

Figure 1. Perspective drawing of the anion 7,7':8,8'-bis(di-thio)bis(7,8-dicarbaundecaborate(10)). Hydrogen atoms have been omitted for clarity. Some significant interatomic distances and angles are as follows: S1-S2', 2.061 (1) Å; S1-C7, 1.774 (3) Å; C7-C8, 1.594 (4) Å; C7-B11, 1.634 (5) Å; B10-B11, 1.838 (7) Å; S₁-S₂-C₈, 102.95 (11)°, C₈-C₇-B₁₁, 110.72 (26)°.

It is important to point out that partial degradation and protonation of both sulfurs has taken place in the step indicated in eq 2. The structure of compound 5 has been solved by single-crystal X-ray diffraction and is illustrated in Figure 1, which shows the molecular geometry and atom numbering convention. The crystals of 5 belong to the monoclinic system, space group $P2_1/a$, with unit cell dimensions $a = 13.318$ (4) Å, $b = 15.432$ (5) Å, $c = 7.084$ (2) Å, and $\beta = 100.45$ (2)°; $D_{\text{calcd}} = 1.186$ g cm⁻³ for $Z = 2$.⁸ In this space group this requires that the center of symmetry of each molecule reside at a special position. The molecule consists of two 7,8-dicarbaundecaborate(10) moieties bridged by two -S-S- units in such a way that there is an inversion center in the middle of the molecule. As a consequence the two cluster units are relatively placed in an anti fashion.⁹ The cations NMe₃H (not represented in Figure 1) are placed sideways to the anionic moiety, not above the pentagonal faces with the missing borons. Some selected distances are indicated in Figure 1. It is interesting to comment on the C-S bond lengths. A C-S distance of 1.774 (3) Å is found in 5, which is slightly shorter than the C-S bond length of 1.82 (4) Å reported for the disulfide [C₅H₉NMeHS]₂CuCl₄¹⁰ while being slightly longer than the C-S distance of 1.737 (5) Å in the copper thiolato complex [Cu(cyclam)(SC₅F₅)₂].¹¹ Accordingly, there is an apparent relationship between the carbon hybridization and the C-S distances, with C(sp²) < C(5) < C(sp³), which could imply a certain C=S double bond character in 5 through delocalization of the sulfur lone pair into the cluster open-face orbitals. The ¹¹B NMR spectrum at 115.5 MHz in the Me₂CO-*d*₆ shows six regions of resonance at -4.23 (d, 142), -9.35 (d, 168), -15.01 (d, 139), -17.14 (d, 146), -32.49 (d, 122), and -34.55 ppm (d, 125) with relative intensities 2:1:2:2:1:1. There is an additional splitting of each of the resonances at -32.49 ppm of 49 Hz attributed to bridge hydrogen.¹² All these data are consistent with the structure established by X-ray diffraction. The {¹H} ¹¹B NMR spectrum displays only five well-defined signals,

which indicate fairly clearly the isomeric purity of compound 5.

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Supplementary Material Available: Table of structure factors for 5 (9 pages). Ordering information is given on any current masthead page.

(12) (a) This resonance is attributed to B(10). (b) Siedle, A. R.; Bodner, G. M.; Todd, L. J. *J. Organomet. Chem.* 1971, 33, 137. (c) The ¹¹B NMR data for compound 4 in Me₂CO-*d*₆ is as follows: -5.90 (d, 140), -7.48 (d, 165), -16.20 (d, 142), -33.02 (d, 124), and -34.20 ppm (d, 127), with relative intensities 2:1:4:1:1. There is an additional splitting of each of the resonances at -33.02 ppm of 41 Hz which is attributed to bridge hydrogen.

