The Thermally Stable Silylene Si[{N(CH₂Buᵗ)}₂C₆H₄-1,2]: Reactivity toward CO Double Bonds

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Treatment of the bis(amino)silylene Si[{N(CH₂Buᵗ)}₂C₆H₄-1,2] (**1**) [abbreviated as Si(NN)] with RR′C=O (R = R′ = Ph, R = Me and R′ = Buᵗ, or R = R′ = adamantanyl) yielded the corresponding disilaoxetane RR′COSi(NN)Si(NN) (**2**−**4**); an oxasilacyclopropane, as a formal [2+1] cycloaddition product, is believed to be the intermediate. The reaction of **1** with benzophenone at 60 °C afforded a 2:1 adduct 2-C₆H₄Si(NN)OSi(H){N(R)C₆H₄NR-2}CPh (R = CH₂Buᵗ) **8**, for which a multistep mechanism is proposed. The [4+1] cycloaddition of **1** with PhCHCHC(R)O or PhC(O)C(O)Ph gave the oxasilacyclopentenes Si(NN)OC(R)C(H)-C(H)Ph (**9**, R = Ph; **10**, R = Me) or the dioxasilacyclopentene Si(NN)OC(Ph)C(Ph)O (**11**). X-ray structures for compound **2** and **8** are provided.

Introduction

Ever since silylenes were recognized as transients, there has been a quest to isolate these divalent and two-coordinate silicon species SiXX′. Theoretical studies in 1986 provided suggestions as to factors that may contribute to the stability of a silylene, most effectively by an NH₂ substituent (22.3 kcal mol⁻¹ stabilization of Si(NH₂)₂ relative to SiH₂).[1] Subsequently, the bis(amino)silylene Me₂Si(NBuᵗ)₂Si: was detected in an argon matrix (stable up to 77 K) by Veith *et al.* in 1992 and identified by its IR spectrum.[2] The first thermally stable bis(amino)silylene Si[N(Buᵗ)CHCHNBuᵗ] (**I**) was synthesized by Denk and West *et al.* in 1994 using additional aromatic stabilization, and its structure in the vapor phase was established by electron diffraction.[3] In 1995 we reported the synthesis of the thermally stable, crystalline bis(amino)silylene Si[{N(CH₂-Buᵗ)}₂C₆H₄-1,2] **1** and its X-ray structure.[4] UV-Photoelectron spectra and MO calculations on **I** and **1** have been reported.[5]

The chemical behavior of the stable bis(amino)-silylenes **I** and **1** is relatively unexplored. Treatment of **I** with [Ni(CO)₄] yielded the bis(silylene) complex [Ni-(CO)₂(**I**)₂];[6] with the azide RN₃ (R = Ph₃C or Me₃Si) **I** afforded **I**(=NR)L (R = CPh₃ and L = THF) or **I**(N₃)-[N(SiMe₃)₂].[7] Recently the silylene−borane adduct **I**→B-(C₆F₅)₃ was isolated; it was slowly converted into the silylborane **I**(C₆F₅)[B(C₆F₅)₂].[8] The silylene **1** readily underwent oxidative addition reactions with MeI, EtOH, or a chalcogen to give **1**(Me)I, **1**(OEt)H, or [**1**(μ-E)]₂ (E = S, Se, or Te).[9]

Addition of a transient silylene or SiCp*₂ (Cp* = C₅-Me₅) to a CO double-bonded system is now well-known. Such reactions have involved ketones and aldehydes,[10−14] 1-oxadienes,[14,15] benzil,[14,15] 1-oxatrienes,[16] or α-ket-imines.[15,17] With the exception of SiCp*₂ [a stable Si-(II) compound, behaving as a silylene],[18] the silylenes in these reactions were generated *in situ* either by thermolysis or photolysis.

