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1-Chloro-1,3-dimethyl-1,3-diorganyl-3-(trimethylsilylamino)disiloxanes

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The main products of the reaction of 1,3-dichloro-1,3-dimethyl-1,3-diorganyldisiloxanes with hexamethyldisilazane (molar ratio 1 : 1, 20 °C) are the earlier unknown linear 1-chloro-1,3-dimethyl-1,3-diorganyl-3-(trimethylsilylamino)disiloxanes of the general formula $ClMeRSiOSiMeRNHSiMe_3$ (**1**) (R = $ClCH_2$ (**a**), Me (**b**), Vin (**c**), Ph (**d**)) obtained in up to 53—72% yield and 1,3-dimethyl-1,3-diorganyl-1,3-bis(trimethylsilylamino)disiloxanes of the general formula $(Me_3SiNH)MeRSiOSiMeR(NHSiMe_3)$ (**2a**—**d**) (7—12% yield). Thermolysis of compounds **1a**—**d** leads to the formation of cyclic silazaoxanes. A plausible scheme for their formation was suggested.

Key words: hexamethyldisilazane, 1,3-dichloro-1,3-dimethyl-1,3-diorganyldisiloxanes, 1-chloro-1,3-dimethyl-1,3-diorganyl-3-(trimethylsilylamino)disiloxanes, 1,3-bis(trimethylsilylamino)-1,3-dimethyl-1,3-diorganyldisiloxanes, cyclosilazaoxanes.

Organylsilazaoxanes are used as starting materials for the production of silicone oils and rubbers,[1] surface modifiers of glass, polymers, and biopolymers.[2] Polymers based on them are used as heat-resistant adhesives and components of compositions for the preparation of chemically resistant ceramic coatings for aviation and space technology.[3]

In continuation of our studies[4] in the area of synthesis and examination of reactivity of 1,3-dichloro-1,3-dimethyl-1,3-diorganyldisiloxanes, in the present work we found that, in contrast to the statements made earlier,[5] these compound even at room temperature react with hexamethyldisilazane (molar ratio 1 : 1, 120—240 h) with a predominant formation of earlier unknown 1-chloro-1,3-dimethyl-1,3-diorganyl-3-(trimethylsilylamino)disiloxanes **1a**—**d** (up to 72%) together with 1,3-dimethyl-1,3-diorganyl-1,3-bis(trimethylsilylamino)disiloxanes **2a**—**d** (7—12%) (Scheme 1).

A removal of chlorotrimethylsilane from the reaction mixture by evacuation (40—50 Torr) did not considerably affect the ratio of the reaction products, while its distillation off at normal pressure or the distillation of the reaction products *in vacuo* led to the formation of cyclosilazaoxanes, the yield of which became almost quantitative at a prolonged (16—28 h) reflux. In the presence of nitrogen bases (DMF, pyridine) and DMSO, this process requires 2—5 h. A catalytic effect of nitrogen bases on cyclization reactions was reported earlier in the work.[5]

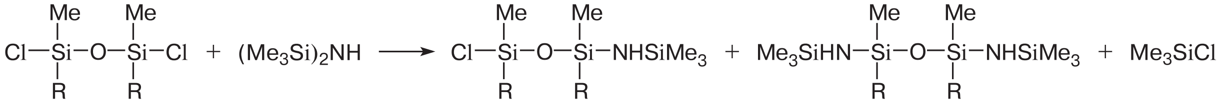
Based on gas chromatography-mass spectrometery (GC-MS) monitoring and using reaction of 1,3-dichloro-1,3-dimethyl-1,3-divinyldisiloxane as a model, we suggested the following pathway for the process (Scheme 2).

It should be noted that in the initial stage, cyclosilazaoxanes **4c** and **5c** are formed in the ratio 2 : 1, however, as the reaction proceeds further only the content of structure **4c** is increased, while the yield of compound **5c** does not exceed 5%.

A prolonged contact (20—25 days) of 1,3-dichlorotetraorganyldisiloxanes with hexamethyldisilazane (molar ratio 1 : 2) at room temperature did not lead to considerable increase in the yield of compounds **2a**—**d**.

