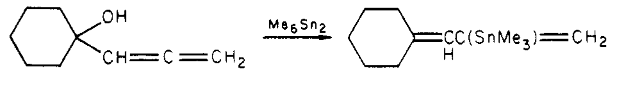
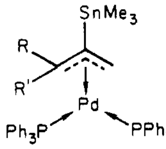
hexyl)allene gives the monostannylbutadiene **3**, possibly due to elimination of trimethyltin hydroxide. The regiospecific or regioselective addition noted here was indicated in the previous report of disilane addition:[2] allene and methylallene (1,2-butadiene) reacted with disilanes $X_{3-m}Me_mSiSiMe_nX_{3-n}$ (X = Cl, OMe; m, n = 0–3), methylallene giving only the product corresponding to the "kinetic" ditin adduct.



3

Though no mechanistic examination of the reaction has been carried out, we assume that (π-allyl)palladium intermediates such as **4** and/or **5** are involved. We can safely presume that the primary palladium species formed are PdL_2·allene[4] and $PdL_2·Me_6Sn_2$ (probably trans, as in the platinum case).[5] The existence of a species such as **4** is supported by the observation that at high temperatures allene itself gives a byproduct that is clearly formed by dimerization of the allyl groups. If **5** is formed with the cis geometry shown and with the π-allyl ligand in the plane of the complex, it can exist in two forms when the allene is not symmetrically substituted. When R and R′ are not too large, the form shown (which leads to the kinetic product) is likely to be favored on steric grounds, but heating can lead via the second form to the thermodynamic product.



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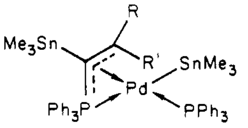

5

The products of addition of hexamethylditin to allenes offer considerable potential in organic synthesis, since they contain both vinylic and allylic trimethyltin residues: the chemistry of the 2,3-bis(trimethylstannyl)-1-propenes is under active investigation.

Acknowledgment. This work was supported by the Deutsche Forschungsgemeinschaft and the "Fonds der Chemischen Industrie".

Registry No. **1** (R = R′ = R″ = H), 90886-11-6; **1** (R = Ph, R′ = R″ = H), 90886-12-7; (*Z*)-[1] (R = R′ = H, R″ = Ph), 90886-13-8; (*E*)-**1** (R = R′ = H, R″ = Ph), 90886-14-9; **1** (R = MeO, R′ = R″ = H), 90886-15-0; (*E*)-**1** (R = R′ = H, R″ = MeO), 90886-16-1; (*Z*)-**1** (R = R′ = H, R″ = MeO), 90886-17-2; **1** (R = Me_3Sn, R′ = R″ = H), 90886-18-3; (*Z*)-**1** (R = R′ = H, R″ = Me_3Sn), 90886-19-4; **1** (R = R′ = Me, R″ = H), 90886-20-7; (*Z*)-**1** (R = R′ = R″ = Me), 90886-25-2; **2** (R = Ph, R′ = R″ = H), 90886-26-3; **2a** (R = Me_3Sn, R′ = R″ = H), 90886-27-4; **3**, 90886-29-6; $Pd(PPh_3)_4$, 14221-01-3; Me_6Sn_2, 661-69-8; $(H_2C{=}C(SnMe_3)(CH_2)_2$, 90886-28-5; $Me_3SnCH_2C(SnMe_3){=}CMe_2$, 90886-21-8; *cis*-$Me_3SnCH_2C(SnMe_3){=}C(Me)Ph$, 90886-22-9; *trans*-$Me_3SnCH_2C(SnMe_3){=}C(Me)Ph$, 90886-23-0; $Me_3SnCH(Me)C(SnMe_3){=}CMe_2$, 90886-24-1; $H_2C{=}C{=}CH_2$, 463-49-0; $PhCH{=}C{=}CH_2$, 2327-99-3; $MeOCH{=}C{=}CH_2$, 13169-00-1; $Me_3SnCH{=}C{=}CH_2$, 4104-88-5; $Me_2C{=}C{=}CH_2$, 598-25-4; $PhC(Me){=}C{=}CH_2$, 22433-39-2; $Me_2C{=}C{=}CHMe$, 3043-33-2; 1-(1,2-propadienyl)cyclohexanol, 34761-56-3.

Supplementary Material Available: Tables of elemental analyses and ^1H and ^{13}C NMR data (7 pages). Ordering information is given on any current masthead page.

