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Iron(II) and Ruthenium(II) Complexes Containing Bidentate, Tied-Back Diphosphonite Ligands. X-ray Structure of *cis*-FeMe$_2$[(−OCH$_2$CEt$_2$CH$_2$O−)PCH$_2$CH$_2$P(−OCH$_2$CEt$_2$CH$_2$O−)]$_2$

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*Summary: trans-RuCl$_2$(PP)$_2$ (**2**) and cis-FeCl$_2$(PP)$_2$ (**4**) have been synthesized by the reaction of the diphosphonite 1,2-bis((2,2-diethyl-1,3-propanedioxy)phosphino)ethane (PP) with RuCl$_2$(Ph$_3$P)$_3$ and FeCl$_2$, respectively. Complex **4** was converted to cis-FeMe$_2$(PP)$_2$ (**5**), for which a molecular structure was obtained.*

Fe(II) and Ru(II) diphosphine complexes have been extensively studied for decades with various applications, including small-molecule activation.[1] However, we have been surprised to find no literature report on analogous Fe(II) or Ru(II) complexes containing diphosphonites. We have been interested in small-molecule activation on electrophilic transition-metal complexes with ligands that cannot offer intramolecular agostic C−H bond interaction.[2] Complexes of this nature can potentially coordinate extremely weak ligands such as alkanes, which otherwise cannot compete with entropically favored agostic interactions. The new diphosphonite 1,2-bis((2,2-diethyl-1,3-propanedioxy)phosphino)ethane ligand (**1**; denoted as PP) appears to be a good ligand for such Fe(II) and Ru(II) complexes because of (1) its lack of internal agostic interaction capability and (2) weaker electron-donating ability compared to diphosphine analogues. We report here on some Fe(II)/Ru(II) complexes containing ligand **1**.

As shown in Scheme 1, the ruthenium(II) dichloride **2** was synthesized by the reaction of RuCl$_2$(Ph$_3$P)$_3$ with **1,** which was synthesized in high yield by the reaction of Cl$_2$PCH$_2$CH$_2$PCl$_2$ with Et$_2$C(CH$_2$OH)$_2$ on the basis of the preparation of similar ligands.[3] The ^{31}P NMR chemical shifts of **1** and **2** are δ 174.0 and 203.8, respectively.

Reaction of **2** with AgPF$_6$ in CH$_2$Cl$_2$ apparently leads to removal of one chloride and formation of a white solid **3** that is not soluble in CH$_2$Cl$_2$ but is slightly soluble in more coordinating acetone (Scheme 1). Complex **3** shows one ^{31}P NMR signal at δ 202.2 in acetone-d_6, very close to the value of δ 203.8 for the neutral complex **2**. The ^{1}H NMR shows two closely spaced triplets near δ 0.9 (methyl protons of two sets of inequivalent Et groups which are integrated as 24H total), and five other multiplet signals integrated as 8H each for the five inequivalent types of methylene protons (including those for the two inequivalent Et groups). This is similar to the pattern for **2**, except the latter shows one signal (16H) at δ 4.03 instead of two for **3** at δ 3.99 and 4.27 (8H each). These signals apparently result from the methylene protons adjacent to oxygen on the diphosphonite ligand, which presumably become inequivalent upon removal of a chloride to form **3**. It is possible that the inequivalency results from one set of methylenes lying closer to the open coordination site than the other. Although the distances from the methylene C−H to the metal are much too long to be considered agostic, there may be a small perturbation leading to an NMR shift to lower field. Another possibility is that the “open” site may be weakly binding solvent or chloride from another molecule of **3**, as will be discussed below.

