Synthesis and Characterization of New Cationic Hexacoordinate Silanes

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The reaction of *N*,*N*,*N*′,*N*′,*N*″-pentamethyldiethylenetriamine (pmdeta) with $HSiCl_3$ and H_2SiCl_2 in dry CH_2Cl_2 affords the novel cationic intermolecular hexacoordinate silanes $[pmdeta{\cdot}HSiCl_2]^+Cl^-$ (**1**) and $[pmdeta{\cdot}H_2SiCl]^+Cl^-$ (**2**), respectively. The addition of *N*,*N*,*N*′,*N*′,*N*″-pentaethyldiethylenetriamine (pedeta) to a solution of $HSiCl_3$ in dry CH_2Cl_2 gives the redistribution product formulated as the salt $[pedeta{\cdot}H_2SiCl]^+Cl^-$ (**3**) and $SiCl_4$. Treatment of H_2SiCl_2 with pedeta and *N*,*N*,*N*′,*N*′,*N*″,*N*‴-hexamethyltriethylenetetramine (hmteta) produces $[pedeta{\cdot}H_2SiCl]^+Cl^-$ (**3**) and $[hmteta{\cdot}H_2SiCl]^+Cl^-$ (HCl) (**4**). In **4**, the fourth amine is not bonded to silicon. Reactions of **2**, **3**, and **4** with $NaBPh_4$ give the ion exchange products $[pmdeta{\cdot}H_2SiCl]^+BPh_4^-$ (**5**), $[pedeta{\cdot}H_2SiCl]^+BPh_4^-$ (**6**), and $[hmteta{\cdot}H_2SiCl]^+BPh_4^-$ (HCl) (**7**), respectively. The salts **1**, **5**, **6**, and **7** were characterized by single-crystal X-ray diffraction analysis. Most notable is that the cations in **1**, **5**, **6**, and **7** are slightly distorted octahedra in which two hydrogen atoms are at the trans positions. The reactions of **3** with tmeda and pmdeta afford the intermolecular ligand exchange products $tmeda{\cdot}H_2SiCl_2$ and **2**, respectively.

Introduction

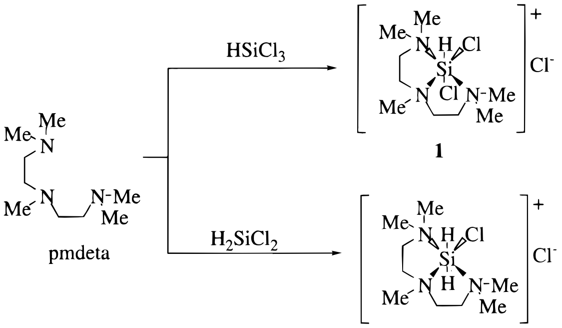
Generally, organosilanes with at least two electronegative groups on silicon will coordinate two donors to give hexacoordinate silicon compounds by inter- or intramolecular coordination.[1] Diamines such as 2,2′-bipyridine and 1,10-phenanthroline easily coordinate intermolecularly to halogenosilanes and give neutral[2] or cationic species,[3] depending on the starting silane. Fleischer recently reported that bis(dichlorosilyl)amine reacts with 3-picoline (3-methylpyridine, 3-pic) to give neutral and cationic species SiH_2Cl_2(3-pic) and $[SiH_2(3\text{-pic})_4]Cl_2$, respectively.[4]

Examples of intermolecular hexacoordinate silanes with aliphatic donor ligands are rare.[5] Recently we reported the syntheses, reactions, and characterizations of new hexacoordinate silanes with *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (tmeda) and *N*,*N*,*N*′,*N*′-tetraethylethylenediamine (teeda) complexed to $HSiCl_3$ and H_2SiCl_2.[6] In this paper we present the results of our continuing efforts in this area: the synthesis and characterization of new cationic intermolecular hexacoordinated silicon compounds containing triamines and a tetramine.