We now describe the chemical behavior of **1** toward selected ketones in order to establish similarities and/or differences from its transient relatives. A preliminary communication, without experimental data, has appeared.[19]

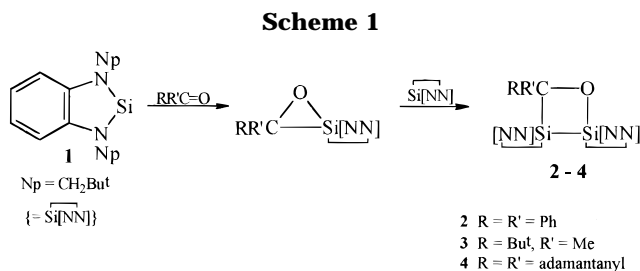⊗ Abstract published in *Advance ACS Abstracts,* September 15, 1997.
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Scheme 1

Np = CH$_2$Bu^t

{= $\overline{\text{Si[NN]}}$}

2   R = R' = Ph
3   R = Bu^t, R' = Me
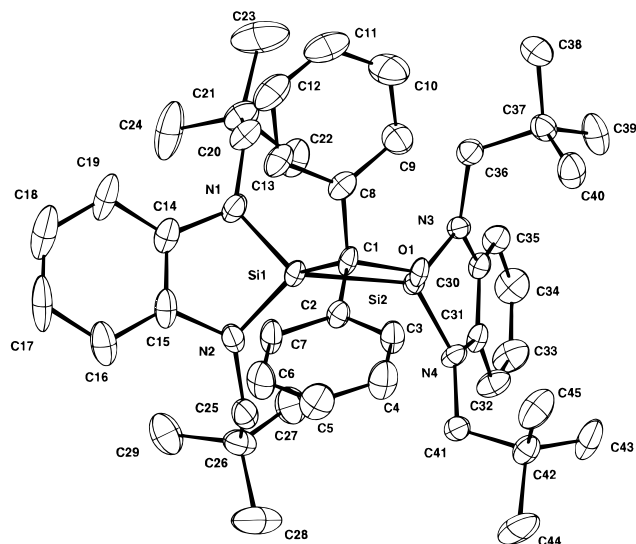4   R = R' = adamantanyl

Results

Treatment of the pale yellow bis(amino)silylene **1** with an equimolar portion of benzophenone at room temperature gave the colorless, crystalline disilaoxetane **2** in good yield. Similar results were obtained with 3,3-dimethylbutan-2-one or 2-adamantan-2-one affording the pale yellow crystalline **3** or **4**, respectively. Related four-membered SiOCSi ring compounds have previously been obtained only by reaction of a disilene with a ketone.[20] Since **1** is monomeric in solution,[4] the likely pathway for these reactions probably involves an initial [2+1] cycloaddition to form an oxasilacyclopropane intermediate which is responsive to attack by a further silylene molecule (Scheme 1).
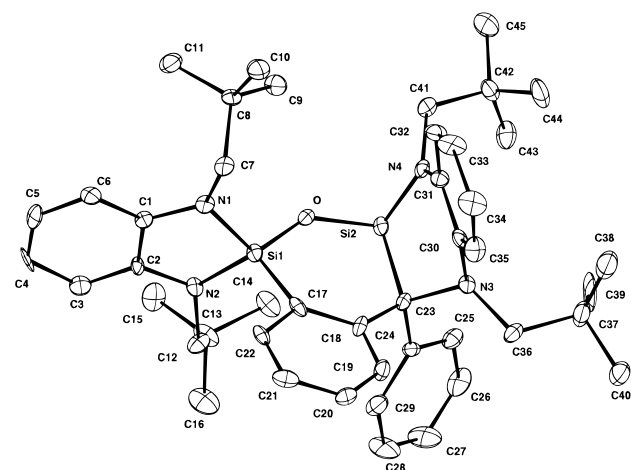
Different reactions of transient silylenes with adamantan-2-one have been described in the literature, which gave (i) a stable oxasilacyclopropane,[13] (ii) a dioxasilacyclopentane,[10,21] or (iii) a dioxadisilacyclohexane.[10] The formation of (ii) was believed to involve a ketone insertion into a silacyclopropane, while (iii) was postulated to involve the latter followed by dimerization of its Si–C cleavage product. As for the reaction of **1** with adamantan-2-one, neither a product analogous to (i), (ii), nor (iii) was observed by adding **1** slowly to a solution of the ketone in benzene at room temperature or under reflux, **4** being the major product.

