Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

Crystal structure of 1,1′-bis(2-methoxycarbonyl-2-methylpropyl)ferrocene

Yan-Feng Guo,[a] Jian-Jun Wang,[a] Wei-Juan Xu,[a] Dong-Hao Sun[a] and Qiang Gao[b]*

[a]College of Chemistry, Chemical Engineering and Materials Science, Soochow University, Suzhou, Jiangsu 215123, People's Republic of China, and [b]Key Laboratory of Eco-textiles, Ministry of Education, College of Textile & Clothing, Jiangnan University, Wuxi 214122, People's Republic of China. *Correspondence e-mail: gaoqiang@jiangnan.edu.cn

Received 26 October 2015; accepted 31 October 2015

Edited by M. Weil, Vienna University of Technology, Austria

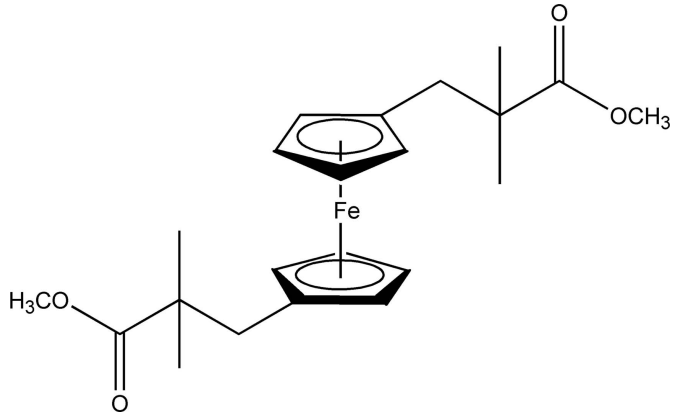
The Fe atom in the title ferrocene derivative, $[Fe(C_{11}H_{15}O_2)_2]$, is situated on an inversion centre. As a result of the point-group symmetry $\overline{1}$ of the molecule, the ferrocene moiety adopts a staggered conformation. The average Fe—C(Cp) bond length (Cp is cyclopentadienyl) is 2.045 (4) Å, in agreement with that of other disubstituted ferrocenes. The Fe—C bond length involving the substituted C atom is slightly longer [2.0521 (17) Å] than the remaining Fe—C bond lengths caused by the inductive effect of the methylene group on the Cp ring. Apart from van der Waals forces, no significant intermolecular interactions are observed in the crystal packing.

Keywords: crystal structure; inversion symmetry; disubstituted ferrocene; ester.

CCDC reference: 1434467

1. Related literature

The interest in disubstituted ferrocene compounds has increased due to their applications in the field of homogeneous catalysis, biology and medicine (Atkinson *et al.*, 2004; Gao *et al.*, 2009; Ferreira *et al.*, 2006). The presence of ester groups on these compounds make them promising candidates for the construction of metal-containing polymers (Wilbert *et al.*, 1995). Related structures have been described by Woodward *et al.* (1952); Cetina *et al.* (2003); Navarro *et al.* (2004); Pérez *et al.* (2015).



2. Experimental

2.1. Crystal data

$[Fe(C_{11}H_{15}O_2)_2]$
M_r = 414.31
Triclinic, $P\overline{1}$
a = 6.273 (3) Å
b = 8.313 (4) Å
c = 10.490 (5) Å
α = 83.833 (6)°
β = 74.405 (7)°
γ = 81.652 (8)°
V = 520.0 (4) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.75 mm^{-1}
T = 296 K
0.15 × 0.12 × 0.12 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.896, T_{max} = 0.916
2753 measured reflections
1793 independent reflections
1688 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.05
1793 reflections
124 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

We are very grateful for the financial support from the Open Project Program of Key Laboratory of Eco-textiles, Ministry of Education, Jiangnan University (No. KLET1303).

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5231).

References

Atkinson, R. C. J., Gibson, V. C. & Long, N. J. (2004). *Chem. Soc. Rev.* **33**, 313–328.

