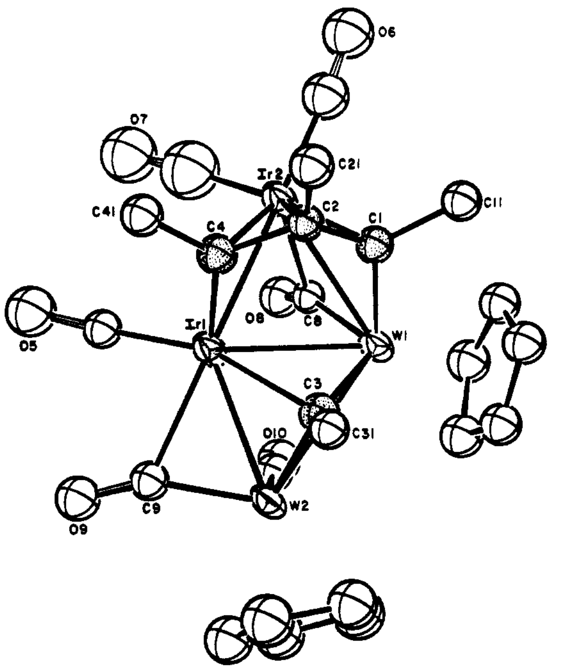

Figure 1. ORTEP-II diagram of the core of the $(\eta^5\text{-}C_5H_5)_2W_2Ir_2(CO)_6(\mu_3\text{-}CPh)(\mu_3\text{-}\eta^3\text{-}C_3Ph_3)$ molecule. Phenyl groups (whose ipso carbons are C(11), C(21), C(31), C(41)) are omitted for clarity. Distances of interest (in Å) are as follows: (a) metal–metal bond lengths, W(1)–W(2) = 3.080 (1), W(1)–Ir(1) = 2.723 (2), W(1)–Ir(2) = 2.852 (2), Ir(1)–Ir(2) = 2.720 (1), W(2)–Ir(1) = 2.665 (2); (b) metal–$(\mu_3\text{-}\eta^3\text{-}C_3Ph_3)$ ligand distances, W(1)–C(1) = 2.231 (24), Ir(2)–C(1) = 2.233 (25), Ir(2)–C(2) = 2.296 (25), Ir(2)–C(4) = 2.302 (26), Ir(1)–C(4) = 2.069 (24); (c) metal–$(\mu_3\text{-}CPh)$ ligand distances, W(1)–C(3) = 2.012 (29), W(2)–C(3) = 2.081 (27), Ir(1)–C(3) = 2.180 (28).

at room temperature.[3] A similar rearrangement process has been observed for related homometallic butterfly complexes.[4]

The NMR data obtained for "$Cp_2W_2Ir_2(CO)_6(C_2Ph_2)_2$" do not define a unique structure, so an X-ray diffraction study has been performed.[5] The results (see Figure 1) show that the molecule contains an almost flat W_2Ir_2 core, consisting of two triangles fused at the W(1)–Ir(1) edge (dihedral angle W(1)–Ir(1)–W(2)/W(1)–Ir(1)–Ir(2) = 170.33°). A $\mu_3\text{-}\eta^3$-1,3-dimetalla-allyl (C_3Ph_3) moiety is π bonded to Ir(2) and σ bonded to Ir(1) and W(1). On the same face of the tetrametallic core, a μ_3-CPh moiety bridges Ir(1), W(1), and W(2). Ir(1) is linked to one terminal carbonyl, whereas Ir(2) has two. Each tungsten atom is attached to an $\eta^5\text{-}C_5H_5$ ring and to a semibridging carbonyl having a signficant interaction with an adjacent iridium atom (W(2)–[C(9)–O(9)]···Ir(1), α = 0.24;[6] W(1)–[C(8)–O(8)]···Ir(2), α = 0.13[6]). Atom W(2) also bears a terminal carbonyl ligand. The carbonyl ^{13}C NMR spectrum is consistent with this structure.

Overall the tetrametallic core is associated with 60 valence electrons, rather than the 62 expected for a (5M–M) triangulated rhombus.

The formation of $Cp_2W_2Ir_2(CO)_6(\mu_3\text{-}CPh)(\mu_3\text{-}\eta^2\text{-}C_3Ph_3)$ involves the scission of one C–C triple bond and the condensation of one of the resulting alkylidyne moieties with a second alkyne. Each of these reactions has recently been observed separately in other systems.[7,8] Most interesting is the fact that a W–Ir bond is cleaved in forming $Cp_2W_2Ir_2(CO)_6(\mu_3\text{-}CPh)(\mu_3\text{-}\eta^3\text{-}C_3Ph_3)$ whereas a W–W bond is cleaved in forming $Cp_2W_2Ir_2(CO)_8(C_2Ph_2)$. These processes occur independently, with the initial reaction apparently determining the final products. [Vahrenkamp[9] has recently observed that H_2 and C_2Ph_2 react at different sites in $Ru_2Co_2(CO)_{13}$.]

