.7
V	3.67 s	3.58 s	3.70 s (9H, 3MeO)	6.8 s	57.7	51.7	59.6 (MeO)	–58.7
VI	3.69 s	3.52 s	3.70 s (9H, 3MeO)	6.4 s	57.8	51.7	59.8 (MeO)	–58.9
VII	3.70 s	3.57 s	1.29 t (9H, 3Me), 3.94 q (6H, $3CH_2O$)	7.2 s	58.5	57.8	18.0 (CH_3 in Et), 59.7 (CH_2O)	–62.3
VIII	3.69 s	3.58 s	1.25 t (9H, 3*Me*CH_2), 3.92 q (6H, $3CH_2O$)	6.4 s	59.5	58.5	18.0 (CH_3 in Et), 59.7 (CH_2O)	–62.7
XI	–	2.63 s	2.89 t (6H, $3NCH_2$), 3.84 t (6H, $3CH_2O$)	–	–	31.1	51.2 (NCH_2), 57.42 (CH_2O)	–77.2[a]
XII	–	1.88	2.87 t (6H, NCH_2), 3.82 t (6H, $3CH_2O$)	–	–	–12.8	51.3 (NCH_2), 57.5 (CH_2O)	–77.0[a]
XIII	3.48 s	2.82 s	3.00 t (6H, $3CH_2N$), 3.85 t (6H, $3CH_2O$)	6.5 s	59.8	64.0	50.8 (NCH_2), 57.1 (CH_2O)	–84.8
XIV	3.50 s	2.82 s	3.04 t (6H, NCH_2), 3.88 t (6H, CH_2O)	6.2 s	60.7	64.7	51.1 (NCH_2), 57.4 (CH_2O)	–85.0

[a] The δ_{Si} values for compounds **XI** and **XII** were taken from [12].

the methyl substituents on nitrogen (2700–2850 cm^{-1}) show that the positive charge is localized on this nitrogen atom [16] and thus provide evidence for the presence in 1,1-dimethyl-1-(trialkoxysilylmethyl)-ydrazinium halides **V**–**VIII** and 1,1-dimethyl-1-(silatranylmethyl)hydrazinium halides **XIII** and **XIV** of a $nMe_2N^+NH_2$ fragment.

EXPERIMENTAL

The ^1H, ^{13}C, and ^{29}Si NMR spectra were registered on a Bruker DPX-400 spectrometer at 400.13, 100.61, and 79.49 MHz, respectively, for 10-15% solutions in $CDCl_3$ (**I**–**IV, VII**) and CD_3OD (**V**–**VIII**). The IR spectra were obtained on Specord 75IR spectrophotometer for KBr pellets. Technical grade 1,1-dimethylhydrazine was kept for 48 h over potassium hydroxide and twice distilled over metallic sodium. The solvents were purified and dried according to conventional procedures [17].

The starting (iodomethyltrimethoxy)-, (iodomethyl)-triethohysilane, and 1-(iodomethyl)silatrane were prepared as described in [18].

1,1-Dimethyl-1-(trimethoxysilylmethyl)hydrazinium chloride (V). A mixture of 1.7 g of (chloromethyl)trimethoxysilane and 1.2 g of 1,1-dimethylhydrazine was kept in a sealed ampule for 14 h until a syrup-like material formed and then placed in a refrigerator. When the syrup crystallized, the ampule was unsealed, and volatile admixtures were removed in a vacuum (1–2 mm Hg). The solid residue was loosened up, washed with anhydrous ether (3 × 5 ml), filtered off, and dried in a vacuum to obtain 2.1 g (91%) of compound **V** as a colorless finely crystalline powder, mp 92–94°C. IR spectrum (KBr), cm^{-1}: 780, 845, 1080, 1180 [δ, ν(SiOC)], 1630 [δ(NH_2)], 3090, 3190 [ν(NH_2)]. Found, %: C 31.02; H 8.17; Cl 15.12; N 11.76; Si 11.88. $C_6H_{19}ClN_2O_3Si$. Calculated, %: C 31.23; H 8.30; Cl 15.36; N 12.14; Si 12.17.

