

***N,N*-Bis[ethoxy(methyl)silylmethyl]methyamines MeN[CH₂SiMe_{*m*}(OEt)_{3–*m*}]₂ (*m* = 0–2). Synthesis and Reactions with Phenol**

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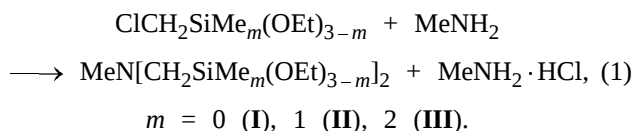
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Abstract—Previously unknown *N,N*-bis[ethoxy(methyl)silylmethyl]amines MeN[CH₂SiMe_{*m*}(OEt)_{3–*m*}]₂ (*m* = 0–2) were synthesized. According to UV spectral data, only MeN[CH₂SiMe₂(OEt)₂]₂ form hydrogen bond with phenol in a heptane solution. The amines with *m* = 0 and 1 fail to forms hydrogen bond with phenol [under the same conditions, *N*-(triethoxysilylmethyl)dimethylamine Me₂NCH₂Si(OEt)₃ forms a strong hydrogen bond with phenol]. All the amines (*m* = 0–2) enter transesterification with phenol to give compounds of the general formula MeN[CH₂SiMe_{*m*}(OPh)_{*n*}(OEt)_{3–*m–n*}]₂ (*m* = 0–2, *n* = 1–3). Refluxing of *N,N*-bis[ethoxy(methyl)silylmethyl]amines with excess phenol results in cleavage of the Si–C bond by phenol, providing phenoxy-silanes Me_{*m*}Si(OPh)_{4–*m*} (*m* = 0–2) and trimethylamine.

The electron-donor ability of the piperidine nitrogen atom in organosilicone amines (CH₂)₅NCH₂·SiMe_{*n*}(OEt)_{3–*n*} (*n* = 2, 3) is reduced by *n*,σ⁺ interaction in the N–CH₂–Si fragment [1, 2]. However, in the R_{*m*}N(CH₂SiMe₃)_{3–*m*} series, no such effect with increasing number of CH₂SiMe₃ groups at the nitrogen atom was observed: The ionization potential of the lone electron pair of the nitrogen atom (7.93, 7.82, and 7.66 at *m* = 2, 1, and 0, respectively [3]) decrease as the electron-donor ability of the latter increases with the increasing number of donor substituents. Compounds containing several CH₂SiMe_{*m*}(OAlk)_{3–*m*} groups at the nitrogen atom have not been studied from this viewpoint.

To assess the effect of CH₂SiMe_{*m*}(OEt)_{3–*m*} groups on nitrogen basicity, we synthesized previously unknown *N,N*-bis[ethoxy(methyl)silylmethyl]methyamines MeN[CH₂SiMe_{*m*}(OEt)_{3–*m*}]₂ by scheme (1):



The physicochemical characteristics of these compounds are listed in Table 1. Their structure was proved by ¹H NMR and IR spectral data (Tables 2 and 3).

Table 1. Physicochemical characteristics of amines MeN[CH₂SiMe_{*m*}(OEt)_{3–*m*}]₂ (*m* = 0–2) and their reaction products with phenol MeN[CH₂SiMe_{*m*}(OPh)_{*n*}(OEt)_{3–*m–n*}]₂ (*m* = 0–2, *n* = 1–3)