Brandenburg, K. & Berndt, M. (1999). *DIAMOND*. University of Bonn, Germany.

Bruker (2012). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cetina, M., Jukić, M., Rapić, V. & Golobič, A. (2003). *Acta Cryst.* C**59**, m212–m214.

Ferreira, C. L., Ewart, C. B., Barta, C. A., Little, S., Yardley, V., Martins, C., Polishchuk, E., Smith, P. J., Moss, J. R., Merkel, M., Adam, M. J. & Orvig, C. (2006). *Inorg. Chem.* **45**, 8414–8422.

Gao, L. M., Hernández, R., Matta, J. & Meléndez, E. (2009). *Metal-based Drugs*, Article ID 420784, doi: 10.1155/2009/420784.

Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.

Navarro, A.-E., Spinelli, N., Moustrou, C., Chaix, C., Mandrand, B. & Brisset, H. (2004). *Nucleic Acids Res.* **32**, 5310–5319.

Pérez, W. I., Rheingold, A. L. & Meléndez, E. (2015). *Acta Cryst.* E**71**, 536–539.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Wilbert, G., Wiesemann, A. & Zentel, R. (1995). *Macromol. Chem. Phys.* **196**, 3771–3788.

Woodward, R. B., Rosenblum, M. & Whiting, M. C. (1952). *J. Am. Chem. Soc.* **74**, 3458–3459.

supporting information

Acta Cryst. (2015). E**71**, m213–m214 [https://doi.org/10.1107/S2056989015020642]

Crystal structure of 1,1′-bis(2-methoxycarbonyl-2-methylpropyl)ferrocene

Yan-Feng Guo, Jian-Jun Wang, Wei-Juan Xu, Dong-Hao Sun and Qiang Gao

S1. Experimental

1,1′-bis(1-methoxy-methyl)ferrocene was first prepared by refluxing 1,1′-bis(hydroxymethyl)ferrocene in methanol and acetic acid (12:1 *v*/*v*) for 16 h. Then a solution of 1,1,-bis(1-methoxy-methyl)ferrocene (3.481 g, 12.7 mmol), 1-methoxy-1-(trimethylsiloxy)-2-methyl-1-propene (10.5 ml, 50.8 mmol) and BF_3—OEt_2 (3.5 ml, 27.9 mmol) in CH_2Cl_2 (180 ml) was stirred at 195 K for 15 min. The reaction was quenched with a satured solution of $NaHCO_3$ and extracted with CH_2Cl_2. The organic phases were combined and dried to give a viscous yellow oil, which was chromatographed over a column of silica gel using ethyl acetate/petroleum ether (1:4 *v*/*v*) as the eluent. Yellow crystals of the title compound were obtained by slow evaporation of a solution in dichloromethane/petroleum ether (333-363 K). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.97 (d, 8H, $C_5H_4FeC_5H_4$), 3.62 (s, 6H, OCH_3), 2.57 (s, 4H, CH_2), 1.08 (s, 12H, $C(CH_3)_2$). HRMS (ESI): $C_{22}H_{30}FeO_4$ calcd for $[M + H]^+$ 415.1572, found 415.1575.

S2. Refinement

H atoms were placed in calculated positions and thereafter treated as riding atoms, with C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) (Cp rings CH), 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) (methylene CH_2) and 0.96 Å U_{iso}(H) = 1.5U_{eq}(C) (methyl CH_3).

