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Synthesis of *N*-[Chloro(diorganyl)silyl]anilines

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Abstract—A series of *N*-[chloro(diorganyl)silyl]anilines RR'Si(NR"Ph)Cl (R, R' = Me, Ph, CH_2=CH, $ClCH_2$, $Cl(CH_2)_3$; R" = H, Me) was prepared via the reaction of diorganyldichlorosilanes with aniline or *N*-ethylaniline in the presence of triethylamine.

Keywords: *N*-silylamine, *N*-[Chloro(diorganyl)silyl]aniline, aniline, silane

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Chemistry of *N*-silylated amines has been intensively developing over the last fifty years [1–6]. The interest to these compounds is due to their unique physico-chemical properties: the organosilicon substituent adjacent to the nitrogen atom causes structural changes of the amino group [7–11] and decreases the nitrogen atom basicity [12–14]. These compounds are widely used as synthons [15–20] and silylating agents [21–24] in synthetic organic and organoelement chemistry. *N*-Silylamines are promising volatile precursors for chemical vapor deposition of silicon nitride and oxynitride as well as for preparation of new materials, including metallated nanomaterials and membranes [25–30]. To date, several thousands of monosilylated amines have been synthesized, and their structure and chemical properties have been characterized. Extending our studies of reactivity of α-silylamines [31, 32] we aimed to investigate chemical properties of the following type of compounds: $RR'NCH_2Si[N(Ar)R"]_nX_{3-n}$ (R, R', R" = Alk, Ar; X = Alk, Ar, OAlk, OAr; n = 1–3). So far, they have been scarcely studied: to the best of our knowledge there are only a few publications on their synthesis and properties [33–36]. Amination of halogenomethylsilanes with amines is known as the most convenient synthetic route to α-silylamines $RR'NCH_2SiX_3$ [37, 38]. Therefore, the target compounds to be studied in this work could be prepared via reaction of the corresponding (halogenomethyl)(*N*-arylamino)silanes $HalCH_2Si(NarR")_nX_{3-n}$ with amines. However, only a few (halogenomethyl)(*N*-arylamino)silanes have been described in the literature: bromo- and chloromethyl(dimethyl)anilinosilanes $PhNHSiMe_2CH_2Hal$ [35, 39–41], chloromethyl(dimethyl)(*N*-methylanilino)silane $PhN(Me)SiMe_2CH_2Cl$ [42], *o*- and *p*-chloromethyl(dimethyl)(*N*-trimethylsiloxyanilino)silane $Me_3SiOC_6H_4NH{\cdot}SiMe_2CH_2Cl$ [43], whereas information on synthesis of polyfunctional amino(chloro)silanes $HalCH_2SiR(NArR')Hal$ has been missing. Noteworthily, even the simplest mixed diorganyl(chloro)aminosilanes $RR'Si(NR_2)Cl$ has been almost not studied. Therefore, the goal of this work has transformed into elaborating the method to prepare *N*-[chloro(diorganyl)silyl]anilines RR'Si(NR"Ph)Cl (R, R' = Me, Ph, CH_2=CH, $ClCH_2$, $Cl(CH_2)_3$; R" = H, Me), new promising synthons for organic synthesis and material chemistry. The only compound of this series described in the literature is (anilino)dimethylchlorosilane [44].

The synthesis conditions were optimized using the reaction of dimethylchlorosilane with *N*-methylaniline as an example, effects of the solvent, the base, temperature and the ratio of the reagents on the yield of the reaction product **I** was investigated (Table 1).

$$Me_2SiCl_2 + PhNHMe \rightarrow PhMeNSiMe_2Cl.$$
I

The reaction was performed by slow addition of the solution of the mixture of *N*-methylaniline and acceptor of HCl (triethylamine, pyridine, or excess of *N*-methylaniline) to the solution of the silane, not allowing the temperature to rise above 25°C, followed by heating (if needed) to the desired reaction

Effect of solvent, base, ratio of reagents, and temperature on the yield of compound **I**

