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Reversible C−C Bond Scission and C−H Bond Activation in the Butterfly Acetylide Clusters $Cp^*WOs_3(CO)_{11}(CCR)$ ($R = Ph, Bu, CH_2OMe$)

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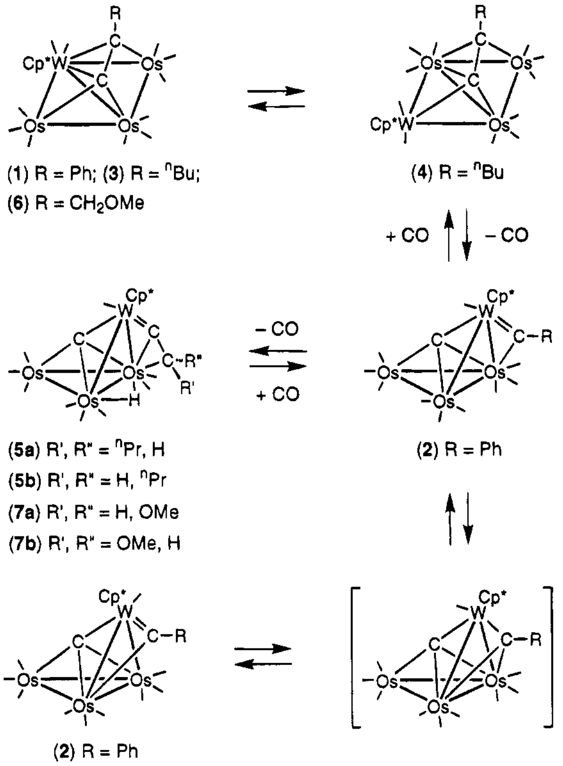
Summary: The tetranuclear acetylide compounds $Cp^*WOs_3(CO)_{11}(CCR)$ ($R = Ph, Bu, CH_2OMe$)*, depending on the substituents of the acetylide ligand, undergo reversible C−C bond scission and C−H activation to afford the carbido−alkylidyne cluster* $Cp^*WOs_3(CO)_{10}(\mu_4\text{-}C)(\mu\text{-}CPh)$ *and the carbido−vinylidene clusters* $Cp^*WOs_3(CO)_9(\mu_4\text{-}C)(\mu\text{-}H)(\mu\text{-}CCHR')$ ($R' = Pr, OMe$)*, respectively.*

The chemistry of metal acetylide complexes has been the subject of intense study in recent years.[1] Condensation with terminal alkynes,[2] phosphinoacetylenes,[3] and mononuclear acetylide complexes[4] has provided versatile synthetic entries for a variety of such complexes. Their successful synthesis has not only led to the observation of fascinating structural chemistry but also formed a basis for understanding the reactivity of chemisorbed acetylides on metal surfaces.[5] Attention is now focused on their novel chemical transformation, which requires multisite interaction of the polynuclear frameworks.[6] Of particular interest in this regard is the conversion between acetylides and alkylidyne carbides.[7] This transformation represents a possible pathway for the conversion of interstitial carbides to hydrocarbon fragments of higher carbon content, a process which has been recognized to play an important role in the CO reduction and activation of hydrocarbons on catalytic surfaces.[8] Here we report the results of our investigation on the reactivity of unique WOs_3 acetylide clusters which offers an informative comparison.

Scheme 1



The butterfly cluster $Cp^*WOs_3(CO)_{11}(CCPh)$ (**1**), which contains a hinge W atom and a multisite-bound acetylide ligand (Scheme 1), was obtained directly from combination of the triosmium cluster $Os_3(CO)_{10}(NCMe)_2$ and the mononuclear acetylide complex $Cp^*W(CO)_3(CCR)$ according to the published method.[9] When a solution of **1** was allowed to react with 1.1 equiv of anhydrous Me_3NO in dichloromethane/acetonitrile solution at room temperature (30 min), followed by heating in refluxing toluene (10 min), a stable orange product (**2**) was formed in 80% yield, which was purified by chromatography followed by recrystallization from a

