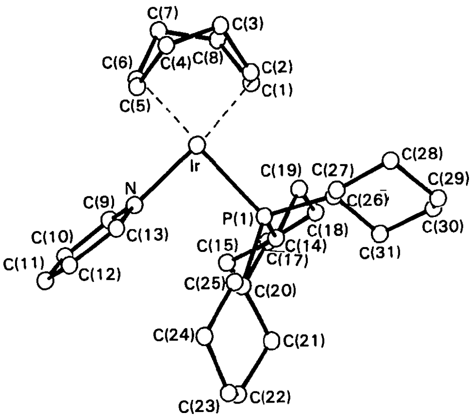

Fig. 1. Molecular structure and numbering scheme for $[Ir(cod)(py)(PCy_3)]^+$.

and $[Ir_3(\mu_3\text{-}H)H_7(py)_3(PCy_3)_3]$, formed by its deactivation, has been isolated and its structure determined (Crabtree, 1979; Crabtree & Davis, 1983). The conformations of cod in $[\{IrCl(cod)\}_2]$ and $[\{Ir_2Cl_2(\mu_2\text{-}O)(\mu_2\text{-}OH)_2(cod)_2]$ (Cotton, Lahuerta, Sanau & Schwotzer, 1986) are similar to those observed here. There are many examples of structures of $[Rh(diene)L_2]^+$ 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 P(1)—Ir—N and $M1$—Ir—P angles.

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References

Anderson, M. P. & Pignolet, L. H. (1981). *Inorg. Chem.* **20**, 4101–4107.
Cotton, F. A., Lahuerta, P., Sanau, M. & Schwotzer, M. (1986). *Inorg. Chim. Acta*, **120**, 153–157.
Crabtree, R. H. (1979). *Acc. Chem. Res.* **12**, 331–338.
Crabtree, R. H. & Davis, M. W. (1983). *Organometallics*, **2**, 681–682.
Crabtree, R. H. & Morehouse, S. M. (1986). *Inorg. Synth.* **24**, 173–176.
Enraf–Nonius (1982). *SDP-Plus Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Knowles, W. S. (1983). *Acc. Chem. Res.* **16**, 106–112.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1989). C**45**, 333–336

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

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Abstract. $[C_{15}H_{22}FeN]_2B_{20}H_{18}$, $M_r = 778{\cdot}8$, monoclinic, $C2/c$, $a = 20{\cdot}387\,(3)$, $b = 12{\cdot}265\,(4)$, $c = 16{\cdot}830\,(4)$ Å, $\beta = 95{\cdot}162\,(16)^\circ$, $V = 4191{\cdot}3\,(17)$ Å^3, $Z = 4$, $D_x = 1{\cdot}234$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu(\text{Mo }K\alpha) = 7{\cdot}18$ cm^{-1}, $F(000) = 1632$, $T = 295$ K. Final $R = 0{\cdot}033$ for 2834 observed reflections. The structure shows that the centrosymmetric $B_{20}H_{18}^{2-}$ ion consists of two B_{10} units, very similar to that in the $B_{10}H_{10}^{2-}$ 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, $B_{(apical)}$—$B_{(basal)}$ and $B_{(basal)}$—$B_{(basal)}$ 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 $B_{(apical)}$—$B_{(basal)}$ and $B_{(basal)}$—$B_{(basal)}$ 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 $[C_5H_5FeC_5H_4CH_2N(CH_3)_2C_2H_5]^+$ is consistent with that of its trimethylammonium cation derivative.

