

2-(4-Ferrocenylphenyl)-4,4,5,5-tetra-methyl-1,3,2-dioxaborolane

Peter D. W. Boyd* and J. D. Paaue

Department of Chemistry, The University of Auckland, Private Bag 92019, Auckland, New Zealand

Correspondence e-mail: pdw.boyd@auckland.ac.nz

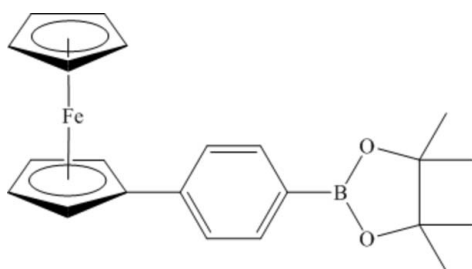
Received 23 October 2008; accepted 28 October 2008

Key indicators: single-crystal X-ray study; $T = 89$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 18.6.

In the title compound, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{17}\text{H}_{20}\text{BO}_2)]$, the two near parallel cyclopentadienyl rings of the ferrocene group are eclipsed. The benzene ring is tilted with respect to the attached cyclopentadiene ring by 17.0 (1)° and by 24.2 (1)° with respect to the dioxaborolane ring. The molecules assemble in the crystal *via* $\text{C}-\text{H}\cdots\pi$ interactions between the cyclopentadienyl H atoms and the benzene and cyclopentadienyl rings of neighbouring molecules.

Related literature

For the related tris(4-ferrocenylphenyl)boroxine benzene solvate, see: Makarov *et al.* (2004). For other related structures, see: Anderson *et al.* (2003); Nyamori & Bala (2008). For related literature, see: Leclerc *et al.* (2003).



Experimental

Crystal data

$[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{17}\text{H}_{20}\text{BO}_2)]$
 $M_r = 388.08$

Monoclinic, $P2_1/c$
 $a = 12.4439$ (3) Å

$b = 12.9832$ (3) Å
 $c = 13.0728$ (3) Å
 $\beta = 117.126$ (1)°
 $V = 1879.75$ (8) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.82$ mm⁻¹
 $T = 89$ (2) K
 $0.37 \times 0.37 \times 0.20$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.717$, $T_{\max} = 0.849$

23134 measured reflections
 4442 independent reflections
 3984 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.085$
 $S = 1.03$
 4442 reflections

239 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.86$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6}\cdots\text{C12}^{\text{i}}$	0.93	2.86	3.6302 (19)	141
$\text{C5}-\text{H5}\cdots\text{C6}^{\text{ii}}$	0.93	2.62	3.538 (2)	168

Symmetry codes: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (ii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP3* (Burnett & Johnson, 1996) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2008).

We thank Tania Groutso for help with the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2435).

References

- Anderson, F. P., Gallagher, J. F., Kenny, P. T. M., Ryan, C. & Savage, D. (2003). *Acta Cryst.* **C59**, m13–m15.
- Bruker (2006). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Burnett, M. N. & Johnson, C. K. (1996). *ORTEP3*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Leclerc, N., Serieys, I. & Attias, A.-J. (2003). *Tetrahedron Lett.* **44**, 5879–5882.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Makarov, M. V., Dyadchenko, V. P. & Antipin, M. Yu. (2004). *Russ. Chem. Bull.* **53**, 2768–2773.
- Nyamori, V. O. & Bala, M. D. (2008). *Acta Cryst.* **E64**, m1376.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Westrip, S. P. (2008). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2008). E64, m1494 [doi:10.1107/S1600536808035228]