1,1-Dimethyl-1-(trimethoxysilylmethyl)hydrazinium iodide (VI). A mixture of 2.5 g of (iodomethyl)trimethoxysilane and 2.0 g of 1,1-dimethylhydrazine was placed in a sealed glass ampule and kept at room temperature until crystals formed (~8 h). The ampule was unsealed, and volatile admixtures were removed in a vacuum ((1–2 mm Hg). The crystalline product was loosened up, washed with anhydrous ether (3 × 5 ml), filtered off, and dried in a vacuum to obtain 4.32 g (96%) of iodide **VII** as a colorless finely crystalline powder, mp 74–76°C. IR spectrum (KBr), cm^{-1}: 785, 815, 840, 1050, 1070, 1185 [δ, ν(SiOC)], 1615 [$\delta(NH_2)$], 3100, 3200 [$\nu(NH_2)$]. Found, %: C 22.08; H 6.06; I 38.78; N 8.46; Si 8.52. $C_6H_{19}\cdot IN_2O_3Si$. Calculated, %: C 22.37; H 5.94; I 39.38; N 8.69; Si 8.72.

1,1-Dimethyl-1-(triethoxysilylmethyl)hydrazinium chloride (VII) was prepared in a similar way from 2.1 g of (chloromethyl)triethoxysilane and 1.2 g of 1,1-dimethylhydrazine. Yield 2.5 g (93%), colorless finely crystalline powder, mp 77–78°C. IR spectrum (KBr), cm^{-1}: 780, 790, 815, 1060, 1155 [δ, ν(SiOC)], 1620 [$\delta(NH_2)$], 3090, 3200 [$\nu(NH_2)$]. Found, %: C 39.37; H 9.07; Cl 12.38; N 10.12; Si 9.89. $C_9H_{25}ClN_2O_3Si$. Calculated, %: C 39.62; H 9.24; Cl 12.99; N 10.26; Si 10.29.

1,1-Dimethyl-1-(triethoxysilylmethyl)hydrazinium iodide (VIII) was prepared in a similar way from 1.5 g of (iodomethyl)triethoxysilane and 0.6 g of 1,1-dimethylhydrazine. Yield 1.9 g (94%), mp 92–94°C. IR spectrum (KBr), cm^{-1}: 780, 790, 815, 1065, 1155 [δ, ν(Si-O-C)], 1610 [$\delta(NH_2)$], 3110, 3210 cm [$\nu[NH_2)$]. Found, %: C 29.23; H 6.68; I 34.26; N 7.28; Si 7.39. $C_9H_{25}IN_2O_3Si$. Calculated, %: 29.67; H 6.92; I 34.8; N 7.69; Si 7.71.

Reaction of 1,1-dimethyl-1-(trimethoxysilylmethyl)hydrazinium chloride (V) with methanol. Chloride **I**, 1 g, was dissolved in 3 ml of anhydrous methanol. The reaction mixture was kept for 1 h and then evaporated in a vacuum (1.5 mm Hg). The solid residue was crystallized from 2-propanol to obtain 0.35 g (73%) of chloride **V**, mp 250–252°C (decomp.) {published data [19]: 255°C (decomp.)}. ^1H NMR spectrum (CD_3OD), δ, ppm: 3.40 s (9H, 3Me). ^{13}C NMR spectrum: δ_C 58.9 ppm. Distillation of the condensate from the vacuum trap gave 0.4 g of tetramethoxysilane, bp 119–120°C {published data [20]: bp 121°C}.

Treatment of the solution of 1.5 g of 1,1-dimethyl-1-(trimethoxysilylmethyl)hydrazinium iodide (**VII**) in methanol according to the same procedure gave 0.75 g (80%) of 1,1,1-trimethoxysilylmethylhydrazinium iodide (**VI**), mp 25°C {published data [21]: mp 229–230°C}. ^1H NMR spectrum (CD_3OD), δ, ppm: 3.38 s (9H, 3Me). In addition, 0.5 g of tetramethoxysilane was isolated.