Comp. no.	<i>m</i>	<i>n</i>	Yield, %	bp, °C (<i>p</i> , mm)	<i>n</i> _D ²⁰	Found, %				Formula	Calculated, %			
						C	H	N	Si		C	H	N	Si
I	0	0	55	124–125 (1)	1.4174	46.65	9.63	3.69	13.75	C ₁₅ H ₃₇ NO ₆ Si ₂	46.96	9.72	3.65	14.64
II	1	0	43	105–106 (1)	1.4098	48.01	9.98	4.35	16.92	C ₁₃ H ₃₃ NO ₄ Si ₂	48.25	10.28	4.32	17.36
III	2	0	52	220–224	1.4054	50.28	11.65	4.98	20.98	C ₁₁ H ₂₉ NO ₂ Si ₂	50.13	11.09	5.32	21.32
IV	0	1	40	200–202 (1)	1.4998	57.78	7.98	3.13	11.70	C ₂₃ H ₃₇ NO ₆ Si ₂	57.58	7.77	2.92	11.71
V	0	2	35	240–242 (1)	1.5225	64.92	6.91	2.75	9.58	C ₃₁ H ₃₇ NO ₆ Si ₂	64.66	6.47	2.43	9.76
VI	0	3	38	295–297 (1)	1.5638	70.15	5.98	2.05	8.38	C ₃₉ H ₃₇ NO ₆ Si ₂	69.72	5.55	2.08	8.36
VII	1	2	41	238–240 (1)	1.5448	67.81	6.93	2.61	11.12	C ₂₉ H ₃₃ NO ₄ Si ₂	67.53	6.45	2.72	10.89
VIII	2	1	35	180–183 (2)	1.5145	63.93	8.58	4.01	15.95	C ₁₉ H ₂₉ NO ₂ Si ₂	63.46	8.13	3.89	15.62

Table 2. Group frequencies (cm^{-1}) in the IR spectra of amines $\text{MeN}[\text{CH}_2\text{SiMe}_m(\text{OPh})_n(\text{OEt})_{3-m-n}]_2$ (**I–VIII**) and model compounds $\text{Me}_m\text{Si}(\text{OPh})_n$ (**IX–XI**)

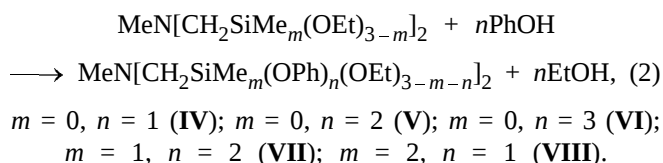
Comp. no.	<i>m</i>	<i>n</i>	CH_3N	SiOC_{Alk}	SiOC_{Ph}	C_6H_5	$\text{CH}_2\text{Si}; \text{CH}_3\text{Si}$
I	0	0	2745, 2755	815, 955, 1070, 1100	—	—	750
II	1	0	2745, 2760	795, 955, 1070, 1100	—	—	750, 845, 1255
III	2	0	2740, 2755	795, 955, 1070, 1100	—	—	750, 845, 1250
IV	0	1	2760	800, 950, 1080, 1100	950, 1240	690, 760, 1495, 1600, 3035, 3055	755
V	0	2	2760	805, 950, 1080, 1100	950, 1240	690, 760, 1495, 1600, 3035, 3055	755
VI	0	3	2760	—	950, 1220	690, 760, 1500, 1600, 3035, 3060	760
VII	1	2	2740, 2755	—	915, 1250	695, 750, 1490, 1595, 3035, 3055	755, 800, 1270
VIII	2	1	2760	—	925, 1240	695, 750, 1490, 1600, 3035, 3055	750, 798, 1270
IX	1	3	—	—	950, 1240	690, 755, 1490, 1595, 3040, 3055	800, 1280
X	2	2	—	—	925, 1240	690, 755, 1490, 1595, 3040, 3060	805, 1270
XI	3	1	—	—	915, 1250	695, 755, 1490, 1595, 3035, 3055	755, 845, 1270

Table 3. ^1H NMR spectra (δ , ppm) of amines $\text{MeN}[\text{CH}_2\text{SiMe}_m(\text{OPh})_n(\text{OEt})_{3-m-n}]_2$ (**I–VIII**) and model compounds $\text{Me}_m\text{Si}(\text{OPh})_n$ (**IX–XI**)^a

