# (Iodomethyl)fluorosilanes: Synthesis and Reactions

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**Abstract**—The methods of synthesis of bifunctional (iodomethyl)fluorosilanes of general formula ICH<sub>2</sub>SiMe<sub>n</sub>F<sub>3-n</sub> (n = 0, 2) have been elaborated; the structure was proved by <sup>1</sup>H, <sup>13</sup>C, <sup>29</sup>Si NMR spectroscopy. The reaction of (iodomethyl)dimethylfluorosilane with *O*-trimethylsilyl derivative of *N*,*N*<sup>-</sup>dimethylhydrazide of trifluoroacetic acid gives rise to the formation of 2,2,4,4-tetramethyl-6-(trifluoromethyl)-3,4-dihydro-2*H*-1,4,5,2-oxadiasilin-4-ium iodide with tetracoordinate silicone atom.

**Keywords:** (iodomethyl)trifluorosilane, (iodomethyl)dimethylfluorosilane, (halogenomethyl)halogenosilanes, α-carbofunctional silanes

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Organosilicon α-carbofunctional compounds  $YCH_2SiR_nX_{3-n}$  containing geminal fragment Y–C–Si (X = Hlg, OR, SR, NRR'; Y = Hal, RO, RR'N, RS; R,R' = Alk, Ar) demonstrate unique reactivity due to their structure and are widely used in synthetic organic chemistry [1-4]. (Chloromethyl)chlorosilanes ClCH<sub>2</sub>SiR<sub>n</sub>Cl<sub>3-n</sub> (n = 0-3) are commercially available compounds and their reactivity is the most studied among (halomethyl) halosilanes HalCH<sub>2</sub>SiR<sub>n</sub>Hal<sub>3-n</sub> (n = 0-3). Therefore, it is not surprising that they serve as a basis for the synthesis of a-carbofunctional silanes with different substituents at the silicon and the  $\alpha$ -carbon atom [1, 5]. However, special interest attract bifunctional (halomethyl)halosilanes containing two different halogen atoms in the molecule. Thus, (iodomethyl)fluorosilanes  $ICH_2SiR_nF_{3-n}$ contain the C-I bond capable of nucleophilic substitution reactions, and one of the strongest bonds in silicon-containing compounds Si-F ( $E_{Si-F} = 159.9$  kcal/mol [6]), therefore, their use in organic synthesis may lead to the synthesis of new compounds and elaboration of unique synthetic procedures. We failed to find information on their reactivity. Apparently, the lack of such information is due to difficulties of preparation and isolation of bifunctional (iodomethyl)fluorosilanes. (Iodomethyl)trifluorosilane ICH<sub>2</sub>SiF<sub>3</sub> was first synthesized by exchange reactions of (iodomethyl)trichlorosilane with KHF<sub>2</sub> and SbF<sub>3</sub> in 48.7 and 87% yield, respectively [7]. When treated with potassium bifluoride, this silane is formed in low yield, and the

procedure with SbF<sub>3</sub> was not described. (Iodomethyl)dimethylfluorosilane was synthesized by splitting the siloxane bond of  $(ICH_2SiMe_2)_2O$  with potassium bifluoride [8], its yield was not reported. The structure of the products was proved by the methods of <sup>1</sup>H NMR and IR spectroscopy.

The goal of the present work was to elaborate an effective laboratory method for the synthesis of (iodomethyl)fluorosilanes ICH<sub>2</sub>SiMe<sub>n</sub>F<sub>3-n</sub> (n = 0, 2) and investigation of their reactivity. The synthesis of (iodomethyl)methyldifluorosilane and its properties will be reported elsewhere. As starting reagents, commercially available (chloromethyl)chlorosilanes ClCH<sub>2</sub>SiMe<sub>n</sub>Cl<sub>3-n</sub> (n = 0, 2) were chosen, which were converted to the target products **4** by a sequence of consecutive reactions via intermediate silanes **1–3** (Scheme 1).

