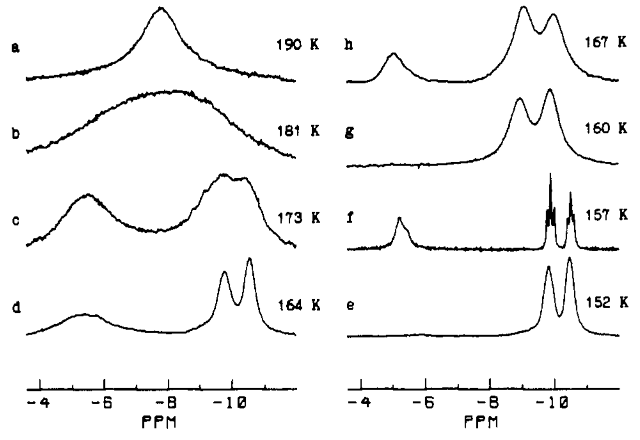


Figure 1. (a–e) Variable-temperature ^{1}H NMR spectra of the hydride region of $Ru(H)_2(H_2)(CO)[P(i\text{-}Pr)_3]_2$ dissolved in a mixture of CH_2Cl_2 and toluene-d_8 (5%) under an atmosphere of H_2 and (f–h) the hydride region of the ^{1}H NMR spectra at lowest attainable temperature of the following samples: (f) partially deuterated **3** (65% D) in CD_2Cl_2; (g) complex **3** in toluene-d_8; (h) partially deuterated **3** (50% D) in toluene-d_8. In the cases of the samples for spectra f and h the sources of deuterium were CD_2Cl_2 and HD gas, respectively.[9] The amount of deuteration was determined by integration of the residual resonance in the hydride region against the resonances of the phosphine protons.

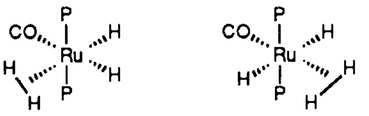
K, irradiating the RuH_4 resonance saturates the resonance of H_2 at δ 4.53. At temperatures above 270 K, this intermolecular exchange destroys $^{1}H-^{31}P$ spin–spin coupling and the hydride signal is broad. However, at temperatures below 250 K, the width of the RuH_4 resonance increases again due to a fast proton relaxation. The minimum T_1 value of 15 ± 0.5 ms found for this resonance in toluene at 200 K clearly indicates[12] the presence of a dihydrogen ligand in **3**, assuming a rapid intramolecular exchange between the dihydrogen and hydride ligands. The T_1 time of 13.9 ± 0.5 ms is also observed in a dichloromethane solution of **3** at 190 K. According to these data, an H–H bond distance in the η^2-H_2 ligand of $Ru(H)_2(H_2)(CO)[P(i\text{-}Pr)_3]_2$ of 0.86–0.84 Å can be calculated.[13]

At 260 K the ^{1}H NMR spectrum of a deuterated (under a 9:1 $D_2{:}H_2$ atmosphere) complex **3** in toluene reveals a pattern at δ −7.64, best described as an AX_2Y_3 spin system ($X = {}^{31}P$, $Y = {}^{2}H$), with $^{2}J(H,P) = 10.8$ Hz and $J(H,D) = 5.4$ Hz. The latter is the value expected (if hydrogen and deuterium are distributed statistically) for the HD_3 isotopomer of **3**, where the $J(H,D)$ value for the η^2-HD ligand is actually 32.4 Hz.[14]

A slowing of the exchange between the dihydrogen and hydride ligands of **3** takes place below 190 K (Figure 1a–d). After that, in the ^{1}H NMR spectra separate resonances are observed at δ −5.0, −8.95, and −9.93 in toluene and at δ −5.2, −9.83, and −10.49 in dichloromethane solutions of **3**. In the case of a CD_2Cl_2 solution of a partially deuterated complex **3**, the two signals between −9 and −11 ppm reveal couplings $^{2}J(H,P)$ of 21.8 and 19.6 Hz (Figure 1f) as expected for cis hydride and phosphine ligands.