The GC-MS data showed (for compound **1c** as an example) that the reflux of the reaction mixture under conditions of incomplete proceeding of the reaction (mo-

Scheme 1



$$Cl-\underset{R}{\overset{Me}{Si}}-O-\underset{R}{\overset{Me}{Si}}-Cl + (Me_3Si)_2NH \longrightarrow Cl-\underset{R}{\overset{Me}{Si}}-O-\underset{R}{\overset{Me}{Si}}-NHSiMe_3 + Me_3SiHN-\underset{R}{\overset{Me}{Si}}-O-\underset{R}{\overset{Me}{Si}}-NHSiMe_3 + Me_3SiCl$$

1a—d **2a—d**

R = $ClCH_2$ (**a**), Me (**b**), Vin (**c**), Ph (**d**)

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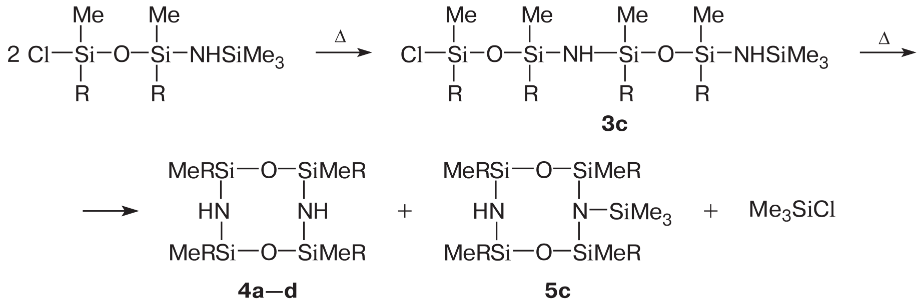
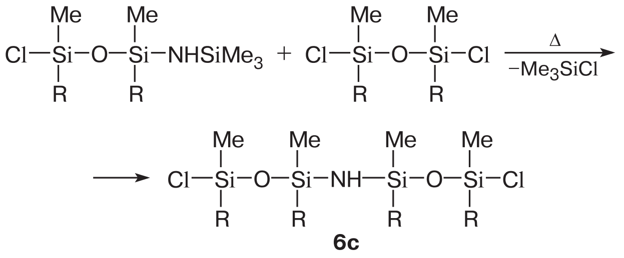
Scheme 2



R = ClCH$_2$ (**a**), Me (**b**), Vin (**c**), Ph (**d**)

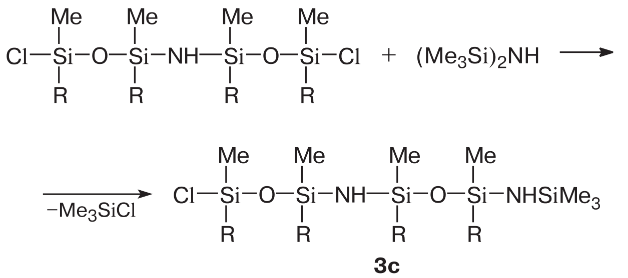
lar ratio 1 : 1), *i.e.*, in the presence of the starting 1,3-dichloro-1,3-dimethyl-1,3-divinyldisiloxane and hexamethyldisilazane, the formation of symmetrical linear α,ω-dichloro(vinyl)silazaoxane (**6c**) is observed (Scheme 3), as well as the elongation of the silazaoxane chain (Scheme 4).

Scheme 3



R = Vin (**c**)

Scheme 4



R = Vin (**c**)

Compound **3c** can be also formed by the reaction of two molecules of **1c** (see Scheme 2). Attempted isolation of these compounds leads to their further conversion with the formation of compound **4c**.

Compounds **1a—d**, **2a—d**, and **4a—d** were characterized by ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopy and gas chromatography-mass spectrometry, while compounds **3c, 5c, 6c** only by GC-MS. Physicochemical constants of octamethylcyclotetrasila-4,8-diaza-2,6-dioxane and 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasila-4,8-diaza-2,6-dioxane agree with the corresponding constants for ammonolysis products of 1,3-dichloro-1,1,3,3-tetramethyldisiloxane[6,7] and 1,3-dichloro-1,3-dimethyl-1,3-divinyldisiloxane[8] obtained by the reaction with liquid ammonia at –71—(–20) °C.

The fragmentation of 1-chloro-1,3-dimethyl-1,3-diorganyl-3-(trimethylsilylamino)disiloxanes, 1,3-dimethyl-1,3-diorganyl-1,3-bis(trimethylsilylamino)disiloxanes and cyclosilazaoxanes follows the same directions which was found for linear and cyclic silazane structures.[9—11]

Experimental

^{1}H, ^{13}C, and ^{29}Si NMR spectra were recorded on a Bruker DPX 400 spectrometer (400, 100, and 161.98 MHz, respectively) in CDCl$_3$, using SiMe$_4$ as an internal standard. Reaction progress was monitored by ^{1}H NMR spectroscopy.