(C₅H₅)₂Zr[Ru(CO)₂C₅H₅]₂. A Metal-Metal Bonded Zirconium-Diruthenium Complex

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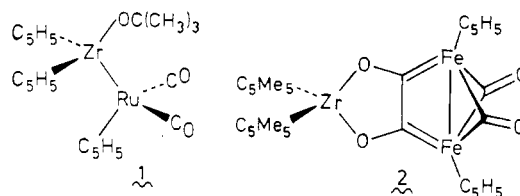
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Summary: K[(C₅H₅)Ru(CO)₂] reacts with (C₅H₅)₂ZrI₂ to give (C₅H₅)₂Zr[Ru(CO)₂(C₅H₅)]₂ (3). A single-crystal X-ray structural analysis of 3 establishes the metal-metal bonded nature of the complex. The space group is *Pbca*; the cell dimensions are $a = 15.000$ (4) Å, $b = 17.425$ (3) Å, $c = 16.957$ (4) Å, and $Z = 8$.

We have become interested in synthesizing heterobimetallic complexes in which an early transition metal is directly bonded to a late transition metal because we believe such compounds might be good precursors for new types of hydrogenation catalysts. Recently we reported the synthesis of a series of metal-metal bonded zirconium-iron and zirconium-ruthenium complexes such as (C₅H₅)₂[(CH₃)₃CO]Zr-Ru(CO)₂C₅H₅ (1) by reaction of



K[(C₅H₅)M(CO)₂] (M = Fe, Ru) with bis(cyclopentadienyl)zirconium chloride complexes.¹ We were interested to see if we could extend this method to the

(8) A crystal with approximate dimensions 0.21 × 0.19 × 0.18 mm was selected for X-ray analysis. Intensity measurements were carried out on a Syntex P2₁ automated four-circle diffractometer using graphite monochromatized Mo K α radiation. Reflections with 2θ up to 55° were collected by the θ - 2θ scan technique. A total of 3970 unique data was collected, of which 1935 had $I < 3\sigma(I)$. The structure was solved by direct methods using standard procedures (cf.: Hilty, T. K.; Thompson, D. A.; Butler, W. M.; Rudolph, R. W. *Inorg. Chem.* 1979, 18, 2642) to give an R_1 value of 0.047 and R_2 value of 0.053.

(9) A priori, it would have been expected that in order to minimize the electrical repulsions an anti geometry would be favored when the cages occupy nearby spatial positions, and a mixture of syn and anti isomers would appear when they were placed in noninteracting positions.

(10) Briansó, M. C.; Briansó, J. L.; Gaete, W.; Ros, J. *Inorg. Chim. Acta* 1981, 49, 263-267.

(11) Addison, A. W.; Sinn, E. *Inorg. Chem.* 1983, 22, 1225-1228.

(1) Casey, C. P.; Jordan, R. F.; Rheingold, A. L. *J. Am. Chem. Soc.* 1983, 105, 665 and references therein.

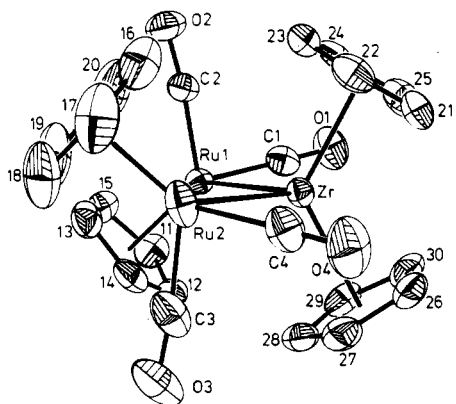
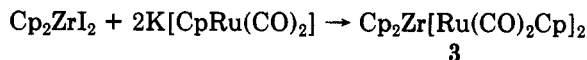


