

**Scission of a Coordinated Acetylidyde Ligand on the Tungsten-Triosmium Framework. Synthesis, Crystal Structure, and Reactivity Studies of  $\text{CpWOs}_3(\text{CO})_{11}(\text{C}\equiv\text{CR})$  ( $\text{R} = \text{Ph}$  and  $^n\text{Bu}$ )**

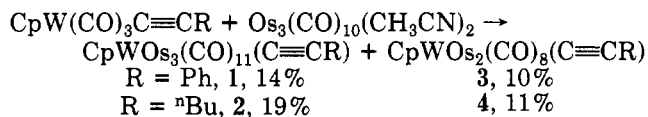
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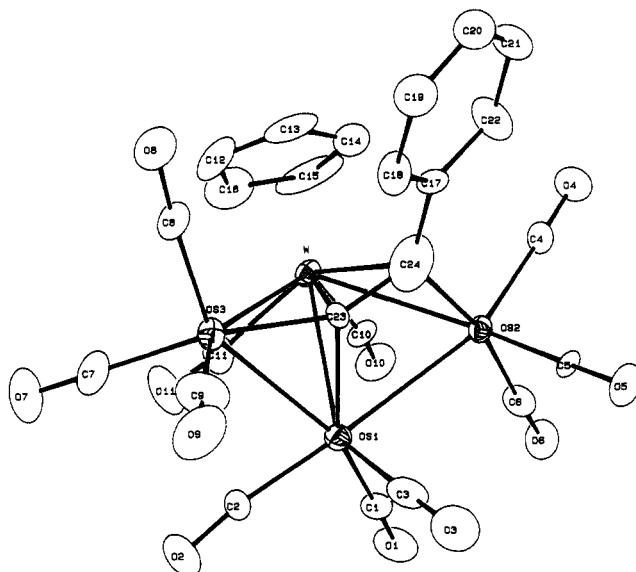
Received February 6, 1989

**Summary:** Reaction of  $\text{CpW}(\text{CO})_3\text{C}\equiv\text{CR}$  with  $\text{Os}_3(\text{CO})_{10}(\text{CH}_3\text{CN})_2$  in refluxing toluene produces tetranuclear mixed-metal acetylidyde complexes  $\text{CpWOs}_3(\text{CO})_{11}(\text{C}\equiv\text{CR})$  (**1**, **2**). Carbonylation of (**1**, **2**) at 120 °C under pressurized CO induces cluster fragmentation, giving  $\text{CpWOs}_2(\text{CO})_8(\text{C}\equiv\text{CR})$  (**3**, **4**) in 80–85% yield. Hydrogenation of **2** produces an alkylidyne complex,  $\text{CpWOs}_3(\text{CO})_{11}(\mu_3\text{-C}^n\text{C}_5\text{H}_{11})$  (**5**), while treatment of **1** with excess ditolylacetylene affects the scission of the acetylidyde ligand to give  $\text{CpWOs}_3(\text{CO})_8(\mu_3\text{-CPh})[\mu_4\text{-}\eta^5\text{-C}(\text{C}_2\text{ToI}_2)_2]$  (**6**). The structures of **1** and **6** were determined by single-crystal X-ray diffraction studies.

The reactivity of mixed-metal clusters has been of interest for many years.<sup>1</sup> Research in this area is stimulated by a belief that combination of metals having very different chemical properties in one compound may induce unique chemical transformations. In seeking to exploit the chemistry of the unsaturated hydrocarbyl ligand in the coordination sphere of mixed-metal polynuclear complexes, we have treated the mononuclear acetylidyde complex  $\text{CpW}(\text{CO})_3\text{C}\equiv\text{CR}$  with the triosmium species  $\text{Os}_3(\text{CO})_{10}(\text{CH}_3\text{CN})_2$  with the aim of generating mixed-metal acetylidyde complexes.<sup>2</sup> We describe herein the preparation, crystal structure, and reaction of these  $\text{WOs}_3$  complexes possessing a novel<sup>3</sup>  $\mu_4\text{-}\eta^2$ -acetylidyde ligand. On reaction with ditolylacetylene, this acetylidyde ligand undergoes carbon-carbon bond scission and alkyne coupling reactions to generate a  $\text{C}_1$  alkylidyne ligand and a  $\text{C}_5$  alkylidene moiety. This reaction is in contrast to those of polynuclear ruthenium complexes containing phosphorus bridging ligands, in which only carbon-carbon bond formation with the acetylidyde ligand were reported.<sup>4</sup>



