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Synthesis and Reactions of $CpFe(CO)_2(\eta^1\text{-}CH_2PEt_2)$ and $CpFe(CO)(\eta^2\text{-}CH_2PEt_2)$. Study of a Photochemically Induced η^1 to η^2 Conversion

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Summary: Displacement of chloride from $CpFe(CO)_2CH_2Cl$ by $HPEt_2$ followed by deprotonation yields the new η^1-phosphinomethyl complex $CpFe(CO)_2(\eta^1\text{-}CH_2PEt_2)$ (**2**). Suprisingly, thermolysis of this complex does not lead either to the CO insertion product acyl **7** or to the cyclic CO loss product $CpFe(CO)_1(\eta^2\text{-}CH_2PEt_2)$ (**5**). Coordination of the phosphorus to the iron atom occurs only upon photolysis to yield the η^2 species **5**, which spectroscopic data and molecular weight data confirm is a cyclic monomer. Thermolysis of this complex fails to yield characterizable product. Protonolysis of the Fe–C bond occurs with $HBF_4{\cdot}OMe_2$, yielding $CpFe(CO)_2PMeEt_2^+$.

Many examples of transition-metal complexes containing the CH_2PR_2 ligand have been reported in the literature. These complexes are typically prepared either through cyclometalation of a methylphosphine ligand[1,2] or reaction of $LiCH_2PR_2$ with a metal halide.[3-6] Reactions of geminal dihalides with metal phosphine dianions[7] or reactions of CH_2N_2 with phosphorus–metal double bonds[8] have also been used to obtain complexes of this type.[9] Except for a few zirconocene derivatives[4-6] and a chromium complex[10] containing the η^1 ligand, the CH_2PR_2 moiety is always bound in the η^2 fashion. Here we report a new, potentially general route to synthesis of the η^1-CH_2PR_2 moiety and the first observation of a controlled η^1-CH_2PR_2 to η^2-

(1) (a) Werner, H.; Gotzig, J. *Organometallics* **1983**, *2*, 547–549. Gotzig, J.; Werner, R.; Werner, H. *J. Organomet. Chem.* **1985**, *290*, 99–114. (b) Harris, T. V.; Rathke, J. W.; Muetterties, E. L. *J. Am. Chem. Soc.* **1978**, *100*, 6966–6977. Rathke, J. W.; Muetterties, E. L. *J. Am. Chem. Soc.* **1975**, *97*, 3272–3273. (c) Bresciani, N.; Calligaris, M.; Delise, P.; Nardin, G. Randaccio, L. *J. Am. Chem. Soc.* **1974**, *96*, 5642–5643. (d) Klein, H. F.; Wenninger, J.; Schubert, U. *Z. Naturforsch.* **1979**, *34B*, 1391-1397. (e) Gibson, V. C.; Grebenik, P. D.; Green, M. L. H. *J. Chem. Soc., Chem. Commun.* **1983**, 1101-1102. (f) Karsch, H. H. *Chem. Ber.* **1978**, *111*, 1650–1652. (g) Chiu, K. W.; Howard, C. G.; Rzepa, H. S.; Sheppard, R. N.; Wilkinson, G. *Polyhedron* **1982**, *1*, 441–451. (h) Karsch, H. H.; Klein, H.-F.; Schmidbaur, H. *Angew. Chem., Int. Ed. Engl.* **1975**, *14*, 637-638.

(2) Mainz, V. V.; Andersen, R. A. *Organometallics* **1984**, *3*, 675–678.

(3) (a) Karsch, H. H.; Schmidbaur, H. *Z. Naturforsch.* **1977**, *32B*, 762–767. (b) Schmidbaur, H.; Blaschke, G. *Z. Naturforsch.* **1979**, *35B*, 584–587. (c) Karsch, H. H. *Chem. Ber.* **1984**, *117*, 783–796. (d) Karsch, H. H.; Deubelly, B.; Hofmann, J.; Pieper, U.; Müller, G. *J. Am. Chem. Soc.* **1988**, *110*, 3654–3656.

(4) (a) Schore, N. E.; Hope, H. *J. Am. Chem. Soc.* **1980**, *102*, 4251–4253. (b) Schore, N. E.; Young, S. J.; Olmstead, M. M. *Organometallics* **1983**, *2*, 1767–1780.

