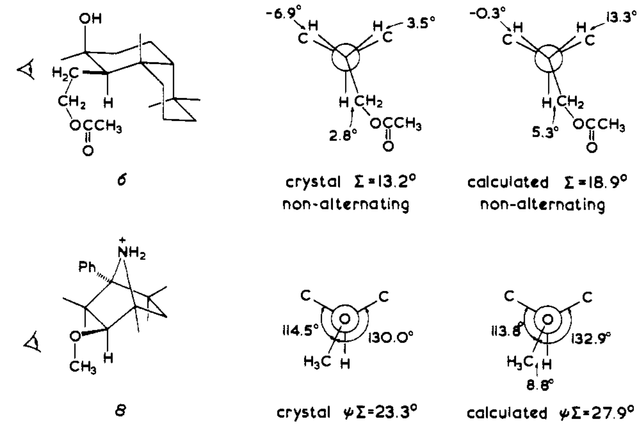
Scheme I



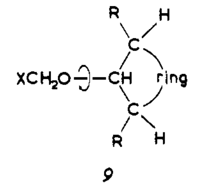
from a 2,2,6,6-tetrasubstituted cyclohexane ring, the bond was more or less eclipsed, with Σ varying from 13.2° to 48.0°. Scheme I shows structure **6** with the smallest of these Σ values.[3a]

Only one structure was found[4] with a fragment of the type $CR_3CH_2{-}{-}{-}CH(CR_3)CR_3$ (**7**), but the bond is nearly eclipsed, with Σ values of 33.6° and 48.0° in the two molecules in the unit cell.

Bonds of carbon with oxygen are shorter than carbon–carbon bonds, and the interactions between substituents which lead to eclipsing conformations may be the greater therefore. We found five examples[5a–e] of the molecular type $CH_3O{-}{-}{-}CH(CR_3)_2$ in the data base, in all of which the conformation is near to eclipsed, with $\psi\Sigma$ ranging from 8.4° to 52.9°. Structure **8** in Scheme I shows one of these five examples.[5a]

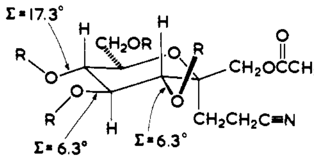
It is a point of caution in discussing crystal structure conformations that lattice forces rather than intramolecular strain may be determining the conformation, although the consistency of the occurrence of eclipsed conformations in the two sets of examples quoted argues against this. We looked at this further by calculating the conformation in the gas phase of the two relatively simple molecules **6** and **8** of Scheme I using Allinger's MM3 molecular mechanics program.[6] Newman projections of the significant bond for these two molecules as in the crystal structure and as calculated for the global minimum conformation are shown in Scheme I, and the good agreement, within 5° on average, suggests that it is intramolecular effects which determine the eclipsing.

Eclipsing is not restricted to the uncommonly branched structural types reported above. The set of 25 molecules of the structural type **9** from the data base includes 16 molecules with



9

at least one eclipsed or nearly eclipsed bond, seven of these involving at least one bond with an eclipsing sum of less than 30°. It is not appropriate to examine this more miscellaneous range in a brief communication, but structure **10** shows a simple molecule with several eclipsed bonds.[7a]



10 R = CH$_2$Ph

This last is one of several examples of a particularly simple type which promises to be a rich source of eclipsed conformations. When there are three adjacent equatorial substituents on a six-membered ring, the middle one being $-X-CH_3$ (or of course $-X-CR^aR^bR^c$), the flanking groups will push the central bond conformation toward one with the $X-CH_3$ bond eclipsing the ring-H bond.

The presence of an eclipsed single bond conformation was noted explicitly in only one of the 34 crystal structure determination reports referred to here, and the importance of vicinal interactions of groups R was in fact suggested.[7b] The number of examples and the relative simplicity of some of them strengthen the original postulate.[1,8]

Acknowledgment. We are grateful to the Ramsay Memorial Fellowships Trust for bringing together this problem and its solution.

