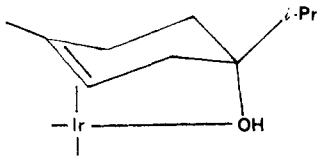
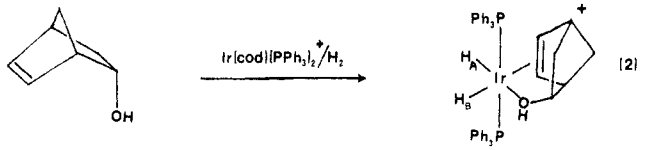
weaker but also catalytic activity is usually insufficient to reduce the trisubstituted C=C group. Complex **3** is a



3

plausible intermediate in this process. We wished to try to observe such a species at low temperature. In our previous work in the area we found that the $[Ir(cod)P\text{-}c\text{-}Hx_3(py)]^+$ system, while it was the most active catalyst, did not allow observation of catalytic intermediates. For this we turned to the closely related $[Ir(cod)(PPh_3)_2]^+$ system, a poor catalyst.[7] Our studies[2] have suggested a close mechanistic parallel between the two cases.

A ^1H NMR study of the chemistry of **1** with $[Ir(cod)(PPh_3)_2]^+$ showed that a number of species was formed on hydrogenation. We imagine that the OH group of **1** binds more efficiently than does the C=C group, as we have found[2] in competition experiments between alcohols and olefins. No complex of type **3** could be detected, and it may never be present in other than trace amounts. In order to increase the binding constant of the C=C group so that a chelate could be formed, we used *endo*-5-norbornen-2-ol, **4**. The strain present in the C=C group was expected to lead to a higher binding constant. We did observe a single organometallic product on hydrogenating $[Ir(cod)(PPh_3)_2]^+$ in the presence of **4** or by treating $[IrH_2(Me_2CO)_2(PPh_3)_2]^+$ with **4**. This product was shown by ^1H NMR spectroscopy at 0 °C to be **5** (see eq. 2).



(2)

4 5

When **5** was prepared from the acetone complex, it was formed in ca. 70% yield in equilibrium with the acetone complex itself, so the binding is not particularly strong. Indeed, too strong a binding would inhibit catalysis. No other norbornenol complexes can be detected, suggesting this ligand has no significant tendency toward monodentate binding.

Complex **5** is the isomer expected by analogy with the known configuration[2] of nonchelating analogs such as $[IrH_2(C_2H_4)(H_2O)(PPh_3)_2]^+$. Two IrH resonances are observed. One, H_A, has a chemical shift (δ −29.4) characteristic for IrH trans to an oxygen ligand (typical range δ −29 to −32) and the other, H_B, a shift (δ −9.6) characteristic[2] for IrH trans to an olefin (typical range δ −9 to −14). The two resonances arise from the same molecule as coupling is observed between H_A and H_B ($^2J(H,H)$ = 5.5 Hz). The coupling to phosphorus shows that two inequivalent phosphorus nuclei are present, as expected on the basis of structure **5** (e.g., $^2J(H_A, P)$ = 11 and 24 Hz). When being warmed to +30 °C the complex decomposes, apparently by simple loss of H_2.

We propose that the key intermediate in the directing effect observed is of type **5**. Since ROH binds more strongly[2] to the catalyst than does an olefin, we do not expect that binding of the catalyst to the face of compound **1** opposite the OH group is likely, although we cannot rule it out completely. The chemical yield of **2a** was 95%. In contrast, much lower yields are usually observed for the reduction of simple trisubstituted olefins not bearing OH groups, due to deactivation of the catalyst by trimerization. The presence of the OH group does, however, decrease the rate of reduction from ca. 4000 mol of H_2 (mol of Ir)$^{-1}$ h^{-1} for 1-methylcyclohexene to 30 mol of H_2 (mol of Ir)$^{-1}$ h^{-1} for **1**. The OH group therefore protects the catalyst but slows the rate of reduction.

We have shown[8] that a hydroxyl group can direct the stereochemistry of hydrogenation with $[Ir(cod)P\text{-}c\text{-}Hx_3(py)]^+$ and that this probably occurs by a chelated intermediate of type **5**, of which we have studied one example.

Further work is in hand on the effects of different functional groups and substitution patterns.

