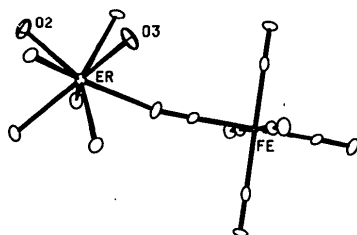


Table 2. *Interatomic distances (Å) and bond angles (°) with e.s.d.'s in parentheses*

Er—N(1)	2.505 (4)	Er—N(1)—C(1)	166.9 (4)
Er—N(2)	2.522 (4)	Er—N(2)—C(2)	149.4 (4)
Er—N(3)	2.511 (4)	Er—N(3)—C(3)	166.8 (4)
Av.	2.513 (7)	Av.	161 (8)
Er—O(2)	2.408 (5)		
Er—O(3)	2.416 (5)		
Av.	2.412 (4)		
Fe—C(1)	1.938 (5)	Fe—C(1)—N(1)	177.3 (4)
Fe—C(2)	1.935 (5)	Fe—C(2)—N(2)	178.8 (4)
Fe—C(3)	1.928 (5)	Fe—C(3)—N(3)	178.1 (4)
Av.	1.934 (4)	Av.	178.1 (6)
C(1)—N(1)	1.141 (6)	C(1)—Fe—C(2)	90.7 (2)
C(2)—N(2)	1.144 (6)	C(1)—Fe—C(3)	90.4 (2)
C(3)—N(3)	1.145 (6)	C(2)—Fe—C(3)	90.8 (2)
Av.	1.143 (2)	Av.	90.6 (2)
O(1)—O(3)	2.811 (5)		

Fig. 1. $\text{ErFe}(\text{CN})_6 \cdot 4\text{H}_2\text{O}$. The square antiprism geometry about Er and the octahedrally arranged cyano groups about Fe. Cyanide bridging links the $\text{ErN}_6(\text{H}_2\text{O})_2$ and FeC_6 groups.

Related literature. Bailey, Williams & Milligan (1973) reported the first single-crystal X-ray diffraction analysis [$\text{LaFe}(\text{CN})_6 \cdot 5\text{H}_2\text{O}$, hexagonal, $P6_3/m$] of the $\text{LnFe}(\text{CN})_6 \cdot n\text{H}_2\text{O}$ series. In the same year, Hulliger, Landolt & Vetsch (1973) reported the $\text{LnT}(\text{CN})_6 \cdot 4\text{H}_2\text{O}$ series ($T = \text{Fe}, \text{Co}$) to have orthorhombic symmetry (nearly pseudo-hexagonal) and Kietabile & Petter (1974), using single crystals from the Hulliger group, reported that $\text{SmFe}(\text{CN})_6 \cdot 4\text{H}_2\text{O}$ was orthorhombic ($Cmcm$). Hulliger *et al.* (1976) reported additional structural and magnetic data for $\text{LnT}(\text{CN})_6 \cdot n\text{H}_2\text{O}$ ($T = \text{Cr}, \text{Co}, \text{Fe}; n = 4, 5$). The pentahydrate forms were classified as hexagonal

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Structure of an Iridium-Containing Hydrogenation Catalyst

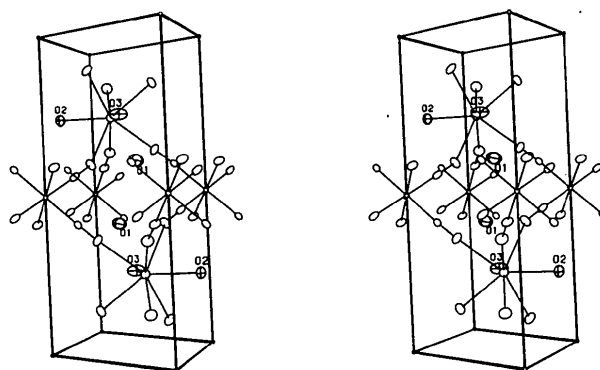
BY M. S. ABBASSIOUN, P. B. HITCHCOCK AND P. A. CHALONER

