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Crystal and molecular structure of (η^5 -1,3,5-trimethyl-6-phenylcyclohexadienyl)(η^5 -cyclopentadienyl)iron, a mixed full sandwich iron(II) complex

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Abstract

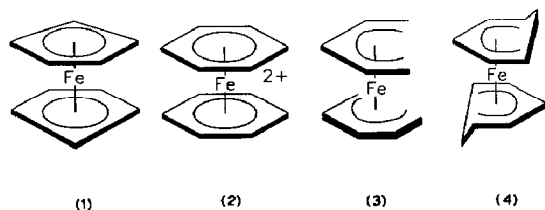
(η^5 -1,3,5-trimethyl-6-phenylcyclohexadienyl)(η^5 -cyclopentadienyl)Fe (**9**) was synthesized via reaction of phenyllithium with [(cyclopentadienyl)(mesitylene)Fe]-[PF₆]. **9** crystallized from hexanes in the monoclinic space group $P2_1/n$ with a 8.5703(5), b 8.5283(9), c 21.8436(23) Å, β 93.868(6)° and D_{calc} 1.327 g cm⁻³ for $Z = 4$. Least-squares refinement gave a conventional R value of 0.035 for 2046 independent observed reflections. The structure reveals that the phenyl moiety added in the expected *exo* fashion and that the Fe-ring distances are statistically identical, averaging 2.059(3) and 2.06(3) Å for the cyclopentadienyl and cyclohexadienyl moieties, respectively. A comparison with other mixed iron(II) full-sandwich complexes is made in an attempt to determine relative π -back bonding abilities of the four isoelectronic π -ligands: arene, cyclopentadienyl, pentadienyl, and cyclohexadienyl.

Introduction

Fe(cyclopentadienyl)₂, FeCp₂ or ferrocene (**1**) is the prototypal [1,2] and perhaps the most generally recognized example of a full-sandwich iron(II) complex. Subse-

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quent work spanning three decades has resulted in synthesis and characterization of several isoelectronic analogues, including the following: $[\text{Fe}(\text{arene})_2]^{2+}$ [3] ($[\text{Fear}_2]^{2+}$, 2), $\text{Fe}(\text{pentadienyl})_2$ [4] (Fepd_2 , 3) and $\text{Fe}(\text{cyclohexadienyl})_2$ [5] (Fech_2 , 4).



3 and 4 have been termed “open ferrocenes” [6] and “pseudoferrocenes” [5], respectively. A number of mixed ligand sandwiches have also been prepared and, as for the symmetric sandwiches, several have been characterized via X-ray crystallography. These include Fepdcp [6] (5), $[\text{Fearcp}]^+$ (6) [7,8], $[\text{Fearch}]^+$ (7) [9,10], but only one example of Fechcp (8) [11]. The latter is not representative since it was not synthesized via the well documented [12–14] carbanion addition to 6, and contains three highly electron-withdrawing CF_3 groups. We have therefore undertaken the X-ray structural characterization of an example of a mixed ch–cp complex containing hydrocarbon ligands; the title compound, $(\eta^5\text{-1,3,5-trimethyl-6-phenylcyclohexadienyl})(\eta^5\text{-cyclopentadienyl})\text{iron(II)}$ (9).

Results and discussion

9 has a melting point that agrees with a previous report [15]. No NMR results were originally reported for 9, however, our data are consistent with what one would expect for metal-cyclohexadienyl complexes and similar to that reported for 8 [11]. The ^1H NMR spectrum exhibits the characteristic [15] range of chemical shifts for cyclohexadienyl ring protons, and the ^{13}C NMR spectrum is likewise consistent with earlier studies [9,10,16,17].

