should be pointed out that in the false *Pcab* space group N(13) and C(13) are symmetry related and cannot be distinguished. It is therefore very probable that these atoms can statistically interchange their positions in the crystal (the refinement of the structure in *Pcab* stopped at $R = 0{\cdot}112$ and $wR = 0{\cdot}113$ and led to unacceptable temperature factors and bond lengths for C—N groups). The disordering of the N(11)—C(11) and N(12)—C(12) cations precludes any interpretation of their interactions with neighbouring anions.

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Structure of 2-(1-Hydroxyethyl)-1-ferrocenecarboxylic Acid Methyl Ester

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Abstract. Methyl 2-(1-hydroxyethyl)-1-ferrocenecarboxylate, $[\mathrm{Fe(C_{14}H_{16}O_3)}]$, $M_r = 288{\cdot}13$, monoclinic, $P2_1/c$, $a = 8{\cdot}378\,(3)$, $b = 9{\cdot}2845\,(8)$, $c = 16{\cdot}669\,(5)$ Å, $\beta = 103{\cdot}13°$, $V = 1262{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}52$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\bar{\alpha}) = 0{\cdot}71069$ Å, $\mu = 11{\cdot}79$ cm^{-1}, $F(000) = 600$, $T = 293\,(2)$ K. $R = 0{\cdot}036$ for 1913 reflections with $I/\sigma(I) \geq 2{\cdot}0$. The configurations of the molecules are (*R*,*S*) and (*S*,*R*). The hydroxyl groups are hydrogen bonded to carbonyl groups on adjacent molecules.

Introduction. Transition-metal catalysts containing chiral ferrocenyl complexes as a ligand have been found to be useful in asymmetric syntheses (Sun, 1986; Cullen & Woollins, 1981; Hayashi & Kumada, 1982). α-Substituted derivatives of *N*,*N*-dimethylaminoethylferrocene possessing either a center, a plane or both a center and a plane of chirality are effective chiral ligands in asymmetric syntheses catalyzed by rhodium, platinum, palladium and nickel complexes (Hayashi, Yamamoto & Kumada, 1974; Hayashi, Mise, Mitachi, Yamamoto & Kumada, 1976; Cullen & Woollins, 1982; Appleton, Cullen, Evans, Kim & Trotter, 1985; Hayashi, Katsumura, Konishi & Kumada, 1979; Hayashi, Tamao, Katsuro, Nakae & Kumada, 1980; Hayashi, Konishi, Fukushima, Mise, Kagotani, Tajika & Kumada, 1982; Hayashi, Yamamoto & Ito, 1988). In attempts to obtain diketone derivatives of *N*,*N*-dimethylaminoferrocene, the title compound was obtained and its crystallographic study is reported here.

Experimental. A crystal sample of the title compound was provided by W. R. Cullen of the University of British Columbia. It was obtained from the reaction of 2-(*N*,*N*-dimethylaminoethylferrocene)carboxylic acid with sodium bicarbonate and methyl iodide in dimethylacetamide followed by hydrolysis by addition of 10% aqueous sodium chloride.

An orange crystal having an irregular shape and approximate dimensions $0{\cdot}1 \times 0{\cdot}1 \times 0{\cdot}15$ mm was sealed in a quartz capillary. Cell dimensions were determined from the refinement of 34 reflections with $31 \leq 2\theta \leq 39°$. Intensity data collected with modified Picker four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan tech-

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nique. Three reference reflections showed less than a 5% decrease, 2915 independent reflections recorded for 2θ values between 3 and 55°. $0 \le h \le 10$, $0 \le k \le 12$, $-21 \le l \le 21$. Of these, 1913 reflections showed $I/\sigma(I) \ge 2{\cdot}0$, and considered as observed and used in the subsequent structural studies. Data were corrected for polarization and Lorentz effects. Absorption corrections applied using the Gaussian integration method; minimum and maximum transmission factors 0·704 and 0·881.

The structure was solved by direct methods using the *XTAL*85 system (Stewart & Hall, 1986). An *E* map gave all non-H atoms. All H atoms were located from difference Fourier syntheses. All non-H atoms were refined anisotropically on *F* and H atoms were refined isotropically using the *XTAL*85 programs on a VAX8600 computer. $w = \sigma(F)^{-2}$, where σ is based on counting statistics and the machine instability factor. The final *R* and *wR* values were 0·036 and 0·025 for 1913 observed reflections. The maximum fluctuation of electron density found at the termination of refinement was 0·46 e Å^{-3} at a position 1·21 Å from the Fe atom. The maximum and average shift/e.s.d. were $8{\cdot}8 \times 10^{-4}$ and $5{\cdot}3 \times 10^{-5}$ respectively. Scattering factors taken from Cromer & Mann (1968); *International Tables for X-ray Crystallography* (1974).

