$[Hg(CH_2CH_2OCH_2CH_2)]_2$. The Hg atoms in **1a** lie on the *c* axis in the lattice with a Hg–Hg separation of 4.59 Å and with weak intermolecular Hg–S contacts, 3.20 and 3.16 Å, respectively. We believe that **1a** is the first structurally characterized two-coordinate ylide Hg^{II} complex. A linear two-coordinate geometry was suggested for the cations $[Hg(CH_2PR_3)_2]^{2+}$ (R = Ph, Me) reported previously by Seyferth[9] and Schmidbaur et al.[9]

As found for **1b**, **1a** is an excellent synthetic precursor for bimetallic complexes. For example, **1b** reacts readily with Au(THT)Cl (THT = tetrahydrothiophene) in CH_2Cl_2 to product[1,10] $Au_2(CH_2P(S)Ph_2)_2$ (**3b**),[1,10] while **1a** also reacts readily with Au(THT)Cl in the presence of $TlPF_6$ to yield the new bimetallic compound $[HgAu(CH_2P(S)Ph_2)_2]PF_6$ (**2a**) in 60% yield.[5] However, unlike **1b** which shows a rearrangement of the Au–C bond to form **3b**, **1a** yields **2a** without the rearrangement of Hg–C bonds (Scheme I). **2a** was fully characterized by ^1H NMR, ^{31}P NMR, elemental,[5] and X-ray diffraction analyses.[7] The molecular structure of **2a** is shown in Figure 2. As found in **1a**, the Hg^{II} center is linearly coordinated to two carbon atoms. This result is consistent with the ^1H NMR spectrum of **2a** which shows only one methylene signal and ^{199}Hg coupling satellites (Table I). The Au^I center in **2a** is bonded linearly to two sulfur atoms with normal Au–S bond distances. The Au–Hg separation is 3.088 Å, similar to the sum of metallic radii of Au and[11] Hg, 3.009 Å. The fact that **2a** is the only product from the reaction of **1a** with Au(THT)Cl and **3b** the only product from **1b** with Au(THT)Cl suggests that the Au–C bonds in **1b** are more labile than the Hg–C bonds in **1a**. This lability of Au–C bonds in **1b** is further demonstrated by the specific synthesis of compound **3a**, $[HgAu(CH_2P(S)Ph_2)_2]PF_6$, from **1b**.

Compound **3a** was obtained as the only product from the reaction of **1b** with $HgCl_2$ in the presence of $TlPF_6$ in CH_2Cl_2 in 69% yield.[5] **3a** is isomeric with **2a** and similar to **3b** in structure. A drawing of its structure is shown in Figure 3. Each metal atom in **3a** is bonded linearly to a methylene group and a sulfur atom with similar M–S and M–C bond lengths. This bonding mode agrees with the ^1H NMR spectrum of **3a** in solution in which two strikingly different chemical shifts of methylene groups are observed. Only one methylene group is coupled to the ^{199}Hg nucleus (Table I). The Hg–Au distance, 2.989 (1) Å, is shorter than that found in **2a** or the sum of metallic radii of Hg and Au atoms. Interestingly, **3a** dimerizes in the solid and forms a tetranuclear species. One-half of the dimer is related to the other half by a 2-fold symmetry axis. Hg and Au atoms are experimentally crystallographically indistinguishable. The intermolecular metal–metal distance of 3.150 (2) Å is similar to the intermolecular Au–Au separations in[1,3] $Au_2Pt(CH_2P(S)Ph_2)_4$, $Au_2Pb(CH_2P(S)Ph_2)_4$, and[10] $Au_2(CH_2PnS)Ph_2)_2$. On the basis of the fact that Au^I complexes tend to form short intermolecular Au–Au contacts in the solid[12] and that Hg atoms in **3a** are in a higher oxidation state (+2) than the Au atoms (+1) and therefore they would prefer to be further apart, the intermolecular M–M distance was assigned to a Au^I–Au^I distance.

2a and **3a** are the first examples of organometallic complexes containing both of Au and Hg atoms with $5d^{10}$ electronic configurations. These two compounds do not interconvert under reflux in THF.

