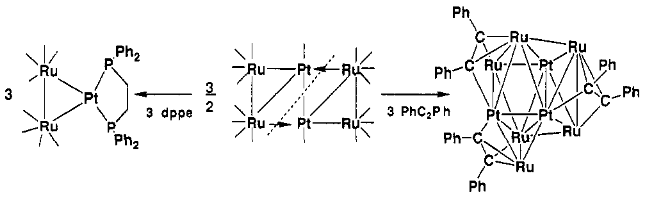
Scheme I



(Ru(1), Ru(5), Ru(6)) octahedron (Ru(2), Ru(3), Ru(4), Pt(1), Pt(2), Pt(3)) with a bond between the capping atoms Ru(1) and Ru(5). With 22 metal-metal bonds, 18-electron configurations can be assigned to each metal atom, although some of the metal-metal bonds (e.g. Ru(3)-Ru(1)) must be viewed formally as donor/acceptor bonds. Two of the metal-metal bonds are unusually long, Ru(1)-Ru(5) = 3.125 (2) Å and Pt(1)-Pt(3) = 3.124 (1) Å, but bonds of similar lengths have been observed in other platinum[11] and ruthenium[2d] cluster complexes. Compound **3** contains three triply bridging PhC_2Ph ligands that occupy triangular $PtRu_2$ groupings and 14 carbonyl ligands. The molecule could be viewed as an assembly derived from three $PtRu_2(\mu_3\text{-}PhC_2Ph)$ groupings, although details of the mechanism of formation are not available at this time. Both the dppe and PhC_2Ph reactions with **1** suggest that a splitting of **1** to $PtRu_2$ groupings is occurring. With dppe, the $PtRu_2$ grouping is stabilized, and the complex **2** is isolable. With PhC_2Ph three $PtRu_2$ groups condense to **3** (see Scheme I). In the presence of carbonyl ligands only, the dimer **1** is the most stable form of the complex.

It has been proposed that the series of oligomeric platinum cluster complexes $[Pt_3(CO)_6]_n^{2-}$ (n = 2-5)[11] interconvert by the transfer of unsaturated "$Pt_3(CO)_6$" groupings.[12] As illustrated by the reactions of **1**, this behavior may extend to other platinum-containing cluster complexes and may be influenced and possibly controlled by the ligands.

Acknowledgment. These studies were supported by the National Science Foundation under Grant CHE8919786.

Supplementary Material Available: Tables of crystal data, positional and anisotropic thermal parameters, and bond distances and angles for the structural analysis of compounds **1** and **3** (25 pages); tables of structure factor amplitudes (47 pages). Ordering information is given on any current masthead page.

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Synthesis and Characterization of Monooxo Alkyl Complexes of Ruthenium(VI) and Osmium(VI)

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Summary: Monooxo alkyl complexes of osmium(VI) and ruthenium(VI) with the molecular formula $M(O)(CH_2SiMe_3)_4$ have been synthesized from ((trimethylsilyl)methyl)magnesium compounds and $[PPh_4]_2[Os(O)_2Cl_4]$, $[PPN]_2[Ru(O)_2Cl_4]$, or $[PPh_4]_2[Os(O)_2(OSiMe_3)_4]$. The complexes were characterized by IR, NMR, and UV-visible spectroscopy and electrochemistry. The molecular structure of $Os(O)(CH_2SiMe_3)_4$ was determined by single-crystal X-ray diffraction.

Transition-metal oxo complexes are important for their properties as materials, as catalysts and reagents for oxidation reactions, and as models for certain metalloenzymes (cytochrome P450, isopenicillin N synthase). Because of this, activity on the synthesis, structure, and reaction chemistry of oxo complexes has increased dramatically in recent years.[1]

