those of the structurally characterized alkylidyne complex $CpWOs_3(CO)_{11}(\mu_3\text{-}CTol)$.[16] Interestingly, hydrogenation of 1 under similar conditions failed to generate the expected alkylidyne complex $CpWOs_3(CO)_{11}(\mu_3\text{-}CCH_2Ph)$.

Reaction of 1 with excess ditolylacetylene in refluxing toluene (110 °C, 2 h) led to the isolation of a dark green complex, $CpWOs_3(CO)_8(\mu_3\text{-}CPh)[\mu_4\text{-}\eta^5\text{-}C(C_2Tol_2)_2]$ (**6**), as the only isolable product (62%), and no intermediate was observed during the reaction. This complex has been characterized by spectroscopic methods[17] and by a single-crystal X-ray diffraction study (Figure 2).[18]

The molecule contains an almost planar triangulated rhomboidal arrangement with W and Os(3) atoms at the bridgehead position, the angle between W–Os(1)–Os(3) and W–Os(2)–Os(3) planes being 3.32 (3)°. The W atom and the Os(3) atoms of the bridgehead position are coordinated to a Cp ligand and two CO ligands, respectively, while each of the terminal Os(1) and Os(2) atoms is linked to three terminal CO ligands. There is a triply bridging alkylidyne group (μ_3-CPh) which is associated with the triangular W–Os(2)–Os(3) system, and, on the opposite side of the WOs_3 plane is a C_5 hydrocarbyl moiety which interacts with all of the four transition-metal atoms. This C_5 ligand is best described as a bis(2-metallovinyl)alkylidene ligand. The center alkylidene carbon atom C(37) is bridged across the shortest W–Os(3) bond, with the C(21)–C(29) fragment bound to the Os(3) atom via a σ-bond and to the Os(1) atom via a π-bond and with the C(38)–C(46) fragment bound to Os(2) atom and the W atom via a σ-bond and a π-bond, respectively.

The aromatic substituents on the alkylidyne and the alkylidene moieties suggests that the alkylidyne originates from the phenylacetylide ligand of complex 1. Therefore, the reaction mechanism can be considered as a scission of the acetylide carbon–carbon bond to form an alkylidyne unit and a coordinated carbide carbon. The carbide carbon then couples with two molecules of ditolylacetylene to generate the observed alkylidene moiety. Studies of the scission of coordinated alkyne ligand,[19] the coupling of a coordinated carbide atom and alkyne ligand,[20] and the consideration of acetylide ligands as alkylidyne carbides[21] provide precedence for this postulated reaction pathway. However, other possibilities, such as a prior coupling of the coordinated alkynes and acetylide ligands followed by the cleavage of a C–C bond, cannot be eliminated at present. We are currently involved in studying the reactions with other disubstituted acetylenes to establish the mechanism.

Acknowledgment. We are grateful to the National Science Council of the Republic of China for generous financial support.

Registry No. 1, 120637-06-1; 2, 120637-07-2; 3, 120637-11-8; 4, 120665-59-0; 5, 120637-09-4; 6, 120637-10-7; Os, 7440-04-2; W, 7440-33-7; $CpW(CO)_3C{\equiv}CPh$, 69140-93-8; $CpW(CO)_3C{\equiv}CBu$, 120637-08-3; $Os_3(CO)_{10}(CH_3CN)_2$, 61817-93-4; ditolylacetylene, 2789-88-0.

Supplementary Material Available: Tables of bond distances and angles, atomic coordinates, and thermal parameters (11 pages); listings of observed and calculated structure factors (62 pages). Ordering information is given on any current masthead page.

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[16] Busetto, L.; Green, M.; Hesser, B.; Howard, J. A. K.; Jeffery, J. C.; Stone, F. G. A. *J. Chem. Soc., Dalton Trans.* **1983**, 519.

