

The procedure described above must also work to introduce ^{14}C into the carbonyl position. To avoid handling ^{14}CO , we tried $\text{CH}_3^{14}\text{COCl}^{16}$ as an in situ source of ^{14}CO . Thus, $\text{RhCl}(\text{CO})(\text{PPh}_3)_2$ (0.10 mmol), benzoyl chloride (2.7 mmol), and $\text{CH}_3^{14}\text{COCl}$ (3.3 mmol, 13 mCi/mol) were reacted in benzene (5 mL) for 22 h at 100 °C. The normal workup gave benzoic acid (97%) with a specific activity of 8 mCi/mol (theoretical for statistical distribution = 7 mCi/mol). The recovered $\text{RhCl}(\text{CO})(\text{PPh}_3)_2$ was also radioactive. The success of this exchange experiment suggests that nonaromatic acid chlorides might also be successfully labeled under our "typical" conditions. In fact, *trans*-cinnamoyl chloride¹⁸ was smoothly labeled in 11 h at 100 °C (recovered acid, 96% yield, ef = 21%). We are actively pursuing experiments with simple aliphatic substrates.

Several observations suggest that the slow step in the labeling mechanism is the initial reaction of $\text{RhCl}(\text{CO})(\text{PPh}_3)_2$ with the acid chloride. Thus, the rate of the labeling depends qualitatively on the concentration of 4 and on the structure of the acid chloride. For example, a simple competition experiment revealed that 4-nitrobenzoyl chloride is labeled more rapidly than benzoyl chloride. The time courses of the ^{13}C NMR spectra show that cinnamoyl chloride labels more rapidly than does benzoyl chloride. The most important mechanistic conclusions follow from the success of these labeling experiments; the barrier to product formation (3 or 5 \rightarrow ArCl) must be greater than the barriers 5 \rightleftharpoons 3 \rightleftharpoons 2 \rightleftharpoons 6 \rightarrow 4 (see Scheme I). The labeling reaction shows that the aryl chlorides react with $\text{RhCl}(\text{CO})(\text{PPh}_3)_2$ at 100 °C. The "activation" of $\text{RhCl}(\text{CO})(\text{PPh}_3)_2$ is, therefore, not the reason that the catalytic decarbonylation of aryl chlorides requires high temperatures. Rather, the high temperature is required to form the aryl chloride product. The most important synthetic conclusion is that these reactions provide a simple, one-pot, catalytic procedure for preparing aryl chlorides labeled with ^{13}C or ^{14}C in the carbonyl group.

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for partial support of this work and to Grant IN-18 from the American Cancer Society. We are grateful to Matthey-Johnson, Inc., for a generous loan of $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ and to Professor W. D. Jones and Frank Feher for helpful discussions and technical advice.

Registry No. 4, 13938-94-8; ^{13}C -4, 88548-50-9; PhCOCl , 98-88-4; ^{13}CO , 1641-69-6; $\text{Ph}^{13}\text{COCl}$, 52947-05-4; $\text{Ph}^{13}\text{CO}_2\text{H}$, 3880-99-7; 2- $\text{ClC}_6\text{H}_4\text{COCl}$, 609-65-4; 3- $\text{NO}_2\text{C}_6\text{H}_4\text{COCl}$, 121-90-4; 4- $\text{NO}_2\text{C}_6\text{H}_4\text{COCl}$, 122-04-3; 4- $\text{CH}_3\text{OC}_6\text{H}_4\text{COCl}$, 100-07-2; 4- $\text{CH}_3\text{COC}_6\text{H}_4\text{COCl}$, 31076-84-3; 4- $\text{ClCOC}_6\text{H}_4\text{COCl}$, 100-20-9; 2- $\text{ClC}_6\text{H}_4^{13}\text{COCl}$, 88525-75-1; 3- $\text{NO}_2\text{C}_6\text{H}_4^{13}\text{COCl}$, 88525-76-2; 4- $\text{NO}_2\text{C}_6\text{H}_4^{13}\text{COCl}$, 88525-77-3; 4- $\text{CH}_3\text{OC}_6\text{H}_4^{13}\text{COCl}$, 88525-78-4; 4- $\text{CH}_3\text{COC}_6\text{H}_4^{13}\text{COCl}$, 88525-79-5; 4- $\text{Cl}^{13}\text{COC}_6\text{H}_4^{13}\text{COCl}$, 88525-80-8; $\text{CH}_3^{14}\text{COCl}$, 676-77-7; $\text{Ph}^{14}\text{CO}_2\text{H}$, 1589-66-8; *trans*- $\text{C}_6\text{H}_5\text{CH}=\text{CHCOCl}$, 17082-09-6; *trans*- $\text{C}_6\text{H}_5\text{CH}=\text{CH}^{13}\text{COCl}$, 88525-81-9.

