ternative disproportionation stoichiometry that gives [{Fp}$_2$] (eq 3) is more reasonable and has been reported for

$$2Na[Fp] + 2CO_2 \rightarrow [\{Fp\}_2] + CO + Na_2CO_3 \quad (3)$$

Na[Fp(CO$_2$)],[3] but we have not observed an analogous decomposition of Li[Fp(CO$_2$)] in our laboratory. Decomposition of Li[Fp(CO$_2$)] in THF becomes significant at ambient temperatures ($t_{1/2}$ = ca. 4 h), giving [{Fp}$_2$] contaminated by <10% [FpH] (IR). Excess CO$_2$ accelerates decomposition, although it is not consumed and does not affect the course of reaction (IR of solution and of the precipitate). A D$_2$O solution of the precipitate from the decomposition of Li[Fp(^{13}CO$_2$)] in the presence of excess ^{13}CO$_2$ initially contains (^{13}C NMR) a ca. 1:1:2 ratio of formate, carbonate, and a mixture of unidentified products (possibly iron carbonates) and hydrolyzes after 12 h to a ca. 1:3 mixture of HCO$_2^-$ and CO$_3^{2-}$. Quantification was complicated by the THF polymerization which invariably accompanied decomposition, but Ba^{2+} precipitation indicated an eventual CO$_3^{2-}$ yield of ca. 60%.

The most remarkable feature of the reaction is that only traces of free[21] CO are formed (0–10%; GC). The formation of labeled [{Fp}$_2$] from Na[Fp(^{13}CO$_2$)] has previously been taken to imply that the decomposition produces CO,[3] but oxide transfer from CO$_2$ to CO provides an alternative source of ^{13}CO, and we conclude that the published data do not establish reductive disproportionation of Na[Fp(CO$_2$)] and that reductive disproportionation provides at best a minor decomposition pathway for Li[Fp(CO$_2$)].

Formate ion could reasonably be formed in this decomposition by homolysis of the Fe–CO$_2$ bond in [Fp(CO$_2$)]$^-$ followed by reaction of CO$_2^{-}$·[22] with the solvent, but the observation of CO$_3^{2-}$ formation without CO evolution is less readily interpreted since something other than CO$_2$ must be reduced. The only obvious possibility is that there is an unidentified organic product derived from the solvent: this is not unreasonable, since the formation of HCO$_2^-$ and, more directly, the invariable THF polymerization observed suggest the intermediacy of solvent-derived radicals.

The experiments above lead to two general conclusions about the organometallic chemistry of CO$_2$. First, monoanionic CO$_2$ complexes can be sufficiently nucleophilic to transfer oxide from CO$_2$ to CO ligands, suggesting that oxide transfer is a general reaction of CO$_2$ complexes that can be reasonably described as metallocarboxylates; second, carbonate can be formed from CO$_2$ and transition-metal complexes by routes other than reductive disproportionation: only in cases in which both CO and CO$_3^{2-}$ formation have been established[7] can reductive disproportionation be regarded as proven.

Acknowledgment. We thank the Office of Naval Research (Contract No. N00014-83-K-0292) and the donors of the Petroleum Research Fund, administered by the American Chemical Society, for financial support of this research. We thank John M. Maher for preliminary experiments.

Registry No. Li[Fp(CO$_2$)], 33361-37-4; NBu$_4$[Fp(CO$_2$)], 95156-00-6; K[Fp(CO$_2$)], 95156-01-7; Na[Fp(CO$_2$)], 95156-02-8; Li[Fp], 33361-37-4; NBu$_4$[Fp], 65836-70-6; [FpCH$_3$], 12080-06-7; [{Fp}$_2$], 12154-95-9; [FpH], 35913-82-7; K[Fp], 60039-75-0; HCO$_2^-$, 71-47-6; CO$_3^{2-}$, 3812-32-6; CO$_2$, 124-38-9; CH$_3$OSO$_2$F, 421-20-5; O$_2$, 7782-44-7.

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[21] We also see no IR evidence for the formation of [Fe(CO)$_5$] or other potential CO sinks.

[22] This could also give rise to the small amount of CO observed.