Finally, the reactions of alkynes with $Cp_2W_2Ir_2(CO)_{10}$ occur under much milder conditions and proceed more cleanly than analogous reactions with the isoelectronic $Ir_4(CO)_{12}$.[10]

Preliminary work has shown that similar reactions occur also with $CpW(CO)_2(CTol)$; however, the products are not yet fully defined.

Acknowledgment. This work was supported at the University of Illinois by National Science Foundation Grant DMR 80-20250 (to J.R.S.) and at SUNY-Buffalo by Grant CHE 80-23448 (to M.R.C.). Instruments supported by the Grants NSF CHE 79-16100 and NIH GM-27029 were utilized for NMR and mass spectra, respectively.

Registry No. I (R = R′ = Ph), 91687-09-1; I (R = r′ = *p*-tol), 91687-10-4; I (R = R′ = CF_3), 91687-11-5; I (R = Ph, R′ = CO_2Et), 91687-12-6; I (R = R′ = CO_2Et), 91687-13-7; I (R = Ph, R′ = Me), 91687-14-8; $Cp_2W_2Ir_2(CO)_6(\mu_3\text{-}CPh)(\mu_3\text{-}\eta^3\text{–}C_3Ph_3)$, 91687-15-9; $Cp_2W_2Ir_2(CO)_{10}$, 80398-82-9; $CpW(CO)_2(Ctol)$, 60260-15-3; C_2Ph_2, 501-65-5; $C_2(p\text{-}tol)_2$, 2789-88-0; $C_2(CF_3)_2$, 692-50-2; $C_2Ph(CO_2Et)$, 2216-94-6; $C_2(CO_2Et)_2$, 762-21-0; C_2PhMe, 673-32-5.

Supplementary Material Available: Tables of final positional parameters for non-hydrogen atoms, anisotropic thermal parameters, and observed and calculated structure factors (23 pages). Ordering information is given on any current masthead page.

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(3) $Cp_2W_2Ir_2(CO)_8(C_2PhMe)$: ^{13}C NMR $(CD_2Cl_2$, 20 °C) δ 222.6 (2 C, *J*(WC) = 171 Hz), 221.7 (2 C, *J*(WC) = 170 Hz), 176.2 (1 C), 175.3 (1 C), 174.5 (1 C), 171.6 (1 C).

(4) Stuntz, G. F.; Shapley, J. R.; Pierpont, C. G. *Inorg. Chem.* **1978**, *17*, 2596.

(5) This complex crystallizes in the centrosymmetric monoclinic space group $P2_1/c$ with a = 14.2534 (88) Å, b = 15.6285 (39) Å, c = 18.7311 (36) Å, β = 96.860 (38)°, V = 4142.7 (29) Å^3, and Z = 4. Diffraction data for 2θ = 4.5–45.0° (Mo Kα) were collected on a Syntex $P2_1$ diffractometer and were corrected for absorption and a linear decay (to 72% initial intensity). The structure was solved by direct methods (MULTAN 76) and refined to R_F = 8.5% for 237 parameters against 4887 data (R_F = 5.8% for those 3395 data with $|F_o| > 3.0\sigma(|F_o|)$.

(6) $\alpha = (d_1 - d_2)/d_1$, where d_1 is the "short" M–CO distance and d_2 is the "long" M–CO distance. See: Curtis, M. D.; Han, K. R.; Butler, W. M. *Inorg. Chem.* **1980**, *19*, 2096.

(7) Alkyne scission: (a) Clauss, A. D.; Shapley, J. R.; Wilker, C. N.; Hoffmann, R. *Organometallics* **1984**, *3*, 619 (and references therein). (b) Park, J. T.; Shapley, J. R.; Churchill, M. R.; Bueno, C. *J. Am. Chem. Soc.* **1983**, *105*, 6182.

