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Decamethylferrocenium hexafluorophosphate

Alejandro Sánchez Perucha and Michael Bolte*

Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany
Correspondence e-mail: bolte@chemie.uni-frankfurt.de

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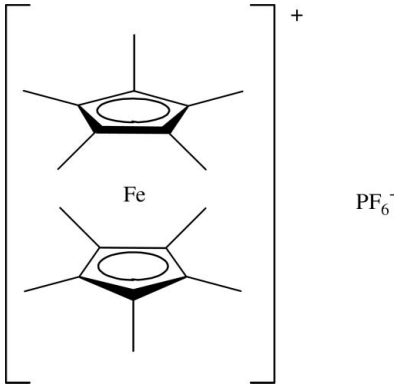
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.092; data-to-parameter ratio = 13.8.

The structure of the title compound, $[Fe(C_{10}H_{15})_2]PF_6$, is composed of discrete decamethylferrocenium cations and hexafluorophosphate anions. The Fe and P atoms are located on special positions of site symmetry 2/m, two C atoms and two F atoms are located on a mirror plane, and one F atom is located on a twofold rotation axis. The compound is isostructural with the cobaltocenium complex.

Related literature

For related literature, see: Braga *et al.* (1999); Heise *et al.* (2002).



Experimental

Crystal data

$[Fe(C_{10}H_{15})_2]PF_6$
M_r = 471.26
Monoclinic, $C2/m$
a = 14.186 (2) Å
b = 8.9579 (11) Å
c = 8.9902 (13) Å
β = 110.277 (13)°
V = 1071.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.83 mm^{-1}
T = 173 (2) K
0.34 × 0.32 × 0.19 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.765, T_{max} = 0.858
8050 measured reflections
1050 independent reflections
980 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.092
S = 1.11
1050 reflections
76 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2302).

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supplementary materials

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Decamethylferrocenium hexafluorophosphate

A. Sánchez Perucha and M. Bolte

Comment

Ferrocenium salts have been extensively studied to better understand the mechanisms of reactions involving ferrocene and ferrocenium-type systems.

The structure of the title compound, $[C_{20}H_{30}Fe]^{+}[PF_6]^{-}$, is composed of discrete decamethylferrocenium cations and hexafluorophosphate anions. The Fe and P atoms are located on special positions of site symmetry 2/m, two carbon atoms and two F atoms are located on a mirror plane and one F atom is located on a twofold rotation axis. The title compound is isostructural with the cobaltocenium complex (Braga *et al.*, 1999; Heize *et al.*, 2002).

Experimental

Decamethylferrocenium hexafluorophosphate was synthesized from the reaction between decamethylferrocene and $AlCl_3$ in refluxing cyclohexane followed by aqueous workup with NH_4PF_6. Single crystals of the title compound were obtained by slow diffusion of diethyether in a concentrated solution of the ferrocenium phosphate in acetone.

Refinement

H atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.5U_{eq}(C)] using a riding model with C—H = 0.98 Å. Two methyl groups were allowed to rotate but not to tip.

Figures

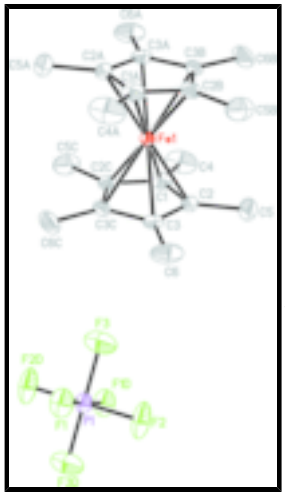


Fig. 1. Perspective view of the title compound with the atom numbering scheme; displacement ellipsoids are at the 50% probability level. H atoms are omitted for clarity. Symmetry operators for generating equivalent atoms: (A) −*x*, −*y*, −*z*; (B) −*x*, *y*, −*z*; (C) *x*, −*y*, *z*; (D) 1 − *x*, *y*, 1 − *z*.