Homo- and Heterobimetallic Complexes Connected by Peralkylated Cyclopentadienyl Rings Having a Two-Carbon Bridge between Them

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Summary: The reaction between the tungstenacyclobutadiene complex W(C$_3$Et$_3$)(OCMe$_2$CMe$_2$O)(OCMe$_3$) and 3,7-decadiyne yields [WO$_2$(OCMe$_3$)]$_2$(η^5,η^5-Et$_4$C$_5$CH$_2$CH$_2$C$_5$Et$_4$) (**1**, η^5,η^5-Et$_4$C$_5$CH$_2$CH$_2$C$_5$Et$_4$ = Cp′CH$_2$CH$_2$Cp′). **1** was was treated with HCl and Me$_3$SiCl to give [W(O)Cl$_3$]$_2$(Cp′CH$_2$CH$_2$Cp′) (**2**). Treatment of **2** with PCl$_5$ gave (WCl$_4$)$_2$(Cp′CH$_2$CH$_2$Cp′) (**3**). An X-ray structural study of a PMe$_3$ adduct of **3**, [(PMe$_3$)Cl$_4$W]$_2$(Cp′CH$_2$CH$_2$Cp′) (**4**), confirmed the presence of the Cp′CH$_2$CH$_2$Cp′ ring system. **4** crystallizes in the space group $p\bar{1}$ with a = 9.562 (4) Å, b = 10.690 (8) Å, c = 14.007 (13) Å, α = 110.10 (6)°, β = 104.55 (5)°, γ = 95.14 (5)°, and Z = 1. A total of 4458 reflections were collected in the range 3° < 2θ < 50° with the 3371 having $F_o > 4\sigma(F_o)$ being used for refinement by full-matrix least-squares techniques to a final R_1 = 0.046 and R_2 = 0.050. HC$_5$Me$_4$CH$_2$CH$_2$C≡CEt was prepared by addition of LiCH$_2$CH$_2$C≡CEt to tetramethylcyclopentenone. LiC$_5$Me$_4$CH$_2$CH$_2$C≡CEt was added to [Rh(CO)$_2$Cl]$_2$ to give Rh(η^5-C$_5$Me$_4$CH$_2$CH$_2$C≡CEt)(CO)$_2$ which was subsequently added to W(C$_3$Et$_3$)(OCMe$_2$CMe$_2$O)(OMe$_3$) to give [(Me$_3$CO)O$_2$W](η^5,η^5-Et$_4$C$_5$CH$_2$CH$_2$C$_5$Me$_4$)[Rh(CO)$_2$].

Approaches to the reduction of carbon monoxide employing complexes in which donor ligands link two metal centers, so far, for the most part, have not anticipated the fact that CO will likely displace donor ligands at the temperatures (≳150 °C) and pressures (≳100 atm of CO/H$_2$) typically necessary to reduce CO. Bimetallic complexes containing linked cyclopentadienyl rings,[1] on the other hand, may be likely to survive such relatively severe reaction conditions. A molecular model of Ta$_2$(η^5-C$_5$Me$_4$Et)$_2$Cl$_3$(CH$_3$)H$_2$,[2] a relative of compounds of the type [Ta(η^5-C$_5$Me$_4$R)Cl$_2$H]$_2$ which will react with CO to give [Ta(η^5-C$_5$Me$_4$R)Cl$_2$]$_2$(μ-CHO)(μ-H) complexes,[3] suggested to us that a two-carbon link between peralkylated rings would distort this bimetallic complex little. We report here some potentially general approaches to bimetallic complexes containing two-carbon-linked peralkylated cyclopentadienyl ring systems in which at least one of the metals is tungsten.

3,7-Decadiyne reacts smoothly with a tungstenacyclobutadiene complex as shown in eq 1.[4,5] The most im-

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[1] Many bimetallic complexes containing cyclopentadienyl rings linked by one or two (or more) units (usually CR$_2$ or SiR$_2$) have been prepared. To our knowledge, however, they all contain only one linking unit (often SiMe$_2$), or unalkylated rings, or both.

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[3] Belmonte, P. A.; Cloke, F. G. N.; Schrock, R. R. *J. Am. Chem. Soc.* **1983**, *105*, 2643.

