both yields and product ratios is interesting[25] and is under further investigation.

That a η^3-cyclopropenyl ligand can serve as a direct precursor to a η^3-oxocyclobutenyl ligand is evidenced by the phosphine-promoted conversion of **2a** to **2b** and **3b** (or **2c** and **3c**) and by transformation of **4** to **5b**. These reactions are presumably initiated by pathway c (Scheme I) followed by competition between path b or a sequence of paths e and i; in the case of the iron compounds, both pathways are competitive, for molybdenum the ring expansion route dominates, whereas for cobalt no ring expansion occurs. A similar thermal conversion of a η^3-cyclopropenyl to a η^3-oxocyclobutenyl ligand on molybdenum is evidenced by formation of only **4** under mild conditions, but a mixture of **4** and **5a** under more vigorous thermal conditions.[24] The reasons for the different reactivities of the anions $[Mo(CO)_3(\eta\text{-}C_5R_5)]^-$ (R = H, Me) toward $[C_3Ph_3]^+$ are unclear; notably $[Mo(CO)_3(\eta\text{-}C_5H_5)]^-$ has been shown previously to react with $[C_3\text{-}t\text{-}Bu_3]^+$ via attack at the cyclopentadienyl ring.[27]

Once again, it cannot be distinguished whether the manganese compound **6** arises via direct attack at Mn (path a, e, i) or by attack at CO (path f, i). Whichever path is followed, formation of **6** demonstrates a notable difference between Mn and Re chemistry; the (2-cyclopropene-1-carbonyl)rhenium system undergoes exclusive cyclopropenyl migration to Re (path d), with no observable ring expansion (path i), and (3-η^1-cyclopropenyl)rhenium complexes show no tendency to form oxocyclobutenyl compounds (path e, i).[11,12]

Reactions of metal carbonyl anions with cyclopropenium cations clearly proceed via an array of reaction pathways, but the underlying reasons for choice of reaction path are still obscure. Nevertheless, under suitable conditions these reactions can provide good synthetic routes to oxocyclobutenyl compounds, which are useful precursors to cationic cyclobutadiene compounds[28,29] and to new η^1- and η^3-cyclopropenyl compounds, whose chemistry is currently under continuing investigation.

Acknowledgment. We are grateful to the National Science Foundation (Grants CHE 7717877 and CHE 8022854) and the Alfred P. Sloan Foundation for support of this research. Helpful discussions with Dr. Neil G. Connelly (Bristol University) are also acknowledged with gratitude.

Registry No. 2a, 82661-39-0; **2b**, 82661-40-3; **2c**, 82661-41-4; **3a**, 82661-42-5; **3b**, 82661-43-6; **3c**, 82661-44-7; **4**, 82661-45-8; **5a**, 82661-46-9; **5b**, 82661-47-0; **5c**, 82665-10-9; **6**, 82661-48-1; $Na[Fe(CO)_3NO]$, 25875-18-7; $[C_3Ph_3]Br$, 4919-51-1; $Mo(CO)_3(MeCN)_3$, 15038-48-9; $[C_3Ph_3]Cl$, 82661-38-9; $[Mo(CO)_3(\eta\text{-}C_5H_5)]Na$, 12107-35-6; $[Mo(CO)_3(\eta\text{-}C_5H_5)]PPN$, 67486-18-4; $[C_3Ph_3]PF_6$, 82661-49-2; $[Mo(CO)_3(\eta\text{-}C_5H_5)]_2$, 12091-64-4; $[C_3Ph_3]_2$, 4997-62-0; $Na[Mo(CO)_3(\eta\text{-}C_5Me_5)]$, 82661-50-5; $Na[Mn(CO)_5]$, 13859-41-1; $[C_3Ph_3]BF_4$, 741-16-2; $PPN[Mn(CO)_5]$, 52542-59-3.