[17] Selected spectroscopic data for complex **6**: MS (FAB, ^{192}Os, ^{184}W), m/z 1562 (M^+); IR (C_6H_{12}) ν(CO) 2073 (s, sh), 2070 (vs), 2036 (vs, sh), 2034 (vs), 2019 (m, sh), 2014 (s), 1999 (s), 1991 (m), 1971 (vs), 1960 (s) cm^{-1}; ^1H NMR ($CDCl_3$, 317 K) δ 7.38 (br, 2 H), 7.30 (t, 2 H), 7.10 (t, 1 H), 6.97 (d, 2 H), 6.87 (d, 2 H), 6.78 (m, 4 H), 6.69 (d, 2 H), 6.56 (d, 2 H), 6.34 (d, 2 H), 6.03 (d, 2 H), 5.56 (s, 5 H), 2.26 (s, 3 H), 2.26 (s, 3 H), 2.12 (s, 3 H), 2.04 (s, 3 H). Anal. Calcd for $W_1Os_3C_{53}H_{38}O_8$: C, 40.88; H, 2.46. Found: C, 40.83; H, 2.50.

[18] Crystal data for complex **6**: $W_1Os_3C_{53}H_{38}O_8$; $M = 1557.34$; monoclinic, space group $P\bar{1}$; $a = 10.351$ (3), $b = 12.576$ (3), $c = 18.098$ (3) Å; $\alpha = 94.44$ (2), $\beta = 91.07$ (2), $\gamma = 96.00$ (2)°; $V = 2335.18$ Å^3; $Z = 2$; $D_{calcd} = 2.215$ mg/cm^3; $F(000) = 1443.5$; Nonius CAD-4 diffractometer with graphite-monochromated Mo Kα radiation; $\lambda = 0.70930$ Å; μ(Mo Kα) = 10.71 mm^{-1}. Ψ scan absorption correction has been made, 8201 unique reflections were measured, and 5283 reflections with $I > 2\sigma(I)$ were used in refinement. Refinement of 65 atoms and 587 parameters converged to $R_F = 0.041$, $R_w = 0.042$, and GOF = 2.397.

[19] (a) Park, J. T.; Shapley, J. R.; Bueno, C.; Ziller, J. W.; Churchill, M. R. *Organometallics* **1988**, *7*, 2307. (b) Eaton, B.; O'Connor, J. M.; Vollhardt, K. P. C. *Organometallics* **1986**, *5*, 394. (c) Chi, Y.; Shapley, J. R. *Organometallics* **1985**, *4*, 1900. (d) Listemann, M. L.; Schrock, R. R. *Organometallics* **1984**, *4*, 74. (e) Cotton, F. A.; Schwotzer, W.; Shamshoum, S. E. *Organometallics* **1984**, *3*, 1770. (f) Shapley, J. R.; McAteer, C. H.; Churchill, M. R.; Biondi, L. V. *Organometallics* **1984**, *3*, 1595.

[20] Dutton, T.; Johnson, B. F. G.; Lewis, J.; Owen, S. M.; Raithby, P. *J. Chem. Soc., Chem. Commun.* **1988**, 1423.

[21] Nucciarone, D.; Taylor, N. J.; Carty, A. J. *Organometallics* **1986**, *5*, 1179.

Preparation and Structure Determination of a Dinuclear Ruthenacyclopentadiene Complex. Coupling of Coordinated Vinyl Ligands

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Received December 30, 1988

Summary: Heating the divinyl complex $(\eta^5\text{-}C_5Me_5)Ru(PMe_3)(CH{=}CH_2)_2Ru(\eta^5\text{-}C_5Me_5)$ in toluene at 70 °C leads to the formation of a novel dinuclear ruthenacyclopentadiene complex, $(\eta^5\text{-}C_5Me_5)Ru(PMe_3)(\mu\text{-}\eta^4\text{-}C_4H_4)Ru(\eta^5\text{-}C_5Me_5)$ (**3**). The structure of the complex has been determined by single-crystal X-ray diffraction.

The activation of C–H bonds of olefins by transition-metal complexes and the reactivities of the resultant σ-alkenyl–metal complexes have been relatively unexplored though the catalytic conversions of alkenes to functionalized molecules via such a mechanism would be of substantial interest.[1]

In the previous paper, we reported that the dinuclear divinyl complex $(\eta^5\text{-}C_5Me_5)Ru(CH_2{=}CH_2)(CH{=}CH_2)_2Ru(\eta^5\text{-}C_5Me_5)$ (**1**) was obtained by treatment of the tetrahydride-bridged dinuclear ruthenium complex $(\eta^5\text{-}C_5Me_5)Ru(\mu\text{-}H)_4Ru(\eta^5\text{-}C_5Me_5)$ with ethylene via an acti-