(5) Engelhardt, L. M.; Jacobsen, G. E.; Raston, C. L.; White, A. H. *J. Am. Soc., Chem. Commun.* **1984**, 220–222.

(6) Karsch, H. H.; Müller, G.; Krüger, C. *J. Organomet. Chem.* **1984**, *273*, 195–212.

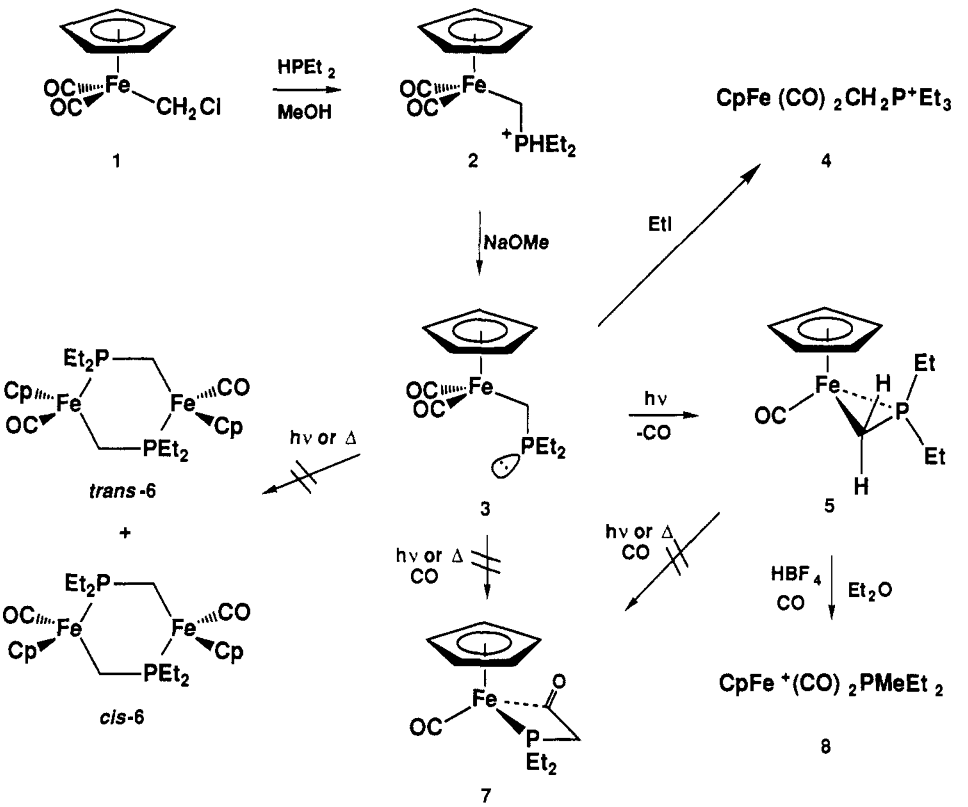
(7) (a) Lindner, E.; Starz, K. A.; Eberle, H.-J.; Hiller, W. *Chem. Ber.* **1983**, *116*, 1209–1218. (b) Lindner, E.; Goth, D. *J. Organomet. Chem.* **1987**, *319*, 149–160. (c) Lindner, E.; Stangle, M.; Hiller, W.; Fawzi, R. *Chem. Ber.* **1989**, *122*, 823-827.

(8) (a) Karsch, H. H.; Reisacher, H.-U.; Hüber, B.; Müller, G.; Malisch, W. *Angew. Chem., Int. Ed. Engl.* **1986**, *25*, 455–456. (b) Karsch, H. H.; Reisacher, H.-U.; Hüber, B.; Müller, G. *New. J. Chem.* **1989**, *13*, 319–327. (c) Karsch, H. H. *Chem. Ber.* **1984**, *117*, 3123–3133. (d) Gross, E.; Jorg, K.; Fiederling, K.; Gottlein, A.; Malisch, W.; Boese, R. *Angew. Chem., Int. Ed. Engl.* **1984**, *23*, 738–739.