[4] (a) The formation of cyclopentadienyl rings from tungstenacyclobutadiene complexes and acetylenes has already been disclosed.[4b,c] (b) Pedersen, S. F.; Schrock, R. R.; Churchill, M. R.; Wasserman, H. J. *J. Am. Chem. Soc.* **1982**, *104*, 6808. (c) Schrock, R. R.; Pedersen, S. F.; Churchill, M. R.; Ziller, J. W. *Organometallics* **1984**, *3*, 1574.

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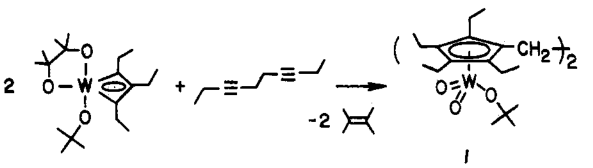

(1)

portant feature of the ^{13}C NMR spectrum of $[WO_2(OCMe_3)]_2(Cp'CH_2CH_2Cp')$ (1) is a 2:2:1 set of singlets (Figure 1a) characteristic of the ring carbon atoms in a η^5-C_5R_4R' cyclopentadienyl complex (cf. 124.7, 122.3, and 119.7 ppm for the cyclopentadienyl ring carbon atoms in $W[\eta^5\text{-}C_5Me_4(CMe_3)](OCMe_3)O_2$[4c]). We were not able to obtain crystals suitable for an X-ray study that would prove the presence of the expected linked cyclopentadienyl system. A suitable derivative (4) was finally obtained by the series of reactions shown in eq 2–4.[6] An X-ray diffraction study[9] confirmed that 4 contained the two-carbon-linked cyclopentadienyl ring system (Figure 2).

$$1 \xrightarrow[\text{Me}_3\text{SiCl}]{\text{HCl}} \underset{2}{[W(O)Cl_3]_2(Cp'CH_2CH_2Cp')} \qquad (2)^{7}$$

$$2 \xrightarrow{\text{PCl}_5} \underset{3}{[WCl_4]_2(Cp'CH_2CH_2Cp')} \qquad (3)^{8a}$$

$$3 \xrightarrow{\text{PMe}_3} \underset{4}{[WCl_4(PMe_3)]_2(Cp'CH_2CH_2Cp')} \qquad (4)^{8b}$$

The key to the successful preparation of bimetallic species containing tungsten and a group 8 or 9[15] metal (preferably Ru or Rh) was the synthesis of HC_5Me_4R where R = $CH_2CH_2C{\equiv}CEt$. That was accomplished by treating $LiCH_2CH_2C{\equiv}CEt$[10] with tetramethylcyclopentenone[11] in ether at −78 °C followed by an aqueous acid workup and isolation of the product by distillation in vacuo.[12] Deprotonation of HC_5Me_4R with butyllithium in ether at −10 °C gave a THF-soluble salt whose ^{13}C NMR spectrum shows (inter alia) a three-signal pattern for the cyclopentadienyl ring carbon atoms (Figure 1b).

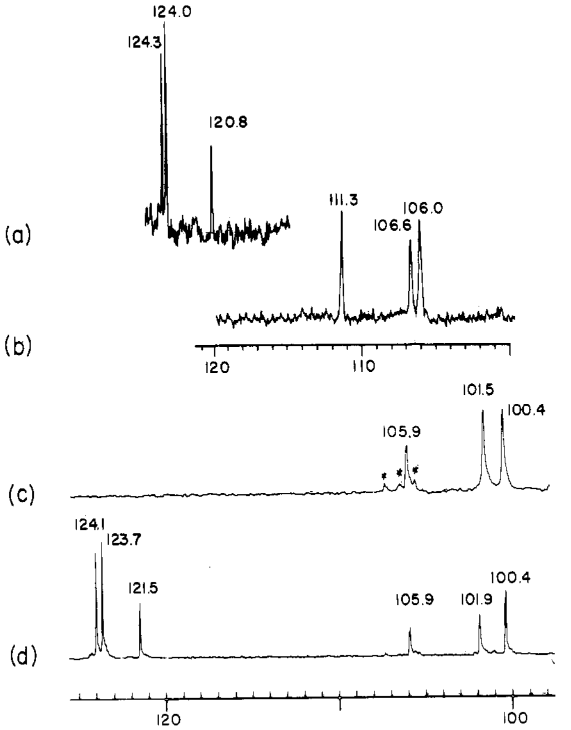

Figure 1. The cyclopentadienyl ring carbon atom signals in ^{13}C NMR spectrum of (a) 1 in C_6D_6; (b) $LiC_5Me_4(CH_2CH_2C{\equiv}CEt)$ in THF-d_8; (c) 5 in C_6D_6 (asterisk, unidentified impurity); (d) 6 in C_6D_6. Coupling to ^{103}Rh (~6 Hz) can just be resolved in most of the signals in c and d.