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(5) (a) Swanson, R.; Stavinova, J. L.; Mariano, P. S. *Cryst. Struct. Commun.* **1982**, *11*, 799. (b) Mazzocchi, P. H.; Somich, C.; Ammon, H. *Tetrahedron Lett.* **1984**, *25*, 3551. (c) Somich, C.; Mazzocchi, P. H.; Ammon, H. L. *J. Org. Chem.* **1987**, *52*, 3614. (d) Miyahara, K.; Kawasaki, T.; Kinjo, J.-E.; Shimokawa, T.; Yamahara, J.; Yamasaki, M.; Harano, K.; Nohara, T. *Chem. Pharm. Bull.* **1986**, *34*, 4166. (e) Bokii, N. G.; Yanovskii, A. I.; Yu, T.; Struchkov, T.; Shemyakin, N. F. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1978**, *95*, 380.

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(7) (a) Giese, B.; Dupuis, J.; Leising, M.; Nix, M.; Lindner, H. J. *Carbohydr. Res.* **1987**, *171*, 329. (b) Luger, P.; Paulsen, H. *Acta Crystallogr., Sect. B: Struct. Crystallogr. Cryst. Chem.* **1981**, *37*, 1693.

(8) Anderson, J. E. Conformational Analysis of Acyclic and Alicyclic Saturated Hydrocarbons. In *The Chemistry of Alkanes and Cycloalkanes*; Patai, S., Ed.; Wiley: Chichester, Chapter 3.II.D, in press.

Transition Metal Complexes Containing Donor-Stabilized Silylyne Ligands. Reductive Dimerization to a Silylene Complex

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Recent progress in the synthesis of transition metal complexes with unsaturated silicon ligands has stimulated interest in development of new transition metal–silicon chemistry.[1,2] In particular, attention has focused on silylene complexes,[1a–c,j,k] which we have obtained via silyl complexes possessing a labile triflate group.[2] For example, Cp*(PMe$_3$)$_2$RuSi(STol-*p*)$_2$(OTf) (Cp* = η^5-C$_5$Me$_5$; OTf = OSO$_2$CF$_3$) dissociates triflate in solution to give Cp*(PMe$_3$)$_2$Ru=Si(STol-*p*)$_2$$^+$.[2b] Availability of the bis(triflate) derivative Cp*(PMe$_3$)$_2$RuSi(STol-*p*)(OTf)$_2$ (**1**),[2b] and its ionization in acetonitrile,[3] suggested possible routes to new silylene

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(1) Leading references: (a) Probst, R.; Leis, C.; Gamper, S.; Herdtweck, E.; Zybill, C.; Auner, N. *Angew. Chem., Int. Ed. Engl.* **1991**, *30*, 1132. (b) Tobita, H.; Ueno, K.; Shimoi, M.; Ogino, H. *J. Am. Chem. Soc.* **1990**, *112*, 3415. (c) Corriu, R.; Lanneau, G.; Priou, C. *Angew. Chem., Int. Ed. Engl.* **1991**, *30*, 1130. (d) Campion, B. K.; Heyn, R. H.; Tilley, T. D. *J. Am. Chem. Soc.* **1990**, *112*, 4079. (e) Pham, E. K.; West, R. *Organometallics* **1990**, *9*, 1517. (f) Berry, D. H.; Chey, J.; Zipin, H. S.; Carroll, P. J. *Polyhedron* **1991**, *10*, 1189. (g) Koloski, T. S.; Carroll, P. J.; Berry, D. H. *J. Am. Chem. Soc.* **1990**, *112*, 6405. (h) Procopio, L. J.; Carroll, P. J.; Berry, D. H. *J. Am. Chem. Soc.* **1991**, *113*, 1870. (i) Zarate, E. A.; Tessier-Youngs, C. A.; Youngs, W. J. *J. Am. Chem. Soc.* **1988**, *110*, 4068. (j) Tilley, T. D. In *The Chemistry of Organic Silicon Compounds*; Patai, S., Rappoport, Z., Eds.; Wiley: New York, 1989; Chapter 24, p 1415. (k) Tilley, T. D. In *The Silicon–Heteroatom Bond*; Patai, S., Rappoport, Z., Eds.; Wiley: New York, 1991; Chapter 10, p 309.

(2) (a) Straus, D. A.; Zhang, C.; Quimbita, G. E.; Grumbine, S. D.; Heyn, R. H.; Tilley, T. D.; Rheingold, A. L.; Geib, S. J. *J. Am. Chem. Soc.* **1990**, *112*, 2673. (b) Straus, D. A.; Grumbine, S. D.; Tilley, T. D. *J. Am. Chem. Soc.* **1990**, *112*, 7801.