2-(4-Ferrocenylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane

P. D. W. Boyd and J. D. Paauwe

Comment

The title compound, (I), was prepared from the reaction of lithiated 4-bromophenylferrocene with 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane in tetrahydrofuran. Unlike the related tris(4-ferrocenylphenyl)boroxine benzene solvate (Makarov *et al.* (2004), the 2-(4-ferrocenyl-phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane is monomeric (Fig. 1). The two cyclopentadienyl rings are nearly eclipsed, average torsion angle $2.1 (1)^\circ$, with a small tilt of the two planes (C1—C5 and C6—C10) of $3.5 (1)^\circ$. The distances of the iron atom to the ring centroids were $1.6514 (2) \text{ \AA}$ and $1.6475 (2) \text{ \AA}$ respectively. The phenyl ring is tilted by $17.0 (1)^\circ$ with respect to the (C6—C10) plane. This value is slightly higher than that observed in similar structures (Anderson *et al.* (2003), Nyamori and Bala (2008)). The dioxaborolane ring is in a half-chair conformation, with an O1—C17—C18—O2 torsion angle of $24.2 (1)^\circ$. The BO_2 group is rotated away from the plane of the phenyl ring system by $11.1 (2)^\circ$, and the angle between the dioxaborolane ring and the phenylplane is $9.9 (1)^\circ$. The molecules pack in the crystal, (Fig. 2), with $\text{C—H}\cdots\pi$ interactions between cyclopentadienyl hydrogen atoms and the phenyl and cyclopentadienyl rings of neighbouring molecules, Table 1.

Experimental

To a solution of 4-bromophenyl ferrocene (0.2 g, 0.59 mmol) in dry THF (10 mL) stirred at -78°C under nitrogen was added dropwise a solution of *n*-BuLi 2.5*M* in hexane (0.51 ml, 0.88 mmol). The mixture was then stirred at -78°C for 20 minutes. Then 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.18 ml, 0.88 mmol) was added, the stirring was kept at -78°C for 2 h and the mixture allowed to warm to -35°C and stir for 1 h and then warmed to room temperature. The reaction mixture was then poured into water and extracted with diethyl ether (2 x 25 ml). The combined organic layers were washed with brine and dried with Na_2SO_4 . The solvent removed under reduced pressure and purified by column chromatography (SiO_2 , Hexane/DCM = 2/1) to give the pure compound 2-(4-ferrocenyl-phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.052 g, 0.129 mmol, 23%) as an orange solid. FAB-MS ($\text{C}_{22}\text{H}_{25}^{10}\text{BFeO}_2$) 387.13293 ($\text{C}_{22}\text{H}_{25}^{11}\text{BFeO}_2$) 388.12934. ^1H NMR (CDCl_3 , 300 MHz) δ 1.36 (CH₃, s, 12H), 4.02 (CpH, s, 5H), 4.33 (CpH, t, $J=1.84$, 2H), 4.68 (CpH, t, $J=1.85$ 2H), 7.46 (ArH, d, $J=8.3$ 2H), 7.72 (ArH, d, $J=8.3$ 2H) p.p.m..

Refinement

Hydrogen atoms were placed in calculated positions and refined using the riding model [C—H 0.93–0.97 \AA], with $U_{\text{iso}}(\text{H}) = 1.2$ and 1.5 times $U_{\text{eq}}(\text{C})$ for aromatic and alkyl groups respectively. In the case of the methyl groups protons were rotated to fit the H-atom positions to the observed electron density.

Figures

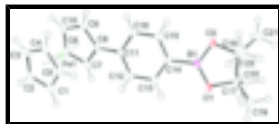


Fig. 1. Structure of (I) showing 50% probability displacement ellipsoids for non-H atoms.