Solvent	Base	Ratio of reagents (silane : aniline : base)	Temperature, °C	Time, h	Yield, %
Et_2O	Et_3N	1 : 1 : 1.1	r.t.	48	56
Et_2O	Et_3N	1 : 1 : 1.1	r.t.	48	32[a]
Et_2O	Et_3N	1 : 1 : 1.1	r.t.	96	62
Et_2O	Et_3N	1 : 1 : 1.1	34	12	42
THF	Et_3N	1 : 1 : 1.1	r.t.	48	52
THF	Et_3N	1 : 1 : 1.1	66	12	49
Hexane	Et_3N	1 : 1 : 1.1	r.t.	48	58
Hexane	Et_3N	1 : 1 : 1.1	68	10	59
Benzene	Et_3N	1 : 1 : 1.1	r.t.	48	72
Benzene	Et_3N	1 : 1 : 1.1	50	24	76
Benzene	Et_3N	1 : 1 : 1.1	50	24	40[a]
Benzene	Et_3N	1 : 1 : 1.1	80	10	73
Benzene	Et_3N	1 : 1 : 1.1	80	24	76
Benzene	pyridine	1 : 1 : 1.1	50	24	75
Benzene	Et_3N	1 : 1 : 2	50	24	77
Benzene	PhMeNH	1 : 2	r.t.	48	19
Benzene	PhMeNH	1 : 2	80	48	44
Toluene	Et_3N	1 : 1 : 1.1	60	24	80
Toluene	Et_3N	1 : 1 : 1.1	110	10	75

[a] The reaction was performed by dropwise addition of the silane to the mixture of amines.

temperature after the reagents we fully mixed. As follows from Table 1, the highest yield of compound **I** was observed when using benzene or toluene as a solvent. The optimal time of heating depends on the temperature (in particular, 10 h at reflux and 24 h at 50°C). Further increase of the reaction time did not improve the target product yield, but led to formation of small amount of bis(*N*-methylanilino)dimethylsilane. Reverse order of the reagents mixing resulted in substantial decrease of the of the target product yield accompanied by formation of large amount (up to 35%) of bis(*N*-methylanilino)dimethylsilane, a side product of the reaction. Using pyridine instead of triethylamine or introduction of large excess of triethylamine had no noticeable effect on the yield of the product.

N-[Chloro(diorganyl)silyl]anilines RR'Si[N(R")Ar]Cl (**II–XI**) were synthesized via the reaction of the corresponding diorganyldichlorosilanes with aniline or *N*-methylaniline in the presence of triethylamine as scavenger of HCl in benzene or toluene as solvent.

$$R^1R^2SiCl_2 + PhNHR^3 \rightarrow PhR^3NSiR^1R^2Cl,$$

R^1 = Me, R^2 = Ph, R^3 = H (**II**); R^1 = Me, R^2 = Ph, R^3 = Me (**III**); R^1 = Me, R^2 = Vinyl, R^3 = H (**IV**); R^1 = Me, R^2 = Vinyl, R^3 = Me (**V**); $R^1 = R^2$ = Ph, R^3 = H (**VI**); $R^1 = R^2$ = Ph, R^3 = Me (**VII**); R^1 = Me, $R^2 = ClCH_2$, R^3 = H (**VIII**); R^1 = Me, $R^2 = ClCH_2$, R^3 = Me (**IX**); R^1 = Me, $R^2 = Cl(CH_2)_3$, R^3 = H (**X**); R^1 = Me, $R^2 = Cl(CH_2)_3$, R^3 = Me (**XI**).

The obtained compounds were viscous, sometimes slowly crystallizing yellowish liquids with specific odor. The refractive index was determined only for a

few selected compounds, as high viscosity and susceptibility to hydrolysis did not allow for reliable results. Noteworthily, the reaction of diorganyldichlorosilanes with *N*-methylaniline proceeded more smoothly than in the case of aniline, leading to formation of the corresponding *N*-[chloro(diorganyl)silyl]-*N*-methylanilines in satisfactory yields (53–80%). As side products, in some cases small amounts (3–5%) of diorganyl-bis(*N*-methylanilino)silanes RR'Si(NMePh)$_2$ could be isolated. The yield of *N*-[chloro(diorganyl) silyl]anilines was much lower (15–64%). Apparently, that was mainly due to side reactions between the NH- and SiCl-containing species at high temperature upon distillation. The residue after distillation of the reaction mixture appeared as resin and contained a number of non-identified Si-containing products. The ^{29}Si NMR spectra of the distillation residue normally contained five to ten signals of varied intensity. Unfortunately, all trials purify *N*-[chloro(diorganyl)silyl]anilines by means of column chromatography failed. Structure of the prepared compounds was confirmed by multinuclear NMR experiments (^{1}H, ^{13}C, and ^{29}Si).