The fractional atomic coordinates and equivalent isotropic temperature factors are listed in Table 1. Table 2 lists the bond lengths and angles.* A stereoscopic view (*ORTEP*II; Johnson, 1976) of the enantiomer with the (*R*,*S*) absolute configuration is given in Fig. 1 with the atomic labeling scheme.†

Discussion. The cyclopentadienyl rings are planar with a maximum displacement from the mean plane of 0·007 Å. The dihedral angle between the two cyclopentadienyl ring planes is 178·4°. The two cyclopentadienyl rings have an arrangement close to an eclipsed conformation. The rotation, defined by the vector from ring C atoms to the centroids of the ring, ranges from 3·4 to 4·6° with an average of 3·8°.

The distance from the Fe atom to the cyclopentadienyl C atoms varies from 2·018 (3) to 2·050 (3) Å, with an average of 2·037 (14) Å, for the substituted ring, and varies less, from 2·032 (4) to 2·046 (4) Å, with an average of 2·041 (6) Å, for the unsubstituted ring. The C—C distances range from 1·409 (4) to

Table 1. *Fractional atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic temperature factors* (Å$^2 \times 10^2$)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
Fe	0·75591 (5)	0·30659 (5)	0·17428 (2)	3·34
O(1)	1·1303 (3)	0·0659 (3)	0·0901 (2)	6·5
O(2)	0·7302 (2)	−0·0539 (2)	0·0552 (1)	4·9
O(3)	0·5934 (2)	−0·0578 (2)	0·1557 (1)	4·8
C(1)	0·8163 (3)	0·0962 (3)	0·1742 (2)	3·4
C(2)	0·9437 (3)	0·1826 (3)	0·1535 (2)	3·5
C(3)	1·0014 (4)	0·2752 (3)	0·2218 (2)	4·2
C(4)	0·9120 (4)	0·2487 (4)	0·2829 (2)	4·6
C(5)	0·7975 (4)	0·1387 (3)	0·2541 (2)	4·1
C(6)	0·5357 (4)	0·3397 (4)	0·0937 (2)	4·8
C(7)	0·6589 (4)	0·4204 (4)	0·0694 (2)	5·0
C(8)	0·7269 (5)	0·5143 (4)	0·1354 (2)	5·6
C(9)	0·6456 (5)	0·4919 (4)	0·1993 (2)	5·8
C(10)	0·5273 (4)	0·3840 (4)	0·1741 (2)	5·3
C(11)	1·0078 (4)	0·1755 (4)	0·0767 (2)	4·0
C(12)	1·0797 (5)	0·3161 (5)	0·0548 (3)	6·2
C(13)	0·7134 (4)	−0·0113 (3)	0·1210 (2)	3·6
C(14)	0·4769 (5)	−0·1574 (4)	0·1079 (3)	5·2

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Fe—C(1)	2·018 (3)	Fe—C(2)	2·041 (3)
Fe—C(3)	2·050 (3)	Fe—C(4)	2·050 (3)
Fe—C(5)	2·028 (3)	Fe—C(6)	2·044 (3)
Fe—C(7)	2·046 (3)	Fe—C(8)	2·032 (4)
Fe—C(9)	2·041 (4)	Fe—C(10)	2·045 (4)
C(1)—C(2)	1·438 (4)	C(1)—C(5)	1·431 (4)
C(1)—C(13)	1·477 (4)	C(2)—C(3)	1·421 (4)
C(2)—C(11)	1·498 (4)	C(3)—C(4)	1·417 (5)
C(4)—C(5)	1·409 (4)	C(6)—C(7)	1·408 (5)
C(6)—C(10)	1·418 (5)	C(7)—C(8)	1·418 (5)
C(8)—C(9)	1·404 (6)	C(9)—C(10)	1·504 (5)
C(11)—C(12)	1·516 (6)	O(1)—C(11)	1·427 (4)
O(2)—C(13)	1·205 (4)	O(3)—C(13)	1·339 (4)
O(3)—C(14)	1·446 (4)		

C(2)—C(1)—C(5)	108·3 (2)	C(2)—C(1)—C(13)	126·7 (3)
C(5)—C(1)—C(13)	124·9 (3)	C(1)—C(2)—C(3)	106·3 (3)
C(1)—C(2)—C(11)	127·6 (3)	C(3)—C(2)—C(11)	126·1 (3)
C(2)—C(3)—C(4)	109·4 (3)	C(3)—C(4)—C(5)	108·1 (3)
C(4)—C(5)—C(1)	107·9 (3)	C(7)—C(6)—C(10)	108·3 (3)
C(6)—C(7)—C(8)	107·2 (3)	C(7)—C(8)—C(9)	108·8 (3)
C(8)—C(9)—C(10)	108·0 (3)	C(6)—C(10)—C(9)	107·9 (3)
O(1)—C(11)—C(2)	106·4 (2)	O(1)—C(11)—C(12)	110·0 (3)
C(2)—C(11)—C(12)	114·0 (3)	O(2)—C(13)—O(3)	122·8 (3)
O(2)—C(13)—C(1)	126·6 (3)	O(3)—C(13)—C(1)	110·6 (3)
C(13)—O(3)—C(14)	116·6 (3)		