Mass spectra were recorded on a Shimadzu GCMS-QP5050A spectrometer, injector temperature 200—250 °C, carrier gas helium, detector temperature 200 °C, a quadrupole mass-analyzer, EI ionization (70 eV). Chromatographic separation of compounds under study was carried out on a SPB-5 capillary column (60 m×0.25 mm×0.25 μm), carrier gas helium, the flow rate 0.7 mL min^{-1}; injector temperature 230 °C, temperature of the source of ions 200 °C, pressure 280 kPa, programmed regime from 60 to 250 °C at the rate of 10 deg min^{-1}.

Chromatographic analysis of reaction products was carried out by GLC on an LKhM-8M chromatograph with a 1000×0.5-mm column filled with Chromaton-N-AW covered with the SE-30 phase (5%), carrier gas He. Injector temperature 260 °C, katharometer temperature 240 °C. The column temperature was linearly programmed from 30 to 250 °C at the rate of 20 deg min^{-1}.

1,3-Dichloro-1,3-dimethyl-1,3-diorganyldisiloxanes were synthesized according to the method described earlier,[4] hexamethyldisilazane, an industrial product, was purified by distillation. Their physical constants corresponded to the literature data.

Reaction of 1,3-dichloro-1,3-dimethyl-1,3-divinyldisiloxane with hexamethyldisilazane. A mixture of 1,3-dichloro-1,3-dimethyl-1,3-divinyldisiloxane (22.6 g, 0.1 mol) with hexamethyldisilazane (16.1 g, 0.1 mol) was stirred for 140 h at ~20 °C. The reaction was carried out in a three-neck flask with a reflux condenser, argon was used as a reaction gas medium. Chlorotrimethylsilane (8.9 g, 82%) was distilled off at reduced pressure (b.p. 57 °C for distillation at normal pressure). The conversion on hexamethyldisilazane was more than 95%. The residue was analyzed by ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopy and chromato-mass spectrometry.

1-Chloro-1,3-dimethyl-3-trimethylsilylamino-1,3-divinyldisiloxane, ClMeVinSiOSiMeVinNHSiMe$_3$ (1c). The yield was 72%. ^{1}H NMR, δ: 0.07 (9 H, NSiMe$_3$); 0.15, 0.20 (3 H, CH$_3$SiN); 0.55, 0.59 (3 H, CH$_3$SiCl); 0.65 (NH); 5.87—6.03 (m, 6 H, CH=CH$_2$). ^{13}C NMR, δ: 1.14 (NSiMe$_3$), 2.73 (CH$_3$SiN), 3.19 (CH$_3$SiCl), 135.11—136.27 (CH=CH$_2$). ^{29}Si NMR, δ: −11.44 (OSiN), −7.57 (SiMeVinCl), 3.53 (NSiMe$_3$). MS, *m/z* (I_{rel} (%)): 264 [M − Me]$^+$ (100), 252 [M − Vin]$^+$ (6), 244 [M − Cl]$^+$ (20), 228 [M − Me − HCl]$^+$ (17), 210 (8), 156 [M − Me − ClSiMe$_3$]$^+$ (7), 144 (19), 132 (20), 97 (13), 85 (10), 73 Me$_3$Si$^+$ (7).

1,3-Dimethyl-1,3-bis(trimethylsilylamino)-1,3-divinyldisiloxane, (Me$_3$SiNH)MeVinSiOSiMeVin(NHSiMe$_3$) (2c). The yield was 7%. ^{1}H NMR, δ: 0.08 (18 H, NSiMe$_3$); 0.13, 0.18 (6 H, CH$_3$SiN); 0.61 (NH); 5.79—6.12 (m, 6 H, CH=CH$_2$). ^{13}C NMR, δ: 1.19 (NSiMe$_3$), 2.79 (CH$_3$SiN), 134.98—136.17 (CH=CH$_2$). ^{29}Si NMR, δ: −10.92 (OSiN), 2.70 (NHSiMe$_3$). MS, *m/z* (I_{rel} (%)): 317 [M − Me]$^+$ (100), 305 [M − Vin]$^+$ (12), 289 [M − Vin − CH$_4$]$^+$ (35), 277 (19), 243 (35), 229 (35), 217 (97), 205 (84), 191 (11), 189 (21), 142 (19), 130 (39), 85 (55), 73 Me$_3$Si$^+$ (55).