The molecular structure of compound **2** was determined by single-crystal X-ray diffraction and with the atom labeling scheme is shown in Figure 1; selected bond lengths and angles are displayed in Table 1. By comparison with the previously described molecular structure of the crystalline diphenyl-1,1,2,2-tetra-*tert*-butyl-1,2-disilaoxetane,[12] we find the planar ring system of **2** less sterically overcrowded, as evident from slightly shorter SiC, SiSi, and SiO bonds but a longer CO bond.

Transient silylenes, generated by thermolysis, have been shown to react with benzophenone to yield siloxaindanes.[10,13] Hence, we added a solution of **1** slowly to a refluxing solution of benzophenone in benzene in order to promote an intramolecular rearrangement of the first formed oxasilacyclopropane intermediate rather than cause it to react by addition of another silylene as in Scheme 1. However, the analytical data of the isolated compound did not support the expected siloxaindane structure **6** (Scheme 2). Instead, mass spectral analysis indicated that the product comprises of silylene/benzophenone fragments in the ratio 2:1, as for **2** (obtained from **1** and Ph$_2$CO at room temperature), but the ^{1}H-NMR data indicated the chemical inequivalence of the four neopentyl groups. Moreover, a signal

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Figure 1.

Figure 2.

Table 1. Selected Bond Lengths (Å) and Angles (deg) for 2

Si(1)–C(1)	1.963(6)	Si(1)–N(1)	1.729(5)
O–C(1)	1.502(6)	Si(1)–N(2)	1.720(5)
Si(2)–O	1.649(4)	Si(2)–N(3)	1.719(5)
Si(1)–Si(2)	2.352(3)	Si(2)–N(4)	1.721(5)
C(1)–Si(1)–Si(2)	72.9(2)	O–Si(2)–Si(1)	79.6(2)
C(1)–O–Si(2)	110.1(3)	O–C(1)–Si(1)	97.2(3)
N(2)–Si(1)–N(1)	92.2(3)	N(3)–Si(2)–N(4)	92.0(2)

for an Si–H proton was consistent with the notion that the second silylene unit had inserted into the CH bond of **6** to give **7**. In order to provide a definitive answer as to its nature, its molecular structure was determined to be **8** by single-crystal X-ray diffraction (Figure 2; Table 2). From these data it is clear that crystalline **8** is a single diastereoisomer [Si(2) and C(23) are chiral centers]; the solution NMR spectroscopic data showed that this was also the case for the bulk sample in solution.

We suggest that the reaction pathway to **8** from **1** and Ph$_2$CO is as represented in Scheme 2. By analogy with earlier studies involving related compounds, the bicyclic molecules **5** or **6** might have been expected to be stable. It had been shown that (i) the transient silylene SiAr$_2$ and Ph$_2$CO gave a siloxaindane, the SiAr$_2$ analogue of the Si(NN) compound **6** [Ar = 2-(CH$_2$-NMe$_2$)C$_6$H$_4$],[13] and (ii) Si(C$_5$Me$_5$)$_2$ and Ph$_2$CO gave an

Table 2. Selected Bond Lengths (Å) and Angles (deg) for 8

Si(1)−O	1.624(8)	Si(1)−N(1)	1.740(9)
Si(2)−O	1.632(8)	Si(1)−N(2)	1.735(9)
Si(2)−C(23)	1.903(11)	Si(2)−N(4)	1.711(9)
C(18)−C(23)	1.53(2)	N(3)−C(23)	1.535(13)
C(17)−C(18)	1.41(2)	C(23)−C(24)	1.52(2)
S(1)−C(17)	1.854(12)		

C(18)−C(17)−Si(1)	129.0(9)	N(2)−Si(1)−N(1)	93.1(4)
C(17)−C(18)−C(23)	122.4(11)	C(30)−N(3)−C(36)	117.4(9)
C(18)−C(23)−Si(2)	117.0(8)	C(30)−N(3)−C(23)	110.1(8)
Si(1)−O−Si(2)	130.9(5)	C(36)−N(3)−C(23)	119.3(8)
O−Si(2)−C(23)	109.7(4)	C(31)−N(4)−C(41)	120.2(9)
O−Si(1)−C(17)	105.3(5)	C(31)−N(4)−Si(2)	117.6(7)
		C(41)−N(4)−Si(2)	120.8(8)