(9) Other methods include the following. (a) Reductive coupling of $X\text{-}MCH_2PR_2\text{-}X$: Lindner, E.; Kütser, E.-U.; Hiller, W.; Fawzi, R. *Chem. Ber.* **1984**, *117*, 127–141. Lindner, E.; Neese, P.; Hiller, W.; Fawzi, R. *Organometallics* **1986**, *5*, 2030–2036. (b) Intramolecular trapping of a carbene by an adjacent phosphide ligand: Fryzuk, M. D.; Joshi, K. *J. Am. Chem. Soc.* **1989**, *111*, 4498–4500.

(10) Noh, S. K.; Heintz, R. A.; Theopold, K. H. *Organometallics* **1989**, *8*, 2071–2073.

Scheme I



CH_2PR_2 cyclization to form a new cyclometalated complex.[11]

Reaction of $CpFe(CO)_2CH_2Cl$[12] (1) with diethyl phosphine in methanol proceeds at reflux to give the chloride salt of $CpFe(CO)_2(CH_2PHEt_2)^+$ (2) as the major product in high yield (Scheme I).[13] Anion metathesis of **2** afforded bright yellow air-stable crystals of **2**-BPh_4.[14] Consistent with the expected[15] pK_a of the phosphonium compound **2**, treatment with NaOMe in methanol or methanol-d_4 solution gave clean deprotonation to yield the η^1-CH_2PEt_2 complex $CpFe(CO)_2(\eta^1$-$CH_2PEt_2)$ (**3**). This complex was formed in quantitative yield as determined by ^{1}H NMR spectroscopy and was isolated as a bright yellow oil[16] that quickly decomposed even at −30 °C. Spectroscopic data[17] conclusively show that this complex has the η^1-CH_2PEt_2 structure as shown in Scheme I. The solution IR spectrum clearly displays two strong bands in the ν_{CO} region at 2020

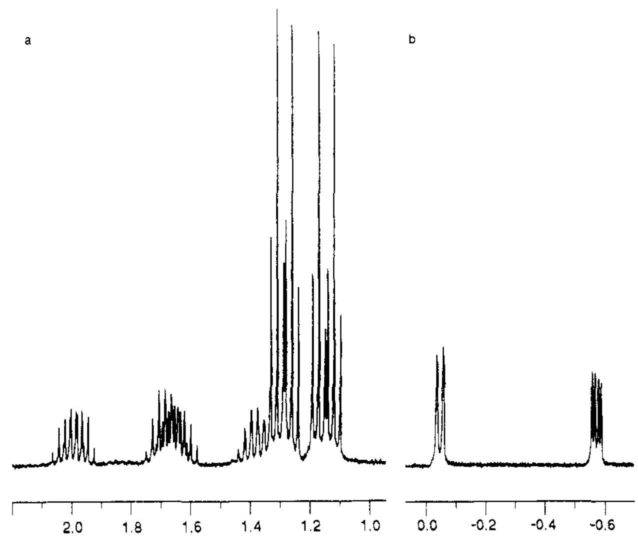


Figure 1. ^{1}H NMR spectrum (360 MHz) of **5** in methanol-d_4: (a) the two doublets of triplets due to diastereotopic methyl groups and four multiplets due to the diastereotopic ethyl group methylene protons (two methylene resonances are nearly coincidental at 1.65 ppm); (b) the ABX pattern assigned to the $FeCH_2P$ protons. For the peak at −0.048 ppm, J_{P-H} = 0.97 Hz. For the peak at 0.57 ppm, J_{P-H} = 3.3 Hz. For both peaks, J_{H-H} = 7.7 Hz.

(11) For related imine chemistry see: (a) Fong, C. W.; Wilkinson, G. *J. Chem. Soc., Dalton Trans.* **1975**, 1100–1104. (b) Sepelak, D. J.; Pierpont, C. G.; Barefield, E. K.; Budz, J. T.; Poffenberger, C. A. *J. Am. Chem . Soc.* **1976**, *98*, 6178–6185.

(12) Green, M. L. H.; Ishaq, M.; Whiteley, R. N. *J. Chem. Soc. A* **1967**, 1508–1515.

(13) Related quaternary (alkylphosphonium)- and (arylphosphonium)iron complexes have been reported previously: Pelling, S.; Botha, C.; Moss, J. R. *J. Chem. Soc., Dalton Trans.* **1983**, 1495–1501.