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Silylene–Disilene Isomerizations. A Theoretical Study

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Summary: Transition states and barrier heights for the title reactions via 1,2-silyl and 1,2-methyl shifts are investigated through ab initio calculations. The 1,2-silyl shift in silylenes to disilenes is found to proceed at room temperature, this being in agreement with the recent experiment. Also discussed are the structures of disilenes.

There is considerable current interest in the possible interconversion of divalent and doubly bonded silicon compounds. Several examples of the silylene–silene isomerizations (via 1,2-hydrogen[1] and 1,2-silyl[2] shifts) have been documented in the last few years. However, the unimolecular reactions are likely to proceed only at high temperature, as the calculated barrier heights[3] as well as the further experimental studies[4] reveal. In contrast, recently Sakurai and co-workers[5] have found that a silylene, $Me\ddot{S}iSiMe_2(SiMe_3)$ (**1**), isomerizes rapidly to a disilene, $(SiMe_3)MeSi{=}SiMe_2$ (**2**): this is the first example at room temperature. We report here the ab initio calculations of the transition states and barrier heights for the interconversion reactions (1) and (2).[6]

$$\underset{\mathbf{3}}{H\ddot{S}iSiH_2(SiH_3)} \rightleftharpoons \underset{\mathbf{4}}{(SiH_3)HSi{=}SiH_2} \tag{1}$$

$$\underset{\mathbf{5}}{H\ddot{S}iSiH_2(CH_3)} \rightleftharpoons \underset{\mathbf{6}}{(CH_3)HSi{=}SiH_2} \tag{2}$$

All calculations were for closed-shell singlets. Geometries were fully optimized at the Hartree-Fock level with the 6-31G[7] energy gradient method. Energies were improved with the larger 6-31G* basis set[7] using third-order Møller–Plesset perturbation theory.[8]

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(3) The barriers (ca. 41 kcal/mol) for $H\ddot{S}iCH_3 \rightleftharpoons H_2Si{=}CH_2$: (a) Goddard, J. D.; Yoshioka, Y.; Schaefer, H. F. *J. Am. Chem. Soc.* **1980**, *102*, 7644. (b) Yoshioka, Y.; Schaefer, H. F. *J. Am. Chem. Soc.* **1981**, *103*, 7366. The barriers (41–47 kcal/mol) for $CH_3\ddot{S}iCH_3 \rightleftharpoons (CH_3)HSi{=}CH_2$: (c) Nagase, S.; Kudo, T. *J. Chem. Soc., Chem. Commun.* **1984**, 141. The barriers (25–26 kcal/mol) for $H\ddot{S}iCH_2(SiH_3) \rightleftharpoons (SiH_3)HSi{=}CH_2$: (d) Nagase, S.; Kudo, T., submitted for publication in *J. Chem.* Soc., *Chem. Commun.* It is to be noted that the barriers for silylenes → silenes are all calculated to be almost equal to those for the reverse reactions. For a germylene–germene isomerization, see: (e) Nagase, S.; Kudo, T. *Organometallics* **1984**, *3*, 324. For a silylene–silanone isomerization, see: (f) Kudo, T.; Nagase, S. *J. Organomet. Chem.* **1983**, *253*, C23; *J. Phys. Chem.*, in press.

(4) (a) Conlin, R. T.; Gill, R. S. *J. Am. Chem. Soc.* **1983**, *105*, 618. (b) Arrington, C. A.; West, R.; Michl, J. *Ibid.* **1983**, *105*, 6176. These authors interpreted that the facile isomerization observed at 100 K is catalyzed rather than being unimolecular.

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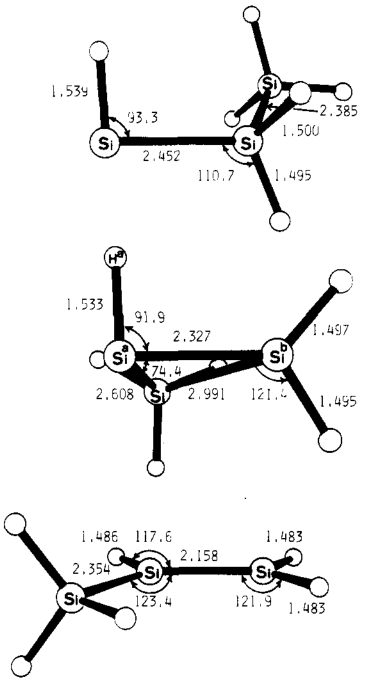

Figure 1. ORTEP drawings of the optimized geometries of **3** (top), **4** (bottom), and the transition state for the 1,2-silyl group shift, in angstroms and degrees.