Solid **3**, which did not react with the tied-back phosphite P(OCH$_2$)$_3$CMe at room temperature, is likely to be a polymeric species bridged by chlorides to form chains, which may be at least partially broken on dissolving **3** in acetone. This is in contrast to analogous cationic diphosphine complexes such as [RuCl(dicyclohexylphosphinoethane)$_2$]$^+$ [4a] and [RuCl(diphenylphosphinoethane)$_2$]$^+$,[4b] which exist as discrete 16e five-coordinate molecules. The difference is possibly due to the fact that the Ru(II) cation in **3** is (1) more electrophilic (phosphonites are not as strongly donating as phosphines), (2) is less sterically crowded, and (3) has no internal agostic protection. All these factors encourage coordination to Ru(II) from the chloride of another Ru(II) molecule, and for steric reasons (bulky diphosphonites), the halide bridge may be linear, a rare but not unprecedented geometry.[5] Replacement of one or both chlorides in **2** with a group that does not contain lone pairs such as hydride or alkyl could lead to a 16e complex such as **3** that presumably would not oligomerize. However, reaction of the diphosphonite ligand **1**

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(1) For selected recent examples, see for Ru(II): (a) Schlaf, M.; Lough, A. J.; Morris, R. H. *Organometallics* **1997**, *16*, 1253. (b) Rocchini, E.; Mezzetti, A.; Ruegger, H.; Burckhardt, U.; Gramlich, V.; Del Zotto, A.; Martinuzzi, P. Rigo, P. *Inorg. Chem.* **1997**, *36*, 711. (c) Six, C.; Gabor, B.; Gorls, H.; Mynott, R.; Philipps, P.; Leitner, W. *Organometallics* **1999**, *18*, 3316. (d) Stoop, R. M.; Bauer, C.; Setz, P.; Worle, M.; Wong, T. Y. H.; Mezzetti, A.; *Organometallics* **1999**, *18*, 5691. (e) Martelletti, A.; Gramlich, V.; Zurcher, F.; Mezzetti, A. *New J. Chem.* **1999**, *23*, 199. For Fe(II): (f) Landau, S. E.; Morris, R. H.; Lough, A. J. *Inorg. Chem.* **1999**, *38*, 6060. (g) Bennett, M. A.; Ditzel, E. J.; Hunter, A. D.; Khan, K.; Kopp, M. R.; Neumann, H.; Robertson, G. B.; Zeh, H. *J. Chem. Soc., Dalton Trans.* **2000**, 1733.

(2) (a) Fang, X.-G.; Vincent, J. H.; Scott, B. L.; Kubas, G. J. *J. Organomet. Chem.* **2000**, *609*, 95. (b) Fang, X.-G.; Scott, B. L.; John, K. D.; Kubas, G. J. *Organometallics* **2000**, *19*, 4141.

(3) Squires, M. E.; Sardellas, D. J.; Kool, L. B. *Organometallics* **1994**, *13*, 2970.

(4) (a) Mezzetti, A.; Del Zotto, A.; Rigo, P.; Pahor, N. B. *J. Chem. Soc., Dalton Trans.* **1989**, 1045. (b) Chin, B.; Lough, A. J.; Morris, R. H.; Schweitzer, C. T.; D'Agostino, C. *Inorg. Chem.* **1994**, *33*, 6278.