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[1] See, for example: (a) Volger, H. C. *Recl. Trav. Chim. Pays-Bas* **1967**, *86*, 677. (b) Keister, J. B.; Shapley, J. R. *J. Organomet. Chem.* **1975**, *85*, C29. (c) Goddard, R.; Knox, S. A. R; Stone, F. G. A.; Winter, M. J.; Woodward, P. *J. Chem. Soc., Chem. Commun.* **1976**, 559. (d) Caddy, P.; Green, M.; Smart, L. E.; White, N. *J. Chem. Soc., Chem. Commun.* **1978**, 839. (e) Bhaduri, S.; Johnson, B. F. G.; Kelland, J. W.; Lewis, J.; Raithby, P. R.; Rehani, S.; Sheldrick, G. M.; Wong, K. *J. Chem. Soc., Dalton Trans.* **1979**, 562. (f) Dyke, A. F.; Guerchais, J. E.; Knox, S. A. R.; Roue, J.; Short, R. L.; Taylor, G. E.; Woodward, P. *J. Chem. Soc., Chem. Commun.* **1981**, 537. (g) Brown, T. L.; Nubel, P. O. *J. Am. Chem. Soc.* **1982**, *104*, 4955. (h) Franzreb, K. H.; Kreiter, C. G. *Z. Naturforsch., B* **1982**, *37B*, 1058. (i) Aumann, R.; Henkel, G.; Krebs, B. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 204. (j) Herrmann, W. A.; Weichmann, J.; Balbach, B.; Ziegler, M. L. *J. Organomet. Chem.* **1982**, *231*, C69. (k) Eisenstadt, A.; Efraty, A. *Organometallics* **1982**, *1*, 1100. (l) King, J. A.; Vollhardt, K. P. C. *J. Am. Chem. Soc.* **1983**, *105*, 4846. (m) Nubel, P. O.; Brown, T. L. *J. Am. Chem. Soc.* **1984**, *106*, 644. (n) Fryzuk, M. D.; Jones, T.; Einstein, F. W. B. *Organometallics* **1984**, *3*, 185. (o) Faller, J. W.; Felkin, H. *Organometallics* **1985**, *4*, 1488. (p) Stoutland, P. O.; Bergman, R. G. *J. Am. Chem. Soc.* **1985**, *107*, 4581. (q) Baker, M. V.; Field, L. D. *J. Am. Chem. Soc.* **1986**, *108*, 7433, 7436. (r) Ting, C.; Messerle, L. *J. Am. Chem. Soc.* **1987**, *109*, 6506.

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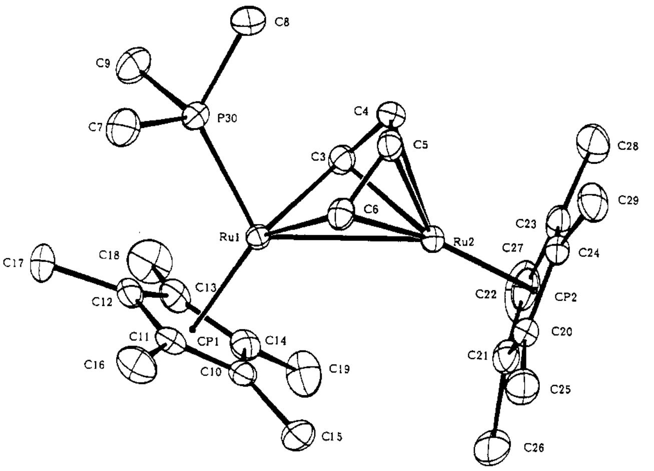
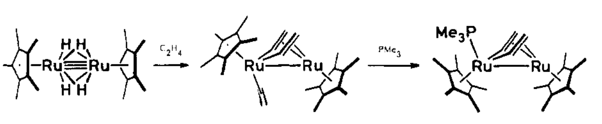


