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Novel Diamagnetic and Paramagnetic Iron(II), Iron(III), and Iron(IV) Classical and Nonclassical Hydrides. X-ray Crystal Structure of [Fe(C₅Me₅)(dppe)D]PF₆

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Summary: The synthesis, characterization, and X-ray crystal structure determination of the unprecedented 17-electron radical cation iron(III) hydride [Fe(Cp*)(dppe)H]PF₆ (2) is reported. The related protonation of the parent iron(II) hydride Fe(Cp*)(dppe)H, including both the synthesis and characterization of the first half-sandwich iron dihydrogen complex, [Fe(Cp*)(dppe)(η²-H₂)]BF₄, and the first organoiron(IV) dihydride, [Fe(Cp*)(dppe)(H)₂][BF₄], are presented.

Transition-metal hydride complexes constitute an important class of compounds because of their involvement in catalytic and stoichiometric processes.¹ Although they have been the subject of intensive investigation, the chemistry of electron-deficient, 17-electron metal hydrides resulting from the oxidation of the corresponding 18-electron neutral species remains a relatively unknown area. Most of the radical cations previously studied were short lived, their major mode of decomposition being deprotonation.² We wish to report (1) the synthesis, characterization, X-ray crystal structure determination, and unusual reactivity of the first 17-electron radical cation iron hydride, [Fe(Cp*)(dppe)H]PF₆ (2, dppe = 1,2-bis(diphenylphosphino)ethane, Cp* = η-C₅Me₅) and (2) the re-

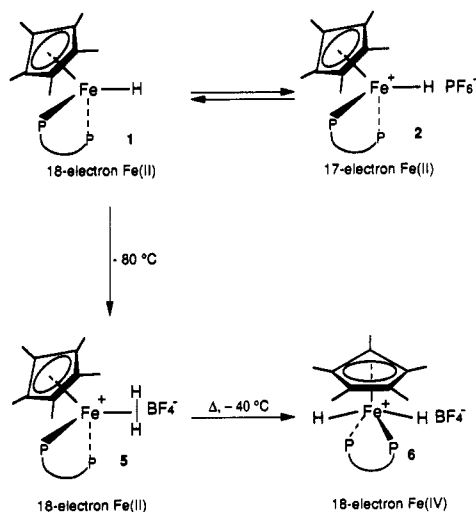
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Scheme I



lated protonation of the parent iron(II) hydride Fe(Cp*)(dppe)H (1), including both the synthesis and the characterization of the nonclassical [Fe(Cp*)(dppe)(η²-H₂)]BF₄ (5) and the classical [Fe(Cp*)(dppe)(H)₂][BF₄] (6) (Scheme I).

When the neutral iron hydride Fe(Cp*)(dppe)H (1) is treated with 1 equiv of ferrocenium hexafluorophosphate salt in CH₂Cl₂ at -80 °C, the solution turns red in 1 h and the novel iron hydride radical cation [Fe(Cp*)(dppe)H]PF₆ (2) is isolated in quantitative yield after its precipitation and washing with pentane. The thermally stable complex 2^{3a} is paramagnetic ($\mu_{\text{eff}} = 2.40 \mu_{\text{B}}$),⁴ and its IR spectrum

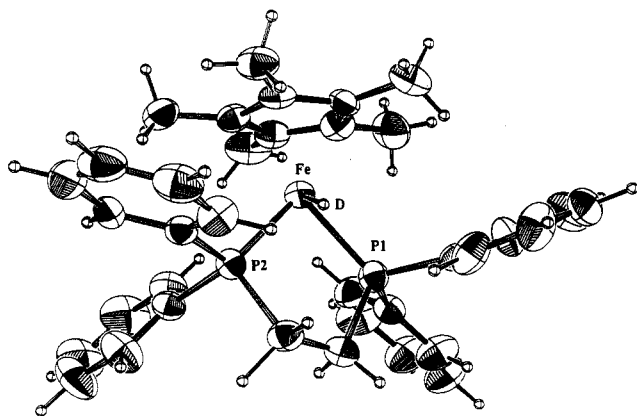
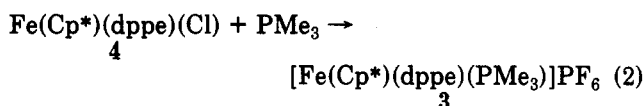
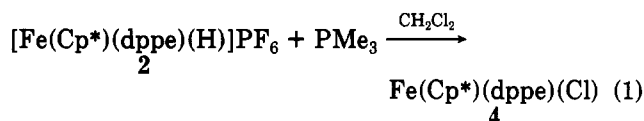