Results and Discussion

Synthesis and Characterization of $[pmdeta{\cdot}HSiCl_2]^+Cl^-$ (1), $[pmdeta{\cdot}H_2SiCl]^+Cl^-$ (2), and $[pmdeta{\cdot}H_2SiCl]^+BPh_4^-$ (5). The addition of *N*,*N*,*N*′,*N*′,*N*″-pentamethyldiethylenetriamine (pmdeta) to a solution of $HSiCl_3$ in CH_3CN proceeds exothermically to give $[pmdeta{\cdot}HSiCl_2]^+Cl^-$ (**1**) (Scheme 1). [7] Under similar conditions, adding pmdeta to a solution of H_2SiCl_2 in CH_2Cl_2 produces $[pmdeta{\cdot}H_2SiCl]^+Cl^-$ (**2**). To the best of our knowledge, this is the first example in which three nitrogen electron pairs have been used to form a hexacoordinate silane.

Scheme 1. Reaction of Pmdeta with $HSiCl_3$ and H_2SiCl_2



The ^1H NMR spectrum of **2** shows two SiH peaks at 4.04 and 4.55 ppm. The methyl group on the internal nitrogen atom is not in the plane of the three nitrogen atoms, leading to a different environment for each hydrogen attached to silicon (Figure 1). Five signals, 40.2, 47.2, 47.5, 52.6, and 57.4 ppm, for the pmdeta fragment in ^{13}C NMR indicate that the triamine is coordinated rigidly to the silicon center. ^{29}Si NMR reveals a triplet at −111.87 ppm ($^1J_{SiH}$ = 358 Hz) consistent with a hexacoordinated silicon bound to two hydrogens. The infrared and elemental analysis data also support the structure of **2**. VT-NMR analysis over the range −60 to +35 °C shows no change in the ^1H NMR, consistent with a static framework.

Adding $NaBPh_4$ in CH_3CN to a solution of **2** in CH_2Cl_2 leads to a heterogeneous mixture which yields white crystals of **5**, eq

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10.1021/ic990415p CCC: $19.00

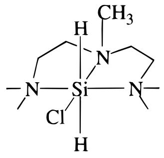


Figure 1.

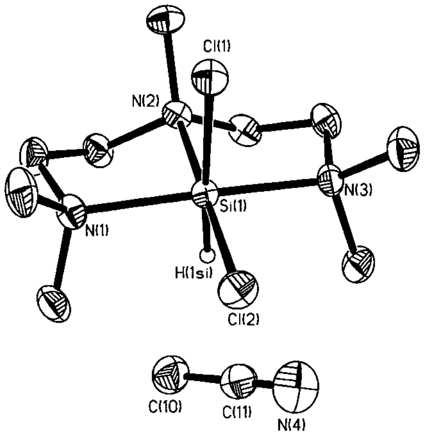


Figure 2. ORTEP diagram of [pmdeta·$HSiCl_2$]$^+$$Cl^-$·$CH_3CN$ (**1**) showing refined atoms at the 30% probability level.

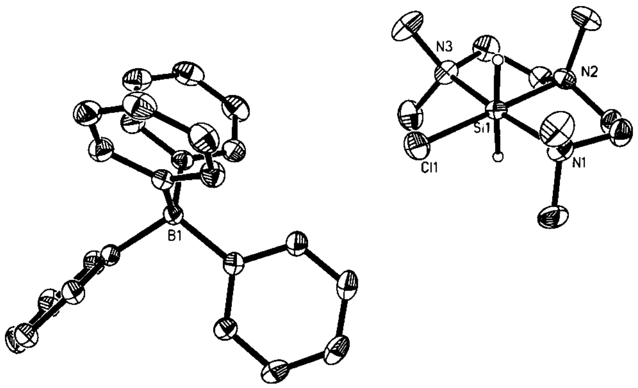
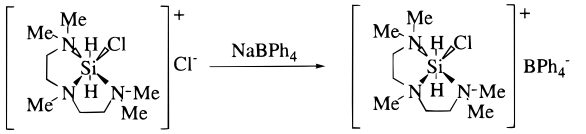


Figure 3. ORTEP diagram of [pmdeta·H_2SiCl]$^+$$BPh_4^-$ (**5**) showing refined atoms at the 30% probability level.