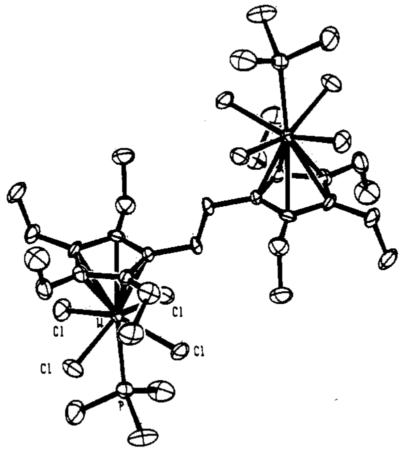

Figure 2. Geometry of $[WCl_4(PMe_3)]_2(Cp'CH_2CH_2Cp')\cdot CH_2Cl_2$ showing thermal ellipsoids at the 40% probability level. Hydrogen atoms and the CH_2Cl_2 molecule are not shown.

The reaction between $[Rh(CO)_2Cl]_2$ and LiC_5Me_4R (R = $CH_2CH_2C{\equiv}CEt$) in THF at −30 °C proceeds smoothly to give a high yield of $Rh(\eta^5\text{-}C_5Me_4R)(CO)_2$ (5) as a red-brown oil. The ^{13}C NMR of 5 shows a 1:2:2 pattern of barely resolved doublets ($J_{CRh} \approx 6$ Hz) in the region expected for cyclopentadienyl carbon atoms (Figure 1c). Addition of 5 to $W(C_3Et_3)(OCMe_2CMe_2O)(OCMe_3)$ gave 6 (eq 5) in high yield.[14] Unfortunately 6 is also an oil. Its

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(5) 3,7-Decadiyne (0.56 g, 4.2 mmol) was added to a solution of $W(C_3Et_3)(OCMe_2CMe_2O)(OCMe_3)$[4c] (4.12 g, 8.3 mmol) in 2 mL of pentane at −30 °C, and the reaction was warmed to room temperature. Over a period of 1 week a total of 2.51 g of 1 (63%) was filtered off. It may be recrystallized from toluene. Anal. Calcd for $WC_{18}H_{31}O_3$: C, 45.11; H, 6.52. Found: C, 44.90; H, 6.41.

(6) The reactions shown in eq 2–4 were developed first in the analogous monomeric system starting with $W(\eta^5\text{-}C_5Et_5)O_2(OCMe_3)$. Details will be reported separately in due course.

(7) A 1.0-g sample of 1 in 10 mL of toluene at 0 °C was treated with 1.0 mL of Me_3SiCl followed by 280 mL of gaseous HCl. After 15 min, 0.93 g of yellow $[WOCl_3]_2(Cp'CH_2CH_2Cp')$ was filtered off. It was recrystallized from dichloromethane. Anal. Calcd for $WC_{14}H_{22}OCl_3$: C, 33.87; H, 4.47; Cl, 21.42. Found: C, 33.84; H, 4.18; Cl, 20.75.

(8) (a) Solid PCl_5 (0.28 g, 1.36 mmol) was added all at once to 0.68 g of $[WOCl_3]_2(Cp'CH_2CH_2Cp')$ dissolved in 10 mL of dichloromethane. After 2 h 0.70 g of copper-colored, impure $[WCl_4]_2(Cp'CH_2CH_2Cp')$ (approximately a quantitative yield) was filtered off and rinsed with dichloromethane and ether. (We believe the pure product should be bright orange, cf. $W(\eta^5\text{-}C_5Me_5)Cl_4$.[8c]) (b) The impure $[WCl_4]_2(Cp'CH_2CH_2Cp')$ was suspended in 10 mL of dichloromethane. It dissolved upon adding 4 equiv of PMe_3 to give a very dark green/red dichroic solution. Two crops (0.69 g total) of dark green crystals of $[WCl_4(PMe_3)]_2(Cp'CH_2CH_2Cp')$ were isolated upon concentration and cooling the solution to −30 °C. (c) Murray, R. C.; Blum, L.; Liu, A.; Schrock, R. R. *Organometallics*, in press.