Scheme 2
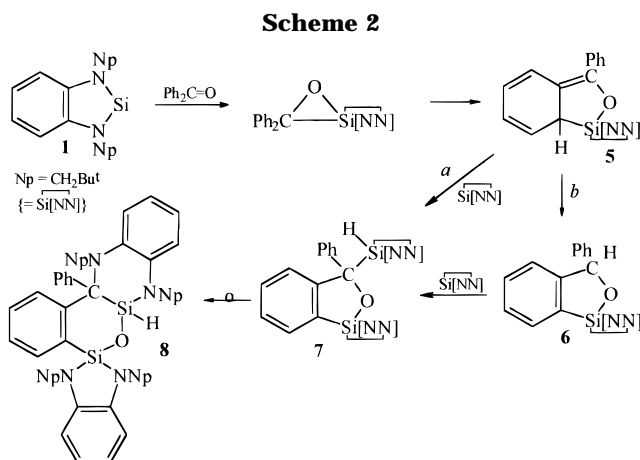
oxasilacyclopentene, the Si(C₅Me₅)₂ analog of the Si(NN) compound **5**.[14] Thus, **5** and **6** are plausible reaction intermediates to **8**, but the failure to isolate such a compound may be due to the facile reaction between **1** and **5** or **6** (paths *a* or *b*, respectively in Scheme 2) to give **7**. The driving force for the ultimate proposed rearrangement step **7** → **8** may well be the oxophilicity of silicon. Heating compound **2** did not yield **8**; hence **2** is an unlikely intermediate in the reaction between **1** and Ph₂CO → **8**.

Scheme 3

Treatment of the stable bis(amino)silylene **1** with a cinnamyl ketone or benzil gave in good yield a 1,4-cycloadduct, the pale yellow oxasilacyclopentene (**9** or **10**) or dioxasilacyclopentene (**11**), **9** and **11** as crystals and **10** as a distillable, viscous liquid, Scheme 3. These reactions are analogous to those observed with the transient silylene SiAr₂[13] and SiMe(OMe).[15]

Experimental Section

General Considerations. The NMR spectra were recorded in [²H₆]benzene at 298 K using a Bruker AC-P 250 [at 250 MHz (¹H) or 62.86 MHz (¹³C)], a Bruker DPX 300 [at 300 MHz (¹H) or 75.48 MHz (¹³C)], or a Bruker AMX 500 [at 500 MHz (¹H) or 99.33 MHz (²⁹Si)] 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 2. Ph₂C=O (0.2 g, 1.1 mmol) was added to a solution of **1** (0.3 g, 1.1 mmol) in benzene (20 mL). After the solution was stirred for 16 h at room temperature, the solvent was evaporated under vacuum. The remaining solid was redissolved in a hexane/toluene mixture to give **2** (0.35 g, 87%) at −25 °C as colorless crystals, mp 209−210 °C. Anal. Calcd for C₄₅H₆₂N₄OSi₂: C, 73.9; H, 8.55; N, 7.66. Found: C, 73.6; H, 8.67; N, 7.52. ¹H NMR (500 MHz): δ = 0.8, 1.13 (CMe₃, 2 s, 36H), 2.43, 2.94, 3.13, 3.50 (CH₂, 4 s, AB-type, 8H), 6.7−7.5 (phenyl, m, 18H). ¹³C NMR (62.86 MHz): δ = 28.73, 29.64

