

Table I

| organic monomer  | platinum monomer  | trans/cis          |
|--|---|--------------------|
| $\text{BrCH}_2\text{CH}_2\text{O}_2\text{CC}(\text{Me})=\text{CH}_2$ | $[\text{PtBrMe}_2(\text{CH}_2\text{CH}_2\text{O}_2\text{CC}(\text{Me})=\text{CH}_2)(\text{bpy})]$ | 60/40 <sup>a</sup> |
| $\text{BrCH}_2\text{C}(\text{CO}_2\text{Me})=\text{CH}_2$            | $[\text{PtBrMe}_2(\text{CH}_2\text{C}(\text{CO}_2\text{Me})=\text{CH}_2)(\text{bpy})]$            | 95/5               |
| $3-(\text{ClCH}_2)\text{C}_6\text{H}_4\text{CH}=\text{CH}_2^b$       | $[\text{PtClMe}_2(3-\text{CH}_2\text{C}_6\text{H}_4\text{CH}=\text{CH}_2)(\text{bpy})]$           | 99/1               |
| $\text{ClCOCH}=\text{CH}_2$  | $[\text{PtClMe}_2(\text{COCH}=\text{CH}_2)(\text{bpy})]$  | 75/25              |

<sup>a</sup> Isomers correspond to the geometry at platinum (see text) and can be separated by recrystallization. The isomer ratio was determined by <sup>1</sup>H NMR spectroscopy. <sup>b</sup> Monomer contains 30% 4-(ClCH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>CH=CH<sub>2</sub>.

ferential scanning calorimetry. However, the polymers 3 and 5 exhibit a broad endothermic transition over the range 60–160 °C, with the peak at 115 °C, and the melting endotherm begins at 210 °C and peaks at 245 °C. When samples of polymers 3 and 5 were heated to 150 °C for 30 min and then cooled and reheated, the broad endotherm in the region 60–160 °C was not observed. Thus, it appears that polymers 3 and 5 precipitate in a strained conformation which relaxes on heating. Polymer 4 undergoes complete weight loss over the temperature range 335–395 °C, but 3 decomposes to leave a residue of platinum over the much wider temperature range 270–450 °C.

The synthetic method outlined above is generally applicable, so long as the organic monomer contains both a functional group which can take part in polymerization and a carbon–bromine or carbon–chlorine bond which can undergo oxidative addition. Some vinyl monomers and their products of oxidative addition with [PtMe<sub>2</sub>(2,2'-bipyridine)] are given in Table I. In all cases, the oxidative addition occurred with mostly trans stereochemistry and, in addition, platinum-containing polymers could be syn-

thesized by oxidative addition to the polymerized organic monomers. It is not necessary for the organic polymer to be soluble in organic solvents in order for oxidative addition to occur. Thus, Merrifield's resin, which is a cross-linked styrene(chloromethyl)styrene copolymer (1% cross-linked with divinylbenzene, with 1 mequiv of Cl/g of polymer) reacted, as a suspension in acetone, with excess [PtMe<sub>2</sub>(bpy)] to give the platinum-containing polymer in which essentially all of the chloromethyl groups were reacted.<sup>6</sup> All of the platinum-containing polymers described above are very sparingly soluble in organic solvents, even when the extent of platinum incorporation is <10% of the available functional groups. However, the corresponding polymers with 4,4'-di-*tert*-butyl-2,2'-bipyridine have sufficient solubility for solution characterization,<sup>5</sup> and efforts are being made to prepare derivatives with more highly alkylated bipyridine derivatives in order to prepare still more soluble polymers.

A major advantage of the oxidative-addition route to metal-containing monomers is the ease of reaction and the wide range of functional groups which can be incorporated. This work has focused on vinyl monomers, but platinum-containing monomers with ester, nitrile, amide, and carboxylic acid functionalities, suitable for studies of condensation polymerization, have also been synthesized by this method.

**Acknowledgment.** We thank the NSERC (Canada) for financial support.

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(6) Anal. Calcd for C<sub>9</sub>H<sub>8</sub>·0.15C<sub>21</sub>H<sub>22</sub>ClN<sub>2</sub>Pt: C, 74.4; H, 5.9; N, 2.1. Found: C, 72.9; H, 6.6; N, 2.1.

