1472 *Organometallics* **2001,** *20,* 1472−1475

# *Notes*

## Generation and Reactivity of a New Heterocyclic Silylene, a Silaazaanthracene

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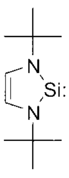
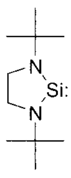
*Summary: The cyclic silylene **6**, a silaazaanthracene derivative, which possesses 14-electrons, has been generated by the reduction of 10,10-dichloro-5-methyl-5,10-dihydrophenazasiline (**5**) with lithium naphthalenide. Reactions of **6** with 2,3-dimethyl-1,3-butadiene, isoprene, and triethylvinylsilane gave the corresponding silylene-trapping products, **7**, **8**, and **9**, in moderate yield. Pseudoaromatic stabilization of the cyclic silylene **6** was not observed, possibly due to the fact that the three rings are not coplanar.*

### Introduction

A new research area in the study of silylenes began with the isolation of the first stable divalent silicon species, $Cp^*_2Si$: ($Cp^* = Me_5C_5$, silico*cene*), by Jutzi and co-workers.[1] Recently, a new class of stable silylenes, **1**−**3**, has been reported.[2−4] Although the flanking *tert*-butyl groups provide some kinetic and steric stabilization to the silylene, electronic factors arising from the pseudoaromaticity of the species or $\pi$-donation from nitrogen to silicon are primarily responsible for the stability of these systems. Quite recently, the first isolable dialkylsilylene, 2,2,5,5-tetrakis(trimethylsilyl)-silacyclopentane-1,1-diyl, **4**, which is stabilized sterically, has been reported.[5]



**1**



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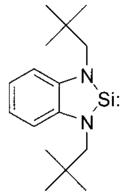

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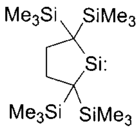

**4**

This prompted us to investigate whether silylene **6** as an anthracene analogue would be stabilized through a special pseudoaromatization with 14 electrons, 12 electrons from two phenyl rings and 2 electrons from a lone electron pair on the N atom. This paper describes our generation and the reactivity of silylene **6**, which contains the silaazaanthracene moiety. Unexpected results regarding stabilities were encountered (Scheme 1).

### Results and Discussion

The reaction of di(2-lithiophenyl)methylamine with $SiCl_4$ in $Et_2O$ gave 10,10-dichloro-5-methyl-5,10-dihydrophenazasiline, **5**, as colorless crystals.[6] The reduction[7,8] of **5** with LiNp (lithium naphthalenide), it was hoped, would lead to silylene **6**. When a LiNp solution was slowly added into **5** in THF at −78 °C, the dark purple LiNp color was discharged very rapidly, indicating that the reduction of **5** had occurred. Such a reaction of **5** with LiNp in the presence of an excess of 2,3-dimethyl-1,3-butadiene or isoprene at −78 °C yielded the corresponding silylene-trapping products, **7** (61%) and **8** (57%), as colorless crystals, respectively. Also, the trapping experiment in which an excess of triethylvinylsilane was used afforded colorless crystals of **9** (48%) (Scheme 2).

Products **7** and **8** could have been formed either via 1,4-cycloaddition or via concerted 1,2-addition of silylene **6** to one of the double bonds of the diene, in the latter case, producing an alkenylsilirane intermediate that

