The Thermally Stable Silylene Si[{N(CH$_2$Bu^t)}$_2$C$_6$H$_4$-1,2]: Reactivity toward CN Double Bonds

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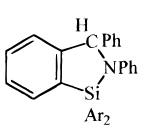
The reaction of the bis(amino)silylene Si[{N(CH$_2$Bu^t)}$_2$C$_6$H$_4$-1,2] (**5**; abbreviated as Si(NN)) with the imine Ph$_2$C=NSiMe$_3$ gave a 1:1 adduct, the fused bicyclic compound 1,2-C$_6$H$_5$[=C(Ph)N(SiMe$_3$)Si(NN)] (**6**). Upon heating, **6** was transformed into the benzosilaazacyclopentene 1,2-C$_6$H$_4$[CH(Ph)N(SiMe$_3$)Si(NN)] (**7**). In contrast, treatment of **5** with PhCH=NBu^t gave a 2:1 adduct, the silyl-substituted benzosilaazacyclopentene 1,2-C$_6$H$_4$-[C(H){Si(NN)H}N(Bu^t)Si(NN)] (**8**). Treatment of **6** with an equivalent of **5** yielded a related benzosilaazacyclopentene, 1,2-C$_6$H$_4$[C(Ph){Si(NN)H}N(SiMe$_3$)Si(NN)] (**9**). Plausible reaction pathways for these reactions are suggested. From **5** and the 1-aza- or 1,4-diazabutadiene PhCH=CHCH=NPh or Bu^{t}N=CHCH=NBu^t, there was obtained the 1-aza-2-sila-4-cyclopentene **15** or the 1,3-diaza-2-sila-4-cyclopentene **16**, respectively, by a formal [4 + 1] cycloaddition. Compounds **6**−**9**, **15**, and **16** have been characterized by multinuclear NMR spectroscopy and mass spectrometry (except for **15**), and the structure of **8** was also determined by a single-crystal X-ray diffraction study.

Introduction

In contrast to reactions between a transient silylene and a compound containing a CO double bond, little is known about silylene behavior toward C=N analogues. Only one paper has described the product obtained from a thermally generated silylene and an imine. Thus, PhCH=NPh and SiAr$_2$ gave the heteroindane (or benzosilaazacyclopentene) **1**, while a similar five-membered-



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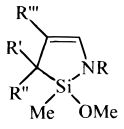

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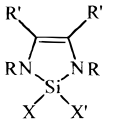

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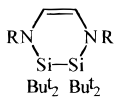

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ring 1:1 adduct was obtained from SiAr$_2$ and a fluorene-derived imine (Ar = C$_6$H$_4$(CH$_2$NMe$_2$)-2).[1] The 1-aza-diene PhC(H)C(H)CH=NPh and Si(OMe)Me gave a mixture of (*E*)- and (*Z*)-1-aza-2-sila-4-cyclopentenes **2** (R = Ph = R′ and R″ = H = R‴).[2] The 1,4-diazabutadiene [RNC(R′)]$_2$ and Si(X)X′ gave the 1,3-diaza-2-sila-4-cyclopentene **3** (X = OMe, X′ = Me, R′ = H, with R = Bu^t, C$_6$H$_{11}$-c;[2] X = Ar = X′, R′ = H, R = Bu^t, C$_6$H$_{11}$-c [1]), while [RNC(H)]$_2$ and (SiBu$^t_2$)$_2$ or SiBu$^t_2$ (from photolysis of (SiBu$^t_2$)$_3$) yielded the 1,4-diaza-2,3-disila-5-cyclohexene **4** (R = Pr^i, C$_6$H$_{11}$-c) or **3** (X = Bu^t = X′, R = Bu^t, R′ = H).[3] The 1-azatriene Pr^{i}NC(H)C(Me)C(H)C(H)C(H)-Ph and Si(OMe)Me gave some **2** (R = Pr^i, R′ = H = R‴, R″ = CHC(H)Ph), but the principal product was the 1-aza-2-sila-3-cyclopentene isomer.[4] Further reactions of a transient in situ generated silylene with a 1,4-(or 1,3-)diaza-1,3-diene have involved (i) 2-NC$_5$H$_4$-(CH=NBu^t), which with SiR$_2$ gave 2-NC$_5$H$_4$C(H)N-(Bu^t)SiR$_2$ (R = Bu^t, C$_6$H$_2$Me$_3$-2,4,6),[5] (ii) 2-NC$_5$H$_4$-(CH=NR′), which with SiR$_2$ gave 2-NC$_5$H$_4$C(NR′)Si(H)R$_2$ (R = C$_6$H$_2$Me$_3$-2,4,6, R′ = C$_6$H$_3$Pr$^i_2$-2,6),[5] and (iii) Bu^t-NC(Ph)NC(CF$_3$)$_2$, which with SiCl$_2$ generated the [4 + 1] adduct Bu^{t}NC(Ph)NC(CF$_3$)$_2$SiCl$_2$.[6]