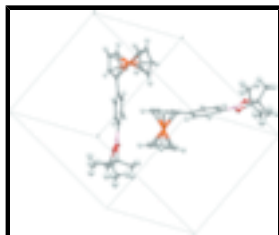


Fig. 2. The crystal packing of (I) showing C—H... π interactions between adjacent molecules. [Symmetry codes: x, y, z and $1 - x, 1 - y, 1 - z$]

2-(4-Ferrocenylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane

Crystal data

[Fe(C₅H₅)(C₁₇H₂₀BO₂)]

$M_r = 388.08$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.4439$ (3) Å

$b = 12.9832$ (3) Å

$c = 13.0728$ (3) Å

$\beta = 117.126$ (1)°

$V = 1879.75$ (8) Å³

$Z = 4$

$F_{000} = 816$

$D_x = 1.371$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 9781 reflections

$\theta = 1.8$ – 27.8 °

$\mu = 0.82$ mm⁻¹

$T = 89$ (2) K

Block, orange

$0.37 \times 0.37 \times 0.2$ mm

Data collection

Bruker SMART APEXII CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 89$ (2) K

ω scans

Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.717$, $T_{\max} = 0.849$

23134 measured reflections

4442 independent reflections

3984 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 27.8$ °

$\theta_{\min} = 1.8$ °

$h = -16 \rightarrow 14$

$k = 0 \rightarrow 17$

$l = 0 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$$R[F^2 > 2\sigma(F^2)] = 0.031$$

$$wR(F^2) = 0.085$$

$$S = 1.04$$

4442 reflections

239 parameters

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 1.0943P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.86 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.244744 (18)	0.661122 (14)	0.209954 (16)	0.01545 (8)
O2	0.36539 (9)	1.21833 (8)	-0.03562 (9)	0.0207 (2)
O1	0.16554 (9)	1.19391 (8)	-0.16076 (9)	0.0195 (2)
C11	0.26427 (12)	0.89643 (10)	0.14427 (11)	0.0157 (3)
C14	0.26540 (13)	1.05937 (10)	0.00034 (12)	0.0167 (3)
C18	0.33635 (13)	1.29767 (11)	-0.12386 (12)	0.0187 (3)
C2	0.16209 (14)	0.52057 (11)	0.16048 (14)	0.0231 (3)
H2	0.1105	0.4901	0.1854	0.028*
C16	0.37235 (12)	0.94523 (10)	0.16343 (12)	0.0178 (3)
H16	0.4447	0.9239	0.2241	0.021*
C7	0.15815 (13)	0.78484 (11)	0.23606 (12)	0.0186 (3)
H7	0.0789	0.8050	0.1895	0.022*
C13	0.15859 (13)	1.00753 (10)	-0.02096 (12)	0.0172 (3)
H13	0.0868	1.0269	-0.0836	0.021*
C8	0.26304 (13)	0.81901 (11)	0.22597 (12)	0.0167 (3)
C9	0.36571 (13)	0.76831 (11)	0.31467 (12)	0.0187 (3)
H9	0.4454	0.7758	0.3281	0.022*
C6	0.19744 (14)	0.71467 (11)	0.33018 (12)	0.0209 (3)
H6	0.1480	0.6811	0.3556	0.025*
B1	0.26500 (15)	1.15783 (11)	-0.06779 (14)	0.0174 (3)
C15	0.37260 (13)	1.02504 (11)	0.09282 (12)	0.0184 (3)
H15	0.4453	1.0564	0.1071	0.022*
C5	0.23381 (15)	0.61510 (11)	0.05503 (13)	0.0243 (3)
H5	0.2370	0.6572	-0.0011	0.029*