Experimental. The title compound (I) was prepared as a yellow solid in a reaction involving $C_5H_5FeC_5H_4CH_2N(CH_3)_2C_2H_5I$ (5·00 mmol) in water (10 mL) and a 20 mL solution of $[(C_2H_5)_4N]_2B_{20}H_{18}$

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

	x	y	z	U_{eq}*
B(1)	4883 (1)	14 (2)	9050 (2)	35 (1)
B(2)	4645 (1)	−354 (2)	9944 (1)	30 (1)
B(3)	4083 (1)	453 (2)	9235 (2)	34 (1)
B(4)	4216 (1)	−563 (2)	8491 (2)	36 (1)
B(5)	4743 (1)	−1405 (2)	9155 (2)	36 (1)
B(6)	3909 (2)	−1806 (2)	8915 (2)	43 (1)
B(7)	4240 (1)	−1673 (2)	9942 (2)	37 (1)
B(8)	3758 (1)	−319 (2)	10001 (2)	37 (1)
B(9)	3452 (1)	−501 (2)	8972 (2)	39 (1)
B(10)	3426 (1)	−1515 (3)	9658 (2)	46 (1)
Fe	1207 (1)	505 (1)	9178 (1)	36 (1)
C(1)	1243 (1)	−660 (2)	8335 (1)	34 (1)
C(2)	1439 (1)	360 (2)	8035 (2)	43 (1)
C(3)	912 (2)	1111 (2)	8073 (2)	53 (1)
C(4)	392 (1)	561 (2)	8389 (2)	51 (1)
C(5)	584 (1)	−525 (2)	8551 (2)	43 (1)
C(6)	1809 (2)	55 (3)	10148 (2)	74 (1)
C(7)	2017 (2)	1042 (3)	9866 (2)	69 (1)
C(8)	1501 (2)	1792 (3)	9882 (2)	61 (1)
C(9)	969 (2)	1271 (3)	10182 (2)	59 (1)
C(10)	1157 (2)	174 (3)	10353 (2)	72 (1)
C(11)	1656 (1)	−1653 (2)	8468 (1)	35 (1)
C(12)	1818 (1)	−1990 (2)	7017 (1)	48 (1)
C(13)	1987 (2)	−3449 (2)	8031 (2)	68 (1)
C(14)	887 (2)	−2790 (3)	7604 (2)	68 (1)
C(15)	2541 (2)	−1716 (3)	7037 (2)	64 (1)
N	1594 (1)	−2463 (2)	7775 (1)	41 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

(Kaczmarczyk, Dobrott & Lipscomb, 1962) (2·50 mmol) in 70/30% mixture of acetone and water at room temperature. The yellow solid was then separated from the product mixture by filtration and washed repeatedly with water to remove last traces of $(C_2H_5)_4NI$. This solid was crystallized and then recrystallized from actone to collect (I) in high purity and in almost quantitative yield.

Crystals of (I) are yellow cubes, unit-cell parameters by least-squares fit of 25 reflections in the range $10 \leq 2\theta \leq 25°$, crystal dimensions $0·4 \times 0·3 \times 0·5$ mm, space group $C2/c$ from systematic absences (hkl, $h+k$ odd; $0k0$, k odd; $h0l$, l odd); automatic Nicolet $R3m/V$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode (scan rate 3·0–15·00° min^{-1}, depending upon the intensity), 4027 measured reflections, 3690 independent reflections in the range $3 \leq 2\theta \leq 50°$, $R_{\text{int}} = 0·022$, hkl range h −24→24, k 0→14, l 0→20; 2834 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change in intensity during the data collection; Lorentz–polarization correction, no extinction corrections. Absorption correction based on ψ scans. Direct-methods *SHELXTL-Plus* (Sheldrick, 1988), refinement by full-matrix least squares using *SHELXTL-Plus,* anisotropic; boron-cage H atoms were located in difference Fourier maps, H atoms included with isotropic temperature factors; primary and secondary H atoms on carbons were constrained tetrahedrally; $w = 1/(\sigma^2F + 0·0004F^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0·033$, $wR = 0·041$; $S = 1·41$, $(\Delta/\sigma)_{\text{max}} = 0·24$, $\Delta\rho_{(\text{max,min})} = 0·28$, $-0·22$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, B, N, Fe, and the real and imaginary parts of the dispersion correction for Fe were those stored in *SHELXTL-Plus.* The final atomic parameters of the non-H atoms are given in Table 1.* The bond lengths, bond angles and selected torsion angles with their standard deviations are given in Table 2. The identification of the atoms and the configuration of the molecule are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1.