1. The molecular structures of [pmdeta·$HSiCl_2$]$^+$$Cl^-$ (**1**) and [pmdeta·H_2SiCl]$^+$$BPh_4^-$ (**5**) have been determined in the solid state (Figures 2 and 3).



(1)

2 **5**

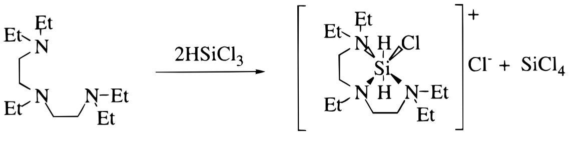
The cations in **1** and **5** exhibit a distorted octahedral geometry. Selected bond lengths and bond angles are given in Table 1, and crystallographic data are given in Table 2. The bond angles for H(1)−Si−Cl(1) in **1** and H(1)−Si−H(2) in **5** are 174.6° and 176.4°, respectively. All the Si−H, Si−N, and Si−Cl bond distances in **1** are similar to the corresponding bond distances in **5**. The Cl^- and BPh_4^- anions are well removed from the silicon centers in **1** and **5** at 7.0 and 7.8 Å, respectively.

Synthesis and Characterization of [pedeta·H_2SiCl]$^+$$Cl^-$ (3) and [pedeta·H_2SiCl]$^+$$BPh_4^-$ (6). We recently reported that while tmeda reacts with $HSiCl_3$ to form the complex tmeda·$HSiCl_3$, *N*,*N*,*N*′,*N*′-tetraethylethylenediamine (teeda) and $HSiCl_3$ give the redistributed products teeda·H_2SiCl_2 and $SiCl_4$ in high yield.[6] Similarly, we observed a dramatic change in the reactivity of the triamines toward $HSiCl_3$ when we switched the pmdeta to pedeta.

Table 1. Selected Bond Distances (Å) and Angles (deg) for **1**, **5**, **6**, and **7**

	1	5	6	7
Si−Cl(1)	2.165(3)	2.2035(7)	2.193(3)	2.2130(15)
Si−Cl(2)	2.176(2)			
Si−N(1)	2.035(5)	2.0365(16)	2.067(7)	2.018(3)
Si−N(2)	2.019(5)	2.0132(15)	2.027(8)	2.019(3)
Si−N(3)	2.035(5)	2.0403(16)	2.089(7)	2.101(3)
Si−H(1)	1.347(48)	1.384	1.353	1.415
Si−H(2)		1.376	1.282	1.395
H(1)−Si−H(2)		176.4	174.6	175.3
H(1)−Si−Cl(1)	174.6(19)			
N(1)−Si−N(3)	169.1(2)	170.75(7)	170.5(3)	169.63(14)

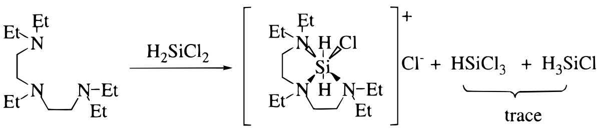
The addition of $HSiCl_3$ to a solution of *N*,*N*,*N*′,*N*′,*N*″-pentaethyldiethylenetriamine (pedeta) in dry CH_2Cl_2 proceeds exothermically to give a homogeneous solution, which, upon the addition of hexane at room temperature, produces [pedeta·H_2SiCl]$^+$$Cl^-$ (**3**), within a few minutes, eq 2. $SiCl_4$ can be removed from the reaction mixture under reduced pressure. No other products are observed.



