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both complexes, O—C bonds of the coordinated ring are shorter than those of the uncoordinated ring.

Comment

The title complexes were synthesized, following a previously described procedure (Sutherland, Piórko, Gill & Lee, 1982), by the reaction of catechol with (η^5 -1,2-dichlorobenzene)(η^5 -Cp)iron(II) hexafluorophosphate and with (η^6 -2,3-dichlorotoluene)(η^5 -Cp)iron(II) hexafluorophosphate to give (1) and (2), respectively. Crystals of both complexes were grown from acetone–dichloromethane–diethyl ether solutions at ca 255 K. Crystals of complex (2) contain molecules of diethyl ether in the complex:solvent ratio 2:1. Results obtained in the course of this study add to earlier studies on the synthesis, reactivity and, in particular, the structure of tricyclic heterocycles complexed with an FeCp moiety. It had been noted earlier that the effect of complexing iron, the presence of substituents on the heterocycle and the packing forces in the crystal all may influence the overall geometry of the heterocycle. Our results provide additional information on the influence of the above factors on the structure of the heterocycle.

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Hexafluorophosphate Salts of Cyclopentadienyliron(II) Complexes of Dibenzodioxin and 1-Methyldibenzodioxin

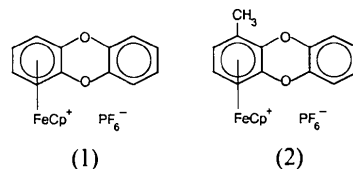
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Abstract

The structures of (η^5 -cyclopentadienyl)[(1,2,3,4,4a,10a- η^6)-dibenzo[*b,e*][1,4]dioxin]iron(II) hexafluorophosphate (1), [Fe(C₅H₅)(C₁₂H₈O₂)]PF₆, and (η^5 -cyclopentadienyl)[(1,2,3,4,4a,10a- η^6)-1-methyldibenzo[*b,e*][1,4]-dioxin]iron(II) hexafluorophosphate diethyl ether solvate (2), [Fe(C₅H₅)(C₁₃H₁₀O₂)]PF₆·0.5C₄H₁₀O, have been determined. The dihedral angle between the outer rings of dibenzodioxin was found to be 175.8 (2)° in (1) and 177.55 (19)° in (2). The planes of the coordinated arene and cyclopentadienyl (Cp) rings are nearly parallel for both complexes. The Fe atom is centered above the Cp ring while Fe—C distances involving the quaternary C atoms of the coordinated arene rings are slightly longer than those involving the other aromatic C atoms, particularly for complex (2). In the central dioxin ring of



Many of the structural features of complexes (1) and (2) resemble those reported for the FeCp complex of 2-methyldibenzodioxin (Abboud, Simonsen, Piórko & Sutherland, 1991). The Fe atom is centered above the Cp ring in both complexes, while bonding to the benzene ring is asymmetrical. The longest Fe—C distances are found for the quaternary C atoms [average 2.107 (6) versus 2.091 (6) Å for the other Fe—C distances in (1) and 2.104 (5) versus 2.079 (5) Å for the other Fe—C distances in (2)]. This phenomenon has been reported earlier for similar complexes (Abboud, Simonsen, Piórko & Sutherland, 1991; Simonsen, Lynch, Sutherland & Piórko, 1985; Sutherland, Piórko, Lee, Simonsen & Lynch, 1988; Lynch, Thomas, Simonsen, Piórko & Sutherland, 1986). The C—C distances within the coordinated rings are slightly longer than those of the uncoordinated rings [average 1.411 (10) versus 1.380 (11) Å in (1) and 1.410 (7) versus 1.383 (12) Å in (2)]. Asymmetry of the central dioxin ring, exemplified by the shortening of the O—C bonds of the coordinated ring, was noted earlier for the FeCp complex of 2-methyldibenzodioxin (Abboud, Simonsen, Piórko & Sutherland, 1991) and is also evident in (1) and (2) [average 1.364 (7) Å for O—C(coordinated) versus 1.399 (7) Å for the others in (1) and 1.367 (6) Å for O—C(coordinated) versus 1.393 (6) Å for the others in