Supplementary Material Available: Table of microanalysis data for **2a–c**, **3a–c**, **5a–c**, and **6** (1 page). Ordering information is given on any current masthead page.

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[25] The nature of the cation is known to affect the IR spectroscopic properties of metal carbonyl anions and can also affect the rates of some of their reactions. The rate of the CO migratory insertion reaction can also be influenced by the nature of cations present in solution.[26]

[26] See: Darensbourg, M. Y.; Jimenez, P.; Sackett, J. R.; Hanckel, J. M.; Kump, R. L. *J. Am. Chem. Soc.* **1982,** *104,* 1521–1530 and references cited therein.

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New Syntheses of Mixed-Metal Clusters from $H_2Os_3(CO)_{10}$. Crystal and Molecular Structure of the Paramagnetic Cluster $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ and Its Diamagnetic Structural Analogue $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$

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Summary: Reactions of $H_2Os_3(CO)_{10}$ with metal carbonyls in the presence of H_2 and also in the absence of H_2 provide convenient routes to a number of new mixed-metal clusters and improved yields of some previously reported clusters. The X-ray structure determination of the paramagnetic cluster $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ and its diamagnetic structural analogue $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ is reported.

An earlier report[1] from this laboratory suggested a relatively simple approach to the preparation of mixed-metal systems containing a triosmium unit and is based upon the use of the electronically unsaturated cluster $H_2Os_3(CO)_{10}$ as a starting material. A simple high-yield preparation of $H_2FeOs_3(CO)_{13}$ and a good yield preparation of $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$ were reported. More recently we have found that when the latter reaction is conducted in the presence of molecular hydrogen, the yield of $H_2(\eta^5\text{–}C_5H_5)CoOs_3(CO)_{10}$ is greatly diminished but two additional products are formed.

$$(\eta^5\text{-}C_5H_5)Co(CO)_2 + H_2Os_3(CO)_{10} + H_2 \xrightarrow[90°\ C,\ 53\ h]{toluene} \underset{4\%}{H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}} + \underset{33\%}{H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9} + \underset{32\%}{H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9} \quad (1)$$

The conditions chosen for reaction 1 permit formation of significant yields of $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$. However, this compound will react with H_2 to convert to $H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$. Thus after a prolonged period of time the principal product from reaction 1 is $H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$. On the other hand, the conversion of $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$ to the other clusters in the presence of H_2 at 1 atm is not favorable.[2]

$$\underset{30\%\ \text{consumed}}{H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}} + H_2 \xrightarrow[90\ °C,\ 3\ days]{toluene} \underset{7\%}{H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9} + \underset{23\%}{H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9} \quad (2)$$

It is possible that in reaction 1 H_2 is assisting in CO displacement from the reactants and that the primary path of the reaction does not proceed through $H_2(\eta^5\text{-}C_5H_5)$-

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[1] Plotkin, J. S.; Alway, D. G.; Weisenberger, C. R.; Shore, S. G. *J. Am. Chem. Soc.* **1980,** *102,* 6157.

[2] This result contrasts with the earlier observation that most of $H_2FeRu_3(CO)_{13}$ (82%) is converted to $H_4FeRu_3(CO)_{12}$ in the presence of H_2. See: Koepke, J. W.; Johnson, J. R.; Knox, S. A. R.; Kaesz, H. D. *Ibid.* **1975,** *97,* 3947.

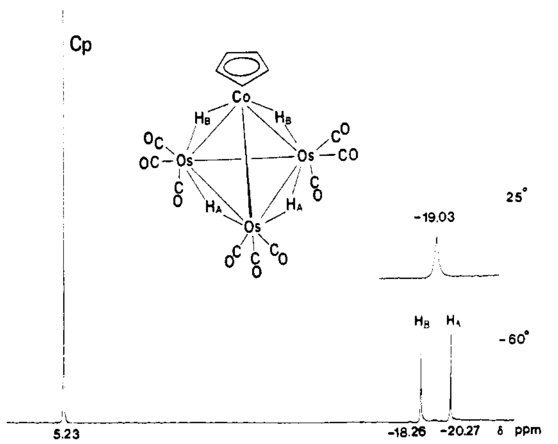

Figure 1. 300-MHz proton NMR spectra of $H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ in $CDCl_3$ at -60 and 25 °C.