(2)

pedeta **3**

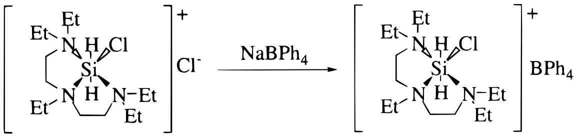
The ^1H NMR spectrum for **3** shows two Si−H peaks at ca. 4.38 and 4.73 ppm, indicating different environments for each hydrogen attached to silicon. ^{29}Si NMR gives a triplet at −106.09 ppm ($^1J_{SiH}$ = 380 Hz). Analysis of the ^{29}Si NMR data for **3** ($^1J_{SiH}$ = 380 Hz) reveals a significant upfield shift (^{29}Si: $\Delta\delta$ = −96.5 ppm) and an increase in the Si−H coupling constant ($\Delta(^1J_{SiH})$ = 12 Hz) from those of the compound $HSiCl_3$. The magnitude of the change in the chemical shift and in the coupling constant can be explained by the strong coordination of pedeta to silicon. In contrast to the total redistribution of $HSiCl_3$ promoted by pedeta, H_2SiCl_2 reacts with pedeta to give [pedeta·H_2SiCl]$^+$$Cl^-$ (**3**), with only traces of the redistribution products, $HSiCl_3$ and H_3SiCl, eq 3. The reaction of pedeta with H_2SiCl_2 probably proceeds along the same path as pmdeta with H_2SiCl_2.



(3)

pedeta **3**

The addition of $NaBPh_4$ in CH_3CN to a solution of **3** in CH_2Cl_2 leads to a heterogeneous mixture from which the ion exchange product [pedeta·H_2SiCl]$^+$$BPh_4^-$ (**6**) is obtained as white crystals, eq 4. The crystal structure of **6** shows a hexacoordinated cation similar to **5**. With the H1−Si−H2 bond



(4)

3 **6**

Table 2. Crystallographic Data for **1**, **5**, **6**, and **7**

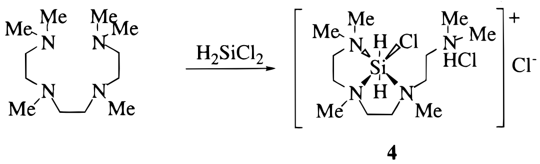
	1	**5**	**6**	**7**
empirical formula	$C_{11}H_{27}Cl_3N_4Si$	$C_{33}H_{45}BClN_3Si$	$C_{38}H_{55}BClN_3Si$	$C_{39}H_{58}BCl_4N_5Si$
fw	349.8	558.07	628.2	770.60
cryst syst	monoclinic	triclinic	monoclinic	triclinic
space group	$P2_1/n$	$P\bar{1}$	$P21/n$	$P\bar{1}$
a, Å	14.104(3)	9.6572(6)	10.6910(10)	9.4452(10)
b, Å	11.995(2)	12.0511(7)	12.200(2)	16.2805(10)
c, Å	11.049(2)	15.0631(9)	27.708(10)	16.4639(10)
α, deg		75.6950(10)		60.8980(10)
β, deg	111.43(3)	84.6380(10)	98.760(10)	87.1410(10)
γ, deg		67.6890(10)		75.3770(10)
V, Å^3	1740.0(6)	1571.52(16)	3571.8(8)	2132.5(3)
Z	4	2	4	2
ρ_{calcd}, Mg/m^3	1.335	1.179	1.168	1.211
$F(000)$	744	600	1360	828
no. of reflns collected	2898	7705	4978	10022
no. of independent reflns	2203	5276	4679	6900
GOF[a]	1.87	1.013	2.59	1.064
$R(F)$[b]	0.05	0.0383	0.068	0.0771
$R_w(F^2)$[c]	0.0641	0.0948	0.069	0.1843

[a] Goodness of fit $= s = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n - p)\}^{1/2}$. [b] $R = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$. [c] $R_w = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$.

angle of 174.6°, its geometry shows a slightly tilted octahedral structure in which two hydrogens occupy the trans position, (Figure 4).

Deviations from this geometry can be seen in the N(1)−Si−N(2) angles of 84.8(3)°, N(2)−Si−N(3) angles of 86.5(3)°, and N(1)−Si−N(3) angles of 170.5(3)°. The Si−Cl bond length is 2.193(3) Å. These bond angles and bond length are consistent with a hexacoordinate silicon compound. In addition, compared to **5**, **6** shows similar Si−H (1.353 and 1.282 Å), Si−N (2.027, 2.067, and 2.089 Å), and Si−Cl (2.193 Å) bond distances (Table 1).