(2)]. The distances between Fe and the Cp ring planes [1.663 (4) for (1) and 1.665 (4) Å for (2)] and between Fe and the coordinated arene ring planes [1.551 (3) for (1) and 1.5454 (21) Å for (2)] are within the range reported for FeCp complexes of arenes (Zaworotko, Sturge & White, 1990; Houlton, Roberts, Silver, Wells & Frampton, 1992). The planes of the coordinated arene rings and Cp rings are nearly parallel [2.1 (3) and 0.8 (8)° for (1) and (2), respectively; within the range of values reported for similar complexes]. The dibenzodioxin ligand in both complexes is slightly folded and the complexing FeCp moiety is located inside the fold in each case. This phenomenon has been observed for all the FeCp complexes of tricyclic heterocycles reported so far with the single exception of the thianthrene complex (Abboud, Simonsen, Piórko & Sutherland, 1991; Abboud, Lynch, Simonsen, Piórko & Sutherland, 1990; Sutherland, Piórko, Lee, Simonsen & Lynch, 1988; Lynch, Thomas, Simonsen, Piórko & Sutherland, 1986; Simonsen, Lynch, Sutherland & Piórko, 1985; Christie, Piórko & Zaworotko, 1994; Piórko, Christie & Zaworotko, 1994). The dihedral angle between the outer rings was found to be 175.8 (2) for (1) and 177.55 (19)°

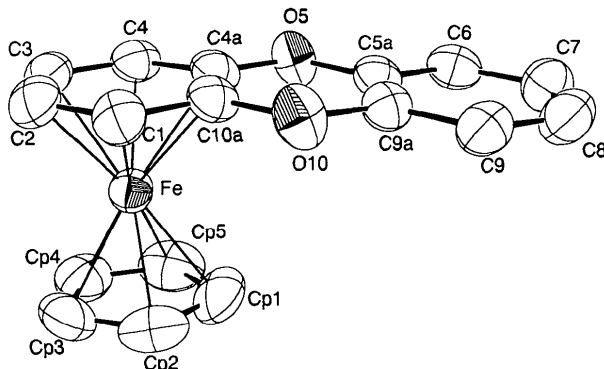


Fig. 1. ORTEP plot (Johnson, 1965) of the cation of complex (1) showing the numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

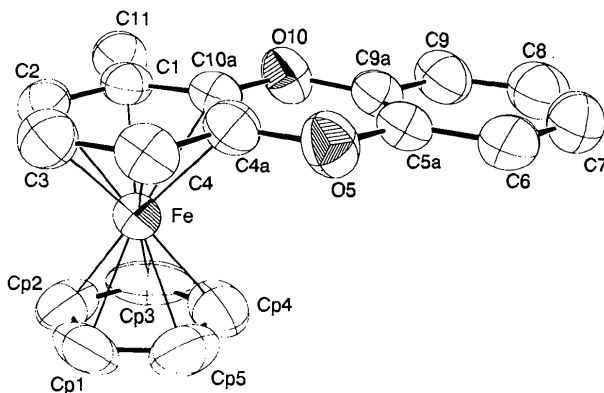


Fig. 2. ORTEP plot (Johnson, 1965) of the cation of complex (2) showing the numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

for (2). These values are comparable to those found for the FeCp complexes of both 2-methyldibenzodioxin [173.8 (1)°; Abboud, Simonsen, Piórko & Sutherland, 1991] and η^6 -[1,4]benzodioxino[2,3-*b*]pyridine [177.4 (3)°; Piórko, Christie & Zaworotko, 1994], and also for 2,7,8-trinitro-1,4-benzodioxino[2,3-*b*]pyridine [174.65 (8)°; Piórko, Christie, Crook & Sampson, 1994], 2,8-dichlorodibenzodioxin [175.2°; Boer, Neuman & Aniline, 1972] and 1,2,3,7,8,9-hexachlorodibenzodioxin [175.7°; Cantrell, Webb & Mabis, 1969], while dibenzodioxin (Senma, Taira, Taga & Osaki, 1973; Singh & McKinney, 1978), 2,7-dichlorodibenzodioxin (Boer & North, 1972), 2,3,7,8-tetrachlorodibenzodioxin (Boer, van Remoortere, North & Neuman, 1972), octachlorodibenzodioxin (Neuman, North & Boer, 1972) and octafluorodibenzodioxin (Rainville, Zingaro & Meyers, 1980) are planar. The above data seem to indicate that FeCp complexation and the introduction of substituents into the dibenzodioxin ring may result in deviation of the molecule from planarity. Such a limited deviation, presumably restricted by the electronic structure of the O atoms, might indicate a trend which would be opposite to that found for other tricyclic heterocycles (for discussion see Abboud, Simonsen, Piórko & Sutherland, 1991; Lynch, Simonsen, Martin, Puig-Torres & Smith, 1984; Hossain, Dwiggins, Sen Gupta, Turley & Martin, 1982; Womack, Turley, Martin, Kimura & Simonsen, 1981).