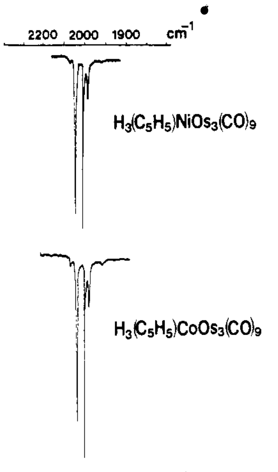

Figure 2. Infrared spectra of $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ and $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ in hexane.

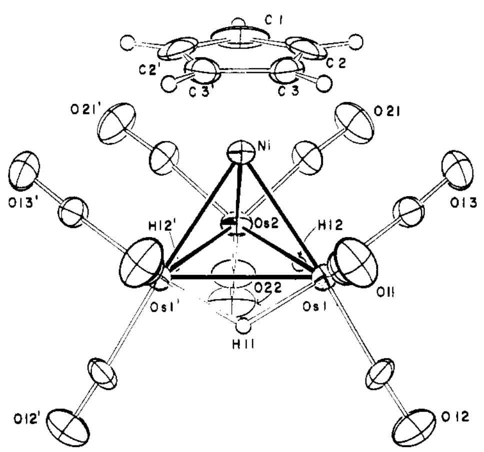

Figure 3. Geometry of an $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ molecule. For each of the crystallographically distinct molecules (molecule A and molecule B) a crystallographic mirror plane passes through atoms C(1), Ni, Os(2), O(22), and H(11). Note that if we neglect direct osmium–osmium bonds, each osmium atom is in an approximately octahedral coordination environment. The $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ molecule has essentially the same geometry with minor variations in the disposition of ligands.

$CoOs_3(CO)_{10}$ as an intermediate. Of the new clusters formed in reaction 1 $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ (mass spectrum, *m/e*(calcd) for $^1H_8{}^{12}C_{14}{}^{16}O_9{}^{59}Co^{192}Os_3$, 954.8336, *m/e*(obsd) 954.8367) is paramagnetic. It contains one unpaired electron, as determined by the Evans method.[3] The ESR signal is too broad (298–4 K) to calculate a *g* value. The structure and infrared spectrum of this compound are discussed below in relation to its structural analogue, the electron-precise cluster $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$.

The cluster $H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ (mass spectrum, *m/e*(calcd) for $^1H_9{}^{12}C_{14}{}^{16}O_9{}^{59}Co^{192}Os_3$, 955.8415, *m/e*(obsd) 955.8387) gives the ^1H NMR spectra shown in Figure 1. At -60 °C two sets of bridging protons of equal intensities are observed. This is consistent with two Os–H–Os bridges and two Os–H–Co bridges in a structure with C_s symmetry. The ^{13}C NMR spectrum at -70 °C under the condition of limiting exchange is consistent with mirror symmetry.

In a reaction analogous to (1) the cluster $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ (mass spectrum, *m/e*(calcd) for $^1H_8{}^{12}C_{14}{}^{16}O_9{}^{59}Ni^{192}Os_3$, 953.8357, (*m/e*)(obsd) 953.8381) was prepared.

$$[(\eta^5\text{-}C_5H_5)Ni(CO)]_2 + H_2Os_3(CO)_{10} + H_2 \xrightarrow[90\ °C,\ 10\ h]{toluene} \underset{93\%}{H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9} \quad (3)$$

This is the only product observed. However, when $[(\eta^5\text{-}C_5H_5)Ni(CO)]_2$ and $H_2Os_3(CO)_{10}$ react in the absence of H_2, the yield of $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ is halved and a brown unstable material is formed. This material may be $H(\eta^5\text{-}C_5H_5)NiOs_3(CO)_{10}$ since it reacts readily with H_2 to give $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$.