In a preceding paper,[7] we showed that the thermally stable silylene Si[{N(CH$_2$Bu^t)$_2$C$_6$H$_4$-1,2}] (**5**; abbreviated as Si(NN)) readily reacts with ketones or oxadienes.[7] We now draw attention to the behavior of **5** with imines or azadienes. A preliminary communication, without experimental data, has appeared.[8]

Results and Discussion

Treatment of the pale yellow bis(amino)silylene **5** with *N*-(trimethylsilyl)diphenylimine, Ph$_2$C=NSiMe$_3$, at ambient temperature afforded (path i in Scheme 1) a pale yellow 1:1 adduct, the fused bicyclic dearomatized compound **6**. The latter was sufficiently stable in benzene at room temperature to be completely charac-

(1) Belzner, J.; Ihmels, H.; Pauletto, L., Noltemeyer, M. *J. Org. Chem*. **1996**, *61*, 3315.
(2) Heinicke, J.; Gehrhus, B. *J. Organomet. Chem*. **1992**, *423*, 13.
(3) Weidenbruch, M.; Lesch, A. *J. Organomet. Chem*. **1991**, *407*, 31.
(4) Heinicke, J.; Gehrhus, B. *Heteroat. Chem*. **1995**, *6*, 461.
(5) Weidenbruch, M.; Piel, H.; Lesch, A.; Peters, K.; von Schnering, H. G. *J. Organomet. Chem*. **1993**, *454*, 35.
(6) Karsch, H. H.; Bienlein, F. in *Organosilicon Chemistry II*; Auner, N., Weis, J., Eds.; VCH: New York, 1996; p 133.
(7) Gehrhus, B.; Hitchcock, P. B.; Lappert, M. F. *Organometallics* **1997**, *16*, 4861.
(8) Gehrhus, B.; Lappert, M. F. *Polyhedron*, in press.

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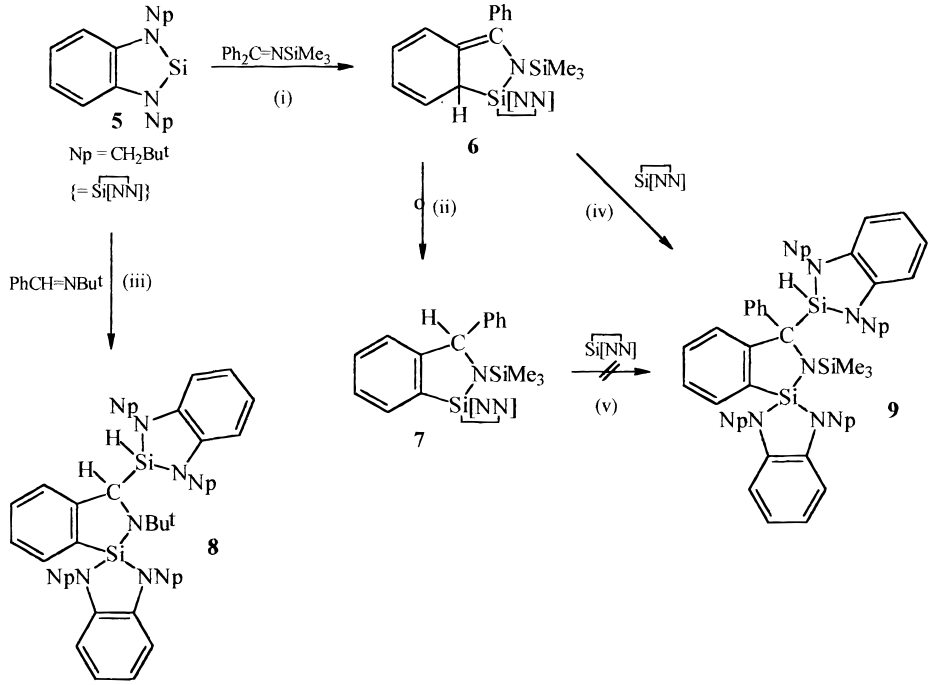
Scheme 1