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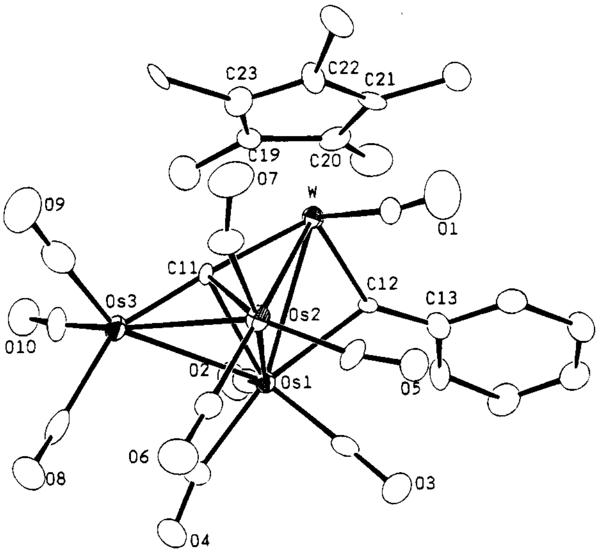

Figure 1. Molecular structure of **2** and the atomic numbering scheme. Selected bond lengths (Å): W−Os(1) = 2.896(3), W−Os(2) = 2.974(3), Os(1)−Os(2) = 2.751(2), Os(1)−Os(3) = 2.899(2), Os(2)−Os(3) = 2.850(3), W−C(11) = 2.10(4), Os(1)−C(11) = 2.22(4), Os(2)−C(11) = 2.12(4), Os(3)−C(11) = 1.81(4), W−C(12) = 1.89(3), Os(1)−C(12) = 2.22(4). Selected bond angles (deg): W−C(11)−Os(3) = 175(3), Os(1)−C(11)−Os(2) = 79(1), W−C(12)−C(13) = 142(3).

mixture of chloroform and methanol.[10] The stoichiometry was initially confirmed by FAB mass analysis, which gave a parent ion at *m/z* 1276, showing that this complex contains one CO less than its precursor. The loss of CO ligand strongly suggested the formation of carbide and alkylidyne species via fission of the acetylide C−C bond. Thus, an X-ray diffraction study was carried out to reveal the identity of this compound.

Crystals of **2** contain two crystallographically distinct, but structurally similar, molecules in the asymmetric unit.[11] A perspective view of one of these molecules is depicted in Figure 1. The overall structure consists of a butterfly WOs_3 arrangement with an acute dihedral angle (99.5(1)°). The W atom is located at a wingtip position. A similar skeletal arrangement has been observed in the carbide clusters $CpWRu_3(CO)_{11}(\mu_4\text{-}C)(\mu\text{-}H)$[12] and $CpWOs_3(CO)_{11}(\mu_4\text{-}C)(\mu\text{-}SMe)$[13] and in the carbonyl clusters $Cp^*MRu_3(CO)_{11}(\mu_4\text{-}CO)(\mu\text{-}H)$ (M = Mo, W), with a quadruply bridging CO ligand.[14] The carbido atom C(11), liberated from the acetylide fragment, is bound to the cluster with short M(wingtip)−C distances (average 1.96(4) Å) and long M(hinge)−C distances (average 2.17(4) Å), typical for such carbides in a butterfly environment.[15] The alkylidyne ligand bridges the W−Os(1) edge with angles W−C(12)−C(13) = 142(3)°, Os(1)−C(12)−C(13) = 127(3)°, and W−C(12)−Os(1) = 89(1)°. Since these structural features invoke a trigonal-planar carbon atom, a substantial degree of W−C double-bond interaction is indicated (W−C(12) = 1.89(3) Å).

In accordance with the solid-state structure, the ^{13}C NMR spectrum showed two signals at δ 357.0 (J_{W-C} = 104 Hz) and 293.9 (J_{W-C} = 150 Hz), which were assigned to the carbido and alkylidyne carbon atoms, respectively. Moreover, the ^{13}C NMR spectrum recorded at 253 K showed one W−CO signal at δ 226.7 and eight Os−CO resonances in the region δ 187.2−164.9 with one signal at δ 182.1 possessing intensity equivalent to two CO ligands. When the temperature was raised to 338 K, the signal at δ 164.9 broadened substantially and the seven Os−CO signals in the range δ 187.2−173.3 coalesced to one broad signal centered at δ 181.3. Although this exchange pattern supports the onset of alkylidyne migration from one W−Os edge to the second via the formation of a μ_3-alkylidyne intermediate on the WOs_2 plane (Scheme 1), it is possible that this migration requires a higher reaction temperature.