Virtually identical infrared spectra of $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ and $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ are observed (Figure 2). Indeed, $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ and $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ have been shown to be isomorphous and their structures each determined via single-crystal X-ray structural analyses[4,5] (Figure 3). The ^1H and ^{13}C NMR data for the $NiOs_3$ complex are consistent with the X-ray determined structure.

The crystal structures of $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ and $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ each contain two unrelated sites for the molecules, each of which has crystallographically imposed C_s symmetry. The crystallographic "asymmetric unit" thus consists of two distinct half molecules of $H_3(\eta^5\text{-}C_5H_5)MOs_3(CO)_9$ (M = Co, Ni); the molecules are, however, equivalent. Each consists of a $(\eta^5\text{-}C_5H_5)M$ unit bonded symmetrically to a basal $(\mu\text{-}H)_3Os_3(CO)_9$ moiety of C_{3v} symmetry (see Figure 3). The heteroatom is bonded to an η^5-cyclopentadienyl ligand and to three osmium atoms, while each osmium atom is linked to the heteroatom, two other osmium atoms, two bridging hydride ligands, and three terminal carbonyl ligands. In the structural study of the $NiOs_3$ derivative, all bridging hydride ligands were located directly; unfortunately, in the study of the $CoOs_3$ derivative, hydride ligands associated with one of the two molecules were located but those of the

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[3] Loliger, J.; Scheffold, R. *J. Chem. Educ.* **1972**, *49*, 646.

[4] Crystal data for $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$: the complex crystallizes in the centrosymmetric orthorhombic space group *Pcam* (No. 57) with $a = 14.554$ (3) Å, $b = 17.845$ (3) Å, $c = 15.008$ (3) Å, $V = 3898$ Å^3, and ρ(calcd) = 3.24 g cm^{-3} for mol wt 949.52 and $Z = 8$. Single-crystal X-ray diffraction data were collected with a Syntex P2$_1$ automated four-circle diffractometer, and the structure was solved by direct methods (MULTAN), difference Fourier syntheses and full-matrix least-squares refinement. The resulting discrepancy indices were $R_F = 5.3\%$ and $R_{wF} = 4.9\%$ for all 2683 independent reflections with $3.5° < 2\theta < 45.0°$ (Mo Kα radiation) and $R_F = 3.4\%$ and $R_{wF} = 4.2\%$ for those 2129 data with $|F_o| > 3\sigma(|F_o|)$.

[5] The quality of crystals obtained for $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ was not as good as for the $NiOs_3$ complex. Data are as follows: space group *Pcam*, $a = 14.632$ (3) Å, $b = 17.872$ (5) Å, $c = 14.991$ (3) Å, $V = 3920$ Å^3, and ρ(calcd) = 3.23 g cm^{-3}. Final discrepancy indices were $R_F = 5.2\%$ and $R_{wF} = 5.0\%$ of all 1937 independent reflections with $4.0° < 2\theta < 40.0°$ (Mo Kα) and $R_F = 4.2\%$ and $R_{wF} = 4.8\%$ for those 1628 data with $|F_o| > 3\sigma(|F_o|)$.