The chemical reactivity of these new compounds and syntheses of other new bimetallic complexes using $Hg(CH_2P(S)Ph_2)_2$ as a precursor are currently under investigation in our laboratory.

Acknowledgment. We thank the Welch Foundation and the National Science Foundation (CHE 8708625) for financial support. The available fund of Texas A&M University has supported the crystallographic studies. We thank a referee for suggesting **1a** be refined in the space group *C*2/*c*.

Supplementary Material Available: Tables of positional and thermal parameters, bond lengths and angles, and crystallographic data for **1a**–**3a** (13 pages); listings of structure factors for **1a**–**3a** (57 pages). Ordering information is given on any current masthead page.

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Reaction of Primary Silanes with a Binuclear Rhodium Complex. Formation of μ-Silylene Complexes and Rapid Si–H/Rh–H Exchange

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Summary: The primary silanes $PhSiH_3$ and $EtSiH_3$ react with the binuclear complex $Rh_2H_2(CO)_2(dppm)_2$ to produce in sequence the μ-SiRH dihydride species $Rh_2(\mu\text{-}SiRH)H_2(CO)_2(dppm)_2$ and the bis μ-SiRH complex $Rh_2(\mu\text{-}SiRH)_2(CO)_2(dppm)_2$. The latter for R = Ph has been found by a single-crystal structure determination [$P\bar{1}$; a = 12.927 (5) Å, b = 15.165 (2) Å, c = 18.280 (8) Å; α = 93.8 (5)°, β = 103.6 (3)°, γ = 107.8 (9)°; Z = 2; d_{calcd} = 1.42 g/cm^3; R = 0.047, R_w = 0.059] to have a cradlelike structure with μ-SiPhH bridges of 2.35 (1) Å average Rh–Si distance and a Rh–Rh single bond of 2.813 (1) Å. The complexes $Rh_2(\mu\text{-}SiRH)H_2(CO)_2(dppm)_2$ are fluxional with Si–H and Rh–H protons undergoing rapid exchange, indicating the relative reactivity of the coordinated Si–H bond in these systems. For R = Ph, $\Delta G^{\ddagger}$ for the fluxional process is 12 ± 1 kcal/mol.

The reactivity of Si–H bonds with transition-metal complexes[1] has stimulated interest in dehydrogenative coupling of primary and secondary silanes, R_nSiH_{4-n} (n = 1, 2; R = alkyl, aryl), as a reaction path to the formation of Si–Si bonds. Recently, Harrod and co-workers have employed this approach using $Cp_2TiR'_2$ (R′ = Me, CH_2Ph) to obtain $(RSiH)_x$ with x = ~10.[2] In the course of this work, they obtained binuclear intermediates having μ-$SiRH_2$ groups (R = Ph) and observed Si–H/hydride ex-

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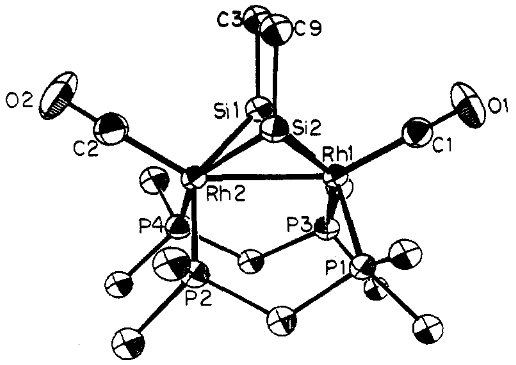

Figure 1. A perspective view of $Rh_2(\mu\text{-}SiPhH)_2(CO)_2(dppm)_2$ (**2**). Only the ipso carbon atoms of the phenyl groups are shown. Important bond distances (Å) and angles (deg): Rh1-Si1, 2.347 (1); Rh2-Si1, 2.354 (2); Rh1-Si2, 2.368 (2); Rh2-Si2, 2.335 (1); Rh1-Rh2, 2.813 (1); Rh1-C1, 1.864 (6); Rh2-C2, 1.863 (6). P1-Rh1-Si1, 148.52 (5); P3-Rh1-Si2, 146.00 (5); P2-Rh2-Si1, 145.70 (5); P4-Rh2-Si2, 148.83 (5); Si1-Rh1-Si2, 73.78 (5); Si1-Rh2-Si2, 74.25 (5); Rh1-Si1-Rh2, 73.52 (4); Rh1-Si2-Rh2, 73.47 (4).