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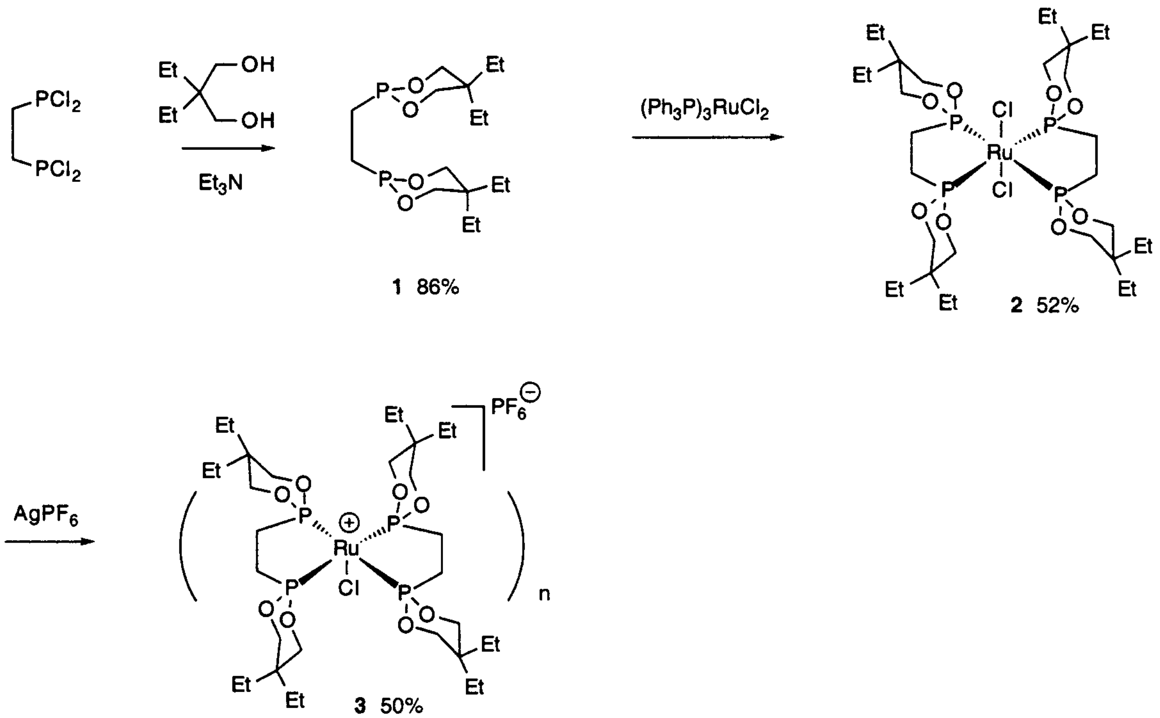
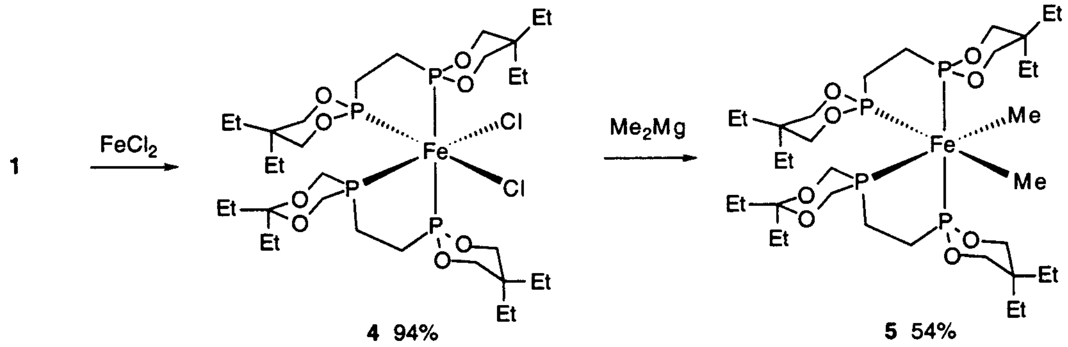
Scheme 1



Scheme 2



with $RuClH(Ph_3P)_3$ under conditions similar to those producing **2** led to intractable materials. Also, in attempts to synthesize an analogue of **3** with alkyl or aryl instead of Cl, neither MeLi, Me_2Mg, Me_2Zn, nor PhLi reacted with **2** in THF at room temperature for days. The lack of reactivity is possibly due to the repulsive interaction of the incoming nucleophile with oxygen lone pairs around the Ru(II). Surprisingly also, reaction of **2** with $AgPF_6$ under 1 atm of CO or in more strongly coordinating MeCN solvent did not lead to clean products with these ligands occupying the sixth coordination site. The elemental analysis for solid **3** was poor because of difficulty in purification (Ag compounds were possible contaminants) and/or partial occupation of the sixth coordination site of **3** by solvent or other molecules. However the ^1H, ^{31}P, and ^{13}C NMR spectra of **3** were clean and consistent with the proposed structure (see Supporting Information). Variation of the anion (e.g. use of SbF_6) and use of $TlPF_6$ instead of $AgPF_6$ did not improve the situation, and oligomeric species were still obtained. Nonetheless, there is potential for alkane binding on these types of systems by further ligand and coordination sphere design (solubility in hydrocarbons would be desirable).

