

Transfer of an aryl group from triphenylphosphine to a diruthenium species has been reported to take place along with a synergic joining of an oxygen atom from a bridging amide to the phosphorus atom.²⁵ Also, in the fast rhodium-catalyzed decomposition of triphenylphosphine in the presence of formaldehyde²⁶ we propose that the first step involves attack of the methoxy ligands at the coordinated phosphine. The rearrangement of 2 to 3 is the first well-defined case of an alkoxy ligand assisted phosphorus-carbon bond cleavage in a triarylphosphine. It shows that ortho metalation or cluster-assisted reactions are not necessarily involved in P-C bond cleavage. The present P-C/Pt-O metathesis may have been somewhat overlooked as a phosphine decomposition mode of homogeneous catalysts.^{27,28} It may be especially important in

syngas reactions where metal alkoxides are likely intermediates.

Registry No. 1, 126135-74-8; 2, 127973-52-8; 3, 127973-53-9; 3-C₇H₈, 127973-55-1; 4, 127973-54-0; *o*-(diphenylphosphino)benzaldehyde, 50777-76-9; 1-phenyl-3*H*-2,1-benzoxaphosphole, 79157-82-7.

Supplementary Material Available: Details of the structure determination and listings of crystal data, positional and thermal parameters, and bond distances and angles for 3 (7 pages); a listing of observed and calculated structure factors for 3 (28 pages). Ordering information is given on any current masthead page.

(27) A reviewer pointed out that there still is the possibility that a classical P-C(phenyl) bond splitting occurs, preceded by Pt-alkoxide dissociation, followed by attack of the alkoxide at the phosphido intermediate. Prolonged hydroformylation experiments did not show any P-C bond breaking of tris(*p*-tolyl)phosphine.¹⁴

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(26) Kaneda, K.; Sano, K.; Teranishi, S. *Chem. Lett.* 1979, 821. In a typical reaction 0.05 mmol of Rh₂(CO)₁₀, 1 mmol of PPh₃, and 2.5 mmol of paraformaldehyde were heated at 80 °C under 6-8 bar of CO in dioxane (1% H₂O) for 24 h. Analysis showed that 1 mmol of benzaldehyde was formed. Phosphorus NMR spectra showed a variety of phenyl- and diphenylphosphorus oxide complexes or, more precisely, their formaldehyde adducts.

Synthesis of Isomeric Complexes (μ-H)W₂(CO)₇(RNC)₂(NO) (R = Me, ^tBu, PhCH₂): X-ray Structures of (μ-H)W₂(CO)₇(THF)₂(NO) and Isomeric (μ-H)W₂(CO)₇(MeNC)₂(NO) with Bent Staggered/Eclipsed Conformations

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Summary: Reactions of (μ-H)W₂(CO)₉(NO) and (μ-H)W₂(CO)₇(THF)₂(NO) with excess RNC (R = Me, ^tBu, PhCH₂) afford the yellow complexes (μ-H)W₂(CO)₇(RNC)₂(NO) and reddish isomers, respectively; X-ray structural studies on (μ-H)W₂(CO)₇(THF)₂(NO) and an isomeric pair of (μ-H)W₂(CO)₇(MeNC)₂(NO) complexes are described. The reddish isomers of the type (μ-H)W₂(CO)₇(MeNC)₂(NO) exhibit bent, eclipsed conformations.

Transition-metal complexes with bridging hydride ligands have attracted considerable interest in the past.¹ It is believed that a linear M-H-M geometry is inherently less stable than a bent geometry since the latter permits a smaller M-M distance with a stronger 3c-2e bonding interaction.² A recent report suggested that the M-H-M bond can in fact be described as flexible in the case of carbonyl dimers with single hydrogen bridges.³ To date

only two complexes, HCr₂(CO)₁₀⁻ and HW₂(CO)₁₀⁻, have been found to have both carbonyl-staggered and carbonyl-eclipsed geometries,^{2,4} however. We now report that both staggered and eclipsed conformations also exist in the isocyanide derivatives of HW₂(CO)₉(NO),⁵ HW₂(CO)₇(RNC)₂(NO). Earlier we⁶ characterized several phosphine-substituted derivatives of HW₂(CO)₉(NO), all of which possess a bent W-H-W linkage with the equatorial groups staggered and the nitrosyl ligand occupying the axial position on the phosphine-substituted tungsten, similar to the case for HW₂(CO)₈(P(OMe)₃)(NO).⁷

