Acknowledgment. Financial support for this research by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie is gratefully acknowledged. We thank a referee for valuable comments.

Registry No. [Ru(NH_3)$_5$Cl]Cl_2, 18532-87-1; K_4[Ru(CN)$_6$], 15002-31-0; Na[(NH_3)$_5$Ru(NC)Ru(CN)$_5$], 81177-85-7.

Bimetallic Acyl Complexes. Use of Transition Organometallic Lewis Acids in Promoting Migratory CO Insertion

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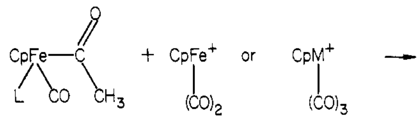
Migratory CO insertion, which generates a metal acyl system via formal CO insertion into the metal–alkyl bond, serves as a fundamental reaction of organometallic chemistry[1] and functions as a key step in several homogeneous catalytic transformations.[2] Lewis acids moreover promote CO insertion[3,4] to produce metal acyl–Lewis acid adducts. We now report that cationic coordinatively unsaturated Cp metal carbonyl complexes (Cp = η^5-C_5H_5) also induce methyl-CO insertion on a second metal center[5] and form an acetyl ligand bridging two metal centers. Although several bimetallic complexes bearing μ-acyl ligands are known,[6] their syntheses entailed neither starting with a mononuclear acyl complex nor Lewis acid facilitation of the CO insertion step. We accordingly found it expeditious to first demonstrate that bimetallic μ-[η^1-C,O]-acetyl compounds can be obtained from mononuclear acetyl complexes.

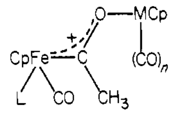
Neutral acetyl complexes **1a,b** and **2** coordinate the appropriate Cp metal carbonyl Lewis acid **3–5** (eq 1 and 2) by generating the bimetallic μ-acetyl adducts **6–9**. Labile isobutylene[7a] or tetrahydrofuran[7b] complexes of CpFe(CO)$_2^+$$PF_6^-$ (**3**) metalated **1a,b** in refluxing CH_2Cl_2 (1–6 h), whereas CpM(CO)$_3$$FPF_5$, a source of CpM(CO)$_3^+$ [**4**, M = Mo; **5**, M = W],[8] consumed **1a,b** and **2** at ~−20 °C (0.5 h) in CH_2Cl_2. All reactions afforded air-stable red powders **6–9** (50–85% yields) after reprecipitating from CH_2Cl_2–ether.[9] Although **6–9** remained intact in CH_2Cl_2 or CH_3NO_2 solution, acetone degraded these μ-acetyl adducts to starting acetyl complexes and acetone solvates of **3–5**. A similar degradative procedure serves as a convenient assay procedure for all μ-[η^1-C,O]-acetyl complexes reported herein: 1 equiv of (n-Bu)$_4$N$^+$I$^-$ in CH_2Cl_2 immediately and quantitatively (via IR and NMR monitoring) reverts them to the starting acetyl complex and CpM(CO)$_n$I.



1a, L = CO
b, L = PPh$_3$

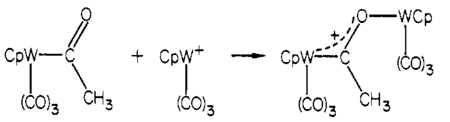
3

4, M = Mo
5, M = W



(1)

6a, L = CO; M = Fe (n = 2)
b, L = PPh$_3$
7a, L = CO; M = Mo (n = 3)
b, L = PPh$_3$
8, L = CO; M = W (n = 3)



(2)

2 **5** **9**

Bimetallic μ-acetyl compounds **6–9**, formulated as carboxonium salts, entail η^1 metal–O bonding that resembles CpFe(CO)$_2^+$ complexation of organic ketones.[10] The carboxonium formulation derives from the substantial delocalization of positive charge from the activating metal M to the Fe in **6–8**. IR [ν(C≡O), CH_2Cl_2] and ^{1}H NMR (Cp in ppm, acetone-d_6) data of CpFe(CO)PPh_3 in **1b** (1910 cm^{-1}, 4.43), **6b** (1941 cm^{-1}, 4.65), and CpFe(CO)PPh_3[C(OCH_3)CH_3]$^+$$PF_6^-$ (1990 cm^{-1}, 5.13) accordingly are consonant with an electronic environment of the Fe in **6b** that is intermediate to the starting acetyl complex **1b** and the methoxyethylidene salt. NMR spectra of **6b** and **7b** additionally support the η^1 bonding of the acetyl complex to an activating metal **3** or **4**, since diastereomeric mixtures were not detected for **6b** or **7b**.[11]