Silanes **1a**, **1b** were synthesized in good yield by the reaction of the corresponding (chloromethyl)chlorosilanes  $ClCH_2SiMe_nCl_{3-n}$  with excess methanol in diethyl ether in the presence of thoroughly dried urea. The choice of urea as a hydrogen chloride acceptor was not accidental: urea hydrochloride formed in the reaction is a viscous mass or solid, which allowed simple decanting of ethereal solution of the product for further treatment. The use of triethylamine as a hydrogen chloride acceptor is less preferable because of two reasons. Firstly, the



formation of voluminous fine-disperse crystalline salt  $Et_3N \cdot HCl$  requires increasing the amount of the used solvent to perform the stage of filtration and to wash the precipitate ( $Et_3N \cdot HCl$ ). Secondly,  $Et_3N \cdot HCl$  is partly soluble in the reaction mixture; it is sublimed to the condenser during distillation and contaminates the product.

The exchange reaction of silanes **1a**, **1b** with NaI was performed in thoroughly dried solvent. Unlike a similar procedure [9], the use of excess NaI, substantial increase in the reaction time, and washing of the precipitate during filtration allowed the preparation of (iodomethyl)(methoxy)silanes **2a**, **2b** in a high yield. (Pentafluorophenyl)ethoxysilanes under the action of thionyl chloride in the presence of pyridine hydrochloride readily form the corresponding (pentafluorophenyl)chlorosilanes [10]. Under these conditions, silanes **2a**, **2b** were quantitatively converted to the corresponding (iodomethyl)chlorosilanes **3a**, **3b**.

Earlier, (iodomethyl)trifluorosilane was synthesized by the exchange reaction of (iodomethyl)trichlorosilane with SbF<sub>3</sub> by addition of SbF<sub>3</sub> to the silane [7]. However, the reaction was highly exothermic, so, we used the reverse order of mixing of the reagents: slow addition of the silane to SbF<sub>3</sub> with vigorous stirring allowed controlling the process.

The reactions of bifunctional silanes XCH<sub>2</sub>SiMe<sub>2</sub>Y with *N*- and/or *O*-trimethylsilylamides, hydrazides of carboxylic acids and related compounds leading to the



formation of (O–Si) chelate or zwitter ionic silicon compounds attract the attention of chemists during last decades [11–17]. *O*-Trimethylsilyl-substituted *N*,*N*dimethylhydrazides of carboxylic acids, when reacting with (chloromethyl)dimethylchlorosilane, form zwitter ionic heterocycles [18, 19]. In particular, from the *O*-trimethylsilyl derivative of *N*,*N*-dimethylhydrazide of trifluoroacetic acid **5**, (O–Si)-chloro-[1-(1,1-dimethyl-2-trifluoroacetylhydrazonium)methyl]dimethylsilane **6** is formed (Scheme 2). The upfield chemical shift of the silicon atom in the <sup>29</sup>Si NMR spectra of the compound (–42.9 ppm) is indicative of pentacoordinate state of the silicon atom [19].

To monitor the reactivity of the synthesized silanes, we have studied the reaction of silanes **2b–4b** with compound **5**. Silane **2b** does not react with compound **5** even on heating in chloroform (100°C, 3 h). The reaction of silane **3b** with *N*,*N*-dimethylhydrazide of trifluoroacetic acid is completed at room temperature after several minutes by the formation of 2,2,4,4-tetramethyl-6-(trifluoromethyl)-3,4-dihydro-2*H*-1,4,5,2-oxadiazasilin-4-ium iodide **7** (Scheme 3). In contrast to compound **6**, the value of chemical shift of the silicon atom in the <sup>29</sup>Si NMR spectrum of compound **7** is indicative of its tetracoordinate state (+22.02 ppm).

Silane **4b** in the reaction with hydrazide **5** also forms compound **7**, but in this case the reaction proceeds much slower. Thus, after 32 h, the <sup>1</sup>H NMR spectrum of the reaction mixture contains only traces of trimethylfluorosilane, and the reaction is completed after  $\sim$ 72 h. The structure of compound **7** was proved by <sup>1</sup>H, <sup>13</sup>C, and <sup>29</sup>Si NMR spectroscopy.