Reaction of **1** with $FeCl_2$ in THF afforded the cis-configured diphosphonite $Fe^{II}Cl_2(PP)_2$ complex **4** (Scheme 2). The cis geometry is evidenced by the two triplet signals in the ^{31}P NMR spectrum at δ 227.7 and 241.0. The change of configuration from that of *trans*-**2** is likely due to the unfavorable steric interactions of two diphosphonite fragments around the smaller Fe(II). However it is noteworthy that most reported diphosphine Fe^{II}-Cl_2 complexes are trans-configured, with only very few being cis.[6] Reaction of **4** with $MgMe_2$ afforded the *cis*-dimethyl complex **5**. The cis geometry is again evidenced by the two triplet signals in its ^{31}P NMR spectrum at δ 232.6 and 238.9 with the coupling constant $J_{PH} = 46.2$ Hz. Complex **5** is stable in solution at room temperature, and no apparent change was observed after several days. This is in sharp contrast to analogous diphosphine

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(5) A linear Cu(μ-Br)Cu bridge has recently been found: Bowmaker, G. A.; Boyd, P. D. W.; Rickard, C. E. F.; Scudder, M. L.; Dance, I. G. *Inorg. Chem.* **1999**, *38*, 5476. We have prepared sterically more hindered *trans*-$RuCl_2(P'P')_2$ to attempt to discourage chloride coordination in the cation analogous to **3**, where $P'P' = (-OCMe_2CMe_2O-)$-$PCH_2CH_2P(-OCMe_2CMe_2O)$. However, removal of one chloride still generates a CH_2Cl_2-insoluble polymeric species.

(6) Field, L. D.; Thomas, I. P.; Hambley, T. W.; Turner, P. *Inorg. Chem.* **1998**, *37*, 612 and references therein.

