it to be a single, π-complexed, bridgehead olefin. Attempts to decomplex **9** in the usual way with NaI/acetone[9] were unsuccessful; however, stirring the complx in CD_2Cl_2 for 5 h at -20 °C with 1.5 equiv of anhydrous trimethylamine oxide[10] and filtering through Celite provided a single olefin whose $^{13}C\{^1H\}$ NMR matches tht reported for (Z)-bicyclo[3.3.1]non-1-ene (**10**),[3,11] Scheme II.

The structure of **9**, determined by single-crystal X-ray diffraction,[12] is shown in Figure 1. As the bond lengths and bond angles indicate, the cationic portion of the complex is octahedral with the olefin occupying one coordination site, the Cp three. The iron is located midway between the olefinic carbons. The double bond is lengthened slightly; its axis is essentially parallel to one Fe–CO bond and perpendicular to the other.[13] This structure confirms that **9** is indeed a π-complexed bicyclo[3.3.1]non-1-ene and establishes the geometry of the double bond as Z.

Our results establish rather clearly a new mode of reaction for transition metal alkylidenes: β- to α-alkyl migration.[14] That **5** is converted to **9** rather than to a different bridgehead olefin complex indicates that ring enlargement is *not* concerted with the loss of ethanol from **7**. The considerable precedent for the formation of Fe(II) alkylidenes by protonation of Fe(II) α-alkoxyalkyls[15] suggests **8** as an intermediate in this case. We formulate the reaction as shown in Scheme II.

The ring enlargement is regiospecific:[16] of the six bridgehead-olefin π-complexes that are possible only **9** is observed. Even through the rearrangement produces what is probably the most stable of these complexes, we presume that the observed specificity is kinetic rather than thermodynamic for there is no indication of the reaction being reversible. The ring enlargement relieves considerable strain[1] and hence is likely to be exothermic and to occur via an early transition state[18] in which the filled iron orbitals are not yet properly positioned for good overlap with the developing empty orbital at C(β).[19] Consequently, the bridgehead cation is probably a better model energetically for the transition state than is the π-complexed olefin. If the barrier to C(α)–C(β) rotation in **8** is small relative to the activation enthalpy of rearrangement,[20] ethano-bridge migration is expected to be about 4 kcal/mol more favorable than methano or propano migration.[21] When it occurs in the most stable conformer[23] (Scheme II), ethano-bridge migration generates the observed product, **9**.[24]

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(13) Krüger, C.; Barnett, B. L.; Brauer, D. In "The Organic Chemistry of Iron"; Koerner von Gustorf, E. A., Grevels, F.-H., Fischler, I., Eds.; Academic Press: New York, 1978; Vol. 1, pp 8–13.

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(15) (a) Brookhart, M.; Nelson, G. O. *J. Am. Chem. Soc.* **1977**, *99*, 6099. (b) Bodnar, T.; Cutler, A. R. *J. Organomet. Chem.* **1981**, *213*, C31. (c) Casey, C. P.; Miles, W. H.; Tukada, H.; O'Connor, J. M. *J. Am. Chem. Soc.* **1982**, *104*, 3761.

(16) Orchin, M.; Kaplan, F.; Macomber, R. S.; Wilson, R. M.; Zimmer, H. "The Vocabulary of Organic Chemistry"; Wiley: New York, 1980; p 146.

(17) The stabilization which results from π-complexation is known only qualitatively but (Z)-bicyclo[3.3.1]non-1-ene (**10**) itself is estimated by molecular mechanics to be at least 6 kcal/mol more stable than any of the other ametallic possibilities, viz., (E)- or (Z)-bicyclo[4.2.1]non-1-ene, (E)-bicyclo[3.3.1]non-1-ene, or (E)- or (Z)-bicyclo[3.2.2]non-1(7)-ene.[1]

(18) Hammond, G. S. *J. Am. Chem. Soc.* **1955**, *77*, 334.