change.[3] Dehydrogenative coupling of silanes using platinum group complexes to yield Si–Si dimers and small oligomers has also been reported by Brown-Wensley,[4] Corey,[5] and Tessier-Youngs and Youngs.[6] In the last of these reports, the silylene-bridged complexes $Pt_2(\mu\text{-}SiXPh)(\mu\text{-}SiYPh)(PEt_3)_4$ (X = Y = H, Cl; X = H, Y = Cl) were crystallographically characterized and shown to be active in the oligomerization reaction. The Si···Si distances in these structures, 2.57–2.60 Å, were taken to suggest nascent Si–Si bond formation. In this paper we describe the reaction of the primary silanes $PhSiH_3$ and $EtSiH_3$ with the binuclear rhodium complex $Rh_2H_2(CO)_2(dppm)_2$ (**1**; dppm = bis(diphenylphosphino)methane),[7] leading to the formation of fluxional μ-SiRH hydride intermediates and stable bis μ-SiRH products which may be relevant to promoting Si–Si bond formation by platinum group complexes.

The reaction of **1** with $PhSiH_3$ in C_6H_6 at 60 °C for 24 h results in the formation of colorless crystals, **2**, whose formulation as $Rh_2(\mu\text{-}SiPhH)_2(CO)_2(dppm)_2$ was established by spectroscopic characterization and X-ray crystallography.[8] The molecular structure of **2** is shown in Figure 1. Complex **2** contains two bridging phenylsilylene units, each bonded to two Rh atoms with an average distance of 2.351 (10) Å. The two Rh atoms are also joined directly by a single bond of length 2.813 (1) Å as well as by bridging dppm ligands. The overall geometry of **2** is of the $Co_2(\mu\text{-}RC{\equiv}CR)(CO)_6$ type assuming two coordination sites for the $\mu\text{-}RC{\equiv}CR$ ligand[9] and is structurally very similar to the "cradle" complexes $Rh_2(\mu\text{-}PhC{\equiv}CPh)(CO)_2(dppm)_2$ and $Ir_2(\mu\text{-}RNC)_2(RNC)_2(dmpm)_2$ where R = 2,6-xylyl.[10] The Si···Si separation in the structure is 2.75 Å which while longer than a Si–Si single bond is substantially shorter than a van der Waals contact. This 2.75 Å value is much closer to the 2.57–2.60 Å range found for $Pt_2(\mu\text{-}SiXPh)(\mu\text{-}SiYPh)(PEt_3)_4$ than the 3.85–4.22 Å range calculated for other bis μ-SiRR′ structures.[6,11] Also notable in the structure is the diaxial or cis orientation of the Ph substituents of the silylene ligands.

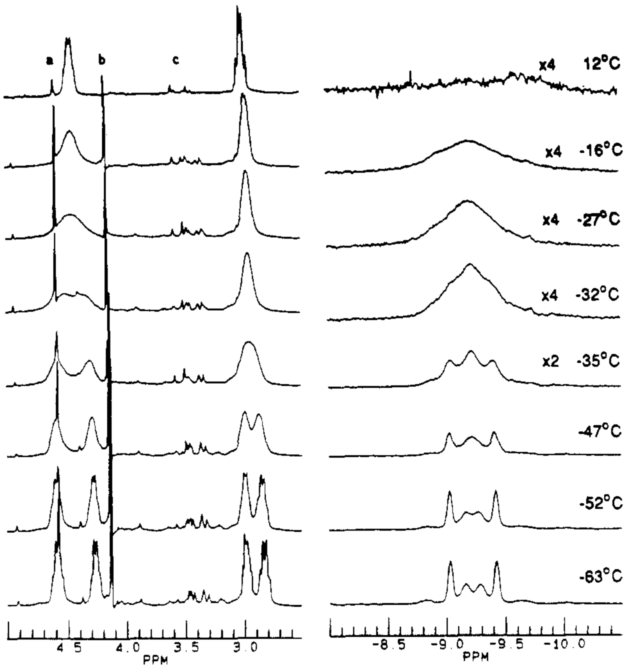

Figure 2. Variable-temperature ^1H NMR spectrum of **3** formed in situ in CD_2Cl_2. The resonances marked a, b, and c correspond to dissolved H_2, free $PhSiH_3$ and a complex impurity, respectively.