Comp. no.	CH_3N	NCH_2Si	OCH_2CH_3	OC_6H_5	CH_3Si
I	2.30 s	2.00 s	1.20 t, 3.83 q	—	—
II	2.31 s	2.01 s	1.20 t, 3.80 q	—	0.18 s
III	2.26 s	1.96 s	1.17 t, 3.68 q	—	0.14 s
IV	2.43 s	2.27 s	1.25 t, 3.94 q	7.04 m, 7.23 m	—
V	2.43 s	2.37 s	1.20 t, 3.90 q	6.96 m, 7.21 m	—
VI	2.44 s	2.36 s	—	6.94 m, 7.15 m	—
VII	2.38 s	2.01 s	—	6.97 m, 7.13 m	0.39 s
VIII	2.36 s	2.18 s	—	6.98 m, 7.22 m	0.38 s
IX	—	—	—	6.83 m, 7.19 m	0.25 s
X	—	—	—	6.98 m, 7.24 m	0.37 s
XI	—	—	—	7.02 m, 7.16 m	0.48 s

^a Solvent CDCl_3 , internal standard TMS (for **I** and **IV–VI**) and cyclohexane (for **II**, **III**, **VII**, and **VIII**).

Compounds **I–III** react with phenol in a heptane solution at room temperature. By vacuum distillation we also isolated viscous yellowish specifically smelling products **IV–VIII**.



Their physicochemical characteristics and elemental analyses are listed in Table 1. The IR spectra of compounds **IV–VIII** contain CH_3N stretching absorption bands [4] (Table 2). The $\text{C}_{\text{Alk}}\text{O-Si}$ absorption bands [5] are present in the spectra of **IV**, **V** and absent in the spectra of **VI–VIII**. In the spectra of

IV–VIII, there are bands of stretching and bending vibrations characteristic of a monosubstituted benzene ring, as well as a strong broad band at 1240 cm^{-1} [$\nu(\text{C}_{\text{Ar}}\text{O})$] [5].

The ^1H NMR spectra of **IV–VIII** compared with those of parent **I–III** show new signals at 6.8–7.2 ppm, characteristic of benzene ring protons (Table 3). The signals of ethoxy groups are preserved only in the ^1H NMR spectra of **IV** and **V**.

Compounds **I–III** have a weak UV absorption maximum below 210 nm, like trialkylamines [6]. The UV spectra of **IV–VIII** show two maxima at 260–280 and 211 nm, assignable to $\pi\text{--}\pi^*$ transitions in the benzene ring (Table 4). The λ_1 and λ_2 values in the UV spectra of the isolated products only slightly differ from those for model compounds $\text{Me}_m\text{Si}\cdot$

(OPh)_{4-m} (**IX–XI**) ($m = 1–3$). The intensities of the λ_1 and λ_2 bands in the spectra of **IV–VIII** grow regularly with increasing number of phenoxy groups. Thus, the IR, ¹H NMR, and UV data give conclusive evidence showing that *N,N*-bis[ethoxy(methyl)silylmethyl]methylamines MeN[CH₂SiMe_m(OEt)_{3-m}]₂ enter transesterification with phenol.

We discovered the transesterification of **I–III** with phenol when studied their basicity by UV spectroscopy in a heptane solution. Compounds containing heteroatoms with lone electron pairs (N, O, S, etc.) enter with phenol hydrogen bonding which have been fairly well studied by IR (in CCl₄) and UV (in hydrocarbon solvents) spectroscopy [7–11]. Earlier by the latter method we determined the electron-donor ability of the nitrogen atom in compounds like (CH₂)₅NCH₂·SiMe_n(OEt)_{3-n} ($n = 2, 3$) [1, 2], which formed hydrogen bond with phenol but did not react with it. The UV spectrum of a solution of phenol and excess compound **III** ($m = 2$) in heptane shows a bathochromically shifted $\pi \rightarrow \pi^*$ band of phenol, implying hydrogen bonding with nitrogen (Fig. 1, spectrum 2). For **III** this shift ($\Delta\nu$) is larger than for Me₂NCH₂SiMe₂·(OEt) (540 and 450 cm⁻¹, respectively). However, already after 10 min the $\pi \rightarrow \pi^*$ bands of free and H-bonded phenol get weaker. On mixing of phenol solutions with excess compounds **I, II** no H-bonded complex is observed at all: The long-wave band at 260–290 nm gets half that of the free phenol band and shifts hypsochromically, after which it no longer changes (Fig. 1, spectrum 3). It should be noted that under the same conditions dimethyl(triethoxysilylmethyl)amine Me₂NCH₂Si(OEt)₃ forms a stable H-bonded complex with phenol ($\Delta\nu$ 490 cm⁻¹), and its UV spectrum is invariable even after 3 h. As follows from our results, compounds **I–III** rapidly react with phenol at room temperature in heptane. Therefore, their basicity cannot be estimated by UV spectroscopy {unlike ethoxypiperidinomethylsilanes C₅H₁₀NCH₂SiMe_n(OEt)_{3-n} [1, 2] and triethoxydimethylaminomethylsilane Me₂NCH₂Si(OEt)₃}.