(19) We thank a referee for his helpful comments on this point.

(20) (a) Curtin, D. Y. *Rec. Chem. Prog.* **1955**, *15*, 111. (b) Cf.: Eliel, E. L. "Stereochemistry of Carbon Compounds"; Mc Graw-Hill: New York, 1962; p 238 ff.

(21) The strain energies of the bridgehead bicyclononyl cations (kcal/mol) estimated from the ethanolysis constants of the bridgehead bromides:[22] [3.3.1] (1.2), [4.2.1] (5.4), [3.2.2] (5.5).

(22) (a) Bingham, R. C.; Schleyer, P. v. R. *J. Am. Chem. Soc.* **1971**, *93*, 3189. (b) Becker, K. B. *Helv. Chim. Acta* **1977**, *60*, 94.

(23) (a) Schilling, B. E. R.; Hoffman, R.; Lichtenberger, D. L. *J. Am. Chem. Soc.* **1979**, *101*, 585. (b) Schilling, B. E. R.; Hoffmann, R.; Faller, J. W. *J. Am. Chem. Soc.* **1979**, *101*, 592. (c) Seeman, J. I.; Davies, S. G. *J. Chem. Soc., Chem. Commun.* **1984**, 1014.

(24) Formation of the less stable E isomer[1,17] would require that ethano-bridge migration occur in the *least* stable conformer of **8**.

We continue to examine both the mechanism and utility of this new reaction of transition-metal alkylidenes, this new route to a stablized bridgehead olefin.

Mercuration of the Se–Se Bond of a Chelating Se₂ Group with Hg⁰: Preparation and Crystal Structure of [(dmpe)₂Ir(μ-Se)₂Hg]₄Cl₄

A. P. Ginsberg* and C. R. Sprinkle

AT&T Bell Laboratories
Murray Hill, New Jersey 07974
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It has been known for some time that Hg^0 can mercurate the S–S and Se–Se bonds of perfluoroalkyl disulfides and diselenides:[1]

$$RXXR + Hg^0 \rightarrow (RX)_2Hg$$

$$R = CF_3, C_3F_7; \quad X = S, Se$$
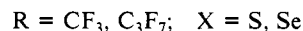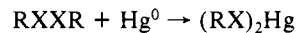
We now report the first example of mercuration of the chalcogen–chalcogen bond of a dichalcogen group chelated to a transition metal in a complex. The mercuration occurs when the compound $[Ir(Se_2)(dmpe)_2]Cl^2$ in acetonitrile solution is treated with metallic mercury and leads to the tetrameric product $[(dmpe)_2Ir(\mu\text{-}Se)_2Hg]_4Cl_4$ in which Hg has inserted into an Se₂ group and also bridges to an adjacent Se₂ group. The reaction corresponds to oxidative addition of Hg across the Se–Se bond and is similar to insertion of low-valent metal complexes into the S–S and Se–Se bonds of S₂ and Se₂ groups chelating[3] or bridging[4] metal atoms. That Hg^0 mercurates Se₂ in $[Ir(Se_2)(dmpe)_2]Cl$, rather than stripping the selenium off as HgSe, is a striking example of the sensitive dependence of the reactivity of the Se₂ group in [M-(Se₂)(L-L)₂]Cl complexes on the nature of the metal M and the basicity of the ditertiary phosphine L-L: for both M = Rh, L-L = dmpe and M = Ir, L-L = dppe the only reaction observed with metallic mercury is