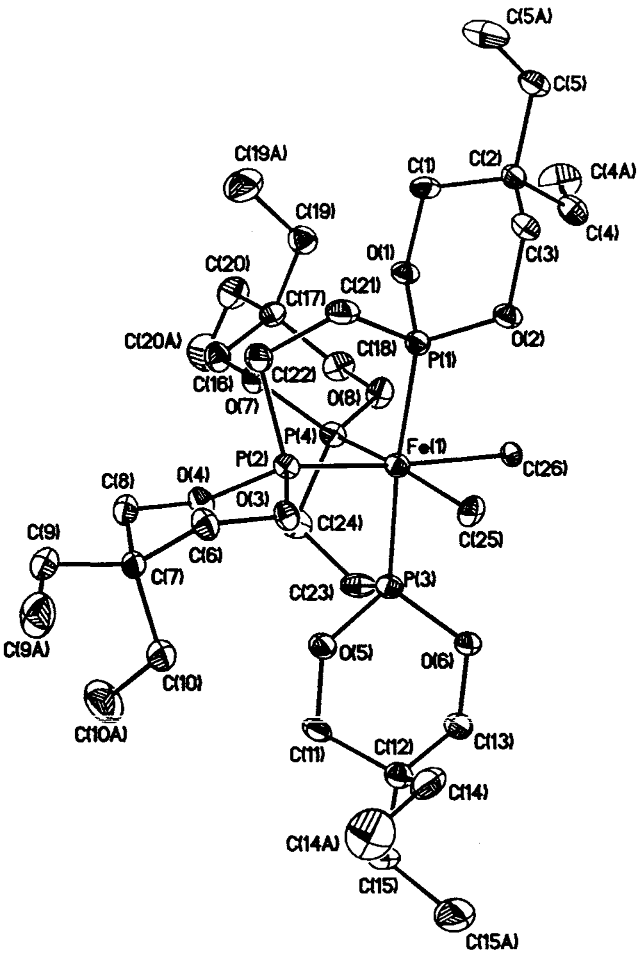

Figure 1. Drawing of $Fe_2Me_2(PP)_2$ (**5**) (thermal ellipsoids are drawn at the 50% level). Selected bond distances (Å) and angles (deg): Fe−C(25), 2.151(5); Fe−C(26), 2.179(5); Fe−P(1), 2.1240(18); Fe−P(2), 2.1186(17); P(1)−O(1), 1.622(4); P(1)−O(2), 1.611(4); P(2)−O(3), 1.633(4); P(2)−O(4), 1.617(4); P(1)−Fe−P(4), 97.33(7); P(4)−Fe−P(3), 86.72(7); P(3)−Fe−C(25), 87.24(17); C(25)−Fe−C(26), 85.44(21); C(26)−Fe−P(4), 91.57(15); P(4)−Fe−C(25), 173.33(17).

dimethyl Fe(II) complexes such as *cis*-$Fe^{II}Me_2(Me_2PCH_2CH_2PMe_2)_2$[7] and *cis*-$Fe^{II}Me_2[o\text{-}C_6H_4(PMe_2)_2]_2$,[1g] which are configurationally unstable and readily convert to a mixture of cis and trans isomers. Unfortunately, attempts to synthesize 16e or weakly ligated species such as $[FeMe(PP)_2]^+$ by treatment of **5** with $H(OEt_2)_2BAr_F$ or Ph_3CBAr_F (BAr_F = $B(3,5\text{-}(CF_3)_2\text{-}C_6H_3)_4$) afforded mixtures from which no useful information could be extracted.

Crystals of **5** suitable for X-ray diffraction were grown from a toluene/hexane mixture. The solid-state structure of **5** was solved by X-ray single-crystal diffraction analysis (Figure 1). The crystallographic data are listed in Table 1.

The structure shows an overall octahedral conformation. The Fe−C(25) and Fe−C(26) distances are 2.151(5) and 2.179(5) Å, respectively. While there is no other structural report on $Fe^{II}Me_2$ complexes containing four phosphorus atoms in the Cambridge Structure Database,[8] the average Fe−C distance is longer than the average mean value of 2.046 Å of other iron−methyl complexes.[9] The Fe−P(1) distance, 2.1240(18) Å, is slighter longer than the Fe−P(2) distance, 2.1186(17) Å, probably reflecting the stronger π-accepting ability of the phosphonite group P(3) (trans to P(1)) than the methyl group (trans to P(2)). The average Fe−P distance, 2.1213(18) Å, is shorter than the mean values, 2.139(2) and 2.239(4) Å, found for the analogous phosphonite complexes $FeH_2(PPh(OEt)_2)_4$[10a] and $[FeCl(4\text{-}CH_3C_6H_4NC)_2(PPh(OEt)_2)_3][ClO_4]$,[10b] respectively. In addition, the Fe−P distance is also shorter than the average value, 2.224(3) Å, found for the cis phosphine complex $Fe(PBE5)_2Cl_2$ (PBE5 = 1,2-diphospholanothane).[6] The P(1)−Fe−P(4) angle, 97.33(7)°, is larger than those for P(4)−Fe−P(3) (86.72(7)°) and P(3)−Fe−C(25) (87.24(17)°). The P(4)−Fe−C(26) angle, 91.57(15)°, is larger than that for C(26)−Fe−C(25) (85.44(21)°). Finally, the P(4)−Fe−C(25) angle is 173.33(17)°, indicating deviation of the structure from perfect octahedral conformation.

Table 1. Crystallographic Data of Compound 5

mol formula	$C_{34}H_{70}FeO_8P_4$	α (deg)	90
fw	786.63	β (deg)	101.176(1)
space group	$P2_1/n$	γ (deg)	90
λ, Å	0.710 73	V (Å^3)	3951.2(5)
temp (K)	203(2)	Z	4
a (Å)	13.605(1)	ρ_{calcd} (g cm^{-3})	1.322
b (Å)	18.373(1)	μ (mm^{-1})	0.590
c (Å)	16.113(1)	final R indices[a]	R1($I > 2\sigma$) = 0.0810 wR2($I > 2\sigma$) = 0.1420

[a] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $wR2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$. The parameter w is equal to $1/[\sigma^2(F_o^2) + (0.0532P)^2]$.