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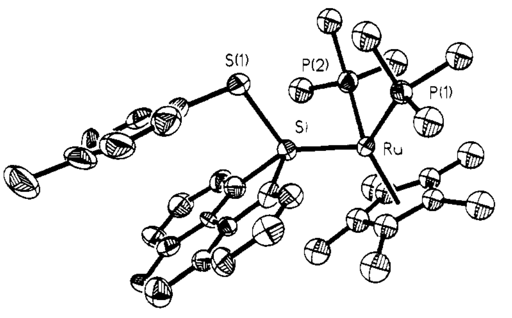
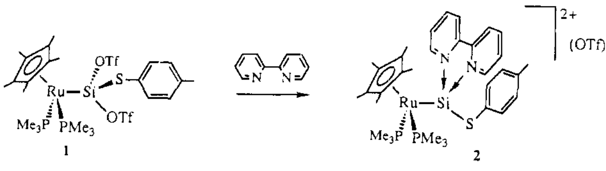


Figure 1. Structure of the cation of **3**. Selected interatomic distances (Å) and angles (deg): Ru–Si 2.269 (5), Si–S (1) 2.179 (7), Si–N(1), 1.91 (1), Si–N(2) 1.95 (1), Ru–P(1) 2.329 (5), Ru–P(2) 2.314 (5); P(1)–Ru–P(2) 91.2 (2), P(1)–Ru–Si 95.4 (2), P(2)–Ru–Si 94.0 (2), Ru–Si–S(1) 126.1 (2), S(1)–Si–N(1) 98.9 (5), S(1)–Si–N(2) 95.9 (5), Si–S–C(17) 106.3 (7), Ru–Si–N(1) 120.9 (4), Ru–Si–N(2) 122.9 (5), N(1)–Si–N(2) 81.5 (6).

(=SiR$_2$) and silylyne (≡SiR) derivatives. Here we report the displacement of both triflate groups in **1** to produce unusual silicon-based ligands that are formally described as base-stabilized silylynes and the reductive dimerization of one such complex via coupling of two phenanthroline rings. The donor-stabilized μ-silylyne complex [Cp$_2$Fe$_2$(CO)$_2$(μ-CO)(μ-Si^{t}Bu·*N*-methylimidazole)]I was very recently described by Ogino.[4]

Bipyridine displaces the triflate groups in **1** to give the air-stable, dark purple adduct **2** in 95% yield (eq 1). The phenanthroline



(1)

derivative [Cp*(PMe$_3$)$_2$RuSi(STol-*p*)(phen)](OTf)$_2$ (**3**) was prepared analogously and possesses very similar properties. Both **2** and **3** display electronic absorptions in the visible region at 534 and 514 nm, respectively, which probably correspond to metal-to-*N*-heterocycle or ligand-to-*N*-heterocycle charge-transfer transitions.[5] Note that the bipyridine adduct of :Si^{t}Bu$_2$ is similar in color (dark violet).[6]

X-ray quality crystals of **3** were grown by diffusion of ether into a concentrated 1,2-difluorobenzene solution. The molecular structure[7] (Figure 1) is characterized by a strong π-stacking interaction between the tolyl and phenanthroline groups (the tolyl centroid to phenanthroline plane distance is 3.30 Å). Intermolecular Cp*···tolyl π interactions also exist in the crystalline lattice (the Cp* centroid to tolyl plane distance is 3.61 Å), resulting in columns of stacked molecules. Evidence for π-stacking interactions in dichloromethane-d_2 solution is provided by ^{1}H NMR spectra, which contain resonances for the aromatic tolyl hydrogens of **2** and **3** that are shifted upfield by ca. 1 ppm compared to analogous resonances for **1**. It is not clear at present to what degree the intermolecular π-stacking interactions are present in solution, but we have noted that the UV–vis spectrum of **2** is not concentration dependent (dichloromethane solution).

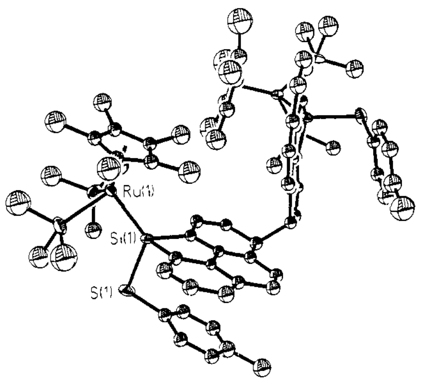
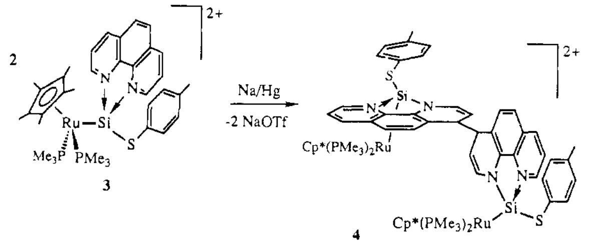