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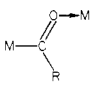
(2) Parshall, G. W. "Homogeneous Catalysis"; Wiley: New York, 1980; *Adv. Organomet. Chem.* **1979**, *17*. Eisenberg, R.; Hendrickson, D. E. *Adv. Catal.* **1979**, *28*, 79.

(3) (a) Butts, S. D.; Richmond, T. G.; Shriver, D. F. *Inorg. Chem.* **1981**, *20*, 278. Butts, S. B.; Strauss, S. H.; Holt, E. M.; Stimson, R. E.; Alcock, N. W.; Shriver, D. F. *J. Am. Chem. Soc.* **1980**, *102*, 5093. (b) Berke, H.; Hoffmann, R. *Ibid.* **1978**, *100*, 7224. (c) Collman, J. P.; Finke, R.; Cawse, J. N.; Brauman, J. I. *Ibid.* **1978**, *100*, 4766. (d) Nitay, M.; Priester, W.; Rosenblum, M. *Ibid.* **1978**, *100*, 3620.

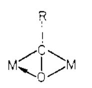
(4) Some electrophilic reagents(e.g., Ag^+) spur migratory insertion by oxidizing alkymetal carbonyl complexes. The resulting cation radical then undergoes rapid alkyl-CO migration and subsequent degradative steps: Magnuson, R. H.; Zulu, S.; Tsai, W.-M.; Giering, W. P. *J. Am. Chem. Soc.* **1980**, *102*, 6887. Johnson, M. D. *Acc. Chem. Res.* **1978**, *11*, 57 and references cited.

(5) We differentiate bimetallic reactions that incur an electron-rich metal center (i.e., a Lewis base) inducing alkyl-CO migratory insertion on a second metal center, concomitant with metal–metal bond formation: (a) Casey, C. P.; Cyre, C. R.; Anderson, R. L.; Marten, D. F. *J. Am. Chem. Soc.* **1975**, *97*, 3053. (b) Collman, J. P.; Rothrock, R. K.; Finke, R. G.; Rose-Munch, F. *Ibid.* **1977**, *99*, 7381. (c) Reference 3d.

(6) Two types of structures have been established for bimetallic structures containing μ-acyl (including formyl) ligands:



μ-[η^1-C,O][6a–f]



μ-[η^2-C,O][6g]

(a) Fischer, E. O.; Kiener, V.; Bunbury, D. St. P.; Frank, E.; Lindley, P. F.; Mills, O. S. *Chem. Commun.* **1968**, 1378. Lindley, P. F.; Mills, O. S. *J. Chem. Soc. A* **1969**, 1279. Fischer, E. O.; Kiener, V. *J. Organomet. Chem.* **1970**, *23*, 215; **1972**, *42*, 447. (b) Blickensderfer, J. R.; Kaesz, H. D. *J. Am. Chem. Soc.* **1975**, *97*, 2681. Blickensderfer, J. R.; Knobler, C. B.; Kaesz, H. D. *Ibid.* **1975**, *97*, 2686. (c) Merlino, S.; Montagnoli, G.; Braca, S.; Sbrana, G. *Inorg. Chim. Acta* **1978**, *27*, 233. (d) Wolczanski, P. T.; Threlkel, R. S.; Bercaw, J. E. *J. Am. Chem. Soc.* **1979**, *101*, 218. Threlkel, R. S.; Bercaw, J. E. *Ibid.* **1981**, *103*, 2650. (e) Longato, J.; Norton, J. R.; Huffman, J. C.; Marsella, J. A.; Caulton, K. G. *Ibid.* **1981**, *103*, 209. (f) Lukehart, C. M. *Acc. Chem. Res.* **1981**, *14*, 109. (g) Belmonte, P.; Schrock, R. R.; Churchill, M. R.; Youngs, W. J. *J. Am. Chem. Soc.* **1980**, *102*, 2858. Churchill, M. R.; Wasserman, H. J. *J. Chem. Soc., Chem. Commun.* **1981**, 274.