Experimental

Complex (1)

Crystal data

[Fe(C₅H₅)(C₁₂H₈O₂)]PF₆

M_r = 450.10

Monoclinic

*P*2₁/*n*

a = 11.0301 (14) Å

b = 13.7114 (8) Å

c = 11.6012 (12) Å

β = 108.603 (6)°

V = 1662.9 (3) Å³

Z = 4

D_x = 1.80 Mg m⁻³

Mo *K*α radiation

λ = 0.7107 Å

Cell parameters from 24 reflections

θ = 17.5–22.5°

μ = 1.07 mm⁻¹

T = 290 K

Cube

0.40 × 0.30 × 0.30 mm

Yellow–green

Data collection

Enraf–Nonius CAD-4

diffractometer

$\omega/2\theta$ scans

Absorption correction:

empirical

T_{min} = 0.914, *T_{max}* = 1.00

3072 measured reflections

2914 independent reflections

2281 observed reflections

[*I* > 2.5σ(*I*)]

R_{int} = 0.012

θ_{\max} = 22.5°

h = -13 → 12

k = 0 → 16

l = 0 → 13

3 standard reflections

frequency: 60 min

intensity variation: ±1%

<i>Refinement</i>		F(3)	0.6587 (3)	0.5046 (4)	0.0558 (7)	13.2 (5)
<i>Refinement on F</i>	$w = 1/[\sigma^2(F_o) + 0.000005(F_o)^2]$	F(4)	0.7817 (7)	0.4890 (4)	-0.0483 (5)	12.1 (5)
$R = 0.061$		F(5)	0.9330 (5)	0.5658 (5)	0.0965 (11)	21.5 (9)
$wR = 0.061$	$(\Delta/\sigma)_{\max} = 0.109$	F(6)	0.7550 (6)	0.6348 (3)	0.0195 (5)	10.7 (4)
$S = 5.38$	$\Delta\rho_{\max} = 0.810 \text{ e } \text{\AA}^{-3}$	O(5)	0.0886 (4)	0.8765 (3)	-0.0618 (4)	4.2 (2)
2281 reflections	$\Delta\rho_{\min} = -0.630 \text{ e } \text{\AA}^{-3}$	O(10)	0.2475 (4)	1.0132 (3)	0.1048 (4)	4.4 (2)
244 parameters	Atomic scattering factors	C(1)	0.3316 (6)	0.8856 (5)	0.2441 (5)	4.1 (3)
H-atom parameters not refined	from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)	C(2)	0.3346 (7)	0.7856 (5)	0.2744 (6)	4.8 (4)
		C(3)	0.2574 (7)	0.7177 (5)	0.1923 (6)	4.6 (3)
		C(4)	0.1737 (6)	0.7489 (4)	0.0776 (6)	4.1 (3)
		C(4a)	0.1688 (6)	0.8486 (4)	0.0496 (6)	3.9 (3)
		C(5a)	0.0940 (6)	0.9746 (4)	-0.0912 (6)	3.5 (3)
		C(6)	0.0161 (6)	1.0036 (5)	-0.2046 (6)	4.2 (3)
		C(7)	0.0185 (7)	1.1022 (5)	-0.2363 (6)	4.8 (4)
		C(8)	0.0975 (7)	1.1682 (5)	-0.1570 (7)	4.9 (4)
		C(9)	0.1729 (6)	1.1371 (4)	-0.0453 (6)	4.1 (3)
		C(9a)	0.1703 (6)	1.0405 (4)	-0.0125 (5)	3.5 (3)
		C(10a)	0.2463 (6)	0.9168 (4)	0.1321 (6)	3.7 (3)
		Cp(1)	0.4328 (9)	0.8562 (6)	-0.0223 (8)	6.3 (5)
		Cp(2)	0.5262 (7)	0.8600 (6)	0.0929 (8)	5.7 (4)
		Cp(3)	0.5483 (7)	0.7652 (7)	0.1368 (7)	6.2 (5)
		Cp(4)	0.4693 (8)	0.7017 (5)	0.0495 (8)	5.9 (5)
		Cp(5)	0.3983 (8)	0.7581 (7)	-0.0484 (7)	6.2 (5)
Complex (2)						
<i>Crystal data</i>						
[Fe(C ₅ H ₅)(C ₁₃ H ₁₀ O ₂)]PF ₆ ·0.5C ₄ H ₁₀ O	Mo K α radiation					
$M_r = 501.22$	$\lambda = 0.7107 \text{ \AA}$					
Monoclinic	Cell parameters from 24 reflections					
$C2/c$	$\theta = 17-20^\circ$					
$a = 18.027 (9) \text{ \AA}$	$\mu = 0.87 \text{ mm}^{-1}$					
$b = 11.706 (5) \text{ \AA}$	$T = 290 \text{ K}$					
$c = 19.845 (4) \text{ \AA}$	Parallelepiped					
$\beta = 99.81 (3)^\circ$	$0.75 \times 0.50 \times 0.45 \text{ mm}$					
$V = 4127 (3) \text{ \AA}^3$	Yellow					
$Z = 8$						
$D_x = 1.61 \text{ Mg m}^{-3}$						
<i>Data collection</i>						
Enraf-Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.017$					
$\omega/2\theta$ scans	$\theta_{\max} = 20^\circ$					
Absorption correction: empirical	$h = -19 \rightarrow 19$					
$T_{\min} = 0.653, T_{\max} = 1.00$	$k = 0 \rightarrow 12$					
2782 measured reflections	$l = 0 \rightarrow 21$					
2684 independent reflections	3 standard reflections					
2173 observed reflections	frequency: 60 min					
$[I > 2.5\sigma(I)]$	intensity variation: -10%					
<i>Refinement</i>						
<i>Refinement on F</i>	$w = 1/[\sigma^2(F_o) + 0.000100(F_o)^2]$					
$R = 0.042$						
$wR = 0.052$	$(\Delta/\sigma)_{\max} = 0.000$					
$S = 3.16$	$\Delta\rho_{\max} = 0.540 \text{ e } \text{\AA}^{-3}$					
2173 reflections	$\Delta\rho_{\min} = -0.480 \text{ e } \text{\AA}^{-3}$					
276 parameters	Atomic scattering factors					
H-atom parameters not refined	from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)					