Table I. Reactions of $H_2Os_3(CO)_{10}$ with Metal Carbonyls

metal carbonyl	product(s) (yield, %)	
	without hydrogen	with hydrogen
$(\eta^5\text{-}C_5H_5)Co(CO)_2$	$H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$ (30)[1]	$H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$ (4) $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ (33)[a] $H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$ (32)[a]
$[(\eta^5\text{-}C_5H_5)Ni(CO)]_2$	$H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ (48)[a]	$H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$ (93)[a]
$Co_2(CO)_8$	$HCoOs_3(CO)_{13}$ (48)[b] $H_3CoOs_3(CO)_{12}$ (5)[c]	$H_3CoOs_3(CO)_{12}$ (60)[c]
$(\eta^5\text{-}C_5H_5)Rh(CO)_2$	$H_2(\eta^5\text{-}C_5H_5)RhOs_3(CO)_{10}$ (42)[a]	
$(\eta^5\text{-}C_5H_5)Ir(CO)_2$	$H_2(\eta^5\text{-}C_5H_5)IrOs_3(CO)_{10}$ (21)[a]	

[a] Unreported previously. [b] Obtained previously in trace amount.[7] [c] Obtained previously, no yield reported.[8]

Table II. Spectral Data of New Mixed-Metal Clusters

cluster	mass (*m/e*)	^1H NMR (in $CDCl_3$), δ	ν_{CO}, cm^{-1}
$H_2(\eta^5\text{-}C_5H_5)RhOs_3(CO)_{10}$	1026	5.95 (5 H), −17.57 (1 H), −20.78 (1 H)[a]	2083 m, 2063 vs, 2042 vs, 2010 vs,[b] 2000 s (sh), 1982 m, 1972 m, 1819 m
$H_2(\eta^5\text{-}C_5H_5)IrOs_3(CO)_{10}$	dec	5.80 (5 H), −19.02 (1 H), −21.40 (1 H)[a]	2082 w, 2068 s, 2040 s, 2018 vs, 2000 w (sh), 1980 vw, 1970 w, 1815 vw[b]
$H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$	956	5.22 (5 H), −19.30 (4 H)[c] 5.23 (5 H), −18.26 (2 H), −20.27 (2 H)[a]	2082 m, 2060 s, 2050 s, 2019 s, 1995 s (sh), 1992 s, 1977 m, 1952 vw[d]
$H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$	955		2082 w, 2060 s, 2008 vs, 1990 m, 1955 vw[d]
$H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$	954	6.05 (5 H), −17.65 (3 H)[c] 6.05 (5 H), −17.64 (3 H)[a]	2085 vw, 2065 s, 2010 vs, 1990 m, 1958 vw[d]

[a] At −60 °C. [b] In cyclohexane. [c] At room temperature. [d] In hexane.

other molecule were not. The structure is, nevertheless, secure.

Metal–metal distances for the $NiOs_3$ complex are as follows: (molecule A) Os(1)–Os(1′) = 2.859 (1) Å, Os(1)–Os(2) = Os(1′)–Os(2) = 2.874 (1) Å, Os(1)–Ni = Os(1′)–Ni = 2.569 (2) Å, Os(2)–Ni = 2.578 (3) Å; (molecule B) Os(1)–Os(1′) = 2.873 (1) Å, Os(1)–Os(2) = Os(1′)–Os(2) = 2.873 (1) Å, Os(1)–Ni = Os(1′)–Ni = 2.563 (2) Å, Os(2)–Ni = 2.562(2) Å. Other distances include Os–CO = 1.866 (15)–1.991 (24) Å and Ni–C(C_5H_5) = 2.089 (22)–2.136 (23) Å.

Metal–metal distances for the $CoOs_3$ complex are as follows: (molecule A) Os(1)–Os(1′) = 2.864 (1) Å, Os(1)–Os(2) = Os(1′)–Os(2) = 2.881 (1) Å, Os(1)–Co = Os(1′)–Co = 2.578 (3) Å, Os(2)–Co = 2.590 (4) Å; (molecule B) Os(1)–Os(1′) = 2.891 (1) Å, Os(1)–Os(2) = Os(1′)–Os(2) = 2.879 (1) Å, Os(1)–Co = Os(1′)–Co = 2.583 (3) Å, Os(2)–Co = 2.597 (4) Å. Other distances include Os–CO = 1.801 (23)–1.943 (20) Å and Co–C(C_5H_5) = 2.060 (38)–2.167 (19) Å.