In order to directly compare the effects of multiply bonded ligands on a metal, it is necessary to prepare isoelectronic and isostructural complexes containing oxo, nitrido, and alkylimido groups. We have previously prepared alkyl complexes of ruthenium(VI) and osmium(VI) that contain terminal nitrido or alkylimido ligands.[2] These complexes have the formula $[M(N)R_4]^-$ and $M(NR')R_4$. The osmium complexes $[NBu^n{}_4][Os(N)(CH_2SiMe_3)_4]$ and $Os(NMe)(CH_2SiMe_3)_4$ were structurally characterized.[3] An isoelectronic oxo complex, $Os(O)(CH_2SiMe_3)_4$, was reported by Wilkinson and co-workers.[4] The oxo compound was prepared in 12% yield by the direct reaction of bis((trimethylsilyl)methyl)magnesium with osmium tetroxide. The ruthenium analogue cannot be prepared by this route, however. As part of our study of the reactivity of high-oxidation-state organometallic complexes, we sought to develop a more general synthetic route to the osmium and ruthenium oxo alkyl complexes $M(O)R_4$. Here we report the synthesis and characterization of $Os(O)(CH_2SiMe_3)_4$ and $Ru(O)(CH_2SiMe_3)_4$ and the structure of the osmium complex by single-crystal X-ray diffraction.

A number of inorganic oxo complexes of ruthenium and osmium in the +6 oxidation state have been prepared, but most of these are inappropriate precursors to oxoalkylmetal complexes since they are insoluble in nonprotic solvents and are easily reduced.[5] The tetraphenylphosphonium and bis(triphenylphosphine)nitrogen(1+) salts of the dioxometal tetrachlorides, $[PPh_4]_2[Os(O)_2Cl_4]$[6] and $[PPN]_2[Ru(O)_2Cl_4]$,[7] are moderately soluble in dichloromethane. More soluble complexes can be prepared by metathesis of the chloride ligands for trimethylsiloxide. Treatment of $[PPh_4]_2[Os(O)_2Cl_4]$ with 4 equiv of sodium trimethylsiloxide in dichloromethane produced $[PPh_4]_2[Os(O)_2(OSiMe_3)_4]$ in good yield. Analytically pure ma-

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(1) For a review of recent research on metal oxo complexes see: Nugent, W. A.; Mayer, J. M. *Metal-Ligand Multiple Bonds*; Wiley: New York, 1988.

(2) (a) Belmonte, P. A.; Own, Z.-Y. *J. Am. Chem. Soc.* **1984**, *106*, 7493-6. (b) Shapley, P. A.; Wepsiec, J. P.; *Organometallics* **1986**, *5*, 1515-7. (c) Shapley, P. A.; Kim, H. S.; Wilson, S. R. *Organometallics* **1988**, *7*, 928-33.

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(6) A solution of $[OsO_2Cl_4]^{2-}$ was prepared by dissolving potassium osmate, $K_2[Os(O)_2(OH)_4]$ (0.229 g, 0.622 mmol), in 50 mL of 2 M HCl. Addition of PPh_4Cl (0.7 g in 45 mL of 2 M HCl) caused immediate precipitation of the buff-colored product $[PPh_4]_2[OsO_2Cl_4]$ in 87% yield. ^1H NMR (CD_2Cl_2, 300 MHz, 19 °C): δ 7.69 (m, 2 H, *o*-H), 7.78 (m, 2 H, *m*-H), 7.89 (m, 1 H, *p*-H). IR (KBr, cm^{-1}): 840 s (OsO_2). Anal. Calcd for $C_{48}H_{40}Cl_4OsO_2P_2$: C, 55.28; H, 3.86. Found: C, 55.09; H, 3.83.

(7) Kochi, J. K.; Perrier, S. *Inorg. Chem.* **1988**, *27*, 4165-73.

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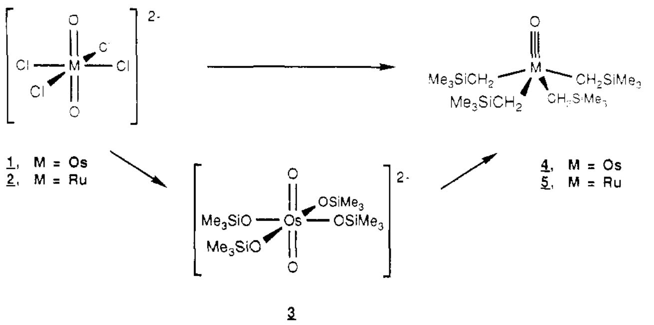
Scheme I



terial was obtained as green crystals by crystallization of the crude product from toluene/hexane solution.[8]