Treatment of  $\text{CpW}(\text{CO})_3\text{C}\equiv\text{CR}$ <sup>5</sup> with the triosmium species  $\text{Os}_3(\text{CO})_{10}(\text{CH}_3\text{CN})_2$  in refluxing toluene (110 °C,



**Figure 1.** ORTEP diagram of complex **1**. Bond lengths (Å): W–Os(1), 2.833 (1); W–Os(2), 2.906 (1); W–Os(3), 2.968 (1); Os(1)–Os(2), 2.866 (1); Os(1)–Os(3), 2.819 (1); W–C(23), 2.27 (2); Os(1)–C(23), 2.13 (2); Os(3)–C(23), 1.95 (2); W–C(24), 2.49 (2); Os(2)–C(24), 2.09 (2); C(23)–C(24), 1.38 (2). Bond angles (deg): Os(3)–C(23)–C(24), 153 (1); C(17)–C(24)–C(23), 121 (2).

30 min) provided, in addition to a pale yellow trinuclear complex,  $\text{CpWOs}_2(\text{CO})_8(\text{C}\equiv\text{CR})$ ,<sup>6</sup> a red tetranuclear acetylidyde complex,  $\text{CpWOs}_3(\text{CO})_{11}(\text{C}\equiv\text{CR})$  ( $\text{R} = \text{Ph}$ , **1**;  $\text{R} = ^n\text{Bu}$ , **2**) in low yield.<sup>7</sup> Infrared studies indicated the presence of only terminal CO ligands. The <sup>13</sup>C NMR spectrum of **1** and **2** each showed two signals at  $\delta$  186.9 and 162.8 and  $\delta$  192.0 and 165.8, respectively, assigned to the acetylidyde  $\alpha$ - and  $\beta$ -carbons. As the chemical shifts of the acetylidyde carbons are in the range of the Os–CO signals, their assignment follows the prior assignment of the CO signals of the <sup>13</sup>CO-enriched samples. The  $\text{C}_\alpha$  and  $\text{C}_\beta$  chemical shifts also suggest that the acetylidyde ligand is quite electron deficient, characteristic of a  $\mu_4\text{-}\eta^2$  bonding mode.<sup>8</sup>

The structure of **1** was determined by a single-crystal X-ray diffraction study.<sup>9</sup> An ORTEP diagram is presented in Figure 1, which also provides some selected data of bond lengths and angles. The molecule consists of a  $\text{WOs}_3$  core

(6) Chi, Y.; Peng, S.-M.; Lee, G.-H.; Liu, B.-J., submitted for publication in *Polyhedron*.

(7) Selected spectroscopic data. Complex **1**: MS (EI, <sup>192</sup>Os, <sup>184</sup>W),  $m/z$  1234 ( $M^+$ ); IR ( $\text{C}_6\text{H}_{12}$ )  $\nu(\text{CO})$  2082 (s), 2058 (vs), 2033 (s), 2016 (s), 2000 (m), 1985 (vw), 1972 (s), 1962 (w)  $\text{cm}^{-1}$ ; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , room temperature)  $\delta$  7.89 (d, 2 H), 7.46 (m, 3 H), 4.96 (s, 5 H); <sup>13</sup>C{<sup>1</sup>H} NMR ( $\text{CD}_2\text{Cl}_2$ , room temperature)  $\delta$  208.5 (W–CO), 204.2 (W–CO), 186.9 (CCPh), 162.8 (CCPh), 90.5 (Cp). Anal. Calcd for  $\text{W}_1\text{Os}_3\text{C}_{24}\text{H}_{10}\text{O}_{11}$ : C, 23.46; H, 0.82. Found: C, 23.38; H, 0.85. Complex **2**: MS (FAB, <sup>192</sup>Os, <sup>184</sup>W),  $m/z$  1214 ( $M^+$ ); IR ( $\text{C}_6\text{H}_{12}$ )  $\nu(\text{CO})$  2082 (m), 2057 (vs), 2032 (s), 2009 (s), 2000 (m), 1983 (w), 1977 (s), 1960 (w), 1938 (vw)  $\text{cm}^{-1}$ ; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , room temperature)  $\delta$  5.21 (s, 5 H, Cp), 2.99 (m, 1 H, CH), 2.71 (m, 1 H, CH), 1.92 (q, 2 H, CH<sub>2</sub>), 1.55 (m, 2 H, CH<sub>2</sub>), 1.00 (t, 3 H, CH<sub>3</sub>); <sup>13</sup>C{<sup>1</sup>H} NMR ( $\text{CD}_2\text{Cl}_2$ , room temperature)  $\delta$  208.9 ( $J_{\text{W-C}} = 145$  Hz, W–CO), 205.2 ( $J_{\text{W-C}} = 150$  Hz, W–CO), 192.0 (CC<sup>n</sup>Bu), 181.4 (Os–CO), 178.2 [br, Os(CO)<sub>3</sub>], 177.6 [Os(CO)<sub>3</sub>], 172.6 (Os–CO), 172.2 (Os–CO), 165.8 (CC<sup>n</sup>Bu), 98.4 (Cp), 55.0 (CH<sub>2</sub>), 31.2 (CH<sub>2</sub>), 22.0 (CH<sub>2</sub>), 13.5 (CH<sub>3</sub>). Anal. Calcd for  $\text{W}_1\text{Os}_3\text{C}_{22}\text{H}_{14}\text{O}_{11}$ : C, 21.86; H, 1.17. Found: C, 21.84; H, 1.20.