School of Chemistry and Molecular Sciences, University of Sussex, Falmer, Brighton, England

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Abstract. (η^4 -1,5-Cyclooctadiene)(pyridine)(tricyclohexylphosphine)iridium(I) hexafluorophosphate, $[\text{Ir}(\text{C}_8\text{H}_{12})(\text{C}_5\text{H}_5\text{N})\{\text{P}(\text{C}_6\text{H}_{11})_3\}][\text{PF}_6]$, $M_r = 804.9$, mono-

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Fig. 2. A stereoscopic view of the contents of the unit cell (a axis horizontal, b axis vertical, c axis into plane of paper). The Er^{3+} and Fe^{3+} ions are shown fully coordinated. O(1) atoms associated with the zeolitic water molecules are seen within the $\text{Er}-\text{N}\equiv\text{C}-\text{Fe}$ network.

and the tetrahydrate forms as orthorhombic. This work was based on powder work. Recent single-crystal structure analyses have revealed that Bi and $\text{SmFe}(\text{CN})_6 \cdot 4\text{H}_2\text{O}$ best fit a monoclinic system ($P2_1/m$) and not hexagonal or orthorhombic (Perkins, 1986; Mullica, Perkins & Sappenfield, 1988).

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clinic, $P2_1$, $a = 9.625 (6)$, $b = 16.440 (8)$, $c = 10.383 (5) \text{ \AA}$, $\beta = 103.13 (4)^\circ$, $V = 1600.1 \text{ \AA}^3$, $Z = 2$, $D_x = 1.67 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71069 \text{ \AA}$, $\mu =$

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43.1 cm⁻¹, $F(000) = 808$, $T = 295$ K. $R = 0.044$ and $wR = 0.056$ for 2426 reflections with $|F^2| > \sigma(F^2)$. The geometry at iridium is approximately square planar, with the expected distortions due to the size of the Cy₃P ligand: Ir–P(1) 2.366 (4), Ir–N 2.064 (11) Å, P(1)–Ir–N = 93.0 (4), M1–Ir–P(1) = 94.0, M1–Ir–M2 = 87.3, M2–Ir–N = 85.8° [M1 and M2 are the midpoints of the C(1)–C(2) and C(5)–C(6) bonds]. One of the angles at phosphorus is appreciably larger than the other two [110.8 (7), 103.1 (7) and 101.6 (6)°], for reasons which are not readily apparent.

Experimental. The complex was prepared by the addition of tricyclohexylphosphine to [Ir(cod)(py)₂][PF₆] (Crabtree & Morehouse, 1986) and well formed crystals obtained by diffusion of ether into a solution in dichloromethane. Data collected using a crystal *ca* 0.4 × 0.4 × 0.1 mm, Enraf–Nonius CAD-4 diffractometer, monochromated Mo K α radiation, in the θ – 2θ mode, with $\Delta\theta = (1.0 + 0.35 \tan\theta)^\circ$ and a maximum scan time of 1 min. Cell parameters from 25 reflections with $15 < 2\theta < 20^\circ$. A total of 2937 unique reflections were measured for $2 < \theta < 25^\circ$ and h 0→11, k 0→19, l –12→12, and 2426 reflections with $|F^2| > \sigma(F^2)$ were used in the refinement where $\sigma(F^2) = [\sigma^2(I) + (0.04I)^2]^{1/2}/Lp$. There was no crystal decay. An absorption correction was applied using *DIFABS* (Walker & Stuart, 1983) after isotropic refinement, and gave maximum and minimum corrections of 1.63 and 0.79. The structure was solved by routine heavy-atom methods and refined by full-matrix least squares on F with non-H atoms anisotropic (369 variables). The origin was defined by fixing the Ir y coordinate. H atoms were fixed at calculated positions with $B_{iso} = 1.3B_{eq}$ for the C atoms to which they are bonded. The weighting scheme was $w = 1/\sigma^2(F)$ and the final residuals were $R = 0.044$ (0.050) and $wR = 0.056$ (0.063), the values in parentheses being for the opposite absolute structure, $(\Delta/\sigma)_{max} = 0.02$, $\Delta\rho_{max} = 1.3$ (near Ir), $\Delta\rho_{min} = -2.3$ e Å⁻³. Programs from the Enraf–Nonius (1982) *SDP-Plus* package were run on a MicroVAX computer. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Atomic parameters are given in Table 1,* intramolecular bond distances and angles in Table 2. Fig. 1 shows the molecular structure and the numbering scheme.