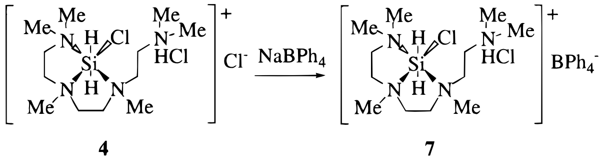
Synthesis and Characterization of [hmteta·$H_2SiCl]^+Cl^-$ (HCl) (4) and [hmteta·$H_2SiCl]^+$(HCl) BPh_4^- (7). Fleischer et al. recently reported that bis(dichlorosilyl)amine or H_2SiCl_2 reacts in a chloroform solution with 3-picoline to form two intermolecular hexacoordinate silicon compounds, H_2SiCl_2(3pic)$_2$ and $[H_2Si(3pic)_4]Cl_2$.[4] With our aliphatic ligands, we investigated the possibility of generating dicationic hexacoordinate and heptacoordinate complexes of silicon. However, the use of tetramine to form those target complexes was not successful. Instead, this reaction affords the monocationic hexacoordinate silicon complex [hmteta·$H_2SiCl]^+Cl^-$ (HCl) (**4**) (hmteta = *N,N,N′,N′,N″,N‴*-hexamethyltriethylenetetramine), in which the fourth amine is not bonded to the silicon atom, eq 5.



(5)

4

As with **5** and **6**, the addition of $NaBPh_4$ to a solution of [hmteta·$H_2SiCl]^+Cl^-$ (HCl) (**7**) in CH_2Cl_2 leads to anion exchange, eq 6. The X-ray crystal structure of **7** was determined (Figure 5).



(6)

4 **7**

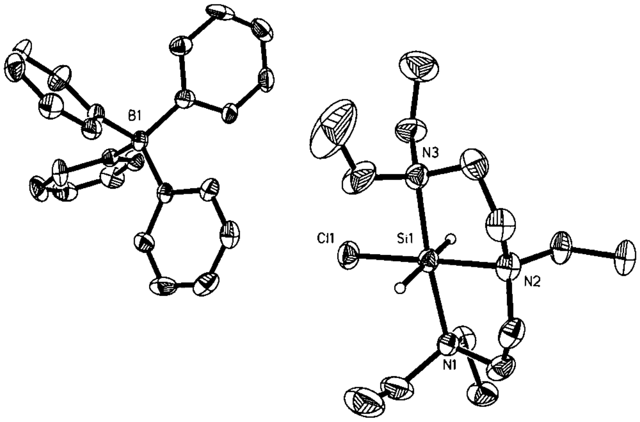


Figure 4. ORTEP diagram of [pedeta·$H_2SiCl]]^+BPh_4^-$ (**6**) showing refined atoms at the 30% probability level.

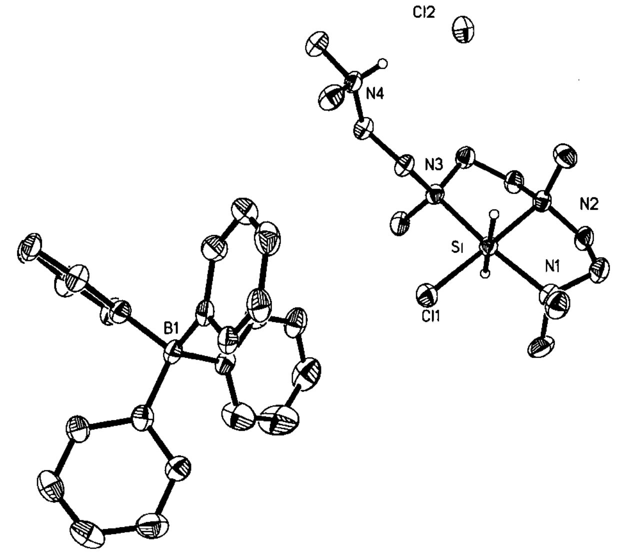
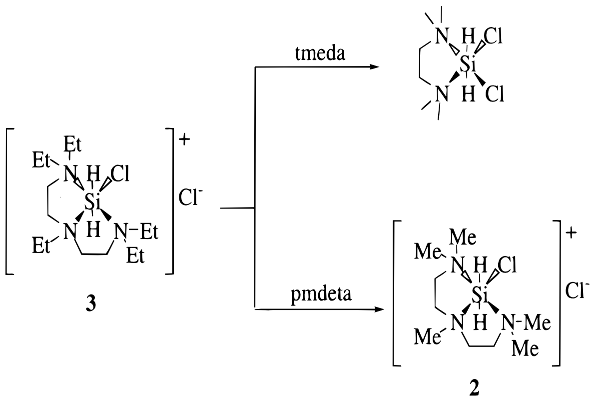