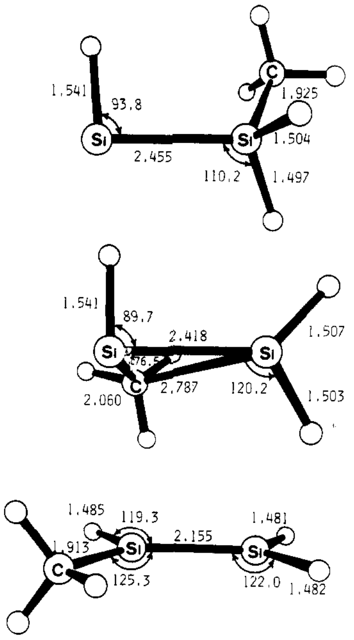

Figure 2. ORTEP drawings of the optimized geometries of **5** (top), **6** (bottom), and the transition state for the 1,2-methyl group shift, in angstroms and degrees.

The ORTEP drawings of optimized geometries of **3**, **4**, and the transition state connecting them are shown in Figure 1. In the notation of $\mathrm{H^a\ddot{S}i^aSi^bH_2(SiH_3)}$ and $\mathrm{(SiH_3)H^aSi^a{=}Si^bH_2}$, the silyl group shifts diagonally across the $\mathrm{Si^aSi^b}$ bond to accomplish the interconversion. In the transition state the shifting group is positioned almost under the $\mathrm{Si^aSi^b}$ bond, as characterized by the $\mathrm{SiSi^aSi^bH^a}$ dihedral angle of 95.6°, but away from the perpendicular bisector of the $\mathrm{Si^aSi^b}$ bond in a way that the Si atom in the silyl group is 0.383 Å nearer to $\mathrm{Si^a}$ than to $\mathrm{Si^b}$. The $\mathrm{SiSi^a}$ distance of 2.608 Å is only 0.254 Å longer than the value (2.354 Å) in **4** while the $\mathrm{SiSi^b}$ distance of 2.991 Å is 0.606 Å longer than the value (2.385 Å) in **3**. As also seen in several of the remaining geometrical parameters except for the $\mathrm{H^aSi^aSi^b}$ angle, the transition state is closer to **4** rather than to **3**.

Nevertheless, the transition state for the 1,2-silyl shift lies 18.2 kcal/mol above **4** and 8.5 kcal/mol above **3**. The barrier for reaction **4** → **3** is 2.14 times larger than that for the reverse reaction. Correction for zero-point vibrational energies[9] can reduce the barriers only very slightly to 17.2 (**4** → **3**) and 8.4 (**3** → **4**) kcal/mol. Most interesting is the small barrier[10] for the 1,2-silyl shift in **3** to **4** that is likely to be surmountable at room temperature with a considerable rate.[11] This finding is comparable to the apparent observation of the rapid isomerization of **1** to **2** at room temperature. In contrast, the barrier for **4** → **3** is somewhat too large for the reaction to occur at room temperature,[11] suggesting that disilenes are kinetically more stable to isomerization than silylenes. In fact, the isomerization of **2** back to **1** was not observed at 15 ± 2 °C by Sakurai et al., though was found to proceed at an elevated temperature (300 °C).[5]

There is the possibility that via the 1,2-methyl shift **1** isomerizes to **2**. To theoretically check this, we have undertaken reaction **5** → **6**. As Figure 2 shows, the feature of the geometrical changes in the 1,2-methyl shift is essentially the same as that in Figure 1. However, the calculated barrier for **5** → **6** is as large as 27.8 kcal/mol and excludes the methyl shift mechanism in the formation of **2** from **1** at room temperature. As the sizable barrier (34.7 kcal/mol) for **6** → **5** also suggests, methyl groups are much more reluctant to migrate in disilenes and silylenes than are silyl groups.[12] Several years ago, Barton and co-workers[13] claimed that tetramethyldisilene isomerized rapidly to (trimethylsilyl)methylsilylene. This is not surprising in the high-temperature experiment (700 °C). However, they could find no evidence for the reverse 1,2-methyl shift under the condition: this may conflict with our expectation that its barrier is rather smaller.

