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Reaction of *N*-[Chloro(diorganyl)silyl]anilines with Isopropanol and Isopropylamine

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Abstract—Reaction of *N*-[chloro(methylvinyl)silyl]-, *N*-[chloro(methylphenyl)silyl]anilines and of *N*-[chloro-(methylvinyl)silyl]-, *N*-[chloro(methylphenyl)silyl]-*N*-methylanilines with isopropylamine and isopropanol yielding the products of chlorine substitution with isopropoxy and isopropylamino group, respectively, has been studied. Structure of new polyfunctional silanes has been confirmed by IR and NMR spectroscopy.

Keywords: *N*-silylamines, *N*-[chloro(diorganyl)silyl]anilines, (diorganyl)diaminosylanes

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N-Silylamines have found wide application in organic synthesis as synthons [1–5] and silylating agents [6–9] as well as in the material science as precursors for preparation and modification of polymer materials [10–13], protecting films [14, 15], and advanced materials based on silicon nitride and oxynitride [16–20]. The first reports on synthesis of polyfunctional silanes of the general formula $R^1R^2Si(Cl)NR^1R^2$ (R^1, R^2 = H, Alk, or Ar) date back to the second half of the XX century [20–25], and some data on their chemical properties have been published [26, 27]. However, only a few of such silanes have been prepared, and their structure and properties have been scarcely studied.

We have recently synthesized a series of *N*-[chloro-(diorganyl)silyl]anilines as promising synthons for preparation of polyfunctional silanes and silanols containing simultaneously alkoxy, hydroxyl, and amino groups at the silicon atom [28]. Herein we report on the reactions of *N*-[chloro(methylvinyl)silyl]- and *N*-[chloro(methylphenyl)silyl]aniline as well as *N*-[chloro(methylvinyl)silyl]- and *N*-[chloro(methyl-phenyl)silyl]-*N*-methylaniline with isopropanol and isopropylamine. The choice of the silanes has been based on the following reasons: the vinyl group at silicon is capable of addition and polymerization reactions [29–32], while the phenyl group at silicon serves as a latent functional group because the $Si–C_{Ar}$ bond is capable of to splitting [33–35], substantially expanding the synthetic potential of the final products and allowing preparation of new derivatives with various substituents at silicon (Scheme 1).

The reactions were carried out in toluene in the presence of triethylamine as a scavenger of the formed hydrogen chloride, either upon heating (the reactions with isopropanol) or at room temperature (the reactions with isopropylamine). The products of chlorine substitution with isopropoxy **I–IV** or isopro-pylamino group **V–VIII** were isolated via vacuum distillation. Noteworthily, the yields of compounds **I**

Scheme 1.

$$PhR^3NSiR^1R^2O\textit{i}\text{-}Pr \xleftarrow[-Et_3N\cdot HCl]{\textit{i}\text{-}PrOH,\ Et_3N,\ 60^\circ C,\ 6\ h} PhR^3NSiR^1R^2Cl \xrightarrow[-Et_3N\cdot HCl]{\textit{i}\text{-}PrNH_2,\ Et_3N,\ T_{room},\ 6\ h} PhR^3NSiR^1R^2NH\textit{i}\text{-}Pr$$

R^1 = vinyl, R^2 = Me, R^3 = H (**I**); R^1 = vinyl, R^2 = Me, R^3 = Me (**II**); R^1 = Ph, R^2 = Me, R^3 = H (**III**); R^1 = Ph, R^2 = Me, R^3 = Me (**IV**); R^1 = vinyl, R^2 = Me, R^3 = H (**V**); R^1 = vinyl, R^2 = Me, R^3 = Me (**VI**); R^1 = Ph, R^2 = Me, R^3 = H (**VII**); R^1 = Ph, R^2 = Me, R^3 = Me (**VIII**).

and **III** were of 65% and 45%, respectively, whereas compounds **II** and **IV** were prepared with yields of 73% and 68%, respectively. Apparently, the lower target products yield in the cases of *N*-silylanilines was owing to the side reactions at the NH giving rise to tarry unidentified polymers and siloxanes. The obtained compounds **I–VIII** were colorless or light-yellow liquids, very sensitive to the air moisture. The silanes **V–VIII** became cloudy and turned yellow or orange upon storage, apparently due to the metathesis reactions. The structure of all synthesized compounds was confirmed by IR and NMR spectroscopy data, and their composition was determined via elemental analysis. NMR spectra of compounds **III** and **VI** contained signals of siloxanes trace because of the high hydrolytic lability. Chemical shifts of the methyl, vinyl, and phenyl groups in the ^{1}H and ^{13}C NMR spectra were close to those for structurally similar *N*-silylamines [36–38]. IR spectra of silanes **I**, **III,** and **V–VIII** contained the absorption bands of NH groups stretching at 3380–3400 cm^{-1}. Yields, physico-chemical parameters, and spectral characteristics are given in the Experimental.