Figure 1. Molecular structure of $(\eta^5\text{-}C_5Me_5)Ru(PMe_3)(\mu\text{-}\eta^4\text{-}C_4H_4)Ru(\eta^5\text{-}C_5Me_5)$ (**3**). Bond lengths (Å) and angles (deg) of interest are as follows: Ru(1)–Ru(2), 2.831 (1); Ru(1)–P(30), 2.297 (1); Ru(1)–C(3), 2.057 (5); Ru(1)–C(6), 2.044 (6); C(3)–C(4), 1.401 (9); C(4)–C(5), 1.426 (9); C(5)–C(6), 1.397 (8); Ru(2)–C(3), 2.155 (5); Ru(2)–C(4), 2.115 (5); Ru(2)–C(5), 2.112 (5); Ru(2)–C(6), 2.149 (4); P(30)–Ru(1)–Ru(2), 116.8 (0); Ru(1)–C(3)–C(4), 114.6 (4); Ru(1)–C(6)–C(5), 115.0 (4); C(3)–Ru(1)–C(6), 77.6 (2); C(3)–C(4)–C(5), 114.0 (5); C(4)–C(5)–C(6), 114.3 (5). CP indicates the centroid of a C_5Me_5 ring.

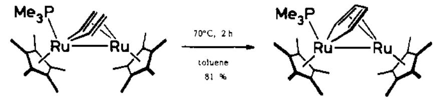
vation of a C–H bond of ethylene and that reaction of **1** with trimethylphosphine leads to *cis*-$(\eta^5\text{-}C_5Me_5)Ru(PMe_3)(CH{=}CH_2)_2Ru(\eta^5\text{-}C_5Me_5)$ (**2**) in excellent yield, with inversion of the stereochemistry at one of the ruthenium centers (eq 1).[2,3] We report here the formation of a dinuclear ruthenacyclopentadiene complex via a thermal coupling of the vinyl ligands in **2**.



(1)

1 **2**

Heating of complex **2** (0.186 g, 0.31 mmol) in toluene (5 mL) at 70 °C for 2 h led to the formation of a ruthenacyclopentadiene complex, $(\eta^5\text{-}C_5Me_5)Ru(PMe_3)(\mu\text{-}\eta^4\text{-}C_4H_4)Ru(\eta^5\text{-}C_5Me_5)$ (**3**) (eq 2). Purification by column chromatography on alumina (Merck, Art. 1097) with toluene followed by crystallization from toluene–pentane afforded analytically pure **3** (0.149 g, 0.25 mmol) in 81% yield as red plates. The structure of **3** is assigned on the basis of ^1H NMR, ^{13}C NMR, FD mass spectrometry, infrared spectroscopy, and elemental analysis.[4]

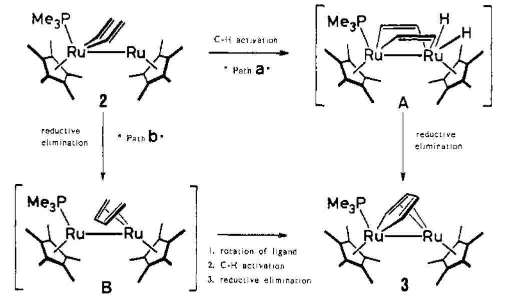


(2)

2 **3**

The ^1H NMR spectrum measured in C_6D_6 revealed two equivalent sets of olefinic protons of the metallacycle at δ 7.25 (H_α) and 4.43 (H_β) and two singlet peaks at δ 2.02 and 1.86 for nonequivalent C_5Me_5 ligands. The ^{13}C resonance signals for the metallacycle framework are observed at δ 142.0 (J_{CH} = 149.9 Hz, J_{CP} = 16.5 Hz, C_α) and 92.7 (J_{CH} = 148.7 Hz, J_{CP} = 9.9 Hz, C_β). These are slightly shielded compared with those, δ 156.6 and 114.4, reported for $Fe(CO)_3(\mu\text{-}\eta^4\text{-}C_4H_4)(\mu\text{-}CO)Fe(CO)_3$.[5] The shielding was presumably arising from the electron donation from the PMe_3 and C_5Me_5 ligands. The signals for the two C_5Me_5 ligands appeared as nonequivalent sets at δ 11.5 (q, J_{CH} = 126.0 Hz) and 91.5 (s) and δ 11.4 (q, J_{CH} = 126.0 Hz) and 86.8, respectively.

Scheme I



The molecular structure of **3** determined by X-ray diffraction study confirms the cyclocoupling of two vinyl ligands and the formation of a five-membered ruthenacycle (Figure 1).[6] Two C_5Me_5 ligands occupy cis positions with respect to the Ru–Ru bond. The complex has a pseudosymmetry plane with the phosphorus, C8, C12, C17, C21, C26, and two ruthenium atoms.