1-Chloro-1,3,5,7-tetramethyl-7-trimethylsilylamino-1,3,5,7-tetravinyl-4-aza-2,6-dioxasilane, ClMeVinSiOSiMeVinNHSiMeVinOSiMeVinNHSiMe$_3$ (3c). MS, *m/z* (I_{rel} (%)): 435 [M − Me]$^+$ (21), 423 [M − Vin]$^+$ (3), 407 [M − Vin − CH$_4$]$^+$ (6), 395 (7), 381 (8), 377 [M − SiMe$_3$]$^+$ (6), 303 (24), 287 (15), 229 (41), 217 (38), 158 (56), 105 (45), 85 (100).

1,3,5,7-Tetramethyl-1,3,5,7-tetravinylcyclotetrasila-4,8-diaza-2,6-dioxane, (MeVinSiO)$_2$(MeVinSiNH)$_2$ (4c). The yield was 92%. ^{1}H NMR, δ: 0.08—0.13 (12 H, Me); 5.89—6.11 (m, 6 H, CH=CH$_2$). ^{13}C NMR, δ: 1.09 (CH$_3$), 134.75—136.77 (CH=CH$_2$). ^{29}Si NMR, δ: −23.15 (OSiN). MS, *m/z* (I_{rel} (%)): 342 M$^+$ (3), 327 [M − Me]$^+$ (100), 315 [M − Vin]$^+$ (30), 299 [M − Vin − CH$_4$]$^+$ (18), 288 (19), 282 (8), 256 (7), 233 (6), 219 (6), 203 (6), 156 (2), 97 (11), 85 (5).

1,3,5,7-Tetramethyl-1,3,5,7-tetravinyl-4-trimethylsilylcyclotetrasila-4,8-diaza-2,6-dioxane, (MeVinSiO)$_2$(MeVinSiNH)(MeVinSiNSiMe$_3$) (5c). MS, *m/z* (I_{rel} (%)): 414 M$^+$ (5), 399 [M − Me]$^+$ (100), 387 [M − Vin]$^+$ (59), 375 (21), 359 (10), 303 (25), 275 (9), 245 (20), 217 (12), 158 (64), 130 (38), 85 (52), 73 (68).

1,7-Dichloro-1,3,5,7-tetramethyl-1,3,5,7-tetravinyl-4-aza-2,6-dioxasilane, ClMeVinSiOSiMeVinNHSiMeVinOSiMeVinCl (6c). MS, *m/z* (I_{rel} (%)): 397 M$^+$ (2), 382 [M − Me]$^+$ (17), 370 [M − Vin]$^+$ (4), 328 (20), 302 (9), 262 (36), 250 (100), 238 (51), 214 (14), 97 (52).

The reaction of 1,3-dichloro-1,3-di(chloromethyl)-1,3-dimethyldisiloxane with hexamethyldisilazane was carried out similarly (molar ratio 1 : 1). The reaction time was 120 h.

1-Chloro-1,3-di(chloromethyl)-1,3-dimethyl-3-trimethylsilylaminodisiloxane, ClMe(ClCH$_2$)SiOSiMe(CH$_2$Cl)NHSiMe$_3$ (1a). The yield was 69%. ^{1}H NMR, δ: 0.05 (s, SiMe$_3$); 0.56 (s, 3 H, MeSiN); 0.63 (s, 3 H, MeSiCl); 2.69—2.81 (q, 2 H, ClCH$_2$SiN); 2.89—2.99 (q, 2 H, ClCH$_2$SiCl). ^{13}C NMR, δ: −1.88, −1.53 (CH$_3$); 0.04 (SiMe$_3$); 29.37, 30.00 (ClCH$_2$). ^{29}Si NMR, δ: −7.23 (OSiN), −3.40 (SiMeCl), 3.57 (NHSiMe$_3$). MS, *m/z* (I_{rel} (%)): 308 [M − Me]$^+$ (19), 274 [M − ClCH$_2$]$^+$ (62), 258 [M − ClCH$_2$ − CH$_4$]$^+$ (2), 238 [M − ClCH$_2$ − HCl]$^+$ (9), 230 (18), 194 (12), 180 (30), 166 (61), 152 (40), 132 (24), 87 (71), 73 [Me$_3$Si$^+$] (100).