Reaction of HW₂(CO)₉(NO) with excess RNC (R = Me, ^tBu, PhCH₂) in CH₂Cl₂ at 25 °C for 3 h affords, after subsequent workup, the yellow complexes (μ-H)W₂(CO)₇(RNC)₂(NO) in 33, 36, and 6% yields for R = ^tBu

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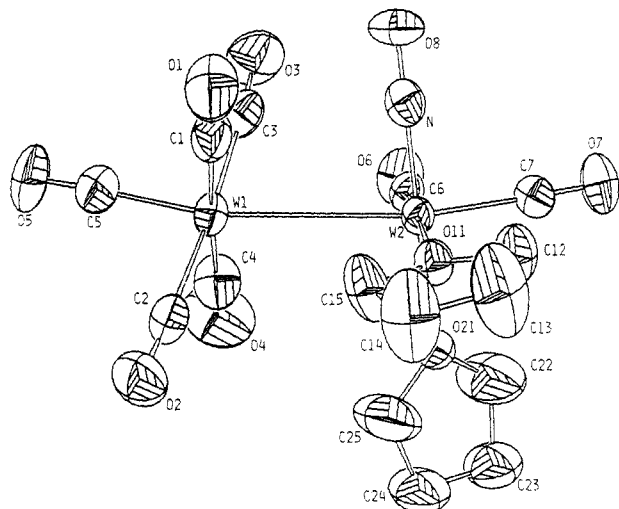


Figure 1. Molecular structure of **7**. Important bond lengths (Å): W(1)–W(2) = 3.418(1), W(1)–C(1) = 2.038 (9), W(1)–C(2) = 2.049 (9), W(1)–C(3) = 2.023 (9), W(1)–C(4) = 2.013 (9), W(1)–C(5) = 1.968 (9), W(2)–N = 1.825 (7), W(2)–O(11) = 2.216 (5), W(2)–O(21) = 2.205 (5), W(2)–C(6) = 1.908 (8), W(2)–C(7) = 2.002 (8). Important bond angles (deg): W(2)–N–O(8) = 177.6 (6), W(2)–O(11)–C(12) = 122.7 (4), W(2)–O(11)–C(15) = 128.1 (4), W(2)–O(21)–C(22) = 124.9 (6), W(2)–O(21)–C(25) = 129.9 (6).