$$[M(Se_2)(L\text{-}L)_2]^+ + 2Hg^0 \xrightarrow{\quad CH_3CN \quad} [M(L\text{-}L)_2]^+ + 2HgSe$$
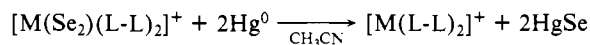
When $[Ir(Se_2)(dmpe)_2]Cl$ (200 mg, 0.292 mmol) in acetonitrile (20 mL) was stirred with mercury (2.0 g, 10.0 mmol), the purple color of the Se₂ complex slowly faded and the solution became yellow.[5] After 4 days the yellow solution was concentrated to 10 mL and excess diethyl ether (70 mL) was added to precipitate the yellow product (208 mg, 80%) with stoichiometry [Ir-(Se₂Hg)(dmpe)₂]Cl.[6] The same mercury adduct is obtained when $[Ir(Se_4)(dmpe)_2]Cl^2$ in acetonitrile is stirred with excess mercury. In this case HgSe precipitates and the red Se₄ complex is converted to the purple Se₂ complex which is then mercurated.

(1) Brandt, G. A. R.; Emeléus, H. J.; Haszeldine, R. N. *J. Chem. Soc.* **1952**, 2198. Dale, J. W.; Emeléus, H. J.; Haszeldine, R. N. *Ibid.* **1958**, 2939. Emeléus, H. J.; Welcman, N. *Ibid.* **1963**, 1268.

(2) Ginsberg, A. P.; Osborne, J. H.; Sprinkle, C. R. *Inorg. Chem.* **1983**, *22*, 1781.

(3) Ginsberg, A. P.; Lindsell, W. E.; Sprinkle, C. R.; West, K. W.; Cohen, R. L. *Inorg. Chem.* **1982**, *21*, 3666.

(4) Seyferth, D.; Henderson, R. S.; Gallagher, M. K. *J. Organomet. Chem.* **1980**, *193*, C75. Lesch, D. A.; Rauchfuss, T. B. *Ibid.* **1980**, *199*, C6.

(5) The reaction and workup were carried out under dry oxygen-free nitrogen or helium. All solvents were dried and distilled under nitrogen.

(6) Anal. Calcd for $C_{12}H_{32}P_4IrSe_2HgCl$: C, 16.26; H, 3.64; P, 13.98; Se, 17.81; Hg, 22.63; Cl, 4.00. Found (by analytische Laboratorien, Engelskirchen, West Germany, after drying at 55 °C (10^{-3} mm)): C, 16.20; H, 3.67; P, 14.14; Se, 17.66; Hg, 22.58; Cl, 3.83.
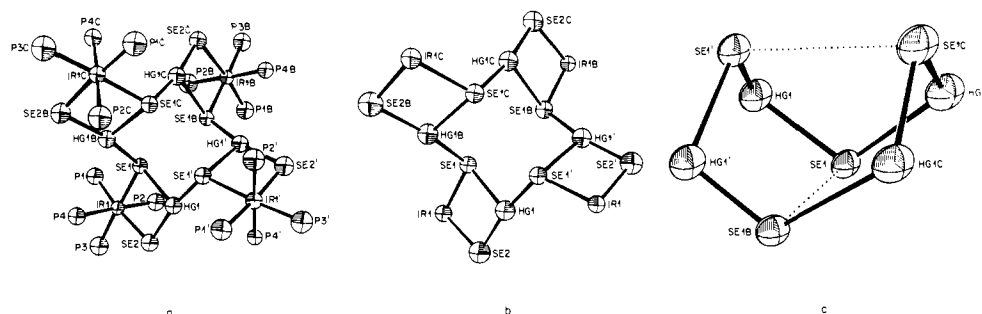
Figure 1. ORTEP drawings of [(dmpe)$_2$Ir(μ-Se)$_2$Hg]$_4$$^{4+}$ with 50% probability thermal spheres: (a) Omitting the organic part of the phosphine ligand. (b) Omitting the entire phosphine ligand to show the windmill-like appearance of the (HgSe)$_4$ ring with its four attached (IrSe$_2$Hg) rings. (c) A view of the (HgSe)$_4$ ring showing its cagelike nature and its resemblance to the structures of As$_4$S$_4$ and S$_4$N$_4$.