(8) Carty, A. J.; Cherkas, A. A.; Randall, L. H. *Polyhedron* 1988, 7, 1045.

(9) Crystal data for complex **1**:  $\text{W}_1\text{Os}_3\text{C}_{24}\text{H}_{10}\text{O}_{11}$ ;  $M = 1228.79$ ; monoclinic, space group  $P2_1/c$ ;  $a = 9.0565$  (16),  $b = 9.1972$  (12),  $c = 29.065$  (4) Å;  $\beta = 95.768$  (12)°;  $V = 2597.25$  Å<sup>3</sup>;  $Z = 4$ ,  $D_{\text{calcd}} = 3.389$  mg/cm<sup>3</sup>;  $F(000) = 2175.12$ ; Nonius CAD-4 diffractometer with graphite-monochromated Mo  $K_\alpha$  radiation;  $\lambda = 0.70930$  Å;  $\mu(\text{Mo } K_\alpha) = 20.72$  mm<sup>-1</sup>.  $\Psi$  scan absorption correction has been made, 4554 unique reflections were measured, and 3374 reflections with  $I > 2\sigma(I)$  were used in refinement. Refinement of 49 atoms and 350 parameters converged to  $R_F = 0.055$ ,  $R_w = 0.060$ , and GOF = 2.757.

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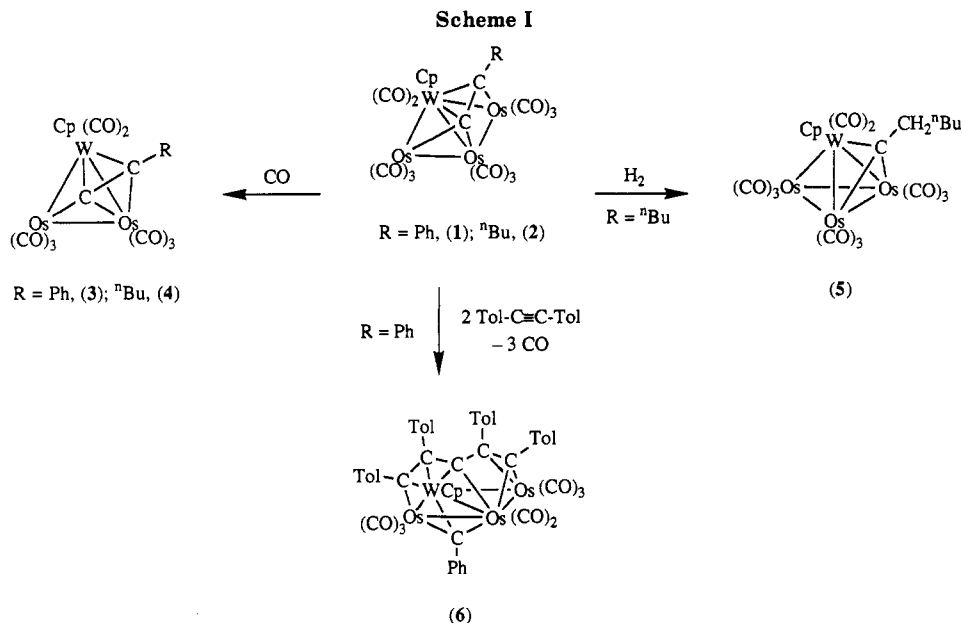
(1) Galdfelter, W. L.; Geoffroy, G. L. In *Comprehensive Organometallic Chemistry*; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon: Oxford, 1982; Vol. 6, Chapter 40.