Figure 1. Molecular structure of $(\eta^5\text{-C}_5\text{H}_5)_2\text{Zr}[\text{Ru}(\text{CO})_2(\eta^5\text{-C}_5\text{H}_5)]_2$ (**3**). Bond lengths (Å) and angles (deg) of interest are as follows: Zr–Ru(1), 2.938 (1); Zr–Ru(2), 2.948 (1); Ru(1)–C(1), 1.850 (6); Ru(2)–C(2), 1.824 (6); C(1)–O(1), 1.146 (8); C(2)–O(2), 1.160 (7); Ru(1)–C(3), 1.942; Zr–C(1), 2.271, 2.273. CNT–Ru(1)–Zr, 123.7; CNT–Ru(1)–C(1), 131.7; CNT–Ru(1)–C(2), 126.4; C(1)–Ru(1)–C(2), 92.4(3); Zr–Ru(1)–C(1), 80.5 (2); Zr–Ru(1)–C(2), 87.1 (2); Ru(1)–Zr–Ru(2), 100.5 (1); CNT–Zr–CNT, 124.7; Ru(1)–C(1)–O(1), 177.4 (6); Ru(1)–C(2)–O(2), 171.9 (5). CNT indicates the centroid of a Cp ring.

synthesis of zirconium compounds with two late transition metals bonded to zirconium. This seemed particularly interesting since the formation of an alternate isomeric structure related to Bercaw's^{2,3} zirconium–diiron complex **2** seemed possible. Here we report the synthesis and crystal structure of the metal–metal bonded complex $(\text{C}_5\text{H}_5)_2\text{Zr}[\text{Ru}(\text{CO})_2(\text{C}_5\text{H}_5)]_2$ (**3**).

Reaction of Cp_2ZrI_2 ⁴ ($\text{Cp} = \eta^5\text{-C}_5\text{H}_5$) with 2 equiv of $\text{K}[\text{CpRu}(\text{CO})_2]$ for 1 h at 25 °C in THF gave $\text{Cp}_2\text{Zr}[\text{Ru}(\text{CO})_2\text{Cp}]_2$ (**3**) (90%), which was isolated as an orange crystalline solid by evaporation of solvent and recrystallization from toluene/hexane.^{5,6} A Zr–Ru bonded structure for **3** was first established by IR spectroscopy. The



ν_{CO} bands of **3** at 1934 (vs) and 1882 (vs) cm^{-1} are shifted 29 and 122 cm^{-1} to higher energy from the corresponding bands of $\text{K}[\text{CpRu}(\text{CO})_2]$,¹ as expected for a metal–metal bonded structure⁷ and are similar to those of **1**, which has been shown by X-ray crystallography to contain a Zr–Ru bond.¹ Low-energy, ν_{CO} bands indicative of Zr–CO bonding were not seen. The two Zr–Cp ligands are equivalent in both the room-temperature and low-temperature ¹H and ¹³C NMR spectra, as are the two Cp ligands on Ru. Two different carbonyl carbon signals are seen at δ 203.7 and 214.0 in the ¹³C NMR of **3** at –60 °C. Upon warming, these signals broaden at 1 °C, disappear at 34 °C, and reappear

(2) Berry, D. H.; Bercaw, J. E.; Jircitano, A. J.; Mertes, K. B. *J. Am. Chem. Soc.* **1982**, *104*, 4712.

(3) Compound **2** was prepared originally by reaction of $[(\text{C}_5\text{Me}_5)_2\text{Zr}(\text{N}_2)]_2$ with $[\text{CpFe}(\text{CO})_2]$ and more recently by reaction of $(\text{C}_5\text{Me}_5)_2\text{ZrI}_2$ with 2 equiv of $[\text{Na}[\text{Fe}(\text{CO})_2\text{Cp}]]$. The isostructural ruthenium compound $(\text{C}_5\text{Me}_5)_2\text{Zr}(\text{O}_2\text{C}_2\text{Ru})_2\text{Cp}_2$ has also been prepared (Berry, D. H.; Bercaw, J. E., personal communication).

(4) Druce, P. M.; Kingston, B. M.; Lappert, M. F.; Spalding, T. R.; Srivastava, R. C. *J. Chem. Soc.* **1969**, 2106.