EXPERIMENTAL

NMR spectra of the solutions in $CDCl_3$ were recorded using a Bruker DPX-400 spectrometer [400.1 (^{1}H), 100.6 (^{13}C), 79.5 MHz (^{29}Si)] with HMDS and cyclohexane as an internal references. IR spectra were registered using a Bruker Vertex 70 spectrometer. All the reactions were performed under argon atmosphere in thoroughly dried solvents purified via the known procedures [39]. Isopropanol and isopropylamine were distilled prior to use.

***N*-[(Isopropoxy)methylvinylsilyl]aniline (I).** A mixture of 0.29 g (4.82 mmol) of isopropanol and 0.6 g (5.93 mmol) of triethylamine in 5 mL of toluene was slowly added dropwise upon vigorous stirring to a solution of 0.96 g (4.85 mmol) of *N*-[methylvinyl(chloro)-silyl]aniline in 10 mL of toluene. The mixture was heated on a water bath to 50–60°C and stirred at that temperature during 6 h. After the reaction was over, the mixture was filtered, the precipitate was washed with 30 mL of toluene, and the solvent removed on a rotary evaporator. The obtained yellow oily residue was distilled in vacuum. Yield 65%, bp 82°C (2 mmHg). IR spectrum (KBr), ν, cm^{-1}: 927, 1282, 1601, 2816, 2972, 3383. ^{1}H NMR spectrum, δ, ppm: 0.32 s (3H, CH_3Si), 1.16 d [6H, ($C\underline{H}_3)_2CH$, *J* 8.1 Hz], 3.74 br.s (1H, PhN<u>H</u>), 4.17 m [1H, $(CH_3)_2C\underline{H}$], 5.93–6.25 m (3H, CH_2=CH), 6.71–7.22 m (5H, PhN). ^{13}C NMR spectrum, δ_C, ppm: –3.2 ($\underline{C}H_3Si$), 25.5 [($\underline{C}H_3)_2CH$], 65.1 [$(CH_3)_2\underline{C}H$], 115.4 (C^{p}), 116.7 (C^{o}), 118.0 ($\underline{C}H_2$=CH), 129.1(C^{m}), 135.2 (CH_2=$\underline{C}H$), 146.1 (C^{ipso}). ^{29}Si NMR spectrum, δ_{Si}: –20 ppm. Found, %: C 64.83; H 8.68; N 5.55; Si 12.59. $C_{12}H_{19}NOSi$. Calculated, %: C 65.11; H 8.65; N 6.33; Si 12.69.

***N*-[Isopropoxymethylvinylsilyl]methylaniline (II).** Yield 73%, bp 130°C (10 mmHg), n_D^{20} 1.5086. IR spectrum (KBr), ν, cm^{-1}: 1027, 1269, 1600, 2816, 2972. ^{1}H NMR spectrum, δ, ppm: 0.35 s (3H, CH_3Si), 1.17 d (6H, $[C\underline{H}_3)_2CH$, *J* 10 Hz], 2.96 s (3H, CH_3N), 4.13 m [1H, $(CH_3)_2C\underline{H}$], 5.79–6.22 m (4H, CH_2=CH), 6.81–7.23 m (5H, PhN). ^{13}C NMR spectrum, δ_C, ppm: –3.0 ($\underline{C}H_3Si$), 25.0 [($\underline{C}H_3)_2CH$], 34.4 (CH_3N), 64.7 [$(CH_3)_2\underline{C}H$], 117.6 (C^{o}), 118.7 (C^{p}), 128.0 (C^{m}), 133.7 ($\underline{C}H_2$=CH), 135.0 (CH_2=$\underline{C}H$), 149.4 (C^{ipso}). ^{29}Si NMR spectrum, δ_{Si}: –15 ppm. Found, %: C 66.37; H 8.68; N 6.23; Si 11.04. $C_{13}H_{21}NOSi$. Calculated, %: C 66.33; H 8.99; N 5.95; Si 11.93.