Table I. Selected Bond Distances (Å) and Angles (deg) For One of the Two [(dmpe)$_2$Ir(μ-Se)$_2$Hg]$_4$$^{4+}$ Cations in the Asymmetric Unit

Hg(1)–Se(1)	2.833 (8)	Se(1)–Hg(1)–Se(2)	84.9 (3)
Hg(1)–Se(2)	2.475 (8)	Se(1′)–Hg(1)–Se(2)	168.6 (3)
Hg(1)–Se(1′)	2.451 (8)	Se(1)–Hg(1)–Se(1′)	106.5 (2)
Ir(1)–Se(1)	2.585 (7)	Se(1)–Ir(1)–Se(2)	88.1 (3)
Ir(1)–Se(2)	2.581 (8)	Se(1)–Ir(1)–P(1)	88.1 (6)
Ir(1)–P(1)	2.32 (2)	Se(1)–Ir(1)–P(2)	85.5 (6)
Ir(1)–P(2)	2.33 (2)	Se(1)–Ir(1)–P(3)	177.0 (7)
Ir(1)–P(3)	2.29 (2)	Se(1)–Ir(1)–P(4)	94.3 (5)
Ir(1)–P(4)	2.30 (2)	P(1)–Ir(1)–P(2)	84.9 (8)
		P(1)–Ir(1)–P(3)	94.9 (9)
		P(1)–Ir(1)–P(4)	97.7 (8)
		P(2)–Ir(1)–P(3)	94.2 (8)
		P(2)–Ir(1)–P(4)	177.4 (8)
		P(3)–Ir(1)–P(4)	85.8 (8)

Single crystals suitable for X-ray diffraction were obtained by crystallization from 2-propanol. The crystal structure[7,8] consists of discrete [(dmpe)$_2$Ir(μ-Se)$_2$Hg]$_4$$^{4+}$ cations and Cl$^-$ anions; the two crystallographically independent cations in the asymmetric unit are quite similar.[9] ORTEP views of the cation, shown in Figure 1, reveal a number of interesting features. Hg has inserted asymmetrically into the Se$_2$ group of [Ir(Se$_2$)(dmpe)$_2$]$^+$ (Hg-(1)–Se(1) = 2.833 (8) Å, Hg(1)–Se(2) = 2.475 (8) Å) and is also bonded to Se(1) of a second [(dmpe)$_2$Ir(μ-Se)$_2$Hg] group (Hg-(1)–Se(1′) = 2.451 (8) Å). The Hg atoms have distorted trigonal-planar coordination with bond angles Se(1)–Hg(1)–Se(2) = 84.9 (3)°, Se(1′)–Hg(1)–Se(2) = 168.6 (3)°, and Se(1)–Hg-(1)–Se(1′) = 106.5 (2)°; for the short Hg–Se bonds there is a deviation of only 11° from the linear arrangement characteristic of Hg^{2+}. The short Hg–Se distances are typical of what is found

(7) The crystal structure determination was carried out by the staff of Molecular Structure Corporation: Dr. M. W. Extine, R. A. Meisner, Dr. J. M. Troup, and B. B. Warrington.

(8) Crystal data: 0.15 × 0.15 × 0.25 mm yellow prism, cubic space group $I\bar{4}3d$, $a = b = c = 40.820$ (10) Å, $\alpha = \beta = \gamma = 90.0$ (0)°, $V = 68\,017.2$ Å^3, $Z = 96$, $\rho_{calcd} = 2.08$ g/cm^3, and μ(Mo Kα) = 135.6 cm^{-1}. 4172 unique reflections were measured at 23 ± 1 °C on an Enraf-Nonius CAD4 diffractometer by using Mo Kα ($\lambda = 0.710\,73$ Å) radiation out to $2\theta = 45$°. Lorenz and polarization corrections as well as an empirical absorption correction (min–max relative transmission coefficients, 0.694–0.972) were applied to the data. The structure was solved by direct methods and difference-Fourier synthesis. Very weak high-resolution data led to difficulties in locating chlorine and carbon atoms; only the Hg, Ir, and Se atoms were well localized. The phosphine groups showed some disorder and the numerous fractionally occupied chlorine atom sites made an absolute determination of the chlorine locations and occupancies impossible; the overall chlorine occupancy in the structure was set to the analytically determined Ir:Cl ratio of 1:1. Only the 1335 unique reflections with $F_o^2 > 3\sigma(F_o^2)$ were used in the full-matrix least-squares refinement, which gave $R = 0.054$ and $R_w = 0.060$. Hydrogen atoms were not included in the calculations. Hg, Ir, and Se were allowed to vibrate anisotropically, but C, P, and Cl were assigned isotropic thermal parameters. The height of the highest peak in the final difference Fourier map was 0.45 (11) e/Å^3.