Related literature. Several salts of $B_{20}H_{18}^{2-}$ ion are now known (Kaczmarczyk, Dobrott & Lipscomb, 1962; Chamberland & Muetterties, 1964). It has been reported that Babcock has obtained single-crystal X-ray data on $[(CH_3)_4N]_2B_{20}H_{18}$, $[(C_6H_5)_3PCH_3]_2B_{20}H_{18}$, and $[(CH_3)_2S]_2B_{20}H_{18}$ (Chamberland & Muetterties, 1964). However, the actual structures of these salts have not been reported to date. The first preliminary results on the disordered structure of icosaborate(2−) were reported for its triethylammonium salt (Schwalbe & Lipscomb, 1969). Least-squares refinement of the 1874 intensities converged to $R = 0·106$, and the H atoms of interest, located and refined in both disordered orientations of $[(C_2H_5)_3NH]_2B_{20}H_{18}$ (monoclinic, space group $P2_1/a$), are 1·13 and 1·25 Å from apices near the center of the ion, but 2·34 and 2·27 Å from the nearest boron atoms of the opposite B_{10} units, respectively (Schwalbe & Lipscomb, 1969). Although a detailed structure of $[(C_2H_5)_3NH]_2B_{20}H_{18}$ has been reported in the early 1970's, the R factor of about 10% was not improved and the located

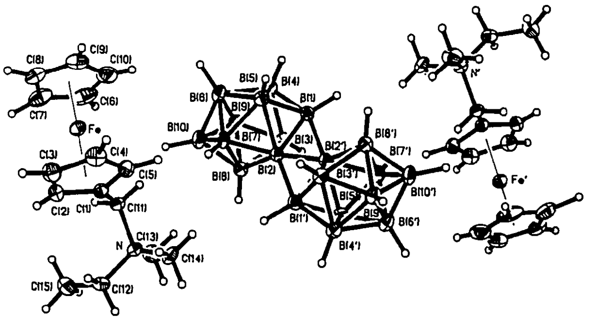

Fig. 1. *ORTEP* drawing of the molecule. Thermal ellipsoids scaled to enclose 30% probability. Hydrogen atoms are represented as spheres of arbitrary radii.

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* Lists of structure factors, anisotropic temperature factors, selected torsion angles and H parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51377 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å), *bond angles and selected torsion angles* (°)

B(1)–B(2)	1·684 (4)	B(1)–B(3)	1·769 (4)
B(1)–B(4)	1·734 (4)	B(1)–B(5)	1·774 (4)
B(1)–B(2′)	1·916 (3)	B(2)–B(3)	1·862 (3)
B(2)–B(5)	1·873 (4)	B(2)–B(7)	1·817 (4)
B(2)–B(8)	1·820 (3)	B(2)–B(1′)	1·916 (3)
B(2)–B(2′)	1·686 (5)	B(3)–B(4)	1·803 (4)
B(3)–B(8)	1·777 (4)	B(3)–B(9)	1·766 (4)
B(4)–B(5)	1·804 (4)	B(4)–B(6)	1·819 (4)
B(4)–B(9)	1·819 (4)	B(5)–B(6)	1·781 (4)
B(5)–B(7)	1·777 (4)	B(6)–B(7)	1·804 (4)
B(6)–B(9)	1·858 (4)	B(6)–B(10)	1·696 (4)
B(7)–B(8)	1·937 (4)	B(7)–B(10)	1·697 (4)
B(8)–B(9)	1·802 (4)	B(8)–B(10)	1·696 (4)
B(9)–B(10)	1·702 (4)	Fe–C(1)	2·019 (2)
Fe–C(2)	2·030 (3)	Fe–C(3)	2·043 (3)
Fe–C(4)	2·034 (3)	Fe–C(5)	2·021 (3)
Fe–C(6)	2·028 (3)	Fe–C(7)	2·038 (3)
Fe–C(8)	2·031 (3)	Fe–C(9)	2·029 (3)
Fe–C(10)	2·030 (3)	C(1)–C(2)	1·421 (3)
C(1)–C(5)	1·431 (3)	C(1)–C(11)	1·486 (3)
C(2)–C(3)	1·421 (4)	C(3)–C(4)	1·401 (4)
C(4)–C(5)	1·408 (4)	C(6)–C(7)	1·381 (6)
C(6)–C(10)	1·411 (6)	C(7)–C(8)	1·399 (5)
C(8)–C(9)	1·394 (5)	C(9)–C(10)	1·421 (5)
C(11)–N	1·527 (3)	C(12)–C(15)	1·509 (4)
C(12)–N	1·510 (3)	C(13)–N	1·492 (4)
C(14) N	1·500 (4)		