Figure 1. ORTEP representation for **2** with the PF_6^- anion, solvent molecule, and hydrogen atoms of the Cp^* and phenyl rings removed for clarity. Selected bond lengths (Å) and bond angles (deg): Fe–D, 1.55 (8); Fe–P(1), 2.213 (3); Fe–P(2), 2.200 (3); P(1)–Fe–P(2), 88.5 (1); P(1)–Fe–D, 77 (5); P(2)–Fe–D, 69 (4).

indicates the presence of a terminal hydride ligand ($\nu_{\text{Fe-H}} = 1869 \text{ cm}^{-1}$). Dark red crystals were grown by slow diffusion of pentane into a CH_2Cl_2 solution of the isotopomer **2-d**, and a single-crystal X-ray structure was determined (Figure 1).⁵ The C_5 ring of the Cp^* group is planar, and the deuterium atom, the iron nucleus, and the Cp^* centroid are located in a plane that is nearly normal at the C_5 ring. The Fe–D bond length (1.55 (8) Å) is much larger than the Fe–H distance in the diamagnetic iron(II) hydride $[\text{Fe}(\eta^2\text{-H}_2)(\text{H})(\text{dppe})]\text{BPh}_4$ (1.30 (3) Å).⁶ The Fe–P separa-

tions (2.213 (3) and 2.200 (3) Å) are rather contracted compared to those observed in the isostructural iron(III) alkyl complex $[\text{Fe}(\text{Cp}^*)(\text{dppe})\text{CH}_2\text{OCH}_3]\text{PF}_6$.⁷

It is commonly believed that metals in the third and second transition series form bonds to hydrogen stronger than those in the first row. Calorimetric^{2a} and chemical reactivity^{1a,8} data support this view. Surprisingly, **2** is stable for several hours in CH_2Cl_2 or THF and does not react with **1** under these conditions, indicating that spontaneous proton transfer is not a kinetically favored process. The proton abstraction from **2** cannot be achieved in CH_2Cl_2 with PPh_3 , PMe_3 , or even Et_3N . However, **2** reacts with PMe_3 , affording $[\text{Fe}(\text{Cp}^*)(\text{dppe})(\text{PMe}_3)]\text{PF}_6$ (**3**).^{3b} We assume that the 17-electron radical resulting from the deprotonation of **2** reacts immediately with CH_2Cl_2 to give the known^{4,9,10} chloro complex **4** (eq 1). The subsequent displacement of the chloride anion by PMe_3 occurs quickly in the presence of hexafluorophosphate ion (eq 2).



The literature provides examples showing that neutral hydrides react as bases with their 17-electron cationic analogues. The absence of reactivity between **1** and **2** contrasts strongly with well-established knowledge.² We therefore examined separately the protonation of **1**. Addition of 1 equiv of $\text{HBF}_4 \cdot \text{Et}_2\text{O}$ to a solution of **1** in Et_2O at -80°C gave the first piano-stool dihydrogen complex of iron, $[\text{Fe}(\text{Cp}^*)(\text{dppe})(\eta^2\text{-H}_2)]\text{BF}_4$ (**5**),^{3c} isolated as lemon yellow microcrystals in 95% yield (Scheme I).¹¹ The ^1H NMR spectrum (300 MHz) of **5** in the hydride region consists of a broad peak at $\delta -12.39$ (CH_2Cl_2 , -80°C) with a minimum T_1 value of 7 ms at 223 K. **1-d** is protonated to **5-d**, which exhibits a resolvable hydrogen–deuterium coupling constant ($J_{\text{HD}} = 27 \text{ Hz}$). Crystals of **5** can be stored conveniently at -50°C , but when they are warmed to room temperature, they convert to the Fe(IV) dihydride $[\text{Fe}(\text{Cp}^*)(\text{dppe})(\text{H})_2]\text{BF}_4$ (**6**).^{3d} When a solution of **5** in CD_2Cl_2 is warmed from -80 to 20°C , ^1H NMR spectroscopy reveals the gradual, complete, and irreversible generation of complex **6**. The latter constitutes the first organoiron(IV) dihydride to be isolated.¹³ It displays a characteristic sharp triplet ($\delta -7.89$, $^2J_{\text{PH}} = 68 \text{ Hz}$) in the ^1H NMR spectrum with a T_1 value of 175 ms at 223 K. Consistent with a trans geometry, the $^{31}\text{P}\{^1\text{H}\}$ NMR