supplementary materials

C21	0.39377 (14)	1.39832 (11)	-0.06503 (13)	0.0238 (3)
H21A	0.3680	1.4143	-0.0079	0.036*
H21B	0.3695	1.4526	-0.1210	0.036*
H21C	0.4801	1.3916	-0.0290	0.036*
C4	0.33403 (14)	0.56821 (11)	0.14749 (13)	0.0226 (3)
H4	0.4144	0.5745	0.1628	0.027*
C10	0.32509 (14)	0.70475 (11)	0.37867 (12)	0.0208 (3)
H10	0.3735	0.6639	0.4413	0.025*
C12	0.15769 (12)	0.92783 (10)	0.04947 (12)	0.0169 (3)
H12	0.0854	0.8949	0.0335	0.020*
C17	0.19486 (13)	1.29760 (11)	-0.18488 (13)	0.0201 (3)
C22	0.39120 (14)	1.26270 (11)	-0.20117 (14)	0.0235 (3)
H22A	0.4761	1.2510	-0.1555	0.035*
H22B	0.3790	1.3150	-0.2573	0.035*
H22C	0.3530	1.2000	-0.2395	0.035*
C3	0.29014 (14)	0.50988 (11)	0.21282 (14)	0.0228 (3)
H3	0.3369	0.4715	0.2783	0.027*
C20	0.14060 (15)	1.37292 (13)	-0.13159 (16)	0.0294 (3)
H20A	0.0554	1.3605	-0.1625	0.044*
H20B	0.1539	1.4422	-0.1489	0.044*
H20C	0.1783	1.3635	-0.0498	0.044*
C19	0.13736 (16)	1.31085 (15)	-0.31429 (14)	0.0312 (4)
H19A	0.1653	1.2574	-0.3471	0.047*
H19B	0.1593	1.3768	-0.3324	0.047*
H19C	0.0511	1.3068	-0.3453	0.047*
C1	0.12727 (14)	0.58627 (12)	0.06311 (13)	0.0249 (3)
H1	0.0489	0.6067	0.0136	0.030*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.02072 (12)	0.00975 (11)	0.01600 (12)	-0.00098 (7)	0.00849 (9)	0.00033 (7)
O2	0.0219 (5)	0.0155 (5)	0.0231 (5)	-0.0012 (4)	0.0087 (4)	0.0059 (4)
O1	0.0234 (5)	0.0134 (5)	0.0205 (5)	-0.0019 (4)	0.0090 (4)	0.0031 (4)
C11	0.0225 (6)	0.0095 (5)	0.0171 (6)	-0.0009 (5)	0.0106 (5)	-0.0026 (5)
C14	0.0228 (7)	0.0119 (6)	0.0174 (6)	0.0003 (5)	0.0110 (5)	-0.0002 (5)
C18	0.0234 (7)	0.0130 (6)	0.0206 (7)	0.0000 (5)	0.0107 (6)	0.0037 (5)
C2	0.0296 (8)	0.0134 (6)	0.0292 (8)	-0.0063 (6)	0.0159 (6)	-0.0055 (6)
C16	0.0196 (6)	0.0135 (6)	0.0192 (6)	0.0003 (5)	0.0078 (5)	-0.0001 (5)
C7	0.0243 (7)	0.0137 (6)	0.0202 (7)	-0.0002 (5)	0.0122 (6)	-0.0005 (5)
C13	0.0214 (7)	0.0144 (6)	0.0155 (6)	0.0007 (5)	0.0080 (5)	-0.0011 (5)
C8	0.0235 (7)	0.0105 (6)	0.0170 (6)	-0.0017 (5)	0.0099 (5)	-0.0018 (5)
C9	0.0232 (7)	0.0140 (6)	0.0176 (6)	-0.0024 (5)	0.0081 (6)	-0.0015 (5)
C6	0.0310 (8)	0.0147 (6)	0.0214 (7)	-0.0024 (5)	0.0158 (6)	0.0000 (5)
B1	0.0228 (8)	0.0131 (7)	0.0182 (7)	-0.0004 (5)	0.0111 (6)	-0.0004 (5)
C15	0.0207 (6)	0.0142 (6)	0.0215 (7)	-0.0014 (5)	0.0106 (6)	-0.0008 (5)
C5	0.0386 (9)	0.0156 (7)	0.0207 (7)	-0.0018 (6)	0.0153 (6)	-0.0020 (5)
C21	0.0284 (7)	0.0160 (7)	0.0263 (7)	-0.0033 (6)	0.0118 (6)	0.0005 (6)