(5) **3**: ¹H NMR (benzene-*d*₆) δ 6.21 (s, 10 H, Cp₂Zr), 4.64 (s, 10 H, CpRu); ¹³C{¹H}NMR (THF-*d*₆, –60 °C) δ 214.0 (s, CO), 203.7 (s, CO), 111.5 (s, Cp₂Zr), 88.0 (s, CpRu); ¹³C{¹H}NMR (THF-*d*₆, 50 °C) δ 207 (s, br, CO), 111.6 (s), 87.9 (s); IR (KBr) 1953 (vw), 1945 (vw), 1934 (vs), 1882 (vs) cm^{-1} ; Anal. Calcd: C, 43.30; H, 3.03. Found: C, 43.44; H, 3.31.

(6) Resonances assignable to the intermediate monosubstituted compound $\text{Cp}_2\text{Zr}(\text{I})\text{Ru}(\text{CO})_2\text{Cp}$ (δ 6.56 (s, 10 H), 4.98 (s, 5 H)) were observed when this reaction was followed by ¹H NMR in THF-*d*₆.

(7) Burlitch, J. M.; Leonowicz, M. E.; Petersen, R. B.; Hughes, R. E. *Inorg. Chem.* **1979**, *18*, 1097.

as a single broad peak at δ 207 at 51 °C. This temperature dependence is consistent with an exchange process with $\Delta G^\ddagger = 13.5 \pm 0.5 \text{ kcal mol}^{-1}$.

To confirm the presence of metal–metal bonding in **3** and to determine the origin of the dynamic behavior observed in its ¹³C spectrum, an X-ray diffraction study was performed⁸ (Figure 1). The two $\text{Ru}(\text{CO})_2\text{Cp}$ groups are linked to the Cp_2Zr group by metal–metal bonds (2.938 and 2.948 Å) and are related by an approximate two-fold rotational axis. Consistent with the low-temperature ¹³C spectrum there are two sets of two equivalent CO ligands: CO(1) and CO(4), which reside nearly in the plane defined by the ZrRu_2 framework (ZrRu₂ plane–C(1) distance = 0.345 Å; ZrRu₂ plane–O(1) distance = 0.565 Å), and CO(2) and CO(3), which are nearly perpendicular to this plane. The dynamic process that causes the COs to become equivalent most likely involves rotation about the Zr–Ru bonds that turns the Cp rings past one another. It is noteworthy that there is no significant interaction between Zr and the CO ligands (Zr–C(1) distance = 3.205 Å; Zr–O(1) distance = 3.875 Å).

The reaction of Cp_2ZrCl_2 with 2 equiv of $\text{K}[\text{Ru}(\text{CO})_2\text{Cp}]$ in THF at 25 °C also gave **3** but at a much slower rate and with significant amounts of $[\text{CpRu}(\text{CO})_2]$ byproduct.⁹ The reaction of Cp_2ZrI_2 with 2 equiv of $\text{K}[\text{CpFe}(\text{CO})_2]$ in THF at 25 °C gave $[\text{CpFe}(\text{CO})_2]$ as the sole Fe-containing product and a mixture of unidentified Zr products. When then reaction was followed by low-temperature ¹H NMR in THF-*d*₆, resonances assignable to a 100% yield of the monosubstituted product $\text{Cp}_2\text{Zr}(\text{I})[\text{Fe}(\text{CO})_2\text{Cp}]$ were observed at δ 6.66 (s, 10 H) and 4.63 (s, 5 H) after 1.5 h at –78 °C. When the reaction mixture was warmed to –20 °C for 15 min, resonances assignable to a 33% yield of the disubstitution product $\text{Cp}_2\text{Zr}[\text{Fe}(\text{CO})_2\text{Cp}]_2$ were observed at δ 6.41 (s, 10 H) and 4.72 (s, 10 H). However, both products decomposed rapidly when the reaction mixture was warmed above –20 °C.