Finally, the structures of disilenes are worth mentioning. The equilibrium structures of **4** and **6** are in C_s symmetry with a planar disilene framework, as shown in Figures 1 and 2. These differ significantly from the equilibrium structure of $\mathrm{H_2Si{=}SiH_2}$ since the parent compound has been predicted to adopt a trans-bent C_{2h} form (not a planar D_{2h} form).[14] At this point, it is interesting to note that the two silicon atoms and four attached carbons in 1,2-di-*tert*-butyl-1,2-dimesityldisilene are recently found to be coplanar by the X-ray crystal study,[15a] while the silicon

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[9] Because of the size of the reaction system, harmonic vibrational frequencies at the 3-21G level were used to compute zero-point energies: 31.5 (**3**), 32.3 (**4**), and 31.3 (transition state) kcal/mol.

[10] Since it is likely that calculations at higher levels will reduce the size of the barrier, the present value may be an upper limit to the experimental one.

[11] On the basis of the structures, energies, and frequencies obtained from the present ab initio calculations, the enthalpy and entropy of activation for reaction **3** → **4** are evaluated to be 7.8 kcal/mol and −5.1 eu, respectively, at room temperature, while those for **4** → **3** are 16.8 kcal/mol and −3.6 eu. Within the framework of conventional transition-state theory, the evaluated thermodynamic quantities allow us to estimate the rate constants on the order of 8.6×10^5 (**3** → **4**) and 4.7×10^{-1} (**4** → **3**) $\mathrm{s^{-1}}$ at high pressures.

[12] In addition, note that silyl groups are much more mobile in disilenes and silylenes than in silenes and silylenes.[3d]

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atoms in tetramesityldisilene are moderately anti-pyramidalized.[15] It seems that the planarity of disilene frameworks is sensitive to substitution.

Acknowledgment. We are grateful to Prof. Sakurai for helpful discussions and to Prof. West for making ref 15 available prior to publication. All calculations were carried out at the Computer Center of the Institute for Molecular Science, using an IMS version of the GAUSSIAN80 series of programs.[16]

Registry No. 3, 90968-70-0; 4, 90968-71-1; 5, 88867-25-8; 6, 88867-24-7.

Supplementary Material Available: A listing of the optimized geometries in Figures 1 and 2 (3 pages). Ordering information is given on any current masthead page.

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Synthesis, Characterization, and Reactivity of $HRu_3(CO)_{10}(CH)$ and $H_2Ru_3(CO)_9(CCO)$

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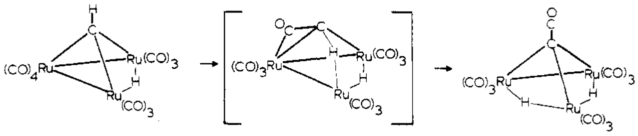
Summary: Treatment of $HRu_3(CO)_{10}(COMe)$ with $LiHBEt_3$ at −78 °C forms a formyl species, which above −60 °C decomposes into a mixture of $[H_2Ru_3(CO)_9(COMe)^-]$ and $[HRu_3(CO)_{10}(CHOMe)^-]$. Low-temperature extraction of this mixture provides $HRu_3(CO)_{10}(CH)$, which rearranges readily ($t_{1/2}$ = 9 min at −12 °C) into $H_2Ru_3(CO)_9(CCO)$. The latter complex appears analogous to $H_2Os_3(CO)_9(CCO)$.

Recent results have focused attention on the connection between methylidyne (≡CH) and carbonylmethylidyne (≡CCO) ligands. Thus, $HOs_3(CO)_{10}(CH)$[1] rearranges thermally into $H_2Os_3(CO)_9(CCO)$[2] and $[Fe_3(CO)_9(CCO)^{2-}]$ gives $[Fe_3(CO)_{10}(CH)^-]$ upon protonation.[3] $HOs_3(CO)_{10}(CH)$[1] was prepared by a hydride addition/protonation sequence from $HOs_3(CO)_{10}(COMe)$. We wish to describe aspects of analogous work with $HRu_3(CO)_{10}(COMe)$, which results in the synthesis of $HRu_3(CO)_{10}(CH)$ and allows observation of its facile transformation into $H_2Ru_3(CO)_9(CCO)$.