The elaborated modified procedures for the synthesis of silanes 1-4 allow the preparation of difficultly accessible bifunctional silanes in high yields (87–94%) after purification and can be used in laboratory practice. At present, the investigation of reactivity of silanes having two different halogen atoms in



resilvlation and alkylation of amides and hydrazides is in progress. We expect that variation of the number of fluorine atoms at silicon atom and the nature of halogens at carbon atom in silanes  $HalCH_2SiMe_nF_{3-n}$ will allow controlling the selectivity of the processes.

### **EXPERIMENTAL**

<sup>1</sup>H, <sup>13</sup>C, and <sup>29</sup>Si NMR spectra were registered on Bruker DPX-400 and Bruker AV-400 spectrometers (400.13, 100.61, and 161.90 MHz respectively) in CDCl<sub>3</sub> and CD<sub>3</sub>CN solutions with TMS as an internal reference.

(Chloromethyl)trichlorosilane and (chloromethyl)dimethylchlorosilane were distilled before use. The solvents were dried by known procedures [20]. All reactions were carried out in dry argon atmosphere.

(Chloromethyl)trimethoxysilane (1a). To a mixture of 86.00 g (1.43 mol) of dry urea, 45.88 g (1.43 mol) of anhydrous methanol, and 100 mL of anhydrous diethyl ether 70.23 g (0.38 mol) of chloromethyltrichloro-silane was added dropwise at stirring in the course of 2 h, and the mixture was refluxed for 2 h. After cooling the ethereal solution was decanted, ether was removed in a vacuum at 40 mmHg, and silane **2** was isolated by vacuum distillation. Yield 56.76 g (0.333 mol, 87%), colorless liquid, bp 65°C (30 mmHg),  $n_D^{20}$  1.4074. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 2.83 s (2H, CH<sub>2</sub>), 3.65 s (9H, MeO). Found, %: C 27.86; H 6.34. C<sub>4</sub>H<sub>11</sub>ClO<sub>3</sub>Si. Calculated, %: C 28.15; H 6.50.

(Chloromethyl)dimethylmethoxysilane (1b). To a mixture of 18.42 g (0.58 mol) of anhydrous MeOH, 37.70 g (0.58 mol) of urea, and 100 mL of anhydrous diethyl ether 71.54 g (0.5 mol) of (chloromethyl)-dimethylchlorosilane was added dropwise in the course of 2 h and the mixture was refluxed for 2 h. Crystallization of urea hydrochloride was initiated by cooling the reaction flask bottom by liquid nitrogen. After decanting of ethereal solution and its distillation at atmospheric pressure 64.70 g (0.467 mol, 93%) of

silane **1b** was isolated, bp 118°C (720 mmHg),  $n_D^{20}$ 1.4172. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 0.24 s (6H, SiMe<sub>2</sub>), 2.81 s (2H, CH<sub>2</sub>Cl), 3.50 s (3H, OMe). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: -3.95 (SiMe), 29.20 (CH<sub>2</sub>Cl), 50.95 (OMe). <sup>29</sup>Si NMR spectrum,  $\delta$ , ppm: 13.45. Found, %: C 34.92; H 7.58. C<sub>4</sub>H<sub>11</sub>ClOSi. Calculated, %: C 34.65; H 7.99.

(Iodomethyl)trimethoxysilane (2a). A mixture of 20.00 g (0.117 mol) of silane 1a, 26.40 g (0.176 mol) of NaI, and 50 mL of anhydrous acetonitrile was refluxed with stirring for 15 h. The precipitate was filtered off, washed with anhydrous  $Et_2O$  (2×20 mL), the ethereal solution was combined with the filtrate. The solvents were removed in a vacuum (20 mmHg), the residue was distilled in a vacuum to obtain 28.90 g (0.11 mol, 94%) of compound 2a as pink liquid (decolorized after addition of 0.1 g of copper powder), bp 76–78°C (10 mmHg),  $n_D^{20}$  1.4691. <sup>1</sup>H NMR spectrum, δ, ppm: 1.97 s (2H, CH<sub>2</sub>), 3.64 s (9H, 3MeO). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: -26.83 (CH<sub>2</sub>), 51.54 (MeO). <sup>29</sup>Si NMR spectrum,  $\delta$ : -53.20 ppm. Found, %: C 18.59; H 4.78. C<sub>4</sub>H<sub>11</sub>IO<sub>3</sub>Si. Calculated, %: C 18.33; H 4.23.