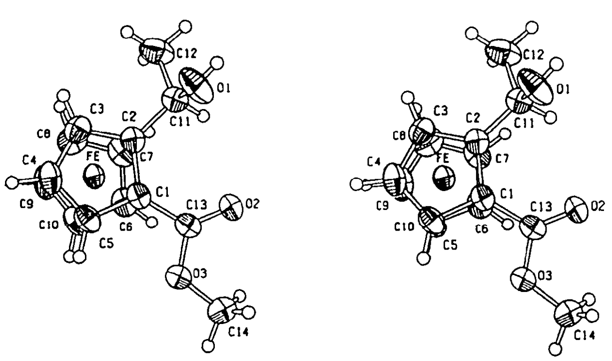

Fig. 1. Stereoview with the atomic labeling scheme.

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* Lists of structure factors, anisotropic temperature factors, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52772 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† The first symbol in '(*R*,*S*)' refers to the asymmetric C atom, and the second refers to the asymmetrically substituted ferrocene moiety.

1·438 (4) Å, with an average value of 1·411 (7) Å, for the unsubstituted ring. The C—C—C bond angles for the unsubstituted ring range from 107·2 (3) to 108·6 (3)°, and for the substituted ring from 106·3 (3) to 109·4 (3)°. Although these distances and angles are normal as compared to other similar structures, the differences among bond lengths and angles for the substituted ring are worth noting.

If we apply a criterion proposed by Taylor & Kennard (1983), it must be concluded that real differences are present. The larger differences are associated with the substituted ring. Therefore, it appears that they are caused by the electronic effects of the substituents rather than by packing effects. Similar variations have been observed in other ferrocenyl derivatives (Appleton *et al.*, 1985; Einstein & Willis, 1980; Cullen, Einstein, Huang, Willis & Yeh, 1980; Cullen, Evans, Han & Trotter, 1987; Battelle, Bau, Gokel, Oyakawa & Ugi, 1973; Butler, Cullen & Rettig, 1986).

The orientation of the substituents is an important structural feature. Atom C(11) is displaced 0·017 (5) Å above the cyclopentadienyl ring plane. The hydroxyethyl group is rotated in a similar way as is the —$CH(CH_3)N(CH_3)_2$ group in the structure of [1-(*N*,*N*-dimethylammonium)ethyl]ferrocene tartrate dihydrate (Luo, Barton & Robertson, 1987). Atom O(1) is above the ring plane while atom C(12) is below the ring plane. The perpendicular distances from these two atoms to the ring plane are 1·388 (6) and 0·618 (8) Å, respectively. The plane formed by atoms C(2), C(11) and O(1) is almost perpendicular to the cyclopentadienyl ring plane with an angle of 87·3 (3)°. The plane through atoms C(2), C(11) and C(12) forms an angle of 27·6 (3)° with the ring plane. There is considerable repulsion between the methyl group of the hydroxyethyl group and the ferrocene moiety. This repulsion pushes the methyl group away which leads to the bond angle C(2)—C(11)—C(12) being 114·0 (3)°.

A calculation based on the structural parameters given by Cotton & Reid (1985) shows that in the structure of ferrocenecarboxylic acid the carboxylic group is almost coplanar with the cyclopentadienyl ring plane to which it is attached. The maximum displacement from the ring plane is 0·09 Å. An X-ray crystallographic analysis of 1,1′-ferrocenedicarboxylic acid by Palenik (1969) also showed that the carboxylic groups are almost coplanar with the cyclopentadienyl ring planes to which they attach. A later neutron-diffraction study (Takusagawa & Koetzle, 1979) showed that the hydroxyl O atoms are displaced from the cyclopentadienyl ring plane toward the Fe atom by 0·126 and 0·187 Å. In our structure, atoms C(13) and O(2) are coplanar with the cyclopentadienyl ring plane within the experimental error. However, atoms O(3) and C(14) are displaced below the cyclopentadienyl ring plane 0·214 (6) and 0·378 (9) Å respectively. Atoms O(2), C(13), O(3), C(14) and attached ring C atom C(1) are coplanar but the plane is twisted with respect to the cyclopentadienyl ring plane with an angle of 6·4 (5)°.