Related literature. This complex has proved to be of considerable significance as a hydrogenation catalyst

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$$B_{eq} = \frac{2}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Ir	8613.0 (4)	2500	5409.5 (4)	1.855 (7)
P(1)	8849 (4)	1975 (2)	7570 (4)	1.67 (7)
P(2)	2988 (6)	576 (3)	4132 (5)	4.6 (1)
F(1)	1791 (16)	456 (11)	2802 (16)	8.4 (4)
F(2)	3046 (19)	–377 (9)	4116 (21)	9.9 (6)
F(3)	4124 (15)	670 (11)	3273 (14)	8.2 (4)
F(4)	4101 (22)	648 (19)	5423 (17)	16.3 (8)
F(5)	1805 (17)	534 (15)	4903 (14)	14.1 (5)
F(6)	2836 (22)	1498 (9)	4041 (22)	14.2 (6)
N	10473 (11)	3167 (8)	5936 (12)	3.2 (2)
C(1)	6398 (17)	2279 (6)	5015 (17)	3.1 (3)
C(2)	6984 (17)	1645 (10)	4379 (16)	3.2 (3)
C(3)	6983 (20)	1571 (10)	2927 (16)	3.3 (4)
C(4)	8305 (21)	1901 (13)	2590 (17)	4.1 (4)
C(5)	8881 (15)	2655 (11)	3392 (12)	3.0 (3)
C(6)	8035 (17)	3271 (9)	3667 (14)	2.7 (3)
C(7)	6475 (19)	3375 (10)	3185 (18)	3.4 (3)
C(8)	5622 (15)	2988 (11)	4103 (14)	2.8 (3)
C(9)	10411 (20)	3975 (9)	6341 (18)	3.9 (4)
C(10)	11613 (21)	4460 (13)	6598 (23)	5.1 (5)
C(11)	12913 (21)	4162 (16)	6530 (25)	6.0 (6)
C(12)	12984 (17)	3362 (14)	6104 (19)	4.5 (4)
C(13)	11760 (16)	2826 (11)	5837 (16)	3.5 (4)
C(14)	8116 (12)	2667 (8)	8657 (11)	2.0 (3)
C(15)	8825 (15)	3495 (9)	8849 (16)	2.7 (3)
C(16)	8277 (19)	3976 (11)	9892 (20)	3.9 (4)
C(17)	6690 (19)	4097 (11)	9555 (17)	3.6 (4)
C(18)	5936 (16)	3267 (10)	9260 (16)	2.9 (3)
C(19)	6491 (14)	2793 (9)	8203 (15)	2.4 (3)
C(20)	10749 (16)	1913 (10)	8475 (15)	2.4 (3)
C(21)	11095 (16)	1845 (11)	9966 (16)	2.8 (3)
C(22)	12721 (18)	1994 (10)	10489 (17)	3.2 (4)
C(23)	13619 (16)	1419 (11)	9907 (20)	3.4 (4)
C(24)	13208 (17)	1423 (12)	8434 (20)	3.8 (4)
C(25)	11623 (16)	1290 (10)	7844 (17)	3.1 (4)
C(26)	7897 (16)	991 (9)	7699 (14)	2.1 (3)
C(27)	8583 (19)	269 (9)	7142 (18)	3.3 (4)
C(28)	7605 (19)	–487 (10)	6997 (20)	3.9 (4)
C(29)	7358 (21)	–701 (10)	8389 (23)	4.5 (4)
C(30)	6783 (21)	25 (11)	9001 (21)	4.7 (4)
C(31)	7700 (21)	772 (11)	9070 (18)	3.8 (4)