The alkylation of $[PPh_4]_2[Os(O)_2Cl_4]$ or $[PPh_4]_2[Os(O)_2(OSiMe_3)_4]$ with bis((trimethylsilyl)methyl)magnesium or ((trimethylsilyl)methyl)magnesium chloride produced the monooxo complex $Os(O)(CH_2SiMe_3)_4$ (4). In a typical reaction, a solution of $(Me_3SiCH_2)MgCl$ in diethyl ether (0.20 mL, 1.0 M) was added dropwise to a dichloromethane solution of $(PPh_4)_2[OsO_2Cl_4]$ (0.052 g, 0.050 mmol, 30 mL of CH_2Cl_2). The color changed from blue to orange over the course of the reaction. The solution was concentrated and filtered, and the product was purified by column chromatography on silica gel with CH_2Cl_2 followed by crystallization from hexane at −30 °C. Orange crystals (0.015 g) were obtained in 54% yield.[9] Orange crystals of the product could also be obtained by sublimation (2×10^{-5} Torr, 75 °C). Compound 4 decomposes only at temperatures greater than 160 °C in solution. It is stable to air and water and does not react with electrophiles.

A similar reaction between ((trimethylsilyl)methyl)magnesium chloride and $[PPN]_2[Ru(O)_2Cl_4]$ produced $Ru(O)(CH_2SiMe_3)$ (5) in approximately 60% yield.[10] The oxoruthenium alkyl complex is quite volatile, distilling at 20 °C and approximately 1 Torr. It is a thermally stable, orange oil, soluble in nonpolar organic solvents. Unlike the osmium analogue $Ru(O)(CH_2SiMe_3)_4$ is sensitive to air and water and must be handled under an inert atmosphere.

The molecular structure of $Os(O)(CH_2SiMe_3)_4$ was determined by X-ray diffraction.[11] A suitable crystal was grown over a period of several weeks by allowing a hexane solution of 4 to slowly evaporate at −30 °C. Data were collected under a cold nitrogen stream. No crystallographic symmetry was imposed on the molecule. An ORTEP drawing of 4 is shown in Figure 1. The osmium–oxo distance of 1.692 (6) Å in $Os(O)(CH_2SiMe_3)_4$ is comparable with that of other monooxo alkyl complexes of the transition metals that are considered to have metal–oxygen triple bonds.[12] It is close to the Os–N distance in $Os(NMe)(CH_2SiMe_3)_4$, 1.686 (5) Å, but longer than the Os–N distance in $[NBu^n_4][Os(N)(CH_2SiMe_3)_4]$, 1.631 (8) Å.

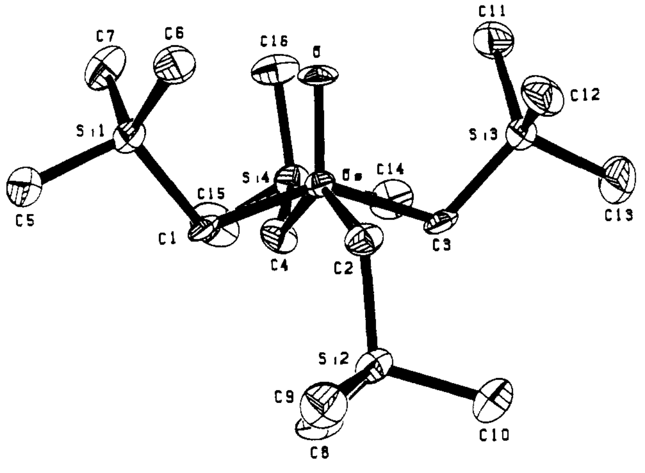


Figure 1. ORTEP diagram of $Os(O)(CH_2SiMe_3)_4$ with hydrogen atoms omitted for clarity. Selected bond distances (Å) and angles (deg) are as follows: Os–O, 1.692 (6); Os–C_1, 2.076 (9); Os–C_2, 2.079 (10); Os–C_3, 2.099 (9); Os–C_4, 2.057 (9); O–Os–C_1, 108.0 (3); O–Os–C_2, 114.0 (3); O–Os–C_3, 107.7 (3); O–Os–C_4, 113.5 (4); C_1–Os–C_3, 144.1 (1); C_2–Os–C_4, 132.5 (4).