C4	0.0291 (7)	0.0154 (6)	0.0273 (7)	-0.0018 (6)	0.0165 (6)	-0.0046 (6)
C10	0.0305 (8)	0.0149 (6)	0.0153 (6)	-0.0012 (5)	0.0090 (6)	0.0011 (5)
C12	0.0210 (6)	0.0127 (6)	0.0185 (6)	-0.0031 (5)	0.0102 (5)	-0.0032 (5)
C17	0.0246 (7)	0.0128 (6)	0.0241 (7)	0.0002 (5)	0.0122 (6)	0.0038 (5)
C22	0.0303 (8)	0.0157 (6)	0.0308 (8)	-0.0003 (6)	0.0193 (7)	0.0017 (6)
C3	0.0316 (8)	0.0103 (6)	0.0272 (7)	0.0015 (5)	0.0140 (6)	0.0007 (5)
C20	0.0302 (8)	0.0184 (7)	0.0438 (10)	0.0026 (6)	0.0205 (8)	-0.0005 (7)
C19	0.0293 (8)	0.0348 (9)	0.0256 (8)	0.0004 (7)	0.0090 (7)	0.0131 (7)
C1	0.0274 (8)	0.0194 (7)	0.0216 (7)	-0.0019 (6)	0.0057 (6)	-0.0056 (6)

Geometric parameters (Å, °)

Fe1—C6	2.0357 (14)	C13—C12	1.3885 (19)
Fe1—C3	2.0388 (14)	C13—H13	0.9300
Fe1—C10	2.0431 (14)	C8—C9	1.435 (2)
Fe1—C4	2.0454 (15)	C9—C10	1.4220 (19)
Fe1—C7	2.0477 (14)	C9—H9	0.9300
Fe1—C2	2.0492 (14)	C6—C10	1.422 (2)
Fe1—C9	2.0488 (14)	C6—H6	0.9300
Fe1—C1	2.0512 (15)	C15—H15	0.9300
Fe1—C5	2.0558 (15)	C5—C4	1.419 (2)
Fe1—C8	2.0626 (14)	C5—C1	1.427 (2)
O2—B1	1.3695 (19)	C5—H5	0.9300
O2—C18	1.4636 (16)	C21—H21A	0.9600
O1—B1	1.3626 (19)	C21—H21B	0.9600
O1—C17	1.4663 (16)	C21—H21C	0.9600
C11—C12	1.4007 (19)	C4—C3	1.424 (2)
C11—C16	1.4020 (19)	C4—H4	0.9300
C11—C8	1.4718 (18)	C10—H10	0.9300
C14—C13	1.3998 (19)	C12—H12	0.9300
C14—C15	1.403 (2)	C17—C19	1.516 (2)
C14—B1	1.557 (2)	C17—C20	1.525 (2)
C18—C21	1.519 (2)	C22—H22A	0.9600
C18—C22	1.524 (2)	C22—H22B	0.9600
C18—C17	1.567 (2)	C22—H22C	0.9600
C2—C3	1.426 (2)	C3—H3	0.9300
C2—C1	1.426 (2)	C20—H20A	0.9600
C2—H2	0.9300	C20—H20B	0.9600
C16—C15	1.3887 (19)	C20—H20C	0.9600
C16—H16	0.9300	C19—H19A	0.9600
C7—C6	1.426 (2)	C19—H19B	0.9600
C7—C8	1.4405 (19)	C19—H19C	0.9600
C7—H7	0.9300	C1—H1	0.9300
C6—Fe1—C3	119.34 (6)	C11—C8—Fe1	129.97 (9)
C6—Fe1—C10	40.82 (6)	C10—C9—C8	108.51 (13)
C3—Fe1—C10	104.60 (6)	C10—C9—Fe1	69.45 (8)
C6—Fe1—C4	155.59 (6)	C8—C9—Fe1	70.09 (8)
C3—Fe1—C4	40.80 (6)	C10—C9—H9	125.7
C10—Fe1—C4	120.46 (6)	C8—C9—H9	125.7