(9) Comparison of the two cations in the asymmetric unit shows only two significantly different bond distances: in the first molecule (Figure 1) Ir-(1)–Se(2) = 2.581 (8) Å and P(4)–C(10) = 1.71 (7) Å while the corresponding distances in the second molecule are, respectively, 2.553 (8) and 1.95 (7) Å. Several bond angles show differences of 2–5°; the only larger differences occur for two C–P–C and two Ir–P–C angles where the differences between the two cations range from 9 to 17°.

for linear Se–Hg–Se. Thus, in CuHgSeCl, which contains Se–Hg–Se chains with Se–Hg–Se angles of 171.3°, the Hg–Se distance is 2.477 and 2.486 Å.[10] An Hg coordination geometry similar to that in [(dmpe)$_2$Ir(μ-Se)$_2$Hg]$_4$$^{4+}$, but much less distorted from trigonal planar, is found in CsHg(SCN)$_3$ which has Hg–S bond distances of 2.56, 2.45, and 2.44 Å with bond angles of 109°, 132°, and 116°.[11]

Hg–Se–Hg bridges link the four [(dmpe)$_2$Ir(μ-Se)$_2$Hg] units in the cation and make up an eight-membered (HgSe)$_4$ ring in which long (2.833 (8) Å) and short (2.451 (8) Å) Hg–Se distances alternate. The ring is puckered by folding along the two Se···Se lines (Figure 1c) to give a cage with a square set of Hg atoms and a distorted tetrahedron of Se atoms. Both the Hg···Hg (4.11 Å) and Se···Se (4.24 Å) distances are nonbonding. The (HgSe)$_4$ cage structure resembles the cage structures of S$_4$N$_4$, which has a square of N atoms and a tetrahedron of S atoms, and As$_4$S$_4$, which has a square of S atoms and a tetrahedron of As atoms. In both S$_4$N$_4$ and As$_4$S$_4$, however, the tetrahedrally arranged atoms are close enough to bond to each other.[12]

In addition to the (HgSe)$_4$ ring [(dmpe)$_2$Ir(μ-Se)$_2$Hg]$_4$$^{4+}$ contains four nearly flat four-membered Ir(μ-Se)$_2$Hg rings, one edge of which is also an edge of the (HgSe)$_4$ ring. The folded eight-membered ring with its peripheral four-membered rings has a windmill-like appearance that may be seen in Figure 1b. There are two types of Se atom in the complex: those in (HgSe)$_4$ rings are 3-coordinate and bridge an Ir and two Hg atoms, while those not in (Hg–Se)$_4$ rings are 2-coordinate and bridge Ir and Hg atoms. The Ir–Se distances (Ir(1)–Se(1) = 2.585 (7) Å and Ir(1)–Se(2) = 2.581 (8) Å) are longer than the distances in [Ir(Se$_4$)(dmpe)$_2$]Cl[2] and [Ir(Se$_2$)(dppe)$_2$]Cl[3] by, respectively, 0.04 and 0.05 Å. The Ir–P distances are not significantly different from those in [Ir(Se$_4$)(dmpe)$_2$]Cl, while the P–Ir–P angles show only small differences from the latter compound.