If the reaction between **1** and $PhSiH_3$ is carried out in CH_2Cl_2 at -30 °C and stopped within 10 min, an intermediate in the formation of **2** can be obtained. Specifically, removal of solvent and addition of hexane allows isolation of a yellow-orange solid, **3**, which shows in the ^1H NMR spectrum a broad resonance spanning 2 ppm at $\sim$-4.1 ppm and two dppm methylene resonances.[12a] Upon cooling, the former resonance resolves into two highly coupled patterns at δ -9.23 and 6.33 in a 2:1 intensity ratio assignable to Rh–H and Si–H resonances, respectively, while the dppm CH_2 protons split into four inequivalent signals.[12b]

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(8) (a) **Spectroscopic data for 2**: ^1H NMR (CD_2Cl_2, 298 K, ppm) 7.78 (m, 8 H, Ph), 7.33 (d, 4 H, Ph), 6.7–7.2 (m, 38 H, Ph), 5.65 (m, 2 H, SiH), 4.68 (m, 2 H, CH_2), 3.22 (m, 2 H, CH_2); ^{31}P NMR (CD_2Cl_2, 298 K, ppm) 12.50 (symmetric mult); IR (KBr) $\nu_{SiH} = 2063\ cm^{-1}$, $\nu_{CO} = 1934\ cm^{-1}$. **Crystal and X-ray data for 2**: $Rh_2P_4O_2Si_2C_{76}H_{68}$; space group $P\bar{1}$; a = 12.927 (5) Å, b = 15.165 (2) Å, c = 18.280 (8) Å; α = 93.8 (5)°, β = 103.6 (3)°, γ = 107.8 (9)°; V = 3272 Å^3; Z = 2; d_{calcd} = 1.42 g/cm^3; Mo Kα radiation; ω–2θ scans; $4° < 2\theta < 40°$; observed reflections ($I > 3\sigma$) = 6305; no. of parameters varied = 465; R = 0.047; R_w = 0.059. Calculations were carried out by using the Enraf-Nonius CAD4 and SDP-Plus programs. The function minimized in the least-squares refinements was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma(F_o)^2$. The residuals are defined as $R = \sum(||F_o| - |F_c||)/\sum|F_o|$ and $R_w = \{\sum w(|F_o| - |F_c|)^2/\sum w(F_o)^2\}^{1/2}$. Details of the structure determination are given in the supplementary material. See paragraph regarding supplementary material at the end of the paper.

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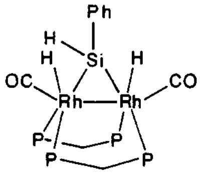
(12) (a) **Spectroscopic data for 3**: ^1H NMR (CD_2Cl_2, −70 °C, ppm) δ 8.29 (d, J = 7 Hz, 2 H, Ph), 7.6–6.8 (m, 43 H, Ph), 6.33 (unresolved mult, 1 H, Si–H), 4.58 (m, 1 H, CH_2), 4.25 (m, 1 H, CH_2), 2.98 (m, 1 H, CH_2), 2.81 (m, 1 H, CH_2), −9.23 (symmetric mult, 2 H, Rh–H); $^{31}P\{^1H\}$ NMR (CD_2Cl_2, −70 °C, ppm) −17.9 (symmetric mult), −28.9 (symmetric mult); IR (Nujol) $\nu_{SiH} = 2031\ cm^{-1}$, $\nu_{CO} = 1949\ cm^{-1}$. (b) Incorporation of chlorine into **3** is extremely unlikely since the 2:1 Rh–H/Si–H integration is completely consistent with addition of one $PhSiH_3$ to **1**, the ^1H NMR spectrum shows no sign of any metal complex other than **3**, and the reaction of **1** with $PhSiH_3$ occurs much more rapidly than the decomposition of **1** alone (**1** does not react with CH_2Cl_2 prior to decomposition).