(16) Prepared from commercial $\text{CH}_3^{14}\text{CO}_2\text{Na}$ (Amersham) by reaction with phthaloyl chloride.¹⁷

(17) Koltai, E.; Esses-Reiter, K.; Banfi, D. *J. Labelled Compd. Radiopharm.* 1982, 19, 13-18.

(18) Kampmeier, J. A.; Harris, S. H.; Rodehorst, R. M. *J. Am. Chem. Soc.* 1981, 103, 1478-1485.

A Facile Equilibrium Involving CO and H₂. The Fe-H-Fe Bond Energy in $(\mu_2\text{-H})_3\text{Fe}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$

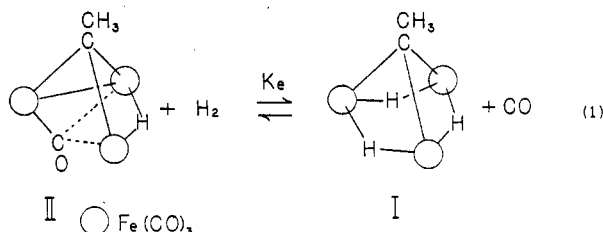
J. Vites and T. P. Fehlner*

Department of Chemistry, University of Notre Dame
Notre Dame, Indiana 46556

Received September 19, 1983

Summary: The reaction $\text{H}_2 + (\mu_2\text{-H})\text{Fe}_3(\text{CO})_{10}(\mu_3\text{-CCH}_3) = \text{CO} + (\mu_2\text{-H})_3\text{Fe}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$ is shown to be an equilibrium process with an equilibrium constant of 0.66 at 60 °C in benzene. This measurement allows an estimate of the enthalpy change of the same reaction of 4 kcal/mol. By utilization of known thermochemical information and this reaction enthalpy, the bond energy for a FeHFe three-center interaction is calculated to lie between 81 and 85 kcal/mol. This number is significantly larger than that for an FeCO interaction and suggests an energetic preference for bridging over terminal FeH bonds.

We have found that the reduction of certain iron carbonyl anions with $\text{BH}_3 \cdot \text{THF}$ leads to the formation of $(\mu_2\text{-H})_3\text{Fe}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$ (I)^{1,2} as well as other iron clusters containing hydrocarbon fragments.³ One of these products, $(\mu_2\text{H})\text{Fe}_3(\text{CO})_{10}(\mu_3\text{-CCH}_3)$ (II),⁴ is converted in the presence of H_2 into I, and I, in the presence of CO, is converted into II (eq 1). Although a number of instances



of the addition of H_2 to metal cluster compounds, with or without the displacement of another ligand, are known,^{5,6}

(1) Wong, K. S.; Fehlner, T. P. *J. Am. Chem. Soc.* 1981, 103, 966.

(2) Wong, K. S.; Haller, K. J.; Dutta, T. K.; Chipman, D. M.; Fehlner, T. P. *Inorg. Chem.* 1982, 21, 3197. DeKock, R. L.; Wong, K. S.; Fehlner, T. P. *Ibid.* 1982, 21, 3203.

(3) Fehlner, T. P.; Vites, J., unpublished work.