(9) A crystal obtained from dichloromethane was coated with epoxy resin. Data were collected on an Enraf-Nonius CAD4F-11 diffractometer using Mo Kα radiation. A total of 4458 reflections ($+h,\pm k,\pm l$) were collected in the range $3° < 2\theta < 50°$ with the 3371 having $F_o > 4\sigma(F_o)$ being used in the structure refinement by full-matrix least-squares techniques (243 variables) using SHELX-76. Final $R_1 = 0.046$ and $R_2 = 0.050$. Data were corrected for a 12% isotropic decay of the three standards. Full details can be found in the supplementary material.

(10) (a) $LiCH_2CH_2C{\equiv}CEt$ was prepared from $ICH_2CH_2C{\equiv}CEt$[10b] by transmetalation with *tert*-butyllithium.[10c] (b) Ansell, M. F.; Emmett, J. C.; Coombs, R. V. *J. Chem. Soc. C* **1968**, 217. (c) Volkmann, R. A.; Andrews, G. C.; Johnson, W. S. *J. Am. Chem. Soc.* **1975**, *97*, 4777.

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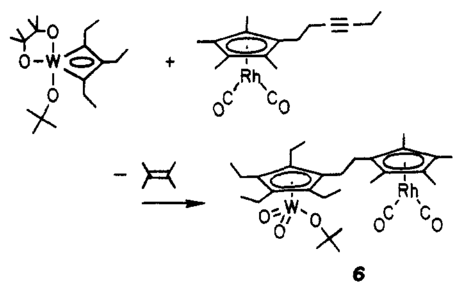
(12) Yield 73%; bp 80–82 °C at 1 mm. The ^{13}C NMR spectrum showed a total of six acetylenic carbon resonances, ten vinyl carbon resonances, and three aliphatic cyclopentadiene carbon resonances, as expected for a mixture of the three possible isomers.

(13) ν_{CO} = 2025 (s) and 1967 (s) cm^{-1} in pentane. No other carbonyl-containing species were observed by IR. ^{1}H and ^{13}C NMR spectra showed only ~5% of some as yet unidentified impurity (see Figure 1c).

(14) $W(C_3Et_3)(OCMe_2CMe_2O)(OCMe_3)$ (0.65 g, 1.33 mmol) reacted with $Rh(\eta^5\text{-}C_5Me_4CH_2CH_2C{\equiv}CEt)(CO)_2$ (0.48 g, 1.33 mmol) in 1 mL of pentane completely in 2 days at 25 °C. All ^{1}H, ^{13}C, and IR data are consistent with the expected product shown in eq 5.



(5)

6

^{13}C NMR spectrum in the cyclopentadienyl carbon signal region shows signals for one η^5-C_5R_4R'-type ring bound to W and one η^5-C_5R_4R'-type ring bound to Rh (Figure 1d). The highest mass pattern in the FD mass spectrum of 6 is found around 744, the mass expected for 6 (^{184}W) minus one carbonyl ligand. The IR spectrum of 6 is a composite of that of 1 and that of 5, suggesting that there is little or no interaction between the two ends of the molecule.

No peralkylated cyclopentadienyl system linked by two carbon atoms is known. The compounds reported here, especially heterobimetallic 6, are not likely to be preparable by traditional techniques. We will be attempting to prepare other heterobimetallic linked cyclopentadienyl systems via approaches analogous to that shown in eq 5 and will be exploring the fundamental chemistry of such species.

Acknowledgment. This work has been supported by the Director, Office of Basic Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division, of the U.S. Department of Energy under Contract DE-AC02-78ER04949-A003. We also thank the Biomedical Research Support Shared Instrumentation Grant Program, Division of Research Resources, for funds to purchase the X-ray diffraction equipment under NIH Grant S10RR02243-01 and the Canadian government for an NSERC postdoctoral fellowship to S.A.M.

Supplementary Material Available: Description of crystallography, crystal data, final positional and thermal parameters, selected interatomic distances, atom labeling scheme, and final observed and calculated structure factors (20 pages). Ordering information is given on any current masthead page.