## Novel Route to C=Si Double Bonds via a Peterson-Type Reaction<sup>1</sup>

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**Summary:** Reaction of (Me<sub>3</sub>Si)<sub>3</sub>SiLi·3THF (1) with adamantane in hexane or benzene leads via a Peterson-type elimination reaction to the silene bis(trimethylsilyl)adamantylidenesilane (3), which spontaneously dimerizes in a head-to-head fashion (80–90% yield) to 1,1,2,2-tetrakis(trimethylsilyl)dispiro[3,3',4,4'-adamantane-1,2-disilacyclobutane] (2). When it is warmed, 2 reverts to 3, which was trapped by 1-methoxybutadiene or methanol. The X-ray structure of 2 is reported. The four-membered ring is strongly puckered and has unusually long C–C and C–Si bond lengths of 1.647 and 2.008 Å, respectively. Reaction of 1 with 4-*tert*-butylcyclohexanone also proceeds via the corresponding silene, which dimerizes to 1,1,2,2-tetrakis(trimethylsilyl)-1-(4-*tert*-butylcyclohexyl)-2-(4-*tert*-butylcyclohexenyl)disilane.

Silicon chemistry is one of the fastest growing fields of organic chemistry.<sup>2</sup> Of special interest are compounds

with multiple bonds to silicon,<sup>3</sup> of which we focus here on silenes, i.e. R<sub>2</sub>C=SiR<sub>2</sub>. Compelling evidence that a silene might exist as a transient intermediate was first presented only in 1967.<sup>4</sup> The first "indefinitely" stable silene was isolated by Brook et al. in 1981,<sup>5a</sup> and this was followed by two X-ray structure determinations<sup>5b,c</sup> and intensive research on silenes which led to many important developments.<sup>3</sup> Yet, the arsenal of methods available for synthesizing silenes is limited.<sup>3</sup> We now report that the reaction of (Me<sub>3</sub>Si)<sub>3</sub>SiLi·3THF (1)<sup>6</sup> with certain ketones

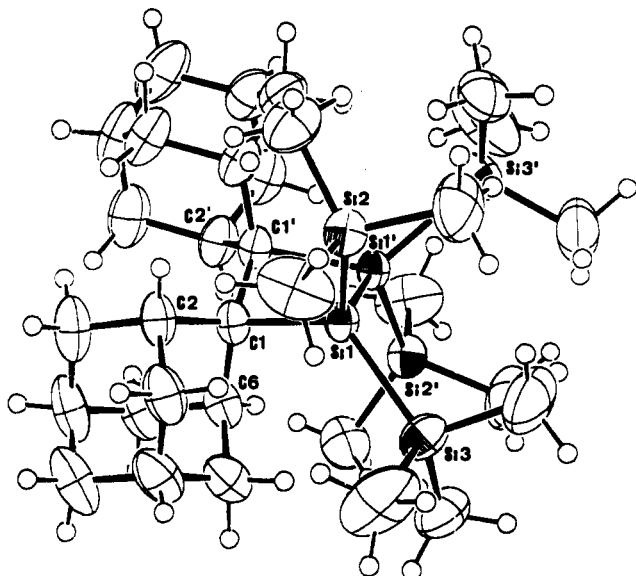
(2) For a comprehensive review, see: *The Chemistry of Organosilicon Compounds*; Patai, S., Rappoport, Z. Eds.; Wiley: Chichester, U.K., 1989.

(3) For reviews see: (a) Raabe, G.; Michl, J. In Ref 2, Chapter 17. (b) Raabe, G.; Michl, J. *Chem. Rev.* 1985, 25, 419. (c) West, R. *Angew. Chem., Int. Ed. Engl.* 1987, 26, 1201. (d) Wiberg, N. *J. Organomet. Chem.* 1984, 273, 141. (e) Gusel'nikov, L. E.; Avakyan, V. G. *Sov. Sci. Rev., Sect. B Chem.* 1989, 13, 39.