Transesterification of silanes containing a SiOC group with alcohols and phenols is one of the most thoroughly studied reactions of organosilicon compounds (see [12] and references therein). The reaction is usually performed with heating in the presence of catalysts. The transesterification of *N,N*-bis[ethoxy(methyl)silylmethyl]amines **I–III** with phenol occurs already at room temperature, and it may be driven by autocatalysis. However, ethoxy(dialkylaminomethyl)silanes R₂NCH₂SiMe_n(OEt)_{3-n} (R₂ = Me₂, C₅H₁₀; $n = 0–2$) whose nitrogen atom is also very basic (and, consequently, they are also able to autocatalysis) do

Table 4. UV spectra [λ , nm (ϵ , l mol⁻¹ cm⁻¹)] of amines MeN[CH₂SiMe_m(OPh)_n(OEt)_{3-m-n}]₂ (**IV–VIII**) and model compounds Me_mSi(OPh)_n (**IX–XI**)

Comp. no.	λ_1 (ϵ_1)	λ_2 (ϵ_2)
IV	265 (1900), 268 (1900), 271 (2800), 278 (2500)	211 (9300)
V	265 (4500), 268 (4000), 271 (6000), 278 (5100)	211 (20900)
VI	265 (7300), 268 (6700), 271 (9100), 278 (8000)	211 (30500)
VII	265 (4350), 268 (4400), 271 (6400), 278 (4650)	211 (24500)
VIII	265 (2500), 268 (2600), 271 (3400), 278 (3100)	211 (16300)
IX	267 (1200), 271 (1590), 277 (1270)	211 (8400)
X	262 (1650), 268 (2250), 275 (1650)	211 (14800)
XI	261 (2250), 267 (3050), 274 (2550)	211 (25300)

not react with phenol under the same conditions. This fact suggests that autocatalysis is not a single driving force of the reaction in question. The unusual facility of transesterification of *N,N*-bis[ethoxy(methyl)silylmethyl]methylamines with phenol can be explained in terms of electronic effects of CH₂SiMe_m(OEt)_{3-m} groups on the basicity of the nitrogen atom. The electron-donor ability of *N*-[ethoxy(methyl)silylmethyl]dimethylamines Me₂NCH₂SiMe_m(OEt)_{3-m} in solutions is determined by a combination of two effects: The +I effect increases the electron density on the

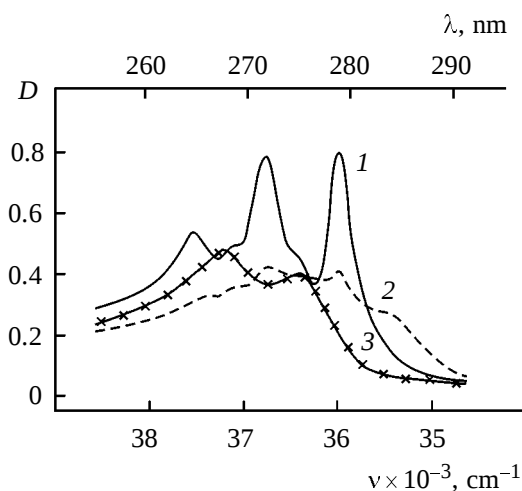


Fig. 1. UV spectra of heptane solutions of (1) phenol, (2) phenol with MeN[CH₂SiMe₂(OEt)]₂ (c_b 3.2×10^{-2} M), and (3) phenol with MeN[CH₂Si(OEt)₃]₂ (c_b 2.0×10^{-2} M).