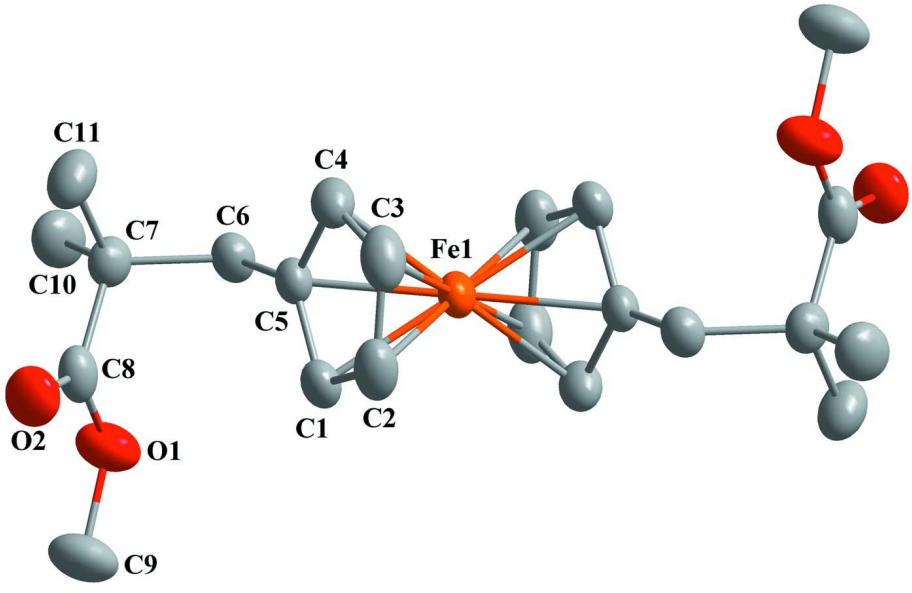


Figure 1

The molecular structure of the title complex, showing displacement ellipsoids drawn at the 50% probability level. All H atoms have been omitted for clarity. Unlabelled atoms are related to labelled ones by the symmetry operation -*x*, -*y* + 1, -*z* + 1.

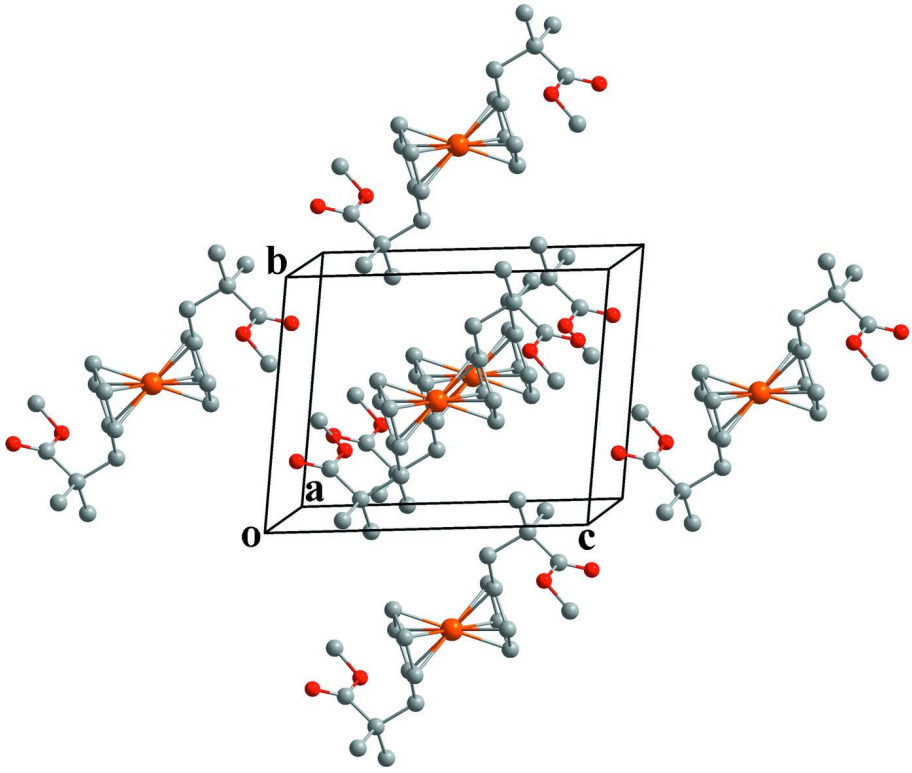


Figure 2

The packing of molecules in the crystal structure of the title compound.