(14) Data for **2**-BPh_4: mp 147.1–148.6 °C; ^{1}H NMR (CD_2Cl_2) δ 7.39 (m, 8 H, BPh_4 ortho H), 7.07 (t, 8 H, BPh_4 meta H, J_{H-H} = 7.2 Hz), 6.93 (t, 4 H, BPh_4 para H, J_{H-H} = 7.2 Hz), 4.57 (d of m, 1 H, PH, J_{P-H} = 455 Hz), 4.73 (s, 5 H, C_5H_5), 1.66 (m, 4 H, CH_2CH_3), 1.04 (d of t, 6 H, CH_3, J_{P-H} = 19.4 Hz, J_{H-H} = 7.7 Hz), 0.18 (d of d, 2 H, $FeCH_2$, J_{P-H} = 12.4 Hz, J_{H-H} = 7.6 Hz); ^{31}P NMR (^{1}H coupled, CD_2Cl_2) δ 37.87 (d of m, J_{P-H} = 455 Hz); ^{13}C NMR (^{1}H coupled, CD_2Cl_2) δ 214.42 (s, CO), 164.30 (q, BPh_4, J_{B-C} = 49.4 Hz), 136.31 (d, BPh_4, J_{C-H} = 152 Hz), 126.14 (d, BPh_4, J_{C-H} = 155 Hz), 122.31 (d of t, BPh_4, J_{C-H} = 157 Hz, 7.7 Hz), 86.58 (d of t, C_5H_5, J_{C-H} = 182, 6.9 Hz), 14.53 (d of t, CH_2CH_3, J_{C-H} = 129 Hz, J_{P-C} = 46.8 Hz), 6.93 (d of q, CH_3, J_{C-H} = 134 Hz, J_{P-C} = 4.98 Hz), −30.82 (d of t, $FeCH_2$, J_{C-H} = 132 Hz, J_{P-C} = 32.65 Hz); IR (CH_2Cl_2) 2035, 1978 cm^{-1}. Anal. Calcd for $C_{36}H_{38}O_2BFeP$: C, 72.03; H, 6.38; P, 5.16. Found: C, 71.68; H, 6.40; P, 5.15.

(15) For a trialkylphosphonium salt, p$K_a \approx$ 8.4. See: Lowry, T. H.; Richardson, K. S. *Mechanism and Theory in Organic Chemistry*, 1st ed.; Harper and Row: New York, 1976.

(16) Attempts to prepare the η^1 $CpFe(CO)_2CH_2PMe_2$ derivative by reaction with $CpFe(CO)_2I$ and $LiCH_2PMe_2$ in either THF or diethyl ether failed to yield either the corresponding η^1 or η^2 complexes.

(17) Data for **3**: ^{1}H NMR (methanol-d_4) δ 4.89 (s, 5 H, C_5H_5), 1.42 (q, 4 H, CH_2CH_3, J_{H-H} = 7.8 Hz), 1.05 (d of t, 6 H, CH_3, J_{P-H} = 13.5 Hz, J_{H-H} = 7.9 Hz), 0.83 (d, 2 H, $FeCH_2$, J_{P-H} = 2.0 Hz); ^{31}P NMR (^{1}H coupled, methanol-d_4) δ −1.66 (septet, J_{P-H} = 13.3 Hz); ^{13}C (^{1}H coupled, methanol-d_4) δ 218.50 (s, CO), 87.18 (d of m, C_5H_5, J_{C-H} = 180 Hz), 23.02 (t of m, CH_2CH_3, J_{C-H} = 130 Hz, J_{P-C} = 12.5 Hz), 9.76 (q of m, CH_3, J_{C-H} = 126 Hz, J_{P-C} = 11.4 Hz), −7.88 (t of d, $FeCH_2$, J_{C-H} = 137 Hz, J_{P-C} = 35.8); IR (hexane) 2020, 1965 cm^{-1}; MS m/z (relative intensity) 252 (M^+ − CO, 14.0), 224 (M^+ − 2CO, 50.7), 194 (24.2), 166 (45.8), 121 ($CpFe^+$, 30.6).

and 1965 cm^{-1}, as expected for two noncollinear CO's. The ^{1}H NMR spectrum exhibits two equivalent ethyl group resonances and a single methylene resonance, consistent with a structure that allows free rotation about the Fe–C–P axis. A single resonance at −1.66 ppm is observed in the ^{31}P NMR spectrum, which is consistent with an uncoordinated trialkylphosphine. Although the η^1 species **3** was difficult to handle as a pure oil, derivatization with CH_3CH_2I yielded the stable phosphonium salt CpFe(CO)$_2$(CH$_2$PEt$_3$)$^+$ (**4**), which was easily characterized.[18]