(4) Gusel'nikov, L. E.; Flowers, M. C. *Chem. Commun.* 1967, 864.

(5) (a) Brook, A. G.; Abdesaken, F.; Gutekunst, B.; Gutekunst, G.; Kallury, R. K. M. *J. Chem. Soc., Chem. Commun.* 1981, 191. (b) Brook, A. G.; Nyburg, S. C.; Abdesaken, F.; Gutekunst, B.; Gutekunst, G.; Kallury, R. K. M.; Poon, Y. C.; Chang, Y.-M.; Wong-Ng, W. *J. Am. Chem. Soc.* 1982, 104, 5667. (c) Wiberg, N.; Wagner, G.; Müller, G. *Angew. Chem., Int. Ed. Engl.* 1985, 24, 229. Wiberg, N.; Wagner, G. *Chem. Ber.* 1986, 119, 1467.

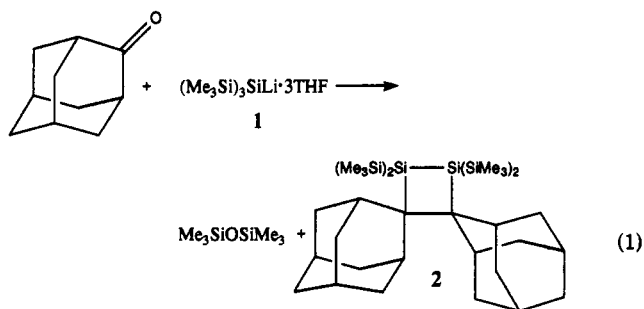
(1) Reported in part at the European Symposium of Organic Reactivity (ESOR III), Gotheburg, Sweden, July 7–12, 1991, proceedings p 124.



**Figure 1.** ORTEP drawing of **2**.<sup>9</sup> Selected bond lengths (Å) and bond angles (deg): C1–C1' = 1.647 (5), Si1–Si1' = 2.333 (1), C1–Si1 = 2.008 (3), C1–C2 = 1.568 (5), Si1–Si2 = 2.422 (1), Si1–Si3 = 2.383 (1); C1'–C1–Si1 = 95.8 (2), C1–Si1–Si1' = 77.3 (1), Si2–Si1–Si1' = 121.20 (5), Si3–Si1–Si1' = 115.55 (5), Si2–Si1–C1 = 114.3 (1), Si3–Si1–C1 = 128.2 (1), Si2–Si1–Si3 = 101.13 (5), Si1–C1–C2 = 110.6 (2), C2–C1–C6 = 104.8 (3), C1–C1'–Si1'–Si1 = –28.0 (2), C1'–Si1'–Si1–C1 = 23.1 (2), Si3–Si1–C1–C1' = –140.6 (2), Si3–Si1–Si1'–Si3' = –83.9 (7), Si2–Si1–Si1'–Si2' = 161.36 (6), Si2–Si1–C1–C2 = –36.7 (3), C2–C1–C1'–C2' = –90.3 (5). The angle between the planes defined by C1–Si1–Si1' (P1) and C1–C1'–Si1' (P2) is 39.6°.

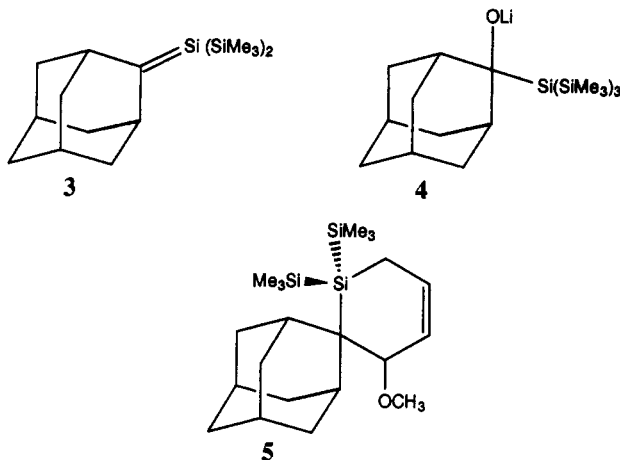
proceeds via a Peterson-type process, providing a new route for generating silenes.<sup>7</sup>