Decamethylferrocenium hexafluorophosphate

Crystal data

$[Fe(C_{10}H_{15})_2]PF_6$	$F_{000} = 490$
$M_r = 471.26$	$D_x = 1.460$ Mg m^{-3}
Monoclinic, $C2/m$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -C 2y	Cell parameters from 2101 reflections
$a = 14.186$ (2) Å	$\theta = 4.1$–25.2º
$b = 8.9579$ (11) Å	$\mu = 0.83$ mm^{-1}
$c = 8.9902$ (13) Å	$T = 173$ (2) K
$\beta = 110.277$ (13)º	Plate, green
$V = 1071.6$ (3) Å^3	0.34 × 0.32 × 0.19 mm
$Z = 2$	

Data collection

Stoe IPDSII two-circle diffractometer	1050 independent reflections
Radiation source: fine-focus sealed tube	980 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 173$(2) K	$\theta_{max} = 25.3$º
ω scans	$\theta_{min} = 2.4$º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	$h = -17\rightarrow16$
$T_{min} = 0.765$, $T_{max} = 0.858$	$k = 0\rightarrow10$
8050 measured reflections	$l = 0\rightarrow10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H-atom parameters constrained
$wR(F^2) = 0.092$	$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.6562P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} < 0.001$
1050 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
76 parameters	$\Delta\rho_{min} = -0.53$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.0000	0.0000	0.0000	0.0139 (2)
C1	0.1113 (2)	0.0000	−0.1024 (3)	0.0247 (6)
C2	0.12225 (13)	0.1297 (2)	−0.0034 (2)	0.0224 (4)
C3	0.14081 (13)	0.0798 (2)	0.1560 (2)	0.0207 (4)
C4	0.0977 (3)	0.0000	−0.2774 (4)	0.0475 (11)
H4A	0.0611	0.0891	−0.3301	0.071*
H4B	0.1666	0.0000	−0.2782	0.071*
C5	0.11647 (17)	0.2895 (3)	−0.0577 (4)	0.0439 (7)
H5A	0.0978	0.3534	0.0162	0.066*
H5B	0.0658	0.2985	−0.1640	0.066*
H5C	0.1820	0.3206	−0.0605	0.066*
C6	0.16194 (17)	0.1773 (3)	0.2996 (3)	0.0410 (6)
H6A	0.1325	0.2762	0.2672	0.061*
H6B	0.2347	0.1870	0.3526	0.061*
H6C	0.1324	0.1323	0.3727	0.061*
P1	0.5000	0.0000	0.5000	0.0218 (3)
F1	0.45456 (15)	0.0000	0.6409 (2)	0.0373 (5)
F2	0.5000	0.1787 (2)	0.5000	0.0512 (6)
F3	0.38896 (16)	0.0000	0.3702 (2)	0.0532 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0142 (3)	0.0147 (3)	0.0126 (3)	0.000	0.0043 (2)	0.000
C1	0.0194 (13)	0.0368 (18)	0.0193 (14)	0.000	0.0084 (11)	0.000
C2	0.0155 (8)	0.0221 (11)	0.0284 (10)	−0.0026 (8)	0.0061 (8)	0.0044 (9)
C3	0.0135 (9)	0.0248 (11)	0.0216 (9)	−0.0017 (8)	0.0035 (7)	−0.0066 (8)
C4	0.0378 (18)	0.088 (3)	0.0218 (15)	0.000	0.0162 (14)	0.000
C5	0.0303 (11)	0.0307 (14)	0.0664 (17)	−0.0022 (10)	0.0114 (11)	0.0206 (13)
C6	0.0292 (11)	0.0513 (17)	0.0370 (12)	−0.0035 (11)	0.0045 (9)	−0.0258 (12)
P1	0.0303 (6)	0.0173 (6)	0.0191 (5)	0.000	0.0101 (4)	0.000
F1	0.0541 (12)	0.0360 (11)	0.0311 (10)	0.000	0.0266 (9)	0.000

F2	0.0998 (18)	0.0194 (11)	0.0481 (12)	0.000	0.0430 (12)	0.000
F3	0.0355 (11)	0.0796 (19)	0.0374 (11)	0.000	0.0037 (9)	0.000

Geometric parameters (Å, °)