(3) (a) $[\text{Fe}(\text{Cp}^*)(\text{dppe})(\text{H})]\text{PF}_6$ (**2**). A solution of **1** (0.590 g, 1 mmol) in CH_2Cl_2 (10 mL) was treated with $[\text{FeCp}_2]\text{PF}_6$ (0.30 g, 0.9 mmol) for 3 h at -60°C . Pentane was added (20 mL) to precipitate **2**, yield 0.75 g (85%) of dark red microcrystals. Anal. Calcd for $\text{C}_{36}\text{H}_{40}\text{FeF}_6\text{P}_2$: C, 58.79; H, 5.48; P, 12.63. Found: C, 58.56; H, 5.31; P, 12.70. IR (Nujol): $\nu_{\text{Fe-H}}$ 1860 cm^{-1} . Mössbauer (4.2 K): IS = 0.260 mm s^{-1} (vs Fe), QS = 0.840 mm s^{-1} . ESR (4.2 K): $g = 2.0441$, 2.3460. (b) $[\text{Fe}(\text{Cp}^*)(\text{dppe})(\text{PMe}_3)]\text{PF}_6$ (**3**). A solution of **2** (0.550 g, 0.75 mmol) in CH_2Cl_2 (10 mL) was treated with PMe_3 (ca. 0.5 mL, 5 mmol) for 24 h at 20°C . Diethyl ether (20 mL) was added to precipitate **3** as an orange solid, yield 0.470 g (77%). Anal. Calcd for $\text{C}_{39}\text{H}_{48}\text{FeF}_6\text{P}_3$: C, 57.79; H, 5.97; P, 15.29. Found: C, 57.39; H, 5.85; P, 16.08. ^1H NMR (20°C , CD_3COCD_3): δ 1.48 (s, Cp^* , 15 H), 0.82 (d, PMe_3 , $^2J_{\text{PH}} = 8 \text{ Hz}$, 9 H). ^{31}P NMR (20°C , CD_3COCD_3): δ 4.3 (t, PMe_3 , $^2J_{\text{PP}} = 42 \text{ Hz}$), 88.8 (d, dppe, $^2J_{\text{PP}} = 42 \text{ Hz}$). (c) $[\text{Fe}(\text{Cp}^*)(\text{dppe})(\eta^2\text{-H}_2)]\text{BF}_4$ (**5**). A solution of **1** (0.59 g, 1 mmol) in diethyl ether was treated with $\text{HBF}_4 \cdot \text{OEt}_2$ (187 μL , 1.5 mmol) for 3 h at -50°C . A yellow powder of **5** precipitated. The solid was filtered off, washed with diethyl ether ($3 \times 10 \text{ mL}$), dried at -50°C , and stored at -50°C ; yield 0.61 g (90%). Mössbauer (4.2 K): IS = 0.317 mm s^{-1} (vs Fe), QS = 1.748 mm s^{-1} . ^1H NMR (-30°C , CD_2Cl_2): δ 1.45 (s, Cp^* , 15 H), -12.39 (b, $\omega_{1/2} = 50 \text{ Hz}$, 2 H). T_1 (300 MHz) values for the high-field signal: 193 K, 10 ms; 203 K, 8 ms; 213 K, 7 ms; 223 K, 7 ms; 233 K, 7 ms; 243 K, 9 ms; 253 K, 9 ms. (d) $[\text{Fe}(\text{Cp}^*)(\text{dppe})(\text{H})_2]\text{BF}_4$ (**6**). A solid sample of **5** was kept at 20°C for 48 h. The sample turned orange and **6** was recovered as a pure compound. Anal. Calcd for $\text{C}_{36}\text{H}_{44}\text{BF}_4\text{FeF}_4\text{P}_2$: C, 63.75; H, 6.09; P, 9.13. Found: C, 63.39; H, 5.85; P, 9.22. Mössbauer (4.2 K): IS = 0.125 mm s^{-1} (vs Fe), QS = 1.516 mm s^{-1} . ^1H NMR (20°C , CD_2Cl_2): δ 1.44 (s, Cp^* , 15 H), -7.84 (t, $^2J_{\text{PH}} = 68 \text{ Hz}$, 2 H). ^{31}P NMR (CD_2Cl_2) 91.5 (t, $^2J_{\text{PH}} = 68 \text{ Hz}$, dppe).