The prepared polyfunctional silanes are of high synthetic potential. In particular, we believe that the presence of labile Si–Cl bond will allow using them as synthons to prepare *N*-([diorganyl]hydroxysilyl)anilines, *N*-([diorganyl]alkoxysilyl)anilines, and *N*-([diorganyl]-aminosilyl)anilines as well as nitrogen- and silicon-containing heterocycles.

EXPERIMENTAL

NMR spectra of the synthesized compounds were registered in $CDCl_3$ solution with the Bruker DPX-400 instrument (^{1}H, 400.1 MHz; ^{13}C 100.6 MHz; ^{29}Si, 79.5 MHz) with HMDS or cyclohexane as internal reference. All reactions were performed using the thoroughly dried solvents purified by the known procedures [45]. Triethylamine, anilines, and silanes were distilled before use.

Synthesis of compounds I–XI *(general procedure).* Three-necked flask equipped with a thermometer, dropping funnel and condenser was placed at water bath and charged with the solvent and the silane (150 mL of benzene or toluene per 0.1 mol of silane). Mixture of the corresponding aniline or *N*-methylaniline with triethylamine in the same solvent (100 mL of the solvent per 0.1 mol of silane) was slowly added dropwise to the flask upon vigorous stirring, not allowing the temperature to rise above 20–25°C. The molar ratio amine:silane was of 1 : 1, and 15–20% excess of triethylamine was used. When the aniline addition was complete, the reaction mixture was heated to 50–60°C with stirring on the water bath during 4–5 days. The formed precipitate was filtered off and carefully washed with the same solvent. The solvent was removed from the filtrate with rotary evaporator, the residue was distilled in vacuum.

***N*-[Chloro(dimethyl)silyl]-*N*-methylaniline (I).** Yield 80%, bp 68°C (2–3 mmHg), n_D^{20} 1.5212. ^{1}H NMR spectrum, δ, ppm: 0.1 s (6H, CH_3Si), 2.96 s (3H, CH_3N), 7.23–7.46 m (5H, PhN). ^{13}C NMR spectrum, δ, ppm: 1.78 ($\underline{C}H_3Si$), 26.7 ($\underline{C}H_3N$), 122.5 (C_o), 129.3 (C_p), 130.2 (C_m), 137 (C_i). ^{29}Si NMR spectrum, δ, ppm: 7.38. Found, %: C 54.36; H 7.41; N 5.12. $C_9H_{14}ClNSi$. Calculated, %: C 54.12; H 7.06; N 7.01.

***N*-[Chloro(phenylmethyl)silyl]aniline (II).** Yield 20%, bp 150°C (2 mmHg). ^{1}H NMR spectrum, δ, ppm: 0.8 s (3H, CH_3), 4.06 br.s (1H, NH), 6.7–7.1 m (5H, PhN), 7.4–7.7 m (5H, PhSi). ^{13}C NMR spectrum, δ_C, ppm: 1.8 ($\underline{C}H_3Si$), 117.3 (C_p, PhN), 120.6 (C_o, PhN), 128.3 (C_m, PhN), 129 (C_m, PhSi), 130.8 (C_p, PhSi), 132.9 (C_i, PhSi), 133.7 (C_o, PhSi), 144.1 (C_i, PhN). ^{29}Si NMR spectrum, δ, ppm: 7.46. Found, %: C 62.74; H 5.28; N 5.43. $C_{13}H_{14}ClNSi$. Calculated, %: C 63.01; H 5.69; N 5.65.