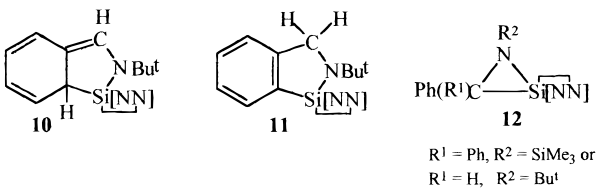


terized by NMR spectroscopy, but the solid was gradually transformed over a period of weeks by a 1,3-hydrogen shift (path ii in Scheme 1) into the thermodynamically more stable rearomatized isomer, the pale yellow azaindane **7**. This rearrangement was more rapidly accomplished by heating a benzene solution of **6** under reflux for 6 h.

In contrast, under similar conditions, **5** with *N-tert*-butylphenylimine, PhCH=NBu^t, gave (path iii in Scheme 1) the 2:1 adduct, the colorless 3-silyl-substituted benzo-1-sila-2-azacyclopentene **8**.

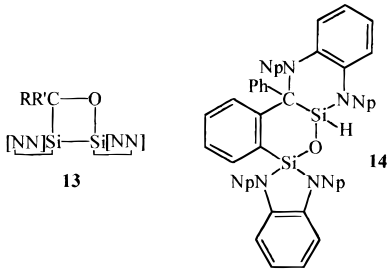
To ascertain whether a 2:1 silylene−imine adduct might also be accessible for the case of $Ph_2C{=}NSiMe_3$ as the imine, **5** was heated with an equimolar portion of the 1:1 adduct **6** in benzene at 70 °C for 24 h. The product of this reaction (path iv in Scheme 1) was the pale yellow compound **9**, an analogue of **8** except for the substituents at N-2 (Bu^t in **8**, Me_3Si in **9**) and C-3 (H in **8**, Ph in **9**) of the 1-sila-2-aza-4-cyclopentene ring. Compounds **7**−**9** have the same central skeletal structure as the heteroindane **1**; whereas **7** is derived from a single silylene unit (like **1**), two were required for **8** and **9**.

As a contribution to a study of the reaction pathway to the 2:1-adduct, it was established by NMR spectroscopic experiments (using the identical procedure which from **6** + **5** gave **9** (path iv in Scheme 1)) that the rearranged 1:1 adduct **7** did not react (path v in Scheme 1) with **5** to give **9**. Hence, **7** is not an intermediate along the pathway **6** → **9**. Although in the aldimine system leading from **5** to **8**, no intermediates have been isolated, it is plausible that an analogue **10** of **6** has a role but is kinetically more labile than **6** for stereoelectronic reasons, the latter being both more bulky and more extensively conjugated. The further intermediacy between **10** and **8** of an analogue **11** of **7** cannot be ruled out. It is proposed that the initial product of the interaction of the silylene **5** and an imine is the azasilacyclopropane **12**, formed by a [1 + 2] cycloaddition.



Each of the crystalline compounds **6**−**9** was characterized by multinuclear NMR spectroscopy, electron impact mass spectrometry (parent ions observed), and microanalysis. Compound **8** is particularly unusual, and hence, a single-crystal X-ray diffraction study was carried out. The molecular structure is shown in Figure 1 (with the atom labeling scheme), and selected bond lengths and angles are listed in Table 1. The Si−N bonds (average 1.745(4) Å) are normal, as is the Si(1)−C(33) bond, but the Si(2)−C(39) bond (1.924(4) Å) is significantly longer.