1,1′-Bis(2-methoxycarbonyl-2-methylpropyl)ferrocene

Crystal data

$[Fe(C_{11}H_{15}O_2)_2]$
$M_r = 414.31$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.273$ (3) Å
$b = 8.313$ (4) Å
$c = 10.490$ (5) Å
$\alpha = 83.833$ (6)°
$\beta = 74.405$ (7)°
$\gamma = 81.652$ (8)°
$V = 520.0$ (4) Å^3
$Z = 1$
$F(000) = 220$
$D_x = 1.323$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1589 reflections
$\theta = 3.3$–$28.2°$
$\mu = 0.75$ mm^{-1}
$T = 296$ K
Block, yellow
0.15 × 0.12 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
$T_{min} = 0.896$, $T_{max} = 0.916$
2753 measured reflections
1793 independent reflections
1688 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
$h = -7 \rightarrow 5$
$k = -9 \rightarrow 9$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.074$
$S = 1.05$
1793 reflections
124 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 0.1517P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.0000	0.5000	0.5000	0.03254 (14)
O1	0.6653 (2)	0.24987 (19)	0.15534 (14)	0.0536 (4)
O2	0.4288 (3)	0.2353 (2)	0.03404 (15)	0.0646 (4)
C8	0.4892 (3)	0.1969 (2)	0.13268 (18)	0.0397 (4)
C5	0.1330 (3)	0.3282 (2)	0.36443 (16)	0.0335 (4)
C1	0.1796 (3)	0.4864 (2)	0.30665 (17)	0.0387 (4)
H1A	0.3282	0.5203	0.2685	0.046*
C6	0.2998 (3)	0.1817 (2)	0.37491 (17)	0.0377 (4)
H6A	0.2343	0.1094	0.4496	0.045*
H6B	0.4288	0.2169	0.3931	0.045*
C4	−0.1038 (3)	0.3335 (2)	0.40734 (18)	0.0410 (4)
H4A	−0.1861	0.2426	0.4513	0.049*
C7	0.3785 (3)	0.0848 (2)	0.24845 (18)	0.0372 (4)
C11	0.1817 (4)	0.0227 (3)	0.2186 (2)	0.0547 (6)
H11A	0.2329	−0.0363	0.1400	0.082*
H11B	0.0746	0.1134	0.2049	0.082*
H11C	0.1134	−0.0482	0.2920	0.082*
C10	0.5508 (4)	−0.0594 (3)	0.2709 (2)	0.0519 (5)
H10A	0.6012	−0.1197	0.1930	0.078*
H10B	0.4836	−0.1293	0.3452	0.078*
H10C	0.6753	−0.0192	0.2884	0.078*
C2	−0.0263 (4)	0.5867 (3)	0.31440 (18)	0.0467 (5)
H2A	−0.0442	0.7018	0.2825	0.056*
C9	0.7892 (5)	0.3576 (3)	0.0540 (3)	0.0721 (7)
H9A	0.9106	0.3869	0.0826	0.108*