Reaction of adamantanone with an equimolar amount of **1**<sup>6</sup> in hexane (or benzene) gave an 85% yield of 1,2-disilacyclobutane **2** (eq 1).<sup>8</sup> The structure of **2** was as-



signed by X-ray analysis (Figure 1).<sup>9</sup> The observed

product is best interpreted in terms of the intermediacy of the silene **3**. Addition of **1** across the C=O bond of adamantanone presumably produces the alcoholate **4** (not isolated), which spontaneously eliminates Me<sub>3</sub>SiOLi<sup>10</sup> to yield **3**, which dimerizes in a head-to-head (HH) fashion to **2**.



Further evidence for the intermediacy of **3** is provided by trapping experiments.<sup>3</sup> For example, reaction in the presence of excess butadiene or 1-methoxybutadiene produces the expected<sup>3</sup> Diels–Alder-type products<sup>11a</sup> in 70–80% yield. The reaction with 1-methoxybutadiene is highly regioselective, giving only **5**.<sup>11b</sup>

The dimerization of **3** is highly regioselective, leading only to the thermodynamically less stable HH dimer **2**.<sup>12</sup> The isomeric head-to-tail (HT) dimer is not observed (<5%). This regioselectivity is different from that exhibited by most other simple silenes<sup>3</sup> (e.g., Me<sub>2</sub>Si=CH<sub>2</sub>,<sup>4,13</sup> (Me<sub>3</sub>Si)<sub>2</sub>C=SiMe<sub>2</sub><sup>14</sup>), which yield HT dimers, but it parallels the behavior of (Me<sub>3</sub>Si)<sub>2</sub>Si=C(OSiMe<sub>3</sub>)R (**6**) (where R = Me,<sup>15a</sup> Et,<sup>15a</sup> *i*-Pr,<sup>15a</sup> CH<sub>2</sub>Ph,<sup>15b</sup> *t*-Bu,<sup>5b</sup> CEt<sub>3</sub>,<sup>5b</sup> Ad,<sup>5b</sup> Ph<sup>15b</sup>) and of (Me<sub>3</sub>Si)<sub>2</sub>Si=CMePh (**7**).<sup>16</sup> Experimental

(9) Crystal data for **2**: C<sub>32</sub>H<sub>64</sub>Si<sub>6</sub>, *M*<sub>r</sub> = 617.3, monoclinic, *a* = 9.646 (4) Å, *b* = 20.240 (8) Å, *c* = 19.064 (8) Å, β = 98.60 (4)°, *V* = 3680.1 Å<sup>3</sup>, space group *C2/c*, *Z* = 4, μ(Mo Kα) = 2.4 cm<sup>-1</sup>, ρ<sub>calc</sub> = 1.115 g/cm<sup>3</sup>. The 3142 unique reflections (5 ≤ 2θ ≤ 50°, |F<sub>o</sub>| > 3σ|F<sub>c</sub>|) were measured on a Philips PW 1100/20 four-circle diffractometer using Mo Kα radiation and the ω/2θ scan mode. The structure was solved by SHELXS direct methods.

(10) The presence of Me<sub>3</sub>SiOLi could be detected by its NMR signal at 0.30 ppm. Upon aqueous workup Me<sub>3</sub>SiOLi produces Me<sub>3</sub>SiOH, which spontaneously condenses to Me<sub>3</sub>SiOSiMe<sub>3</sub>.