The related *n*-butyl-substituted acetylide complex **3** and its isomer **4**, in which the W atom resides at the wingtip position, were prepared and subjected to the same decarbonylation reaction in attempts to extend the scope of the reaction. Unexpectedly, both complexes **3** and **4** slowly eliminated two CO ligands in refluxing toluene solution (30 min) to afford the vinylidene complexes $Cp^*WOs_3(CO)_9(\mu_4\text{-}C)(\mu\text{-}H)(\mu\text{-}CCH^nPr)$ as a mixture of two isomers (**5a**:**5b** = 4:3) in 75% yield.[16] Their identities were revealed on the basis of the NMR data, and they are obviously caused by the asymmetric nature of the vinylidene fragment. In the ^1H NMR spectrum, two sets of signals are observed at δ 4.08 and −24.23 and at δ 4.15 and −24.29, indicating the presence of a $C{=}CH^nPr$ vinylidene ligand and a bridging hydride ligand (Scheme 1). The ^{13}C NMR spectrum was

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[10] Spectral data for **2**: MS (FAB, ^{184}W, ^{192}Os) *m/z* 1276 (M^+); IR (C_6H_{12}) ν(CO) 2076 (s), 2040 (vs), 2021 (s), 2003 (s), 1981 (m, br), 1976 (vw), 1958 (w), 1950 (vw), 1922 (vw, br) cm^{-1}; ^1H NMR ($CDCl_3$, 294 K) δ 2.51−2.47 (m, 3H), 7.37 (t, 2H, J_{H-H} = 7.3 Hz), 1.99 (s, 15H, C_5Me_5); ^{13}C NMR ($CDCl_3$, 258 K) δ 357.0 (μ_4-*C*, J_{W-C} = 104 Hz), 293.9 (μ-*C*Ph, J_{W-C} = 150 Hz), 226.7 (CO, J_{W-C} = 178 Hz), 187.2 (CO, br), 182.1 (2CO), 181.3 (CO), 179.4 (CO), 178.8 (CO, br), 175.1 (CO, br), 173.3 (CO), 164.9 (CO), 155.8 (*i*-C_6H_5), 130.1 (*o,m*-C_6H_5), 129.8 (*m,o*-C_6H_5), 128.6 (*p*-C_6H_5), 107.1 (C_5Me_5), 11.0 (C_5Me_5). Anal. Calcd for $C_{28}H_{20}O_{10}Os_3W$: C, 26.46; H, 1.59. Found: C, 26.52; H, 1.63.

[11] Crystal data for **2**: $C_{28}H_{20}O_{10}Os_3W$, M_r = 1270.90, orthorhombic, space group *Pca*21, a = 30.773(5) Å, b = 9.678(2) Å, c = 20.561(3) Å, V = 6124(2) $Å^3$, Z = 8, ρ_{calcd} = 2.757 g cm^{-3}, $F(000)$ = 4518, λ(Mo Kα) = 0.7107 Å, T = 298 K, μ = 163.0 cm^{-1}. The intensities were measured on a Nonius CAD4 diffractometer on a crystal with dimensions 0.20 × 0.20 × 0.25 mm. Of the 5546 unique reflections collected, 3139 reflections with $I > 2\sigma(I)$ were used for the refinement. The structure was solved by using the NRCC-SDP-VAX package and refined to R_F = 0.053, R_w = 0.047, and GOF = 1.21 for 757 parameters, weighting scheme $w^{-1} = \sigma^2(F_o) + 0.00002F_o^2$, and highest Δ/σ ratio 0.07. A difference map following convergence showed residual electron density within the range −1.49 to +2.24 e/$Å^3$ (min−max). In addition, this crystal belongs to the noncentric space group $Pca2_1$. The presence of two crystallographically distinct molecules in the asymmetric unit doubles the number of variables used in the refinement, which gives a relatively poor data-to-parameter ratio (4.2:1) and undesired large standard deviations for the bond distances and angles.