(2) Similar method of using mononuclear metal acetylidyde complexes to prepare acetylidyde clusters has been reported previously; see: Roland, E.; Bernhardt, W.; Vahrenkamp, H. *Chem. Ber.* 1986, 119, 256.

(3) Carty, A. J. *Pure Appl. Chem.* 1982, 54, 113.

(4) (a) Nucciarone, D.; MacLaughlin, S. A.; Taylor, N. J.; Carty, A. J. *Organometallics* 1988, 7, 106. (b) Nucciarone, D.; Taylor, N. J.; Carty, A. J. *Organometallics* 1988, 7, 127.

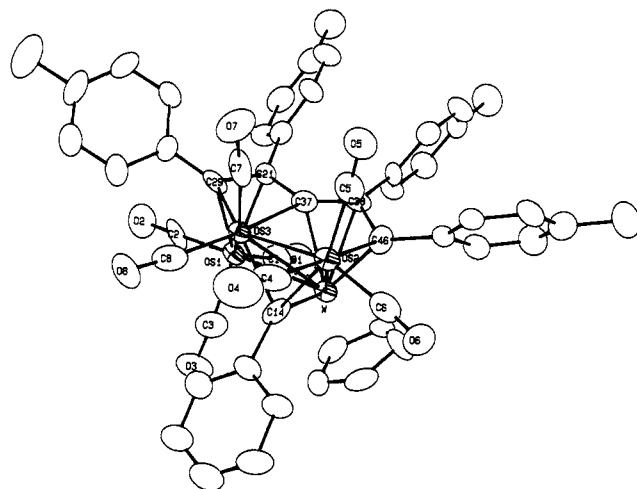
(5) Bruce, M. I.; Humphrey, M. G.; Matison, J. G.; Roy, S. K.; Swincer, A. G. *Aust. J. Chem.* 1984, 37, 1955.



structure of a "butterfly" arrangement, with Os(2) and Os(3) defining the "wing-tip" positions and W and Os(1) atoms, the "hinge" positions. Each osmium atom is associated with three CO ligands, and the tungsten is coordinated by two CO ligands and a Cp ligand. The acetylide ligand occupies the open face of the butterfly framework and is coordinated in multisite fashion with its  $\alpha$ -carbon C(23) atom bound to three metal atoms W, Os(1), and Os(3) and the  $\beta$ -carbon atom C(24) linked to W and Os(2) atoms. Because of the coordination of the acetylide, the dihedral angle between the triangular wings W-Os(1)-Os(2) and W-Os(1)-Os(3),  $131.4(3)^\circ$ , is smaller than the deformation angle of the acetylide,  $\angle\text{Os}(3)\text{-C}(23)\text{-C}(24) = 153(1)^\circ$ .

The bonding interaction of the acetylide ligand in 1 is particularly notable; the  $\mu_4\text{-}\eta^2$ -bonding mode has been documented in spiked tetrametallic systems,<sup>10</sup> and on the square face of square-pyramidal pentametallic derivatives<sup>11</sup> and on the side face of the wing-tip bridged butterfly NiRu<sub>4</sub> complex.<sup>12</sup> However, complex 1 is the only known example of the  $\mu_4\text{-}\eta^2$ -acetylide bonding in the butterfly tetrametallic system, in which we consider that the acetylide ligand functions as a five-electron donor,  $\sigma$ -bonded to a Os-Os edge via a three-center two-electron interaction, while employing its two orthogonal alkyne  $\pi$ -bonds to bridge the opposite W-Os edge. Interestingly, the W-C(24) distance is about 0.4 Å longer than the respective Os(2)-C(24) distance, suggesting that the acetylide  $\pi$ -bond interacts more strongly with the Os(2) atom. This geometry recalls the closely related  $\mu_4\text{-}\eta^2$ -bonding of the vinylidene ligand in butterfly clusters.<sup>13</sup>

Carbonylation of 1 or 2 under pressurized CO (30 psig, 120 °C, 6 h) induced cluster fragmentation, giving the trinuclear complexes CpWOs<sub>2</sub>(CO)<sub>8</sub>(C≡CR) (R = Ph, 3; R = <sup>n</sup>Bu, 4) in 80–85% yield (Scheme I).<sup>14</sup> Nevertheless,