(4) This molecule has been characterized spectroscopically. NMR: ^1H ($\text{C}_6\text{D}_6\text{CD}_3$, -45 °C) δ 4.05 (d, 3 H, $J = 1.2$ Hz), -19.98 (q, 1 H, $J = 1.2$ Hz); ^{13}C [^1H] ($\text{C}_6\text{D}_6\text{CD}_3$, -70 °C) δ 310.0 (1 C, $\mu_3\text{-C}$), 224-205 (10 C, CO), 46.1 (1 C, CH_3). The assignment of for the quaternary carbon was proved by ^{13}CO reaction with I. At higher temperatures the ^{13}CO resonances coalesce at 209.3 and the 310.0 resonance in the ^{13}C spectrum and the -19.98 resonance in the ^1H spectrum undergo broadening. MS: p^+ = 475.785 calcd, 475.786 measd; loss of 9 CO's observed. IR (hexane, cm^{-1}): 2108 (w), 2053 (s), 2045 (s), 2020 (s), 2005 (m, sh), 1988 (w), 1890 (w, br). In Nujol mull bands at 1630 (vw, br) and 1550 (w, br) are also observed. The postulated structure is indicated in eq 1. Note that the deprotonated anion of II ($[\text{Fe}_3(\text{CO})_{10}(\mu_3\text{CCH}_3)]^-$), has been reported as two isomers, one with a $\mu_2\text{-CO}$ and one with a $\mu_3\text{-CO}$ (Lourdichi, M.; Mathieu, R. *Nouv. J. Chim.* 1982, 6, 231). Also note that a compound having the same molecular formula as II has been reported recently but has significantly different spectroscopic properties from the compound characterized here. (Wong, W. K.; Chiu, K. W.; Wilkinson, G.; Galas, A. M. R.; Thornton-Pett, M.; Hursthouse, M. B. *J. Chem. Soc., Dalton Trans.* 1983, 1557). Finally, the structure of $[\text{Fe}_3(\text{CO})_9(\mu_2\text{-CO})(\mu_3\text{-CH})]$ has been published (Kolts, J. W.; Holt, E. M.; Drezdzon, M.; Whitmire, K. H.; Shriver, D. F. *J. Am. Chem. Soc.* 1982, 104, 6134). The spectroscopic properties of II compare favorably with those reported recently for $\text{HFe}_3(\text{CO})_{10}\text{CH}$ (Kolts, J. W.; Holt, E. M.; Shriver, D. F. *J. Am. Chem. Soc.* 1983, 105, 7307).

(5) Eady, C. R.; Johnson, B. F. G.; Lewis, J. J. *J. Chem. Soc., Dalton Trans.* 1977, 477. Roland, E.; Vahrenkamp, H. *Organometallics* 1983, 2, 183. Andrews, M. A.; Kaesz, H. D. *J. Am. Chem. Soc.* 1979, 101, 7255. Keister, J. B.; Payne, M. W.; Muscatella, M. J. *Organometallics* 1983, 2, 219. Farrugia, L. J.; Green, M.; Hankey, D. R.; Orpen, A. G.; Stone, F. G. A. *J. Chem. Soc., Chem. Commun.* 1983, 310. DuBois, M. R. *J. Am. Chem. Soc.* 1983, 105, 3710. L'Éplattien, F.; Calderazzo, F. *Inorg. Chem.* 1968, 7, 1290. Hill, R. H.; Puddephatt, R. J., *J. Am. Chem. Soc.* 1983, 105, 5797.

the equilibrium properties of these reactions have not been exploited. Equilibrium data constitute one important source of thermochemical information, and such data are particularly sparse for compounds containing metal-hydride bonds⁷ even though metal hydrides are involved in many reactions of current interest.⁸ Thus, we have investigated the equilibrium properties of reaction 1 and have measured the equilibrium constant. From it a value of the bond energy of the FeHFe bridge bond is derived.

Reaction 1 was shown to be a reversible equilibrium process in the presence of both gaseous components by examining the ratio [I]/[II] (measured by ¹H NMR) for various [CO]/[H₂] ratios (measured by GLC) in benzene at 60 °C and a total pressure of 3 atm. The equilibrium constant was also measured starting with both pure I and pure II. The average value of the measurements was 0.66.⁹ In the equilibration process some decomposition was observed as the formation of an insoluble precipitate; however, as the equilibrium constant depends only on two ratios, [I]/[II] and [CO]/[H₂], this does not affect the measurements.

Making the reasonable assumption that the heats of solution and vaporization of I and II are similar, the calculated Gibbs free energy at 60 °C for (1) with all species in the gas phase is 0.3 kcal/mol. To obtain the corresponding enthalpy, the entropy must be known. A sufficiently accurate estimate of the latter may be obtained in the manner of Benson.¹⁰ As I and II have the same number of atoms and similar structures and molecular weights, the entropy change for (1) will be nearly equal to the difference in standard entropies of H₂ and CO alone, i.e., 16.0 cal/(K mol). This yields $\Delta H = 5.6$ kcal/mol. Estimating the difference in bond entropy contributions for I and II¹¹ leads to an improved approximation for the overall entropy change of (1) of 12.1 cal/(K mol), yielding $\Delta H = 4.3$ kcal/mol. All estimates¹² suggest that (1) is slightly endothermic.