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[16] Spectral data for **5**: MS (FAB, ^{184}W, ^{192}Os) *m/z* 1228 (M^+); IR (C_6H_{12}) ν(CO) 2075 (m), 2044 (vs), 2008 (s), 2002 (m), 1995 (s), 1976 (w), 1959 (m), 1946 (vw), 1931 (vw, br) cm^{-1}; ^1H NMR ($CDCl_3$, 294 K) isomer **a** δ 4.08 (t, C*H*, J_{H-H} = 7.4 Hz), 2.10 (s, 15H, C_5Me_5), 1.69 (m, 1H), 1.56 (m, 1H), 1.00 (t, CH_3, J_{H-H} = 7.4 Hz), and −24.23 (s, 1H) isomer **b** δ 4.15 (t, C*H*, J_{H-H} = 7.4 Hz), 2.15 (s, 15H, C_5Me_5), 1.68 (m, 1H), 1.48 (m, 1H), 1.02 (t, CH_3, J_{H-H} = 7.4 Hz), and −24.29 (s, 1H); ^{13}C NMR ($CDCl_3$, 294 K) isomer **a** δ 343.1 (μ_4-C, J_{W-C} = 100 Hz), 273.1 (*C*CH^{i}Pr, J_{W-C} = 100 Hz), 222.6 (CO, J_{W-C} = 169 Hz), 186.7 (CO), 180.2 (CO), 175.6 (CO), 172.5 (CO), 166.0 (CO), 106.4 (C_5Me_5), 65.1 (C*C*H^{i}Pr), 36.2 (CH_2), 26.9 (CH_2), 13.3 (CH_3), and 11.0 (C_5Me_5) isomer **b** δ 345.7 (μ_4-C, J_{W-C} = 100 Hz), 269.5 (*C*CH^{n}Pr, J_{W-C} = 100 Hz), 221.2 (CO, J_{W-C} = 169 Hz), 187.3 (CO), 179.5 (CO), 175.3 (CO), 173.3 (CO), 166.1 (CO), 106.5 (C_5Me_5), 65.0 (C*C*H^{i}Pr), 34.2 (CH_2), 27.4 (CH_2), 13.5 (CH_3), 11.2 (C_5Me_5). Anal. Calcd for $C_{25}H_{24}O_9Os_3W$: C, 24.55; H, 1.98. Found: C, 24.53; H, 2.02.

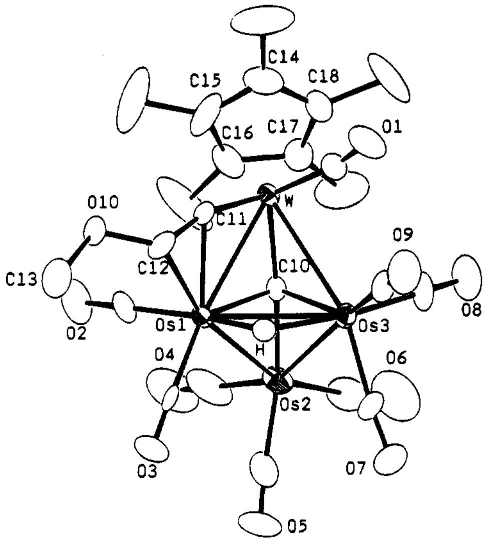

Figure 2. Molecular structure of **7a** and the atomic numbering scheme. Selected bond lengths (Å): W–Os(1) = 2.840(2), W–Os(3) = 2.985(1), Os(1)–Os(2) = 2.815(1), Os(1)–Os(3) = 2.883(1), Os(2)–Os(3) = 2.857(1), W–C(10) = 2.02(2), Os(1)–C(10) = 2.13(1), Os(2)–C(10) = 1.91(2), Os(3)–C(10) = 2.18(1), W–C(11) = 1.93(2), Os(1)–C(11) = 2.18(2), Os(1)–C(12) = 2.27(2), C(11)–C(12) = 1.36(2). Selected bond angles (deg): W–C(10)–Os(2) = 174.4(8), Os(1)–C(10)–Os(3) = 84.0(5), W–C(11)–C(12) = 163(1).

interpreted accordingly. In addition to the carbide and the W–CO resonances, the ^{13}C resonances arising from the α- and β-carbons of the vinylidene group in **5a** appeared at δ 273.1 (J_{W-C} = 100 Hz) and 65.1, while those of **5b** occurred at δ 269.5 (J_{W-C} = 100 Hz) and 65.0. We propose that the dominant isomer **5a** contains the *exo* hydrogen, while the less abundant **5b** has an *endo* hydrogen atom, as a positive NOE enhancement (≥3.6%) was observed for the hydride signal of **5b** upon irradiating the respective vinylidene hydrogen signal.