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[15] The group notation is being changed in accord with recent actions by IUPAC and ACS nomenclature committees. A and B notation is being eliminated because of wide confusion. Group I becomes groups 1 and 11, group II becomes groups 2 and 12, group III becomes groups 3 and 13, etc.

Preparation, Structure, and Reactivity of $[Fe\{CH(PPh_2)_3\}(C_5H_5)][PF_6]$, a Monometallic, η^3 Complex of Tris(diphenylphosphino)methane

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Summary: Ultraviolet irradiation of $[Fe(p\text{-xylene})(Cp)][PF_6]$ and tris(diphenylphosphino)methane (tppm) gives $[Fe\{HC(PPh_2)_3\}(Cp)][PF_6]$ in 96% yield. Crystal structure analysis (R = 6.2%) shows that all three phosphorus atoms are coordinated to the iron atom, providing the first example of η^3 coordination of tppm on a single metal. There is considerable strain in the tricyclic system, with all three P–C–P angles less than 90°. One Fe–P bond is easily opened by refluxing acetonitrile, giving $[Fe(MeCN)(\eta^2\text{-tppm})(Cp)][PF_6]$.

Tris(diphenylphosphino)methane (tppm)[1] has received considerable attention as a ligand for transition metals.[2] In particular, tppm has been shown to promote "spontaneous self-assembly" of the metal cluster $[Ni_3(\mu_3\text{-tppm})(CO)_6]$ from $Ni(CO)_4$.[2a] In other studies, η^1, η^2, η^2-μ, and η^3-μ_3 coordination, and rearrangement, have been demonstrated,[2] but in no case has tppm been found with all three phosphorus atoms coordinated to a single transition-metal center. We report the first instance of tppm acting as a η^3 ligand.

Ultraviolet irradiation[3] of a mixture of $[Fe(p\text{-xylene})(Cp)][PF_6]$[4] (418 mg, 1.12 mmol) and tppm (671 mg, 1.18 mmol) in ~80 mL of dichloromethane for 8 h, followed by filtration, evaporation to ~10 mL in vacuo, and slow addition of ~15 mL of ethyl ether gives $[Fe\{HC(PPh_2)_3\}(Cp)][PF_6]$[4] (I, 903 mg, 96% yield) as orange crystals. Spectroscopic characterization[5] suggested that η^3 coordination of tppm had occurred, but the shift of the cyclopentadienyl protons in the ^{1}H NMR spectrum of I was δ 5.60, at anomalously low field. Ordinarily, phosphine substituents cause an upfield shift of the cyclopentadienyl resonance in $[FeL_3(Cp)]^+$: e.g., $[Fe(\eta^1\text{-dppm})(CO)_2(Cp)]^+$ (dppm = bis(diphenylphosphino)methane), δ 5.52,[6] $[Fe(\eta^2\text{-dppm})(CO)(Cp)]^+$, δ 5.25,[6] $[Fe(\eta^1\text{-dppm})(\eta^2\text{-dppm})(Cp)]^+$, δ 4.76,[7] $Fe\{\eta^3\text{-MeC}(CH_2PPh_2)_3\}(Cp)]^+$, δ 5.06,[8a] and $[Fe\{\eta^3\text{-PhP}(CH_2CH_2PPh_2)_2\}(Cp)]^+$, δ 4.23.[8b] In order to confirm η^3 coordination of tppm in I and to gain some insight into this NMR anomaly, a crystal structure analysis of I was carried out.[9]

Single crystals of I were obtained by slow evaporation of a dichloromethane solution of I with a stream of ethyl ether-saturated nitrogen. An ORTEP[10] plot of the resulting structure is shown in Figure 1, and selected bond lengths

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[5] ^{1}H NMR ($(CD_3)_2CO$, 90 MHz) δ 7.7–6.9 (30 H, m, Ph), (1 H, q, $^2J_{P-H}$ = ~5 Hz, CH), 5.60 (5 H, q, $^3J_{P-H}$ = 1.6 Hz, Cp); ^{31}P NMR ($(CD_3)_2CO$, 80.96 MHz) δ_P 30.05 (3 P, s, Fe–P), –142.21 (1 H, sept, J = 708 Hz, PF_6^-).

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