Final fractional coordinates for 9 are presented in Table 1 and important bond distances and angles are presented in Table 2. A SNOOPI [18] perspective view of the complex, depicted in Fig. 1, shows that the two η^5 -rings are essentially parallel making a dihedral angle of only $2.38(15)^\circ$, and addition of the phenyl moiety has occurred in the expected [19] *exo*-fashion. The cp and phenyl rings are close to planarity with deviations of less than 0.01 \AA , while the dienyl portion of ch ring deviates from planarity by a maximum of 0.016 \AA . The three methyl carbon atoms attached to the ch ligand point significantly towards the iron atom, 0.195 , 0.193 , and 0.069 \AA from the C_5 plane for carbon atoms C(27), C(29), and C(28), respectively. The sp^3 carbon atom lies $0.677(4) \text{ \AA}$ out of the C_5 plane and the angle of upward tilt of the C_3 plane formed by C(22), C(21) and C(26) with respect to the C_5 plane is $44.1(2)^\circ$. Both these values are consistent with those observed for the prototypical cyclohexadienyl complex, $(\text{C}_6\text{H}_7)\text{Mn}(\text{CO})_3$ [20]. The phenyl moiety is twisted with respect to the mirror plane of the ch ring by 40° (based on the torsional angle between C(36):C(31) and C(21):C(23)). Comparison of the results of this study with those obtained for 8 reveal surprising little structural differences between 8 and 9. Indeed, the net effect of replacing 1,3,5-methyl groups with 1,3,5-trifluoromethyl

Table 1

Atomic parameters x , y , z , and B_{iso} for g (esd's refer to the last digit printed)

	x	y	z	B_{iso}^a
Fe(1)	0.81731(4)	0.75388(5)	0.113578(18)	3.296(16)
C(11)	1.0341(4)	0.6508(5)	0.12430(17)	5.89(19)
C(12)	0.9236(5)	0.5414(4)	0.09977(21)	6.92(21)
C(13)	0.8600(4)	0.6016(5)	0.04343(18)	6.38(18)
C(14)	0.9307(4)	0.7459(4)	0.03327(16)	5.52(17)
C(15)	1.0391(3)	0.7767(4)	0.08371(16)	5.12(16)
C(21)	0.5483(3)	0.9104(3)	0.09612(12)	3.13(11)
C(22)	0.7131(3)	0.9752(3)	0.10779(12)	3.18(11)
C(23)	0.7935(3)	0.9484(3)	0.16545(12)	3.23(12)
C(24)	0.7602(3)	0.8127(4)	0.20028(13)	3.49(11)
C(25)	0.6498(3)	0.7061(3)	0.17214(14)	3.74(12)
C(26)	0.5738(3)	0.7407(3)	0.11432(13)	3.48(12)
C(27)	0.7631(4)	1.1080(4)	0.06801(14)	4.53(14)
C(28)	0.8497(4)	0.7759(4)	0.26067(15)	4.89(15)
C(29)	0.4718(4)	0.6185(4)	0.08163(17)	5.03(16)
C(31)	0.4213(3)	1.0025(3)	0.12663(12)	3.13(11)
C(32)	0.3621(3)	0.9553(4)	0.18127(13)	3.90(13)
C(33)	0.2488(4)	1.0426(4)	0.20833(14)	4.52(14)
C(34)	0.1936(3)	1.1806(4)	0.18078(15)	4.71(14)
C(35)	0.2496(4)	1.2281(4)	0.12687(17)	4.84(15)
C(36)	0.3624(4)	1.1397(3)	0.09956(13)	4.03(13)

^a B_{iso} is the mean of the principal axes of the thermal ellipsoid.

substituents is statistically non-existent as the solid state structural parameters of **8** and **9** are almost identical.

Structural parameters for a range of symmetrical and mixed iron (II) full-sandwich complexes obtained via single crystal X-ray crystallography are presented in Table 3 and form the basis for the following discussion. Table 3 permits estimate of the relative interaction of the ar, cp, pd, and ch ligands by facilitating comparison of the structural parameters for the mixed complexes with those for the bis(ligand) complexes. Three parameters may be considered as measures of the iron–ligand interaction: average Fe–C distance, Fe–ligand plane distance, and average C–C ring distance. One might initially expect Fe–C bond distance to be the most effective measure of Fe–ligand interaction. However, as will become clear, this does not necessarily appear to be the case for this study. For example, comparison of the iron–carbon distances in Fepd_2 , ferrocene and Fepdcp complexes indicates that in the mixed complex the pd ligand is 0.03 Å closer than it is in Fepd_2 whereas the cp ligand is the same distance as those in ferrocene. Therefore, there is a suggestion that pd interacts more favourably with iron(II) than cp; the conclusion reached earlier by Ernst et. al. [6] from their investigation of Fepdcp [21*].

A fact that must be considered, however, is that as the ligand plane approaches the Fe atom the C–C bond distances in the C_6 ring increase (as one would expect if increased π -back bonding from the metal to a ligand antibonding orbital occurs) thereby partially offsetting the decrease in the M–C bond distance. It might

* Reference number with asterisk indicates a note in the list of references

Table 2

Important bond distances (Å) and angles (°) for 9.