***N*-[Isopropoxymethylphenylsilyl]aniline (3).** Yield 45%, bp 155°C (2 mmHg). n_D^{20} 1.5308. IR spectrum (KBr), ν, cm^{-1}: 1026, 1120, 1603, 2972, 3383 (NH). ^{1}H NMR spectrum, δ, ppm: 0.47 s (3H, CH_3Si), 1.16 d [6H, $(C\underline{H}_3)_2CH$, *J* 17.4 Hz], 3.90 br.s (1H, PhN<u>H</u>), 4.21 m [1H, $(CH_3)_2C\underline{H}$], 6.60–7.11 m (5H, PhN), 7.28–7.69 m (5H, PhSi). ^{13}C NMR spectrum, δ_C, ppm: –2.3 ($\underline{C}H_3Si$), 25.7 [($\underline{C}H_3)_2CH$], 65.4 [$(CH_3)_2\underline{C}H$], 117.0 (C^{o}, PhN), 118.4 (C^{p}, PhN), 128.0 (C^{m}, PhN), 129.1 (C^{m}, PhSi), 130.4 (C^{p}, PhSi), 134.2 (C^{o}, PhSi), 135.7 (C^{ipso}, PhSi), 146.2 (C^{ipso}, PhN). ^{29}Si NMR spectrum, δ_{Si}: –19 ppm. Found, %: C 70.79; H 7.79; N 4.28; Si 11.49. $C_{16}H_{21}NOSi$. Calculated, %: C 70.8; H 7.8; N 5.16; Si 10.35.

***N*-[Isopropoxymethylphenylsilyl]methylaniline (IV)**. Yield 68%, bp 152°C (2 mmHg), n_D^{20} 1.5392. IR spectrum (KBr), ν, cm^{-1}: 1027, 1118, 1600, 1269, 2816, 2972. ^{1}H NMR spectrum, δ, ppm: 0.48 s (3H, CH_3Si), 1.22 d (6H, $(C\underline{H}_3)_2CH$, *J* 19 Hz), 3.00 s (3H, CH_3N), 4.18 m [1H, $(CH_3)_2C\underline{H}$], 6.76–7.16 m (5H, PhN), 7.29–7.61 m (5H, PhSi). ^{13}C NMR spectrum, δ_C, ppm: –1.2 ($\underline{C}H_3Si$), 25.6 [($\underline{C}H_3)_2CH$], 35.4 ($\underline{C}H_3N$), 65.6 [$(CH_3)_2\underline{C}H$], 118.6 (C^{o}, PhN), 119.3 (C^{p}, PhN), 127.8 (C^{m}, PhN), 128.5 (C^{m}, PhSi), 129.6 (C^{p}, PhSi), 134.1 (C^{o}, PhSi), 136.6 (C^{ipso}, PhSi), 150.0 (C^{ipso}, PhN). ^{29}Si NMR spectrum, δ_{Si}: –15 ppm. Found, %: C 71.5; H 8.14; N 4.64; Si 10.1. $C_{17}H_{23}NOSi$. Calculated, %: C 71.53; H 8.12; N 5.6; Si 9.84.

***N*-[Isopropylaminomethylvinylsilyl]aniline (V).** A solution of 0.11 g (1.86 mmol) of isopropylamine and 0.22 g (2.1 mmol) of triethylamine in 5 mL of toluene was slowly added dropwise at vigorous stirring to a solution of 0.38 g (1.8 mmol) of *N*-[methylvinyl(chloro)-silyl]aniline in 7 mL of toluene. The reaction mixture was stirred at room temperature during 6 h, the precipitate of triethylamine hydrochloride was filtered off and washed with toluene (15 mL), the filtrate was combined with the toluene extract; the solvent was removed on a rotary evaporator, and the residue was distilled in vacuum. Yield 71%, bp 82°C (2 mmHg). IR spectrum (KBr), ν, cm^{-1}: 901, 1019, 1291, 1602, 2868, 2959, 3384 (NH). ^{1}H NMR spectrum, δ, ppm: 0.25 s (3H, CH_3Si), 1.03 d [6H, $(C\underline{H}_3)_2CH$, *J* 6.4 Hz], 3.11 m [1H, $(CH_3)_2C\underline{H}$], 3.50 br.s (1H, *i*-PrN<u>H</u>), 3.68 br.s (1H, PhN<u>H</u>), 5.82–6.34 m (3H, CH_2=CH), 6.71–7.23 m (5H, PhN). ^{13}C NMR spectrum, δ_C, ppm: –2.9 ($\underline{C}H_3Si$), 27.3 [$(\underline{C}H_3)_2CH$], 116.1 (C^p), 117.0 (C^o), 118.3 ($\underline{C}H_2$=CH), 129.0 (C^p), 135.8 (CH_2=$\underline{C}$H), 145.7 (C^{ipso}). ^{29}Si NMR spectrum, δ_{Si}: –25 ppm. Found, %: C 65.24; H 9.24; N 13.41; Si 12.38. $C_{12}H_{20}N_2Si$. Calculated, %: C 65.4; H 9.15; N 12.71; Si 12.74.