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$B_{\text{eq}} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$				
Compound (1)	x	y	z	B_{eq}
Fe	0.36010 (9)	0.80092 (6)	0.10488 (7)	3.39 (4)
P	0.7999 (2)	0.53409 (14)	0.07874 (18)	4.98 (11)
F(1)	0.8369 (7)	0.4313 (4)	0.1312 (5)	5.1 (5)
F(2)	0.8083 (10)	0.5798 (5)	0.1989 (5)	17.1 (7)

Table 2. Selected geometric parameters ($\text{\AA}, ^\circ$)

	(1)	(2)
Fe—C(1)	2.093 (6)	2.107 (5)
Fe—C(2)	2.084 (6)	2.076 (5)
Fe—C(3)	2.085 (6)	2.080 (5)
Fe—C(4)	2.103 (6)	2.082 (5)
Fe—C(4a)	2.104 (6)	2.104 (5)
Fe—C(10a)	2.110 (6)	2.103 (4)
Fe—Cp(1)	2.037 (7)	2.044 (6)
Fe—Cp(2)	2.048 (7)	2.036 (5)
Fe—Cp(3)	2.048 (7)	2.032 (5)
Fe—Cp(4)	2.051 (7)	2.039 (6)
Fe—Cp(5)	2.041 (7)	2.033 (6)
Cp(1)—Cp(5)	1.405 (13)	1.353 (14)
Cp(1)—Cp(2)	1.404 (13)	1.375 (11)
Cp(2)—Cp(3)	1.389 (13)	1.401 (12)
Cp(3)—Cp(4)	1.407 (13)	1.408 (14)
Cp(4)—Cp(5)	1.391 (13)	1.357 (15)