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Hydrogen Bonding in Organometallic Ligands and Possible Implications for CO/H_2 Chemistry. First Carbonylation of an Isolable Metal α-Hydroxyalkyl Complex

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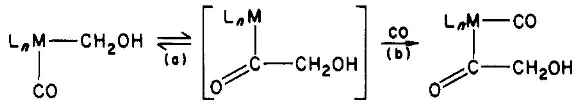
Summary: The metallacyclic α-hydroxyalkyl complex $(CO)_4\overline{MnNH_2CHR_1CHR_2C}HOH$ (**2c**, $R_1 = R_2 = -(CH_2)_4-$) carbonylates (−3 ± 1 °C, 250–360 psi of CO) to an acyl

complex, $\overline{(CO)_4MnNH_2CHR_1CHR_2CH(OH)C}{=}O$ (3c), over 16 times faster than its α-(trimethylsilyl)oxy analogue. IR and ^{13}C NMR spectra show an intramolecular hydrogen bond in 3c; this is proposed to account, at least in part, for the faster carbonylation of 2c.

Homogeneous α-hydroxyalkyl complexes $L_nMCH(R)OH$ have been proposed as intermediates in the metal-catalyzed conversion of CO/H_2 to ethylene glycol and related products,[1] as well as other processes of industrial interest.[2] Consequently, considerable recent attention has been focused upon their synthesis and reactivity.[3,4] In particular, several key questions remain regarding the mechanism of ethylene glycol formation. The best precedented carbon–carbon bond forming sequence,[5] carbonylation via α-hydroxyalkyl migration/CO capture, is shown in eq 1. However, in nearly all model alkyl complexes examined to date, electron-withdrawing α-substituents *retard* the rate of carbonylation.[6] Furthermore, no isolated α-hydroxyalkyl complexes have yet been shown to undergo carbonylation.[7]



(1)

Accordingly, we have sought an isolable α-hydroxyalkyl complex on which quantitative carbonylation rate measurements could be made. In this communication, we report (1) a general synthetic entry into a new class of metallacyclic α-hydroxyalkyl complexes, (2) the facile, <0 °C carbonylation of one of these complexes, and (3) carbonylation rate comparisons with the analogous α-((trimethylsilyl)oxy)alkyl complex. We interpret our data as providing *the first evidence for a significant intramolecular hydrogen bond induced rate acceleration in an organometallic reaction.*

Manganese acyl complexes **1a–c** were prepared from $(CO)_5MnH$ and the corresponding aziridines by the route of Beck.[8] Subsequent reduction of **1a–c** with BH_3·THF (2 equiv, 20 °C, THF, 35–40 min) and workup (CH_3OH, cold room) gave α-hydroxyalkyl complexes **2a–c** as yellow powders in >90% yields (eq 2). Complexes **2a–c** exhibited

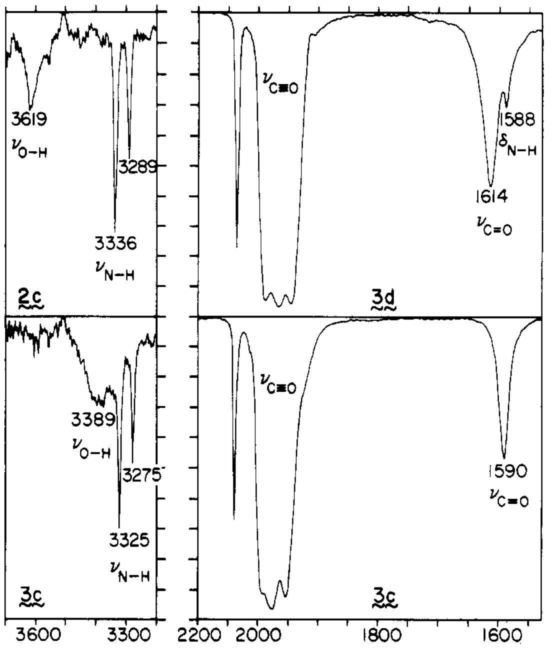
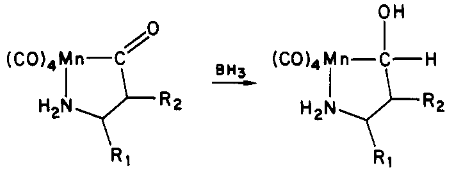


Figure 1. IR spectra (*T* vs. cm^{-1}) of **2c** and **3c** (left, eightfold *T* expansion) and **3d** and **3c** (right) in CH_2Cl_2.