Considering that closure of a three-membered ring might be a facile process, we expected that **3** might react either by loss of CO upon ring closure to yield the η^2 complex CpFe(CO)(η^2-CH$_2$PEt$_2$) (**5**) or by CO insertion induced by ring closure to yield the acyl compound **7**. However, heating a sealed tube of **3** in methanol-d_4 under vacuum at 50 °C in the dark produced no reaction over 3 h.[19] At higher temperatures, a number of unidentified products were formed, with no evidence of either η^2 **5** or acyl **7**.[20] In contrast, when η^1 **3** was photolyzed in methanol-d_4 with a tungsten filament lamp, gas was rapidly evolved (presumably CO) as the solution changed from yellow to orange. The ^{1}H NMR spectrum showed that quantitative conversion to a single compound had occurred. The alkyl region is shown in Figure 1. Most noticeable are the methylenic FeCH_2P protons, which appear as a clear ABX pattern between 0 and −0.6 ppm. The methyl groups appear as two doublets of triplets due to the coupling to the adjacent methylenes and the phosphorus atom. The complexity of the ^{1}H NMR spectrum was consistent with a cyclic structure which would result in six diastereotopic methylene and two diastereotopic methyl resonances in the alkyl region. The solution IR spectrum exhibited a single sharp band in the ν_{CO} region at 1920 cm^{-1}. The possibility that **3** dimerizes upon photolysis to form the cyclic dimer **6** is not consistent with the observations that only one geometric isomer of the product is formed and that the product has a molecular weight in agreement with the monomeric structure (isothermal M_r calcd 252, found 225, 265). The cyclic η^2 structure **5** is consistent with the IR, ^{1}H NMR, and molecular weight data, and thus we assign this structure to the photolysis product. The η^2 complex **5** was isolated as an air-sensitive amber oil that slowly decomposed at room temperature.[21,22]

The ^{31}P NMR spectrum displayed a new phosphorus resonance occurring at 17.5 ppm, which is only 20 ppm lower field than for the uncoordinated structure **3**. In contrast, in similar (C$_5$H$_4$R)Fe(CO)(PPh$_3$)R′ derivatives the ^{31}P coordination shift of PPh$_3$ is nearly 90 ppm.[23] Perhaps the relatively small coordination shift seen in η^2 **5** and in other η^2-CH$_2$PR$_2$ structures is characteristic of three-membered η^2-CH$_2$PR$_2$ structures.[1-3,7,8,9a] The ^{13}C NMR spectrum of η^2 **5** exhibits the phosphinomethylenic carbon resonance at −28.5 ppm with J_{C-H} = 155.8 Hz.

Although a small ring structure might suggest some strain in the molecule, the three-membered ring in **5** did not seem to be exceptionally prone to ring opening or ring expansion. When it was heated in a sealed tube under vacuum in methanol-d_4, **5** began to decompose at 50 °C to yield a number of unidentified products that appeared to be similar to the thermal decomposition products of η^1 **3**. Similar results were obtained when **5** was heated under 2 atm of CO. In neither case was there any evidence of CO insertion leading to acyl complex **7**[20] nor was there any ring opening to give the η^1-CH$_2$PEt$_2$ complex **3**.[24] The reluctance of the η^2-CH$_2$PR$_2$ ligand to undergo CO insertions has been discussed by other workers.[3c,7a,8c,9a] The ^{1}H NMR spectrum was static up to 100 °C, demonstrating no fluxionality in the three-membered ring up to this temperature.

When a Et$_2$O solution of η^2 complex **5** was treated with HBF$_4$·OMe$_2$ under an atmosphere of CO at 0 °C, the initially yellow solution immediately turned a deep green. The remaining yellow solid isolated after removal of solvent and trituration with CH$_2$Cl$_2$/hexane was identified as CpFe(CO)$_2$PEt$_2$Me$^+$ (**8**).[25] Thus, upon protonation of **5**, protonolysis of the Fe–C bond is preferred over protonolysis of the Fe–P bond.