The four carbon atoms of the metallacyclopentadiene framework are close to coplanar, and the torsional angle between the C_4 plane and that defined by Ru1, C3, and C6 atoms is 18.3°. The diene moiety in the ruthenacycle is coordinated to another ruthenium center, Ru(2). The Ru(1)–C(3) and Ru(1)–C(6) bond lengths of 2.057 (5) and 2.044 (6) Å are comparable to that of the Ru–C single bond length reported for ruthenacyclopentadiene.[7] The C(3)–C(4), C(5)–C(6), and C(4)–C(5) bond lengths of 1.401 (9), 1.397 (8), and 1.426 (9) Å also reflect the proposed ruthenacyclopentadiene structure. The 18-electron rule applied to the dinuclear complex **3** requires a single bond between the ruthenium atoms. The Ru(1)–Ru(2) distance of 2.831 (1) Å just represents a single-order metal–metal bond.

The following two mechanisms shown in Scheme I possibly account for the formation of the ruthenacycle **3** from the dinuclear divinyl complex **2**. Activation of the $C(sp^2)$–H bond at the terminus of the vinyl group by one of the ruthenium centers would be expected, on the basis of precedent,[8] to form the dihydrido bis(μ-ethene) species A, which would undergo reductive elimination to give ru-

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(2) Suzuki, H.; Omori, H.; Lee, D. H.; Yoshida, Y.; Moro-oka, Y. *Organometallics* **1988**, *7*, 2243.

(3) Suzuki, H.; Omori, H.; Moro-oka, Y. *Organometallics* **1988**, *7*, 2579.

(4) **3**: IR (KBr) 2988, 2956, 2882, 1465, 1409, 1369, 1215, 1022, 948, 908, 826 cm^{-1}; ^1H NMR (C_6D_6) δ 0.93 (d, J_{PH} = 8.2 Hz, 9 H, PMe_3), 1.86 (s, 15 H, C_5Me_5), 2.02 (s, 15 H, C_5Me_5), 4.43 (dd, $J_{\alpha\beta}$ = 2.1 Hz, J_{PH} = 2.3 Hz, 2 H, Ru—CH=CH_β–), 7.25 (dd, $J_{\alpha\beta}$ = 2.1 Hz, J_{PH} = 4.0 Hz, 2 H, Ru—CH_α=CH–); ^{13}C NMR (C_6D_6) δ 11.4 (q, J_{CH} = 126.0 Hz, C_5Me_5), 11.5 (q, J_{CH} = 126.0 Hz, C_5Me_5), 20.2 (qd, J_{CH} = 127.8 Hz, J_{PC} = 29.8 Hz, PMe_3), 86.8 (s, C_5Me_5), 91.5 (s, C_5Me_5), 92.7 (dd, J_{CH} = 148.7 Hz, J_{PC} = 9.9 Hz, Ru—CH=*C*H–), 142.0 (ddd, J_{CH} = 149.9 and 9.9 Hz, J_{PC} = 16.5 Hz, Ru—*C*H=CH–), $^{31}P\{^1H\}$ NMR (C_6D_6, external PPh_3) δ 16.5; mp 215 °C dec. Anal. Calcd for $C_{27}H_{43}PRu_2$: C, 53.98; H, 7.39. Found: C, 53.97; H, 7.68.

(5) Dettlef, G.; Weiss, E. *J. Organomet. Chem.* **1976**, *108*, 213.

(6) Complex **3** crystallized from toluene–pentane in the monoclinic system, space group $P2_1/n$, with a = 21.597 (5) Å, b = 8.782 (1) Å, c = 14.964 (3) Å, β = 109.06 (2)°, and Z = 4. Intensity data were collected at 25 °C on a Rigaku AFC-5 diffractometer with graphite-monochromated Mo Kα radiation in the 2° < 2θ < 60° range. Absorption correction was not applied. Data processing was performed on a FACOM A-70 computer by using the R-CRYSTAN structure solving program library obtained from Rigaku Corp. All non-hydrogen atoms were refined anisotropically by a full-matrix least-squares technique. The final R value is 0.0435 for 6009 independent reflections with $F_o < 3\sigma(F_o)$.