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(5) A prismatic dark red block ($0.20 \times 0.15 \times 0.15$) of **2-d** (CH_2Cl_2) was used for data collection. Crystal data: monoclinic, space group $P2_1/n$, $a = 10.425$ (3) Å, $b = 22.189$ (7) Å, $c = 17.043$ (5) Å, $\beta = 104.52$ (4) $^\circ$, $V = 3876$ (1) Å 3 , $M_r = 819.3$, $Z = 4$, $\rho_{\text{calc}} = 1.43 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 7.5 \text{ cm}^{-1}$, $F(000) = 1688$, $T = 293 \text{ K}$. Intensity data were collected using an Enraf-Nonius CAD4 diffractometer. A total of 9316 reflections were measured to $2\theta_{\text{max}} = 50^\circ$; 6517 reflections were unique, of which 2516 with $I = 3\sigma(I)$ were used in all calculations. The structure was solved by Patterson and conventional Fourier methods. All non-hydrogen atoms (except solvent) were refined first with isotropic and then with anisotropic thermal parameters. The hydrogen bonded to the metal was treated as a normal isotropic atom and refined with free positional and fixed thermal parameters. Final $R = 0.073$ and $R_w = 0.072$.

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spectrum shows a singlet at δ 91.5, while the corresponding uncoupled spectrum shows a triplet with $J_{\text{PH}} = 68$ Hz. It is noteworthy that complexes **5** and **6** have been obtained in a spectroscopically pure form as established by Mössbauer spectrometry in the solid state and by NMR spectroscopy in solution. These results constitute direct evidence that the protonation of an M-H bond to give an $\eta^2\text{-H}_2$ complex is faster than the protonation of the same complex at the metal, as previously predicted.¹⁴

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Mössbauer and EPR facilities, Drs. P. Guénot and S. Sinbandhit (CRMPO, Rennes, France) for their assistance in the measurements of mass and NMR spectra, and Laboratoires Standa (Caen, France) for their financial support.

Registry No. 1, 117696-31-8; 1-*d*, 132856-43-0; 2, 139524-38-2; 2-*d*, 139524-40-6; 2-*d*-CH₂Cl₂, 139524-41-7; 3, 139524-43-9; 4, 117696-30-7; 5, 139524-45-1; 5-*d*, 139524-47-3; 6, 139524-49-5.

Supplementary Material Available: A table of crystal data and data collection and refinement details and complete tables of atomic coordinates and their estimated standard deviations, bond lengths and angles, and general temperature factor expressions for **2** (12 pages); a listing of observed and calculated structure factors (18 pages). Ordering information is given on any current masthead page.

Structural and Spectroscopic Characterization of a Paramagnetic Isocyanide Complex of Rhodium(II)

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Summary: The remarkably stable paramagnetic Rh(II) complex $[\text{Rh}(\eta^3\text{-TMPP})_2][\text{BF}_4]_2$ (TMPP = tris(2,4,6-trimethoxyphenyl)phosphine) reacts with 2 equiv of *tert*-butyl isocyanide to yield the air-stable mononuclear Rh(II) complex $[\text{Rh}(\eta^3\text{-TMPP})_2(\text{CNBu}^t)_2][\text{BF}_4]_2$. A single-crystal X-ray diffraction study reveals that the cation adopts a distorted-square-planar geometry in which the phosphine ligands are *trans* rather than *cis* as in the parent complex. The paramagnetism of this unusual four-coordinate Rh(II) metallo radical was probed by a variety of spectroscopic and magnetic techniques.

The highly basic ether-phosphine ligand TMPP (TMPP = tris(2,4,6-trimethoxyphenyl)phosphine) forms unusual substitution products with a variety of transition-metal complexes, including those containing carbonyl, carboxylate, and acetonitrile ligands.¹ Of particular interest is the remarkable complex $[\text{Rh}(\eta^3\text{-TMPP})_2][\text{BF}_4]_2$ (**1**), which represents the first mononuclear Rh(II) complex to be fully characterized by X-ray diffraction, magnetic susceptibility, and spectroscopic techniques.² Most of the documented research involving mononuclear rhodium complexes has focused on the +1 and +3 oxidation states, due, in large part, to their demonstrated involvement in homogeneous catalytic processes.³ Only a limited number of reports have addressed the coordination chemistry of paramag-

netic Rh(II),⁴ and even fewer have focused on mononuclear organometallic complexes of Rh(II).⁵ The unusual stability of **1** has presented us with a rare opportunity to investigate these elusive classes of compounds.⁶ In light of the recent reports by Wayland and co-workers of carbon monoxide and methane activation by Rh(II) metallo radicals,⁷ this is a particularly attractive area of research. We recently demonstrated that $[\text{Rh}(\eta^3\text{-TMPP})_2][\text{BF}_4]_2$ reacts reversibly with CO through a pathway that involves the formation of Rh(I) carbonyl and Rh(III) intermediates.⁸ Intrigued by this unusual chemistry, we set out to explore

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