Treatment of $HRu_3(CO)_{10}(COMe)$ with $LiHBEt_3$ or $KHB(O\text{-}i\text{-}Pr)_3$ in THF at −78 °C appears to form the formyl complex $[HRu_3(CO)_9(COMe)(CHO)^-]$ (^1H NMR δ −12.2 (1 H), 14.1 (1 H)). This species is stable indefinitely below −60 °C (no change after 20 h at −65 °C), but above −60 °C both decarbonylation to give $[H_2Ru_3(CO)_9(COMe)^-]$ (δ −17.2 (s, 2 H)) and hydride transfer to give $[HRu_3(CO)_{10}(CHOMe)^-]$ (δ −12.5 (1 H), 8.4 (1 H)) occur concomitantly. The yield of $[HRu_3(CO)_{10}(CHOMe)^-]$ is maximized (55% by NMR) by maintaining the mixture at <−40 °C for an extended period (up to 160 h). Evaporating the THF and adding dry ether to the dark red residue (maintaining a low temperature) provides an orange solution and an immiscible red oil. The latter is mostly $[H_2Ru_3(CO)_9(COMe)^-]$ together with $[HRu_3(CO)_{10}(CHOMe)^-]$, and addition of $HBF_4 \cdot Et_2O$ gives an inseparable mixture of $HRu_3(CO)_{10}(CH)$ and $H_3Ru_3(CO)_9(COMe)$.[4] However, evaporation of the orange ether solution (−30 °C) provides essentially pure $HRu_3(CO)_{10}(CH)$ as an orange-yellow solid, which has been characterized spectroscopically.[5]

Scheme I



The methylidyne proton NMR resonance for $HRu_3(CO)_{10}(CH)$ occurs at δ 12.64, which is upfield of that for $HOs_3(CO)_{10}(CH)$ (δ 14.16)[1] but comparable with that for $[Fe_3(CO)_{10}(CH)^-]$ (δ 12.24).[3] The infrared spectrum of $HRu_3(CO)_{10}(CH)$ shows a band at 1902 cm^{-1}, which indicates the presence of a weakly bridging carbonyl and points to an essentially symmetric, triply bridging position for the methylidyne ligand. Such a symmetric, triply bridging structure has been postulated for $HFe_3(CO)_{10}(CCH_3)$[6] and established for $HRu_3(CO)_8(1,3\text{-}C_6H_8)(COMe)$;[7] the infrared spectrum in each case also exhibits a low-frequency band near 1900 cm^{-1}. The methylidyne ligand in $HOs_3(CO)_{10}(CH)$ is "semi" triply bridging, and the compound reacts readily at −60 °C with 1 equiv of 4-methylpyridine to yield $HOs_3(CO)_{10}(CHNC_5H_4CH_3)$.[1] In contrast, $HRu_3(CO)_{10}(CH)$ does not react with even large excesses of 4-methylpyridine or similar nucleophiles.

Near 0 °C $HRu_3(CO)_{10}(CH)$ rearranges rapidly to $H_2Ru_3(CO)_9(CCO)$.[8] The half-life at −12 °C is 9 min, $k = 1.3 \times 10^{-3}\ s^{-1}$ or $\Delta G^{\ddagger}_{261}$ = 19.5 kcal/mol. The analogous $HOs_3(CO)_{10}(CH)$ to $H_2Os_3(CO)_9(CCO)$ rearrangement has $\Delta G^{\ddagger}_{333}$ = 26 kcal/mol.[9] Scheme I illustrates an intermediate for the transformation [Ru–CO; ≡CH] → [Ru–H; ≡CCO], in which both migrating ligands (H and CO) are bridged between carbon and ruthenium. An analogous pathway can be proposed for the osmium system, which in addition would explain why neither $HOs_3(CO)_{10}(CSiMe_3)$[10a] nor $HOs_3(CO)_{10}(CPh)$[10b] shows any tendency to couple intramolecularly with a CO ligand. Furthermore, a closely similar intermediate could be the first species formed in the protonation of $[Fe_3(CO)_9(CCO)^{2-}]$[3b] (pro-

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(5) $HRu_3(CO)_{10}(CH)$: ^1H NMR (360 MHz, $CDCl_3$, −30 °C) δ −15.89 (s, 1 H), 12.64 (s, 1 H); IR (−20 °C, C_5H_{12}) ν_{CO} 2085 (sh), 2066 (s), 2054 (m), 2031 (vs), 2018 (s), 2004 (m), 1902 (m) cm^{-1}.

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(8) $H_2Ru_3(CO)_9(CCO)$: FI mass spectrum (^{102}Ru), m/z 600 (M^+); IR (C_6H_{12}) ν_{CO} 2120 (m), 2084 (s), 2056 (vs), 2038 (w), 2030 (vw), 2010 (m), 2000 (w), 1965 (vw) cm^{-1}; ^1H NMR ($CDCl_3$) δ −18.08 (s). Anal. Calcd for $Ru_3C_{11}H_2O_{10}$: C, 22.11; H, 0.33. Found: C, 22.32; H, 0.50.

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