The above results may be compared with those we reported in a preceding paper dealing with the reaction of the silylene **5** with the ketones RR′C=O (R = Ph = R′; R = Me, R′ = Bu^t; R = R′ = adamantanyl).[7] At ambient temperature in benzene the product was the 2:1 adduct, the disilaoxetane **13**, while at 60 °C, the



isomeric substituted disilabenzpyran **14** (a single diastereoisomer) was obtained. It was proposed that intermediates in the formation of **14** from 2(**5**) + Ph_2CO were an oxirane (an O analogue of **12**, R^1 = Ph) followed by

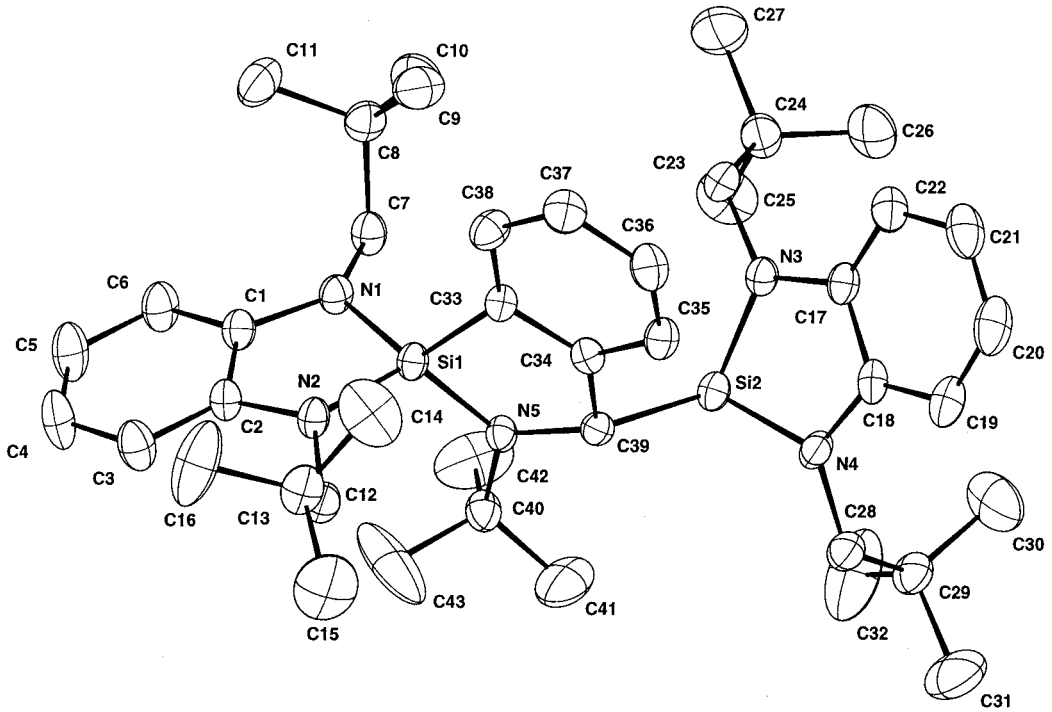


Figure 1. Molecular structure of **8** showing the atom-labeling scheme. The thermal ellipsoids are drawn at the 50% probability level.

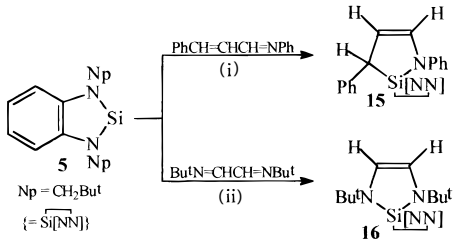
Table 1. Selected Bond Lengths (Å) and Angles (deg) for 8

Si(1)−N(5)	1.723(4)	Si(1)−N(1)	1.729(4)
Si(1)−N(2)	1.738(4)	Si(1)−C(33)	1.828(4)
Si(2)−N(3)	1.716(4)	Si(2)−N(4)	1.729(4)
Si(2)−C(39)	1.924(4)	N(1)−C(1)	1.403(5)
N(1)−C(7)	1.457(5)	N(2)−C(2)	1.399(5)
N(2)−C(12)	1.466(5)	N(3)−C(17)	1.414(5)
N(3)−C(23)	1.449(5)	N(4)−C(18)	1.415(6)
N(4)−C(28)	1.451(6)	N(5)−C(39)	1.483(5)
N(5)−C(40)	1.491(5)		
N(5)−Si(1)−N(1)	117.9(2)	N(5)−Si(1)−N(2)	119.3(2)
N(1)−Si(1)−N(2)	91.8(2)	N(5)−Si(1)−C(33)	93.2(2)
N(1)−Si(1)−C(33)	122.9(2)	N(2)−Si(1)−C(33)	114.1(2)
N(3)−Si(2)−N(4)	92.5(2)	N(3)−Si(2)−C(39)	116.7(2)
N(4)−Si(2)−C(39)	111.0(2)	C(1)−N(1)−C(7)	121.9(4)
C(1)−N(1)−Si(1)	110.4(3)	C(7)−N(1)−Si(1)	125.1(3)
C(2)−N(2)−C(12)	121.0(4)	C(2)−N(2)−Si(1)	110.9(3)
C(12)−N(2)−Si(1)	123.7(3)	C(17)−N(3)−C(23)	121.2(4)
C(17)−N(3)−Si(2)	110.3(3)	C(23)−N(3)−Si(2)	128.0(3)
C(18)−N(4)−C(28)	121.1(4)	C(18)−N(4)−Si(2)	109.3(3)
C(28)−N(4)−Si(2)	129.6(3)	C(39)−N(5)−C(40)	119.8(3)
C(39)−N(5)−Si(1)	113.2(3)	C(40)−N(5)−Si(1)	125.8(3)