B(2)–B(1)–B(3)	65·2 (2)	B(2)–B(1)–B(4)	95·8 (2)
B(3)–B(1)–B(4)	61·9 (2)	B(2)–B(1)–B(5)	65·5 (2)
B(3)–B(1)–B(5)	97·0 (2)	B(4)–B(1)–B(5)	61·9 (1)
B(2)–B(1)–B(2′)	55·4 (2)	B(3)–B(1)–B(2′)	100·2 (2)
B(4)–B(1)–B(2′)	151·2 (2)	B(5)–B(1)–B(2′)	101·4 (2)
B(1)–B(2)–B(3)	59·6 (1)	B(1)–B(2)–B(5)	59·6 (1)
B(3)–B(2)–B(5)	90·5 (2)	B(1)–B(2)–B(7)	113·9 (2)
B(3)–B(2)–B(7)	102·7 (2)	B(5)–B(2)–B(7)	57·6 (1)
B(1)–B(2)–B(8)	114·1 (2)	B(3)–B(2)–B(8)	57·7 (1)
B(5)–B(2)–B(8)	102·9 (2)	B(7)–B(2)–B(8)	64·4 (1)
B(1)–B(2)–B(1′)	124·6 (2)	B(3)–B(2)–B(1′)	133·0 (2)
B(5)–B(2)–B(1′)	134·3 (2)	B(7)–B(2)–B(1′)	112·8 (2)
B(8)–B(2)–B(1′)	111·8 (2)	B(1)–B(2)–B(2′)	69·3 (2)
B(3)–B(2)–B(2′)	105·6 (2)	B(5)–B(2)–B(2′)	106·7 (2)
B(7)–B(2)–B(2′)	147·7 (2)	B(8)–B(2)–B(2′)	146·1 (2)
B(1′)–B(2)–B(2′)	55·3 (2)	B(1)–B(3)–B(2)	55·2 (1)
B(1)–B(3)–B(4)	58·1 (1)	B(2)–B(3)–B(4)	87·5 (2)
B(1)–B(3)–B(8)	112·1 (2)	B(2)–B(3)–B(8)	59·9 (1)
B(4)–B(3)–B(8)	103·0 (2)	B(1)–B(3)–B(9)	114·6 (2)
B(2)–B(3)–B(9)	101·6 (2)	B(4)–B(3)–B(9)	61·3 (2)
B(8)–B(3)–B(9)	61·1 (2)	B(1)–B(4)–B(3)	60·0 (1)
B(1)–B(4)–B(5)	60·2 (1)	B(3)–B(4)–B(5)	94·7 (2)
B(1)–B(4)–B(6)	114·2 (2)	B(3)–B(4)–B(6)	103·2 (2)
B(5)–B(4)–B(6)	58·9 (1)	B(1)–B(4)–B(9)	113·7 (2)
B(3)–B(4)–B(9)	58·4 (1)	B(5)–B(4)–B(9)	103·5 (2)
B(6)–B(4)–B(9)	61·4 (2)	B(1)–B(5)–B(2)	54·9 (1)
B(1)–B(5)–B(4)	58·0 (1)	B(2)–B(5)–B(4)	87·2 (2)
B(1)–B(5)–B(6)	114·1 (2)	B(2)–B(5)–B(6)	100·9 (2)
B(4)–B(5)–B(6)	61·0 (1)	B(1)–B(5)–B(7)	111·4 (2)
B(2)–B(5)–B(7)	59·6 (1)	B(4)–B(5)–B(7)	102·5 (2)
B(6)–B(5)–B(7)	60·9 (2)	B(4)–B(6)–B(5)	60·1 (1)
B(4)–B(6)–B(7)	100·9 (2)	B(5)–B(6)–B(7)	59·4 (2)
B(4)–B(6)–B(9)	59·3 (1)	B(5)–B(6)–B(9)	102·9 (2)
B(7)–B(6)–B(9)	91·2 (2)	B(4)–B(6)–B(10)	110·9 (2)
B(5)–B(6)–B(10)	112·4 (2)	B(7)–B(6)–B(10)	57·9 (2)
B(9)–B(6)–B(10)	57·0 (2)	B(2)–B(7)–B(5)	62·8 (1)
B(2)–B(7)–B(6)	102·3 (2)	B(5)–B(7)–B(6)	59·7 (2)
B(2)–B(7)–B(8)	57·9 (1)	B(5)–B(7)–B(8)	102·0 (2)
B(6)–B(7)–B(8)	88·8 (2)	B(2)–B(7)–B(10)	109·4 (2)
B(5)–B(7)–B(10)	112·6 (2)	B(6)–B(7)–B(10)	57·9 (2)
B(8)–B(7)–B(10)	55·1 (1)	B(2)–B(8)–B(3)	62·3 (1)
B(2)–B(8)–B(7)	57·7 (1)	B(3)–B(8)–B(7)	101·2 (2)
B(2)–B(8)–B(9)	101·8 (2)	B(3)–B(8)–B(9)	59·1 (1)
B(7)–B(8)–B(9)	88·7 (2)	B(2)–B(8)–B(10)	109·3 (2)
B(3)–B(8)–B(10)	112·2 (2)	B(7)–B(8)–B(10)	55·2 (1)
B(9)–B(8)–B(10)	58·1 (2)	B(3)–B(9)–B(4)	60·4 (1)
B(3)–B(9)–B(6)	103·1 (2)	B(4)–B(9)–B(6)	59·3 (2)