1,3-Di(chloromethyl)-1,3-dimethyl-1,3-bis(trimethylsilylamino)disiloxane, (Me$_3$SiNH)Me(ClCH$_2$)SiOSi(CH$_2$Cl)Me(NHSiMe$_3$) (2a). The yield was 10%. ^{1}H NMR, δ: 0.04 (s, SiMe$_3$); 0.58 (s, 6 H, MeSi); 2.84—3.04 (q, 4 H, ClCH$_2$Si). ^{13}C NMR, δ: −1.78 (Me), 0.04 (SiMe$_3$), 29.71 (ClCH$_2$). ^{29}Si NMR, δ: −6.56 (OSiN), 3.31 (NHSiMe$_3$). MS, *m/z* (I_{rel}(%)): 361 [M − Me]$^+$ (13), 327 [M − ClCH$_2$]$^+$ (100), 311 [M − ClCH$_2$ − CH$_4$]$^+$ (2), 291 [M − ClCH$_2$ − HCl]$^+$ (4), 283 (2), 252 (26), 238 (23), 218 (39), 189 (17), 146 (57), 73 [Me$_3$Si$^+$] (91).

1,3,5,7-Tetra(chloromethyl)-1,3,5,7-tetramethylcyclotetrasila-4,8-diaza-2,6-dioxane, [Me(ClCH$_2$)SiO]$_2$[Me(ClCH$_2$)SiNH]$_2$ (4a). The yield was 83%. ^{1}H NMR, δ: 0.88 (s, 6 H, MeSi); 2.74—3.91 (q, 4 H, ClCH$_2$Si). ^{13}C NMR, δ: −0.18 (Me), 29.10 (ClCH$_2$). ^{29}Si NMR, δ: −16.49 (OSiN). MS, *m/z* (I_{rel} (%)): 415 [M − Me]$^+$ (2), 381 [M − ClCH$_2$]$^+$ (100), 345 (12), 289 (19), 210 (13), 180 (23), 152 (13), 138 (22), 93 (14).

The reaction of 1,3-dichloro-1,1,3,3-tetramethyldisiloxane with hexamethyldisilazane was carried out similarly (molar ratio 1 : 1). The reaction time was 200 h.

1-Chloro-1,1,3,3-tetramethyl-3-trimethylsilylaminodisiloxane, ClMe$_2$SiOSiMe$_2$NHSiMe$_3$ (1b). The yield was 57%. ^{1}H NMR, δ: 0.06 (s, 9 SiMe$_3$); 0.21 (s, 6 H, OSiMe$_2$N); 0.47 (s, 6 H, 2 ClSiMe$_2$O). ^{13}C NMR, δ: 1.53 (SiMe$_3$), 2.43 (OSiMe$_2$NH), 3.85 (ClSiMe$_2$O). ^{29}Si NMR, δ: −11.16 (OSiN), 1.99 (NSiMe$_3$), 7.23 (ClSiMe$_2$). MS, *m/z* (I_{rel} (%)): 240 [M − Me]$^+$ (100), 224 [M − Me − CH$_4$]$^+$ (7), 220 [M − Cl]$^+$ (7), 204 [M − Me − HCl]$^+$ (4), 188 [M − Me − HCl − CH$_4$]$^+$ (12), 167 [M − Me − NHSiMe$_2$]$^+$ (4), 132 [M − Me − ClSiMe$_3$]$^+$ (87), 93 (3), 73 [Me$_3$Si$^+$] (28).

1,1,3,3-Tetramethyl-1,3-bis(trimethylsilylamino)disiloxane, (Me$_3$SiNH)Me$_2$SiOSiMe$_2$(NHSiMe$_3$) (2b). The yield was 10%. ^{1}H NMR, δ: 0.17 (s, 18 H, SiMe$_3$); 0.44 (s, 12 H, 2 SiMe$_2$). ^{13}C NMR, δ: 1.43 (SiMe$_3$), 3.81 (SiMe$_2$). ^{29}Si NMR, δ: −8.78 (OSiN), 2.79 (NHSiMe$_3$). MS, *m/z* (I_{rel} (%)): 308 M$^+$ (3), 293 [M − Me]$^+$ (100), 277 [M − Me − CH$_4$]$^+$ (7), 261 (5), 227 (3), 221 (2), 205 (51), 189 (6), 146 (10), 132 (5), 130 (17), 73 [Me$_3$Si$^+$] (20).