The fact that the width of the signal at δ −5 is substantially reduced by partial deuteration (due to weakening of dipole–dipole interactions) allows us to assign the signal to the dihydrogen ligand. The lack of observable $J(H,D)$ couplings in the spectra of the partially deuterated **3** (Figure 1f,h) can be explained taking into account deuterium relaxation, which results in the scalar relaxation of the second kind of the proton in the HD ligand. Preliminary ^{2}H T_1 data for $Ru(D)_2(D_2)(CO)[P(i\text{-}Pr)_3]_2$ give the T_1 value of ca. 12 ms at 157 K.[15] The relaxation rate ($1/T_1$) of 83 s^{-1} is sufficient to cause the strong broadening of the HD triplet and can preclude observation of the expected 32.4 Hz $J(H,D)$ in **3** at low temperatures.[16]

Chart I



structure **3a**

structure **3b**

There are two possible structures (**3a** and **3b**) for $Ru(H)_2(H_2)(CO)[P(i\text{-}Pr)_3]_2$, which are depicted in Chart I. In the case of the most probable structure **3a**, three hydride signals are expected in the slow-exchange spectra in the ratio 2:1:1 and an averaged resonance must be observed at $\delta = 0.5(\delta(H_2)) + 0.25(\delta_1(H) + \delta_2(H))$. According to the data discussed above, the averaged resonances are expected at δ −7.22 and −7.68 in toluene and dichloromethane solutions of **3**, respectively. These values are in good agreement with the experimental δ of −7.30 and −7.75 at 190 K. The same averaging is valid for the H–P couplings, and the observed $^{2}J(H,P) = 10.8$ Hz can be obtained if $^{2}J(H_2\text{–}P) = 0.9$ Hz. Thus, our data are most consistent with the structure **3a** and provide no evidence for the formation of any reliably detectable amounts of **3b** or the classical tautomer, $RuH_4(CO)[P(i\text{-}Pr)_3]_2$.

Acknowledgment. We thank Professor R. H. Crabtree for helpful discussion.

Supplementary Material Available: Figures S1 and S2, showing the temperature-dependent behavior of the RuH and H_2 resonances in the ^{1}H NMR spectra of **1** in toluene solution under an atmosphere of H_2 (2 pages). Ordering information is given on any current masthead page.

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Di-*tert*-butylphosphido versus Diphenylphosphido as a Bridging Ligand in Binuclear Iron Carbonyl Complexes: Synthesis and Structural Characterization of $Fe_2(CO)_5(\mu\text{-}P(t\text{-}Bu)_2)_2$ and $Fe_2(CO)_6(\mu\text{-}PPh_2)(\mu\text{-}P(t\text{-}Bu)_2)$

Binuclear iron carbonyls containing two 3-electron bridging ligands, such as phosphido (PR_2), thiolato, amido, certain hydrocarbyls, and several other inorganic and organic groups, represent a numerous class of structurally related complexes.[1,2] The μ-phosphido members of this class are invariably hexacarbonyls,

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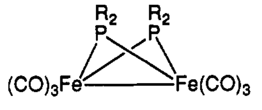
$Fe_2(CO)_6(\mu\text{-}PR_2)_2$ (I), or appropriate substitution derivatives



I

thereof with other 2-electron ligands, L, e.g., phosphines.[1,2b,f] These complexes have been the object of considerable recent research activity with respect to structure,[1b,3] ^{31}P NMR spectroscopic properties,[4,5] and reaction chemistry.[1c,2d,6] Two particularly noteworthy features of their structure are a "butterfly" shape of the Fe_2P_2 core and a metal–metal distance that is consistent with the presence of an Fe–Fe single bond. In this communication, we wish to report the synthesis and complete characterization of $Fe_2(CO)_5(\mu\text{-}P(t\text{-}Bu)_2)_2$ (**1**), the first iron bis(μ-phosphido) carbonyl that does not conform to the aforementioned structural behavior. We also report a synthetic method for mixed bis(μ-phosphido) complexes, $Fe_2(CO)_6(\mu\text{-}PR_2)(\mu\text{-}PR'_2)$. This method enabled us to prepare $Fe_2(CO)_6(\mu\text{-}PPh_2)(\mu\text{-}P(t\text{-}Bu)_2)$ (**2**) for a comparative study of spectroscopic and structural properties of **1**, **2**, and the known[7] $Fe_2(CO)_6(\mu\text{-}PPh_2)_2$ (**3**).