(2)

1a, $R_1 = R_2 = H$ **2a**
b, $R_1 = CH_3$, $R_2 = H$ **2b**
c, $R_1 = R_2 = -(CH_2)_4-$ **2c**

characteristic IR ν_{OH} (3619–3622 (w) cm^{-1}, CH_2Cl_2) and ^{13}C NMR MnCHROH carbon resonances (82.6–89.0 ppm, CD_3OD)[9] and underwent OH/OD exchange in CD_3NO_2. Whereas **2c** was obtained >95% diastereomerically pure, **2b** was a mixture of epimers. Complex **2c** was further characterized by microanalysis and mass spectrometry. Coupled with earlier observations from our laboratory,[4b] it appears that the BH_3·THF reduction of *metallacylic* acyl complexes provides a general route to α-hydroxyalkyl complexes. In contrast, the BH_3·THF reduction of *acyclic* metal acyl complexes affords metal alkyls.[10]

When α-hydroxyalkyl complex **2c** was treated with 350–360 psi of CO at -3 ± 1 °C in CH_3NO_2 for 44 h, carbonylation to α-hydroxyacyl complex **3c** (74% isolated)[9] occurred (eq 3). A reference compound, α-((trimethylsilyl)oxy)alkyl complex **2d**, was subsequently synthesized from **2c** ($(CH_3)_3SiCl/[(CH_3)_3Si]_2NH$; 52% isolated)[9] and found to similarly carbonylate to α-(silyloxy)acyl complex **3d** (eq 3), but much more slowly. The carbonylation rates of **2c** and **2d** were monitored by IR spectroscopy at -3 ± 1 °C for 14 and 73 h, respectively, and found to be (k_{obsd}, 350–360 psi) $(125 \pm 5) \times 10^{-7}$ and $(7.5 \pm 0.4) \times 10^{-7}$ s^{-1}. Under 250–260 psi of CO, k_{obsd} of $(66.0 \pm 3.2) \times 10^{-7}$ (**2c**)

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[7] Berke has reported that the complex $((CH_3O)_3P)_2(CO)_2(Cl)FeCH_2OH$, which is *unstable* above 0 °C, undergoes carbonylation (1 atm CO, ether) to $((CH_3O)_3P)_2(CO)_2(Cl)FeCOCH_2OH$.[3d] Complexes $(C_5R_5)M(CO)_2CH_2OH$ (M = Fe, Ru) are described as inert to >4000 psi of CO (THF, 80 °C).[3i,j] The reaction of $HCo(CO)_4$, $H_2C{=}O$, and CO to give glycolaldehyde is believed to involve the carbonylation of intermediate $(CO)_4CoCH_2OH$: Roth, J. A.; Orchin, M. *J. Organomet. Chem.* **1979**, *172*, C27.

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[9] Spectroscopic charactierzation is provided in the Supplementary Material.

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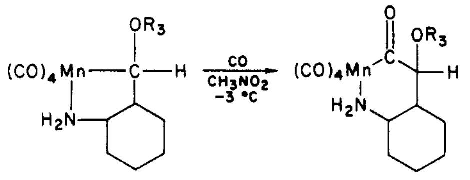


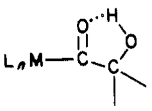
(3)

2c, R3 = H
d, R3 = Si(CH3)3

3c
3d

and (4.0 ± 0.2) × 10^{-7} s^{-1} (**2d**) were measured. Hence, at −3 °C and 250–360 psi of CO in CH_3NO_2, *α-hydroxyalkyl complex 2c carbonylates over 16 times faster than its α-(trimethylsilyl)oxy analogue.*

Previously, we reported that α-((trimethylsilyl)oxy)alkyl complex $(CO)_5MnCH_2OSi(CH_3)_3$ carbonylates ca. 3.5 times *faster* (24 °C, 750–1500 psi) than α-methoxyalkyl complex $(CO)_5MnCH_2OCH_3$.[6b] Hence, there is no evidence that the bulk of the α-(trimethylsilyl)oxy substitute inhibits carbonylation. How then can the more rapid carbonylation of the α-hydroxyalkyl ligand be accounted for? We believe that an important clue is furnished by the IR spectra shown in Figure 1. A comparison of the ν_{OH} region of **3c** and **2c** and the $\nu_{C=O}$ region of **3c** and **3d** clearly indicates the presence of a hydrogen bond in **3c**.[11] This is shown schematically in **4**. Furthermore, the ^{13}C NMR spectra of **3c** (CD_3OD) and **3d** (CD_2Cl_2) are virtually superimposable,[9] except that the acyl resonance of **3c** is 11 ppm downfield of that in **3d**. We suggest that the faster rate of carbonylation of **2c** may be due to *intramolecular hydrogen bond stabilization of the transition state or equilibrium* for the first step of eq 1.[12] As precedent, we note that Shriver found that proton acids can accelerate the carbonylation of $(CO)_5MnCH_3$;[13] he attributed this to *intermolecular* hydrogen bond stabilization of the transition state.