***N*-[Chloro(phenylmethyl)silyl]-*N*-methylaniline (III).** Yield: 55%, bp 116°C (2 mmHg). ^{1}H NMR spectrum, δ, ppm: 0.67 s (3H, CH_3Si), 2.9 s (3H, CH_3N), 6.9–7.2 m (5H, PhN), 7.3–7.6 m (5H, PhSi). ^{13}C NMR spectrum, δ, ppm: 2.6 ($\underline{C}H_3Si$), 37 ($\underline{C}H_3N$), 112.4 (C_o, PhN), 122.2 (C_p, PhN), 128 (C_m, PhSi), 128.7 (C_m, PhN), 130.6 (C_p, PhSi), 132.9 (C_i, PhSi), 133.9 (C_o, PhSi), 148.6 (C_i, PhN). ^{29}Si NMR spectrum, δ, ppm: –3.7. Found, %: C 64.53; H 6.17; N 5.29. $C_{14}H_{16}ClNSi$. Calculated, %: C 64.22; H 6.16; N 5.35.

***N*-[Chloro(methyl)(vinyl)silyl]aniline (IV).** Yield 15.4%, bp 78–82°C (2 mmHg). ^{1}H NMR spectrum, δ, ppm: 0.65 s (3H, CH_3), 3.9 br.s (1H, NH), 6.16 t (1H, HC-Si), 6.6–6.7 d.d (2H, CH_2=), 6.8–7.2 m (5H, PhN). ^{13}C NMR spectrum, δ_C, ppm: 0.6 ($\underline{C}H_3Si$), 117 (C_p), 119.5 (C_o), 129 (C_m), 133.3 (H$\underline{C}$-Si), 136.6 ($\underline{C}H_2$=), 143.9 (C_i). ^{29}Si NMR spectrum, δ_{Si}, ppm: 8.7. Found, %: C 54.46; H 6.51; N 6.96. $C_9H_{12}ClNSi$. Calculated, %: C 54.67; H 6.12; N 7.08.

***N*-[Chloro(methyl)(vinyl)silyl]-*N*-methylaniline (V).** Yield 72%, bp 63°C (2 mmHg). ^{1}H NMR spectrum, δ, ppm: 0.57 s (3H, CH_3Si), 3 s (3H, CH_3N), 5.9–6.2 m (3H, H_2C=CH), 7–7.2 m (5H, PhN). ^{13}C NMR spectrum, δ_C, ppm: 1.8 ($\underline{C}H_3Si$), 36.7 ($\underline{C}H_3N$), 118.2 (C_p), 122.1 (C_o), 128.9 (C_m), 134.2 (H$\underline{C}$-Si), 136.2 ($\underline{C}H_2$=), 148.9

(C_i). Found, %: C 56.43; H 6.71; N 6.59. $C_{10}H_{14}ClNSi$. Calculated, %: C 56.72; H 6.66; N 6.61.

***N*-[Chloro(diphenyl)silyl]aniline (VI).** Yield: 64%, bp 170–188°C (2 mmHg), mp 80.7°C. ^{1}H NMR spectrum, δ, ppm: 4.1 s (1H, NH), 6.7–7.0 m (5H, PhN), 7.3–7.7 m (10H, PhSi). ^{13}C NMR spectrum, δ_C, ppm: 117.5 (C_o, PhN), 119.5 (C_p, PhN), 128.1 (C_m, PhSi), 128.9 (C_m, PhN), 130.9 (C_p, PhSi), 132.1 (C_i, PhSi), 134.4 (C_o, PhSi), 144 (C_i, PhN). ^{29}Si NMR spectrum, δ, ppm: 0.7. Found, %: C 70.05; H 5.37; N 4.37. $C_{18}H_{16}ClNSi$. Calculated, %: C 69.77; H 5.20; N 4.52.

***N*-[Chloro(diphenyl)silyl]-*N*-methylaniline (VII).** Yield: 68%, bp 175°C (2–3 mmHg). ^{1}H NMR spectrum, δ, ppm: 3.0 s (3H, CH_3N), 6.8–7.0 m (5H, PhN), 7.3–7.6 m (10H, PhSi). ^{13}C NMR spectrum, δ_C, ppm: 37.4 ($\underline{C}H_3$), 120.1 (C_p, PhN), 121.7 (C_o, PhN), 128.1 (C_m, PhSi), 128.5 (C_m, PhN), 130.8 (C_p, PhSi), 133 (C_i, PhSi), 135.1 (C_o, PhSi), 148.6 (C_i, PhN). ^{29}Si NMR spectrum, δ, ppm: –4.4. Found, %: C 70.27; H 5.68; N 4.40. $C_{19}H_{18}ClNSi$. Calculated, %: C 70.46; H 5.60; N 4.32.