The variable-temperature ^1H NMR spectrum of **3** is shown in Figure 2. The −9.23 ppm resonance shows the same splitting at both 400 and 500 MHz indicating that the two hydride ligands have the same chemical shift and that the splitting pattern is second order. The $^{31}P\{^1H\}$ NMR spectrum of **3** exhibits resonances due to two inequivalent sets of phosphine donors, while the hydride-coupled spectrum reveals that only one of these sets has significant phosphorus–hydride coupling. From these results and other spectroscopic characterization,[12] complex **3** is formulated as the μ-phenylsilylene hydride $Rh_2H_2(\mu\text{-}SiPhH)(CO)_2(dppm)_2$.

A satisfactory fit of the hydride resonance of **3** at −70 °C was obtained assuming an AA′MM′XX′ pattern with the following parameters: $J_{AA'} = 5$ Hz, $J_{AM} = J_{A'M'} = 143$ Hz, $J_{AX} = J_{A'X'} = 21.7$ Hz, $J_{AM'} = J_{A'M} = 14$ Hz, $J_{AX'} = J_{A'X} = -8.6$ Hz, $J_{MM'} = 144$ Hz, $J_{MX} = J_{M'X'} = 93$ Hz, $J_{M'X} = J_{MX'} = -24$ Hz, $J_{XX'} = 61$ Hz. On the basis of this fit with its one large hydride-phosphorus coupling and the crystallographically determined structure of **2**, complex **3** is proposed to have a cradlelike structure with terminal hydrides and a single μ-SiPhH bridge.



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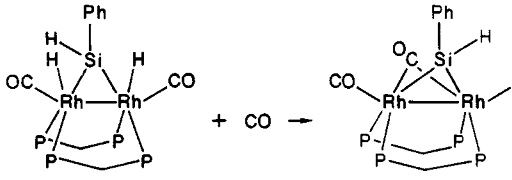
The fluxional process of Figure 2 involves exchange of the hydrogen atoms bound to rhodium and silicon. If the basic cradle structure is preserved throughout the exchange, then the dppm CH_2 protons coalesce from four resonances into two as is readily seen. From the coalescence temperatures of the two sets of dppm methylene protons and the Rh–H/Si–H exchange, $\Delta G^{\ddagger}$ for the process is calculated to be 12 ± 1 kcal/mol at both −30 and 25 °C, indicating a virtually negligible $\Delta S^{\ddagger}$ for the fluxional process. Harrod et al. have observed a similar exchange, but in their case the equilibration was between μ-$SiPhH_2$ and μ-H hydrogen atoms.[3] The fluxionality for **3** may be due to rapid reductive elimination and oxidative addition of Si–H bonds via a $Rh_2H(SiH_2Ph)(CO)_2(dppm)_2$ intermediate.

The reaction of **3** with $PhSiH_3$ leading to the stable bis μ-SiPhH product **2** undoubtedly proceeds by loss of H_2. This is supported by trapping the resultant intermediate with CO yielding the complex $Rh_2(\mu\text{-}SiPhH)(\mu\text{-}CO)(CO)_2(dppm)_2$ (**4**), which contains a bridging carbonyl ligand with ν_{CO} of 1770 cm^{-1}.[13] Complex **4** can also be prepared in high yield by reaction of $Rh_2(CO)_3(dppm)_2$ with $PhSiH_3$ in C_6H_6 at 25 °C for 24 h. On the basis of the fact that the dppm methylene protons of **4** exhibit four different resonances and that the μ-SiPh*H* resonance appears as a triplet of triplets with the larger J_{PH} similar to that seen for **2**, the structure of **4** is presumed to be of the cradle-type as shown in eq 1.



3 **4** (1)

When $EtSiH_3$ is used as the substrate in reaction with **1** under similar conditions, exactly analogous chemistry is observed with sequential formation of $Rh_2H_2(SiEtH)(CO)_2(dppm)_2$ (**5**) and $Rh_2(SiEtH)_2(CO)_2(dppm)_2$ (**6**). Characterization of **5** and **6** is by ^1H and ^{31}P NMR and IR spectroscopies.[14] Complex **6** has also been isolated.[15] From the characterization data, **5** contains a bridging ethylsilylene ligand and terminal hydrides and exhibits fluxional behavior similar to that of **3** in which Si–H/Rh–H interchange is occurring.