The $\text{CpRu}(\text{CO})_2$ groups of **3** exchange with halide and $\text{CpM}(\text{CO})_2$ ligands of other Zr complexes. For example, reaction of **3** with Cp_2ZrCl_2 (THF, 25 °C, 24 h) gave $\text{Cp}_2\text{Zr}(\text{Cl})[\text{Ru}(\text{CO})_2\text{Cp}]$ in quantitative (NMR) yield. Similarly reaction of **3** with $\text{Cp}_2[(\text{CH}_3)_3\text{CO}]\text{Zr}-\text{Fe}(\text{CO})_2\text{Cp}^1$ (THF, 25 °C, 5 h) gave a quantitative (NMR) yield of $\text{Cp}_2[(\text{CH}_3)_3\text{CO}]\text{Zr}-\text{Ru}(\text{CO})_2\text{Cp}^1$ (**1**) as well as a mixture of products dominated by $[\text{CpFe}(\text{CO})_2]$ (possibly resulting from decomposition of $\text{Cp}_2\text{Zr}[\text{Ru}(\text{CO})_2\text{Cp}][\text{Fe}(\text{CO})_2\text{Cp}]$).

Bercaw has now synthesized $(\text{C}_5\text{Me}_5)_2\text{Zr}-\text{Ru}_2$ compounds with structures similar to **3**.³ The differences between our compounds with direct Zr–Ru bonds and Bercaw's compounds are probably due to some combination of the steric and electronic effects of the C_5Me_5 ligand. Steric hindrance might prevent bonding of ruthenium to the more crowded $(\text{C}_5\text{Me}_5)_2\text{Zr}$ center, and the better electron donor properties of the C_5Me_5 ligand might enhance the reducing power of the $(\text{C}_5\text{Me}_5)_2\text{Zr}$ unit and lead to products similar to **2** in which the carbonyl units are reductively coupled and bonded to zirconium through oxygen. Metal–metal bonded structures have now been found for a substantial series of compounds of the type $\text{Cp}_2\text{Zr}(\text{X})\text{Ru}(\text{CO})_2\text{Cp}$ (X = CH_3 , Cl, $\text{OC}(\text{CH}_3)_3$, $\text{Ru}(\text{CO})_2\text{Cp}$) as well as some Fe

(8) Suitable crystals of **3** were grown by slow cooling of a hot toluene solution. Crystallographic data: $a = 15.000$ (4) Å, $b = 17.425$ (3) Å, $c = 16.957$ (4) Å, $V = 4432$ (1) Å³, $Z = 8$ in space group *Pbca*; $R_F = 0.0404$, $R_{wF} = 0.0420$ for 3232 unique reflections ($I \geq 3\sigma(I)$) with anisotropic thermal parameters for all non-hydrogen atoms.

(9) In an NMR tube reaction the first substitution to give $\text{Cp}_2\text{Zr}(\text{Cl})\text{Ru}(\text{CO})_2\text{Cp}^1$ was complete within 15 min at 25 °C with no detectable $[\text{CpRu}(\text{CO})_2]$ formation. After 5 h the yield of disubstituted product **3** was 30% and that of $[\text{CpRu}(\text{CO})_2]$ was 10%.

analogues.¹ The insensitivity of these structures to the electronic properties of the ligand X (and hence the electronic properties of the Zr center¹⁰) suggests that the steric difference between the C₅Me₅ and C₅H₅ ligands is the major effect responsible for the remarkable structural difference between 2 and 3.¹²⁻¹⁴

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

Registry No. 1, 84303-39-9; 3, 88657-67-4; Cp₂ZrCl₂, 1291-32-3; K[CpFe(CO)₂], 60039-75-0; Cp₂Zr(I)[Fe(CO)₂Cp], 88657-68-5; Cp₂Zr[Fe(CO)₂Cp]₂, 88657-69-6; Cp₂Zr(Cl)[Ru(CO)₂Cp], 84303-40-2; Cp₂[(CH₃)₃CO]Zr-Fe(CO)₂Cp, 84303-42-4; Cp₂ZrI₂, 1298-41-5; K[CpRu(CO)₂], 84332-45-6; Cp₂Zr(I)Ru(CO)₂Cp, 88657-70-9.

Supplementary Material Available: Details of the X-ray structure solution, listings of fractional coordinates, thermal parameters, bond distance, bond angles, and structure factors (27 pages). Ordering information is given on any current masthead page.