analogues (O in place of $NSiMe_3$) of successively **6** and **9**.[7] The failure to obtain a disilaazabutane from 2(**5**) + $Ph_2C{=}NR$ (R = Bu^t, $SiMe_3$), in contrast to the formation of **13** from 2(**5**) + Ph_2CO,[7] is attributed to steric hindrance. The oxygen analogue of **8** (O in place of NBu^t) was not identified in the Si(NN)−Ph_2CO system; if formed, it readily isomerized into **14**, the driving force being the oxophilicity of silicon, since **14** is a cyclic disiloxane.

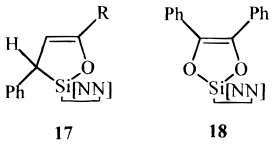
Treatment of the silylene **5** with an equimolar portion of the 1-azabutadiene PhC(H)C(H)C(H)=NPh in benzene at ambient temperature gave (path i in Scheme 2) the 1-aza-2-sila-4-cyclopentene **15** as a pale yellow, high-boiling oil. Under similar conditions, **5** and *N*,*N*′-di-*tert*-butyl-1,4-diazabutadiene ([$Bu^tNC(H)]_2$) gave (path ii in Scheme 2) the colorless, crystalline 1,3-diaza-2-sila-4-cyclopentene **16**. Both compounds were characterized

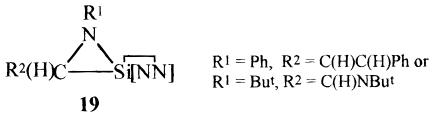
Scheme 2



by multinuclear NMR spectra and **16** also by electron impact mass spectrometry (parent ion) and microanalysis. Compounds **15** and **16** have skeletal similarities to **2** and **3**, respectively. Hence, their formation differs from similar reactions of transient silylenes SiX(X′) only in the important respect that they were obtained under very mild conditions.

The formation of **15** and **16** from **5** and the above azabutadiene or diazabutadiene is similar to that of the oxa and dioxa analogues **17** and **18** obtained[7] from **5** and PhC(H)C(H)C(R)O (R = Ph, Me) and $[PhC(O)]_2$, respectively. The reaction pathway leading to **15** and **16** may well have implicated the initial 1 + 2 cycloadduct **19**.





Experimental Section

General Considerations. The NMR spectra (^1H-decoupled, unless otherwise stated) were recorded in [$^2H_6$]benzene

at 298 K using a Bruker AC-P 250 (at 250 MHz (^{1}H) or 62.86 MHz (^{13}C)), a Bruker DPX 300 (at 300 MHz (^{1}H) or 75.48 MHz (^{13}C)), or a Bruker AMX 500 (at 500 MHz (^{1}H) or 99.33 MHz (^{29}Si)) and referenced internally to residual solvent resonances (chemical shift data in δ). Electron impact mass spectra were taken from solid samples using a Kratos MS 80 RF instrument. Melting points were taken in sealed capillaries and are uncorrected.