Xα-Sw molecular orbital calculations on [Ir(Se$_2$)(PH$_3$)$_4$]$^+$ have shown that the chelated Se$_2$ group is best described as a neutral molecule with excited configuration ··· (pσ)^2(π)^3(π*)^3 and an Se–Se bond order of 1.[13] Hg insertion into the Se–Se bond of this group may be described formally as an oxidative addition in which the Hg is oxidized to Hg^{2+} and the two Se atoms are reduced to Se$^-$. The short, nearly colinear, Hg–Se bonds in [(dmpe)$_2$Ir(μ-Se)$_2$Hg]$_4$$^{4+}$ are in agreement with this description and correspond to overlap with predominantly sp hybrid orbitals on Hg^{2+}. The long Hg–Se bond is roughly orthogonal to the two short bonds and must be due to overlap with a nearly pure p orbital on Hg^{2+}. Lengthening of Hg–ligand bonds with decreasing s character in the Hg orbitals has been noted previously (compare Hg–Se = 2.44 Å for sp hybridization and Hg–Se = 2.62 Å for sp^3 hybridization), and a pair of anomalously long Hg–Cl bridge bonds (2.781 and 2.596 Å) with an Cl–Hg–Cl angle of 88.3° has

(10) Guillo, M.; Mercey, B.; Labbé, P.; Deschanvres, A. *Acta Crystallogr., Sect. B* **1980**, *B36*, 2520.

(11) Thiele, G.; Messer, D. *Z. Anorg. Allg. Chem.* **1976**, *421*, 24.

(12) Cotton, F. A.; Wilkinson, G. "Advanced Inorganic Chemistry"; 4th ed.; Wiley: New York, 1980, p 516.

(13) Ginsberg, A. P.; Osborne, J. H.; Sprinkle, C. R. *Inorg. Chem.* **1983**, *22*, 254.

been inferred to involve mercury orbitals of predominantly p character.[14]

Supplementary Material Available: Tables of bond distances and angles, structure factors, and positional and thermal parameters (10 pages). Ordering information is given on any current masthead page.

(14) Dent Glasser, L. S.; Ingram, L.; King, M. G.; McQuillan, G. P. *J. Chem. Soc. A* **1969**, 2501.

Tetrasilabicyclo[1.1.0]butane System: Preparation and Characterization of 1,3-Di-*tert*-butyl-2,2,4,4-tetrakis-(2,6-diethylphenyl)tetrasilabicyclo[1.1.0]butane

Satoru Masamune,* Yoshio Kabe, and Scott Collins

Department of Chemistry
Massachusetts Institute of Technology
Cambridge, Massachusetts 02139

David J. Williams* and Ray Jones

Chemical Crystallography Laboratory
Department of Chemistry, Imperial College
London SW7 2AY, England

We wish to report herein the synthesis and characterization of the title compound **1** (R = *tert*-butyl in Scheme I), which represents the first tetrasilabicyclo[1.1.0]butane derivative. Although no mention of this system has been made in the literature,[1-3] the anticipated reactivity attributed to its intrinsic strains attracts great interest.[4] It has now been found that the central Si(1)–Si(3) bond of compound **1** is readily susceptible to a variety of external attacks and the puckered ring system itself undergoes rapid inversion (**1** ⇌ **1a**).

Synthesis of 1 (Scheme II). Reaction of trichlorosilane **2** (1 equiv) with (2,6-diethylphenyl)lithium (2 equiv), prepared from the corresponding bromide and *tert*-butyllithium, led to the formation of chlorobis(2,6-diethylphenyl)silane **3** (73% yield),[5] which after metalation with lithium was condensed with *tert*-butyl-chlorosilane (1 equiv)[6] to provide the disilane **4** (41%).[5] Treatment of **4** with chlorine (ca. 3.5 equiv, 2 M, CCl$_4$) yielded the corresponding trichloro compound **5** of 88% purity (71%)[5] which was