supplementary materials

C6—Fe1—C7	40.89 (6)	Fe1—C9—H9	126.3
C3—Fe1—C7	156.19 (6)	C10—C6—C7	108.43 (12)
C10—Fe1—C7	68.79 (6)	C10—C6—Fe1	69.87 (8)
C4—Fe1—C7	162.19 (6)	C7—C6—Fe1	70.01 (8)
C6—Fe1—C2	105.52 (6)	C10—C6—H6	125.8
C3—Fe1—C2	40.82 (6)	C7—C6—H6	125.8
C10—Fe1—C2	121.05 (6)	Fe1—C6—H6	125.9
C4—Fe1—C2	68.57 (6)	O1—B1—O2	114.03 (12)
C7—Fe1—C2	121.64 (6)	O1—B1—C14	123.86 (13)
C6—Fe1—C9	68.60 (6)	O2—B1—C14	122.06 (13)
C3—Fe1—C9	121.93 (6)	C16—C15—C14	121.35 (13)
C10—Fe1—C9	40.67 (5)	C16—C15—H15	119.3
C4—Fe1—C9	107.46 (6)	C14—C15—H15	119.3
C7—Fe1—C9	68.77 (6)	C4—C5—C1	108.05 (13)
C2—Fe1—C9	157.80 (6)	C4—C5—Fe1	69.37 (8)
C6—Fe1—C1	123.42 (6)	C1—C5—Fe1	69.50 (9)
C3—Fe1—C1	68.54 (6)	C4—C5—H5	126.0
C10—Fe1—C1	158.56 (6)	C1—C5—H5	126.0
C4—Fe1—C1	68.41 (6)	Fe1—C5—H5	126.7
C7—Fe1—C1	108.84 (6)	C18—C21—H21A	109.5
C2—Fe1—C1	40.69 (6)	C18—C21—H21B	109.5
C9—Fe1—C1	160.07 (6)	H21A—C21—H21B	109.5
C6—Fe1—C5	161.33 (7)	C18—C21—H21C	109.5
C3—Fe1—C5	68.38 (6)	H21A—C21—H21C	109.5
C10—Fe1—C5	157.54 (7)	H21B—C21—H21C	109.5
C4—Fe1—C5	40.47 (6)	C5—C4—C3	108.11 (13)
C7—Fe1—C5	126.07 (6)	C5—C4—Fe1	70.16 (9)
C2—Fe1—C5	68.40 (6)	C3—C4—Fe1	69.36 (8)
C9—Fe1—C5	123.65 (6)	C5—C4—H4	125.9
C1—Fe1—C5	40.67 (6)	C3—C4—H4	125.9
C6—Fe1—C8	68.88 (5)	Fe1—C4—H4	126.1
C3—Fe1—C8	159.79 (6)	C9—C10—C6	108.03 (12)
C10—Fe1—C8	68.77 (5)	C9—C10—Fe1	69.88 (8)
C4—Fe1—C8	124.87 (6)	C6—C10—Fe1	69.31 (8)
C7—Fe1—C8	41.03 (5)	C9—C10—H10	126.0
C2—Fe1—C8	158.96 (6)	C6—C10—H10	126.0
C9—Fe1—C8	40.85 (6)	Fe1—C10—H10	126.4
C1—Fe1—C8	124.36 (6)	C13—C12—C11	120.85 (13)
C5—Fe1—C8	110.09 (6)	C13—C12—H12	119.6
B1—O2—C18	107.39 (11)	C11—C12—H12	119.6
B1—O1—C17	107.07 (11)	O1—C17—C19	107.88 (12)
C12—C11—C16	118.08 (12)	O1—C17—C20	106.56 (12)
C12—C11—C8	121.46 (12)	C19—C17—C20	110.78 (14)
C16—C11—C8	120.36 (13)	O1—C17—C18	102.89 (11)
C13—C14—C15	117.52 (12)	C19—C17—C18	114.64 (12)
C13—C14—B1	121.73 (13)	C20—C17—C18	113.31 (12)
C15—C14—B1	120.50 (13)	C18—C22—H22A	109.5
O2—C18—C21	108.44 (11)	C18—C22—H22B	109.5
O2—C18—C22	106.79 (11)	H22A—C22—H22B	109.5