In summary, the first examples of Ru(II) and Fe(II) diphosphonite complexes have been synthesized. They exhibit different chemical properties from the respective phosphine-based complexes. Work on other diphosphonite ligands and their electrophilic (and hopefully unsaturated) transition-metal complexes will continue to be studied in this laboratory.

Experimental Section

All manipulations were performed either under a helium atmosphere in a Vacuum Atmospheres drybox or under an argon atmosphere using standard Schlenk techniques unless otherwise specified. Hexane, toluene, ether, and THF were purified by passing through columns of activated alumina and activated Cu-0226 S copper catalyst (Engelhard). CH_2Cl_2 was purchased from Aldrich and dried under 4 Å molecular sieves. $RuClH(Ph_3P)_3$[11] and $MgMe_2$[12] were prepared as reported. $AgPF_6$, $Cl_2PCH_2CH_2PCl_2$, and $RuCl_2(Ph_3P)_3$ were purchased from Strem Chemicals and used as received. Other reagents

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[7] Girolami, G. S.; Wilkinson, G.; Galas, A. M. R.; Thornton-Pett, M.; Hursthouse, M. B. *J. Chem. Soc., Dalton Trans.* **1985**, 1345.

[8] CSD version 5.20 (October 2000). The structural analysis of one compound $FeMe_2(dmpm)_2$ (dmpm = $Me_2PCH_2PMe_2$) was mentioned: Wong, W.-K.; Chiu, K.; Wilkinson, G.; Howes, A. J.; Motevalli, M.; Hursthouse, M. B. *Polyhedron* **1985**, *4*, 603. However, no meaningful geometrical parameters were reported because of extensive orientational disorder of the molecule.

[9] Kisko, J. L.; Hascall, T.; Parkin, G. *J. Am. Chem. Soc.* **1998**, *120*, 10561.

[10] (a) Guggenberger, L. J.; Titus, D. D.; Flood, M. T.; Marsh, R. E.; Orio, A. A.; Gray, H. B. *J. Am. Chem. Soc.* **1972**, *94*, 1135. (b) Albertin, G.; Orio, A. A.; Calogero, S.; Sipio, L. D.; Pelizzi, G. *Acta Crystallogr.* **1976**, *B32*, 3023.

[11] Hudson, B.; Webster, D. E.; Wells, P. B. *J. Chem. Soc., Dalton Trans.* **1972**, 1204.

[12] Strohmeier, W.; Siefert, F. *Chem. Ber.* **1961**, *94*, 2356.

were purchased from Aldrich and Acros Chemicals and used as received. ^{1}H, ^{31}P, and ^{13}C spectra were recorded on a Varian Unity 300 spectrometer with field strengths of 300, 121, and 75 MHz, respectively. ^{1}H and ^{13}C chemical shifts were referenced to the residual solvent resonance relative to TMS; ^{31}P chemical shifts were referenced to external 85% H_3PO_4. Elemental analyses were performed in house on a Perkin-Elmer Series II CHNS/O model 2400 analyzer.