Octamethylcyclotetrasila-4,8-diaza-2,6-dioxane, (Me$_2$SiO)$_2$(Me$_2$SiNH)$_2$ (4b). The yield was 62%. ^{1}H NMR, δ: 0.09 (s, 24 H, 4 SiMe$_2$). ^{13}C NMR, δ: 3.77 (SiMe$_2$). ^{29}Si NMR, δ: −12.56 (OSiN). MS, *m/z* (I_{rel} (%)): 279 [M − Me]$^+$ (100), 263 [M − Me − CH$_4$]$^+$ (25), 247 [M − Me − 2 CH$_4$]$^+$ (10), 233 (3), 229 (3), 227 (4), 189 (4), 175 (3), 146 (10), 132 (15), 93 (2), 73 [Me$_3$Si$^+$] (21).

The reaction of 1,3-dichloro-1,3-dimethyl-1,3-diphenyldisiloxane with hexamethyldisilazane was carried out similarly (molar ratio 1 : 1). The reaction time was 240 h.

1-Chloro-1,3-dimethyl-1,3-diphenyl-3-trimethylsilylaminodisiloxane, ClMePhSiOSiMePhNHSiMe$_3$ (1d). The yield was 53%. ^{1}H NMR, δ: 0.07 (9 H, NSiMe$_3$); 0.68, 0.73 (6 H, CH$_3$); 7.25—7.53 (m, 10 H, C$_6$H$_5$). ^{13}C NMR, δ: 1.17 (SiMe$_3$), 2.72 (CH$_3$), 127.78, 130.45, 132.65, 134.21 (C$_6$H$_5$). ^{29}Si NMR, δ: −10.14 (OSiN), −6.53 (ClSiMe), 3.09 (NSiMe$_3$). MS, *m/z* (I_{rel} (%)): 364 [M − Me]$^+$ (100), 344 [M − Cl]$^+$ (18), 328 [M − Me − HCl]$^+$ (4), 312 [M − Me − HCl − CH$_4$]$^+$ (7), 286 [M − Ph − CH$_4$]$^+$ (9), 270 [M − Ph − 2 CH$_4$]$^+$ (5), 256 [M − Me − ClSiMe$_3$]$^+$ (6), 197 (25), 194 (17), 135 (24), 73 [Me$_3$Si$^+$] (3).

1,3-Dimethyl-1,3-diphenyl-1,3-bis(trimethylsilylamino)disiloxane, (Me$_3$SiNH)MePhSiOSiMePh(NHSiMe$_3$) (2d). The yield was 12%. ^{1}H NMR, δ: 0.06 (18 H, $NSiMe_3$); 0.83 (6 H, CH_3); 7.45—7.54 (m, 10 H, C_6H_5). ^{13}C NMR, δ: 1.29 ($SiMe_3$), 2.72 (CH_3), 127.78, 130.45, 132.65, 134.21 (C_6H_5). ^{29}Si NMR, δ: −9.69 (OSiN), 3.71 ($NHSiMe_3$). MS, *m/z* (I_{rel} (%)): 432 $M^{+\cdot}$ (11), 417 $[M - Me]^+$ (100), 401 $[M - Me - CH_4]^+$ (1), 339 $[M - Ph - CH_4]^+$ (29), 328 (14), 323 $[M - Ph - 2\ CH_4]^+$ (13), 267 (49), 250 (15), 205 (25), 197 (15), 192 (26), 135 (41), 73 $[Me_3Si^+]$ (10).

1,3,5,7-Tetramethyl-1,3,5,7-tetraphenylcyclotetrasila-4,8-diaza-2,6-dioxane, $[Me(Ph)SiO]_2[Me(Ph)SiNH]_2$ (4d). The yield was 88%. ^{1}H NMR, δ: 0.82 (6 H, CH_3); 7.44—7.54 (m, 10 H, C_6H_5). ^{13}C NMR, δ: 2.66 (CH_3), 127.98, 130.84, 132.93, 134.30 (C_6H_5). ^{29}Si NMR, δ: −10.97 (OSiN). MS, *m/z* (I_{rel} (%)): 542 $M^{+\cdot}$(8), 527 $[M - Me]^+$ (100), 511 $[M - Me - CH_4]^+$ (3), 465 $[M - Ph]^+$ (12), 449 $[M - Ph - CH_4]^+$ (39), 432 (28), 416 (13), 371 (18), 281 (22), 217 (54), 207 (41), 178 (30), 96 (17), 69 (26).

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