Figure 2. Structure of the cation of **4**. Selected interatomic distances (Å) and angles (deg): Ru(1)–Si(1) 2.281 (5), Ru(1)–P(1) 2.32 (1), Ru(1)–P(2) 2.31 (1), Ru(2)–Si(2) 2.281 (6), Ru(2)–P(3) 2.31 (1), Ru(2)–P(4) 2.30 (1), S(1)–Si(1) 2.19 (1), S(2)–Si(2) 2.18 (1), Si(1)–N(1) 1.82 (2), Si(1)–N(2) 1.93 (2), Si(2)–N(3) 1.81 (2), Si(2)–N(4) 1.91 (2); P(1)–Ru(1)–P(2) 92.5 (3), P(3)–Ru(2)–P(4) 91.8 (3), P(1)–Ru(1)–Si(1) 92.8 (2), P(2)–Ru(1)–Si(1) 94.0 (2), P(3)–Ru(2)–Si(2) 94.9 (3), P(4)–Ru(2)–Si(2) 92.9 (2), Ru(1)–Si(1)–S(1) 121.6 (3), Ru(2)–Si(2)–S(2) 123.5 (3), S(1)–Si(1)–N(1) 103.7 (6), S(1)–Si(1)–N(2) 96.2 (6), S(2)–Si(2)–N(3) 103.1 (5), S(2)–Si(2)–N(4) 94.6 (6), Si(1)–S(1)–C(29) 104 (1), Si(2)–S(2)–C(64) 104 (1), Ru(1)–Si(1)–N(1) 123.0 (5), Ru(1)–Si(1)–N(2) 121.1 (6), Ru(2)–Si(2)–N(3) 123.1 (6), Ru(2)–Si(2)–N(4) 119.1 (5), N(1)–Si(1)–N(2) 82.3 (7), N(3)–Si(2)–N(4) 83.7 (7).

The silicon atom in **3** has a severely distorted tetrahedral environment, such that the N–Si–N angle is 81.5 (6)° and the Ru–Si–S angle is 126.1 (2)°. Compound **3** is therefore structurally similar to the bis(HMPA) adduct (CO)$_4$FeSi(HMPA)$_2$Fe(CO)$_4$ ($\angle$O–Si–O = 92.1 (1)°, $\angle$Fe–Si–Fe = 122.6 (1)°),[8] and to the bipyridine adduct [(C$_5$H$_4$Me)(CO)$_2$Mn]Ge(bipy)[Mn(CO)$_2$-(C$_5$H$_4$Me)] ($\angle$N–Ge–N = 75.0 (3)°, $\angle$Mn–Ge–Mn = 138.7 (1)°).[9] The Ru–Si distance in **3** (2.269 (5) Å) is quite short,[2b] and the rather long Si–N separations of 1.95 (1) and 1.91 (1) Å reflect coordinate dative bond distances. For comparison, the phenanthroline adduct (Cl$_3$Si)$_2$SiCl$_2$·phen has Si–N distances of 1.991 (9) and 2.012 (11) Å,[10] whereas the normal covalent bond distances in (c-C$_6$H$_{11}$)NCH=CHN(c-C$_6$H$_{11}$)Si^{t}Bu$_2$ are 1.740 (2) and 1.744 (2) Å.[11] It therefore appears that **3** may be regarded as possessing a Si(IV) center, which is bonded to Ru and S via normal covalent bonds, and bonded to the two nitrogens via dative bonds.

In an attempt to produce a lower valent silicon center, complex **3** was stirred with sodium amalgam. After 9 days, this reaction gave compound **4** in 90% yield (eq 2), which results from a re-



(2)

ductive coupling of **3** via carbon–carbon bond formation. This compound was identified by X-ray crystallography, which due

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[3] Infrared ν(SO$_3$) vibrations for **1** are observed at 1271 cm^{-1} in acetonitrile solution (indicating ionic triflate) and at 1377 cm^{-1} in dichloromethane solution (indicating covalently bound triflate). Conductivities in dichloromethane and acetonitrile are 3 and 131 Ω cm^2 equiv^{-1}, respectively.[2a]

[4] Kawano, Y.; Tobita, H.; Ogino, H. *Angew. Chem., Int. Ed. Engl.* **1991**, *30*, 843.