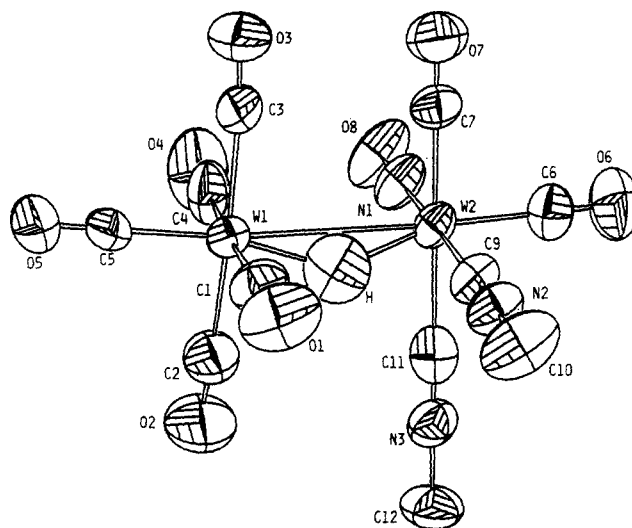


Figure 3. Molecular structure of **5**. Important bond lengths (Å): W(1)–W(2) = 3.4800 (9), W(1)–C(1) = 2.04 (2), W(1)–C(2) = 2.01 (1), W(1)–C(3) = 2.03 (1), W(1)–C(4) = 1.98 (2), W(1)–C(5) = 1.94 (1), W(1)–H = 1.8 (1), W(2)–N(1) = 1.84 (1), W(2)–C(6) = 1.99 (1), W(2)–C(7) = 1.99 (1), W(2)–C(9) = 2.18 (1), W(2)–C(11) = 2.17 (2), W(2)–H = 1.8 (1). Important bond angles (deg): W(2)–N(1)–O(8) = 178 (1), W(2)–C(9)–N(2) = 178 (1), W(2)–C(11)–N(3) = 174 (1), C(9)–N(2)–C(10) = 177 (1), C(11)–N(3)–C(12) = 179 (2), W(1)–H–W(2) = 143 (7).

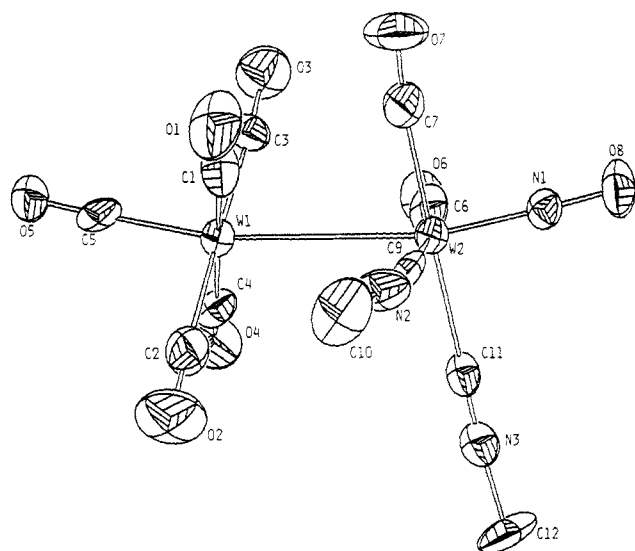


Figure 2. Molecular structure of **2**. Important bond lengths (Å): W(1)–W(2) = 3.389 (1), W(1)–C(1) = 2.06 (2), W(1)–C(2) = 2.07 (3), W(1)–C(3) = 1.96 (2), W(1)–C(4) = 2.00 (2), W(1)–C(5) = 1.95 (2), W(2)–N(1) = 1.83 (2), W(2)–C(6) = 2.00 (2), W(2)–C(7) = 2.03 (2), W(2)–C(9) = 2.19 (2), W(2)–C(11) = 2.18 (2), N(2)–C(9) = 1.12 (3), N(3)–C(11) = 1.13 (3). Important bond angles (deg): W(2)–N(1)–O(8) = 173 (2), W(2)–C(9)–N(2) = 170 (2), C(9)–N(2)–C(10) = 177 (2), W(2)–C(11)–N(3) = 176 (2), C(11)–N(3)–C(12) = 175 (2), N(1)–W(2)–C(9) = 97.4 (7), N(1)–W(2)–C(11) = 89.5 (7), N(1)–W(2)–C(6) = 93.5 (8), N(1)–W(2)–C(7) = 89.3 (8).

(1), R = Me (2), and R = PhCH₂ (3), respectively. Much lower yields of red isomers are also obtained: a 4% yield for R = ^tBu (4) and trace amounts for R = Me (5) and R = PhCH₂ (6).⁸ However, with (μ-H)W₂(CO)₉(THF)₂(NO) (**7**) instead of HW₂(CO)₉(NO) as starting material, the red isomers can be isolated in much higher yields (82, 88, and 67%, respectively).⁹ The orange starting material **7** can

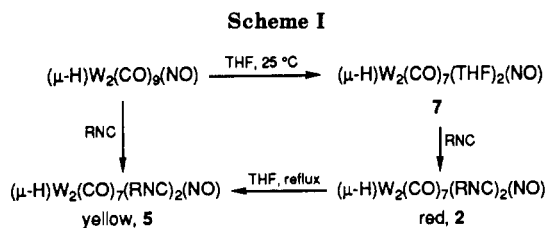
be conveniently prepared from the reaction of HW₂(CO)₉(NO) with THF at room temperature in 76% yield.