B(3)–B(9)–B(8)	59·7 (1)	B(4)–B(9)–B(8)	101·4 (2)
B(6)–B(9)–B(8)	91·4 (2)	B(3)–B(9)–B(10)	112·4 (2)
B(4)–B(9)–B(10)	110·6 (2)	B(6)–B(9)–B(10)	56·7 (2)
B(8)–B(9)–B(10)	57·8 (2)	B(6)–B(10)–B(7)	64·2 (2)
B(6)–B(10)–B(8)	101·1 (2)	B(7)–B(10)–B(8)	69·7 (2)
B(6)–B(10)–B(9)	66·3 (2)	B(7)–B(10)–B(9)	100·6 (2)
B(8)–B(10)–B(9)	64·1 (2)	C(1)–Fe–C(2)	41·1 (1)
C(1)–Fe–C(3)	69·1 (1)	C(2)–Fe–C(3)	40·8 (1)
C(1)–Fe–C(4)	69·1 (1)	C(2)–Fe–C(4)	68·3 (1)
C(3)–Fe–C(4)	40·2 (1)	C(1)–Fe–C(5)	41·5 (1)
C(2)–Fe–C(5)	68·9 (1)	C(3)–Fe–C(5)	68·4 (1)
C(4)–Fe–C(5)	40·6 (1)	C(1)–Fe–C(6)	108·4 (1)
C(2)–Fe–C(6)	124·3 (1)	C(3)–Fe–C(6)	159·9 (1)
C(4)–Fe–C(6)	159·2 (1)	C(5)–Fe–C(6)	123·8 (1)
C(1)–Fe–C(7)	123·3 (1)	C(2)–Fe–C(7)	108·7 (1)
C(3)–Fe–C(7)	123·8 (1)	C(4)–Fe–C(7)	158·9 (1)
C(5)–Fe–C(7)	159·6 (1)	C(6)–Fe–C(7)	39·7 (2)
C(1)–Fe–C(8)	158·8 (1)	C(2)–Fe–C(8)	122·6 (1)
C(3)–Fe–C(8)	107·2 (1)	C(4)–Fe–C(8)	122·4 (1)
C(5)–Fe–C(8)	158·3 (1)	C(6)–Fe–C(8)	67·5 (1)
C(7)–Fe–C(8)	40·2 (1)	C(1)–Fe–C(9)	159·7 (1)
C(2)–Fe–C(9)	157·4 (1)	C(3)–Fe–C(9)	121·1 (1)
C(4)–Fe–C(9)	106·6 (1)	C(5)–Fe–C(9)	122·5 (1)
C(6)–Fe–C(9)	68·2 (1)	C(7)–Fe–C(9)	67·7 (1)
C(8)–Fe–C(9)	40·2 (1)	C(1)–Fe–C(10)	123·4 (1)
C(2)–Fe–C(10)	160·4 (1)	C(3)–Fe–C(10)	157·4 (1)
C(4)–Fe–C(10)	122·2 (1)	C(5)–Fe–C(10)	107·3 (1)
C(6)–Fe–C(10)	40·7 (2)	C(7)–Fe–C(10)	67·8 (2)
C(8)–Fe–C(10)	68·1 (1)	C(9)–Fe–C(10)	41·0 (1)
Fe–C(1)–C(2)	69·9 (1)	Fe–C(1)–C(5)	69·3 (1)
C(2)–C(1)–C(5)	106·9 (2)	Fe–C(1)–C(11)	122·0 (1)
C(2)–C(1)–C(11)	126·9 (2)	C(5)–C(1)–C(11)	126·0 (2)
Fe–C(2)–C(1)	69·0 (1)	Fe–C(2)–C(3)	70·0 (2)
C(1)–C(2)–C(3)	108·4 (2)	Fe–C(3)–C(2)	69·1 (2)
Fe–C(3)–C(4)	69·5 (2)	C(2)–C(3)–C(4)	107·8 (2)
Fe–C(4)–C(3)	70·3 (2)	Fe–C(4)–C(5)	69·2 (1)
C(3)–C(4)–C(5)	108·9 (2)	Fe–C(5)–C(1)	69·2 (1)
Fe–C(5)–C(4)	70·2 (2)	C(1)–C(5)–C(4)	108·0 (2)
Fe–C(6)–C(7)	70·5 (2)	Fe–C(6)–C(10)	69·7 (2)
C(7)–C(6)–C(10)	108·6 (3)	Fe–C(7)–C(6)	69·8 (2)
Fe–C(7)–C(8)	69·6 (2)	C(6)–C(7)–C(8)	108·4 (3)
Fe–C(8)–C(7)	70·2 (2)	Fe–C(8)–C(9)	69·8 (2)
C(7)–C(8)–C(9)	108·4 (3)	Fe–C(9)–C(8)	70·0 (2)
Fe–C(9)–C(10)	69·6 (2)	C(8)–C(9)–C(10)	107·7 (3)
Fe–C(10)–C(6)	69·6 (2)	Fe–C(10)–C(9)	69·5 (2)
C(6)–C(10)–C(9)	106·9 (3)	C(1)–C(11)–N	114·3 (2)
C(15)–C(12)–N	115·9 (2)	C(11)–N–C(12)	112·6 (2)
C(11)–N–C(13)	107·5 (2)	C(12)–N–C(13)	111·1 (2)
C(11)–N–C(14)	109·4 (2)	C(12)–N–C(14)	107·5 (2)
C(13)–N–C(14)	108·7 (2)		