Table 2. Intramolecular distances (Å) and angles (°) with e.s.d.'s in parentheses

Ir–P(1)	2.366 (4)	Ir–N	2.064 (11)
Ir–C(1)	2.11 (2)	Ir–C(2)	2.20 (2)
Ir–C(5)	2.182 (13)	Ir–C(6)	2.175 (15)
P(1)–C(14)	1.850 (14)	P(1)–C(20)	1.859 (15)
P(1)–C(26)	1.88 (2)	N–C(9)	1.40 (2)
N–C(13)	1.38 (2)	C(9)–C(10)	1.38 (3)
C(10)–C(11)	1.36 (3)	C(11)–C(12)	1.40 (3)
C(12)–C(13)	1.45 (2)	C(1)–C(2)	1.42 (2)
C(1)–C(8)	1.58 (2)	C(2)–C(3)	1.51 (2)
C(3)–C(4)	1.50 (3)	C(4)–C(5)	1.52 (3)
C(5)–C(6)	1.37 (2)	C(6)–C(7)	1.48 (2)
C(7)–C(8)	1.53 (3)	Ir–M1	2.032
Ir–M2	2.069		
P(1)–Ir–N	93.0 (4)	Ir–P(1)–C(14)	112.7 (4)
Ir–P(1)–C(20)	111.8 (5)	Ir–P(1)–C(26)	116.0 (4)
C(14)–P(1)–C(20)	101.1 (6)	C(14)–P(1)–C(26)	103.1 (7)
C(20)–P(1)–C(26)	110.8 (7)	Ir–N–C(9)	119 (1)
Ir–N–C(13)	120 (1)	C(9)–N–C(13)	120 (1)
N–C(9)–C(10)	121 (2)	C(9)–C(10)–C(11)	121 (2)
C(10)–C(11)–C(12)	118 (2)	C(11)–C(12)–C(13)	123 (2)
N–C(13)–C(12)	116 (2)	C(2)–C(1)–C(8)	117 (1)
C(1)–C(2)–C(3)	128 (1)	C(2)–C(3)–C(4)	113 (1)
C(3)–C(4)–C(5)	113 (2)	C(4)–C(5)–C(6)	124 (1)
C(5)–C(6)–C(7)	128 (1)	C(6)–C(7)–C(8)	113 (1)
C(1)–C(8)–C(7)	116 (1)	M1–Ir–N	172 (1)
M1–Ir–M2	87.3	M2–Ir–P(1)	178.7
M2–Ir–N	85.8	M1–Ir–P(1)	94.0

M1 and M2 are the midpoints of the C(1)–C(2) and C(5)–C(6) bonds.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, complete intramolecular distances and angles, least-squares planes and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51399 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

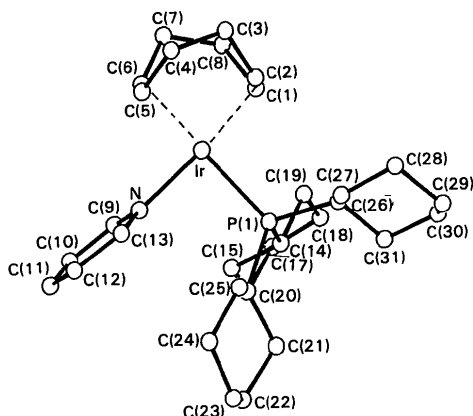


Fig. 1. Molecular structure and numbering scheme for $[\text{Ir}(\text{cod})(\text{py})(\text{PCy}_3)]^+$.