Table 3. Details of the X-ray Structure Determinations of 2 and 8

	2	**8**
empirical formula	C₄₅H₆₂N₄OSi₂	C₄₅H₆₂N₄OSi₂
fw	731.2	731.2
temp (K)	293(2)	173(2)
wavelength (Å)	0.710 73	0.710 73
cryst system	monoclinic	triclinic
space group	*P*2₁/*c* (No.14)	*P*1̄ (No. 2)
a (Å)	20.075(11)	9.783(4)
b (Å)	10.915(12)	10.178(6)
c (Å)	21.981(10)	21.552(11)
α (deg)	90	97.65(5)
β (deg)	115.98(4)	96.04(4)
γ (deg)	90	91.32(4)
V (Å³)	4330(6)	2114(2)
Z	4	2
D$_{(calcd)}$ (Mg/m³)	1.12	1.15
abs coeff (mm⁻¹)	0.12	0.12
F(000)	1584	792
cryst size (mm)	0.30 × 0.20 × 0.15	0.15 × 0.10 × 0.05
θ range for data collcn (deg)	2−25	2−22
index ranges	0 ≤ *h* ≤ 23, 0 ≤ *k* ≤ 12, −26 ≤ *l* ≤ 23	0 ≤ *h* ≤ 10, −10 ≤ k ≤ 10, −22 ≤ *l* ≤ 22
reflcns collcd	7800	5158
indepdt reflcns	7582 [*R*(int) = 0.0948]	5158
reflcns with *I* > 2σ(*I*)	3551	2291
data/restraints/params	7578/0/469	5141/0/469
goodness-of-fit on *F*²	1.042	1.110
final *R* indices [*I* > 2σ(*I*)]	*R*1 = 0.089, w*R*2 = 0.172	*R*1 = 0.109, w*R*2 = 0.246
R indices (all data)	*R*1 = 0.198, w*R*2 = 0.229	*R*1 = 0.235, w*R*2 = 0.344
largest diff peak and hole (e Å⁻³)	0.31 and −0.27	0.45 and −0.45

(CMe_3), 35.08, 35.16 (CMe_3), 53.55, 53.58 (CH$_2$), 110.75, 110.99, 118.12, 118.17, 126.38, 128.29, 138.21, 139.59, 145.05 (phenyl). ^{29}Si NMR: δ = 9.66, 21.96. MS [m/z (%)]: 730 (7), [M]$^+$.

Synthesis of 3. Bu^t(Me)C=O (0.25 mL, 1.825 mmol) was added to a solution of **1** (0.5 g, 1.825 mmol) in benzene (40 mL). After the solution was stirred for 16 h, the solvent was removed under vacuum and the remaining solid was recrystallized from *n*-hexane at −25 °C yielding **3** (0.51 g, 86%) as pale yellow crystals, mp 136−137 °C. Anal. Calcd for C$_{38}$H$_{64}$Si$_2$-ON$_4$: C, 70.3; H, 9.94; N, 8.63. Found: C, 70.3; H, 9.98; N, 8.68. ^{1}H NMR (250 MHz): δ = 0.83, 0.89, 0.94, 1.05, and 1.10 (CMe_3, 4 s, 45H), 1.33 (CH$_3$, s, 3H), 3.09, 3.15, 3.22, 3.25, 3.28, 3.31, 3.48, 3.53, 3.57, 3.59, 3.60, 3.62, and 3.66 (CH$_2$, AB-type, 8H), 6.75−6.82 (phenyl, m, 8H). ^{13}C NMR (62.86 MHz): δ = 26.86, 28.79, 28.82, 29.34, and 29.47 (CMe_3), 35.31, 35.37, 35.77, and 37.12 (CMe_3), 53.42, 53.71, 54.90, and 55.41 (CH$_2$), 22.62 (CH$_3$), 109.66, 110.48, 110.94, 111.74, 117.59, 117.62, 117.99, 118.02, 138.31, 139.12, 139.48, and 140.15 (phenyl). ^{29}Si NMR: δ = 6.44 and 19.97. MS [m/z (%)]: 648 (12), [M]$^+$.

Synthesis of 4. Adamantan-2-one (0.15 g, 0.985 mmol) was added to a solution of **1** (0.54 g, 1.97 mmol) in benzene (30 mL), and the mixture was stirred for 16 h at room temperature. After removal of the solvent under vacuum, the remaining solid was recrystallized from *n*-hexane at −25 °C to afford **4** (0.51 g, 74.1%) as a pale yellow microcrystalline solid, mp 269−270 °C. Anal. Calcd for C$_{42}$H$_{67}$N$_4$OSi$_2$: C, 72.0; H, 9.64; N, 8.00. Found: C, 72.2; H, 9.44; N, 8.06. ^{1}H NMR (300 MHz): δ = 0.91 and 1.04 (CMe_3, 2 s, 36H), 3.07, 3.11, 3.18, 3.22, 3.50, 3.55, 3.58, and 3.63 (CH$_2$, AB-type, 8H), 6.68−6.80 (phenyl, m, 8H). ^{13}C NMR (75.48 MHz): δ = 28.82 and 29.55 (CMe_3), 35.00 and 35.88 (CMe_3), 54.86 and 53.88 (CH$_2$), 26.8, 32.7, 37.89, 87.66 (adamantyl), 109.88, 110.61, 117.74, 117.86, 138.89, and 139.81 (phenyl). ^{29}Si NMR: δ = 5.03 and 21.04. MS [m/z (%)]: 699 (26), [M]$^+$.