The structures of **7**, **2**, and **5** in the solid state were characterized by X-ray diffraction studies,¹⁰ and ORTEP drawings are shown in Figures 1–3, respectively. The metal–metal distances (**7**, 3.418 (1) Å; **2**, 3.389 (1) Å; **5**,

(9) The new complexes were identified by satisfactory elementary analyses and by IR and ¹H NMR spectroscopy. Selected data (IR in CH₂Cl₂, NMR in acetone-*d*₆ with coupling constants in Hz): **1**, ν(CN) at 2190 w and 2170 w cm⁻¹, ν(CO) at 2065 w, 2028 s, 1977 s, and 1925 vs cm⁻¹, ν(NO) at 1648 m cm⁻¹; ¹H NMR δ 1.86 (s, 18 H) and -12.6 (s with satellites, *J* = 32, 43; 1 H); **2**, ν(CN) at 2220 w and 2201 w cm⁻¹, ν(CO) at 2067 w, 2033 s, 1980 s, and 1926 vs cm⁻¹, ν(NO) at 1651 m cm⁻¹; ¹H NMR δ 3.88 (s, 6 H) and -12.6 (s with satellites, *J* = 32, 43; 1 H); **3**, ν(CN) at 2204 w and 2183 w cm⁻¹, ν(CO) at 2066 w, 2032 s, 1982 s, and 1925 vs cm⁻¹, ν(NO) at 1655 m cm⁻¹; ¹H NMR δ 7.63–7.54 (m, 10 H), 5.55 (s, 4 H), and -12.3 (s with satellites, *J* = 34, 43; 1 H); **4**, ν(CN) at 2202 w and 2179 w cm⁻¹, ν(CO) at 2063 w, 2019 s, 1949 s, 1925 vs, and 1896 sh cm⁻¹, ν(NO) at 1682 m cm⁻¹; ¹H NMR δ 1.78 (s, 9 H), 1.69 (s, 9 H), and -9.56 (s with satellites, *J* = 42, 50; 1 H); **5**, ν(CN) at 2230 w and 2210 w cm⁻¹, ν(CO) at 2064 w, 2022 s, 1947 s, 1921 vs, and 1892 sh cm⁻¹, ν(NO) at 1688 m cm⁻¹; ¹H NMR δ 3.88 (s, 3 H), 3.78 (s, 3 H), and -9.49 (s with satellites, *J* = 44, 53; 1 H); **6**, ν(CN) at 2217 w, 2188 w cm⁻¹, ν(CO) at 2064 w, 2021 s, 1951 s, 1924 vs, and 1900 sh cm⁻¹, ν(NO) at 1685 m cm⁻¹; ¹H NMR δ 7.44–7.24 (m, 10 H), 5.11 (s, 2 H), 4.86 (s, 2 H), and -9.46 (s with satellites, *J* = 41, 54; 1 H); **7**, ν(CO) (in THF) at 2064 w, 2002 m, 1969 w, 1928 vs, and 1904 m-s cm⁻¹, ν(NO) at 1662 m cm⁻¹; ¹H NMR δ 4.61–4.23 (m, 4 H), 4.05–3.92 (m, 4 H), 2.20–2.10 (m, 4 H), 2.08–1.96 (m, 4 H), and -5.21 (s with satellites, *J* = 50, 60; 1 H).