and $[\text{Ir}_3(\mu_3\text{-H})\text{H}_7(\text{py})_3(\text{PCy}_3)_3]$, formed by its deactivation, has been isolated and its structure determined (Crabtree, 1979; Crabtree & Davis, 1983). The conformations of cod in $[\{\text{IrCl}(\text{cod})\}_2]$ and $[\{\text{Ir}_2\text{-Cl}_2(\mu_2\text{-O})(\mu_2\text{-OH})_2(\text{cod})_2\}]$ (Cotton, Lahuerta, Sanau & Schwotzer, 1986) are similar to those observed here. There are many examples of structures of

$[\text{Rh}(\text{diene})\text{L}_2]^+$ complexes (Knowles, 1983; Anderson & Pignolet, 1981); the geometries about the metal are similar, although in this case there are some distortions due to the steric requirements of the phosphine ligand, resulting in the opening of the $\text{P}(1)\text{-Ir-N}$ and M1-Ir-P angles.

We thank Johnson Matthey for the loan of iridium complexes.

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Bis[ethyl(ferrocenylmethyl)dimethylammonium] Octadecahydroicosaborate(2-)

BY UPALI SIRIWARDANE, SHIRLEY S. C. CHU AND NARAYAN S. HOSMANE

Departments of Chemistry and Electrical Engineering, Southern Methodist University, Dallas, Texas 75275, USA

AND HONG ZHU AND GUOMIN ZHANG

Department of Chemistry, Wuhan University, Wuhan, The People's Republic of China

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Abstract. $[\text{C}_{15}\text{H}_{22}\text{FeN}]_2\text{B}_{20}\text{H}_{18}$, $M_r = 778.8$, monoclinic, $C2/c$, $a = 20.387$ (3), $b = 12.265$ (4), $c = 16.830$ (4) Å, $\beta = 95.162$ (16)°, $V = 4191.3$ (17) Å³, $Z = 4$, $D_x = 1.234$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu(\text{Mo } K\alpha) = 7.18$ cm⁻¹, $F(000) = 1632$, $T = 295$ K. Final $R = 0.033$ for 2834 observed reflections. The structure shows that the centrosymmetric $\text{B}_{20}\text{H}_{18}^{2-}$ ion consists of two B_{10} units, very similar to that in the $\text{B}_{10}\text{H}_{10}^{2-}$ ion, connected between one pair of adjacent apical and basal boron atoms from each B_{10}H_9 cage via two localized three-center two-electron B–B–B bonds. Location and isotropic refinement of all the H atoms indicate that no B–H–B bridges exist in the molecule. The average bond distances, $\text{B}_{(\text{apical})}\text{-B}_{(\text{basal})}$ and

$\text{B}_{(\text{basal})}\text{-B}_{(\text{basal})}$ in the upper half of the cage and the corresponding distances in the lower half of the cage are 1.740 (4), 1.835 (4), 1.698 (4) and 1.850 (4) Å, respectively. The $\text{B}_{(\text{apical})}\text{-B}_{(\text{basal})}$ and $\text{B}_{(\text{basal})}\text{-B}_{(\text{basal})}$ connecting bond distances between the two B_{10}H_9 cages are approximately 1.916 (3) and 1.686 (5) Å, respectively. The geometry of the cation $[\text{C}_5\text{H}_5\text{FeC}_5\text{H}_4\text{-CH}_2\text{N}(\text{CH}_3)_2\text{C}_2\text{H}_5]^+$ is consistent with that of its trimethylammonium cation derivative.

Experimental. The title compound (I) was prepared as a yellow solid in a reaction involving $\text{C}_5\text{H}_5\text{FeC}_5\text{-H}_4\text{CH}_2\text{N}(\text{CH}_3)_2\text{C}_2\text{H}_5\text{I}$ (5.00 mmol) in water (10 mL) and a 20 mL solution of $[(\text{C}_2\text{H}_5)_4\text{N}]_2\text{B}_{20}\text{H}_{18}$