The reaction enthalpy can be expressed in terms of bond energies and, with cancellation of like terms, the net result is¹⁴

$$\Delta H^\circ = E(\text{FeCO}) + 2E(\text{FeFe}) + E(\text{HH}) - 2E(\text{FeHFe}) \quad (2)$$

(6) Recently a closely related equilibrium system involving ruthenium has been reported along with a mechanistic study of the reaction. The equilibrium constant for eq 1 with Ru and OCH₃ replacing Fe and CH₃, respectively, is about 1 and can be subjected to the same type of analysis reported here. Bavaro, L. M.; Montangero, P.; Keister, J. B. *J. Am. Chem. Soc.* 1983, 105, 4977.

(7) Connor, J. A. *Top. Curr. Chem.* 1977, 71, 71.

(8) See, for example: Gladysz, J. A. *Adv. Organomet. Chem.* 1982, 20, 1.

(9) Note that this equilibrium constant is calculated from concentrations of I and II in solution and partial pressures of CO and H₂ in the gas phase. It is estimated that *K* is known within a factor of 10 or 1.5 kcal/mol in ΔG .

(10) Benson, S. W. "Thermochemical Kinetics"; Wiley: New York, 1968.

(11) In terms of nearest neighbor interactions the pertinent difference between I and II is the replacement of two FeHFe bridges by one FeCO interactions and two FeFe interactions. The contribution of the last term was assumed negligible and the difference in entropy was estimated by using the main group bond analogues BHB and C-C(O)-C for which entropic contributions are known.¹⁰ That is, $\Delta S^\circ = S(\text{CO}) + 2S(\text{BHB}) - S(\text{H}_2) - S(\text{CC}(\text{O})\text{C}) = 47.2 - 4.3 - 31.2 + 0.1 = 12.1$ cal/(K mol). Although the absolute entropy contributions of the transition-metal units are probably poorly estimated by the main group models, the differences should be reasonably accurate.

(12) Alternately, the Nernst approximation formula¹³ yields 3.2 kcal/mol for ΔH .

(13) Prutton, C. F.; Maron, S. "Fundamental Principles of Physical Chemistry"; Macmillan: New York, 1951; p 359.

(14) A complication would arise if the structure of II is not that proposed; however, given the spectroscopic data, it is clear that the main enthalpy difference will be expressed by (2) even if there are subtle features of the actual structure not revealed spectroscopically.



where $E(\text{FeCO})$ is the net interaction energy of the CO lost with the iron cage, $2E(\text{FeFe})$ is the metal-metal interaction energy lost, and $2E(\text{FeHFe})$ is the bridging interaction energy gained. Note that $E(\text{FeHFe})$ applies to the entire interaction energy of the three-atom unit. The value $E(\text{HH})$ is known (104.7 kcal/mol), and the energetics of iron carbonyl clusters have been analyzed previously^{7,15} in two independent manners. In one⁷ (method I), the heats of disruption were assigned to three types of term values, giving $E(\text{FeCO}) = 30.6$ kcal/mol (bridging) and $E(\text{FeFe}) = 19.6$ kcal/mol. In the other (method II), allowance was made for variation in the FeFe bond length, yielding for $\text{Fe}_3(\text{CO})_{12}$ $E(\text{FeCO}) = 30.0$ kcal/mol and $E(\text{FeFe}) = 15.5$ kcal/mol for a bridged $d(\text{FeFe}) = 2.56$ Å (the known FeFe unbridged distance in $[\text{Fe}_3(\text{CO})_9(\mu_2\text{-CO})(\mu_3\text{-CH})]^{-4}$). Note that both analyses of the disruption energies yield $E(\text{FeCO})$ values that are independent (within 2 kcal/mol) of whether the CO is terminal, μ_2 bridging, or μ_3 bridging. Utilization of these energy term values yields $E(\text{FeHFe})$ of 85.1 and 80.7 kcal/mol, respectively. To our knowledge, this is the first determination of the strength of the hydrido-transition-metal bridge bond. The striking aspect of this energy is its size; i.e., it is considerably larger than $E(\text{FeCO}) + E(\text{FeFe})$. This conclusion is independent of the method of analysis of the iron carbonyl cluster disruption energies.