The average hydrido-bridged osmium–osmium distances are 2.871 [6] Å[6] for the $NiOs_3$ complex and 2.879 [9] Å for the $CoOs_3$ complex; the average osmium–nickel bond length of 2.567 [6] Å appears to be slightly shorter than the average osmium–cobalt bond length of 2.585 [6] Å.

The types of reactions cited above have been extended to a number of other metal–triosmium cluster systems by simply reacting metal carbonyls with $H_2Os_3(CO)_{10}$ in the absence and in the presence of molecular hydrogen to provide new cluster compounds and to also provide improved yields of some previously reported compounds. Results from the systems which have been examined in the absence and presence of molecular hydrogen are given in Table I.

All of the compounds reported in Table I were prepared in toluene at 90 °C with the exception of the clusters produced from $Co_2(CO)_8$ which were obtained from CH_2Cl_2 at room temperature. The additional condition of an atmosphere of hydrogen was employed for the preparation of clusters of higher hydrogen content. All of the compounds were isolated as air-stable solids by TLC (silica gel; benzene/hexane solution). Spectral data from the new compounds prepared are listed in Table II.

The new compounds $H_2(\eta^5\text{-}C_5H_5)RhOs_3(CO)_{10}$ and $H_2(\eta^5\text{-}C_5H_5)IrOs_3(CO)_{10}$ are isoelectronic with $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$, a compound of known structure.[9] They are believed to be isostructural in that proton NMR and IR spectra of all three compounds are very similar in appearance.

Acknowledgment. We thank the National Science Foundation for support of this work through Grants CHE79-18149 (S.G.S.) and CHE80-23448 (M.R.C.). We thank Dr. Matthew S. Platz for obtaining ESR spectra. The 300-MHz FT-NMR spectra were obtained at The Ohio State University Chemical Instrument Center (funded in part by National Science Foundation Grant CHE-7910019).

Registry No. $H_2(\eta^5\text{-}C_5H_5)CoOs_3(CO)_{10}$, 74594-41-5; $H_3(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$, 82678-94-2; $H_4(\eta^5\text{-}C_5H_5)CoOs_3(CO)_9$, 82678-95-3; $H_3(\eta^5\text{-}C_5H_5)NiOs_3(CO)_9$, 82678-96-4; $H_3CoOs_3(CO)_{12}$, 68022-12-8; $HCoOs_3(CO)_{13}$, 72275-14-0; $H_2(\eta^5\text{-}C_5H_5)RhOs_3(CO)_{10}$, 82678-97-5; $H_2(\eta^5\text{-}C_5H_5)IrOs_3(CO)_{10}$, 82678-98-6; $(\eta^5\text{-}C_5H_5)Co(CO)_2$, 12078-25-0; $[(\eta^5\text{-}C_5H_5)Ni(CO)]_2$, 12170-92-2; $Co_2(CO)_8$, 10210-68-1; $(\eta^5\text{-}C_5H_5)Rh(CO)_2$, 12192-97-1; $(\eta^5\text{-}C_5H_5)Ir(CO)_2$, 12192-96-0; Os, 7440-04-2; Co, 7440-48-4; Ni, 7440-02-0; Rh, 7440-16-6; Ir, 7439-88-5.

Supplementary Material Available: Tables of positional parameters, anisotropic thermal parameters, and observed and calculated structure factor amplitudes (32 pages). Ordering information is given on any current masthead page.

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(6) Numbers in square brackets are estimated standard deviations of the mean values.

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(8) Bhaduri, S.; Johnson, B. F. G.; Lewis, J.; Raithby, P. R.; Watson, D. J. *J. Chem. Soc., Chem. Commun.* **1978**, 260.

(9) Churchill, M. R.; Bueno, C.; Kennedy, S.; Bricker, J. C.; Plotkin, J. S.; Shore, S. G. *Inorg. Chem.* **1982**, *21*, 627.