***N*-[Isopropylaminomethylvinylsilyl]methylaniline (VI).** Yield 70%, bp 86°C (2 mmHg), n_D^{20} 1.5186. IR spectrum (KBr), ν, cm^{-1}: 879, 1017, 1269, 1590, 2814, 2958, 3398 (NH). ^{1}H NMR spectrum, δ, ppm: 0.29 s (3H, CH_3Si), 1.01 d (6H, $(C\underline{H}_3)_2CH$, *J* 11 Hz), 2.78 br.s (1H, *i*-PrN<u>H</u>), 2.94 s (3H, CH_3N), 3.11 m (1H, $[CH_3)_2C\underline{H}$], 5.81–6.27 m (4H, CH_2=CH), 6.80–7.26 m (5H, PhN). ^{13}C NMR spectrum, δ_C, ppm: –2.3 ($\underline{C}H_3Si$), 26.4 [$(\underline{C}H_3)_2CH$], 34.8 (CH_3N), 42.3 [$(CH_3)_2\underline{C}H$], 117.1 (C^o), 118.0 (C^p), 127.8 (C^m), 128.2 ($\underline{C}H_2$=CH), 132.4 (CH_2=$\underline{C}$H), 137.1 (C^{ipso}). ^{9}Si NMR spectrum, δ_{Si}: –17 ppm. Found, %: C 65.41; H 9.34; N 12.08; Si 11.59. $C_{13}H_{22}N_2Si$. Calculated, %: C 66.61; H 9.46; N 11.95; Si 11.98.

***N*-[Isopropylaminomethylphenylsilyl]aniline (VII).** Yield 65%, bp 182°C (2 mmHg), n_D^{20} 1.5586. IR spectrum (KBr), ν, cm^{-1}: 1125, 1290, 1601, 2868, 2958, 3387 (NH). ^{1}H NMR spectrum, δ, ppm: 0.52 s (3H, CH_3Si), 1.15 d [6H, $(C\underline{H}_3)_2CH$, *J* 11 Hz], 3.27 m [1H, $(CH_3)_2C\underline{H}$], 3.82 br.s (1H, PhN<u>H</u>), 6.39–7.21 m (5H, PhN), 7,40–7.71 m (5H, PhSi). ^{13}C NMR spectrum, δ_C, ppm: –1.9 ($\underline{C}H_3Si$), 27.8 [$(\underline{C}H_3)_2CH$)], 43.3 [$(CH_3)_2\underline{C}H$], 117.2 (C^o, PhN), 118.0 (C^p, PhN), 128.3 (C^m, PhN), 129.1 (C^m, PhSi), 129.7 (C^p, PhSi), 134.0 (C^o, PhSi), 137.7 (C^{ipso}, PhSi), 145.9 (C^{ipso}, PhN). ^{29}Si NMR spectrum, δ_{Si}: –20 ppm. Found, %: C 70.07; H 7.92; N 10.59; Si 10.11. $C_{16}H_{22}N_2Si$. Calculated, %: C 71.06; H 8.2; N 10.36; Si 10.38.

***N*-[Isopropylaminomethylphenylsilyl]methylaniline (VIII).** Yield 62%, bp 166°C (2 mmHg), n_D^{20} 1.5556. IR spectrum (KBr), ν, cm^{-1}: 1018, 1126, 1272, 1590, 2813, 2959, 3393 (NH). ^{1}H NMR spectrum, δ, ppm: 0.46 s (3H, CH_3Si), 1.02 br. d [6H, $(C\underline{H}_3)_2CH$, *J* 7.4 Hz], 3.00 s (3H, CH_3N), 3.13 m [1H, $(CH_3)_2C\underline{H}$], 6.75–7.16 m (5H, PhN), 7.28–7.61 m (5H, PhSi). ^{13}C NMR spectrum, δ_C, ppm: –1.1 ($\underline{C}H_3Si$), 27.1 [$(\underline{C}H_3)_2CH$], 35.7 ($\underline{C}H_3N$), 43.1 [$(CH_3)_2\underline{C}H$], 118.7 (C^o, PhN), 119.0 (C^p, PhN), 127.9 (C^m, PhN), 128.3 (C^m, PhSi), 129.3 (C^p, PhSi), 134.2 (C^o, PhSi), 138.3 (C^{ipso}, PhSi), 150.8 (C^{ipso}, PhN). ^{29}Si NMR spectrum, δ_{Si}: –14.5 ppm. Found, %: C 71.4; H 8.56; N 9.27; Si 9.88. $C_{17}H_{24}N_2Si$. Calculated, %: C 71.78; H 8.5; N 9.85; Si 9.87

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