(10) Crystals of complexes **2** and **5** suitable for X-ray diffraction measurements were grown by cooling a concentrated solution of the complex in CH₂Cl₂/hexane (1:5) at -5 °C for several days. Crystals of complex **7** were grown by slow diffusion of hexane into a concentrated solution of **7** in THF. Crystal data for **2**: C₁₁H₆N₃O₈W₂, *M_r* = 675.9, orthorhombic, space group *Pna*2₁, *a* = 13.402 (3) Å, *b* = 8.386 (1) Å, *c* = 15.510 (2) Å, *V* = 1743.2 (5) Å³, *Z* = 4, *D_c* = 2.58 g cm⁻³, *F*(000) = 1223.5, graphite-monochromated Mo Kα X-radiation (λ = 0.709 30 Å), μ(Mo Kα) = 13.51 mm⁻¹. The structure was solved by heavy-atom methods and refined by least squares to *R* = 0.030 for 1569 unique, observed reflections (*I* > 2.0σ(*I*)), absorption-corrected intensities with 0 < 2θ < 56.8°, collected on an Enraf-Nonius CAD-4 diffractometer. **5**: C₁₁H₆N₃O₈W₂, *M_r* = 675.9, triclinic space group *P1*, *a* = 8.457 (2) Å, *b* = 8.8284 (9) Å, *c* = 12.757 (1) Å, α = 90.789 (8)°; β = 96.34 (1)°, γ = 104.99 (1)°, *V* = 892.8 (3) Å³, *Z* = 2, *D_c* = 2.51 g cm⁻³, *F*(000) = 305.9, μ(Mo Kα) = 6.60 mm⁻¹, *R* = 0.027 for 2206 unique, observed reflections (*I* > 2.0σ(*I*)), ω < 2θ < 49.8°. **7**: C₁₅H₁₆N₃O₁₀W₂, *M_r* = 737.7, triclinic, space group *P1*, *a* = 8.454 (2) Å, *b* = 9.396 (2) Å, *c* = 14.951 (3) Å, α = 97.68 (2)°, β = 98.99 (2)°, γ = 113.48 (3)°, *V* = 1050.4 (4) Å³, *Z* = 2, *D_c* = 2.33 g cm⁻³, *F*(000) = 683.8, μ(Mo Kα) = 11.23 mm⁻¹, *R* = 0.023 for 2941 unique, observed reflections (*I* > 2.5σ(*I*)), 0 < 2θ < 49.8°.

(8) The crude residue was chromatographed on silica gel column under nitrogen. Careful elution with hexane and then with CH₂Cl₂/hexane (1:4) served to remove the yellow band, which was evaporated to yield the yellow product **1** (or **2**, **3**). Further elution with CH₂Cl₂/hexane (1:1) gave the red product **4** (or **5**, **6**).



3.4800 (9) Å) are somewhat longer than that in $\text{HW}_2(\text{C}-\text{O})_9(\text{NO})$ (3.329 (1) Å)⁵ or in the unsupported complex $\text{Cp}_2\text{W}_2(\text{CO})_6$ (3.222 (1) Å).¹¹ To our knowledge, compound **7** is the first structurally characterized tungsten(0) complex that contains THF ligands. The following structural features for **7** are all very similar to those in disubstituted phosphine derivatives⁶ of $\text{HW}_2(\text{CO})_9(\text{NO})$: (a) the two tungsten atoms with a W-H-W linkage reside in roughly octahedral environments, (b) the overall structure is bent (with the framework bend¹² of 23.9 (2)°) and the equatorial groups are staggered, and (c) the two mutual cis THF ligands are coordinated to the tungsten atom containing the NO ligand and occupy the equatorial exo positions which are less sterically hindered. However, the NO ligand is trans to one of the THF ligands instead of being in the axial position. The overall structure of **2** closely resembles that of **7** except that the NO ligand is in the axial position. It has a framework bend of 24.9 (7)° and an average degree of staggering at 45 (2)°. Complex **5** has an almost perfect eclipsed conformation with an average torsional angle of 2 (1)°. The two MeNC ligands are also mutually cis, and the NO ligand resides in an equatorial site. Unlike the complexes $[\text{Et}_4\text{N}][\text{HCr}_2(\text{CO})_{10}]$,^{4b} $[\text{Et}_4\text{N}][\text{DCr}_2(\text{CO})_{10}]$,^{4a} and $[\text{Et}_4\text{N}][\text{HW}_2(\text{CO})_{10}]$,^{4c} molecule **5** is not subjected to crystallographic symmetry constraints, and its slightly bent structure (8.4 (4)° for the framework bend) is unambiguous. The position of $\mu\text{-H}$ in **2** was located in the final difference Fourier maps and refined. The observed W-H distances (1.8 (1) Å) are in good agreement with the reported values (1.8–1.9 Å) for the analogous complexes, and the W-H-W angle (143 (7)°) appears to be larger than those frequently observed (115–135°).^{2b} The interatomic