Acknowledgment. We thank the National Science Foundation, the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the Exxon Educational Foundation for support. M.W.D. gratefully acknowledges support as an F. W. Heyl and Elsie L. Heyl Fellow, and R.H.C. thanks the A.P. Sloan and Henry and Camille Dreyfus Foundations for fellowships.

Registry No. **1**, 562-74-3; **2a**, 3239-02-9; **2b**, 3239-03-0; **4**, 694-97-3; **5**, 84558-26-9; $[Ir(cod)P(c\text{-}Hx_3)(py)]PF_6$, 64536-78-3.

(6) Osborn, J. A.; Jardine, F. H.; Young, J. F.; Wilkinson, G. *J. Chem. Soc. A* **1966**, 1711.

(7) Crabtree, R. H.; Felkin, H.; Fellebeen-Khan, T.; Morris, G. E. *J. Organomet. Chem.* **1979**, *168*, 183.

(8) Stork et al.[9] and Brown and Naik[10] have independently found similar directing effects in the reduction of unsaturated alcohols with $[Ir(cod)Pc\text{-}Hx_3(py)]PF_6/CH_2Cl_2$ and $[Rh(nbd)(PPh_2C_4H_8PPh_2)]BF_4/CH_2Cl_2$, respectively. In the latter case only 1,1-disubstituted olefins were reduced; the iridium catalyst, being more active, is free from this limitation.

(9) Stork, G., personal communication, 1982.

(10) Brown, J. M.; Naik, R. G. *J. Chem. Soc., Chem. Commun.* **1982**, 348.

Stereoselective Formation of Iridium(III) Amides and Ligand-Assisted Heterolytic Splitting of Dihydrogen

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Received November 9, 1982

Summary: A series of octahedral iridium(III) amides are formed stereoselectively from four- and five-coordinate precursors. In addition, an unusual ligand-assisted heterolytic splitting of dihydrogen is observed.

We have recently reported[1] that the iridium(I) amido phosphine complex $[Ir(COE)N(SiMe_2CH_2PPh_2)_2]$ (**1**), where COE = η^2-cyclooctene, acts as an efficient catalyst precursor for the homogeneous hydrogenation of simple olefins. In an effort to delineate the mechanism of this process, we investigated a number of stoichiometric oxidative addition and substitution processes to monitor the fate of the square-planar iridium(I) amide precursor. In this communication, we describe (i) the first examples of iridium(III) amides, (ii) a series of completely stereose-

(1) Fryzuk, M. D.; MacNeil, P. A. *Organometallics* **1983**, *2*, 355.

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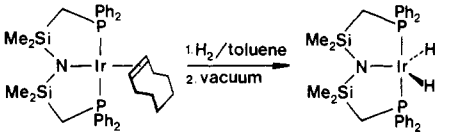
lective reactions that generate octahedral complexes from four- and five-coordinate starting materials, and (iii) a ligand-assisted heterolytic splitting of dihydrogen (H_2).

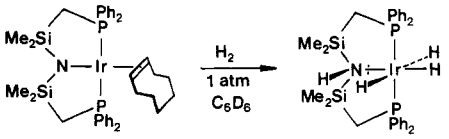
When a toluene solution of **1** is allowed to stir under 1 atm of H_2 for 1 h, the coordinatively unsaturated iridium(III) dihydride **2** can be isolated in high yield after removal of solvent and H_2 in vacuo (eq 1). The most



1 2 (1)

compelling evidence[2] for **2** is the sharp triplet at −24.9 ppm (^2J_P = 13.2 Hz) in the ^1H NMR spectrum for the IrH resonance; in addition, the ^1H NMR spectrum is invariant down to −80 °C, suggesting that the proposed trigonal-bipyramidal structure of **2** is stereochemically rigid. Additional evidence for a rigid structure is provided by the stereoselective addition reactions of **2** (vide infra).