Synthesis of 8. A solution of **1** (0.4 g, 1.46 mmol) in benzene (30 mL) was added slowly to a refluxing solution of Ph$_2$C=O (0.27 g, 1.46 mmol) in benzene (20 mL). After 1 h of additional heating, the solvent was removed under vacuum. The residue was dissolved in *n*-hexane and cooled to −25 °C to give **8** (0.4 g, 75%) as a colorless microcrystalline solid, mp 202−203 °C. X-ray single crystals have been obtained by recrystallization from hexane/toluene. Anal. Calcd for C$_{45}$H$_{62}$N$_4$OSi$_2$: C, 73.9; H, 8.55; N, 7.66. Found: C, 73.1; H, 8.32; N, 7.82. ^{1}H NMR (500 MHz): δ = 0.65, 0.86, 0.89, and 1.01 (CMe_3, 4 s, 36H), 2.39, 2.42, 2.73, 2.76, 3.05, 3.08, 3.13, 3.16, 3.17, 3.20, 3.21, 3.24, 3.72, 3.75, 3.77, and 3.80 (CH$_2$, AB-type, 8H), 5.69 (SiH, s, 1H, $^1J_{SiH}$ 271.60 Hz), 6.57−7.55 (phenyl, m, 17H), 6.4 and 8.3 (phenyl, br s {at −45 °C in [^{2}H$_8$]toluene}, 6.36 and 8.43, 2 d). ^{13}C NMR (75.48 MHz): δ = 28.49, 28.91, 29.08, and 30.20 (CMe_3), 34.01, 34.08, 34.54, and 35.47 (CMe_3), 53.70, 54.14, 55.59, and 56.81 (CH$_2$), 65.08 (PhC), 108.99, 109.18, 116.79, 117.76, 117.88, 118.85, 122.08, 124.58, 126.07, 126.71, 127.38, 130.56, 131.05, 131.56, 136.14, 138.92, 140.49, 140.63, 141.13, 142.87, and 151.7 (phenyl). ^{29}Si NMR: δ = −15.45 ($^1J_{SiH}$ 272.17) and −28.18. MS [m/z (%)]: 730 (14), [M]$^+$.

Synthesis of 9. PhCH=CHC(O)Ph (0.45 g, 2.15 mmol) was added to a solution of **1** (0.5 g, 2.15 mmol) in benzene (30 mL). The mixture was stirred at room temperature for 16 h. The solvent was removed under vacuum, and the remaining solid was recrystallized from *n*-hexane at −25 °C to afford **9** (0.73 g, 70.2%) as a pale yellow, microcrystalline solid, mp 158−159 °C. Anal. Calcd for C$_{31}$H$_{38}$SiN$_2$O: C, 77.1; H, 7.93; N, 5.80. Found: C, 77.6; H, 8.05; N, 5.74. ^{1}H NMR (300 MHz): δ 0.93 and 1.06 (CMe$_3$, 2s, 18 H), 2.04, 2.09, 2.93, 2.98, 3.15,

3.19, 3.52, and 3.57 (CH$_2$, AB-type, 4H), 3.68 (PhCH, d, 1H, ^3J = 3.26 Hz), 5.95 (=CH, d, 1H, ^3J = 3.24 Hz), 6.84−7.33 and 7.91−7.94 (phenyl, m, 9H). ^{13}C NMR (75.48 MHz): δ 28.43 and 28.59 (CMe_3), 33.63 and 34.03 (CMe_3), 54.09 and 55.82 (CH$_2$), 30.48 (CHPh), 101.93 (=CH), 155.33 (=CO), 109.48, 109.55, 118.33, 118.40, 139.88, 139.69 and 125.16, 125.65, 127.15, 128.68, 128.92, 128.96, 133.63, 139.49 (phenyl). ^{29}Si NMR: δ −5.76. MS [m/z (%)]: 482 (32), [M]$^+$.