(7) (a) Giering, W. P.; Rosenblum, M. *Chem. Commun.* **1971**, 441. (b) Reger, D. L.; Coleman, C. *J. Organomet. Chem.* **1977**, *131*, 153.

(8) Beck, W.; Schloter, K. *Z. Naturforsch., B* **1978**, *33B*, 1214. Sünkel, K.; Ernst, H.; Beck, W. *Ibid.* **1981**, *36B*, 474.

(9) All new compounds gave satisfactory C, H elemental analyses and gave IR and ^{1}H and ^{13}C NMR data in accord with the proposed structures.

(10) (a) Foxman, B.; Klemarczyk, P. T.; Liptrot, R. E.; Rosenblum, M. *J. Organomet. Chem.* **1980**, *187*, 253. (b) Schmidt, E. K. G.; Thiel, C. H. *Ibid.* **1981**, *209*, 373.

(11) This conclusion is prediced upon η^2 complexation of a prochiral acetyl complex creating a chiral center. Diastereomeric mixtures would then result because of the second chiral Fe center within the CpFe(CO)PPh_3 group on **6b** and **7b**. For example, η^2 complexation of prochiral propene[12a] or 1-butene[12b] to CpFe(CO)PPh_3^+ gave diastereomeric mixtures that were easily discerned by NMR analysis. We also observed only a single resonance doublet (δ 1.03, J = 6.0 Hz in CD_2Cl_2) in the ^{1}H NMR spectrum of Cp(CO)$_2$Fe{C[OMo(CO)$_3$Cp]CH(CH_3)$_2$}$^+$$PF_6^-$; whereas η^2 coordination of the isobutyryl complex would render the *gem*-methyl groups diastereotopic.[10a]

(12) (a) Aris, K. R.; Brown, J. M. *J. Chem. Soc., Dalton Trans.* **1974**, 2222. (b) Reger, D. L.; Coleman, C. J.; McElligott, P. J. *J. Organomet. Chem.* **1979**, *177*, 73.

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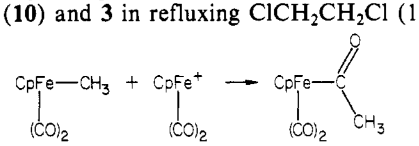
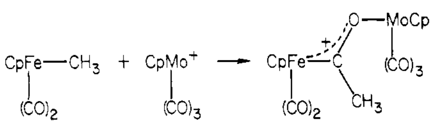
The organometallic Lewis acids **3**–**5** also promote CO insertion on their methyl complexes. An equimolar mixture of $CpFe(CO)_2CH_3$ (**10**) and **3** in refluxing $ClCH_2CH_2Cl$ (1 h) (eq 3)


(3)

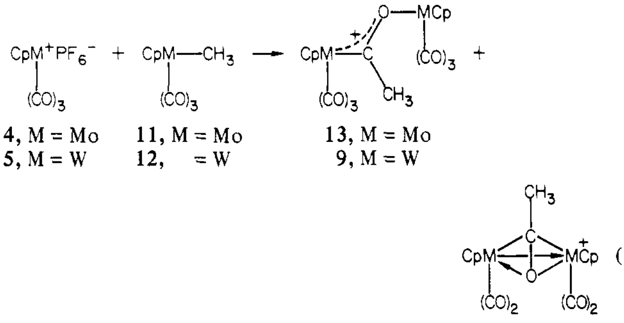
10 **3** **1a**

accordingly gave a green suspension containing insoluble $CpFe(CO)_3{}^+PF_6{}^-$ (58% yield) and the acetyl (**1a**)[13] and methyl (**10**) complexes (41% and 38% yields, respectively) after ether extraction and chromatography as the only organometallic products. Although the putative μ-acetyl compound **6a** was not detected, its facile decomposition to **1a** under these reaction conditions was independently established. The extra CO required for converting **10a** to **1a** could derive from disproportionation of the unsaturated intermediate $Cp(CO)Fe\{CO[Fe(CO)_2Cp]CH_3\}^+$ to **6a**.[14] Milder reaction conditions engendered in using the Mo Lewis acid **4** (eq 4) (~−20 °C), however, permitted isolation of bimetallic μ-acetyl compounds from the Lewis acid promoted CO insertion on a methyl complex. Thus **10** and **4** (−20 to +20 °C) gave **7a** in 42% yield. Further evidence for intermediacy of μ-acetyl complexes during organometallic Lewis acid induced CO insertions came from scrutiny of the reactions between **4** or **5** and their methyl complexes.