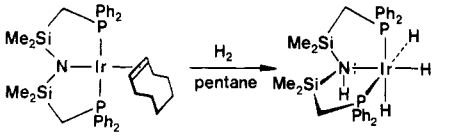
If the reaction between **1** and H_2 is monitored (under excess H_2) by ^1H NMR spectroscopy, one observes the presence of *free* cyclooctene in the initial stages which is subsequently hydrogenated to cyclooctane. More importantly, no resonances assignable to **2** are observed; in fact, the solution spectroscopic data[3] are consistent with the presence of an iridium(III) amine trihydride, **3** (eq 2). The



1 3 (2)

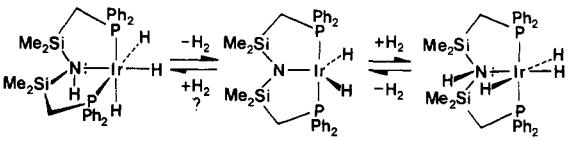
^1H NMR spectrum of a solution of **3** shows three multiplets to high field for the three different Ir–H moieties; in addition the IR (C_6D_6 under H_2) has two moderate intensity bands at 2175 and 1705 cm^{-1} for the Ir–H stretching frequencies, the lower energy absorption characteristic of a trans-H–Ir–H configuration,[4] as well as a weak N–H stretch at 3210 cm^{-1}. All of these absorptions shift appropriately upon deuteration (with D_2). Removal of H_2 from solutions of **3** quantitatively generates the iridium(III) amide dihydride **2**; in fact, when C_6D_6 solutions of **2** are sealed under H_2 (≤1 atm), **3** is formed instantaneously.

In an attempt to isolate **3**, we stirred a concentrated pentane solution of the iridium(I) cyclooctene derivative **1** under H_2 and observed the formation of a fine, yellow precipitate (>70% isolated yield). The spectral and analytical data[5] are consistent with the *facial stereoisomer* of **3**, a new iridium(III) amine trihydride **4** (eq 3). In par-



1 4 (3)

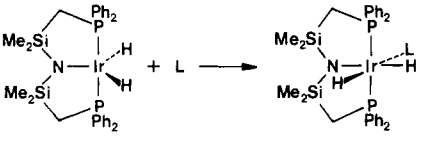
ticular, the infrared (KBr) shows two Ir–H absorptions at 2180 and 2115 cm^{-1} and a weak N–H stretch at 3200 cm^{-1}. Although stable in the solid state, **4** slowly isomerizes in C_6D_6 solution under H_2, to the meridional isomer **3** (~24 h). Under N_2 in solution, **4** decomposes to give a mixture of **2** and **3**, thus suggesting that the stereoisomerization of **4** to **3** occurs via dissociation of H_2 from the *fac* trihydride **4** to generate the dihydride **2** which, in the presence of H_2, forms the *mer* trihydride **3** (eq 4). The for-



4 2 3 (4)

mation of **3** and **4** from the dihydride **2** is, formally, an intramolecular, ligand-assisted, heterolytic[6] splitting of H_2. Whether this occurs in a concerted process or in a stepwise process involving oxidative addition to generate an Ir(V) intermediate, followed by reductive elimination, is unknown.

The iridium(III) amide dihydride **2** easily adds simple ligands such as PMe_3 and CO in a completely stereoselective fashion to generate the isomerically pure meridional-cis dihydride complexes **5** (L = PMe_3) and **6** (L = CO) (eq 5). The formation of the *mer*-cis stereochemistry



2 5 L = PMe3
6 L = CO (5)

requires that the ligand L approach **2** cis to the iridium–amide bond and coordinate to either of the N–Ir–H edges. For reasons that are obscure at present, approach of L trans to the iridium–amide bond of **2**, to generate the unobserved *mer*-trans isomer, is completely inhibited. Furthermore, isomerization of the unobserved *mer*-trans isomer to the observed *mer*-cis isomer (**5** or **6**) can be ruled out on the basis of the following experiment:[8] if paraformaldehyde $(HCHO)_n$ is stirred with the iridium cyclooctene complex **1**, the isomerically pure meridional-trans

(2) **2**: ^1H NMR (C_6D_6, ppm) $SiCH_3$ 0.24 (s), CH_2P 1.89 (t, J_{app} = 5.2 Hz), $P(C_6H_5)_2$ 7.02 (m, para/meta), 7.92 (m, ortho), IrH −24.86 (t, $^2J_{H,P}$ = 13.2 Hz); $^{31}P\{^1H\}$ NMR (C_6D_6, $P(OMe)_3$ internal reference, ppm) 23.9 (s); IR (KBr, cm^{-1}) ν_{Ir-H} 2200 (m). Anal. Calcd for $C_{30}H_{38}IrNP_2Si_2$: C, 49.86; H, 5.26; N, 1.94. Found: C, 50.20; H, 5.56; N, 2.00.