1,2-Bis((2,2-diethyl-1,3-propanedioxy)phosphino)ethane (1, PP). A solution of $Cl_2PCH_2CH_2PCl_2$ (1.000 g, 4.32 mmol) in Et_2O (25 mL) was added slowly to a solution of $Et_2C(CH_2OH)_2$ (1.140 g, 8.64 mmol) and Et_3N (2.52 mL, 18.1 mmol) in Et_2O at −78 °C to give a white suspension. The suspension was slowly warmed to room temperature and stirred overnight at room temperature. The mixture was then filtered through Celite, and the solid was washed with Et_2O. The filtrate was dried to give the product (1.300 g, 86%) as a white solid. ^{1}H NMR ($CDCl_3$): δ 0.80 (t, 6H, $J = 7.6$ Hz), 0.89 (t, 6H, $J = 7.6$ Hz), 1.17 (quartet, 4H, $J = 7.6$ Hz), 1.73 (quartet, 4H, $J = 7.6$ Hz), 1.84 (t, 4H, $J = 6.6$ Hz), 3.68 (t, 4H, $J = 9.6$ Hz), 3.89 (dd, 4H, $J = 11.2$, 3.9 Hz). ^{31}P NMR ($CDCl_3$): δ 174.0. ^{13}C NMR ($CDCl_3$): δ 6.6, 7.5, 18.7 (dd, $J = 42.4$, 19.9 Hz), 22.7, 25.0, 38.1, 68.3. Anal. Calcd for $C_{16}H_{32}O_4P_2$: C, 54.86; H, 9.14. Found: C, 55.27; H, 9.40.

***trans*-RuCl$_2$(PP)$_2$ (2).** The ligand PP (77.0 mg, 0.22 mmol) was added to a black solution of $RuCl_2(Ph_3P)_3$ (95.9 mg, 0.10 mmol) in CH_2Cl_2 (2.5 mL) at room temperature to give a light yellow solution. The solution was stirred at room temperature for 15 min. Hexane (ca. 15 mL) was then added, and the mixture was cooled to −30 °C to give the product (46.0 mg, 52%) as light yellow crystals. ^{1}H NMR ($CDCl_3$): δ 0.78−0.88 (m, 24H), 1.20 (quartet, 8H, $J = 7.6$ Hz), 1.93 (quartet, 8H, $J = 7.6$ Hz), 2.53 (br s, 8H, PCH_2), 4.03 (br s, 16H). ^{31}P NMR ($CDCl_3$): δ 203.8. ^{13}C NMR (CD_2Cl_2): δ 6.7, 7.8, 22.7, 24.2 (t, $J_{PC} = 13.1$ Hz), 24.6, 38.2, 71.5. Anal. Calcd for $C_{32}H_{64}Cl_2O_8P_4Ru$: C, 43.54; H, 7.26. Found: C, 43.66; H, 7.66.

***trans*-[RuCl(PP)$_2$][PF$_6$] (3).** $AgPF_6$ (25.2 mg, 0.10 mmol) was added to a solution of **2** (88.2 mg, 0.10 mmol) in CH_2Cl_2 (3 mL) at room temperature to give a yellow suspension. The suspension was stirred at room temperature for 2 h, and volatiles were removed under vacuum. The residue was extracted with acetone and filtered through Celite. The acetone solution was concentrated, and hexane was added. The solution was then cooled to −30 °C to give the product (50.0 mg, 50%) as white solids. ^{1}H NMR (acetone-d_6): δ 0.82 (t, 12H, $J = 7.5$ Hz), 0.91 (t, 12H, $J = 7.5$ Hz), 1.27 (m, 8H, $J = 7.5$ Hz), 1.96 (m, 8H, $J = 7.5$ Hz), 2.59 (br s, 8H), 3.99 (br s, 8H), 4.27 (d, 8H, $J = 11.3$ Hz). ^{31}P NMR (acetone-d_6): δ −130.7 (hept, $J_{PF} = 291.2$ Hz), 202.2. ^{13}C NMR (acetone-d_6): δ 6.6, 8.0, 23.1, 24.4 (br), 38.8, 72.4. Anal. Calcd for $C_{32}H_{64}ClF_6O_8P_5Ru$: C, 38.69; H, 6.45. Found: C, 36.92; H, 6.44.

***cis*-FeCl$_2$(PP)$_2$ (4).** THF (ca. 5 mL) was added to a mixture of $FeCl_2$ (34.9 mg, 0.27 mmol) and **1** (0.2068 g, 0.59 mmol) at room temperature. The resulting mixture was stirred overnight at room temperature to give an orange solution. Hexane (15 mL) was added, and the mixture was cooled to −30 °C to give the product (0.210 g, 94%) as orange solids. ^{31}P NMR (CD_2Cl_2): δ 227.7 (t, $J = 67.6$ Hz), 241.0 (t, $J = 67.6$ Hz). ^{13}C NMR (CD_2Cl_2): δ 7.9, 8.2, 8.6, 8.8, 23.0, 24.8, 24.9, 28.7, 30.6, 38.6, 39.9, 72.8, 73.3, 73.7, 74.0.