distances between equatorial ligands on the halves of the dimer are as follows: C(2)–C(11), 3.79 Å; C(1)–C(9), 3.42 Å; C(4)–N(1), 3.32 Å; C(3)–C(7), 3.22 Å. Dahl suggested that interatomic van der Waals forces caused the conformational change in the $\text{HCr}_2(\text{CO})_{10}^-$ and $\text{Cr}_2(\text{CO})_{10}^{2-}$ anions,^{4d} whether the different conformations in the isomeric pair **2**, **5** can be accounted for solely by the same factor will be the subject of future study.

The spectroscopic properties⁹ for complexes **1**–**7** are consistent with their formulation. Being trans to CO and NO, respectively, the two RNC or THF ligands in **4**–**7** are magnetically inequivalent in the ¹H NMR spectra, in contrast to those in **1**–**3**.

In refluxing THF solution, complex **5** is converted to **2** completely within 5 h, whereas there is no interconversion between the two under ambient conditions. Apparently the isomeric complexes form from $\text{HW}_2(\text{CO})_{10}(\text{NO})$ via different pathways. Whether cleavage of the W-H-W linkage occurs in any aforementioned reactions is not clear at present. Our successful isolation of $\text{W}(\text{CO})_5(\text{py})$ from the reaction of $\text{HW}_2(\text{CO})_9(\text{NO})$ with pyridine indicates that cleavage of the dimer might occur in certain circumstances.

The principal reactions in this study are summarized in Scheme I. The synthetic application of these new complexes is currently under investigation. Our preliminary results indicate that complex **7** can initiate the polymerization of norbornadiene and 2,3-dihydrofuran. Treatment of **3** with iodine results in the formation of $\text{cis-}(\text{tBuNC})_2\text{W}(\text{CO})_2(\text{NO})\text{I}$, reported by King.¹³

Acknowledgment. We wish to thank the National Science Council of the Republic of China for support and Professor M. Y. Darensbourg for helpful discussions.

Registry No. **1**, 127973-56-2; **2**, 127973-57-3; **3**, 127973-58-4; **4**, 128050-99-7; **5**, 128051-00-3; **6**, 128051-01-4; **7**, 127973-59-5; $\text{HW}_2(\text{CO})_9(\text{NO})$, 40270-21-1; $\text{W}(\text{CO})_5(\text{py})$, 14586-49-3; $\text{cis-}(\text{tBuNC})_2\text{W}(\text{CO})_2(\text{NO})\text{I}$, 52699-24-8; MeNC, 593-75-9; ^tBuNC, 7188-38-7; PhCH_2NC , 10340-91-7; norbornadiene, 121-46-0; 2,3-dihydrofuran, 1191-99-7.

Supplementary Material Available: Tables of atomic coordinates, all bond distances and angles, and anisotropic thermal parameters for **2**, **5**, and **7** and a table of elemental analyses for **1**–**7** (12 pages); tables of structure factors for **2**, **5**, and **7** (45 pages). Ordering information is given on any current masthead page.

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(12) The framework bend is defined as the dihedral angle between the two least-squares planes through the two sets of equatorial ligands. For example, atoms W(1), C(1), C(2), C(3), and C(4) in **2** constitute one of the planes, and atoms W(2), C(6), C(7), C(9), and C(11) constitute the other.

Nucleophilic Attack of Pentacarbonylrhenate(–I) on Sulfur: Formation of the Doubly Bridged Tetrasulfido Dimer $[(\text{OC})_3\text{Re}(\eta^2\text{-S}_4)]_2^{2-}$

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Summary: The title complex is formed by addition of pentacarbonylrhenate(–I) to elemental sulfur and has been characterized by X-ray diffraction.

The nucleophilicity of metalates is well documented in the literature.² As part of our continuing effort to examine

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