(7) (a) Noda, I.; Yasuda, H; Nakamura, A. *Organometallics* **1983**, *2*, 1207. (b) Noda, I.; Yasuda, H.; Nakamura, A. *J. Organomet. Chem.* **1983**, *250*, 447. (c) Bruce, M. I.; Matisons, J. G.; Skelton, B. W.; White, A. H. *J. Organomet. Chem.* **1983**, *251*, 249.

(8) Deeming, A. J.; Hasso, S.; Underhill, M. *J. Chem. Soc., Dalton Trans.* **1975**, 1614.

thenacycle **3** and dihydrogen (path a). Another possible route to **3** (path b) would involve a reductive coupling of the two vinyl ligands to yield the diruthenium-1,3-butadiene complex B. Rotation of the coordinated butadiene and activation of C-H bonds at the terminal carbons of the butadiene followed by reductive elimination would give the ruthenacycle **3**.

While an example of the reductive elimination of 1,3-butadiene from a monomeric ruthenium divinyl complex has been reported,[9] path b could be excluded on the basis of the result of a control experiment. Thermolysis of **2** in benzene even in the presence of a 10-fold excess of isoprene exclusively afforded **3**, and the formation of a 2-methylated ruthenacyclopentadiene was not observed. Furthermore, an intermediary hydride species observed at δ -9.32 as a singlet by monitoring the reaction by means of ^1H NMR spectroscopy suggests the validity of path a.[10]

Further reactivity studies of **3** and mechanistic studies pertaining to the formation of **3** will be reported in due course.

Acknowledgment. Support from Ministry of Education, Science and Culture of the Japanese government (No. 63550618) is gratefully acknowledged.

Supplementary Material Available: Tables of atomic coordinates, interatomic distances and angles, and thermal parameters (18 pages); a listing of calculated and observed structure factors (29 pages). Ordering information is given on any current masthead page.

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[9] (a) Czisch, P.; Erker, G.; Korth, H.-G.; Sustmann, R. *Organometallics* **1984**, *3*, 945. (b) Chang, J.; Bergman, R. G. *J. Am. Chem. Soc.* **1987**, *109*, 4298.

[10] The reaction was carried out in C_6D_6 (c = 0.33 M) at 65 °C and was monitored by means of ^1H NMR spectroscopy. After the mixture was heated for 30 min, resonance signals due to the intermediary hydride species were observed at δ -9.32 (s, Ru-*H*) and 2.06 (s, C_5Me_5) in the intensity ratio of 2:15 together with signals for **2** and **3**. One of the resonances for C_5Me_5 ligands of intermediate might overlap that of **2**. The molar ratio of the intermediate, **2**, and the ruthenacycle **3** was 3:40:1. The ratio changed to 1.5:8:1 after heating for 2 h.

Synthesis and Characterization of $[HgAu(CH_2SPPh_2CH_2)_2]PF_6$, the First Example of Structurally Characterized Carbene Double Insertions into Metal-Sulfur Bonds

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Received April 10, 1989

Summary: The reaction of CH_2N_2 with the heterobimetallic complex $HgAu(SPPh_2CH_2)_2PF_6$, **1**, in THF/diethyl ether at 0 °C yields a novel complex, $HgAu(CH_2SPPh_2CH_2)_2PF_6$, **2**, in 40% yield. **2** is the result of methylene group double insertions into the Au-S bonds in **1**. The structure of **2** was determined by single-crystal X-ray diffraction. **2** crystallizes in the monoclinic space group $P2_1/c$ with a = 11.218 (4) Å, b = 24.59 (2) Å, c = 12.598 (7) Å, and β = 109.42 (4)°. **2** is the first example of CH_2 insertion into metal-sulfur bonds.