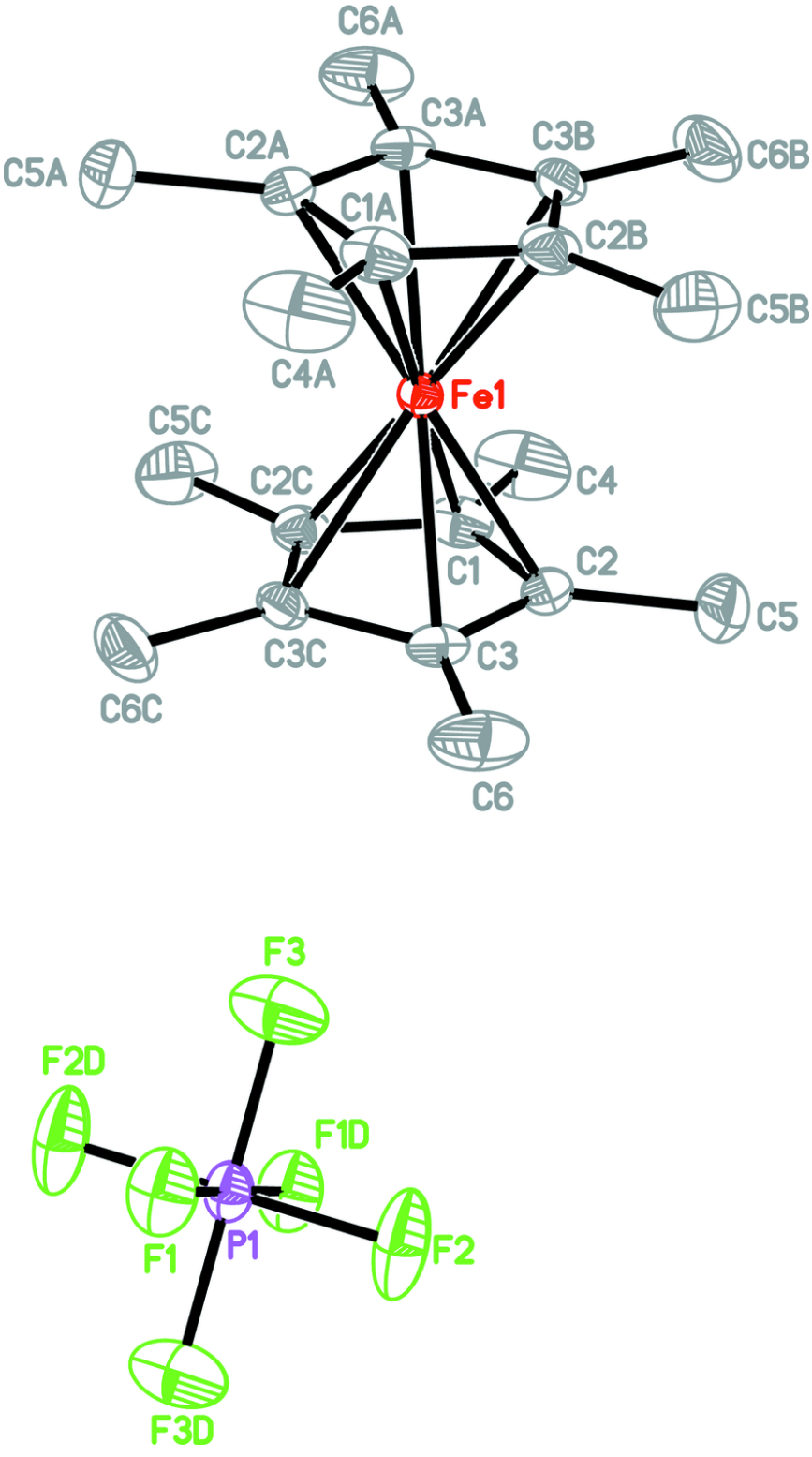
Fe1—C1	2.085 (3)	C3—C6	1.500 (3)
Fe1—C1^{i}	2.085 (3)	C4—H4A	0.9798
Fe1—C2^{ii}	2.0962 (18)	C4—H4B	0.9804
Fe1—C2^{i}	2.0962 (18)	C5—H5A	0.9800
Fe1—C2^{iii}	2.0962 (18)	C5—H5B	0.9800
Fe1—C2	2.0963 (18)	C5—H5C	0.9800
Fe1—C3^{ii}	2.1275 (18)	C6—H6A	0.9800
Fe1—C3	2.1275 (18)	C6—H6B	0.9800
Fe1—C3^{i}	2.1276 (18)	C6—H6C	0.9800
Fe1—C3^{iii}	2.1276 (18)	P1—F2^{iv}	1.601 (2)
C1—C2^{ii}	1.439 (3)	P1—F2	1.601 (2)
C1—C2	1.439 (3)	P1—F3^{iv}	1.603 (2)
C1—C4	1.517 (4)	P1—F3	1.603 (2)
C2—C3	1.435 (3)	P1—F1^{iv}	1.6085 (17)
C2—C5	1.506 (3)	P1—F1	1.6085 (17)
C3—C3^{ii}	1.430 (4)		
C1—Fe1—C1^{i}	180.00 (6)	C2—C1—C4	126.10 (12)
C1—Fe1—C2^{ii}	40.25 (7)	C2^{ii}—C1—Fe1	70.29 (12)
C1^{i}—Fe1—C2^{ii}	139.75 (7)	C2—C1—Fe1	70.29 (12)
C1—Fe1—C2^{i}	139.75 (7)	C4—C1—Fe1	127.9 (2)