B(3)–B(1)–B(2)–B(5)	110·9 (2)	B(3)–B(1)–B(2)–B(7)	91·1 (2)
B(3)–B(1)–B(2)–B(8)	19·7 (2)	B(3)–B(1)–B(2)–B(1′)	−123·7 (2)
B(3)–B(1)–B(2)–B(2′)	−123·7 (2)	B(4)–B(1)–B(2)–B(3)	−55·4 (2)
B(4)–B(1)–B(2)–B(5)	55·5 (2)	B(4)–B(1)–B(2)–B(7)	35·6 (2)
B(4)–B(1)–B(2)–B(8)	−35·7 (2)	B(4)–B(1)–B(2)–B(1′)	−179·2 (2)
B(4)–B(1)–B(2)–B(2′)	−179·2 (2)	B(5)–B(1)–B(2)–B(3)	−110·9 (2)
B(5)–B(1)–B(2)–B(7)	−19·8 (2)	B(5)–B(1)–B(2)–B(8)	−91·2 (2)
B(5)–B(1)–B(2)–B(1′)	125·3 (2)	B(5)–B(1)–B(2)–B(2′)	125·3 (2)
B(2′)–B(1)–B(2)–B(3)	123·7 (2)	B(2′)–B(1)–B(2)–B(5)	−125·3 (2)
B(2′)–B(1)–B(2)–B(7)	−145·2 (3)	B(2′)–B(1)–B(2)–B(8)	143·5 (3)
B(2′)–B(1)–B(2)–B(1′)	0·0 (0)		