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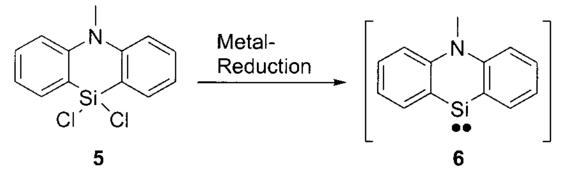
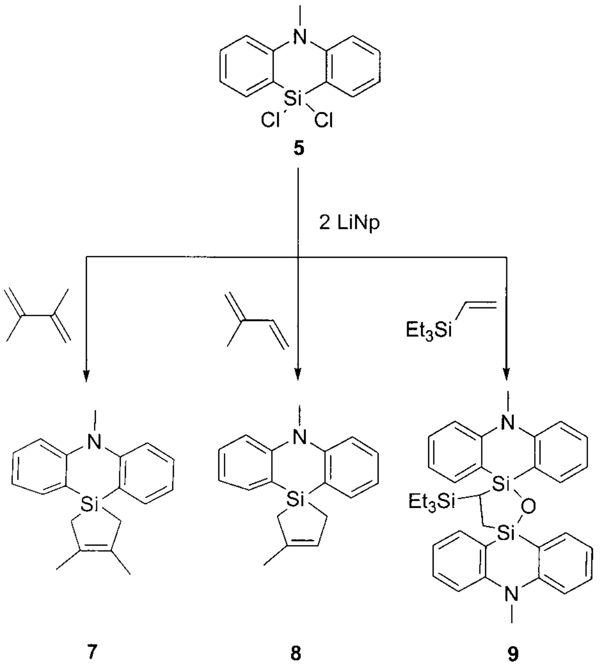
**Scheme 1**



**Scheme 2**



undergoes rearrangement to the observed products.[9] On the basis of studies by Gaspar and co-workers,[10] the latter is more likely. Unexpected product **9** with an Si−O−Si bond most likely was formed by silylene insertion into the Si−C bond of the strained, very reactive silacyclopropane initially formed in the **6**/$Et_3SiCH{=}CH_2$ reaction followed by oxidation by adventitious $O_2$ of the Si−Si bond of the 1,2-disilacyclobutane thus formed.[11] The Si−C bond of the silacyclopropane must be much more reactive toward the silylene than the double bond of triethylvinylsilane which was present in 10-fold exess.[12] The results of these trapping experiments suggest that the expected silylene **6** was indeed generated by the reduction of **5** with LiNp at low temperatures.

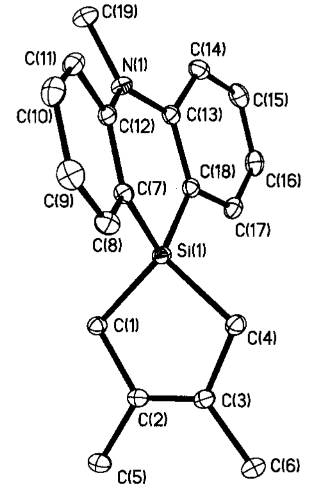


**Figure 1.** Structural drawings of **7** showing the atom-numbering scheme. Selected bond distances (Å) and angles (deg): Si(1)−C(18), 1.8476(14); Si(1)−C(7), 1.8521(14); Si(1)−C(4), 1.8758(14); Si(1)−C(1), 1.8885(14); N(1)−C(12), 1.4075(17); N(1)−C(13), 1.4106(17); N(1)−C(19), 1.4633(18); C(7)−C(12), 1.4138(18); C(13)−C(18), 1.4133(19); C(18)−Si(1)−C(7), 101.41(6); C(18)−Si(1)−C(4), 114.85(6); C(7)−Si(1)−C(1), 115.97(6); C(4)−Si(1)−C(1), 94.94(6); C(12)−N(1)−C(13), 125.13(11); C(12)−N(1)−C(19), 117.16(11); C(13)−N(1)−C(19), 117.39(11); N(1)−C(12)−C(11), 118.79(12); N(1)−C(12)−C(7), 122.59(12); N(1)−C(13)−C(14), 118.79(12); N(1)−C(13)−C(18), 122.63(12).