C21—C18—C22	110.03 (12)	C18—C22—H22C	109.5
O2—C18—C17	102.54 (10)	H22A—C22—H22C	109.5
C21—C18—C17	114.71 (12)	H22B—C22—H22C	109.5
C22—C18—C17	113.65 (12)	C4—C3—C2	108.11 (13)
C3—C2—C1	107.76 (13)	C4—C3—Fe1	69.85 (8)
C3—C2—Fe1	69.20 (8)	C2—C3—Fe1	69.98 (8)
C1—C2—Fe1	69.73 (8)	C4—C3—H3	125.9
C3—C2—H2	126.1	C2—C3—H3	125.9
C1—C2—H2	126.1	Fe1—C3—H3	125.8
Fe1—C2—H2	126.5	C17—C20—H20A	109.5
C15—C16—C11	120.74 (13)	C17—C20—H20B	109.5
C15—C16—H16	119.6	H20A—C20—H20B	109.5
C11—C16—H16	119.6	C17—C20—H20C	109.5
C6—C7—C8	107.89 (13)	H20A—C20—H20C	109.5
C6—C7—Fe1	69.10 (8)	H20B—C20—H20C	109.5
C8—C7—Fe1	70.04 (8)	C17—C19—H19A	109.5
C6—C7—H7	126.1	C17—C19—H19B	109.5
C8—C7—H7	126.1	H19A—C19—H19B	109.5
Fe1—C7—H7	126.4	C17—C19—H19C	109.5
C12—C13—C14	121.36 (13)	H19A—C19—H19C	109.5
C12—C13—H13	119.3	H19B—C19—H19C	109.5
C14—C13—H13	119.3	C2—C1—C5	107.97 (14)
C9—C8—C7	107.14 (12)	C2—C1—Fe1	69.58 (8)
C9—C8—C11	126.85 (12)	C5—C1—Fe1	69.84 (9)
C7—C8—C11	125.90 (13)	C2—C1—H1	126.0
C9—C8—Fe1	69.06 (8)	C5—C1—H1	126.0
C7—C8—Fe1	68.93 (8)	Fe1—C1—H1	126.1

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6—H6 \cdots C12 ⁱ	0.93	2.86	3.6302 (19)	141
C5—H5 \cdots C6 ⁱⁱ	0.93	2.62	3.538 (2)	168

Symmetry codes: (i) $x, -y+3/2, z+1/2$; (ii) $x, -y+3/2, z-1/2$.

Fig. 1

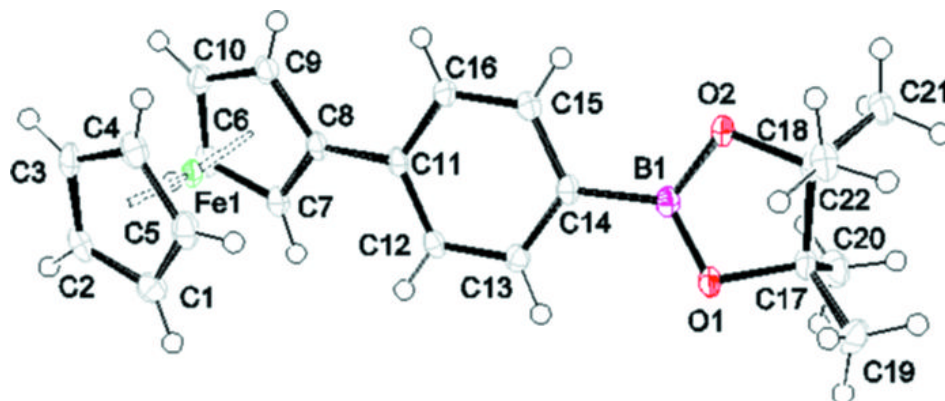


Fig. 2