The geometry about the osmium is distorted square pyramidal, with the four α-carbon atoms forming the base of the pyramid and the osmium atom slightly above the plane of the base. Unlike $[NBu^n_4][Os(N)(CH_2SiMe_3)_4]$, which has nearly equivalent N–Os–C_α angles (108.8–107.0°), there are two sets of O–Os–C_α angles in $Os(O)(CH_2SiMe_3)_4$. This angle averages 113.8° for one set of trans alkyl ligands and 107.9° for the other two. This results in a slight distortion toward a trigonal-bipyrimidal geometry in this molecule. The reason for this must be electronic in nature, rather than steric. The nitrido ligand in $[NBu^n_4][Os(N)(CH_2SiMe_3)_4]$ is bonded to the osmium through a full triple bond. The empty osmium d_{xz} and d_{yz} orbitals are equivalent in the square-pyramidal metal complex and can be used to form the two π bonds with nitrogen.[13] The distortion in $Os(O)(CH_2SiMe_3)_4$ removes the equivalence of the d_{xz} and d_{yz} orbitals.

The complexes $M(O)(CH_2SiMe_3)_4$ are fluxional, and only one type of alkyl resonance is observed in the room-temperature NMR spectra of each complex. At low temperature, the resonance for the trimethylsilyl group in the ^1H NMR spectra splits into two lines and significant broadening of the methylene peak is observed. The Gibb's free energy of activation for equilibration of the alkyl groups in 4 was found to be 9.6 ± 0.2 kcal/mol.[14] There are two types of alkyl groups at low temperature, but this infor-

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(8) $[PPh_4]_2[Os(O)_2(OSiMe_3)_4]\cdot C_6H_5CH_3$: ^1H NMR ($CDCl_3$, 300 MHz, 19 °C) δ 0.112 (s, 36 H, $SiMe_3$), 2.335 (s, 3 H, $C_6H_5CH_3$), 7.100 (m, 5 H, $C_6H_5CH_3$), 7.4–7.8 (m, 40 H, PPh); IR (KBr, cm^{-1}) 838 s (OsO_2). Anal. Calcd for $C_{67}H_{84}O_6OsP_2Si_4$: C, 59.62; H, 6.27. Found: C, 59.91; H, 6.38.

(9) $Os(O)(CH_2SiMe_3)_4$: ^1H NMR (300 MHz, 18 °C, $CDCl_3$) δ 0.078 (s, 9 H, $CH_2Si(CH_3)_3$), 3.739 (s, 2 H, $CH_2Si(CH_3)_3$); ^1H NMR (300 MHz, 18 °C, C_6D_6) δ 0.192 (s, 9 H, $CH_2Si(CH_3)_3$), 3.870 (s, 2 H, $CH_2Si(CH_3)_3$); $^{13}C\{^1H\}$ NMR (C_6D_6, 18 °C, 75.4 MHz) δ 38.64 ($CH_2Si(CH_3)_3$), 1.673 ($CH_2Si(CH_3)_3$); IR (KBr, cm^{-1}) 1054 m (OsO) 2953 m, 1248 s, 1019 m, 853 vs, 840 vs, 757 m, 695 m, 672 m (CH_2SiMe_3); MS (EI, 70 eV) *m/z* 556 (M^+), 541 ($M^+ - CH_3$), 469 ($M^+ - CH_2SiMe_3$). Anal. Calcd for $C_{12}H_{44}OOsSi_4$: C, 34.62; H, 7.98. Found: C, 34.84; H, 7.76.

(10) $Ru(O)(CH_2SiMe_3)_4$: ^1H NMR (360 MHz, 19 °C, $CDCl_3$) δ 3.1 (s, 8 H, CH_2), 0.10 (s, 36 H, CH_3); ^{13}C NMR (125.7 MHz, 19 °C, $CDCl_3$) δ 45 (CH_2), 0.8 (CH_3); IR (KBr, cm^{-1}) 1045 w (Ru–O), 2944 s, 1244 s, 1016 m, 839 vs, 756 m, 694 m, 683 m (CH_2SiMe_3); MS (EI, 70 eV) *m/z* 466 (M^+). Anal. Calcd for $RuOSi_4C_{16}H_{44}$: C, 41.24; H, 9.52. Found: C, 40.90; H, 9.24.