C1^{i}—Fe1—C2^{i}	40.25 (7)	C3—C2—C1	108.03 (18)
C2^{ii}—Fe1—C2^{i}	112.69 (11)	C3—C2—C5	126.2 (2)
C1—Fe1—C2^{iii}	139.75 (7)	C1—C2—C5	125.8 (2)
C1^{i}—Fe1—C2^{iii}	40.25 (7)	C3—C2—Fe1	71.31 (11)
C2^{ii}—Fe1—C2^{iii}	180.00 (6)	C1—C2—Fe1	69.45 (13)
C2^{i}—Fe1—C2^{iii}	67.30 (11)	C5—C2—Fe1	125.40 (14)
C1—Fe1—C2	40.25 (7)	C3^{ii}—C3—C2	108.13 (12)
C1^{i}—Fe1—C2	139.75 (7)	C3^{ii}—C3—C6	125.59 (15)
C2^{ii}—Fe1—C2	67.31 (11)	C2—C3—C6	126.2 (2)
C2^{i}—Fe1—C2	180.0	C3^{ii}—C3—Fe1	70.36 (6)
C2^{iii}—Fe1—C2	112.69 (11)	C2—C3—Fe1	68.96 (10)
C1—Fe1—C3^{ii}	67.01 (9)	C6—C3—Fe1	128.67 (14)
C1^{i}—Fe1—C3^{ii}	112.99 (9)	C1—C4—H4A	110.9
C2^{ii}—Fe1—C3^{ii}	39.72 (8)	C1—C4—H4B	103.8
C2^{i}—Fe1—C3^{ii}	113.37 (7)	H4A—C4—H4B	111.1
C2^{iii}—Fe1—C3^{ii}	140.28 (8)	C2—C5—H5A	109.5
C2—Fe1—C3^{ii}	66.63 (7)	C2—C5—H5B	109.5