(10) Marsella, J. A.; Moloy, K. G.; Caulton, K. G. *J. Organomet. Chem.* **1980**, *201*, 389.

(11) Skora, D. J.; Rausch, D. J.; Rogers, R. D.; Atwood, J. L. *J. Am. Chem. Soc.*, **1981**, *103*, 1265 and references therein.

(12) Martin and Moise have noted that the degree of substitution of the cyclopentadienyl rings Cp¹ strongly affects the nature of the V-CO complexes formed in the reaction of Cp¹₂V with Co₂(CO)₈.¹³ Vanadocenes with unsubstituted or monosubstituted Cp¹ rings gave as initial products V-CO complexes formulated as Cp¹₂V-Co(CO)₄ on the basis of their IR spectra. Vanadocenes with peralkylated Cp¹ rings gave as initial products species with low νCO's (1755 cm⁻¹) indicative of V-OC interactions, possibly of the type observed in (C₅Me₅)₂Yb(THF)OCCo(CO)₃.¹⁴

(13) Martin, J.; Moise, C. *J. Organomet. Chem.* **1982**, *232*, C55.

(14) Tilley, T. D.; Anderson, R. A. *J. Chem. Soc., Chem. Commun.* **1981**, 985.

Synthesis and Crystal Structure of *syn, syn*-Bis(η³-pentadienyl)bis(trimethylphosphine)iron

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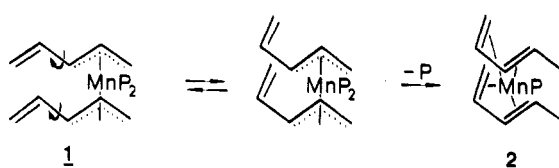
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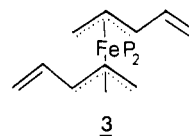
Summary: Reaction of FeCl₂[P(CH₃)₃]₂ with potassium pentadienide results in the formation of *syn, syn*-bis(η³-pentadienyl)bis(trimethylphosphine)iron (3). The crystal structure of 3, as determined by a single-crystal X-ray diffraction study, is reported. Unlike the analogous manganese system, coupling of the pentadienyl ligands is not observed. This stability with respect to coupling is thought to derive from the staggered orientation of the two pentadienyl ligands.

Recently, we reported the stereoselective coupling of two acyclic pentadienyl ligands on a manganese center and the isolation of a manganese-trimethylphosphine complex of the coupled product (2, Scheme I).¹ The stereochemistry of the coupled product, *trans, trans*-1,3,7,9-decatetraene, led us to postulate *syn, syn*-bis(η³-pentadienyl)bis(trimethylphosphine)manganese (1, Scheme I) as the critical intermediate. The two η³-pentadienyl ligands in 1 are

Scheme I



required to have the eclipsed orientation shown. We now report the isolation and full structural characterization of *syn, syn*-bis(η³-pentadienyl)bis(trimethylphosphine)iron (3),² the first structurally characterized mononuclear



syn-(η³-pentadienyl)metal complex.^{3,4} 3 differs from the postulated coupling intermediate in the manganese system (1) in that the two pentadienyl ligands have a staggered orientation. Apparently, the barrier associated with rearrangement from this staggered orientation to the required eclipsed one is sufficient to stabilize 3 with respect to pentadienyl ligand coupling.⁵

The reaction of FeCl₂[P(CH₃)₃]₂⁶ with potassium pentadienide⁷ in tetrahydrofuran at -78 °C, followed by warming to room temperature, produced red, pentane-soluble 3 in high yield.^{8,10} A single-crystal X-ray diffraction study of 3 was carried out.¹¹ An ORTEP drawing

(2) The simple allyl analogue of 3, bis(η³-allyl)bis(trimethylphosphine)iron, has not been reported.

(3) An unusual dinuclear complex, bis(pentadienyl)nickel, has W-shaped pentadienyl ligands which are coordinated to two nickel atoms: Rienacker, R.; Yoshiura, H. *Angew. Chem., Int. Ed. Engl.* **1969**, *8*, 677.