The present study thus shows that primary silanes can react with **1** to form μ-SiRH species and that if hydride ligands are also present, rapid exchange of the hydrides with Si–H occurs. These results suggest that Si–H bonds of silylene bridges are relatively labile, a feature which is under continuing investigation.

Acknowledgment. We wish to thank the National Science Foundation (CHE 83-08064) for support of this work and the Johnson Matthey Co., Inc., for generous loan of rhodium salts. We also wish to acknowledge helpful discussions with Prof. D. H. Berry.

Supplementary Material Available: Tables of refined positional and thermal parameters, calculated hydrogen positional parameters, and complete bond distances and angles for **2** (11 pages); a tabulation of observed and calculated structure factor amplitudes for the structure determination of **2** (32 pages). Ordering information is given on any current masthead.

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(13) **Spectroscopic data for 4**: ^1H NMR (C_6D_6, 298 K, ppm) 8.48 (d, *J* = 7 Hz, 2 H), 7.90 (m, 4 H), 7.73 (m, 4 H), 7.28 (t, *J* = 7 Hz, 2 H), 7.09 (m, 8 H), 6.9–6.5 (m, 25 H), 5.31 (tt, J_{PH} = 23, 7 Hz, 1 H), 4.20 (dt, *J* = 15, 9 Hz, 2 H), 3.72 (AB q, *J* = 10 Hz), 3.28 (dt, *J* = 15, 9 Hz), 2.47 (AB q, *J* = 10 Hz); $^{31}P\{^1H\}$ NMR (C_6D_6, H_3PO_4 reference, ppm) 24.06 (symmetric mult), 44.86 (symmetric mult); IR (CH_2Cl_2) ν_{CO} = 1946, 1770 cm^{-1}, ν_{SiH} = 2012 cm^{-1}.

(14) **Spectroscopic data for 5**: ^1H NMR (C_6D_6, 298 K, ppm) 7.62 (s, 8 H), 7.22 (s, 8 H), 6.85–6.65 (m, 24 H), 4.29 (AB q, *J* = 10 Hz, 2 H), 3.10 (AB q, *J* = 10 Hz, 2 H), 2.15 (br s, 2 H), 1.92 (t, *J* = 8 Hz, 3 H), −3.8 (very br); $^{31}P\{^1H\}$ NMR ($CDCl_3$, −55 °C, H_3PO_4 reference, ppm) 17.6 (symmetric mult), 28.98 (symmetric mult). **Spectroscopic data for 6**: ^1H NMR (C_6D_6, 298 K, ppm) 7.87 (s, 8 H), 7.12 (s, 8 H), 6.93 (t, *J* = 7 Hz, 10 H), 6.79 (t, *J* = 7 Hz, 2 H), 6.65 (t, *J* = 7 Hz, 2 H), 6.56 (t, *J* = 7 Hz, 10 H), 5.47 (symmetric mult, 2 H), 4.58 (m, 2 H), 3.25 (m, 2 H), 2.00 (br s, 4 H), 1.37 (t, *J* = 7 Hz, 6 H); $^{31}P\{^1H\}$ NMR (C_6D_6, H_3PO_4 reference, ppm) 32.61 (symmetric mult); IR (CH_2Cl_2) ν_{SiH} = 2054 cm^{-1}, ν_{CO} = 1932 cm^{-1}.

(15) Anal. Calcd for $Rh_2P_4Si_2O_2C_{56}H_{56}$: C, 58.6, H, 4.9. Found: C, 57.8, H, 5.1. The structure of **6** has been determined crystallographically and is virtually identical with that of **2**. Wang, W.-D.; Eisenberg, R., to be submitted for publication.

Cyclopropane Formation from the Reaction of (γ-Haloalkyl)iron Compounds with Silver(1+)

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Summary: Reaction of $C_5H_5(CO)_2Fe(CH_2)_3Br$ with $AgBF_4$ in benzene leads to formation of cyclopropane in 73% yield.

The reaction of electrophilic carbene complexes with alkenes leads to the formation of cyclopropanes in high yield.[1,2] One mechanism for cyclopropane formation involves electrophilic attack of the carbene carbon on the alkene which produces an intermediate with an electro-

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