Reaction of $Li[Fe_2(CO)_5(PPh_2H)(\mu\text{-}PPh_2)(\mu\text{-}CO)]$[8] with 5 equiv of $P(t\text{-}Bu)_2Cl$ in THF at room temperature affords green **1**[9] (trace), deep red **2**[10] (43%), yellow **3** (11%), and three PPh_2H-substitution derivatives of **2** and **3**[11] (total yield 16%), all of which were separated by chromatography on Florisil. This apparently general reaction of $Li[Fe_2(CO)_5(PPh_2H)(\mu\text{-}PPh_2)(\mu\text{-}CO)]$ with PR_2Cl[12] is obviously quite complex; however, the formation of mixed **2** may proceed by dissociation of PPh_2H from a binuclear anion followed by nucleophilic attack of the resultant diiron species at $P(t\text{-}Bu)_2Cl$.[13]

Complex **1** can be synthesized in a much better yield[14] by reaction of $P(t\text{-}Bu)_2Cl$ with $Na_2[Fe_2(CO)_8]$, also in THF at room temperature. Its unexpected IR spectrum in the ν(CO) region,[9] which differs considerably from that of the hexacarbonyls **2**[10] and various $Fe_2(CO)_6(\mu\text{-}PR_2)_2$,[15] and a lone $^{31}P\{^1H\}$ NMR signal at δ 362.2 prompted an X-ray crystallographic investigation. The solid-state structure of **1**[16] (Figure 1) reveals several unusual features. First, as suggested by the $^{13}C\{^1H\}$ NMR spectrum and supported by the mass spectrum, which shows only five carbonyl ligands,[9] the two iron atoms have different CO coordination environments: $Fe(CO)_3$ and $Fe(CO)_2$. Second, the Fe_2P_2 core is essentially planar. Third, the Fe–Fe bond distance of 2.4870 (6) Å is considerably shorter than that in the hexacarbonyls $Fe_2(CO)_6(\mu\text{-}PR_2)_2$ (2.62–2.82 Å).[3] However, it is longer than the Fe–Fe bond distance in some binuclear iron carbonyl alkyne complexes and in $[CpFe(\mu\text{-}NO)]_2$ (2.215–2.326 (4) Å),[17] where metal–metal double bonding has been proposed. The Fe–Fe bond in **1** is supported by an unsymmetrical arrangement of the two phosphido bridges, the phosphorus atoms being closer to $Fe(CO)_2$ than to $Fe(CO)_3$.

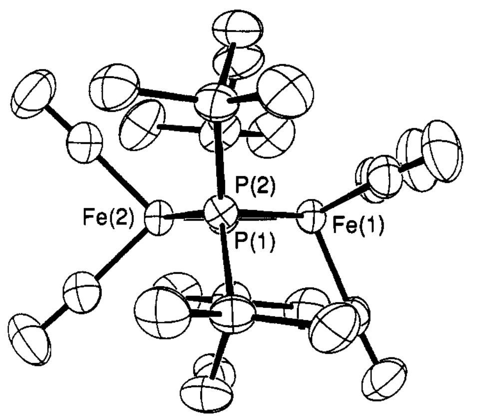


Figure 1. ORTEP view of **1**. The non-hydrogen atoms are represented by 50% probability thermal ellipsoids. The hydrogen atoms have been omitted for clarity. Selected bond distances (Å): Fe–Fe = 2.4870 (6), Fe(1)–P(av) = 2.3687 (8), Fe(2)–P(av) = 2.1368 (8). Flap angle = 176.28 (5)°.

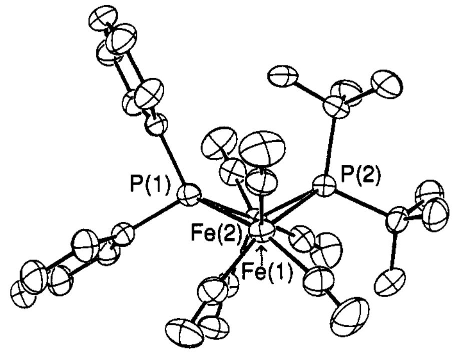


Figure 2. ORTEP view of **2**. The non-hydrogen atoms are represented by 50% probability thermal ellipsoids. The hydrogen atoms have been omitted for clarity. Selected bond distances (Å): Fe–Fe = 2.7017 (9), Fe(1)–P(av) = 2.246 (1), Fe(2)–P(av) = 2.269 (1). Flap angle = 117.07 (5)°.