(4) We recently synthesized and carried out a single-crystal X-ray diffraction study of η³-2,4-dimethylpentadienyltris(trimethylphosphine)cobalt, the first example of a complex possessing an *anti*-η³-pentadienyl ligand: Bleeke, J. R.; Peng, W.-J., submitted for publication.

(5) 3 decomposes to iron metal and unidentified organic products upon heating for several hours at 50 °C in benzene.

(6) Harris, T. V.; Rathke, J. W.; Muettterties, E. L. *J. Am. Chem. Soc.* **1978**, *100*, 6966.

(7) Yasuda, H.; Ohnuma, Y.; Yamauchi, M.; Tani, H.; Nakamura, A. *Bull. Chem. Soc. Jpn.* **1979**, *52*, 2036.

(8) In a typical reaction, 1.89 g (1.5 × 10⁻² mol) of FeCl₂ and 2.28 g (3.0 × 10⁻² mol) of P(CH₃)₃ were refluxed in tetrahydrofuran for 1 h to form a pale green solution of FeCl₂[P(CH₃)₃]₂. This solution was added to 3.42 g (4.5 × 10⁻² mol) of P(CH₃)₃, and the mixture was cooled to -78 °C; 4.46 g (2.5 × 10⁻² mol) of potassium pentadienide-tetrahydrofuran (KC₅H₇-C₄H₉O) was then added over a period of 25 min, producing a dark reddish brown solution. This solution was stirred for 3 h while warming to room temperature, filtered through Celite, and evaporated to dryness. The dark red product, 3, was extracted with pentane and crystallized from pentane at -30 °C; yield (based on limiting reagent, KC₅H₇-C₄H₉O) 3.85 g (90%). Alternatively, 3 can be synthesized by adding P(CH₃)₃ to a solution of bis(pentadienyl)iron⁹ at -78 °C and warming to room temperature. In a typical reaction, 1.78 g (1.0 × 10⁻² mol) of KC₅H₇-C₄H₉O in 30 mL of tetrahydrofuran was added dropwise to a slurry of 0.75 g (5.92 × 10⁻³ mol) of FeCl₂ in 30 mL of tetrahydrofuran at -78 °C, producing a red solution. After stirring at -78 °C for 20 min, a solution of 0.80 g (1.0 × 10⁻² mol) of P(CH₃)₃ in 10 mL of tetrahydrofuran was added dropwise. The mixture was warmed to room temperature and stirred for 3 h. The resulting dark reddish brown solution was filtered through Celite and evaporated to dryness. 3 was extracted with pentane and crystallized from pentane at -30 °C; yield (based on limiting reagent, KC₅H₇-C₄H₉O) 1.2 g (70%).

(9) (a) The synthesis of bis(η⁵-pentadienyl)iron has been reported.^{9b} However, the species which is formed at -78 °C in our reaction system may be bis(η³-pentadienyl)iron. (b) Wilson, D. R.; Ernst, R. D.; Cymbaluk, T. H. *Organometallics* **1983**, *2*, 1220.

(10) ¹H NMR (22 °C, benzene-*d*₆, 100 MHz) δ 0.86-0.98 (m, 9, P-(CH₃)₃), 1.09-1.31 (m, 4, allylic H's), 5.02-5.23 (m, 3, vinylic H's); ¹H¹³C NMR (22 °C, benzene-*d*₆, 25.14 MHz) δ 145.08 (C₄), 106.82 (C₅), 86.85 (C₂), 55.69 (C₃), 36.38 (C₁), 18.60 (P(CH₃)₃); IR (toluene) 1607 (C=C stretch), 937, 949 cm⁻¹ (P-C stretch). Anal. Calcd for FeP₂C₁₆H₃₂: C, 56.14; H, 9.44. Found: C, 55.84; H, 9.24.

(1) Bleeke, J. R.; Kotyk, J. J. *Organometallics* **1983**, *2*, 1263.