The identification of the previously mentioned vinylidene complexes was further confirmed by the characterization of the two methoxy derivatives Cp*$WOs_3(CO)_9(\mu_4\text{-}C)(\mu\text{-}H)(\mu\text{-}CCHOMe)$ (**7a,b**), which were obtained from the thermolysis of the acetylide derivative Cp*$WOs_3(CO)_{11}(CCCH_2OMe)$ (**6**).[17] The methoxy derivatives **7a,b** were isolated in pure form by TLC separation and recrystallization. An X-ray diffraction study on **7a** confirmed that the molecule possesses the WOs_3 butterfly arrangement with dihedral angle 104.24(3)° (Figure 2),[18] which is essentially identical with that of **2**. The hydride, which is located on the difference Fourier map, spans the slightly elongated hinge Os–Os bond. The vinylidene ligand, containing a linear W–C–C skeleton and an *endo* hydrogen atom, is best visualized as possessing a W=C double bond and as π-bonding to the adjacent Os atom. These parameters are in agreement with those observed for the "side-on" coordinated vinylidene ligands of dinuclear and polynuclear systems.[19]

The pathways leading to the formation of vinylidene from acetylide ligands were easily established (Scheme 1), which consist of (i) cluster skeletal rearrangement, (ii) C–C bond cleavage, and (iii) C–H bond activation. Our previous experiments suggested that the W atom in this butterfly system can undergo migration between the wingtip and the hinge sites through a process involving reversible cleavage of the M–M bond.[17] After the occurrence of cluster skeletal rearrangement, the acetylide complexes then liberate one CO ligand to induce the scission of the C–C bond and to afford the carbide and alkylidyne fragments. If the acetylide ligands contained a methylene group adjacent to the C_2 fragment, the reaction proceeded further to the production of vinylidene via hydrogen activation, a process which is akin to that observed in the metal clusters and crystal surfaces.[20] In the present system, two configurations are anticipated due to the poor selectivity in abstracting the diastereotopic methylene hydrogen atoms.

Finally, in view of these pronounced changes starting from the coordinated acetylide, it is even more remarkable that the carbide in both **2** and **5** can be transformed back to the acetylide upon addition of CO. Thus, exposure of **2** to CO (110 °C, 5 min) resulted in the formation of **1** in 85% yield, whereas the reaction of **5** proceeded much more slowly (40 min, 65% completion) to afford **3** and **4** in 61% combined yield and a trace amount of the trinuclear acetylide cluster Cp*$WOs_2(CO)_8(CCBu)$ due to fragmentation.[9] This rarely reported pattern of reactivity highlights the exciting chemistry of carbide in reacting with small organic hydrocarbon fragments.[21] An extensive investigation regarding C–C bond formation with these carbido cluster compounds will be described in the future.

Acknowledgment. We are grateful to the National Science Council of the Republic of China for financial support (Grant No. NSC 85-2113-M007-008).

Supporting Information Available: Text describing the experimental details and the spectroscopic data for **1**, **3**, **4**, and **7** and full details of crystal structure analyses, including tables of bond distances, atomic coordinates, and anisotropic thermal parameters, for **2** and **7a** (14 pages). Ordering information is given on any current masthead page.

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(18) Crystal data for **7a**: $C_{23}H_{20}O_{10}Os_3W$, M_r = 1210.84, monoclinic, space group $P2_1/n$, a = 10.666(5) Å, b = 14.779(3) Å, c = 18.540(5) Å, β = 103.62(3)°, V = 2841(2) Å^3, Z = 4, ρ_{calcd} = 2.831 g cm^{-3}, $F(000)$ = 2139, λ(Mo Kα) = 0.7107 Å, T = 298 K, μ = 175.7 cm^{-1}. The intensities were measured on a crystal with dimensions 0.25 × 0.30 × 0.50 mm. Of the 4998 unique reflections collected, 3635 reflections with $I > 2\sigma(I)$ were used for the refinement. The structure was refined to R_F = 0.037, R_w = 0.037, and GOF = 1.83 for 339 parameters, weighting scheme $w^{-1} = \sigma^2(F_o) + 0.00005F_o^2$, and highest Δ/σ ratio 0.008. A difference map following convergence showed residual electron density within the range −1.38 to +2.49 e/Å^3 (min−max).

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