(11) (a) With butadiene, ca. 10% of the [2 + 2] products was also observed. (b) <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 0.08, 0.01 (each 9H, s, Me<sub>3</sub>Si), 1.41, 1.82 (each 1 H, m, CH<sub>2</sub>Si), 1.25–2.19 (14 H, m, Ad), 3.14 (3 H, s, OCH<sub>3</sub>), 4.41 (1 H, d, *J* = 6 Hz, HCO), 5.70, 6.15 (each 1 H, m, –CH=CH–). <sup>13</sup>C NMR: δ 1.13, 1.14 (Me<sub>3</sub>Si), 10.74 (CH<sub>2</sub>Si), 27.73–39.57 (Ad), 79.23 (OC–H<sub>3</sub>), 128.32, 132.99 (–CH=CH–), 160.83 (HCO). <sup>29</sup>Si NMR: δ –13.31, –14.50 (Me<sub>3</sub>Si), –44.60 (ring Si). IR: 1628 cm<sup>-1</sup> (C=C). MS (CI; *m/e* (relative intensity)): 393 (M<sup>+</sup> + H, 13), 3.91 (M<sup>+</sup> – H, 21), 377 (M<sup>+</sup> – Me, 30), 361 (M<sup>+</sup> – MeO, 100), 319 (M<sup>+</sup> – Me<sub>3</sub>Si, 78). Anal. Calcd for C<sub>22</sub>H<sub>40</sub>O<sub>2</sub>Si<sub>2</sub>: C, 64.22; H, 10.27. Found: C, 64.10; H, 10.60. A [2 + 2] addition product was excluded by the fact that 2D-COSY experiments showed that the two vinylic protons are coupled to aliphatic protons. The regiochemistry of **5** was secured by comparison with the <sup>1</sup>H NMR spectrum of the analogous [4 + 2] adduct of **3** and butadiene, where the indicative chemical shifts are δ 1.43 (2 H, m, SiCH<sub>2</sub>–CH=CH) and 2.36 (2 H, m, Ad–CH<sub>2</sub>–CH<sub>2</sub>–CH).

(12) MM2 (87) force-field calculations (Allinger, N. L. *J. Am. Chem. Soc.* 1977, 99, 8127) suggest that **2** is less stable than the corresponding HT dimer by 36 kcal mol<sup>-1</sup>. A similar energy difference (i.e., 30–40 kcal mol<sup>-1</sup>) is obtained by using several semiempirical methods (PM3, AM1, MNDO). Details will be presented elsewhere.

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(6) Gutekunst, G.; Brook, A. G. *J. Organomet. Chem.* 1982, 225, 1.

(7) Previous similar attempts have failed, leading to rearranged products: Wustrack, R.; Oehme, H. *J. Organomet. Chem.* 1988, 352, 95.

(8) Reactions were performed in THF with enolizable ketones, and **1** was generated in situ from (Me<sub>3</sub>Si)<sub>2</sub>Si and MeLi.

(9) A typical procedure is as follows: 2.2 g (4.7 mmol) of (Me<sub>3</sub>Si)<sub>2</sub>SiLi·3THF (**1**)<sup>6</sup> in 20 mL of dry benzene was placed in a Schlenk flask under vacuum, and 0.6 g (4.2 mmol) of adamantanone in 10 mL of dry benzene was added. After the mixture was stirred at room temperature for 2 h, 20 mL of a saturated aqueous solution of NH<sub>4</sub>Cl was added to the reaction mixture. The organic layer was separated and dried over MgSO<sub>4</sub>, the volatile materials were evaporated under vacuum, and the crude product was washed with 2 × 10 mL of methanol, yielding 1.2 g (85.7%) of a clean white powder of **2**. Colorless crystals of **2** (mp 165–166 °C) suitable for X-ray analysis were obtained by recrystallization from hexane at 0 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 0.23 (18 H, s, Me<sub>3</sub>Si), 0.29 (18 H, s, Me<sub>3</sub>Si) 1.60–2.20 (14 H, m, Ad), 27.96, 29.21, 32.81, 34.15, 36.07, 40.68, 43.08, 44.24 (Ad). <sup>29</sup>Si NMR: δ –10.64, –11.02 (SiMe<sub>3</sub>), –45.14 (ring Si). MS (CI; *m/e* (relative intensity)): 616 (M<sup>+</sup>, 100), 543 (M<sup>+</sup> – Me<sub>3</sub>Si, 7), 348 ((Me<sub>3</sub>Si)<sub>2</sub>Si)<sub>2</sub><sup>+</sup>, 90), 308 (M<sup>+</sup>/2, 82), 268 (Ad<sub>2</sub><sup>+</sup>, 30), 73 (Me<sub>3</sub>Si, 5). MS (EI; *m/e*): (M<sup>+</sup> – Me<sub>3</sub>Si) 543.3111; C<sub>32</sub>H<sub>64</sub>Si<sub>6</sub> requires 543.3150.