(1) Other strained silicon systems which are known include cyclotrisilanes (a–h) and spiropentasilane (i): (a) Masamune, S.; Hanzawa, Y.; Murakami, S.; Bally, T.; Blount, J. F. *J. Am. Chem. Soc.* **1982**, *104*, 1150. (b) Masamune, S.; Tobita, H.; Murakami, S. *Ibid.* **1983**, *105*, 6524. (c) Murakami, S.; Collins, S.; Masamune, S. *Tetrahedron Lett.* **1984**, *25*, 2131. (d) Masamune, S.; Murakami, S.; Snow, J. T.; Tobita, H.; Williams, D. J. *Organometallics* **1984**, *3*, 333. (e) Dewan, J. C.; Murakami, S.; Snow, J. T.; Collins, S.; Masamune, S. *J. Chem. Soc., Chem. Commun.* **1985**, 892. (f) Watanabe, H.; Okawa, T.; Kato, M.; Nagai, Y. *Ibid.* **1983**, 781. (g) Watanabe, H.; Kougo, Y.; Nagai, Y. *Ibid.* **1984**, 66. (h) Schafer, A.; Weidenbruch, M.; Peters, K.; v. Schnering, H. G. *Angew. Chem., Int. Ed. Engl.* **1984**, *23*, 302. (i) Boudjouk, P.; Sooriyakumaran, R. *J. Chem. Soc., Chem. Commun.* **1984**, 777.

(2) For the 2,4-disilabicyclo[1.1.0]butane system, see: (a) Fritz, G.; Thomas, J. *J. Organomet. Chem.* **1984**, *271*, 107. (b) Fritz, G.; Wartanessian, S.; Matern, E.; Honle, W.; V. Schnering, H. G. *Z. Anorg. Allg. Chem.* **1981**, *475*, 87.
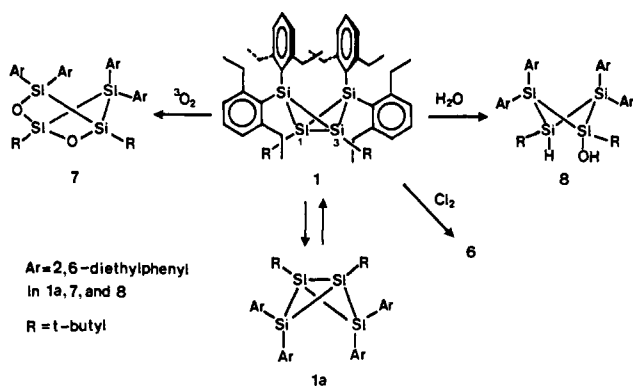
(3) (a) Tetraphospha- and (b) tetraarsabicyclo[1.1.0]butane derivatives have been reported: (a) Niecke, E.; Ruger, R.; Krebs, B. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 544. (b) Baudler, M.; Wietfeltd-Haltenhoff, S. *Ibid.* **1984**, *23*, 379.

(4) Cf. the chemistry of (carbon) bicyclo[1.1.0]butane. Wiberg, K. B. In "Advances in Alicyclic Chemistry"; Hart, H., Karabatsos, G. J., Eds.; Academic Press: New York, 1968; pp 185–205; vol. 2.
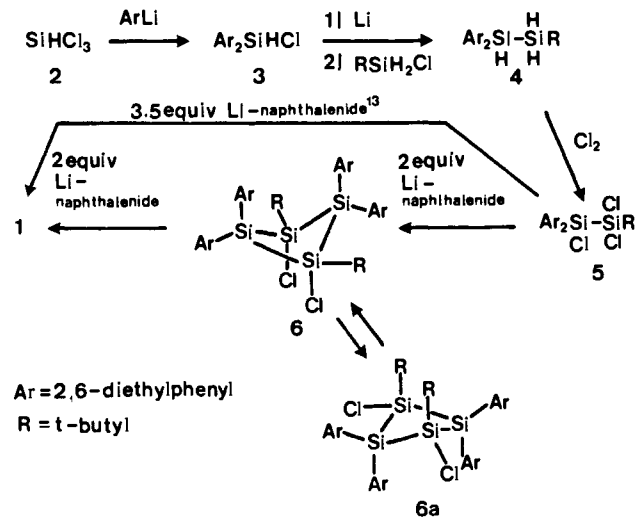
(5) Physical properties of all new compounds and UV and temperature-dependent NMR spectra of **1** are provided in the supplementary material. Complete experimental details for Scheme II are available from S.M. upon request.