(4)

10 **4** **7a**

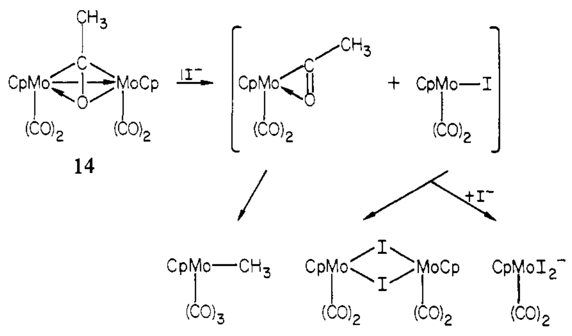
$CpM(CO)_3{}^+$ salts **4** and **5** convert their methyl complexes $CpM(CO)_3CH_3$ (**11**, M = Mo; **12**, M = W) into parallel mixtures of μ-acetyl compounds (eq 5). Mo complexes **4** and **11** at −20 °C (0.5 h) afforded a 1:1 mixture of **13** and **14**, which precipitated from ether (25 °C) as a pink solid. Structural assignment of **13**, although not obtained analytically pure, follows from consideration of IR and NMR spectra[15] and from results of the aforementioned I^- assay procedure, which cleaved **13** into $CpMo(CO)_3COCH_3$[3a] and $CpMo(CO)_3I$. Treatment of the **13**–**14** mixture with acetone decomposed **13**, but ether precipitation left analytically pure **14** in 36–56% yields. Our assignment of **14** as a symmetrical μ-[η^2-C,O]-acetyl complex rests on its spectral properties:[16] IR [(CH_3NO_2) ν(C≡O)] 2000–1910 (br) cm^{-1}; ^1H NMR (200 MHz, unchanged at −40 °C) (CD_3NO_2) δ 6.00 (s, 10, Cp), 3.00 (s, 3, CH_3); ^{13}C NMR (CH_3NO_2) δ 218.0, 216.8 (C≡O, 1:1 intensity), 92.5 (s, Cp), 20.6 (s, CH_3). Compounds **13** and **14** evidently result from separate pathways; they were not interconverted in refluxing in CH_2Cl_2, with or without 1 atm of CO. Tungsten complexes **5** and **12** reacted analogously, but **9** and **15** were not separated. Results of I^- degradative and $P(OMe)_3$ derivatization studies on **14** also support its μ-[η^2-C,O]-acetyl assignment.


(5)

4, M = Mo **11**, M = Mo **13**, M = Mo
5, M = W **12**, = W **9**, M = W

14, M = Mo
15, M = W

One equivalent of I^- in CH_2Cl_2 degraded **14** to the methyl complex **11** (88% after chromatography) and $[CpMo(CO)_2I]_2$[17a] (**17**) (eq 6) (80% by quantitative IR: ν(CO) 1961, 1877 cm^{-1}).

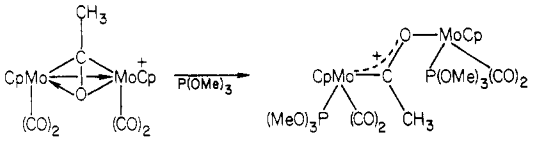

(6)

14

11 **17**

The η^1-acetyl complex $CpMo(CO)_3COCH_3$ was not detected, but variable amounts (combined yields less than 20%) of $CpMo(CO)_3I$ [IR: ν(CO) 2038, 1963 cm^{-1}] and $CpMo(CO)_2I_2{}^-$[17b] [IR: ν(CO) 1939, 1843 cm^{-1}] were present. Two equivalents of I^-, however, quantitatively converted **14** within 10 min into **11** and $CpMo(CO)_2I_2{}^-$. No gas was evolved, as ascertained by gasimetric analysis, during either I^- reaction. Dimeric **17** also consumed 2 equiv of I^- under similar reaction conditions to give $2CpMo(CO)_2I_2{}^-$, but the reaction progressed only 80% after 1 h. Taken together, these observations are consistent with I^- cleavage of **14** to the mononuclear complexes $CpMo(CO)_2(\eta^2\text{-}COCH_3)$ and $CpMo(CO)_2I$; the former rearranges to **11** and the latter either dimerizes to **17**, traps I^- (giving $CpMo(CO)_2I_2{}^-$), or decomposes to $CpMo(CO)_3I$.