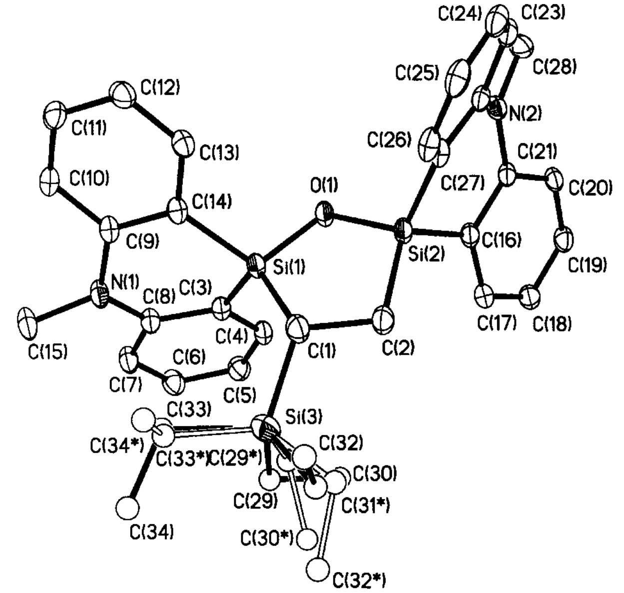


**Figure 2.** Structural drawings of **9** showing the atom-numbering scheme. Selected bond distances (Å) and angles (deg): Si(1)−O(1), 1.6572(18); Si(1)−C(14), 1.846(3); Si(1)−C(3), 1.847(3); Si(1)−C(1), 1.891(3); N(1)−C(8), 1.407(3); N(1)−C(9), 1.412(4); N(1)−C(15), 1.469(3); C(3)−C(8), 1.415(4); C(9)−C(14), 1.414(4); O(1)−Si(1)−C(14), 110.64(11); C(14)−Si(1)−C(3), 100.53(12); O(1)−Si(1)−C(1), 100.40(11); C(3)−Si(1)−C(1), 116.48(14); Si(1)−O(1)−Si(2), 118.20(11); C(8)−N(1)−C(9), 123.0(2); C(8)−N(1)−C(15), 118.1(2); C(9)−N(1)−C(15), 117.7(2).

The X-ray structures and selected bond lengths and bond angles of **7** and **9** are shown in Figures 1 and 2. Crystallographic data are given in Tables 1 and 2. In the structure of **7**, although the sum of the bond angles around N(1) is almost 360° (359.68°), the angle between the two C(11)−C(12)−C(7) and N(1)−C(13)−C(18) planes

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(11) (a) In the reaction of **5**/LiNp/$Et_3SiCH{=}CH_2$, oxygen was bubbled into the reaction mixture for 10 minutes before the workup. The yield of **9** was 52%, which supported the mechanism of oxidation of the Si−Si bond. (b) West, R. In *The Chemistry of Organic Silicon Compounds*; Patai, S., Rappoport, Z., Eds.; John Wiley & Sons: Chichester, U.K., 1989; Chapter 19. (c) Alanimi, I. S.; Weber, W. P. *Organometallics* **1983**, *2*, 903. (d) Carison, C. W.; West, R. *Organometallics* **1983**, *2*, 1798. (e) Kabe, Y.; Ohkubo, K.; Ishikawa, H.; Ando, W. *J. Am. Chem. Soc.* **2000**, *122*, 3775. (f) Seyferth, D.; Annarelli, D. C. *J. Am. Chem. Soc.* **1975**, *97*, 2273. (g) Seyferth, D.; Annarelli, D. C.; Vick, S. C.; Duncan, D. P. *J. Organomet. Chem.* **1980**, *201*, 179.

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**Table 1. Crystal Data and Structure Refinement for 7**

| | |
|---|---|
| empirical formula | $C_{19}H_{21}NSi$ |
| fw | 291.46 |
| temperature | 150(2) K |
| wavelength | 0.71073 Å |
| cryst syst | orthorhombic |
| space group | *Pbca* |
| unit cell dimens | $a = 14.5835(2)$ Å, $\alpha = 90°$<br>$b = 8.2666(4)$ Å, $\beta = 90°$<br>$c = 25.8717(8)$ Å, $\gamma = 90°$ |
| volume | 3118.99(18) Å$^3$ |
| *Z* | 8 |
| density (calcd) | 0.934 Mg/m$^3$ |
| abs coeff | 0.126 mm$^{-1}$ |
| *F*(000) | 960 |
| cryst size | $0.35 \times 0.35 \times 0.20$ mm$^3$ |
| $\theta$ range for data collection | 2.94−27.47° |
| index ranges | $0 \le h \le 18$, $0 \le k \le 10$, $-33 \le l \le 0$ |
| no. of reflns collected | 16 927 |
| no. of ind reflns | 3552 [*R*(int) = 0.0030] |
| completeness to $\theta = 27.47°$ | 99.5% |
| abs corr | multiscan (Denzo-SMN) |
| max. and min. transmn | 0.9752 and 0.9571 |
| refinement method | full-matrix least-squares on $F^2$ |
| no. of data/restraints/params | 3552/0/193 |
| goodness-of-fit on $F^2$ | 1.040 |
| final *R* indices [$I > 2\sigma(I)$] | R1 = 0.0383, wR2 = 0.0981 |
| *R* indices (all data) | R1 = 0.0468, wR2 = 0.1037 |