C1—Fe1—C3	67.01 (9)	H5A—C5—H5B	109.5
C1^{i}—Fe1—C3	112.99 (9)	C2—C5—H5C	109.5
C2^{ii}—Fe1—C3	66.63 (7)	H5A—C5—H5C	109.5
C2^{i}—Fe1—C3	140.28 (8)	H5B—C5—H5C	109.5
C2^{iii}—Fe1—C3	113.37 (7)	C3—C6—H6A	109.5
C2—Fe1—C3	39.72 (8)	C3—C6—H6B	109.5
C3^{ii}—Fe1—C3	39.27 (11)	H6A—C6—H6B	109.5
C1—Fe1—C3^{i}	112.99 (9)	C3—C6—H6C	109.5
C1^{i}—Fe1—C3^{i}	67.01 (9)	H6A—C6—H6C	109.5
C2^{ii}—Fe1—C3^{i}	113.37 (7)	H6B—C6—H6C	109.5
C2^{i}—Fe1—C3^{i}	39.72 (8)	F2^{iv}—P1—F2	179.999 (1)
C2^{iii}—Fe1—C3^{i}	66.63 (7)	F2^{iv}—P1—F3^{iv}	90.0
C2—Fe1—C3^{i}	140.27 (8)	F2—P1—F3^{iv}	90.0
C3^{ii}—Fe1—C3^{i}	140.73 (11)	F2^{iv}—P1—F3	90.0
C3—Fe1—C3^{i}	180.0	F2—P1—F3	90.0
C1—Fe1—C3^{iii}	112.99 (9)	F3^{iv}—P1—F3	180.0
C1^{i}—Fe1—C3^{iii}	67.01 (9)	F2^{iv}—P1—F1^{iv}	90.0
C2^{ii}—Fe1—C3^{iii}	140.28 (8)	F2—P1—F1^{iv}	90.0
C2^{i}—Fe1—C3^{iii}	66.63 (7)	F3^{iv}—P1—F1^{iv}	90.73 (11)
C2^{iii}—Fe1—C3^{iii}	39.72 (8)	F3—P1—F1^{iv}	89.27 (11)
C2—Fe1—C3^{iii}	113.37 (7)	F2^{iv}—P1—F1	90.001 (1)
C3^{ii}—Fe1—C3^{iii}	180.0	F2—P1—F1	90.0
C3—Fe1—C3^{iii}	140.73 (11)	F3^{iv}—P1—F1	89.27 (11)
C3^{i}—Fe1—C3^{iii}	39.27 (11)	F3—P1—F1	90.73 (11)
C2^{ii}—C1—C2	107.7 (2)	F1^{iv}—P1—F1	179.999 (1)
C2^{ii}—C1—C4	126.11 (12)		
C1^{i}—Fe1—C1—C2^{ii}	59.05 (8)	C3—Fe1—C2—C1	118.41 (18)
C2^{i}—Fe1—C1—C2^{ii}	−61.9 (2)	C3^{i}—Fe1—C2—C1	−61.59 (18)
C2^{iii}—Fe1—C1—C2^{ii}	180.0	C3^{iii}—Fe1—C2—C1	−98.51 (14)
C2—Fe1—C1—C2^{ii}	118.1 (2)	C1—Fe1—C2—C5	120.0 (2)
C3^{ii}—Fe1—C1—C2^{ii}	37.63 (12)	C1^{i}—Fe1—C2—C5	−60.0 (2)
C3—Fe1—C1—C2^{ii}	80.46 (14)	C2^{ii}—Fe1—C2—C5	158.17 (18)
C3^{i}—Fe1—C1—C2^{ii}	−99.54 (14)	C2^{i}—Fe1—C2—C5	11 (16)
C3^{iii}—Fe1—C1—C2^{ii}	−142.36 (12)	C2^{iii}—Fe1—C2—C5	−21.83 (18)
C1^{i}—Fe1—C1—C2	−59.05 (17)	C3^{ii}—Fe1—C2—C5	−158.5 (2)
C2^{ii}—Fe1—C1—C2	−118.1 (2)	C3—Fe1—C2—C5	−121.6 (3)
C2^{i}—Fe1—C1—C2	180.0	C3^{i}—Fe1—C2—C5	58.4 (3)
C2^{iii}—Fe1—C1—C2	61.9 (2)	C3^{iii}—Fe1—C2—C5	21.5 (2)
C3^{ii}—Fe1—C1—C2	−80.46 (14)	C1—C2—C3—C3^{ii}	−0.34 (17)
C3—Fe1—C1—C2	−37.64 (12)	C5—C2—C3—C3^{ii}	−179.64 (16)
C3^{i}—Fe1—C1—C2	142.36 (12)	Fe1—C2—C3—C3^{ii}	59.68 (5)