H9B	0.6925	0.4542	0.0387	0.108*
H9C	0.8468	0.3033	−0.0267	0.108*
C3	−0.1995 (4)	0.4931 (3)	0.3759 (2)	0.0488 (5)
H3A	−0.3593	0.5316	0.3944	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0326 (2)	0.0351 (2)	0.0286 (2)	−0.00167 (15)	−0.00463 (15)	−0.00833 (14)
O1	0.0475 (8)	0.0685 (10)	0.0448 (8)	−0.0180 (7)	−0.0123 (7)	0.0122 (7)
O2	0.0833 (12)	0.0734 (11)	0.0426 (8)	−0.0069 (9)	−0.0298 (8)	0.0029 (8)
C8	0.0437 (11)	0.0398 (10)	0.0332 (10)	0.0076 (8)	−0.0094 (8)	−0.0109 (8)
C5	0.0359 (9)	0.0366 (9)	0.0269 (8)	−0.0039 (7)	−0.0040 (7)	−0.0089 (7)
C1	0.0447 (11)	0.0408 (10)	0.0270 (9)	−0.0049 (8)	−0.0019 (8)	−0.0060 (7)
C6	0.0409 (10)	0.0396 (10)	0.0306 (9)	−0.0009 (8)	−0.0075 (8)	−0.0034 (7)
C4	0.0371 (10)	0.0479 (11)	0.0395 (10)	−0.0079 (8)	−0.0069 (8)	−0.0138 (8)
C7	0.0375 (10)	0.0338 (9)	0.0394 (10)	0.0018 (8)	−0.0098 (8)	−0.0073 (8)
C11	0.0513 (12)	0.0519 (13)	0.0651 (14)	−0.0067 (10)	−0.0150 (11)	−0.0224 (11)
C10	0.0533 (13)	0.0396 (11)	0.0557 (12)	0.0082 (9)	−0.0100 (10)	−0.0017 (9)
C2	0.0610 (13)	0.0430 (11)	0.0338 (10)	0.0073 (10)	−0.0139 (9)	−0.0068 (8)
C9	0.0694 (16)	0.0802 (18)	0.0595 (15)	−0.0259 (14)	−0.0050 (13)	0.0195 (13)
C3	0.0414 (11)	0.0622 (13)	0.0449 (11)	0.0074 (10)	−0.0155 (9)	−0.0199 (10)

Geometric parameters (Å, º)

Fe1—C2^{i}	2.043 (2)	C6—C7	1.554 (3)
Fe1—C2	2.043 (2)	C6—H6A	0.9700
Fe1—C4^{i}	2.044 (2)	C6—H6B	0.9700
Fe1—C4	2.044 (2)	C4—C3	1.418 (3)
Fe1—C3^{i}	2.044 (2)	C4—H4A	0.9800
Fe1—C3	2.044 (2)	C7—C11	1.522 (3)
Fe1—C1^{i}	2.0445 (19)	C7—C10	1.536 (3)
Fe1—C1	2.0445 (19)	C11—H11A	0.9600
Fe1—C5^{i}	2.0521 (17)	C11—H11B	0.9600
Fe1—C5	2.0521 (17)	C11—H11C	0.9600
O1—C8	1.333 (3)	C10—H10A	0.9600
O1—C9	1.446 (3)	C10—H10B	0.9600
O2—C8	1.192 (2)	C10—H10C	0.9600
C8—C7	1.522 (3)	C2—C3	1.400 (3)
C5—C1	1.424 (3)	C2—H2A	0.9800
C5—C4	1.428 (3)	C9—H9A	0.9600
C5—C6	1.501 (3)	C9—H9B	0.9600
C1—C2	1.420 (3)	C9—H9C	0.9600
C1—H1A	0.9800	C3—H3A	0.9800
C2^{i}—Fe1—C2	180.0	C2—C1—C5	108.24 (18)
C2^{i}—Fe1—C4^{i}	67.98 (9)	C2—C1—Fe1	69.61 (10)
C2—Fe1—C4^{i}	112.02 (9)	C5—C1—Fe1	69.95 (10)