<i>Distances</i>			
Fe(1)–C(11)	2.055(3)	C(12)–C(13)	1.409(6)
Fe(1)–C(12)	2.059(3)	C(13)–C(14)	1.397(5)
Fe(1)–C(13)	2.060(3)	C(31)–C(32)	1.388(4)
Fe(1)–C(14)	2.063(3)	C(31)–C(36)	1.391(4)
Fe(1)–C(15)	2.060(3)	C(14)–C(15)	1.417(5)
Fe(1)–C(22)	2.088(3)	C(32)–C(33)	1.387(4)
Fe(1)–C(23)	2.027(3)	C(33)–C(34)	1.390(5)
Fe(1)–C(24)	2.050(3)	C(21)–C(22)	1.522(4)
Fe(1)–C(25)	2.028(3)	C(21)–C(26)	1.512(4)
Fe(1)–C(26)	2.091(3)	C(34)–C(35)	1.363(5)
C(24)–C(25)	1.422(4)	C(21)–C(31)	1.531(4)
C(24)–C(28)	1.514(4)	C(35)–C(36)	1.391(4)
C(25)–C(26)	1.413(4)	C(22)–C(23)	1.412(4)
C(26)–C(29)	1.509(4)	C(22)–C(27)	1.507(4)
C(11)–C(12)	1.409(6)	C(23)–C(24)	1.425(4)
C(11)–C(15)	1.395(6)		
<i>Angles</i>			
C(22)–C(21)–C(26)	100.80(20)	C(25)–C(26)–C(29)	119.7(3)
C(22)–C(21)–C(31)	114.80(21)	C(12)–C(11)–C(15)	108.4(3)
C(26)–C(21)–C(31)	118.11(22)	C(11)–C(12)–C(13)	107.6(3)
C(21)–C(22)–C(23)	118.79(23)	C(21)–C(31)–C(32)	122.51(25)
C(21)–C(22)–C(27)	118.22(23)	C(21)–C(31)–C(36)	119.70(24)
C(23)–C(22)–C(27)	119.67(24)	C(32)–C(31)–C(36)	117.8(3)
C(22)–C(23)–C(24)	120.28(24)	C(31)–C(32)–C(33)	121.2(3)
C(23)–C(24)–C(25)	116.11(24)	C(32)–C(33)–C(34)	119.8(3)
C(23)–C(24)–C(28)	121.7(3)	C(33)–C(34)–C(35)	119.7(3)
C(25)–C(24)–C(28)	121.8(3)	C(34)–C(35)–C(36)	120.3(3)
C(24)–C(25)–C(26)	120.38(25)	C(31)–C(36)–C(35)	121.1(3)
C(21)–C(26)–C(25)	119.01(24)	C(11)–C(15)–C(14)	107.8(3)
C(21)–C(26)–C(29)	117.90(25)		

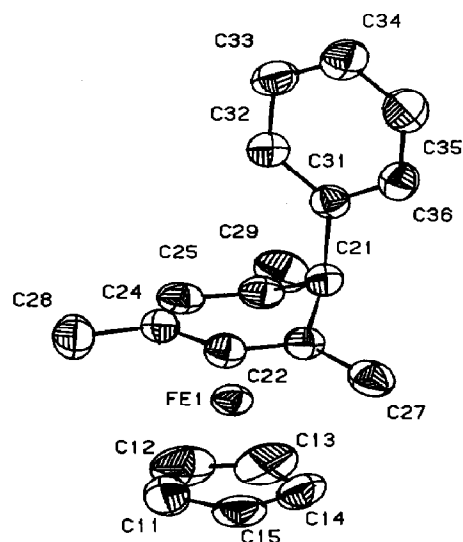
Fig. 1. SNOOPI [18] perspective view of $(C_5H_5)(C_6H_5Me_3C_6H_5)Fe$.