(3) **3**: ^1H NMR (C_6D_6, ppm) $SiCH_3$ −0.02 (s), 0.05 (s), CH_2P, 1.70 (dt, J_{app} = 4.9 Hz, J_{gem} = 14.1 Hz), 2.36 (dt, J_{app} = 4.1 Hz), $P(C_6H_5)_2$ 6.99, 7.12 (m, para/meta), 8.20, 8.35 (m, ortho), IrH, −8.97 (td, $^2J_{P,H}$ = 19.5 Hz, $^2J_{H,H}$ = 5.0 Hz), −9.69 (td, $^2J_{P,H}$ = 18.0 Hz), −24.6 (tt, $^2J_{P,H}$ = 15.5 Hz); $^{31}P\{^1H\}$ NMR (C_6D_6, $P(OMe)_3$ external reference, ppm) 11.44 (s); IR (C_6D_6, cm^{-1}) ν_{N-H} 3210 (w), ν_{Ir-H} 2175 (m), 1705 (m).

(4) Adams, D. M. "Metal-Ligand and Related Vibrations"; Edward Arnold (Publishers) Ltd.: London, 1967; p 6.

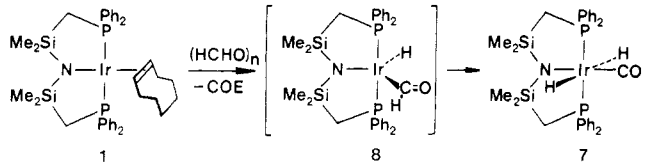
(5) **4**: ^1H NMR (C_6D_6, ppm) $SiCH_3$ 0.23 (s), 0.25 (s), CH_2P 1.91 (dd, $^2J_{H,P}$ = 9.00 Hz, J_{gem} = 14.1 Hz), 2.09 (dd, $^2J_{H,P}$ = 11.00 Hz), $P(C_6H_5)_2$ 6.98 (m), 8.28 (m), IrH_A (trans to PPh_2), −8.5 (m, from spectral simulation of AA'MXX' pattern, $^2J_{A,A'}$ = 2.2 Hz, $^2J_{A,M}$ = $^2J_{A',M}$ = 5.5 Hz, $^2J_{A,X}$ = $^2J_{A',X'}$ = −19.0 Hz, $^2J_{M,X}$ = $^2J_{M,X'}$ = 14.0 Hz, $^2J_{A,X'}$ = $^2J_{A',X}$ = 130.0 Hz, $^2J_{X,X'}$ = 1.0 Hz), IrH_M (trans to NH) −24.3 (tt); IR (KBr, cm^{-1}) ν_{N-H} 3200 (w), ν_{Ir-H} 2115 (m), 2180 (w). Anal. Calcd for $C_{30}H_{40}IrNP_2Si_2$: C, 49.72; H, 5.52; N, 1.93. Found: C, 49.86; H, 5.66; N, 1.97.

(6) Halpern, J. *J. Organomet. Chem.* **1980**, *200*, 133.