***cis*-FeMe$_2$(PP)$_2$ (5).** $MgMe_2$ (23.0 mg, 0.43 mmol) was added to a solution of **4** (0.168 g, 0.20 mmol) in THF (2.5 mL) at −25 °C to give a green solution. The solution was stirred at room temperature for 1 h. Volatiles were then removed under vacuum, and the residue was extracted with CH_2Cl_2 and filtered. The CH_2Cl_2 solution was concentrated, and hexane was added. The mixture was cooled to −30 °C to give the product (0.085 g, 54%) as light yellow crystals. ^{1}H NMR (CD_2Cl_2): δ −0.76 (q, 6H, $J = 6.5$ Hz), 0.71−0.90 (m, 24H), 1.03−1.15 (m, 8H), 1.58 (br, 4H), 1.76−1.87 (m, 8H), 2.20−2.31 (m, 4H), 3.61−3.98 (m, 6H). ^{31}P NMR (CD_2Cl_2): δ 232.6 (t, $J = 46.2$ Hz), 238.9 (t, $J = 46.2$ Hz). ^{13}C NMR (CD_2Cl_2): δ −10.6 (d, $J = 27.2$ Hz), 6.8, 6.9, 7.5, 7.7, 21.6, 22.0, 23.4 (br, PCH_2), 24.8, 24.9, 37.2, 37.4, 70.8, 71.0, 71.3, 71.3. Anal. Calcd for $C_{34}H_{70}O_8P_2Fe$: C, 51.78; H, 8.88. Found: C, 51.72; H, 9.20.

X-ray Structure Determination of 5. A colorless, rod-shaped crystal was attached to a glass fiber using a spot of silicone grease. The crystal was placed on a Bruker P4/CCD/PC diffractometer and cooled to 203 K using a Bruker LT-2 temperature device. The data were collected using a sealed, graphite-monochromated Mo Kα X-ray source. A hemisphere of data was collected using a combination of φ and ω scans, with 30 s frame exposures and 0.3° frame widths. Data collection, initial indexing, and cell refinement were handled using SMART[13] software. Frame integration and final cell parameter calculation were carried out using SAINT[14] software. The data were corrected for absorption using the SADABS[15] program. Decay of the reflection intensity was not observed.

The structure was solved in space group $P2_1/n$ using direct methods and difference Fourier techniques. The initial solution revealed the iron and the majority of all non-hydrogen atom positions. The remaining atomic positions were determined from subsequent Fourier synthesis. All hydrogen-atom positions were idealized, with C−H = 0.96 Å (methyl) or 0.97 Å (methylene). The idealized hydrogen atoms were refined using a riding model, with isotropic temperature factors set to 1.2 (methylene) or 1.5 (methyl) times the equivalent isotropic U value of the atom they were bonded to. The final refinement included anisotropic temperature factors on all non-hydrogen atoms and converged with final residuals of R1($I > 2\sigma$) = 0.0810 and wR2($I > 2\sigma$) = 0.1420. Structure solution, refinement, graphics, and creation of publication materials were performed using SHELXTL NT.[16] Additional details of the data collection and structure refinement are listed in Table 1.

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Supporting Information Available: X-ray crystallographic data of the structure of **5** and NMR spectra of **3**. This material is available free of charge via the Internet at http://pubs.acs.org.

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[14] SAINT Version 4.05; Bruker Analytical X-ray Systems, Inc., Madison, WI 53719, 1996.

[15] Sheldrick, G. M. SADABS; University of Göttingen, Göttingen, Germany, 1996.

[16] SHELXTL NT Version 5.10; Bruker Analytical X-ray Instruments, Inc., Madison, WI 53719, 1997.