Figure 5. ORTEP diagram of [hmteta·$H_2SiCl]^+BPh_4^-$ (HCl) (**7**) showing refined atoms at the 30% probability level.

The Si−H bond distances are 1.415 and 1.395 Å; Si−N bond distances are 2.018, 2.019, and 2.101 Å; and the Si−Cl bond distance is 2.2130 Å (Table 1). The BPh_4^- ion is well removed from the silicon center of the complex at 10.003 Å.

Ligand Exchange Reactions. In a manner similar to that of the ligand exchange reaction of teeda·H_2SiCl_2,[6] the addition of

Scheme 2. Ligand Exchange Reactions of [pedeta·H$_2$SiCl]$^+$Cl$-$ (3)



tmeda and pmdeta to [pedeta·H$_2$SiCl]$^+$Cl$^-$ (**3**) in CH$_2$Cl$_2$ led to the precipitation of tmeda·H$_2$SiCl$_2$ and [pmdeta· H$_2$SiCl]$^+$Cl$^-$ (**2**), respectively (Scheme 2). Compound **3** is very soluble in CH$_2$Cl$_2$, while compounds tmeda·H$_2$SiCl$_2$ and **2** are only slightly soluble. This low solubility is probably the driving force for the ligand exchange reaction. Surprisingly, these exchanges occur while the stereochemistry at the silicon atom is preserved. Variable-temperature NMR analysis for stereochemical rigidity of compound **3** in the range from −60 to +35 °C reveals no change in geometry.

Experimental Section

General Methods. All experiments were performed under dry nitrogen. Tmeda, teeda, pmdeta, pedeta, and hmteta were dried by refluxing over sodium for at least 10 h before distillation. CH$_3$CN was heated over CaH$_2$ for more than 12 h, distilled into a flask containing P$_2$O$_5$, and then stirred over gentle heat overnight before final distillation. CH$_2$Cl$_2$ was distilled from CaH$_2$ prior to use. Hexane was first stirred over H$_2$SO$_4$, followed by stirring over sodium before distillation. Trichlorosilane was degassed through several freeze−pump−thaw cycles before bulb-to-bulb distillation. Dichlorosilane in xylene (25%) was used as obtained from Gelest Co. IR samples were prepared in a nitrogen-filled drybox using dry KBr and a screw-fitted press. IR spectra were recorded on a Mattson Cygnus 25FT-IR. ^{1}H, ^{13}C, and ^{29}Si NMR spectra were obtained on a JEOL GSX270 or JEOL GSX400 spectrometer using CDCl$_3$ as the solvent, except where otherwise indicated. ^{1}H and ^{13}C chemical shifts were reported relative to CDCl$_3$. ^{29}Si NMR spectra are referenced relative to TMS at δ 0.00. Crystal structures were performed using Siemens SHELXTL programs.[8] Elemental analyses were obtained on a Perkin-Elmer Series II CHNO/O Analyzer 2400. Melting points were taken on a Thomas-Hoover capillary melting point apparatus and are uncorrected.