(7) **5**: ^1H NMR (C_6D_6, ppm) $SiCH_3$ 0.18 (s), 0.53 (s), $P(CH_3)_3$ 0.76 (d, $^2J_{H,P}$ = 8.0 Hz), CH_2P 1.79 (dt, J_{app} = 5.2 Hz, J_{gem} = 13.0 Hz), 2.22 (dt, J_{app} = 5.2 Hz), $P(C_6H_5)_2$ 6.95, 7.05 (m, para/meta), 7.83, 8.14 (m, ortho), IrH (trans to PPh_2) −10.21 (ddt, $^2J_{H,P}$(cis) = 17.6 Hz, $^2J_{H,P}$(trans) = 135.0 Hz, $^2J_{H,H}$ = 5.1 Hz), IrH (trans to N) −19.96 (dquart, $^2J_{H,P}$ = 19.5 Hz); $^{31}P\{^1H\}$ NMR (C_6D_6, $P(OMe)_3$ internal reference, ppm) PPh_2 11.00 (d, $^2J_{PMe_3,PPh_2}$ = 19.0 Hz), PMe_3 −56.83 (br t); IR (KBr, cm^{-1}) ν_{Ir-H} 2110 (s, br). Anal. Calcd for $C_{33}H_{47}IrNP_3Si_2$: C, 49.62; H, 5.89; N, 1.75. Found: C, 50.00; H, 6.00; N, 1.74. **6**: ^1H NMR (C_6D_6, ppm) $SiCH_3$ 0.31 (s), 0.34 (s), CH_2P 1.80 (dt, J_{app} = 5.4 Hz, J_{gem} = 13.8 Hz), 2.09 (dt, J_{app} = 6.5 Hz), $P(C_6H_5)_2$ 6.96, 7.04 (m, para/meta), 7.70, 7.95 (m, ortho), IrH (trans to CO) −7.86 (dt, $^2J_{H,P}$ = 17.6 Hz, $^2J_{H,H}$ = 4.4 Hz), IrH (trans to N), −16.09 (dt, $^2J_{H,P}$ = 12.5 Hz); $^{31}P\{^1H\}$ NMR (C_6D_6, $P(OMe)_3$ internal reference, ppm) 24.3 (s); IR (KBr, cm^{-1}) ν_{Ir-H} 2075 (s), 1925 (s), ν_{CO} 1965 (s). Anal. Calcd for $C_{31}H_{38}IrNOP_2Si_2$: C, 49.60; H, 5.07; N, 1.87. Found: C, 49.90; H, 5.13; N, 1.90.

(8) Thorn, D. L. *Organometallics* **1982**, *1*, 927.

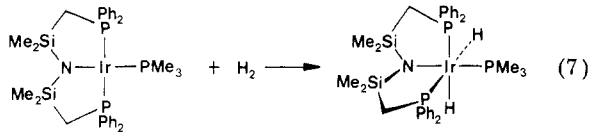
dihydride derivative[9] 7 is formed in virtually quantitative yield (eq 6). Although the intermediate cis formyl hydride



1 8 7 (6)

8 was not detected, it is reasonable to suggest that a stereoselective migratory deinsertion process[10] occurs to generate the *mer*-trans complex 7. Solutions of 7 do not isomerize to the *mer*-cis complex 6 even under 1 atm of CO.

Both oxidative additions of H_2 and paraformaldehyde to 1 have similar features; in each case, dissociation of cyclooctene accompanies oxidative addition to generate a rigid, five-coordinate derivative, 2 or 8, which undergoes further reaction to 3 and 7, respectively, depending on the reaction conditions. In the absence of dissociation, straightforward oxidative addition is observed; thus the reaction of H_2 with the analogous iridium(I) complex[1] $[Ir(PMe_3)N(SiMe_2CH_2PPh_2)_2]$ (9) proceeds with complete stereoselectivity to generate the facial-cis dihydride 10 in quantitative yield (eq 7). The IrH resonance[11] appears



9 10 (7)

as a second-order, symmetrical multiplet at −11.04 ppm, which can be simulated as an AA′XX′Y spin system. In addition, confirmation of the *fac*-cis stereochemistry was provided by an X-ray crystal structure[12] of 10. That 9 does not undergo dissociation of PMe_3 is presumably the reason it is not a catalyst precursor for hydrogenation reactions.[1] The related iridium(I) carbonyl[1] $[Ir(CO)N(SiMe_2CH_2PPh_2)_2]$ does not oxidatively add dihydrogen.

This study has shown that a number of heretofore unknown[13] iridium(III) amides (complexes 2, 5, 6, 7, and 10) can be isolated by a series of completely stereoselective reactions. In addition, the formation of both 3 and 4 is an example of a novel ligand-assisted heterolytic splitting of H_2. Further studies to determine the importance of these reactions to the mechanism of hydrogenation are underway.

Acknowledgment. Financial support for this research was generously provided by the UBC Department of Chemistry and the Natural Sciences and Engineering Research Council of Canada. We also thank Johnson Matthey for the loan of $IrCl_3$. P.A.M. acknowledges the Walter C. Sumner Memorial Foundation for a graduate scholarship.

Registry No. 1, 84074-30-6; 2, 84751-23-5; 3, 84751-24-6; 4, 84799-44-0; 5, 84751-25-7; 6, 84751-26-8; 7, 84799-45-1; 9, 84074-32-8; 10, 84799-46-2; H_2, 1333-74-0.