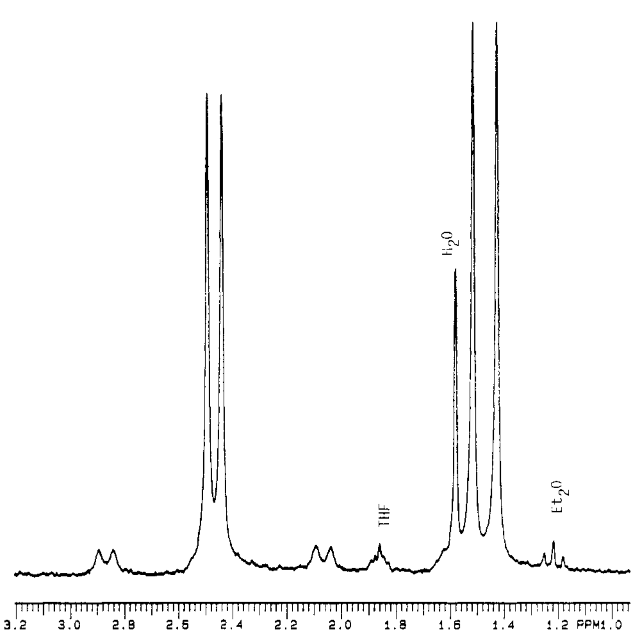

Figure 1. ^1H NMR spectrum of **2** in $CDCl_3$.

Studies of the syntheses, characterization, and reactivity of carbene ligands and their metal complexes are among the most attractive and active areas of investigation in organometallic chemistry in the last two decades.[1] The important application of metal carbene complexes in organic syntheses has given rise to many studies of the reactions of carbene complexes with organic substrates. Nucleophilic and electrophilic substitution reactions on the carbene ligands have been the well-studied topics of numerous papers.[1] In contrast, carbene insertion reactions into metal-ligand bonds, reactions which play a critical role in organic syntheses, have been recognized and investigated only relatively recently.[2] Although there are numerous examples of carbene insertions into metal-carbon bonds,[2,3] very little is known about carbene insertions into M-X bonds where X is a main group element such as O, S, P, or halide.[4] Our interest in studying the interactions of the methylene group with bimetallic complexes caused investigation of the reaction of CH_2N_2 with the heterobimetallic compound[5] $[HgAu(SPPh_2CH_2)_2]PF_6$, **1**. A novel complex with the formula of $[HgAu(CH_2SPPh_2CH_2)_2]PF_6$, **2**, resulted from the carbene double insertion into Au-S bonds in **1**. This is the first example of a structurally characterized complex produced from carbene insertions into metal-sulfur bonds. We report here the synthesis and characterization of this remarkable product.

The reaction[6,7] of CH_2N_2 with **1** in the mixed solvents THF/diethyl ether at 0 °C yielded the air-stable colorless compound **2** as a major product. **2** was isolated in crys-

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[1] (a) Cardin, D. J.; Cetinkaya, B.; Doyle, M. J.; Lappert, M. F. *Chem. Soc. Rev.* **1973**, *2*, 99. (b) Cardin, D. J.; Cetinkaya, B.; Lappert, M. F. *Chem. Rev.* **1972**, *72*, 545. (c) Fischer, E. O. *Adv. Organomet. Chem.* **1976**, *14*, 1. (d) Herrmann, W. A. *Adv. Organomet. Chem.* **1982**, *20*, 159.

[2] Collman, J. P.; Hegedus, L. S.; Norton, J. R.; Finke, R. G. *Principles and Applications of Organotransition Metal Chemistry*, University Science Books: Mill Valley, CA, 1987; p 119-134, 379-381.

[3] (a) Kleitzein, H.; Werner, H.; Serhadli, P.; Ziegler, M. L. *Angew. Chem., Int. Ed. Engl.* **1983**, *22*, 46. (b) Jernakott, P.; Cooper, N. J. *J. Am. Chem. Soc.* **1984**, *106*, 3026. (c) Thorn, D. L.; Tulip, T. H. *J. Am. Chem. Soc.* **1981**, *103*, 5984.

[4] (a) Seyferth, D. *Chem. Rev.* **1955**, *55*, 1155 and references therein. (b) McCrindle, R.; Arsenault, G. J.; Farwaha, R. *J. Organomet. Chem.* **1985**, *296*, C51. (c) Hubbard, J. L.; McVicar, W. K. *J. Am. Chem. Soc.* **1986**, *108*, 6422.

[5] Wang, S.; Fackler, J. P., Jr. *Organometallics* **1988**, *7*, 2415.

[6] CH_2N_2 was generated from the reagent 1-methyl-3-nitro-1-nitrosoguanidine (MNNG) purchased from Aldrich by using the method reported in literature.[7]

[7] Black, T. H. *Aldrichim. Acta* **1983**, *16*, 3.

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