Synthesis of 6. A solution of **5** (0.9 g, 3.3 mmol) in benzene (20 mL) was added to a solution of $Ph_2C{=}N(SiMe_3)$ (0.84 g, 3.3 mmol) in benzene (20 mL), and the mixture was stirred for 16 h at room temperature. The solvent was removed under vacuum, and the remaining solid was redissolved in *n*-pentane. Cooling to −25 °C afforded **6** (1.35 g, 78%) as a pale yellow solid. Anal. Calcd for $C_{32}H_{45}N_3Si_2$: C, 72.8; H, 8.59; N, 7.96. Found: C, 72.4; H, 8.41; N 8.04. ^{1}H NMR (500 MHz): δ −0.18 ($SiMe_3$, s, 9H), 0.95 and 1.08 (CMe_3, 2 s, 18H), 3.25, 3.27, 3.30, 3.33, 3.34, 3.36, and 3.37 (CH_2, AB-type, 4H), 3.96 (H^5-CSi, n, 1H), 5.49 (H^3, 4 dd, 1H), 5.87 (*H*^{1}−CH^5−Si, 2 dt, 1H), 5.91 (H^2, 4 dd, 1H), 5.98 (H^4−C−C=CPh, dquint, 1H), 6.81−7.29 (phenyl, m, 9H). ^{13}C NMR (62.86 MHz): δ 2.39 ($SiMe_3$), 28.93, and 29.45 (C*Me*$_3$), 33.84 and 34.37 (*C*Me$_3$), 55.78 and 57.52 (CH_2), 28.45 (CH), 109.39, 110.75, 115.74, 118.29, 118.58, 119.57, 123.74, 125.28, 125.37, 128.33, 131.29, 131.44, 136.89, 140.49, 140.89, and 142.45 (phenyl). ^{29}Si NMR: δ 5.42, 6.78. ^{1}H NMR coupling constants (Hz): H^1, $^3J_{H^1H^2}$ = 9.3, $^3J_{H^1H^5}$ = 3.11, and $^4J_{H^1H^3}$ = 1.16; H^2, $^3J_{H^2H^1}$ = 9.3, $^3J_{H^2H^3}$ = 5.34, $^4J_{H^2H^4}$ = 0.77, and $^4J_{H^2H^5}$ = 3.42; H^3, $^3J_{H^3H^2}$ = 5.33, $^3J_{H^3H^4}$ = 9.58, $^4J_{H^3H^1}$ = 1.1, and $^5J_{H^3H^5}$ = 2.2; H^4, $^3J_{H^4H^3}$ = 9.55, $^4J_{H^4H^2}$ = 0.98, and $^4J_{H^4H^5}$ = 2.25; H^5, $^3J_{H^5H^1} \approx {}^4J_{H^5H^2} \approx$ 3.3, and $^4J_{H^5H^4} \approx {}^5J_{H^5H^3} \approx$ 2.3. MS (*m*/*z* (%)): 527 (80), [M]$^+$.

Synthesis of 7. Compound **6** (1 g, 1.89 mmol) was dissolved in benzene (30 mL) and the solution refluxed for 6 h. After removal of the solvent under vacuum, the remaining solid was recrystallized in *n*-hexane at −25 °C to give **7** (0.7 g, 70%) as a pale yellow microcrystalline solid, mp 189 °C. Anal. Calcd for $C_{32}H_{45}N_3Si_2$: C, 72.8; H, 8.59; N, 7.96. Found: C, 72.5; H, 8.49; N 8.01. ^{1}H NMR (500 MHz): δ −0.15 ($SiMe_3$, s, 9H), 0.84 and 0.98 (CMe_3, 2 s,18H), 3.02, 3.07, 3.25, 3.31, 3.37, 3.54, and 3.59 (CH_2, AB-type, overlapped, 4H), 5.26 (CH, s, 1H), 6.81−7.24 and 7.54−7.58 (phenyl, m, 9H). ^{13}C NMR (75.48 MHz): δ 0.56 ($SiMe_3$), 28.52 and 29.62 (C*Me*$_3$), 33.88 and 34.49 (*C*Me$_3$), 54.5 and 57.17 (CH_2), 65.12 (CH), 109.53, 109.74, 118.03, 118.22, 125.62, 127.11, 127.77, 128.57, 129.25, 130.71, 133.18, 140.58, 141.09, 145.58, and 155.19 (phenyl). ^{29}Si NMR: δ 6.44, 5.49. MS (*m*/*z* (%)): 527 (45) [M]$^+$.