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Registry No. **1**, 12107-38-9; **2**-BPh$_4$, 131276-15-8; **2**-Cl, 131276-22-7; **3**, 131276-14-7; **4**-BPh$_4$, 131276-17-0; **5**, 131276-18-1; **8**-BPh$_4$, 131276-20-5; HPEt$_2$, 627-49-6; HPPh$_2$, 829-85-6; CpFe(CO)(η^2-CH$_2$PPh$_2$), 131276-21-6.

(18) Data for **4**-BPh$_4$: ^{1}H NMR (CD$_2$Cl$_2$) δ 7.33 (m, 8 H, BPh$_4$ ortho H), 7.04 (t, 8 H, BPh$_4$ meta H, J_{H-H} = 7.3 Hz), 6.90 (t, 4 H, BPh$_4$ para H, J_{H-H} = 7.1 Hz), 4.87 (s, 5 H, C$_5$H$_5$), 1.75 (d of q, 6 H, CH$_2$CH$_3$, J_{P-H} = 11.8 Hz, J_{P-H} = 7.6 Hz), 1.09 (d of t, 6 H, CH$_3$, J_{P-H} = 17.3 Hz, J_{H-H} = 7.7 Hz), 0.45 (d, 2 H, FeCH$_2$, J_{P-H} = 12.6 Hz); ^{31}P{^{1}H} NMR (CD$_2$Cl$_2$) δ 52.56 (s); ^{13}C{^{1}H} NMR (CD$_2$Cl$_2$) δ 214.42 (s, CO), 164.30 (q, BPh$_4$, J_{B-C} = 49.4 Hz), 136.33 (s, BPh$_4$), 126.02 (s, BPh$_4$), 122.16 (s, BPh$_4$), 87.00 (s, C$_5$H$_5$), 15.58 (d, CH$_2$CH$_3$, J_{P-C} = 50.1 Hz), 6.15 (d, CH$_3$, J_{P-C} = 4.62 Hz), −28.74 (d, FeCH$_2$, J_{P-C} = 36.49 Hz); IR (CH$_2$Cl$_2$) 2035, 1980 cm^{-1}. Anal. Calcd for C$_{38}$H$_{42}$O$_2$BFeP: C, 72.64; H, 6.74. Found: C, 72.28; H, 6.70.

(19) The stability of **3** in methanol solution is surprising. The decomposition of the neat oil may be accelerated by autocatalytic processes, which may be quenched in methanol solution. The methanol may further serve to stabilize **3** through hydrogen bonding to the phosphorus lone pair. In agreement with these observations, solutions of **3** in non-H-bonding solvents such as hexane decompose within minutes at room temperature.

(20) The lack of an acyl stretch near 1600 cm^{-1} indicated that **7** had not been formed.

(21) Data for **5**: ^{1}H NMR (methanol-d_4) δ 4.45 (d, 5 H, C$_5$H$_5$, J_{P-H} = 1.14 Hz), 1.99, 1.67, 1.64, 1.36 (4 multiplets, each 1 H, ethyl CH$_2$), 1.14 (d of t, 3 H, CH$_3$, J_{P-H} = 17.8 Hz, J_{H-H} = 7.7 Hz), 1.28 (d of t, CH′$_3$, 3 H, J_{P-H} = 18.4 Hz, J_{H-H} = 7.6), −0.048 (d of d, 1 H, FeCH$_2$, J_{H-H} = 7.7 Hz, J_{P-H} = 0.96 Hz), −0.57 (d of d, 1 H, FeCH$_2$, J_{H-H} = 7.7 Hz, J_{P-H} = 3.3 Hz); ^{31}P NMR (^{1}H coupled, methanol-d_4) δ 17.50 (m); ^{13}C NMR (^{1}H coupled, methanol-d_4) δ 220.44 (d, CO, J_{P-C} = 24.6 Hz), 79.50 (d, C$_5$H$_5$, J_{C-H} = 180 Hz), 18.73 (d, *C*H$_2$CH$_3$, J_{P-C} = 18.2 Hz, J_{C-H} not resolved), 18.04 (d, *C*H$_2$CH$_3$, J_{P-C} = 24.6 Hz, J_{C-H} not resolved), 10.58 (q, CH$_3$, J_{C-H} = 132 Hz), 9.75 (q, CH$_3$, J_{C-H} = 128 Hz), −28.44 (t of m, FeCH$_2$, J_{C-H} = 156 Hz); IR (hexane) 1920 cm^{-1}; MS m/z (relative intensity) 252 (M$^+$, 42.4), 224 (M$^+$ − CO, 76.9), 194 (29.3), 166 (57.5), 121 (CpFe$^+$, 67.3).