H atoms other than on the central B atoms were not refined, presumably due to disorder in its crystal lattice (Schwalbe & Lipscomb, 1971).

The molecular structure of ferrocene has been investigated by X-ray crystallography (Fischer & Fritz, 1959; Wilkinson & Cotton, 1959) and by electron diffraction in the gas phase (Bohn & Haaland, 1966). The single-crystal X-ray diffraction data on several derivatives of ferrocene such as 1,1′-tetramethyleneferrocene (Laing & Trueblood, 1965), 1,1′-(α-oxotrimethylene)ferrocene (Jones, Marsh & Richards, 1965) and the quinidine salt of (−)-1,1′-dimethylferrocene-3-carboxylic acid (Carter, McPhail & Sim, 1967) have also been reported. The recent X-ray investigations of a number of ferrocenealkyltrialkylammonium salts of boron hydrides $[C_5H_5FeC_5H_4CH_2N(CH_3)_3]_2B_{10}H_{10}$ (Zhang, Cai, Chen, Pan, Lu, Zhang & Zhu, 1982), $[C_5H_5FeC_5H_4CH_2N(CH_3)_3]B_3H_8$ (Fu, Pan, Lu, Zhang & Zhu, 1982), $[C_5H_5FeC_5H_4CH_2NH(CH_3)_2]_2B_{12}H_{12}$ (Zhang, Chen, Cai, Pan, Zhang & Zhang, 1983), and of a neutral derivative $C_5H_5FeC_5H_4CH_2N(CH_3)_2BH_3$ (Fu, Chen, Cai, Pang, Zhang & Zhu, 1985) are consistent with those of ferrocene derivatives and their corresponding borohydride cage anion fragments.

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References

Bohn, R. K. & Haaland, A. (1966). *J. Organomet. Chem.* **5**, 470–476.

Carter, O. L., McPhail, A. T. & Sim, G. A. (1967). *J. Chem. Soc. A*, pp. 365–373.

Chamberland, B. L. & Muetterties, E. L. (1964). *Inorg. Chem.* **3**, 1450–1456.

Fischer, E. O. & Fritz, H. P. (1959). *Adv. Inorg. Chem. Radiochem.* **1**, 55–115.

Fu, Z., Chen, Z., Cai, Z., Pang, K., Zhang, G. & Zhu, H. (1985). *J. Struct. Chem. (Chin.)*, **4**, 203–205.

Fu, Z., Pan, K., Lu, J., Zhang, G. & Zhu, H. (1982). *J. Struct. Chem. (Chin.)*, **1**, 57–62.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Jones, N. D., Marsh, R. E. & Richards, J. H. (1965). *Acta Cryst.* **19**, 330–336.