4

In summary, we have established the viability of intramolecular hydrogen bonding in metal α-hydroxyacyl complexes. This, when coupled with the above rate data, suggests some possible, previously unrecognized, controlling factors in the conversion of CO/H_2 to ethylene glycol. Further studies of the chemistry of metallacyclic α-hydroxyalkyl complexes are in progress.

Acknowledgment. We thank the Department of Energy for support of this research.

Registry No. **1a**, 41742-59-0; **1b**, 41742-60-3; **1c**, 91743-29-2; **2a**, 91743-30-5; **2b**, 91743-31-6; **2c**, 91743-32-7; **2d**, 91743-33-8; **3c**, 91743-34-9; **3d**, 91759-05-6; CO, 630-08-0; ethylene glycol, 107-21-1.

Supplementary Material Available: Tables of IR, ^{13}C NMR, and ^1H NMR spectra and other characterizations of **2a–d** and **3c–d** (4 pages). Ordering information is given on any current masthead page.

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[11] See: Silverstein, R. M.; Bassler, G. C.; Morrill, T. C. "Spectrometric Identification of Organic Compounds"; 4th ed.; Wiley: New York, 1981; pp 112–115, 118.

[12] The expected rate equation,[5,6] $-d[\mathbf{2c}]/dt = k_1k_2[CO][\mathbf{2c}]/(k_{-1} + k_2[CO])$, has two limiting cases. When $k_2[CO] >> k_{-1}$, the proposed hydrogen bond induced rate acceleration would reflect stabilization of a transition state (k_1), whereas when $k_{-1} >> k_2[CO]$, the rate acceleration would arise from stabilization of an intermediate (preequilibrium shift). In our experiments, these limiting conditions have apparently not yet been reached. Also, note that in substrates such as **2a–c**, hydrogen bonding would direct the alkyl migration to only *one* of the four inequivalent CO ligands.

[13] Butts, S. B.; Richmond, T. G.; Shriver, D. F. *Inorg. Chem.* 1981, *20*, 278.

Synthesis and Characterization of Sulfur-Capped Trinuclear Group 6B Metal Clusters

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Received June 18, 1984

Summary: The sulfur-capped trinuclear chromium carbonyl cluster $S[Cr(CO)_4]_3^{2-}$ has been synthesized and shown to arise via carbon monoxide loss from the CO-saturated $S[Cr(CO)_5]_3^{2-}$ species. The carbon monoxide ligands display complete stereochemical nonrigidity at ambient temperature. In a low-energy process that occurs below −60 °C the bridging carbon monoxide groups and the set of terminal CO groups that are cis to the capping sulfide moiety undergo fast exchange when compared to their chemical shift differences (1218 Hz at 50.3 MHz). A subsequent, higher energy exchange process of these carbon monoxide ligands with the remaining terminal carbonyl sites (those trans to the sulfide cap) occurs at temperatures greater than −20 °C. Addition of $Cr(^{13}CO)_5THF$ to the $S[Cr(CO)_4]_3^{2-}$ trimer results in formation of $(CO)_5CrS[Cr(CO)_4]_3^{2-}$, where the ^{13}CO ligands are shown to be statistically distributed throughout the tetrameric species.

Our interest in metal carbonyl sulfide and oxide complexes that might serve as useful models for metal carbonyls adsorbed at sulfide or oxide sites on surfaces[1] includes fundamental studies utilizing sulfur and oxygen as good nucleating ligands for group 6B metal carbonyls. A large variety of metal carbonyl derivatives containing oxygen or sulfide donor ligands display facile carbon monoxide exchange; i.e., these ancillary ligands are extremely CO labilizing.[1,2] Indeed this property of group 6B derivatives most readily accounts for the formation of metal species of higher nuclearity. For example, the tetranuclear chromium derivatives $[Cr(CO)_3OR]_4^{4-}$ (R = CH_3, Ph, H), which contain no Cr–Cr bonds, are presumably formed by aggregation of $Cr(CO)_5OR^-$ species.[3,4] Similar synthetic methodology is pivotal in the formation of the tetrameric chromium cluster $[PPN]_2[(\mu\text{-}CO)_3(CO)_9Cr_3(\mu_4\text{-}S)Cr(CO)_5]$,[5] which has recently been reported. This derivative represents the first fully characterized group 6B metal carbonyl cluster, although an early report of $Na_2[Cr_3(CO)_{14}]$ by Behrens may prove to be the first such species prepared.[6,7] We disclose herein further studies aimed at obtaining a better understanding of what promises to be a general route to the elusive group 6B metal carbonyl clusters.

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0276-7333/84/2303-1598$01.50/0