**Figure 2.** ORTEP diagram of complex 6. Bond lengths (Å): W-Os(1), 3.004 (1); W-Os(2), 2.807 (1); W-Os(3), 2.766 (1); Os(1)-Os(3), 2.784 (1); Os(2)-Os(3), 2.743 (1); W-C(14), 1.95 (1); Os(2)-C(14), 2.14 (2); Os(3)-C(14), 2.15 (1); W-C(37), 2.30 (1); Os(3)-C(37), 2.14 (1); Os(1)-C(21), 2.25 (1); W-C(38), 2.36 (1); Os(1)-C(29), 2.22 (1); Os(3)-C(29), 2.07 (1); W-C(37), 2.29 (1); W-C(38), 2.36 (1); Os(2)-C(46), 2.19 (2); W-C(46), 2.09 (2); C(29)-C(21), 1.44 (2); C(21)-C(37), 1.54 (2); C(37)-C(38), 1.33 (2); C(38)-C(46), 1.51 (2). Bond angles (deg): C(29)-C(21)-C(37), 100 (1); C(21)-C(37)-C(38), 135 (1); W-C(37)-Os(3), 77 (1); C(37)-C(38)-C(46), 115 (1).

the reaction conditions are so severe that the degradation reaction cannot be used to fully account for the formation of these trinuclear species as the coproduct, during the initial preparation of complex 1 or 2. Hydrogenation of 2 in refluxing toluene solution (1 atm, 110 °C, 90 min) provided an alkylidyne complex, CpWOs<sub>3</sub>(CO)<sub>11</sub>( $\mu_3\text{-C}^{\text{C}}\text{C}_5\text{H}_{11}$ ) (5), in 45% yield. Characterization of the latter is provided by comparison of its spectroscopic data<sup>15</sup> with

(10) (a) Weatherell, C.; Taylor, N. J.; Carty, A. J.; Sappa, E.; Tiripicchio, A. *J. Organomet. Chem.* **1985**, *291*, C9. (b) Roland, E.; Vahrenkamp, H. *Organometallics* **1983**, *2*, 1048.

(11) Carty, A. J.; MacLaughlin, S. A.; Taylor, N. J. *J. Am. Chem. Soc.* **1981**, *103*, 2456.

(12) Lanfranchi, M.; Tiripicchio, A.; Sappa, E.; MacLaughlin, S. A.; Carty, A. J. *J. Chem. Soc., Chem. Commun.* **1982**, 538.

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(14) Selected spectroscopic data. Complex 3: see ref 6. Complex 4: MS (FAB, <sup>192</sup>Os, <sup>184</sup>W), *m/z* 938 (M<sup>+</sup>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, room temperature)  $\delta$  5.33 (s, 5 H, Cp), 3.03 (m, 2 H, CH<sub>2</sub>), 1.73 (m, 1 H, CH<sub>2</sub>), 1.60 (m, 1 H, CH<sub>2</sub>), 1.38 (m, 2 H, CH<sub>2</sub>), 0.94 (t, 3 H, CH<sub>3</sub>). Anal. Calcd for W<sub>1</sub>Os<sub>2</sub>C<sub>19</sub>H<sub>14</sub>O<sub>8</sub>: C, 24.42; H, 1.51. Found: C, 24.47; H, 1.54.

(15) Selected spectroscopic data for complex 5: MS (FAB, <sup>192</sup>Os, <sup>184</sup>W), *m/z* 1216 (M<sup>+</sup>); IR (C<sub>6</sub>H<sub>12</sub>)  $\nu$ (CO) 2079 (s), 2037 (vs), 2032 (vs), 2007 (s), 1994 (m), 1984 (s), 1973 (m), 1856 (w, br) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, room temperature)  $\delta$  5.33 (s, 5 H, Cp), 3.26 (t, 2 H, CH<sub>2</sub>), 2.06 (q, 2 H, CH<sub>2</sub>), 1.58 (q, 2 H, CH<sub>2</sub>), 1.46 (q, 2 H, CH<sub>2</sub>), 0.97 (t, 3 H, CH<sub>3</sub>). Anal. Calcd for W<sub>1</sub>Os<sub>3</sub>C<sub>22</sub>H<sub>16</sub>O<sub>11</sub>: C, 21.82; H, 1.33. Found: C, 21.80; H, 1.39.

those of the structurally characterized alkylidyne complex  $\text{CpW}(\text{Os}_3(\text{CO})_{11}(\mu_3\text{-CTol}))^{16}$ . Interestingly, hydrogenation of **1** under similar conditions failed to generate the expected alkylidyne complex  $\text{CpW}(\text{Os}_3(\text{CO})_{11}(\mu_3\text{-CCH}_2\text{Ph}))$ .