It was observed from the ^{1}H NMR spectra that the rotation of the hydroxyethyl group was restricted and an intramolecular hydrogen bond between the H atom on the hydroxyl group and the O atom on the carbonyl group was therefore suggested (Cullen & Wickenheiser, 1988). Although the possibility of the formation of such hydrogen bonding in solution cannot be ruled out, the analysis of the structure does not show such hydrogen bonding in the solid state. The carboxylic-acid methyl-ester group in the structure is rotated in such a way that the methoxy group is positioned far from the hydroxyethyl group in the direction opposite to it, probably to reduce steric repulsion. The carbonyl O atom is oriented toward the hydroxyethyl group. Such an orientation of the carboxylic-acid methyl-ester group would favour the formation of an intramolecular hydrogen bond between the carbonyl O atom and an H atom from the hydroxyethyl group. However, considerable repulsion between atom O(2) and the hydroxyethyl group is observed. The bond angle C(1)—C(13)—O(2), 126·8 (4)°, is much larger than the ideal value of 120·0° for sp^2-hybridized C atoms. The enlargement of this angle appears to be the result of steric repulsion. The repulsion also pushes the hydroxyethyl group slightly away from the ideal position.

The C(1)—C(2)—C(11) bond angle, 127·6 (3)°, is larger than the value of 126·1 (3)° observed for C(3)—C(2)—C(11). This repulsion may also contribute to the restricted rotation of the hydroxyethyl group as observed in the ^{1}H NMR spectra in addition to the effect of intermolecular hydrogen bonds which will be discussed below. Since atom O(2) is forced toward the methoxy group in order to reduce internal repulsion, the methoxy group is consequently pushed away from the carbonyl O atom in the direction toward the cyclopentadienyl ring. The bond angle C(1)—C(13)—O(3) is only 110·6 (3)°. The C=O bond length, which is only 1·205 (4) Å, is much shorter than 1·261 (15) Å in ferrocenecarboxylic acid (Cotton & Reid, 1985), and 1·228 (3) Å in ferrocenedicarboxylic acid (Palenik, 1969). However, it is comparable with the value of 1·218 (7) Å found in acetylferrocene (Palenik, 1970). The C—O bond length, 1·339 (4) Å, is longer than the value of 1·313 Å found in both ferrocenecarboxylic acid and ferrocenedicarboxylic acid.

Each of the molecules in the crystal is a stereoisomer with an (*S*,*R*) or (*R*,*S*) configuration related by the center of symmetry. There are two intermolecular hydrogen bonds associated with the two enantiomers; from O(2) in one enantiomer to H(1) in the other enantiomer with hydrogen-bond lengths and angle [O(2)···H(1)] = 2·15 (3), [O(2)···O(1)] = 2·919 (4) Å and [O(2)···H(1)···O(1)] = 178·8 (3)°. These hydrogen bonds are probably a major factor in restricting the free rotation of the hydroxyethyl group.

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1-(2,2′-Bipyridine)-2-methyl-3-(trimethylsilyl)-2,3-dicarba-1-plumba-*closo*-heptaborane(6)

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Abstract. $C_{16}H_{24}B_4N_2PbSi$, $M_r = 522{\cdot}9$, triclinic, $P\bar{1}$, $a = 7{\cdot}007\,(3)$, $b = 9{\cdot}318\,(6)$, $c = 16{\cdot}462\,(13)$ Å, $\alpha = 103{\cdot}84\,(6)$, $\beta = 96{\cdot}40\,(5)$, $\gamma = 95{\cdot}21\,(4)°$, $V = 1029{\cdot}4\,(11)$ Å^3, $Z = 2$, $D_x = 1{\cdot}69$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, μ(Mo $K\alpha$) $= 83{\cdot}12$ cm^{-1}, $F(000) = 500$, $T = 295$ K. Final $R = 0{\cdot}035$ for 2781 observed reflections. The structure shows that the title compound (V) consists of a distorted pentagonal bipyramid with the apical Pb atom displaced significantly from the centroidal position toward the basal borons above the C_2B_3 face. The Pb-bound Lewis base, 2,2′-bipyridine, is not exactly opposite the C—C$_{\text{(cage)}}$ bond, rather it is tilted significantly toward the two basal borons above the C_2B_3 face with B—Pb—N orientation angles of about 81–141°. Bond distances in the cage structure are Pb—C 2·835 (8) and 2·770 (8), Pb—B 2·566 (10), 2·466 (8) and 2·601 (9), and Pb—N 2·615 (7) and 2·663 (7) Å. The crystal packing in the unit cell reveals that the intermolecular dipole–dipole type of interactions within the dimeric plumbacarborane precursor (II) have been broken during the formation of its donor–acceptor complex (V) with the Lewis base, 2,2′-bipyridine.

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