1,1-Dimethyl-1-(silatranylmethyl)hydrazinium chloride (XIII). *a [by scheme (3)].* 1-(Chloromethyl)-silatrane, 6.7 g, was treated with 40 ml of anhydrous DMF. The resulting mixture was heated to 90–100°C and stirred until 1-(chloromethyl)silatrane dissolved completely. The solution was cooled to 60°C, and 4.5 g of 1,1-dimethylhydrazine was added to it dropwise over the course of 20 min. The mixture was then stirred for 4 h with gradual heating to 80–90°C and then cooled 50°C. The flaky precipitate of polymeric admixtures was filtered off. The filtrate was evaporated by half in a vacuum (1–2 mm Hg), and the residue was kept for 12 h in a refrigerator (ca. –10°C). Crystals formed and were filtered off, washed with several portions of anhydrous ether, and dried in a vacuum to obtain 5.5 g of chloride **XIII**. The mother liquor was evapotared in a vacuum to obtain an additional 1.2 g of compound **XIII**. Total yield of product **VII** 6.7 g (79%), mp 184–187°C (decomp.). IR spectrum (KBr), cm^{-1}: 865, 890, 915, 1065, 1150 [δ,ν(Si–O–C)], 1620 [$\delta(NH_2)$], 3090, 3200 [$\nu(NH_2)$]. Found, %: C 38.29; H 7.86; Cl 12.31; N 14.56; Si 9.78. $C_{10}H_{22}ClN_3O_3Si$. Calculated, %: C 38.17; H 8.18; Cl 12.52; N 14.84; Si 10.27.

b. [by scheme (4)]. To a stirred solution of 2.3 g of 1,1-dimethyl-1-(trimethoxysilylmethyl)hydrazinium chloride **V** in 5 ml of chloroform, a solution of 1.5 g of tris(2-hydroxyethyl)amine in 3 ml of chloroform was added dropwise with stirring. After heat release was no longer observed (40°C, ~2 min), the solution was cooled to 20°C, and the precipitate of 1,1,1-trimethylhydrazinium chloride was filtered off to give a chloroform solution of chloride **XIII**. The chloroform was distilled off, and the solid residue was crystallized from 2-propanol to obtain 2.1 g (74.6%) of compound **XIII**, mp 194–195°C (decomp.). IR spectrum (KBr), cm^{-1}: 865, 890, 915, 1065, 1150 [δ, ν(Si–O–C)], 1620 [$\delta(NH_2)$], 3090, 3200 [$\nu(NH_2)$]. Found, %: C 38.29; H 7.86; Cl 12.31; N 14.56; Si 9.78. $C_{10}H_{22}\cdot ClN_3O_3Si$. Calculated, %: C 38.17; H 8.18; Cl 12.52; N 14.84; Si 10.27.

1,1-Dimethyl-1-(silatranylmethyl)hydrazinium iodide (XIV). *a [by scheme (3)].* 1-(Iodomethyl)silatrane, 8.0 g, was treated with 50 ml of anhydrous DMF. The mixture was stirred with heating until 1-(iodomethyl)silatrane dissolved completely. The solution was cooled to 60°C, and 4.0 g of 1,1-dimethylhydrazine was added to it dropwise over the course of 30 min. The reaction mixture was stirred for extra 4 h with gradual heating to 80–100°C and then cooled to 20–25°C and evaporated by half in a vacuum (1–2 mm Hg). The residue was kept for 12 h in a refrigerator at ca. –10°C. Crystals formed and were filtered off, washed with several portions of anhydrous ether, and dried in a vacuum to give 4 g of iodide **XIV**. Evaporation of the mother liquor in a vacuum gave an additional 4 g of compound **XIV**.

Total yield of product **XIV** 8 g (84%), mp 184–186°C (decomp.). Found, %: C 28.38; H 5.47; I 32.19; N 11.21; Si 6.95. $C_9H_{22}IN_3O_3Si$. Calculated, %: C 29.00; H 5.89; I 33.49; N 11.69; Si 7.46.

b [by scheme (4)]. To a stirred solution of 3 g of 1,1-dimethyl-1-(triethoxysilylmethyl)hydrazinium iodide (**VIII**) in 5 ml of chloroform, a solution of 1.2 g of tris(2-hydroxyethyl)amine in 3 ml of chloroform was added dropwise. After heat release was no longer observed (35°C, ~2 min), the reaction mixture was cooled to 20°C, and the precipitate of 1,1,1-trimethylhydrazinium chloride was removed from the chloroform solution of iodide **XIV**. The chloroform was distilled off, and the solid residue was crystallized from 2-propanol to obtain 2.3 g (76%) of compound **XIV**, mp 175–177°C (decomp.).