Reaction of **1** with excess ditolylacetylene in refluxing toluene (110 °C, 2 h) led to the isolation of a dark green complex,  $\text{CpW}(\text{Os}_3(\text{CO})_8(\mu_3\text{-CPh})[\mu_4\text{-}\eta^5\text{-C}(\text{C}_2\text{Tol})_2]_2)$  (**6**), as the only isolable product (62%), and no intermediate was observed during the reaction. This complex has been characterized by spectroscopic methods<sup>17</sup> and by a single-crystal X-ray diffraction study (Figure 2).<sup>18</sup>

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 ( $\mu_3\text{-CPh}$ ) which is associated with the triangular W–Os(2)–Os(3) system, and, on the opposite side of the  $\text{W}(\text{Os}_3)$  plane is a  $\text{C}_5$  hydrocarbyl moiety which interacts with all of the four transition-metal atoms. This  $\text{C}_5$  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  $\sigma$ -bond and to the Os(1) atom via a  $\pi$ -bond and with the C(38)–C(46) fragment bound to Os(2) atom and the W atom via a  $\sigma$ -bond and a  $\pi$ -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,<sup>19</sup> the coupling of a coordinated carbide atom and alkyne ligand,<sup>20</sup> and the consideration of acetylide ligands as alkylidyne carbides<sup>21</sup> 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;  $\text{CpW}(\text{CO})_3\text{C}\equiv\text{CPh}$ , 69140-93-8;  $\text{CpW}(\text{CO})_3\text{C}\equiv\text{CBu}$ , 120637-08-3;  $\text{Os}_3(\text{CO})_{10}(\text{CH}_3\text{CN})_2$ , 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.

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

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**Summary:** Heating the divinyl complex  $(\eta^5\text{-C}_5\text{Me}_5)\text{Ru}(\text{PMe}_3)(\text{CH}=\text{CH}_2)_2\text{Ru}(\eta^5\text{-C}_5\text{Me}_5)$  in toluene at 70 °C leads to the formation of a novel dinuclear ruthenacyclopentadiene complex,  $(\eta^5\text{-C}_5\text{Me}_5)\text{Ru}(\text{PMe}_3)(\mu\text{-}\eta^4\text{-C}_4\text{H}_4)\text{Ru}(\eta^5\text{-C}_5\text{Me}_5)$  (**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  $\sigma$ -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.<sup>1</sup>

In the previous paper, we reported that the dinuclear divinyl complex  $(\eta^5\text{-C}_5\text{Me}_5)\text{Ru}(\text{CH}_2=\text{CH}_2)(\text{CH}=\text{CH}_2)_2\text{Ru}(\eta^5\text{-C}_5\text{Me}_5)$  (**1**) was obtained by treatment of the tetrahydride-bridged dinuclear ruthenium complex  $(\eta^5\text{-C}_5\text{Me}_5)\text{Ru}(\mu\text{-H})_4\text{Ru}(\eta^5\text{-C}_5\text{Me}_5)$  with ethylene via an acti-

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(17) Selected spectroscopic data for complex **6**: MS (FAB,  $^{192}\text{Os}$ ,  $^{184}\text{W}$ ),  $m/z$  1562 ( $M^+$ ); IR ( $\text{C}_6\text{H}_{12}$ )  $\nu(\text{CO})$  2073 (s, sh), 2070 (vs), 2036 (vs, sh), 2034 (vs), 2019 (m, sh), 2014 (s), 1999 (s), 1991 (m), 1971 (vs), 1960 (s)  $\text{cm}^{-1}$ ;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 317 K)  $\delta$  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  $\text{W}_1\text{Os}_3\text{C}_{53}\text{H}_{38}\text{O}_8$ : C, 40.88; H, 2.46. Found: C, 40.83; H, 2.50.

(18) Crystal data for complex **6**:  $\text{W}_1\text{Os}_3\text{C}_{53}\text{H}_{38}\text{O}_8$ ;  $M = 1557.34$ ; monoclinic, space group  $P2_1$ ;  $a = 10.351$  (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<sup>3</sup>;  $Z = 2$ ;  $D_{\text{calc}} = 2.215$   $\text{mg}/\text{cm}^3$ ;  $F(000) = 1443.5$ ; Nonius CAD-4 diffractometer with graphite-monochromated Mo K $\alpha$  radiation;  $\lambda = 0.70930$  Å;  $\mu(\text{Mo K}\alpha) = 10.71$   $\text{mm}^{-1}$ .  $\psi$  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.

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