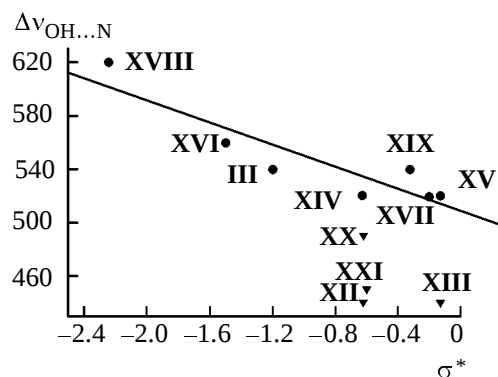


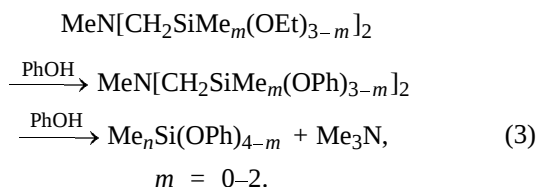
Fig. 2. $\Delta\nu_{\text{OH}\cdots\text{N}}$ (cm^{-1}) vs. Taft inductive σ^* constants for the $(\text{CH}_2)_m\text{SiX}_3$ groups in $\text{MeN}[(\text{CH}_2)_m\text{SiMe}_2(\text{OEt})]_2$ (**III**), $(\text{CH}_2)_5\text{NCH}_2\text{Si}(\text{OEt})_3$ (**XII**), $(\text{CH}_2)_5\text{NCH}_2\text{MeSi}(\text{OEt})_2$ (**XIII**), $(\text{CH}_2)_5\text{N}(\text{CH}_2)_3\text{Si}(\text{OEt})_3$ (**XIV**), $(\text{CH}_2)_5\text{N}(\text{CH}_2)_3\text{MeSi}(\text{OEt})_2$ (**XV**), $(\text{CH}_2)_5\text{NCH}_2\text{MeSi}(\text{OCH}_2\text{CH}_2)_2\text{NMe}$ (**XVI**), $(\text{CH}_2)_5\text{N}(\text{CH}_2)_3\text{MeSi}(\text{OCH}_2\text{CH}_2)_2\text{NMe}$ (**XVII**), $(\text{CH}_2)_5\text{NCH}_2\text{Si}(\text{OCH}_2\text{CH}_2)_3\text{N}$ (**XVIII**), $(\text{CH}_2)_5\text{N}(\text{CH}_2)_3\text{Si}(\text{OCH}_2\text{CH}_2)_3\text{N}$ (**XIX**), $\text{Me}_2\text{NCH}_2\text{Si}(\text{OEt})_3$ (**XX**), and $\text{Me}_2\text{NCH}_2\text{SiMe}_2(\text{OEt})$ (**XXI**). The σ^* constants were taken from [10].

nitrogen atom, while the n, σ^* -conjugation effect acts in the opposite direction [10]. Compounds $\text{R}_2\text{NCH}_2\cdot\text{SiMe}_m(\text{OEt})_{3-m}$, where the latter effect is operative, do not fit the linear dependence $\Delta\nu = f(\sigma^*_{\text{CH}_2\text{SiX}_3})$ [12]. Unlike $\text{Me}_2\text{NCH}_2\text{SiMe}_2(\text{OEt})$ (**XXI**), the $\Delta\nu$ value for compound **III** does not deviate from the $\Delta\nu = f(\sigma^*_{\text{CH}_2\text{SiX}_3})$ dependence (Fig. 2). This fact suggests prevailing inductive effect of the two CH_2SiX_3 groups on the nitrogen atom (apparently, the n, σ^* effect in **I–III** is almost inoperative). As a result, the partial positive charge on the silicon atom in these compounds increases. Apparently, this favors faster transesterification of **I–III** with phenol, as compared with $\text{Me}_2\text{NCH}_2\text{SiMe}_m(\text{OEt})_{3-m}$, since here it occurs as $\text{S}_{\text{N}}2$ substitution at silicon [13, 14].