[pmdeta·HSiCl$_2$]$^+$Cl$^-$·CH$_3$CN (1). Pmdeta (0.83 g, 4.79 mmol) was added to a solution of trichlorosilane (0.649 g, 4.79 mmol) in CH$_3$CN (15 mL), causing the precipitation of white solids. Filtration of the solids followed by washing with hexane (10 mL × 2) and drying by vacuum gave **1** (1.22 g, 73%). Mp = 231−232 °C. ^{1}H NMR: δ 1.95 (s, 3H), 2.77 (s, 3H), 2.81 (s, 6H), 2.86 (s, 6H), 3.12 (m, 4H), 3.38 (m, 4H), 4.03 (s,1H). ^{29}Si NMR: δ −130.3 (d, $J_{\rm SiH}$ = 358 Hz). IR: 2248 cm^{-1} (υ(CN)), 2127 cm^{-1} (υ(SiH)). Anal. Calcd for C$_{10}$H$_{27}$Cl$_3$N$_4$Si: C, 37.77; H, 7.78; N, 16.02. Found: C, 37.75; H, 7.78; N, 15.87.

[pmdeta·H$_2$SiCl]$^+$Cl$^-$ (2) and [pmdeta·H$_2$SiCl]$^+$BPh$_4$$^-$ (5). Pmdeta (4.14 g, 23.97 mmol) was added to a solution of dichlorosilane (3.63 g, 35.95 mmol) in CH$_2$Cl$_2$ (20 mL), causing the precipitation of white solids. Filtration of the solids followed by washing with hexane (30 mL × 3) and drying by vacuum gave **2** (5.8 g, 88%). Mp = 200 °C dec. ^{1}H NMR: δ 2.73 (s, 12H), 2.78 (s, 3H), 3.10−3.69 (m, 8H), 4.04 (s, 1H), and 4.55 (s,1H). ^{13}C NMR: δ 40.2, 47.2, 47.5, 52.6, and 57.40. ^{29}Si NMR: δ −111.9 (t, $J_{\rm SiH}$ = 358 Hz). IR: 2115 cm^{-1} (υ(SiH)). Anal. Calcd for C$_9$H$_{25}$Cl$_2$N$_3$Si: C, 39.41; H, 9.19; N, 15.32. Found: C, 39.49; H, 9.15; N, 14.24.

A solution of **2** (2 g, 7.29 mmol) in CH$_2$Cl$_2$ (20 mL) was added to a solution of NaBPh$_4$ (2.49 g, 7.29 mmol) in CH$_3$CN (8 mL), resulting in the precipitation of white solids after 8 h of stirring. Hexane (3 mL) was added to the filtrate. After 3 days, this solution produced white crystals of **5** (2.67 g, 66%). Mp = 195−197 °C. ^{1}H NMR: δ 2.63 (s, 12H), 2.75 (s, 3H), 2.84−3.17 (m, 8H), 3.96 (s, 1H), 4.56 (s,1H), 6.86 (t, 4H), 7.01 (t, 8H), and 7.21 (s, 8H). ^{13}C NMR: δ 39.5, 46.3, 46.5, 52.3, 56.4, 121.9 (C$_p$), 125.7 (C$_m$), 135.8 (C$_o$), and 163.9 (q, C$_{ipso}$). IR: 2133 cm^{-1} (υ(SiH)). Anal. Calcd for C$_{33}$H$_{45}$BClN$_3$Si: C, 71.02; H, 8.13; N, 7.53. Found: C, 69.95; H, 8.05; N, 7.41.