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REFERENCES

1. Voronkov, M.G., Lukina, Yu.A., D’yakov, V.M., Frolov, Yu.L., and Tandura S.N., *Zh. Obshch. Khim.*, 1982, vol. 52, no. 2, p. 349.
2. Demidov, M.P., Shklover, V.E., Lukina, Yu.L., D’yakov, V.M., Struchkov, Yu.T., and Voronkov, M.G., *Zh. Strukt. Khim.*, 1991, vol. 32, no. 1, p. 177.
3. Sorokin, M.S. and Voronkov, M.G., *Zh. Obshch. Khim.*, 1979, vol. 49, no. 12, p. 2671.
4. Voronkov, M.G. and Sorokin, M.S., *Zh. Obshch. Khim.*, 1986, vol. 56, no. 8, p. 1818.
5. Voronkov, M.G. and Sorokin, M.S., *Zh. Obshch. Khim.*, 1989, vol. 59, no. 3, p. 590.
6. Voronkov, M.G., Kirpichenko, S.V., Abrosimova, A.T., Albanov, A.I., and Lavrent’ev, V.I., *Zh. Obshch. Khim.*, 1982, vol. 52, no. 9, p. 2055.
7. Gurkova, S.N., Gusev, A.I., Abrosimova, A.T., Alekseev, N.V., and Voronkov, M.G., *Dokl. Akad. Nauk SSSR*, 1981, vol. 260, no. 5, p. 1115.
8. Sorokin, M.S. and Voronkov, M.G., *Zh. Obshch. Khim.*, 2001, vol. 71, no. 12. p. 1988.
9. Demidov, M.P., Shklover, V.E., Lukina, Yu.L., D’yakov, V.M., Struchkov, Yu.T., and Voronkov M.G., *Zh. Strukt. Khim.*, 1985, vol. 26, no. 4, p. 103.
10. Shklover, V.E., Struchkov, Yu.T., Sorokin, M.S., and Voronkov, M.G., *Dokl. Akad. Nauk SSSR,* 1984, vol. 274, no. 3, p. 612.
11. Kirilin, A.D., Dokuchaev, A.A., Sokova, N.B., and Chernyshov, E.A., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1999, no. 1, p. 169.
12. Pestunovich, V.A., Tandura, S.N., Voronkov, M.G., Engel’gardt, G., Lipmaa, E., Pekh, T., Sidorkin, V.F., Zelchyan, G.I., and Baryshok, V.P., *Dokl. Akad. Nauk SSSR,* 1978, vol. 240, no. 4, p. 914.
13. Pestunovich, V.A., Popelis, Yu.Yu., Lukevits, E.Ya., and Voronkov, M.G., *Izv. Akad. Nauk Latv. SSR, Ser. Khim.*, 1973, p. 365.
14. Tandura, S.N., Pestunovich, V.A., Zelchyan, G.I., Baryshok, V.P., Lukina, Yu.A., Sorokin, M.S., and Voronkov, M.G., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1981, no. 2, p. 295.
15. Voronkov, M.G., Tandura, S.N., Sorokin, M.S., Shterenberg, B.Z., and Pestunovich, V.A., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1979, no. 2, p. 464.
16. Nakanishi, K., *Infrared Absorption Spectroscopy. Practical,* San Francisco: Holden-Day, 1962.
17. Gordon, A.J. and Ford, R.A., *The Chemist’s Companion,* New York: Wiley, 1972.
18. Sorokin, M.S., Lopyrev, V.A., Chipanina, N.N., Sherstyannikova, L.V., and Voronkov, M.G., *Zh. Obshch. Khim.*, 2004, vol. 74, no. 4, p. 603.
19. Klages, K. and Wolf, H., *Chem. Ber.*, 1959, vol. 92, no. 8, p. 1842.
20. Forbes, G.S. and Anderson, N.N., *J. Am. Chem. Soc.*, 1944, vol. 66, p. 1703.
21. Wawzonek, S. and Kellen, J., *J. Org. Chem.*, 1973, vol. 38, no. 11, p. 2058.