Additional appreciation of the magnitude of this bond energy can be obtained by considering Scheme I.¹⁷ (When two energies are given, the first number is derived via method I, the second (in parentheses) via method II.) The scheme suggests an energetic preference for bridging hydride of 10–11 kcal/mol. This energy loss is independent of the method of partitioning the iron carbonyl disruption energies but obviously depends on the rearrangement of the Fe-Fe bonding, if any, in the conversion of bridging hydrogen to terminal hydrogen as well as on the value assigned to terminal $E(\text{FeH})$.¹⁷ The energy necessary to create an FeH terminal bond and an unsaturated Fe center from an FeHFe bond is even larger but also depends on the value assigned to $E(\text{FeFe})$. Despite this, the qualitative conclusion is clear. Just as a three-center two-electron BHB bond is energetically favored over a two-center, two-electron BH bond and an empty boron valence orbital

(15) Housecroft, C. E.; Wade, K.; Smith, B. C. *J. Chem. Soc., Chem. Commun.* 1978, 765. Housecroft, C. E.; O'Neill, M. E.; Wade, K. *J. Organomet. Chem.* 1981, 213, 35.

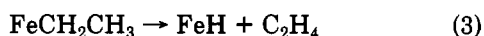
(16) This supports the contention based on chemistry that M-H bonds in this area of the periodic table have a high thermodynamic stability. Lewis, J.; Johnson, B. F. G. *Pure Appl. Chem.* 1975, 44, 43.

(17) Although the value of $E(\text{FeH})$ (terminal) is uncertain, estimates lie between 50 and 60 kcal/mol^{7,8} and a value of 55 kcal/mol is used here.

by 18 kcal/mol,¹⁸ so too is the FeHFe interaction the favored one. The observation that in clusters bridging hydrides are the rule, terminal hydrides the exceptions, is consistent with these energetics.¹⁹

It is educational to compare the cycle in Scheme I with a similar cycle for the FeCO interaction (Scheme II). This comparison emphasizes the weakness of the FeCO interaction compared to FeHFe, the difference in the energies for CO and H necessary to create an unsaturated Fe center and a terminal bond, and the difference in the energies for CO and H in creating a terminal interaction from a bridging one.

The M-H bond strength is one factor important in determining the thermodynamics of several interconversions involving metals and hydrocarbon fragments.⁷ The numerical value of $E(\text{FeHFe})$ reported here has several implications in this regard. For example, $E(\text{FeC})$ and $E(\text{FeH})$ (terminal) suggest that β -hydrogen elimination (eq 3) on



a mononuclear metal center is an endothermic reaction,²⁰ albeit with a favorable entropy change. In a polynuclear system, the hydride has the possibility of ending up bridging, which will favor the elimination reaction by about another 10-11 kcal/mol (Scheme I). Thus, hydride "eliminations" from carbon resulting in MHM hydrides are possible in the absence of a favorable mole change. One example is provided by an internal rearrangement in a triosmium system,²¹ where MC(O) and CH bonds are converted into MHM and C=C(O) bonds in a unimolecular process.

The additional stability of a bridging hydride over a terminal hydride suggests the possibility of a real difference between the chemistry of mono- and polynuclear metal hydride systems. Further exploration of this and related systems is in progress.

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Registry No. I, 69440-00-2; II, 88610-52-0.

(18) Fehlner, T. P. In "Boron Hydride Chemistry"; Muettterties, E. L., Ed.; Academic Press: New York, 1975; p 175.

(19) Muettterties, E. L.; Rhodin, T. N.; Band, E.; Brucker, C. F.; Pretzer, W. R. *Chem. Rev.* 1979, 79, 91.

(20) With $E(\text{FeC}) = 30$ kcal/mol, $E(\text{FeH}) = 55$ kcal/mol, $E(\text{CH}) = 98$ kcal/mol, and $E(\text{C}=\text{C}) - E(\text{C}-\text{C}) = 62$ kcal/mol, $\Delta H = 11$ kcal/mol.

(21) Shapley, J. R.; Strickland, D. S.; St. George, G. M.; Churchill, M. R.; Bueno, C. *Organometallics* 1983, 2, 185.

Oxidative Carbonylation of Alcohols: Dialkyl Oxalates from (Dialkoxycarbonyl)rhodium Complexes

Patrick L. Burk,* Donna Van Engen,* and Kathryn S. Campo

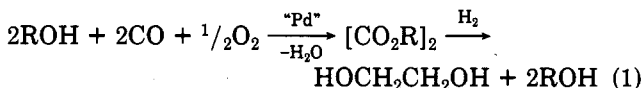
Exxon Research and Engineering Company
Annandale, New Jersey 08801

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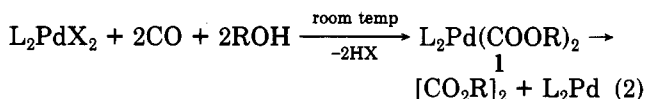
Summary: The oxidative carbonylation of alcohols to generate dialkyl oxalates has, for the first time, been accomplished with rhodium-based complexes.