**Table 2. Crystal Data and Structure Refinement for 9**

| | |
|---|---|
| empirical formula | $C_{34}H_{40}N_2OSi_3$ |
| fw | 576.95 |
| temperature | 100(1) K |
| wavelength | 0.71073 Å |
| cryst syst | rhombohedral |
| space group | $R\bar{3}$ |
| unit cell dimens | $a = 36.9229(8)$ Å, $\alpha = 90°$<br>$b = 36.9229(8)$ Å, $\beta = 90°$<br>$c = 11.8596(4)$ Å, $\gamma = 120°$ |
| volume | 14002.1(6) Å$^3$ |
| *Z* | 18 |
| density (calcd) | 1.232 Mg/m$^3$ |
| abs coeff | 0.182 mm$^{-1}$ |
| *F*(000) | 5544 |
| cryst size | $0.35 \times 0.25 \times 0.20$ mm$^3$ |
| $\theta$ range for data collection | 2.87−27.48° |
| index ranges | $0 \le h \le 47$, $-40 \le k \le 0$, $-15 \le l \le 15$ |
| no. of reflns collected | 21 779 |
| no. of ind reflns | 7121 [*R*(int) = 0.027] |
| completeness to $\theta = 27.48°$ | 99.8% |
| abs corr | multiscan |
| max. and min. transmn | 0.9645 and 0.9390 |
| refinement method | full-matrix least-squares on $F^2$ |
| no. of data/restraints/params | 7121/18/385 |
| goodness-of-fit on $F^2$ | 1.025 |
| final *R* indices [$I > 2\sigma(I)$] | R1 = 0.0641, wR2 = 0.1470 |
| *R* indices (all data) | R1 = 0.0982, wR2 = 0.1652 |

is large, 22.89°, indicating a lack of planarity among the three rings. As expected, the angle between the two C(7)−Si(1)−C(18) and C(1)−Si(1)−C(4) planes is nearly 90° (89.90°), which shows a spiro type structure. The nitrogen atom is situated at a distance of 3.15 Å from the silicon atom, which is somewhat larger than the normal coordination distance of 2.8 Å but shorter than the sum of the van der Waals radii of 3.60 Å.[13] The silaazaanthracene skeleton of **9** is similar to those of **7**. In the five-membered ring in **9**, O(1), Si(1), and Si(2) form a plane and C(1) and C(2) are 0.136(5) Å below and 0.217(5) Å above the plane, respectively. The Si−O bond distance and the Si(1)−O(1)−Si(2) bond angle are 1.6572(18) Å and 118.20(11)°, respectively.

In these trapping experiments, we did not observe any disilene-trapping products. We obtained only unidentified high-boiling materials when the **5**/LiNp reaction was carried out in the absence of a trapping agent, which suggests that silylene **6** is not stable. To study the stability of silylene **6**, we attempted to observe the silylene absorption band at −78 °C using UV−vis spectrometry. However, we did not observe any absorption band for the cyclic silylene, only absorption bands arising from LiNp and the silaazaanthracene moiety. When trapping agents such as MeOH, EtOH, or butadienes were added after the **5**/LiNp reaction had occurred at −78 °C, the corresponding silylene-trapping products were not obtained. The fact that silylene **6** could only be trapped in situ suggests that it is not stable even at −78 °C. We suggest that there might be no aromatic delocalization of 14 electrons in silylene **6** due to the lack of coplanarity among the three rings of the anthracene derivative.