(Iodomethyl)dimethylmethoxysilane (2b). A mixture of 40.0 g (0.288 mol) of silane 1b, 67.50 g (0.438 mol) of NaI in 50 mL of anhydrous ethyl acetate was refluxed at stirring for 93 h. The reaction mixture was filtered, the precipitate was washed with anhydrous ether (40 mL), combined filtrates were evaporated at a reduced pressure, the residue was distilled with 3 g of copper powder to obtain 55.60 g (0.242 mol, 83.8%) of silane 2b as colorless liquid, bp 74°C (40 mmHg),  $n_D^{20}$  1.4870. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 0.29 s (6H, SiMe<sub>2</sub>), 2.04 s (2H, CH<sub>2</sub>I), 3.49 s (3H, MeO). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: -15.60 (CH<sub>2</sub>I), -3.09 (SiMe), 50.82 (MeO). <sup>29</sup>Si NMR spectrum,  $\delta$ , ppm: 14.41. Found, %: C 21.17; H 4.47. C<sub>4</sub>H<sub>11</sub>IOSi. Calculated, %: C 20.88; H 4.82.

(**Iodomethyl)trichlorosilane (3a).** To a mixture of 18.30 g (0.07 mol) of silane **2a** and 1.04 g (9.0 mmol) of pyridine hydrochloride 50.60 g (0.425 mol) of

thionyl chloride was slowly added dropwise at vigorous stirring in the course of 2 h. Slow addition is required because of evolution of two gaseous products (SO<sub>2</sub> and HCl). The reaction mixture was stirred for 2 h until the evolution of gaseous products ceased, then it was refluxed for another 4 h. Such a long reflux is needed because after a shorter period (2 h) the reaction mixture, according to <sup>1</sup>H NMR, contains ~20% of ICH<sub>2</sub>Si(OMe)Cl<sub>2</sub>. <sup>1</sup>H NMR spectrum, δ, ppm: 2.41 s (2H, CH<sub>2</sub>), 3.73 s (3H, MeO). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: -20.25 (CH<sub>2</sub>), 52.59 (MeO). Excess of thionyl chloride and volatile products were removed at 20 mmHg, the residue was distilled in a vacuum to obtain 17.10 g (0.062 mol, 89%) of silane 3a. Colorless liquid, bp 64–67°C (20 mmHg),  $n_{\rm D}^{20}$ 1.5322. <sup>1</sup>H NMR spectrum, δ, ppm: 2.67 s (2H, CH<sub>2</sub>). <sup>13</sup>C NMR spectrum, δ, ppm: -16.21. <sup>29</sup>Si NMR spectrum, δ, ppm: 1.81.

(Iodomethyl)dimethylchlorosilane (3b). To a mixture of 26.02 g (0.219 mmol) of thionyl chloride and 1.66 g (0.014 mol) of Py·HCl 27.60 g (0.120 mol) of silane **2b** was added dropwise at stirring for 2 h. The reaction mixture was refluxed for 3 h. Volatile products and excess of thionyl chloride were removed at 40 mmHg, the precipitate of Py·HCl was filtered off, the residue was distilled over ~3 g of copper powder. Yield 20.13 g (0.086 mol, 71.6 %), colorless liquid, bp 74°C (42 mmHg),  $n_D^{20}$  1.5073. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 0.60 s (6H, SiMe<sub>2</sub>), 2.24 s (2H, CH<sub>2</sub>I). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: -14.20 (CH<sub>2</sub>I), 0.96 (SiMe). <sup>29</sup>Si NMR spectrum,  $\delta$ , ppm: 24.68. Found, %: C 14.91; H 2.98. C<sub>3</sub>H<sub>8</sub>CIISi. Calculated, %: C 15.36; H 3.44.