Synthesis of 10. PhCH=CHC(O)CH$_3$ (0.33 g, 2.29 mmol) was added to a solution of **1** (0.63 g, 2.29 mmol) in benzene (30 mL). The mixture was stirred for 16 h at room temperature. The solvent was removed under vacuum, and the remaining residue was distilled to give **10** (0.82 g, 85%) as a pale yellow, viscous oil, bp 140−150 °C, 0.05 mmHg. Anal. Calcd for C$_{26}$H$_{35}$SiON$_2$: C, 74.4; H, 8.41; N, 6.67. Found: C, 73.5; H, 8.70; N, 6.48. ^{1}H NMR (300 MHz): δ 0.82 and 1.00 (CMe$_3$, 2s, 18H), 1.85, 1.90, 2.76, 2.81, 2.99, 3.04, 3.38, and 3.42 (CH$_2$, AB-type, 4H), 1.83 (CH$_3$, t, 3H), 3.35 (PhCH, m, H), 4.99 (CH, m, H) and 6.67−7.07 (Ph, m, 10H). ^{13}C NMR (75.48 MHz): δ 28.55 and 28.37 (CMe_3), 33.60 and 34.04 (CMe_3), 54.03 and 55.78 (CH$_2$), 18.42 (CH$_3$), 30.09 (CHPh), 102.23 (=CH), 155.46 (CO), 109.31, 109.38, 118.18, 118.29, 139.96, 140.02 and 125.45, 126.98, 128.82, 139.52 (phenyl). ^{29}SiNMR: δ −5.33. MS [m/z (%)]: 420 (19), [M]$^+$.

Synthesis of 12. Benzil (0.46 g, 2.19 mmol) was added to a solution of **1** (0.6 g, 2.19 mmol) in benzene (30 mL), and the mixture was stirred for 16 h at room temperature. After removal of the solvent under vacuum, the resulting solid was recrystallized from *n*-hexane to afford **12** (0.8 g, 76%) as a pale yellow, microcrystalline compound, mp 165−167 °C. Anal. Calcd for C$_{30}$H$_{36}$SiON$_2$: C, 74.3; H, 7.49; N, 5.48. Found: C, 74.3; H, 7.29; N, 5.62. ^{1}H NMR (300 MHz): δ 0.91 (CMe$_3$, s, 18H), 3.23 (CH$_2$, s, 4H), 6.79−7.69 (phenyl, m, 14H). ^{13}C NMR (75.48 MHz): δ 27.97 (CMe_3), 33.61 (CMe_3), 55.20 (CH$_2$), 137.81 (C=C), 109.98, 119.01, 132.87 and 127.44, 128.67, 129.99, 132.87 (phenyl). ^{29}Si NMR: δ −32.25. MS [m/z (%)]: 484 (37), [M]$^+$.

X-ray Structure Determination of 2 and 8. Crystallographic data for compounds **2** and **8** are displayed in Table 3. Unique data sets were collected with an Enraf-Nonius CAD4 diffractometer, using a crystal sealed in a Lindemann capillary under argon at 293(2) K for **2**, or for **8**, the crystal was coated with a layer of hydrocarbon oil, attached to a glass fiber, and cooled to 173(2) K. Crystallographic programs used for structure solution and refinement were from SHELXS-86[22] and SHELXL-93.[23] Refinements were based on full-matrix least squares on all F^2. All non-H atoms were anisotropic, and the hydrogen atoms were included in a riding mode.

Supporting Information Available: Packing diagrams and tables of data collection and processing parameters, atomic coordinates and isotropic temperature factors, bond lengths and angles, anisotropic thermal parameters, and hydrogen atom coordinates and assigned isotropic temperature factors (20 pages). Ordering information is given on any current masthead page.

(22) Sheldrick, G. M. In *Crystallographic Computing 3*; Sheldrick, G. M., Krüger, C., Goddard, R., Eds.; Oxford University Press: Oxford, U.K.; 1985; p 175.

(23) Sheldrick, G. M. SHELX 93, University of Göttingen, 1993.