C2^{i}—Fe1—C4	112.02 (9)	C2—C1—H1A	125.9
C2—Fe1—C4	67.98 (9)	C5—C1—H1A	125.9
C4^{i}—Fe1—C4	180.0	Fe1—C1—H1A	125.9
C2^{i}—Fe1—C3^{i}	40.08 (9)	C5—C6—C7	113.97 (15)
C2—Fe1—C3^{i}	139.92 (9)	C5—C6—H6A	108.8
C4^{i}—Fe1—C3^{i}	40.61 (8)	C7—C6—H6A	108.8
C4—Fe1—C3^{i}	139.39 (8)	C5—C6—H6B	108.8
C2^{i}—Fe1—C3	139.92 (9)	C7—C6—H6B	108.8
C2—Fe1—C3	40.08 (9)	H6A—C6—H6B	107.7
C4^{i}—Fe1—C3	139.39 (8)	C3—C4—C5	108.21 (18)
C4—Fe1—C3	40.61 (8)	C3—C4—Fe1	69.70 (12)
C3^{i}—Fe1—C3	180.0	C5—C4—Fe1	69.92 (10)
C2^{i}—Fe1—C1^{i}	40.65 (8)	C3—C4—H4A	125.9
C2—Fe1—C1^{i}	139.35 (8)	C5—C4—H4A	125.9
C4^{i}—Fe1—C1^{i}	68.18 (8)	Fe1—C4—H4A	125.9
C4—Fe1—C1^{i}	111.82 (8)	C11—C7—C8	110.12 (17)
C3^{i}—Fe1—C1^{i}	68.00 (9)	C11—C7—C10	109.95 (17)
C3—Fe1—C1^{i}	112.00 (9)	C8—C7—C10	108.85 (16)
C2^{i}—Fe1—C1	139.35 (8)	C11—C7—C6	110.52 (16)
C2—Fe1—C1	40.65 (8)	C8—C7—C6	108.53 (15)
C4^{i}—Fe1—C1	111.82 (8)	C10—C7—C6	108.84 (16)
C4—Fe1—C1	68.18 (8)	C7—C11—H11A	109.5
C3^{i}—Fe1—C1	112.00 (9)	C7—C11—H11B	109.5
C3—Fe1—C1	68.00 (9)	H11A—C11—H11B	109.5
C1^{i}—Fe1—C1	180.0	C7—C11—H11C	109.5
C2^{i}—Fe1—C5^{i}	68.48 (8)	H11A—C11—H11C	109.5
C2—Fe1—C5^{i}	111.52 (8)	H11B—C11—H11C	109.5
C4^{i}—Fe1—C5^{i}	40.80 (8)	C7—C10—H10A	109.5
C4—Fe1—C5^{i}	139.20 (8)	C7—C10—H10B	109.5
C3^{i}—Fe1—C5^{i}	68.51 (8)	H10A—C10—H10B	109.5
C3—Fe1—C5^{i}	111.49 (8)	C7—C10—H10C	109.5
C1^{i}—Fe1—C5^{i}	40.67 (7)	H10A—C10—H10C	109.5
C1—Fe1—C5^{i}	139.33 (7)	H10B—C10—H10C	109.5
C2^{i}—Fe1—C5	111.52 (8)	C3—C2—C1	108.31 (18)
C2—Fe1—C5	68.48 (8)	C3—C2—Fe1	69.99 (12)
C4^{i}—Fe1—C5	139.20 (8)	C1—C2—Fe1	69.73 (11)
C4—Fe1—C5	40.80 (8)	C3—C2—H2A	125.8
C3^{i}—Fe1—C5	111.49 (8)	C1—C2—H2A	125.8
C3—Fe1—C5	68.51 (8)	Fe1—C2—H2A	125.8
C1^{i}—Fe1—C5	139.33 (7)	O1—C9—H9A	109.5
C1—Fe1—C5	40.67 (7)	O1—C9—H9B	109.5
C5^{i}—Fe1—C5	180.00 (7)	H9A—C9—H9B	109.5
C8—O1—C9	117.84 (18)	O1—C9—H9C	109.5
O2—C8—O1	123.17 (19)	H9A—C9—H9C	109.5
O2—C8—C7	125.6 (2)	H9B—C9—H9C	109.5
O1—C8—C7	111.19 (16)	C2—C3—C4	108.28 (18)
C1—C5—C4	106.96 (17)	C2—C3—Fe1	69.92 (12)
C1—C5—C6	126.84 (17)	C4—C3—Fe1	69.69 (11)

C4—C5—C6	126.17 (17)	C2—C3—H3A	125.9
C1—C5—Fe1	69.38 (10)	C4—C3—H3A	125.9
C4—C5—Fe1	69.28 (10)	Fe1—C3—H3A	125.9
C6—C5—Fe1	127.75 (13)		

Symmetry code: (i) $-x$, $-y+1$, $-z+1$.