***N*-[Chloro(chloromethyl)(methyl)silyl]aniline (VIII).** Yield: 34.5%. bp 64–66°C (1 mmHg). ^{1}H NMR spectrum, δ, ppm: 0.4 s (3H, CH_3), 3.0–3.2 m (2H, CH_2Cl), 4.2 br.s (1H, NH), 6.9–7.3 m (5H, PhN). ^{13}C NMR spectrum, δ_C, ppm: 0.4 ($\underline{C}H_3$), 27 ($\underline{C}H_2Cl$), 115 (C_o), 119 (C_p), 125.6 (C_i), 129.2 (C_m). -^{29}Si NMR spectrum, δ, ppm: 5.3. Found, %: C 43.08; H 5.01; N 6.18. $C_8H_{11}Cl_2NSi$. Calculated, %: C 43.64; H 5.04; N 6.36.

***N*-[Chloro(chloromethyl)(methyl)silyl]-*N*-methylaniline (IX).** Yield 63.8%, bp 74–77°C (2 mmHg). ^{1}H NMR spectrum, δ, ppm: 0.61 s (3H, CH_3Si), 2.9–3.1 d.d (2H, CH_2Cl), 2.9 s (3H, CH_3N), 7.0–7.3 m (5H, PhN). ^{13}C NMR spectrum, δ_C, ppm: –0.8 ($\underline{C}H_3Si$), 27 ($\underline{C}H_2Cl$), 29.3 ($\underline{C}H_3N$), 112.5 (C_p), 123.5 (C_o), 129.2 (C_m), 148.3 (C_i). ^{29}Si NMR spectrum, δ, ppm: -16.0. Found, %: C 46.56; H 5.71; N 6.21. $C_9H_{13}Cl_2NSi$. Calculated, %: C 46.16; H 5.59; N 5.98.

***N*-[Chloro(3-chloropropyl)(methyl)silyl]aniline (X).** Yield 34%, bp 135–140°C (2 mmHg). ^{1}H NMR spectrum, δ, ppm: 0.6 s (3H, CH_3), 1.1 br.t (2H, CH_2Si), 1.9 m (2H, $–CH_2–$), 3.5 m (2H, CH_2Cl), 3.8 br.s (1H, NH), 6.7–7.2 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 2 ($\underline{C}H_3$), 15.3 ($\underline{C}H_2Si$), 24.3 ($–\underline{C}H_2–$), 47 ($\underline{C}H_2Cl$), 117.5 (C_o), 120 (C_p), 129.4 (C_m), 144.2 (C_i). ^{29}Si NMR spectrum, δ, ppm: –17.5. Found, %: C 48.21; H 6.44; N 5.72. $C_{10}H_{15}Cl_2NSi$. Calculated, %: C 48.39; H 6.09; N 5.64.

***N*-[Chloro(3-chloropropyl)(methyl)silyl]-*N*-methylaniline (XI).** Yield 53%, bp°C (1 mmHg). ^{1}H NMR spectrum, δ, ppm: 0.5 s (3H, CH_3Si), 1.1 m (2H, CH_2Si), 1.8 m (2H, $–CH_2–$), 3 s (3H, CH_3N), 3.4 m (2H, CH_2Cl), 7.0–7.2 m (5H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 2 ($\underline{C}H_3Si$), 15.8 ($\underline{C}H_2Si$), 26.5 ($–\underline{C}H_2–$), 26.9 ($\underline{C}H_3N$), 47 ($\underline{C}H_2Cl$), 112.4 (C_p), 123.0 (C_o), 129.0 (C_m), 149.0 (C_i). ^{29}Si NMR spectrum, δ, ppm: -14.4. Found, %: C 49.92; H 6.08; N 5.09. $C_{11}H_{17}Cl_2NSi$. Calculated, %: C 50.38; H 6.53; N 5.34.

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