Table 3

Selected interatomic distances for iron(II) sandwich complexes

Complex	Type ^a	Average M-C distance (Å)			Average C-C distance (Å)			M-C plane distance (Å)			Reference
		cp	ar	ch	cp	ar	ch	cp	ar	ch	
(C ₅ H ₅) ₂ Fe	A			2.05(2)			1.40(4)			1.66	25
[(C ₈ H ₃ Me ₃) ₂ Fe](PF ₆) ₂	B			2.13(1)			1.39(2)			1.615(4)	26
(t-Butyl-C ₆ H ₃ Me ₃) ₂ Fe	C			2.09(4)			1.409(6)			1.571(3)	27
(C ₆ H ₅ -C ₆ H ₃ Me ₃) ₂ Fe	C			2.07(4)			1.41(1)			1.55	28
(2,3,4-C ₆ H ₁₃) ₂ Fe	D			2.087(1)			1.412(2)			1.46(1)	29
(2,4-C ₇ H ₁₁) ₂ Fe	D			2.089(1)			1.411(8)			1.458(8)	30

Complex	Type ^a	Average M-C Distance (Å)			Average C-C Distance (Å)			M-C Plane Distance (Å)			Ref.
		cp	ar	ch	cp	ar	ch	cp	ar	ch	
[(C ₅ H ₅)(C ₆ Et ₆)Fe]PF ₆	E	2.05(1)	2.11(2)	-	1.38(1)	1.43(1)	-	1.683(1)	1.55(1)	-	7
[(C ₅ H ₅)(C ₆ Et ₆)Fe]BPh ₄	E	2.05(1)	2.10(1)	-	1.40(1)	1.423(9)	-	1.68	1.54	-	8
(C ₅ H ₅)(C ₆ Me ₆)Fe	F	2.144(2)	2.100(7)	-	^b	^b	-	1.79(1)	1.58(1)	-	31
(C ₅ H ₅)(C ₆ H ₃ (CF ₃) ₃ O)CH ₃]Fe	G	2.06(1)	-	2.05(2)	1.397(12)	-	1.415(5)	1.68	-	1.52	11
(C ₅ H ₅)(C ₆ H ₃ Me ₃ C ₆ H ₃)Fe	G	2.059(3)	-	2.06(3)	1.405(9)	-	1.418(6)	1.677(2)	-	1.526(1)	this study
(C ₅ H ₅)(2,4-C ₇ H ₁₁)Fe	H	2.05(1)	-	2.06(2)	1.37(6)	-	-	1.43(1)	1.69	-	1.42 21
[(C ₆ H ₃ Me ₃ Et)(C ₆ H ₃ Me ₃)Fe]PF ₆	I	-	2.11(2)	2.09(5)	-	1.41(1)	1.41(2)	-	1.570(4)	1.586(4)	9
[(C ₆ H ₃ Me ₃ CH ₂ Cl)(C ₆ H ₃ Me ₃)Fe]PF ₆	I	-	2.11(2)	2.09(5)	-	1.40(1)	1.42(1)	-	1.555(4)	1.570(4)	10

^a A = FeCP₂, B = Fear₂²⁺, C = FeCh₂, D = FePd₂, E = Fearcp⁺, F = 19e⁻ Fearcp, G = FeChcp, H = FePdcp, I = Fechar⁺. ^b Information not available.

therefore be anticipated that the metal–ligand plane distance would be a more effective measure of metal–ligand interaction as it should be less influenced by changes in C–C bond distance. The Fe–cp plane distance for **9** is 0.02 Å greater than that observed for ferrocene whereas the Fe–ch plane distance is 0.02–0.045 Å less than that seen for two Fech₂ complexes. The Fe–C bond distances exhibit a similar trend but it is not as extreme. Similarly, in mixed complexes containing the cp and ar ligands the cp ligand plane moves away from the metal vs. ferrocene (by as much as 0.13 Å in the 19-electron complex cp(C₆Me₆)Fe) while the M–ar plane distance contracts by 0.03 Å vs. Fear₂²⁺. Finally, in [Fearch]⁺ complexes the Fe–ch plane distance increases by up to 0.03 Å vs. Fech₂, while the Fe–ar plane distance decreases by as much as 0.06 Å from that of the Fear₂²⁺ complexes. Similar trends are observed for Fepdcp vs. ferrocene and Fepd₂, with the Fe–plane distances showing a greater change than the Fe–C bond distances. The metal plane distance therefore does indeed appear to be the most sensitive measure of Fe–ligand interaction.