Kaczmarczyk, A., Dobrott, R. D. & Lipscomb, W. N. (1962). *Proc. Natl Acad. Sci. USA*, **48**, 729–733.

Laing, M. B. & Trueblood, K. N. (1965). *Acta Cryst.* **19**, 373–381.

Schwalbe, C. H. & Lipscomb, W. N. (1969). *J. Am. Chem. Soc.* **91**, 194–196.

Schwalbe, C. H. & Lipscomb, W. N. (1971). *Inorg. Chem.* **10**, 151–160.

Sheldrick, G. M. (1988). *SHELXTL-Plus* 88. Structure determination software programs. Nicolet Instrument Corp., 5225-5 Verona Road, Madison, Wisconsin, USA.

Wilkinson, G. & Cotton, F. A. (1959). *Prog. Inorg. Chem.* **1**, 1–124.

Zhang, Y., Cai, Z., Chen, Z., Pan, K., Lu, J., Zhang, G. & Zhu, H. (1982). *J. Struct. Chem. (Chin.)*, **1**, 46–53.

Zhang, Y., Chen, Z., Cai, Z., Pan, K., Zhang, G. & Zhang, Z. (1983). *J. Struct. Chem. (Chin.)*, **2**, 201–206.

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Structure of (2,2′-Bipyrimidine)tetracarbonylchromium(0)–2,2′-Bipyrimidine

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Abstract. $[Cr(C_8H_6N_4)(CO)_4].C_8H_6N_4$, $M_r = 480{\cdot}36$, triclinic, $P\bar{1}$, $a = 11{\cdot}331\,(3)$, $b = 13{\cdot}701\,(4)$, $c = 7{\cdot}178\,(5)$ Å, $\alpha = 91{\cdot}51\,(4)$, $\beta = 105{\cdot}76\,(4)$, $\gamma = 105{\cdot}34\,(4)^\circ$, $V = 1028{\cdot}41\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}55$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu_1 = 5{\cdot}86$ cm^{-1}, $F(000) = 488$, $T = 294$ K, final $R = 0{\cdot}042$ for 2609 reflections with $I > \sigma(I)$. The tetracarbonyl complex has approximate C_{2v} symmetry and the bipyrimidine molecule is nearly planar. Both the 2,2′-bipyrimidine and the metal complex showing stacking along the crystallographic c axis. The C—O, C—C and C—N bond lengths within the complex and 2,2′-bipyrimidine molecules are normal. Both diimine molecules are essentially planar, with dihedral angles of 9·64 and 3·74° for 2,2′-bipyrimidine and the Cr complex, respectively.

Experimental. The complex was formed by refluxing equal molar equivalents of 2,2′-bipyrimidine and $Cr(CO)_6$ in THF for 12 h. Upon cooling, dark crystals deposited from solution. Black prism, $0{\cdot}25 \times 0{\cdot}25 \times 0 \times 15$ mm, Enraf–Nonius CAD-4 diffractometer, graphite monochromator, Mo $K\alpha$ radiation, lattice parameters from least-squares refinement using 25 accurately centered reflections with $14 < 2\theta < 35^\circ$, space group $P\bar{1}$ determined from intensity data and successful solution and refinement of structure; 3611 unique reflections collected of which 2609 were considered observed at greater than one $\sigma(I)$ level [$\sigma(I)$ from counting statistics] and used in refinement of structure; $\theta_{max} = 25^\circ$, $\omega/2\theta$ scans with scan range $0{\cdot}6^\circ + 0{\cdot}34^\circ\tan\theta$ and variable scan speeds of 1–5° min^{-1}, three standard reflections $(25\bar{1}, \bar{1}40, 0\bar{1}3)$ were measured every 180 min of X-ray exposure, no significant deviation noted (less than 1%); collected data: h, $\pm k$, $\pm l$, to max. indices of 13, 16, 18. Data corrected for Lp and background but not for absorption, intensities of equivalent reflections were averaged, agreement factor for averaging of 522 observed reflections was 1·4% based on intensity. Structure solved by direct methods (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and

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