(9) 7: ^1H NMR (C_6D_6, ppm) $SiCH_3$ 0.18 (s), CH_2P 2.04 (t, J_{app} = 5.7 Hz), $P(C_6H_5)_2$ 6.98 (m, para/meta), 7.87, (m, ortho), IrH −6.00 (t, $^2J_{H,P}$ = 14.7 Hz), $^{31}P\{^1H\}$ NMR (C_6D_6, $P(OMe)_3$ internal reference, ppm) 9.68 (s); IR (KBr, cm^{-1}) ν_{CO} 1990 (s), ν_{Ir-H} 1725 (s). Anal. Calcd for $C_{31}H_{38}IrNOP_2Si_2$: C, 49.60; H, 5.07; N, 1.87. Found: C, 50.00; H, 5.15; N, 1.88.

(10) Gladysz, J. A.; Johnson, D. L.; Tam, W.; Williams, G. M. *J. Organomet. Chem.* **1977**, *140*, C1.

(11) 9: ^1H NMR (C_6D_6, ppm) $SiCH_3$ 0.32 (s), 0.72 (s), CH_2P 1.95 (m), 2.05 (m), $P(C_6H_5)_2$ 6.89 (m), 7.08 (m), IrH (m, from spectral simulation of AA′XX′Y pattern: $^2J_{A,X} = {}^2J_{A',X'} = -21.0$ Hz, $^2J_{A,Y} = {}^2J_{A',Y} = 21.0$ Hz, $^2J_{A,X'} = 147.0$ Hz, $^2J_{A,A'} = 4.0$ Hz, $^2J_{X,Y} = 9.0$ Hz, $^2J_{X,X'} = 4.0$ Hz); $^{31}P\{^1H\}$ NMR (C_6D_6, $P(OMe)_3$ internal reference, ppm) PPh_2 −1.67 (br d, $^2J_{PMe_3,PPh_2} = 9.0$ Hz), PMe_3 −51.62 (t); IR (KBr, cm^{-1}) ν_{Ir-H} 2065 (s), 2020 (s). Anal. Calcd for $C_{33}H_{47}IrNP_3Si_2$: C, 49.62; H, 5.89; N, 1.75. Found: C, 49.91; H, 5.87; N, 1.84.

(12) Rettig, S. J., personal communication.

(13) Lappert, M. F.; Power, P. P.; Sanger, A. R.; Srivastava, R. C. "Metal and Metalloid Amides"; Horwood-Wiley: Chichester-New York, 1980; p 488.

Photochemical Synthesis and Structure of (μ-η^4-*syn*-1,3-Butadiene)(μ-carbonyl)bis(η^5-cyclopentadienyl)dicobalt(*Co-Co*), a Dinuclear Butadiene Complex

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Received November 16, 1982

Summary: Irradiation of (η^4-1,3-butadiene)(η^5-C_5H_5)Co in the presence of (η^5-C_5H_5)$Co(CO)_2$ results in the title compound, the first dinuclear parent η^4-butadiene complex adopting a syn configuration to be characterized by X-ray crystallography.

Transition-metal-mediated transformations involving butadiene are of considerable current academic and industrial interest.[1] Whereas there are a number of structurally characterized mononuclear diene complexes known, molecules in which the diene unit is bound to more than one metal are scarce,[2] a possible reflection of the lack of facile synthetic approaches to such compounds. Nevertheless, their bonding characteristics should command attention as potential indicators of ligand-surface interactions,[3] of relevance to heterogeneous catalysis, and as key structures with which to investigate the organometallic chemistry of higher nuclear clusters.[4] We wish to report the synthesis of the title compound 3 by a novel route, which promises to be general, and its X-ray structural features. Complex 3 is the first μ-η^4-*syn*-1,3-butadiene complex to be unambiguously characterized in this fashion.

Our approach to 3 was modeled after the photochemical addition of CpCoCO (Cp = η^5-C_5H_5) to $CpCo(CO)_2$ (1), which furnishes $Cp_2Co_2(CO)_3$ containing one bridging and two terminal carbonyl ligands.[5] It was of interest to de-

† University of California.

(1) G. W. Parshall, "Homogeneous Catalysis. The Applications and Chemistry of Catalysis by Soluble Transition Metal Complexes", Wiley-Interscience, New York, 1980. P. W. Jolly and G. Wilke, "The Organic Chemistry of Nickel", Vol. 2, Academic Press, New York, 1975.

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