Excess $P(OMe)_3$ in CH_2Cl_2 readily derivatized **14** (eq 7) and left the bimetallic unit intact as the μ-[η^1-C,O]-acetyl complex **18a**,[9] obtained in 71% yield as orange crystals after precipitation in ether. This reaction furthermore stereoselectively produced *trans*- and *cis*-**18a** on the Mo–C (acetyl) and Mo–O (acetyl) centers, respectively. Independent preparation of **18a** (65% yield) proved possible by CO substitution on **19**,[9] through cis labilization[18] on the (acetyl) O–Mo center, with excess $P(OMe)_3$ (eq 8).


(7)

14 **18a**, trans, cis

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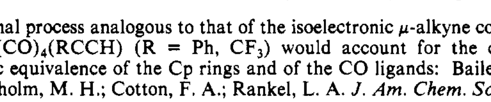
[13] (a) Thermal CO insertion into **10**, giving **1a**, normally requires very high pressures: King, R. B.; King, A. D. Jr.; Iqbal, M. Z.; Frazier, C. C. *J. Am. Chem. Soc.* **1978**, *100*, 1687. (b) $CpFe(CO)_2COCH_3$ (**1a**) also resists thermal decarbonylation to **10**: King, R. B. *Ibid.* **1963**, *85*, 1918.

[14] Similar disproportionation of **3** to $CpFe(CO)_3{}^+$ is documented.[7a] Presence of excess **10**, **3a**, $Fe(CO)_5$, or $[CpFe(CO)_2]_2$ during the reaction of **10a** and **3** did not improve yields of **1a**.

[15] **9**: IR (CH_2Cl_2) 2056, 2045, 1967 (br) cm^{-1}; ^1H NMR (CD_3NO_2) δ 6.32 (s, 5, CpWO), 5.90 (s, 5, CpWC), 2.68 (s, 3, Cp); ^{13}C NMR (CH_3NO_2) δ 91.6 and 90.3 (CpW), 221.3 and 218.5 (1:2 intensity), 215.3 and 213.1 (1:2) (C≡O), 293.0 (W–C acetyl), 52.0 (CH_3). **13**: IR (CH_2Cl_2) 2061, 2048, 1971 (br) cm^{-1}; ^1H NMR (CD_3NO_2) δ 6.22 (s, 5, CpMoO), 5.79 (s, 5, MoC), 2.75 (s, 3, CH_3); ^{13}C NMR (CH_3NO_2) δ 93.7, 91.7 (CpMo), 228.4 and 225.2 (1:2 intensity), 222.8 and 220.8 (1:2) (C≡O), 49.3 (CH_3).

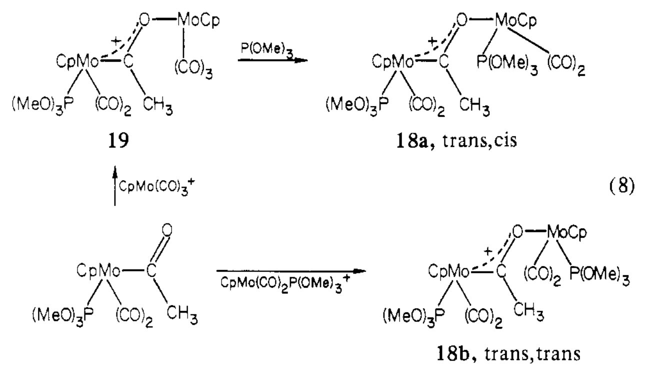
[16] Symmetrical μ-[η^2-C,O]acetyl complexes **14** and **15**, 36-electron structures, are adequately described by the ensemble of resonance forms also including



A fluxional process analogous to that of the isoelectronic μ-alkyne complexes $Cp_2Mo_2(CO)_4(RCCH)$ (R = Ph, CF_3) would account for the observed magnetic equivalence of the Cp rings and of the CO ligands: Bailey, W. I. Jr.; Chisholm, M. H.; Cotton, F. A.; Rankel, L. A. *J. Am. Chem. Soc.* **1978**, *100*, 5764.