To explore further the effect of replacing μ-PPh_2 with μ-$P(t\text{-}Bu)_2$ on the structure of these binuclear iron carbonyls, we also carried out an X-ray crystallographic analysis of the mixed complex **2**[18] (Figure 2). Here the salient structural features of

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(9) **1**: IR (C_6H_{12}) ν(CO) 1998 (m), 1950 (s), 1945 (s), 1911 (m) cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.23 (d, J_{PH} = 14 Hz, Me); $^{13}C\{^1H\}$ NMR ($CDCl_3$) δ 220.2 (s, $Fe(CO)_3$), 215.9 (t, J_{PC} = 10 Hz, $Fe(CO)_2$), 45.2 (br, PC), 32.9 (br, Me); $^{31}P\{^1H\}$ NMR ($CDCl_3$) δ 362.2 (s); mass spectrum (EI) *m/e* (ion) 542 (M^+). Anal. Calcd for $C_{21}H_{36}Fe_2O_5P_2$: C, 46.52; H, 6.69. Found: C, 46.54; H, 6.66.

(10) **2**: mp 158 °C; IR (C_6H_{12}) ν(CO) 2033 (m), 1998 (s), 1977 (sh), 1965 (s), 1959 (s), 1948 (m) cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.62–7.28 (m, 2Ph), 1.36 (d, J_{PH} = 14 Hz, 6Me); $^{13}C\{^1H\}$ NMR ($CDCl_3$) δ 214.0 (t, J_{PC} = 5.5 Hz, CO), 142.0–127.8 (m, Ph), 44.27 (d, J_{PC} = 5.6 Hz, PC), 33.99 (d, J_{PC} = 1.9 Hz, Me); $^{31}P\{^1H\}$ NMR ($CDCl_3$) δ 290.8 (d), 173.3 (d, J_{PP} = 63 Hz); mass spectrum (EI) *m/e* (ion) 582 ($M^+ - CO$), 554 ($M^+ - 2CO$), and consecutive loss of 4 remaining CO's. Anal. Calcd for $C_{26}H_{28}Fe_2O_6P_2$: C, 51.15; H, 4.59. Found: C, 50.94; H, 4.80.

(11) Red-brown $Fe_2(CO)_5(PPh_2H)(\mu\text{-}PPh_2)(\mu\text{-}P(t\text{-}Bu)_2)$, yellow $Fe_2(CO)_5(PPh_2H)(\mu\text{-}PPh_2)_2$, and orange $Fe_2(CO)_4(PPh_2H)_2(\mu\text{-}PPh_2)_2$. For their characterization, see the supplementary material.

(12) A similarly conducted reaction of $Li[Fe_2(CO)_5(PPh_2H)(\mu\text{-}PPh_2)(\mu\text{-}CO)]$ with PEt_2Cl afforded $Fe_2(CO)_6(\mu\text{-}PPh_2)(\mu\text{-}PEt_2)$, in addition to $Fe_2(CO)_6(\mu\text{-}PPh_2)_2$, $Fe_2(CO)_5(PPh_2H)(\mu\text{-}PPh_2)_2$, and $Fe_2(CO)_4(PPh_2H)_2(\mu\text{-}PPh_2)_2$.

(13) Use of only 1 equiv of $P(t\text{-}Bu)_2Cl$ in the reaction with $Li[Fe_2(CO)_5(PPh_2H)(\mu\text{-}PPh_2)(\mu\text{-}CO)]$ considerably decreases the yields of the di-*tert*-butylphosphido-containing products.

(14) After chromatography on Florisil, crystallization of the resultant oil from pentane at ca. −78 °C, and recrystallization from CH_2Cl_2/hexane at −10 °C, the yield of analytically pure **1** is 28%.

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(16) Crystal data for **1**: space group $P\bar{1}$, a = 11.741 (1) Å, b = 13.662 (2) Å, c = 8.652 (1) Å, α = 101.21 (1)°, β = 103.64 (1)°, γ = 76.63 (1)°, V = 1297.8 (3) $Å^3$, Z = 2, D_{calc} = 1.39 g cm^{-3}; data collected at 296 K with Mo Kα radiation, $\lambda(K\bar{\alpha})$ = 0.71069 Å, 2θ limits 4–55°; data corrected for absorption by ψ-scan method, μ = 12.65 cm^{-1}, transmission factors 0.81–1.00. The structure was solved by a combination of the Patterson and direct methods. After anisotropic refinement, the H atoms were fixed at idealized positions with C–H = 0.98 Å. The final refinement cycle resulted in $R(F)$ = 0.032 and $R_w(F)$ = 0.037 for the 4182 unique reflections with $F_o^2 > 3\sigma(F_o^2)$ and the 272 variables. A secondary extinction coefficient refined to a value of 1.7 (1) × 10^{-6}. Details will be published in our full paper.