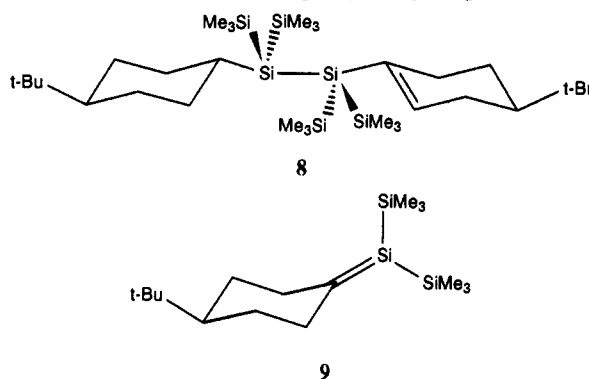
and theoretical studies of the electronic and steric factors which control these regiochemistries are in progress.<sup>17</sup>

The X-ray structure of **2** (Figure 1) reveals the significant consequences of its extreme steric congestion. (a) The ring C-C and C-Si bond lengths ( $r$ ) of 1.647 and 2.008 Å, respectively, are among the longest known distances for such bonds.<sup>18</sup> Similar  $r(\text{C-C})$  and  $r(\text{C-Si})$  values were reported for the HH dimer of **6** ( $R = t\text{-Bu}$ ).<sup>19</sup> (b) The two adamantyls are nearly perpendicular (i.e.,  $\angle\text{C2-C1-C1'-C2}' = -90.3^\circ$ ). (c) The 1,2-disilacyclobutane ring is strongly puckered; the angle between the P1 and P2 planes (Figure 1) is  $39.6^\circ$ . In contrast, in the HH dimer of **6** ( $R = t\text{-Bu}$ ) the four-membered ring is nearly planar.<sup>19</sup> According to temperature-dependent NMR measurements,<sup>20a</sup>  $\Delta H^\ddagger$  and  $\Delta S^\ddagger$  for inversion of the 1,2-disilacyclobutane ring are  $16.1 \pm 0.8 \text{ kcal mol}^{-1}$  and  $-2.5 \pm 2.0 \text{ eu}$ , respectively.<sup>20b</sup>

The dimer **2** is a convenient precursor for generating the silene **3**; at  $70^\circ\text{C}$  (benzene) **2** reverts to **3**, as is indicated by its trapping with 1-methoxybutadiene (yielding **5**<sup>11b</sup>) and with methanol (producing the expected 2-Ad-Si( $\text{SiMe}_3$ )<sub>2</sub>OCH<sub>3</sub>).

Enolizable ketones can also lead to silenes.<sup>7</sup> Reaction of **1** with 4-*tert*-butylcyclohexanone at  $-78^\circ\text{C}$ <sup>21</sup> followed by 2 h at room temperature and aqueous workup produces the noncyclic dimer **8** (80% yield).<sup>22,23</sup> This is consistent with the intermediacy of silene **9**, which can dimerize to **8** via an ene-type reaction or via coupling followed by disproportionation. Dimers analogous to **8** were obtained

from other silenes possessing allylic hydrogens.<sup>15,24</sup>



In conclusion, we have shown that a Peterson-type reaction presents a novel route for synthesizing silenes.<sup>25</sup> We are currently studying the scope of this reaction and its possible extension to other types of multiple bonds to Si as well as to the generation of C=Ge multiple bonds.<sup>26</sup>

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**Registry No.** **1**, 81859-95-2; **2**, 141397-68-4; **3**, 141397-69-5; **5**, 141397-70-8; **8**, 141397-72-0; 2-Ad-Si( $\text{SiMe}_3$ )<sub>2</sub>OCH<sub>3</sub>, 141397-71-9; adamantanone, 700-58-3; 1,1-bis(trimethylsilyl)spiro[silacyclohex-4-ene-2,2'-tricyclo[3.3.1.1<sup>3,7</sup>]decane], 141397-73-1; butadiene, 106-99-0; 1-methoxybutadiene, 3036-66-6; 4-*tert*-butylcyclohexanone, 98-53-3; 1,1-bis(trimethylsilyl)-4-ethenylspiro[silacyclobutane-2,2'-tricyclo[3.3.1.1<sup>3,7</sup>]decane], 141397-74-2.