[17] (a) Curtis, M. D.; Klingler, R. J. *J. Organomet. Chem.* **1978**, *161*, 23. (b) Burkett, A. R.; Meyer, T. J.; Whitten, D. G. *Ibid.* **1974**, *67*, 67.

[18] Atwood, J. D.; Brown, T. L. *J. Am. Chem. Soc.* **1976**, *98*, 3160.



19

18a, trans,cis

(8)

18b, trans,trans

Treatment of $CpMo(CO)_2[P(OMe)_3]COCH_3$ with $CpMo(CO)_2P(OMe)_3^+$ at −20 °C stereoselectively furnished *trans*,*trans*-**18b**[9] (71% yield) as an orange solid after ether precipitation. Stereochemical assignments resulted from established IR and ^1H NMR correlations;[19] spectroscopically distinctive **18a** and **18b** evidently do not interconvert at room temperature. Results of I^- cleavage reactions further corroborated stereochemical assignments: **18a** gave *cis*-$CpMo(CO)_2P(OMe)_3I$ and **18b** formed *trans*-$CpMo(CO)_2P(OMe)_3I$, in addition to *trans*-$CpMo(CO)_2P(OMe)_3(COCH_3)$, as the initial products.

Acknowledgment. Support from the Department of Energy, Office of Basic Energy Sciences, is gratefully acknowledged.

Registry No. **1a**, 12108-22-4; **1b**, 12101-02-9; **2**, 64666-36-0; **3**, 81141-37-9; **4**, 68868-80-4; **5**, 81141-36-8; **6a**, 81141-29-9; **6b**, 81132-99-2; **7a**, 81133-01-9; **7b**, 81133-03-1; **8**, 81133-05-3; **9**, 81133-07-5; **10**, 12080-06-7; **11**, 12082-25-6; **12**, 12082-27-8; **13**, 81133-09-7; **14**, 81133-11-1; **15**, 81133-13-3; **17**, 56731-33-0; **18a**, 81132-96-9; **18b**, 81177-17-5; **19**, 81141-27-7; $[CpFe(CO)_3]PF_6$, 38834-26-3; $CpMo(CO)_3I$, 12287-61-5; $CpMo(CO)_2I_2^-$, 52418-55-0; $CpMo(CO)_2(P(OMe)_3)COCH_3$, 12110-00-8; $[CpMo(CO)_2P(OMe)_3]PF_6$, 81141-35-7.

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[19] (a) Barnett, K. W.; Slocum, D. W. *J. Organomet. Chem.* **1972**, *44*, 1. Faller, J. W.; Anderson, A. S. *J. Am. Chem. Soc.* **1970**, *92*, 5852. Trans configuration of the Mo–C (acetyl) center is consistent with thermodynamic preference for trans orientation in analogous phosphine and phosphite-substituted acyl[20b] and cationic 2-oxacyclopentylidene complexes:[20c] (b) Craig, P. J.; Green, M. *J. Chem. Soc. A* **1969**, *157*; **1968**, 1978. Craig, P. J.; Edwards, J. *J. Organomet. Chem.* **1972**, *46*, 335. (c) Cotton, F. A.; Lukehart, C. *J. Am. Chem. Soc.* **1971**, *93*, 2672; **1973**, *95*, 3552.