## Experimental Section

In all reactions where air-sensitive chemicals were used, the reagents and solvents were dried prior to use. THF, ether, and hexane were distilled from Na/$Ph_2CO$. Other starting materials were purchased in reagent grade and used without further purification. Glassware was dried by flame under nitrogen or argon flushing prior to use. Experiments were performed under nitrogen or argon atmosphere. $^1H$ and $^{13}C$ NMR spectra were recorded on a Bruker DPX 250 FT-NMR spectrometer and Bruker AMX 500 NMR spectrometer and referenced to residual protons of the solvent with chemical shifts being reported as $\delta$ ppm. Analyses of product mixtures were accomplished using a HP 5890 II with FID (HP-1, 15 m column) and with TCD (OV-1, 1/8 in. 6 ft column). Mass spectra were recorded on a low-resolution Shimadzu GCMS QP-2000A mass spectrometer and a high-resolution VG ANALITICLA 70-VSEQ mass spectrometer.

**Synthesis of 10,10-Dichloro-5-methyl-5,10-dihydrophenazasiline, 5.** To a solution of 8.6 g (0.047 mol) of methyldiphenylamine and TMEDA (tetramethylethylenediamine) (14.3 mL, 0.094 mol) in hexane was added slowly 58.7 mL (0.094 mol) of *n*-BuLi diluted to 1.6 M in hexane at room temperature. After the mixture had been heated at reflux for 4 h and the liquid portion was removed by filtration, di(2-lithiophenyl)methylamine, a white solid, was obtained. A solution of the latter in $Et_2O$ was cooled at −78 °C, and 4.4 mL (0.038 mol) of $SiCl_4$ was added slowly. The reaction mixture was allowed to warm slowly to room temperature and stirred for 12 h, whereupon the precipitated LiCl was removed by filtration and volatiles were distilled under vacuum. The crude reaction product was dissolved in $Et_2O$. Cooling to −10 °C gave colorless crystals of **5** (8.5 g, 65%). MS: *m*/*z* (relative intensity) 279 ($M^+$, 44.2), 264 (0.9), 215 (5.3), 181 (100.0), 166 (17.3), 139 (10.7), 63 (12.6). Mp: 113−115 °C. $^1H$ NMR ($CDCl_3$, 250 MHz): $\delta$ 3.67 (s, 3H), 7.17−7.92 (m, 8H). $^{13}C$ NMR ($CDCl_3$, 63 MHz): $\delta$ 38.78 (NMe), 116.11, 117.22, 120.81, 132.63, 133.37, 150.32 (aryl). HRMS: $C_{13}H_{11}Cl_2NSi$ 279.0038 (calcd), 279.0040 (found).

**Reaction of LiNp with 5 in the Presence of an Excess of 2,3-Dimethyl-1,3-butadiene.** To a stirred solution of 1.4 g (5.0 mmol) of **5** and 5.7 mL (50 mmol) of 2,3-dimethyl-1,3-butadiene in THF at −78 °C was added dropwise a LiNp solution (10 mmol) in THF. The reaction mixture was allowed to warm slowly to room temperature and stirred for 4 h. After

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[13] Corriu, R. J. P.; Young, J. C. In *the Chemistry of Organic Silicon Compounds*; Patai, S., Rappoport, Z., Eds.; John Wiley & Sons: Chichester, U.K., 1989; Chapter 20.

filtration of the precipitated LiCl, volatiles were distilled under vacuum. The residue was recrystallized from $Et_2O$ to give **7** as colorless crystals (0.89 g, 61%). MS: *m*/*z* (relative intensity) 291 ($M^+$, 100.0), 276 (7.7), 209(36.1), 194 (17.3), 181 (84.1), 132 (9.6), 110 (12.3). $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 1.69 (s, 4H), 1.75 (s, 6H), 3.48 (s, 3H), 6.91−7.47 (m, 8H). $^{13}C$ NMR ($CDCl_3$, 126 MHz): $\delta$ 19.55 ($CH_2$), 25.53 ($CH_3$), 38.88 (NMe), 115.54, 120.50, 121.30, 130.79, 131.10, 134.24, 151.59 (olefin and aryl). HRMS: $C_{19}H_{21}NSi$ 291.1443 (calcd), 291.1441 (found). Anal. Calcd for $C_{19}H_{21}NSi$: C, 78.30; H, 7.26; N, 4.81. Found: C, 78.38; H, 6.73; N, 4.45.