**Supplementary Material Available:** Complete information on the X-ray crystal analysis of **2**, which includes a description of the data collection and refinement, tables of positional and thermal parameters, full lists of bond lengths and angles, and a figure showing the atom-numbering scheme (8 pages). Ordering information is given on any current masthead page.

OM920154Q

(16) Ohahita, J.; Masauka, Y.; Ishikawa, M. *Organometallics* 1991, 10, 3775.

(17) For previous discussions see ref 5b and 15a and: Apeloig, Y.; Karni, M. *J. Am. Chem. Soc.* 1984, 106, 6676.

(18) (a) C-C: Osawa, E. In *Molecular Structure and Energetics*; Liebman, J. F., Greenberg, A., Eds.; VCH: Deerfield Beach, FL, 1986; Vol. 3, p 329. (b) C-Si: Sheldrick, W. S. In ref 2, Chapter 3.

(19) Brook, A. G.; Nyburg, S. C.; Reynolds, W. P.; Poon, Y. C.; Chang, Y.-M.; Lee, J.-S.; Picard, J.-P. *J. Am. Chem. Soc.* 1979, 101, 6750.

(20) (a) The DYNAMAR program was used: Meakin, P. Z. (modified by McKenna, S. T.), Department of Chemistry, University of California, Berkeley, CA 94720. (b)  $r = 0.996$  (five temperatures).

(21) Only  $\alpha$ -lithiation occurs at room temperature.

(22) <sup>1</sup>H NMR ( $\text{C}_6\text{D}_6$ ):  $\delta$  0.23 (1 H, s, ring CH-Si), 0.36 (36 H, br s,  $\text{Me}_3\text{Si}$ ), 0.79, 0.87 (9 H each, s, *t*-Bu), 1.2-2.4 (18 H, br m, cyclohexane + cyclohexene), 6.02 (1 H, s, CH=C). <sup>13</sup>C NMR:  $\delta$  3.19, 3.55 ( $\text{SiMe}_3$ ), 20.05, 25.51, 25.73, 27.19, 28.03, 29.94, 30.00, 34.98, 43.73, 43.98 (*tert*-butylcyclohexane + *tert*-butylcyclohexene), 133.73, 140.77 (-C=C-). <sup>29</sup>Si NMR:  $\delta$  -12.78, -13.07 ( $\text{SiMe}_3$ ), 64.60, 71.82 ( $\text{SiSiMe}_3$ ). IR: 1607 (C=C)  $\text{cm}^{-1}$ . MS (CI;  $m/e$  (relative intensity)): 624 ( $\text{M}^+$ , 83), 551 ( $\text{M}^+ - \text{Me}_3\text{Si}$ , 51), 485 ( $\text{M}^+ - \text{tert-butylcyclohexane}$ , 30), 415 ( $\text{M}^+ - \text{tert-butylcyclohexane} - \text{C}_6\text{H}_{10}$ , 100). MS (EI):  $\text{M}^+$ , 624.4176;  $\text{C}_{32}\text{H}_{72}\text{Si}_6$  requires 624.4249.

(23) The structure of **8** was secured by X-ray analysis.

(24) Barton, T. J.; Hoekman, S. K. *J. Am. Chem. Soc.* 1980, 102, 1584.

(25) While our work was in progress,<sup>1</sup> Ishikawa et al. reported<sup>16</sup> that reaction of  $(\text{Me}_3\text{Si})_2\text{SiCOR}$  ( $R = \text{Me}, \text{Ph}$ ) with MeLi also produces transient silenes.

(26) For a recent review, see: Barrau, J.; Escudie, J.; Stage, J. *Chem. Rev.* 1990, 90, 283.