Synthetic Approaches to Coordinatively Unsaturated Heterobimetallic Complexes

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Received October 15, 1981

The design and synthesis of coordination compounds that contain two different metal ions are priority goals of contemporary inorganic chemistry.[1] One of the most challenging objectives of such research is the preparation of coordinatively unsaturated heterobimetallic complexes.[2] With this in mind we have examined the coordination chemistry of the recently reported chelating agent [*o*-(diphenylphosphino)benzoyl]pinacolone (HacacP).[3] This compartmentalized ligand possesses electronically dissimilar metal binding sites that facilitate the assembly of a variety of novel compounds containing both "hard" and "soft" metals.[4]

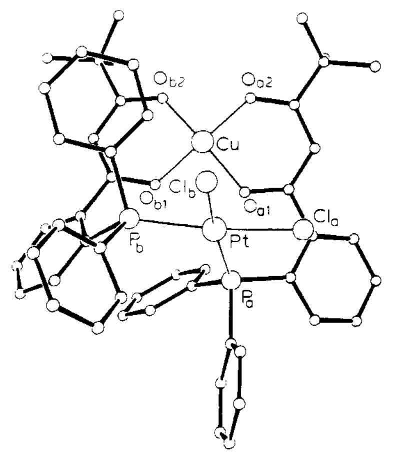
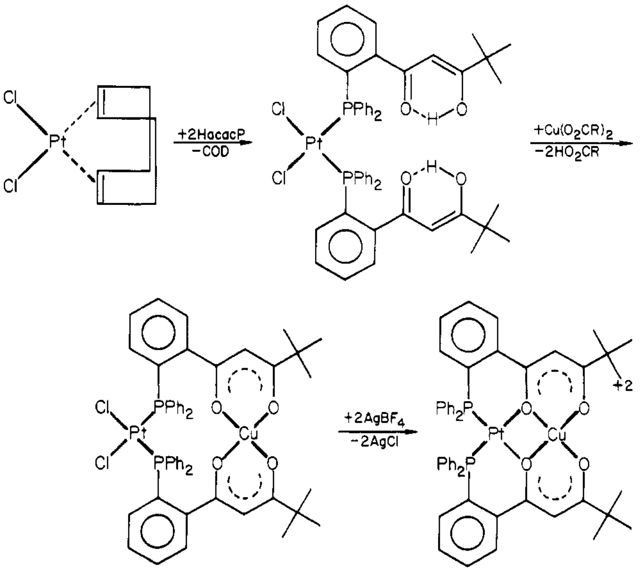


Figure 1. ORTEP plot for the nonhydrogen atoms of the $PtCl_2[Cu(acacP)_2]$ molecule. For purposes of clarity, metal atoms are represented by large open circles, chlorine and phosphorus atoms by medium-sized open circles, and carbon and oxygen atoms by small open circles.

Scheme I



Red, crystalline Ir(acacP)(COD)[5] (COD = 1,5-cyclooctadiene) was readily prepared from the reaction of KacacP (generated from HacacP and KO-*t*-Bu) with $[IrCl(COD)]_2$ in THF. Its ^{31}P NMR chemical shift of 24 ppm downfield from 85% H_3PO_4 and an intense IR band at 1665 cm^{-1} indicate that here the acacP moiety functions as a PO chelating agent with a pendant α,β-unsaturated ketone substituent. Mild (25 °C, 1 atm, 5 min) displacement of the COD with carbon monoxide resulted in an abrupt color change to yellow, a high-field shift in the ^{31}P NMR spectrum, and the disappearance of the 1665-cm^{-1} band in the IR spectrum. The CO stretching frequencies of the carbonylated Ir(acacP) derivative are virtually identical with those for $Ir(acac)(CO)_2$,[6,7] consistent with the binding of the $Ir(CO)_2$ moiety by the O⋯O site of the

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[5] All new compounds described in this paper analyze satisfactorily for the elements indicated. Anal. C, H; IR (mull) 1665 (s), 1610 (m) cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 8.0–7.0 (m, 14 H), 5.7 (d, 1 H), 3.7 (m, 4 H), 1.8 (m, 8 H), 0.8 (s, 9 H); $^{31}P\{^1H\}$ NMR (40.5 MHz, CD_2Cl_2) +24.0 ppm (downfield from 85% H_3PO_4); UV–vis (CH_2Cl_2) 555 nm (446 cm^{-1} M^{-1}), 472 nm (2530 cm^{-1} M^{-1}), 396 nm (1600 cm^{-1} M^{-1}).

[6] IR (mull) 2070 (vs), 1998 (vs) cm^{-1}; $^{31}P\{^1H\}$ NMR (40.5 MHz, CD_3Cl_2) −2.2 ppm; UV–vis (CH_2Cl_2) 408 nm (2260 cm^{-1} M^{-1}).

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