[5] Vogler, A.; Kunkely, H. *Comments Inorg. Chem.* **1990**, *9*, 201.

[6] Weidenbruch, M.; Lesch, A.; Marsmann, H. *J. Organomet. Chem.* **1990**, *385*, C47.

[7] X-ray structure analysis of **3**: M_r = 1018.1; purple crystal (0.40 × 0.52 × 0.20 mm); monoclinic; space group $C2/c$; a = 18.212 (7), b = 17.817 (5), c = 28.049 (9) Å, β = 90.42 (3)° at 23 °C; V = 9100 (5) Å^3; Z = 8; D_x = 1.486 g cm^{-3}; λ(Mo Kα) = 0.71073 Å; $F(000)$ = 4176. A total of 5953 independent reflections ($2\theta_{max}$ = 45°), of which 3262 with $F > 6\sigma(F)$ were used for structure solution (direct methods) and refinement (full-matrix least squares); R = 8.66, R_w = 11.58. All non-hydrogen atoms except those of the PMe$_3$, Cp*, and OTf groups were refined anisotropically. The hydrogen atoms were calculated and fixed in idealized positions (d(C–H) = 0.96 Å, $U = 1.2U_{iso}$ for the carbon to which it was attached). One of the triflates was disordered with respect to rotation about the S–C bond. The disorder was modeled by refinement of the two distinct CF$_3$ configurations with site occupancy factors of 59% and 41%. Each disordered CF$_3$ was refined using fixed lengths for the S–C, C–F, and F–F distances.

[8] Zybill, C.; Wilkinson, D. L.; Müller, G. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 583.

[9] Ettel, F.; Huttner, G.; Imhof, W. *J. Organomet. Chem.* **1990**, *397*, 299.

[10] Kummer, D.; Chaudhry, S. C.; Depmeier, W.; Mattern, G. *Chem. Ber.* **1990**, *123*, 2241.

[11] Weidenbruch, M.; Lesch, A.; Peters, K. *J. Organomet. Chem.* **1991**, *407*, 31.

to significant crystal decay during data collection provided metrical parameters of limited accuracy.[12] However, a number of key structural features are readily apparent. Compound **4** may be described as a dimeric, base-stabilized silylene complex, as indicated by distinct Si–N distances for each silicon center (1.82 (2) and 1.93 (2) Å; 1.81 (2) and 1.91 (2) Å), corresponding to covalent and dative bonds. The length of the new C–C bond that links the two halves of the dimer is 1.57 (3) Å, which is most consistent with sp^3 character at the two carbon centers. Figure 2 illustrates how the monomer units are positioned in the dication of **4** and reveals the presence of tolyl group–phenanthroline π-stacking.

Note that this carbon–carbon coupling reaction of two phenanthroline rings is made possible by the conversion of one of the Si $\leftarrow$ N dative bonds in **3** to a Si–N normal covalent bond. Related processes, such as the reduction of alkylpyridinium salts by sodium amalgam to afford 4,4′-tetrahydrobipyridyl, have been reported.[13]

Acknowledgment is made to the National Science Foundation for their generous support. We thank Johnson Matthey Inc. for a loan of ruthenium, and T.D.T. thanks the Alfred P. Sloan Foundation for a research fellowship (1988–1992).

Supplementary Material Available: Experimental procedures and characterization data for **2–4**, a packing diagram for **4**, and tables of crystal, data collection, and refinement parameters, bond distances and angles, anisotropic displacement parameters, and hydrogen atom coordinates for **3** and **4** (25 pages); listings of observed and calculated structure factors for **3** and **4** (59 pages). Ordering information is given on any current masthead page.