P—F(1)	1.538 (5)	1.586 (3)
P—F(2)	1.505 (5)	1.575 (3)
P—F(3)	1.548 (6)	1.569 (4)
P—F(4)	1.551 (5)	1.566 (3)
P—F(5)	1.481 (6)	1.577 (3)
P—F(6)	1.552 (5)	1.557 (4)
C(1)—C(2)	1.413 (9)	1.423 (7)
C(2)—C(3)	1.408 (10)	1.414 (7)
C(3)—C(4)	1.423 (10)	1.410 (7)
C(4)—C(4a)	1.402 (9)	1.403 (7)
C(4a)—C(10a)	1.414 (9)	1.406 (7)
C(1)—C(10a)	1.406 (9)	1.403 (6)
O(5)—C(4a)	1.368 (7)	1.365 (6)
O(5)—C(5a)	1.393 (7)	1.396 (6)
O(10)—C(9a)	1.406 (7)	1.389 (6)
O(10)—C(10a)	1.360 (7)	1.369 (6)
C(5a)—C(6)	1.381 (9)	1.381 (7)
C(6)—C(7)	1.404 (10)	1.394 (10)
C(7)—C(8)	1.382 (11)	1.368 (12)
C(8)—C(9)	1.366 (10)	1.388 (9)
C(9)—C(9a)	1.382 (8)	1.387 (7)
C(5a)—C(9a)	1.367 (8)	1.381 (8)
C(1)—C(11)		1.471 (7)
O(5)—C(15)		1.411 (9)
O(5)—C(15')		1.411 (9)
C(15)—C(25)		1.298 (16)
C(4a)—O(5)—C(5a)	115.9 (4)	115.8 (4)
C(9a)—O(10)—C(10a)	115.8 (4)	116.5 (4)
C(2)—C(1)—C(10a)	118.7 (6)	118.5 (4)
C(1)—C(2)—C(3)	120.8 (6)	121.3 (4)
C(2)—C(3)—C(4)	120.4 (6)	119.7 (4)
C(3)—C(4)—C(4a)	118.4 (6)	119.5 (4)
O(5)—C(4a)—C(4)	117.2 (5)	116.9 (4)
O(5)—C(4a)—C(10a)	121.5 (5)	122.7 (4)
C(4)—C(4a)—C(10a)	121.2 (6)	120.3 (4)
O(5)—C(5a)—C(6)	116.7 (5)	117.2 (5)
O(5)—C(5a)—C(9a)	122.7 (5)	121.7 (4)
C(6)—C(5a)—C(9a)	120.6 (6)	121.1 (5)
C(5a)—C(6)—C(7)	118.0 (6)	118.0 (6)
C(6)—C(7)—C(8)	121.1 (6)	120.9 (5)
C(7)—C(8)—C(9)	119.4 (6)	121.1 (6)
C(8)—C(9)—C(9a)	120.0 (6)	118.2 (5)
O(10)—C(9a)—C(5a)	121.6 (5)	122.0 (4)
O(10)—C(9a)—C(9)	117.5 (5)	117.3 (5)
C(5a)—C(9a)—C(9)	120.8 (6)	120.7 (5)
O(10)—C(10a)—C(1)	117.2 (5)	117.0 (4)
O(10)—C(10a)—C(4a)	122.3 (5)	121.2 (4)
C(1)—C(10a)—C(4a)	120.4 (6)	121.8 (4)
C(2)—C(1)—C(11)		122.0 (4)
C(10a)—C(1)—C(11)		120.5 (4)
C(15)—O(5)—C(15')		116.6 (9)
O(5)—C(15)—C(25)		116.2 (9)

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

Single crystals of compounds (1) and (2) were mounted independently, sealed in separate glass capillaries and then optically centered in the X-ray beam of the diffractometer. The standard reflections were measured to monitor both crystal and electronic stability. The structures were solved by direct methods and refined using full-matrix least-squares techniques. Ring H atoms and methylene H atoms were placed in calculated positions (C—H = 1.08 Å) and assigned isotropic displacement factors equivalent to those calculated for the C atoms to which they are bonded. Methyl H atoms were located *via* inspection of difference Fourier maps and were fixed to the attached C atom. Computer program used was *NRCVAX* (Gabe, Le Page, Charland, Lee & White (1989)).

AP acknowledges the financial support of Saint Mary's University through Senate and General Research Grants.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, least-squares-planes data and unit-cell packing diagrams have been deposited with the IUCr (Reference: BK1012). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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