The employment of synthesis gas (mixtures of CO and H₂) as a reactant for the generation of organic products

that contain new carbon-carbon bonds encompasses such diverse reactions as olefin hydroformylation,¹ Fischer-Tropsch catalysis,² alcohol and carboxylic acid homologation,^{3,4} and, of particular interest to us, ethylene glycol synthesis.⁵ The direct production of ethylene glycol from synthesis gas has required both high pressures and elevated temperatures. Another attractive, less vigorous route to this diol makes sequential rather than concomitant use of the gases CO and H₂. First, CO and an alcohol are oxidatively coupled to generate a dialkyl oxalate, which is subsequently reduced by H₂ to produce ethylene glycol and the starting alcohol (eq 1). The oxalate-producing step

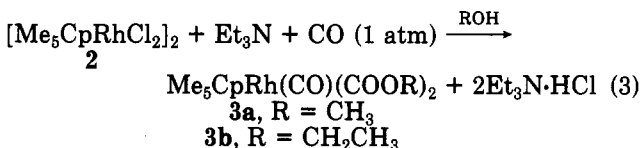


has thus far been accomplished only with palladium-based catalysts.^{6,7} The key palladium carbonylation intermediate appears to be a dicarboalkoxy complex 1 which releases oxalate to the system by reductive coupling of the two alkoxy-carboxylato ligands (eq 2).⁸ We now report the



synthesis of a new class of rhodium dicarboalkoxy complexes which upon gentle prodding produce dialkyl oxalates.

Treatment of a methanol suspension of $[\text{Me}_5\text{CpRhCl}_2]_2$ (2) with bubbling CO and an excess of triethylamine at room temperature (eq 3) produced, after 3 h, a deep red-



orange solution. Evaporation of the solvent in vacuo gave a solid which was extracted with diethyl ether. Addition of hexane to this ether solution followed by cooling to -40 °C overnight gave the yellow crystalline solid 3a (40%).⁹ The ¹H NMR (CDCl₃-Me₄Si) of 3a contained two singlets at 3.6 and 1.9 ppm in a ratio of 2:5, which suggested the product contained two methoxy groups per Me₅Cp ligand. The IR spectrum (Nujol) of 3a showed three absorption bands between 2200 and 1600 cm⁻¹: 2056, 1668, and 1645 cm⁻¹. The bands between 1700 and 1600 cm⁻¹ are characteristic of carboalkoxy ligands,¹² however, bridging carbonyls and carboxylates also absorb in this region. The

(1) Pruett, R. L. "Advances in Organometallic Chemistry"; Stone, F. G. A., West, R., Eds.; Academic Press: New York, 1979; Vol. 17, pp 1-60.

(2) Rofer-DePoorter, C. K. *Chem. Rev.* 1981, 81, 447-474.

(3) Doyle, G. J. *Mol. Catal.* 1981, 13, 237-247.

(4) Knifton, J. F. "Catalytic Activation of Carbon Monoxide"; Ford, P. C., Ed.; American Chemical Society: Washington, DC, 1981; pp 225-242.

(5) Pruett, R. L. "The Place of Transition Metals in Organic Synthesis"; Slocum, D. W., Ed.; The American Academy of Sciences: New York, 1977; Vol. 295, pp 239-248.

(6) Rivetti, F.; Romano, U. *Chim. Ind. (Milon)* 1980, 62, 7-12.

(7) Fenton, D. M.; Steinwand, P. J. *J. Org. Chem.* 1974, 39, 701-704.

(8) (a) Rivetti, F.; Romano, U. *J. Organomet. Chem.* 1978, 154, 323-328. (b) Rivetti, F.; Romano, U. *Ibid.* 1979, 174, 221-226.

(9) The other major carbonyl-containing organometallic compounds produced were $[\text{Me}_5\text{CpRh}(\text{CO})]_2$ ¹⁰ and $[\text{Me}_5\text{CpRh}(\text{CO})]_2$ ¹¹

(10) Kang, J. W.; Maitlis, P. M. *J. Organomet. Chem.* 1971, 26, 393-399.

(11) Nulton, A.; Maitlis, P. M., *Ibid.* 1979, 166, C21-C22.

(12) Angelici, R. J. *Acc. Chem. Res.* 1972, 5, 335-341.