(11) X-ray data for $Os(O)(CH_2SiMe_3)_4$: A transparent, orange, tabular crystal grown from hexane solution was mounted at −20 °C on a glass fiber. Data were collected at −50 °C on a Enraf-Nonius CAD4 automated k-axis diffractometer with graphite-monochromated Mo Kα radiation: $\lambda = 0.71073$ Å; triclinic, space group $P\bar{1}$ (C_i^1), No. 2; $a = 10.460$ (4) Å, $b = 13.331$ (4) Å, $c = 10.320$ (5) Å; $\alpha = 96.10$ (3)°, $\beta = 107.86$ (4)°, $\gamma = 104.37$ (3)°; $V = 1301$ (2) Å^3; $Z = 2$; calculated density $\rho = 1.417$ g/cm^3. A total of 3999 reflections were collected, of which 3336 were used to determine the structure (intensity $>2.58\sigma$). The structure was solved by Patterson methods (SHELX-86); correction positions for all non-hydrogen atoms were deduced from a Patterson map. Subsequent least-squares–difference Fourier calculations revealed positions for all hydrogen atoms; however, owing to the high residual electron density in the vicinity of the osmium atom, hydrogen atoms were included as fixed contributors in "idealized" positions. Absorption numerical correction was applied, and maximum and minimum transmission factors were 0.483 and 0.072, respectively. Anisotropic refinement of all non-hydrogen atoms by full-matrix least squares resulted in $R = 0.055$ and $R_w = 0.067$.

(12) Reference 1, Table 5.2.

(13) Mayer, J. M.; Thorn, D. L.; Tulip, T. H. *J. Am. Chem. Soc.* **1985**, *107*, 7454–62 and references therein.

(14) ^1H NMR spectra were obtained between +19.6 and −90.0 °C on a 300-MHz GE Instruments FT NMR spectrometer. Rates and activation parameters were calculated from line widths determined from a line-fit program available in the CHARM software program.

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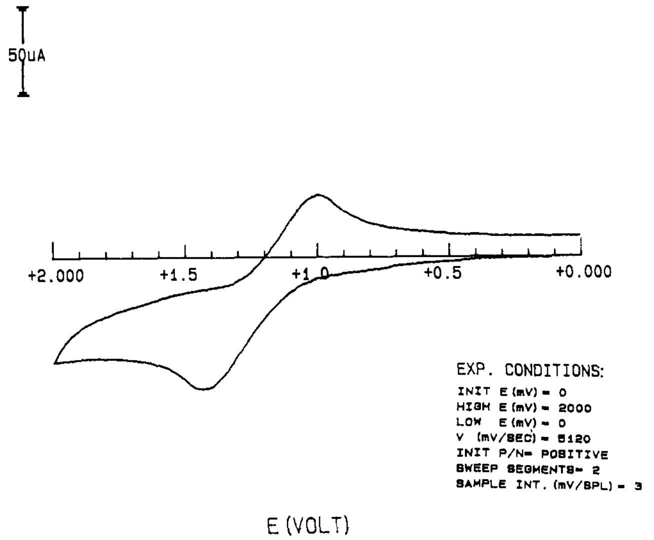

Figure 2. Cyclic voltammogram of $Ru(O)(CH_2SiMe_3)_4$ in CH_2Cl_2, 0.1 M $[NBu_4][BF_4]$.

mation does not allow us to distinguish between isomers due to the distortion from square-pyramidal geometry observed in the crystal structure of 4 or to rotational isomers about the $Os-CH_2SiMe_3$ bond.

The electronic natures of 4 and 5 were probed with UV–visible spectroscopy and electrochemical techniques. Both complexes had intense charge-transfer transitions for the metal–oxo moiety and relatively weak d to d transitions.[15] Each of these complexes exhibited a one-electron-oxidation wave by cyclic voltammetry, Osteryoung square-wave voltammetry, and coulometry.[16] In $Os(O)(CH_2SiMe_3)_4$, the oxidation wave is found at 1.19 V vs Ag/AgCl in methylene chloride. The oxidation is reversible with equal anodic and cathodic currents and a peak to peak separation of 72 mV. The oxidation wave of the ruthenium complex at 1.20 V is quasi-reversible with approximately equal anodic and cathodic currents only at fast scan rates (2000–5000 mV/s) and a peak to peak separation of 400 mV. Neither complex exhibited reversible reduction waves.