$C3^{iii}$—Fe1—C1—C2	99.54 (14)	C1—C2—C3—C6	176.75 (19)
$C1^{i}$—Fe1—C1—C4	180.00 (7)	C5—C2—C3—C6	−2.6 (3)
$C2^{ii}$—Fe1—C1—C4	120.95 (11)	Fe1—C2—C3—C6	−123.23 (19)
$C2^{i}$—Fe1—C1—C4	59.05 (11)	C1—C2—C3—Fe1	−60.02 (15)
$C2^{iii}$—Fe1—C1—C4	−59.05 (11)	C5—C2—C3—Fe1	120.7 (2)
C2—Fe1—C1—C4	−120.95 (11)	C1—Fe1—C3—$C3^{ii}$	−81.29 (5)
$C3^{ii}$—Fe1—C1—C4	158.59 (6)	$C1^{i}$—Fe1—C3—$C3^{ii}$	98.71 (5)
C3—Fe1—C1—C4	−158.59 (6)	$C2^{ii}$—Fe1—C3—$C3^{ii}$	−37.33 (7)
$C3^{i}$—Fe1—C1—C4	21.41 (6)	$C2^{i}$—Fe1—C3—$C3^{ii}$	60.58 (11)
$C3^{iii}$—Fe1—C1—C4	−21.41 (6)	$C2^{iii}$—Fe1—C3—$C3^{ii}$	142.67 (7)
$C2^{ii}$—C1—C2—C3	0.5 (3)	C2—Fe1—C3—$C3^{ii}$	−119.42 (11)
C4—C1—C2—C3	−175.7 (3)	$C3^{i}$—Fe1—C3—$C3^{ii}$	−91 (2)
Fe1—C1—C2—C3	61.19 (13)	$C3^{iii}$—Fe1—C3—$C3^{ii}$	180.0
$C2^{ii}$—C1—C2—C5	179.85 (14)	C1—Fe1—C3—C2	38.13 (11)
C4—C1—C2—C5	3.6 (4)	$C1^{i}$—Fe1—C3—C2	−141.87 (11)
Fe1—C1—C2—C5	−119.5 (2)	$C2^{ii}$—Fe1—C3—C2	82.09 (16)
$C2^{ii}$—C1—C2—Fe1	−60.65 (17)	$C2^{i}$—Fe1—C3—C2	180.0
C4—C1—C2—Fe1	123.1 (3)	$C2^{iii}$—Fe1—C3—C2	−97.91 (16)
C1—Fe1—C2—C3	−118.41 (18)	$C3^{ii}$—Fe1—C3—C2	119.42 (11)
$C1^{i}$—Fe1—C2—C3	61.59 (18)	$C3^{i}$—Fe1—C3—C2	28 (2)
$C2^{ii}$—Fe1—C2—C3	−80.24 (12)	$C3^{iii}$—Fe1—C3—C2	−60.58 (11)
$C2^{i}$—Fe1—C2—C3	132 (16)	C1—Fe1—C3—C6	158.3 (2)
$C2^{iii}$—Fe1—C2—C3	99.76 (12)	$C1^{i}$—Fe1—C3—C6	−21.7 (2)
$C3^{ii}$—Fe1—C2—C3	−36.92 (13)	$C2^{ii}$—Fe1—C3—C6	−157.7 (2)
$C3^{i}$—Fe1—C2—C3	180.0	$C2^{i}$—Fe1—C3—C6	−59.8 (3)
$C3^{iii}$—Fe1—C2—C3	143.08 (13)	$C2^{iii}$—Fe1—C3—C6	22.3 (2)
$C1^{i}$—Fe1—C2—C1	180.0	C2—Fe1—C3—C6	120.2 (3)
$C2^{ii}$—Fe1—C2—C1	38.16 (14)	$C3^{ii}$—Fe1—C3—C6	−120.4 (2)
$C2^{i}$—Fe1—C2—C1	−109 (16)	$C3^{i}$—Fe1—C3—C6	149 (2)
$C2^{iii}$—Fe1—C2—C1	−141.84 (14)	$C3^{iii}$—Fe1—C3—C6	59.6 (2)
$C3^{ii}$—Fe1—C2—C1	81.49 (14)		

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) *x*, −*y*, *z*; (iii) −*x*, *y*, −*z*; (iv) −*x*+1, −*y*, −*z*+1.

Fig. 1


C6A
C3A
C2A
C3B
C6B
C5A
C1A
C2B
C5B
C4A
Fe1
C5C
C2C
C4
C1
C2
C5
C3C
C3
C6C
C6
F3
F2D
F1D
F1
P1
F2
F3D