For better yields of **IV–VIII**, a mixture of **I–III** with excess phenol was heated under reflux (without solvent). The products that were isolated contained no N–CH_3 and $\text{N–CH}_2\text{–Si}$ groups (by ^1H NMR and IR spectroscopy). The ^1H NMR, IR, and UV spectra of the reaction products of compounds **II**, **III** with phenol coincide with the spectra of compounds **X**, **XI**, while the ^1H NMR spectrum of the reaction product of compound **I** coincides with that of tetraphenoxy-silane.

The resulting data suggest that the reaction initially involves substitution of ethoxy groups by phenoxy to afford compounds **VI–VIII**, followed by cleavage of the Si–C bond in the latter by phenol [scheme (3)].

Triethylamine was isolated as hydrochloride.



Heterolytic Si–C bond cleavage is commonly encountered in the silicon chemistry [15–25]. Lewis acids catalyze the Si–C bond cleavage with various electrophiles (see [15] and references therein). Bases, too, catalyze Si–C bond cleavage [20–23]. The Si–C bond cleavage in tetraalkylsilanes occurs under rigid conditions under the action of concentrated sulfuric acid [24]. However, this reaction is much facilitated by functional substitution in the alkyl group [25]. As shown above, the partial positive charge on the silicon atom in the compounds in study is increased compared with $\text{Me}_2\text{NCH}_2\text{SiMe}_m(\text{OEt})_{3-m}$. The Si–C bond cleavage, being $\text{S}_{\text{N}}2$ substitution at silicon [25, 26], is facilitated by increased positive charge on the latter. Moreover, the Si–O bond formation is favored thermodynamically, since Si–O is a stronger bond than Si–C (E_{dis} 530 and 360 kJ/mol, respectively [27]).

EXPERIMENTAL

The IR spectra were obtained on a Specord IR-75 instrument in thin films. The UV spectra of heptane solutions of the compounds in study and their complexes with phenol were obtained on a Specord UV-Vis spectrophotometer at 200–450 nm. The concentration of phenol was 4.5×10^{-4} M. The ^1H NMR spectra were recorded on a JEOL Q-90 spectrometer. Heptane and phenol were purified by standard procedures [5].

Compounds I–III. A mixture of 0.2 mol of $\text{ClCH}_2\text{SiMe}_m(\text{OEt})_{3-m}$ ($m = 0-2$) and 0.5 mol of methylamine was kept in a sealed ampule for 48 h. The ampule was then unsealed, excess methylamine was evaporated, and the precipitate was filtered off and washed with dry ether. The ether solution was combined with the filtrate and distilled.

Compounds IV–VIII. Compound **I–III**, 0.01 mol, was mixed with 10 ml of dry heptane and equimolar amount of phenol, and the mixture was kept at room temperature for 48 h. The ethanol that formed and heptane were removed by distillation, and the residue was distilled in a vacuum to isolate compounds **IV–VIII**. Further are given **I–III**:phenol ratio (product no.): 1:2 (**IV**); 1:4 (**V**); 1:6 (**VI**); 1:4 (**VII**); and 1:2 (**VIII**).

Si–C bond cleavage. Compound **I–III**, 0.01 mol, was heated with excess phenol (silane:phenol ratio 1:9 (**I**), 1:7 (**II**), and 1:5 (**III**)) in a flask equipped with a reflux condenser attached to a trap

containing a benzene solution of hydrogen chloride. Heating was continued for 1.5 h until triethylamine no longer evolved (the presence of trimethylamine in the vapor was determined using a wet universal indicator paper placed at the top of the reflux condenser). The residue was distilled in a vacuum to isolate the following products (yield, %): $(\text{PhO})_4\text{Si}$ (89) for compound **I**; $\text{MeSi}(\text{OPh})_3$ (92) for compound **II**; and $\text{Me}_2\text{Si}(\text{OPh})_2$ (85) for compound **III**. Their melting points and n_D^{20} values were consistent with those reported in [28]. Trimethylamine hydrochloride was isolated from benzene, mp 276–277°C.

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