(22) Although the ^{1}H NMR spectrum indicated that **5** was pure, we were unable to obtain a satisfactory elemental analysis for this compound. However, use of diphenylphosphine in place of diethylphosphine in Scheme I provided the crystalline diphenyl analogue of **5** CpFe(CO)(η^2-CH$_2$PPh$_2$), for which a satisfactory elemental analysis has been obtained and which has been fully characterized. Data for CpFe(CO)(η^2-CH$_2$PPh$_2$): ^{1}H NMR (CD$_2$Cl$_2$) δ 7.54 (m, 10 H, C$_6$H$_5$), 4.44 (d, 5 H, C$_5$H$_5$, J_{P-H} = 1.2 Hz), 0.75 (d, 1 H, FeCH$_2$, J_{H-H} = 7.9 Hz), 0.01 (d of d, 1 H, FeCH$_2$, J_{P-H} = 7.8 Hz, J_{P-H} = 2.5 Hz); ^{31}P NMR (^{1}H coupled, CD$_2$Cl$_2$) δ 11.93 (m); ^{13}C NMR (^{1}H coupled, CD$_2$Cl$_2$) δ 219.69 (d, CO, J_{P-C} = 23.7 Hz), 132.29 (m, C$_6$H$_5$), 79.58 (d, C$_5$H$_5$, J_{C-H} = 176.5 Hz), −27.49 (d of t, FeCH$_2$, J_{C-H} = 156.4 Hz, J_{P-H} = 10.1 Hz); IR (hexane) 1925 cm^{-1}. Anal. Calcd for C$_{19}$H$_{17}$OFeP: C, 65.55; H, 4.92. Found: C, 65.47; H, 4.90.

(23) Shade, J. E.; Wojcicki, A. *J. Organomet. Chem.* **1987**, *319*, 391–406.

(24) Preliminary evidence suggests that PMe$_3$ can affect η^2 to η^1 conversion of the CH$_2$PEt$_2$ ligand, yielding CpFe(CO)(PMe$_3$)(η^1-CH$_2$PEt$_2$); however, no CO insertion products were observed.

(25) Data for **8**-BPh$_4$: ^{1}H NMR (DMSO-d_6) δ 7.17 (m, 8 H, BPh$_4$ ortho H), 6.91 (t, 8 H, BPh$_4$ meta H, J_{H-H} = 7.3 Hz), 6.78 (t, 4 H, BPh$_4$ para H, J_{H-H} = 7.2 Hz), 5.63 (d, 5 H, C$_5$H$_5$, J_{P-H} = 1.52 Hz), 2.04 (m, 4 H, CH$_2$CH$_3$), 1.60 (d, 3 H, CH$_3$, J_{P-H} = 10.7 Hz), 1.08 (d of t, 6 H, CH$_3$, J_{P-H} = 18.4 Hz, J_{H-H} = 7.6 Hz); ^{31}P NMR (^{1}H coupled, DMSO-d_6) δ 54.53 (m); ^{13}C{^{1}H} NMR (DMSO-d_6) δ 210.42 (d, CO, J_{P-C} = 24.2 Hz), 163.31 (q, BPh$_4$, J_{B-C} = 49.8 Hz), 135.48 (s, BPh$_4$), 125.22 (s, BPh$_4$), 121.43 (d, BPh$_4$), 87.54 (s, C$_5$H$_5$), 23.32 (d, CH$_2$CH$_3$, J_{P-C} = 31.9 Hz), 13.26 (d, CH$_3$, J_{P-C} = 33.1 Hz), 7.61 (d, CH$_2$CH$_3$, J_{P-C} = 3.9 Hz); IR (CH$_2$Cl$_2$) 2040, 2000 cm^{-1}. Anal. Calcd for C$_{36}$H$_{38}$O$_2$BFeP: C, 72.03; H, 6.38. Found: C, 71.67; H, 6.49.