Synthesis of 8. A solution of **5** (0.5 g, 1.825 mmol) in benzene (40 mL) was slowly added to PhCH=NBu^t (0.29 g, 1.825 mmol) in benzene (20 mL) at room temperature. After the solution was stirred for 16 h, the solvent was removed under vacuum to give a yellow residue that was redissolved in *n*-hexane to yield at −25 °C **8** (0.4 g, 62%) as a microcrystalline solid. Recrystallization from *n*-hexane afforded colorless crystals, mp 179−180 °C. Anal. Calcd for $C_{43}H_{67}N_2Si$: C, 72.7; H, 9.51; N, 9.86. Found: C, 72.4; H, 9.68; N, 9.65. ^{1}H NMR (500 MHz): δ 0.73 (N−CMe_3, s, 9H), 0.97, 0.98, 1.13, and 1.17 (CMe_3, 4 s, 36H), 2.33, 2.36, 2.83, 2.86, 2.95, 2.98, 2.96, 2.99, 3.00 3.03, 3.36, 3.39, 3.42, and 3.45 (CH_2, 2 × AB-type, 8H), 4.29 (CH, s, 1H, $^1J_{CH}$ = 130.9 Hz), 6.42 (SiH, s, 1H, $^1J_{SiH}$ = 233.2 Hz, $^2J_{CSiH}$ = 23.6 Hz), 6.68−7.75 (phenyl, m, 13H). ^{13}C NMR (75.48 MHz): δ 31.8 (N−C*Me*$_3$), 52.07 (N-*C*Me$_3$), 28.86, 28.9, 29.15, and 30.12 (C*Me*$_3$), 33.73, 34.83, 35.73, and 35.78 (*C*Me$_3$), 53.24, 53.81, 54.65, and 56.79 (CH_2), 55.72 (^{13}C proton coupled: $^1J_{CH}$ = 130.9 Hz, $^2J_{CSiH}$ = 23.6 Hz, CH). ^{29}Si NMR: δ −4.16, −12.66 (^{29}Si proton coupled: $^1J_{SiH}$ = 242.6 Hz). MS (*m*/*z* (%)): 668 (63), [M]$^+$.

Synthesis of 9. A solution of **5** (0.23 g, 0.84 mmol) in benzene (20 mL) was added to a solution of **6** (0.44 g, 0.84 mmol) in benzene (20 mL), and the mixture was stirred for 24 h at 70 °C. After removal of the solvent under vacuum, the residue was redissolved in *n*-pentane and cooled at −25 °C to afford **9** (0.37 g, 55%) as pale yellow crystals, mp 201−202 °C. Anal. Calcd for $C_{48}H_{71}N_5Si_3$: C, 71.85; H, 8.92; N, 8.72. Found: C, 72.1; H, 9.19; N, 8.92. ^{1}H NMR (300 MHz): δ −0.15 ($SiMe_3$, s, 9H), 0.73, 0.8, 0.97, and 1.14 (CMe_3, 4s, 36H), 2.73, 2.78, 3.16, 3.18, 3.21, 3.23, 3.41, 3.45, 3.50, 3.51, 3.56, 3.80 and 3.85 (CH_2, AB-type, 8H), 5.64 (SiH, s, 1H, $^1J_{SiH}$ = 125.6 Hz), 6.82−7.21 and 7.93 (phenyl, m and d, 17H). ^{13}C NMR (75.48 MHz): δ 2.96 ($SiMe_3$), 28.79, 29.57, 29.83, and 29.99 (C*Me*$_3$), 34.22, 35.02, 35.51 and 36.16 (*C*Me$_3$), 53.68, 55.02, 55.34, and 55.71 (CH_2), 69.88 (Ph*C*), 109.27, 110.32, 110.94, 112.39, 117.97, 118.03, 118.10, 118.58, 119.43, 125.96, 127.48, 128.42, 129.18, 130.28, 130.39, 136.22, 139.44, 139.51, 141.26, 145.68 and 156.01 (phenyl). ^{29}Si NMR: δ 5.81 ($SiMe_3$), −8.71 and −12.52 (SiH, ^{29}Si proton coupled: $^1J_{SiH}$ = 252 Hz). MS (*m*/*z* (%)): 801 (55) [M]$^+$.