[pedeta·H$_2$SiCl]$^+$Cl$^-$ (3) and [pedeta·H$_2$SiCl]$^+$BPh$_4$$^-$ (6). Compound **3** can be prepared by two methods: (1) Pedeta (2.43 g, 10 mmol) and hexane (25 mL) were added to a solution of trichlorosilane (3.36 g, 25 mmol) in CH$_2$Cl$_2$ (25 mL). The precipitated solids were separated, washed with hexane (20 mL × 3), and dried under vacuum to give **3** (2.14 g, 63%). (2) Pedeta (2.5 g, 10.27 mmol) and hexane (20 mL) were added to a solution of dichlorosilane (1.14 g, 11.3 mmol) in CH$_2$Cl$_2$ (20 mL). The precipitated solids were separated, washed with hexane (20 mL × 3), and dried under vacuum to give **3** (3.14 g, 89%). mp = 87−89 °C. ^{1}H NMR: δ 1.08 (t, 3H), 1.21−1.25 (m, 12H), 2.83−3.33 (m, 18H), 4.38 (s, 1H), and 4.73 (s,1H). ^{13}C NMR: δ 6.4, 9.1, 41.4, 47.2, 47.7, and 48.8. ^{29}Si NMR: δ −106.09 (t, $J_{\rm SiH}$ = 380 Hz). IR: 2155 cm^{-1} (υ(SiH)). Anal. Calcd for C$_{14}$H$_{35}$Cl$_2$N$_3$Si: C, 48.82; H, 10.24; N, 12.20. Found: C, 49.27; H, 9.96; N, 12.10.

A solution of **3** (3 g, 8.7 mmol) in CH$_2$Cl$_2$ (20 mL) was added to a solution of NaBPh$_4$ (2.98 g, 8.7 mmol) in CH$_3$CN (8 mL), resulting in the precipitation of white solids after 8 h of stirring. Hexane (3 mL) was added to the filtrate. After 3 days, this solution produced white crystals of **6** (3.9 g, 71%). Mp = 185−187 °C. ^{1}H NMR: δ 1.05 (t, 3H), 1.23 (m, 12H), 2.73−3.33 (m, 18H), 4.36 (s, 1H), 4.72 (s,1H), 6.85 (t, 4H), 7.00 (t, 8H), and 7.28 (s, 8H). ^{13}C NMR: δ 6.3, 9.1, 41.3, 46.6, 47.58, 5.86, 50.6, 121.9 (C$_p$), 125.7 (C$_m$), 135.8 (C$_o$), and 163.2 (q, C$_{ipso}$). IR: 2189 cm^{-1} (υ(SiH)). Anal. Calcd for C$_{38}$H$_{55}$BClN$_3$Si: C, 72.65; H, 8.82; N, 6.69. Found: C, 72.92; H, 8.77; N, 16.70.

[hmteta·H$_2$SiCl]$^+$Cl$^-$ (HCl) (4) and [hmteta·H$_2$SiCl]$^+$ (HCl) BPh$_4$$^-$ (7). Hmteta (2.36 g, 10.27 mmol) was added to a solution of dichlorosilane (2.02 g, 20.5 mmol) in CH$_2$Cl$_2$ (20 mL), resulting in the precipitation of white solids. Isolation of the solids by filtration followed by washing with hexane (20 mL × 3) and drying by vacuum gave **4** (3.0 g, 80%). ^{1}H NMR: δ 2.60−2.68 (m, 18H), 2.98−3.71 (m, 12H), 4.03 (s, 1H), and 4.53 (s,1H). ^{29}Si NMR: δ −108.7 (t, $J_{\rm SiH}$ = 364 Hz). IR: 2133 cm^{-1} (υ(SiH)).

A solution of **4** (1.35 g, 3.67 mmol) in CH$_2$Cl$_2$ (10 mL) was added to a solution of NaBPh$_4$ (1.25 g, 3.67 mmol) in CH$_3$CN (4 mL), resulting in the precipitation of white solids after 8 h of stirring. Hexane (3 mL) was added to the filtrate. After 3 days, this solution generated white crystals of **7** (1.1 g, 46%). Mp = 130−132°C. IR: 2133 cm^{-1} (υ(SiH)).

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Supporting Information Available: Tables giving crystal data and structure refinement, atomic coordinates, isotropic and anisotropic displacement parameters, and bond lengths and angles for **1**. This material is available free of charge via the Internet at http://pubs.acs.org.

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(8) Sheldrick, G. M. SHELXTL v 5.10 software package, Bruker-axs, Madison, WI.