(Iodomethyl)trifluorosilane (4a). To 10.00 g (0.056 mol) of SbF<sub>3</sub> 15.00 g (0.055 mol) of silane **3a** was added dropwise at vigorous stirring in the course of 1 h and the mixture was stirred for 1 h. After recondensation to an evacuated Schlenk flask at 0.1 mmHg, 11.36 g (0.050 mol, 92%) of silane **4a** was isolated as a slightly pink liquid,  $n_D^{20}$  1.4112. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 2.15 q (2H, CH<sub>2</sub>, <sup>3</sup> $J_{HF}$  = 2.8 Hz). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: -37.12 q (CH<sub>2</sub>, <sup>2</sup> $J_{CF}$  = 23.6 Hz). <sup>29</sup>Si NMR spectrum,  $\delta$ , ppm: -68.41 q (<sup>3</sup> $J_{SiF}$  = 262.4 Hz). Found, %: C 4.96; H 0.81. CH<sub>2</sub>F<sub>3</sub>ISi. Calculated, %: C 5.31; H 0.89.

**Iodomethyldimethylfluorosilane (4b).** To 3.73 g (62.3 mmol) of SbF<sub>3</sub> 14.61 g (62.3 mmol) of silane **3b** was added dropwise at stirring for 2 h, the mixture was refluxed for 1 h, the product was isolated by distillation. Yield 13.00 g (56.9 mmol,

95.7 %), colorless liquid, bp 69°C (100 mmHg),  $n_D^{20}$ 1.4720. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 0.42 d (6H, SiMe<sub>2</sub>, <sup>3</sup>*J*<sub>HF</sub> = 7.2 Hz), 2.09 d (2H, CH<sub>2</sub>I, <sup>3</sup>*J*<sub>HF</sub> = 3.0 Hz). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: -16.97 d (CH<sub>2</sub>I, <sup>2</sup>*J*<sub>CF</sub> = 17.4 Hz), -2.23 d (MeSi, <sup>2</sup>*J*<sub>CF</sub> = 14.8 Hz). <sup>29</sup>Si NMR spectrum,  $\delta$ , ppm: 26.53 d (*J*<sub>SiF</sub> = 281.6 Hz). Found, %: C 16.19; H 3.24. C<sub>3</sub>H<sub>8</sub>FISi. Calculated, %: C 16.52; H 3.69.

2,2,4,4-Tetramethyl-6-(trifluoromethyl)-3,4-dihydro-2H-1,4,5,2-oxadiazasilin-4-ium iodide (7). A mixture of silane **3b** 1.36 g (5.96 mmol), 10 mL of anhydrous chloroform, and 1.27 g (5.41 mmol) of compound 5 was stirred for 30 min at 20°C. Volatile compounds were removed at a reduced pressure, solid residue was washed with anhydrous diethyl ether  $(2 \times 30 \text{ mL})$  and dried in a vacuum (0.1 mmHg). Yield 1.64 g (4.63 mmol, 85.6%), colorless crystals, mp (evacuated capillar) 128.5°C. <sup>1</sup>H NMR spectrum (CD<sub>3</sub>CN),  $\delta$ , ppm: 0.76 s (6H, SiMe<sub>2</sub>), 3.64 s (6H, NMe<sub>2</sub>), 4.07 s (2H, CH<sub>2</sub>). <sup>13</sup>C NMR spectrum (CD<sub>3</sub>CN),  $\delta$ , ppm: -0.84 (MeSi), 54.56 (H<sub>2</sub>CSi), 59.86 (MeN), 116.31 q (CF<sub>3</sub>,  $J_{CF} = 278.4$  Hz), 152.80 q (OC=N,  ${}^{2}J_{CF} = 39.4$  Hz).  ${}^{29}Si$ NMR spectrum (CD<sub>3</sub>CN),  $\delta$ , ppm: 22.02. Found, %: C 23.80; H 3.76; N 7.82; Si 8.01; F 15.96; I 36.02. C<sub>7</sub>H<sub>14</sub>N<sub>2</sub>OSiF<sub>3</sub>I. Calculated, %: C 23.74; H 3.98; N 7.91; Si7.93; F 16.09; I 35.83.

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# CONFLICT OF INTERESTS

No conflict of interests was declared by the authors.

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