Synthesis of 15. PhCH=CHCH=NPh (0.35 g, 1.715 mmol) was added to a solution of **5** (0.47 g, 1.715 mmol) in benzene (30 mL) and stirred for 16 h at room temperature. After removal of the solvent under vacuum, the remaining oil was distilled to give **15** (0.78 g, 93%) as a pale yellow oil, bp 190−195 °C/0.01 Torr. ^{1}H NMR (300 MHz): δ 0.67 and 0.93 (CMe_3, 2s, 18H), 1.91, 1.97, 2.72, and 2.77 (CH_2, AB-type, 2H), 3.18 (CH_2, s, 2H), 3.83 (PhCH, t, H), 5.30 (CH, q, H), 6.61−7.97 (CH and Ph, m, 15H). ^{13}C NMR (75.48 MHz): δ 28.07 and 28.85 (C*Me*$_3$), 33.64 and 33.94 (*C*Me$_3$), 32.10 (PhCH), 55.04 and 56.48 (CH_2), 104.06 (CH), 110.00, 110.32, 116.72, 118.70, 118.85, 120.99, 125.60, 127.36, 128.94, 129.74, 136.07, 139.78, 140.26, 140.66, and 143.80 (CH and Ph). ^{29}Si NMR: δ −9.25.

Synthesis of 16. Bu^{t}N=CHCH=NBu^t (0.55 g, 3.28 mmol) was added to a solution of **5** (0.9 g, 3.28 mmol) in benzene (30 mL) the mixture and stirred for 16 h at room temperature. After removal of the solvent under vacuum, the remaining solid was recrystallized from Et_2O at −25 °C to afford **16** (1.1 g, 76%) as colorless plates, mp 191 °C. Anal. Calcd for $C_{26}H_{46}N_4Si$: C, 70.5; H, 10.47; N, 12.65. Found: C, 70.2; H, 10.41; N 12.62. ^{1}H NMR (300 MHz): δ 1.03 and 1.20 (CMe_3, 2s, 18H), 3.3 (CH_2, s, 4H), 5.72 (CH, s, 2H), 6.8−6.95 (phenyl, m, 4H). ^{13}C NMR (75.48 MHz): δ 28.92 and 30.12 (C*Me*$_3$), 33.62 (*C*Me$_3$), 51.07 (N*C*Me$_3$), 56.78 (CH_2), 109.72 (CH), 110.69, 118.41, and 138.99 (phenyl). ^{29}Si NMR: δ −34.51. MS (*m*/*z* (%)): 442 (100), [M]$^+$.

Table 2. Details of the X-ray Structure Determination of 8

empirical formula	$C_{43}H_{67}N_5Si_2$
fw	710.2
temp (K)	293(2)
wavelength (Å)	0.710 73
cryst system	monoclinic
space group	$P2_1/c$ (No. 14)
a (Å)	15.636(4)
b (Å)	19.383(5)
c (Å)	14.474(4)
α (deg)	90
β (deg)	103.98(2)
γ (deg)	90
V (Å^3)	4257(2)
Z	4
D_{calcd} (Mg/m^3)	1.11
abs coeff (mm^{-1})	0.12
F(000)	1552
cryst size (mm)	0.40 × 0.30 × 0.30
θ range for data collcn (deg)	2−25
index ranges	$-18 \le h \le 18$, $0 \le k \le 23$, $0 \le l \le 17$
no. of rflns collcd	7765
no. of indep rflns	7462 (*R*(int) = 0.0677)
rflns with $I > 2\sigma(I)$	3989
no. of data/restraints/params	7459/0/451
goodness of fit on F^2	1.048
final *R* indices ($I>2\sigma(I)$)	R1 = 0.075, wR2 = 0.165
R indices (all data)	R1 = 0.155, wR2 = 0.211
largest diff peak and hole (e/Å^3)	0.38 and −0.43

X-ray Structure Determination of 8. Crystallographic data for compound **8** are displayed in Table 2. Unique data

sets were collected using an Enraf-Nonius CAD4 diffractometer, using a crystal sealed in a Lindemann capillary under argon at 293(2) K. Crystallographic programs used for structure solution and refinement were from SHELXS-86[9] and SHELXL-93.[10] Refinements were based on full-matrix least squares on all F^2 values. All non-H atoms were anisotropic, and the hydrogen atoms were included in a riding mode.

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Supporting Information Available: Tables giving data collection and processing parameters, atomic coordinates and isotropic temperature factors, bond lengths and angles, and anisotropic thermal parameters for **8** (9 pages). Ordering information is given on any current masthead page.

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