(6) Collins, S.; Duncan, J. A.; Kabe, Y.; Murakami, S.; Masamune, S. *Tetrahedron Lett.* **1985**, *26*, 2837.

Scheme I

Ar=2,6-diethylphenyl
In **1a**, **7**, and **8**
R = t-butyl

Scheme II

Ar = 2,6-diethylphenyl
R = t-butyl

in turn reductively cyclized with 2 equiv of lithium naphthalenide. The resulting compound **6** (see below), the only isolable cyclotetrasilane, has cis-oriented chloro substituents and exists in solution as a ca. 3:1 mixture of two conformers (**6** ⇌ **6a**).[7]

The final step, the formation of the central Si–Si bond of **1**, proceeded quantitatively, but during the entire operation, including workup, the contact of **1** with air and moisture had to be strictly avoided. Thus, addition of a 1 M solution (2 equiv) of lithium naphthalenide in dimethoxyethane to a 6 mM solution of **6** in toluene at 0 °C produced a pale yellow solution containing suspended solids. The solvent was removed in vacuo and dry, degassed hexane was introduced by vacuum transfer. The mixture was filtered, the filtrate concentrated in vacuo, and naphthalene removed by sublimation at 0.005 mmHg. The resulting pale yellow solid residue was dissolved in dry, degassed hexane and filtered again. The filtrate was concentrated upon slow evaporation of the solvent in a glovebox to provide pale yellow thermochromic[8] crystals (decomposition at ~240 °C) that exhibit the following physical and chemical properties fully consistent with the bicyclobutane structure **1**.

Physical Properties of 1. (1) mass spectrum (EI), m/z calcd for C$_{48}$H$_{70}$Si$_4$ 758.4555, for C$_{44}$H$_{61}$Si$_4$ 701.3851; found 758.4558 (M$^+$, 14.9%), 701.3855 (M$^+$-*t*-C$_4$H$_9$, 100%). (2) UV (cyclo-

(7) **6**: 290–300 °C dec; ^{1}H NMR (250 MHz, C$_6$D$_6$, at 25 °C) δ 0.37~0.58 (12 H, m, CH$_3$), 0.78 (4.5 H, s, *t*-Bu), 1.22 (13.5 H, s, *t*-Bu), 1.23 (6 H, t, 7 Hz, CH$_3$), 1.28 (2 H, t, 7 Hz, CH$_3$), 1.45 (1 H, t, 7.4 Hz, CH$_3$), 1.56 (3 H, t, 7.4 Hz, CH$_3$), 2.52~2.94 (8 H, m, CH$_2$), 2.94~3.45 (7 H, m, CH$_2$), 3.58~3.72 (1 H, m, 7.4 Hz, CH$_2$), 6.75~6.93 (8 H, m, meta Ar), 7.10, 7.24 (4 H, m, para Ar); mass spectrum (EI), calcd m/z for C$_{48}$H$_{70}$Si$_4$Cl$_2$ 828.3932, found 828.3925 (M$^+$). Two sets of signals are discernible in the ^{1}H NMR spectrum and any pair of signals that are assignable to the same proton of compound **6** has a relative intensity 3:1 and merges at high temperatures (decalin-d_{18}, 160 °C). This spectral behavior is inconsistent with the assignment of a *trans*-dichloro configuration to **6**.

(8) Intensely yellow at 170 °C, colorless at –196 °C.