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[12] X-ray structure analysis of **4**: M_r = 1738; purple crystal (0.30 × 0.30 × 0.33 mm); monoclinic; space group $P2_1/c$; a = 19.516 (7), b = 21.893 (7), c = 20.639 (7) Å, β = 112.36 (3)° at 23 °C; V = 8155 (5) $Å^3$; Z = 4; D_x = 1.416 g cm^{-3}; λ(Mo Kα) = 0.71073 Å; $F(000)$ = 3592. A total of 10726 independent reflections ($2\theta_{max}$ = 45°). The decay of monitored reflections was ca. 50% during 146 h of X-ray exposure, and an appropriate scale factor was applied to account for the decay. A total of 4280 reflections with $F > 4\sigma(F)$ were observed and used for structure solution (Patterson method) and refinement (full-matrix least squares); R = 9.51, R_w = 10.34. The Ru, P, and Si atoms were refined anisotropically. The hydrogen atoms were calculated and fixed in idealized positions (d(C–H) = 0.96 Å, $U = 1.2U_{iso}$ for the carbon to which it was attached). One of the triflates was disordered and the S–C bond was fixed at 1.80 Å.

[13] Eisner, U.; Kuthan, J. *Chem. Rev.* **1972**, *72*, 1.

Room Temperature Isomerization of Siloxycyclopropanes to Silyl Ethers of 2-Methylenealkanols Catalyzed by Zeise's Dimer

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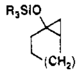
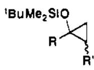
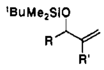
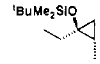
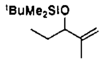
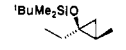
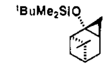
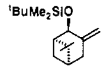
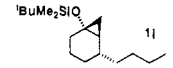
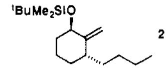
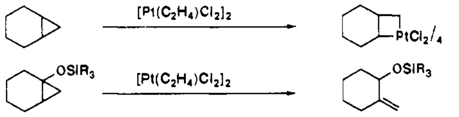
The transition-metal-promoted isomerization of cyclopropanes has attracted much attention for the past two decades.[1] Investigations using rhodium[2] and iridium[3] catalysts have been successful, but the utility of these catalysts often suffered from drastic conditions and poor stereo- and regioselectivity. On the other hand, very few publications have appeared which deal with the catalytic isomerization of cyclopropanes by platinum complexes.[4] The main reason for this may be the formation of well-known *stable* platinacyclobutane complexes (eq 1).[5] In this communication, we report an efficient catalytic isomerization achieved by the introduction of a siloxy group onto a cyclopropane ring (eq 2). This reaction proceeds smoothly *at ambient temperature* and is quite general for 2-alkyl-substituted siloxycyclopropanes **1**.[6] Furthermore, the isomerization exhibits complete regio- and stereoselectivity to give allyl silyl ethers **2**.

Table I. Pt(II)-Catalyzed Isomerization of Siloxycyclopropanes **1** to Allyl Silyl Ethers **2**[a]

entry	substrate	No.	product	No.	yield (%)[b]
					
1	n = 1, R_3Si = ${}^tBuMe_2Si$	1a	n = 1, R_3Si = ${}^tBuMe_2Si$	2a	96
2	n = 2, R_3Si = ${}^tBuMe_2Si$	1b	n = 2, R_3Si = ${}^tBuMe_2Si$	2b	96
3	n = 2, R_3Si = Me_3Si	1c	n = 2, R_3Si = Me_3Si	2c	74
4	n = 3, R_3Si = ${}^tBuMe_2Si$	1d	n = 3, R_3Si = ${}^tBuMe_2Si$	2d	73
					
5	R = Pr, R′ = Et	1e[c]	R = Pr, R′ = Et	2e	88
6	R = H, R′ = ^{i}Pr	1f[d]	R = H, R′ = ^{i}Pr	2f	71
7		1g		2g	83
8		1h		2g	89
9[e]		1i		2i	72
10		1j		2j	89

[a] Reactions were conducted in $CHCl_3$ using 2–5 mol % of $[Pt(C_2H_4)Cl_2]_2$ at 20 °C for 0.5–10 h. [b] Isolated yields after chromatographic purification. [c] E/Z = 50/50. [d] E/Z = 82/18. [e] Using 10 mol % of $[Pt(C_2H_4)Cl_2]_2$.



(1)
(2)

In a preliminary experiment, we attempted the stoichiometric reaction of bicyclic siloxycyclopropane **1b** with Zeise's dimer in $CHCl_3$ at room temperature. Isomerization of **1b** took place immediately to give an *exo*-methylene-type allyl silyl ether **2b**, quantitatively. This result stands in sharp contrast to our earlier study[7] on the reaction of 1-aryl-1-siloxycyclopropanes with Zeise's dimer, wherein β-platinum ketone complexes were formed with liberation of chlorosilane. Thus, we tested the catalytic isomer-

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