Evaluation of these solid-state X-ray structural results therefore leads us to rank the ligands in following order of metal–ligand interaction: ar > ch ~ pd > cp. Although we have not considered the steric differences between the four ligands, particularly the effects of 1,5 substituents in the ch complexes, one should note that the order suggested above corresponds to the relative susceptibility of the bis(ligand)Fe complexes to undergo reduction and/or nucleophilic addition. In this context Fear₂²⁺ complexes are known to be quite reactive ($E^\circ \approx -0.45$ V), Fearch⁺ and Fearcp⁺ complexes undergo attack at the arene ring ($E^\circ \approx -1.55$ V), and Fecp₂ is relatively unreactive [19]. Furthermore, this order is consistent with the $\nu(\text{CO})$ values observed for [arMn(CO)₃]⁺ [22], chMn(CO)₃ [22] and cpMn(CO)₃ [23] complexes, which demonstrate that the latter two complexes vibrate at significantly lower wavenumber than the former. However, Table 3 is incomplete and current work in our laboratory is focusing on completing the table via the synthesis of Fearpd⁺ and Fepdch complexes.

Experimental

Synthesis

1.00 g (2.59 mmol) of [(C₅H₅)(C₆H₃Me₃)Fe]PF₆ was partially dissolved in 30 ml of freshly distilled THF, degassed, and placed under a nitrogen atmosphere. The reaction vessel was then cooled to 0 °C and, under a stream of N₂, 5.18 ml (10.4 mmol) of 2 M phenyllithium solution was added. The mixture was then allowed to warm to room temperature at which point the solvent was removed under reduced pressure and the residue extracted with hexanes. The orange hexanes solution was stirred vigorously with 2 ml of H₂O, dried over anhydrous MgSO₄, and filtered. The hexanes fraction was then concentrated under reduced pressure yielding 0.76 g (92%) of a red oil which crystallized upon cooling. M.p. 99–100 °C. ¹H NMR (C₆D₆): δ 7.08(m, 2H), 6.98(m, 1H), 6.83(m, 2H), 3.87(s, 5H), 3.78(s, 2H), 2.25(s, 3H), 2.02(s, 1H), 1.42(s, 6H); ¹³C NMR (C₆D₆): δ 143.7s, 129.6d, 126.3d, 126.1d, 89.0s, 79.0d, 75.1d, 54.8d, 42.2s, 24.5q, 21.0q.

Cooling of a hexanes solution of **9** to –15 °C afforded moderately air- and solution-sensitive crystals suitable for X-ray crystallography.

Table 4

Crystallographic data collection and structure refinement parameters for $[(C_5H_5)(C_6H_3Me_3C_6H_5)Fe]$

Formula	FeC ₂₀ H ₂₂
Formula wt.	318.24
Radiation, λ	Mo-K α , 0.70926 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
a , Å	8.5703(5)
b , Å	8.5283(9)
c , Å	21.8436(23)
β , °	93.868(6)
V , Å ³	1592.91
Z	4
D_{calc} , g cm ⁻³	1.327
μ , mm ⁻¹	0.94
Crystal dimensions, mm	0.54 × 0.39 × 0.21
Scan type	$\theta - 2\theta$
2θ range, °	4–50
Reflections measured	3025
Reflections observed ^a	2046
Parameters refined	191
Ratio data/parameter	10.7
R	0.035
R_w	0.049
GOF	1.61

^a $I \geq 2.5\sigma(I)$.

X-ray crystallography

An orange crystal of **9** was mounted and sealed in a thin-walled glass capillary and placed on an Enraf-Nonius CAD-4 diffractometer. Cell dimensions were calculated from 33 reflections with 2θ angles in the range 30.00–35.00° and are listed along with other pertinent crystallographic parameters in Table 4. The structure was solved via heavy-atom methods and refined using the NRC VAX Crystal Structure System [24] locally adapted for a Wicat Systems S-1260 computer. Refinement was based on $R = \sum |F_o| - |F_c| / \sum |F_o|$ and $R_w = [\sum (w(F_o - F_c)^2) / \sum wF_o^2]^{1/2}$ and converged at $R = 0.035$ and $R_w = 0.049$ with all non-hydrogen atoms refined with anisotropic thermal parameters. Hydrogen atoms were placed in calculated positions ($d(C-H)$ 1.08 Å) and given isotropic thermal parameters based upon the carbon atoms to which they are bonded. Weights based on counting statistics were used, the weight modifier k in kF_o^2 being 0.0005. No corrections were made for extinction or absorption. Atomic scattering factors and corrections for anomalous dispersion for Fe, C, and H were taken from: International Tables for X-ray Crystallography; Kynoch Press: Birmingham, England; 1974; distributor D. Reidel, Dordrecht, The Netherlands.

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