Now that the ruthenium and osmium complexes $M(O)(CH_2SiMe_3)_4$ have been prepared and characterized, we have series of complexes for these metals that differ only in the nature of the multiply bonded heteroatom ligand. Studies on the comparative reaction chemistry of these complexes is now underway.

Acknowledgment. We gratefully acknowledge the financial support of the National Science Foundation (Grant No. CHE 88-07707) in support of this work. Spectra were obtained on NMR instruments purchased through grants from the National Institutes of Health and the National Science Foundation (Grant Nos. NIH PHS 1532135, NIH 1531957, and NSF CHE 85-14500).

Supplementary Material Available: Tables of crystal data, positional and thermal parameters, and bond distances and angles (8 pages); a table of final observed and calculated structure factor amplitudes (14 pages). Ordering information is given on any current masthead page.

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[15] Ultraviolet and visible spectra were recorded on a Perkin-Elmer Lambda 3 UV–vis spectrometer. UV–vis for $Os(O)(CH_2SiMe_3)_4$ (CH_2Cl_2, 5.6×10^{-5} M, nm): 233 (ϵ = 19000), 386 (ϵ = 23), 476 (ϵ = 16), 500 (ϵ = 18). UV–vis for $Ru(O)(CH_2SiMe_3)_4$ (CH_2Cl_2, 5.9×10^{-4} M, nm): 236 (ϵ = 4900), 514 (ϵ = 390).

[16] Electrochemical experiments were performed on a BAS 100 electrochemical analyzer, with Pt working and auxiliary electrodes and an Ag/AgCl reference electrode. Methylene chloride solutions were 0.1 M in $[NBu_4][BF_4]$ and 10^{-3}–10^{-4} M in sample.

Unsaturated Mixed-Metal Complexes: Syntheses of (η-C_5Me_5)Ni–M$(CO)_3$(η-C_5H_5) (M = Mo, W) and X-ray Diffraction Study of (η-C_5Me_5)Ni–W$(CO)_3$(η-C_5H_4Me)

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Summary: Mild thermolysis of (η-C_5Me_5)(CO)Ni–M$(CO)_3$(η-C_5H_5) (M = Mo, W) afforded the paramagnetic coordinatively unsaturated species (η-C_5Me_5)Ni–M$(CO)_3$(η-C_5H_5). Irreversible decarbonylation to the tetrahedral clusters $Ni_2M_2(CO)_4(\eta\text{-}C_5H_5)_2(\eta\text{-}C_5Me_5)_2$ occurred on prolonged heating. The compound (η-C_5Me_5)Ni–W$(CO)_3$(η-C_5H_4Me) was characterized by an X-ray diffraction study: short nickel–tungsten distances of 2.475 (2) and 2.457 (2) Å for the two independent molecules in the unit cell indicate multiple Ni–W bond character.

Coordinatively unsaturated metal species display a rich chemistry, and their study is of great interest. Dinuclear complexes of this type often contain multiple metal–metal bonds and may be classified into two groups. Molecules in which the metals have formal oxidation states ≥II are found in one set.[1] The other group contains metal in lower formal oxidation states; π-acid ligands such as η-C_5H_5 and CO are generally present. Few molecules exist in this class, and except for the compounds $[M(CO)_2(\eta\text{-}C_5R_5)]_2$ (M≡M, M = Cr, Mo, W; R = H, Me)[2] and $[M(\mu_2\text{-}CO)(\eta\text{-}C_5Me_5)]_2$ (M=M, M = Co, Rh, Ir),[3] their chemistry has not been probed in depth.[4] Mixed-metal multiply bonded compounds are rare in both sets: examples include a few group 6[5] species and the group 9 complexes $CoM(\mu_2\text{-}CO)_2(\eta\text{-}C_5Me_5)_2$ (Co=M, M = Rh, Ir).[3,6] In most cases the metals

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