**Reaction of LiNp with 5 in the Presence of an Excess of Isoprene or an Excess of Triethylvinylsilane.** Using a procedure similar to that described for the reaction of **5** with 2,3-dimethyl-1,3-butadiene, **5** (3.4 g, 12 mmol) in the presence of an excess of isoprene (12.1 mL, 120 mmol) was treated with LiNp (24 mmol) in THF at −78 °C. The crude product was recrystallized from $Et_2O$ to give **8** as colorless crystals (1.9 g, 57%) MS: *m*/*z* (relative intensity) 277 ($M^+$, 58.8), 262 (1.4), 209(18.5), 181 (100), 155 (3.3), 105 (7.1). $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 1.79 (s, 2H), 1.88 (br, 2H), 2.14 (s, 3H), 5.83 (br, 1H), 7.06−7.68 (m, 8H). $^{13}C$ NMR ($CDCl_3$, 126 MHz): $\delta$ 18.13 ($SiCH_2C$), 22.08 ($CCH_3$, $SiCH_2CH$), 37.96 (NMe), 114.68−150.55 (olefin and aryl). HRMS: $C_{18}H_{19}NSi$ 277.1287 (calcd), 277.1286 (found). Anal. Calcd for $C_{18}H_{19}NSi$: C, 77.93; H, 6.90; N, 5.05. Found: C, 77.91; H, 6.84; N, 4.86.

**5** (1.4 g, 5.0 mmol) in the presence of an excess of triethylvinylsilane (7.81 g, 55 mmol) was treated with LiNp (10 mmol) in THF at −78 °C. The crude product was recrystallized from $Et_2O$ to give **9** as colorless crystals (0.69 g, 48%, based on **5**). MS: *m*/*z* (relative intensity) 576 ($M^+$, 100.0), 547 (63.8), 461 (79.0), 434 (12.5), 238 (24.0), 212 (22.9), 181 (10.3). Mp: 212−218 °C. $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 0.387 (m, 6H), 0.714 (t, 9H), 0.947 (m, 2H), 1.262 (t, 1H), 3.580 (s, 3H), 3.631 (s, 3H), 6.978−7.886 (m, 16H). $^{13}C$ NMR ($CDCl_3$, 126 MHz): $\delta$ 3.71 ($SiCH_2CH_3$), 7.85 ($SiCH_2CH_3$), 9.07 ($SiCH_2CH$), 10.80 ($SiCH_2CH$), 38.62, 38.89 (NMe), 115.36−152.32 (aryl). HRMS: $C_{34}H_{40}N_2OSi_3$ 576.2448 (calcd), 576.2439 (found). Anal. Calcd for $C_{34}H_{40}N_2OSi_3$: C, 70.78; H, 6.99; N, 4.86. Found: C, 70.29; H, 6.81; N, 4.57.

**X-ray Structural Characterization.** A summary of selected crystallographic data is given in Tables 1 and 2. Data were collected on a Nonius KappaCCD diffractometer using graphite-monochromated Mo K radiation ($\lambda = 0.71073$ Å). A combination of 1° $\varphi$ and $\omega$ (with $\kappa$ offsets) scans were used to collect sufficient data. The data frames were integrated and scaled using the Denzo-SMN package. The structures were solved and refined using the SHELXTL V5.1 package. Refinement was by full-matrix least-squares on $F^2$ using all data (negative intensities included). Hydrogen atoms were included in calculated positions.

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**Supporting Information Available:** X-ray diffraction data for **7** and **9**. This material is available free of charge via the Internet at http://pubs.acs.org.

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