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the hexacarbonyls $Fe_2(CO)_6(\mu\text{-}PR_2)_2$, and **3** in particular (bond distances: Fe–Fe = 2.623 (2), Fe(1)–P(av) = 2.228 (3), Fe(2)–P(av) = 2.240 (3) Å),[19] are retained; however, the "flap angle" of the Fe_2P_2 core (i.e., angle between the two FeP_2 planes) increases to 117.1° from 100.0° in **3**.[7b]

The Mössbauer spectra of **1** and **2** reflect the aforementioned structural differences. Each shows two quadrupole doublets,[20] indicating nonequivalent iron sites. However, the differences in quadrupole interactions in the two complexes indicate that, as expected, the chemical environments about the two iron sites are substantially more different in **1** than in **2**. One site is similar to those in **2**, whereas the other, $Fe(CO)_2$, site has a markedly increased quadrupole interaction, corresponding to a reduced electronic symmetry as compared with the $Fe(CO)_3$ sites in **2**. This reduction is easily seen in the short Fe(2)–P(1) and Fe(2)–P(2) bond distances of **1** as compared with the same bonds of **2**. Complex **1** also has a smaller average isomer shift. This corresponds to a higher average s-electron density at the iron nuclei, presumably as a result of reduced electron donation to the fewer CO ligands in **1**.

The structural changes that occur on making the progression from **3** to **2** and then to **1** are most likely steric in origin. The bulky *t*-Bu groups force the "butterfly wings" to open until, with two $P(t\text{-}Bu)_2$ bridges, they presumably crowd the carbonyl ligands. This may cause loss of one CO and a shift of both $\mu\text{-}P(t\text{-}Bu)_2$ groups toward the now 16-electron Fe(2).[21] The observed short Fe–Fe bond distance may arise from Fe(1) to Fe(2) π interaction.

Unusual structural properties of binuclear metal complexes containing $\mu\text{-}P(t\text{-}Bu)_2$ ligands have been previously observed by Jones.[22] Particularly relevant to the present study are the structures of $Ni_2(CO)_4(\mu\text{-}PPh_2)_2$[23] and $Ni_2(CO)_3(\mu\text{-}P(t\text{-}Bu)_2)_2$.[22b] The latter contains Ni atoms with different CO coordination environments, unsymmetrical phosphido bridges (Δ(Ni–P) = 0.074 (3) Å), and a shortened Ni–Ni bond compared to that in the bis(μ-diphenylphosphido) tetracarbonyl (Δ(Ni–Ni) = 0.096 (2) Å). These results parallel those observed by us for **1**; however, a larger amount of data on iron carbonyl phosphido complexes than on nickel carbonyl phosphido complexes, as well as a greater number of structural variables for the former complexes, allow a more extensive comparison of the influence of $\mu\text{-}P(t\text{-}Bu)_2$ on structure in the iron system. Studies on these complexes continue in our laboratories.

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Supplementary Material Available: Additional textual details of the synthesis and characterization of new complexes, a figure showing the Mössbauer spectra of **1** and **2**, tables of crystal data and data collection and refinement details, positional parameters, anisotropic thermal parameters, bond distances, and bond angles for **1** and **2**, and figures showing the atom-numbering schemes for **1** and **2** (22 pages). Ordering information is given on any current masthead page.

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(18) Crystal data for **2**: space group $P2_1/c$, a = 10.116 (3) Å, b = 16.810 (4) Å, c = 16.841 (4) Å, β = 105.38 (2)°, V = 2761 (1) Å^3, Z = 4, D_{calc} = 1.47 g cm^{-3}; data collected at 296 K with Mo Kα radiation, $\lambda(K\bar{\alpha})$ = 0.71073 Å, 2θ limits 4–55°; absorption correction by ψ-scan method, μ = 12.00 cm^{-1}, transmission factors 0.88–1.00. The structure was solved by the Patterson method. The H atoms were fixed at idealized positions with C–H = 0.98 Å. The final refinement cycle resulted in $R(F) = 0.037$ and $R_w(F) = 0.036$ for the 3525 unique reflections with $F_o^2 > 3\sigma(F_o^2)$ and the 325 variables. Details will be published in our full paper.

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(20) For **1** at 78 K the isomer shifts are −0.051 and 0.029 mm/s, and the quadrupole splittings are 1.42 and 0.55 mm/s. For **2** at 78 K the isomer shifts are −0.018 and 0.024 mm/s, and the quadrupole splittings are 1.50 and 0.99 mm/s. The isomer shifts are relative to α-iron foil.

(21) The Fe(2)···H intramolecular distances are 3.168 Å and longer to show the absence of agostic interactions.

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