



# Crystal structure of 1,1'-bis(2-methoxy-carbonyl-2-methylpropyl)ferrocene

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Received 26 October 2015; accepted 31 October 2015

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

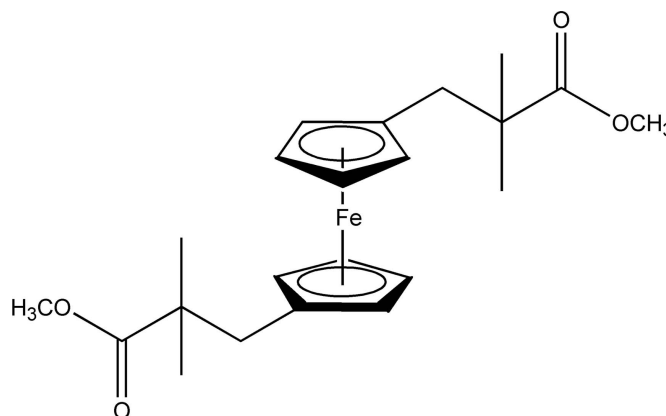
The Fe atom in the title ferrocene derivative,  $[\text{Fe}(\text{C}_{11}\text{H}_{15}\text{O}_2)_2]$ , is situated on an inversion centre. As a result of the point-group symmetry  $\bar{1}$  of the molecule, the ferrocene moiety adopts a staggered conformation. The average Fe—C(Cp) bond length (Cp is 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

$[\text{Fe}(\text{C}_{11}\text{H}_{15}\text{O}_2)_2]$	$\gamma = 81.652 (8)^\circ$
$M_r = 414.31$	$V = 520.0 (4) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 1$
$a = 6.273 (3) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 8.313 (4) \text{ \AA}$	$\mu = 0.75 \text{ mm}^{-1}$
$c = 10.490 (5) \text{ \AA}$	$T = 296 \text{ K}$
$\alpha = 83.833 (6)^\circ$	$0.15 \times 0.12 \times 0.12 \text{ mm}$
$\beta = 74.405 (7)^\circ$	

### 2.2. Data collection

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

### 2.3. Refinement

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

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).

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## supporting information

*Acta Cryst.* (2015). E71, m213–m214 [doi:10.1107/S2056989015020642]

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

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## 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  $\text{BF}_3\text{---OEt}_2$  (3.5 ml, 27.9 mmol) in  $\text{CH}_2\text{Cl}_2$  (180 ml) was stirred at 195 K for 15 min. The reaction was quenched with a saturated solution of  $\text{NaHCO}_3$  and extracted with  $\text{CH}_2\text{Cl}_2$ . 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).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.97 (d, 8H,  $\text{C}_3\text{H}_4\text{FeC}_3\text{H}_4$ ), 3.62 (s, 6H,  $\text{OCH}_3$ ), 2.57 (s, 4H,  $\text{CH}_2$ ), 1.08 (s, 12H,  $\text{C}(\text{CH}_3)_2$ ). HRMS (ESI):  $\text{C}_{22}\text{H}_{30}\text{FeO}_4$  calcd for  $[M + \text{H}]^+$  415.1572, found 415.1575.

## S2. Refinement

H atoms were placed in calculated positions and thereafter treated as riding atoms, with  $\text{C—H} = 0.98 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  (Cp rings CH),  $0.97 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  (methylene  $\text{CH}_2$ ) and  $0.96 \text{ \AA}$   $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  (methyl  $\text{CH}_3$ ).

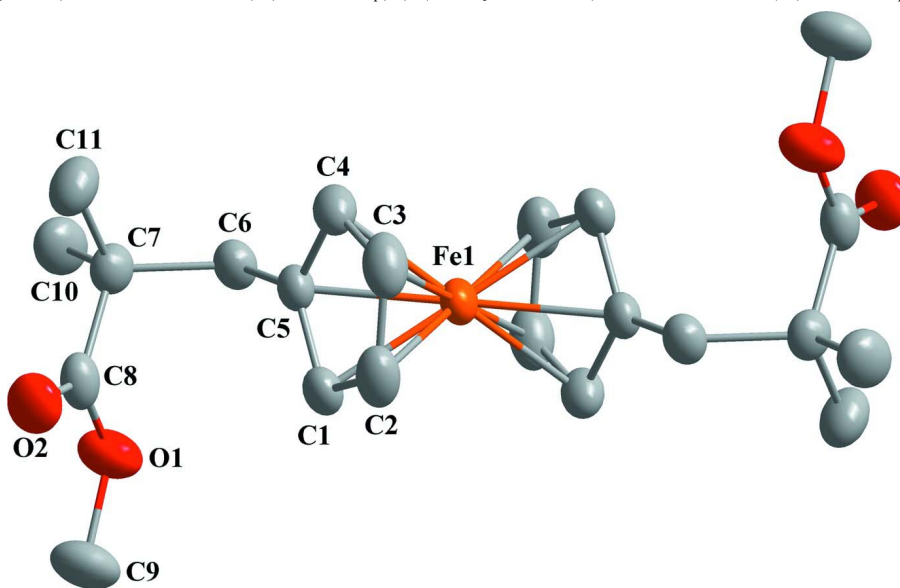


Figure 1

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  $-x, -y + 1, -z + 1$ .

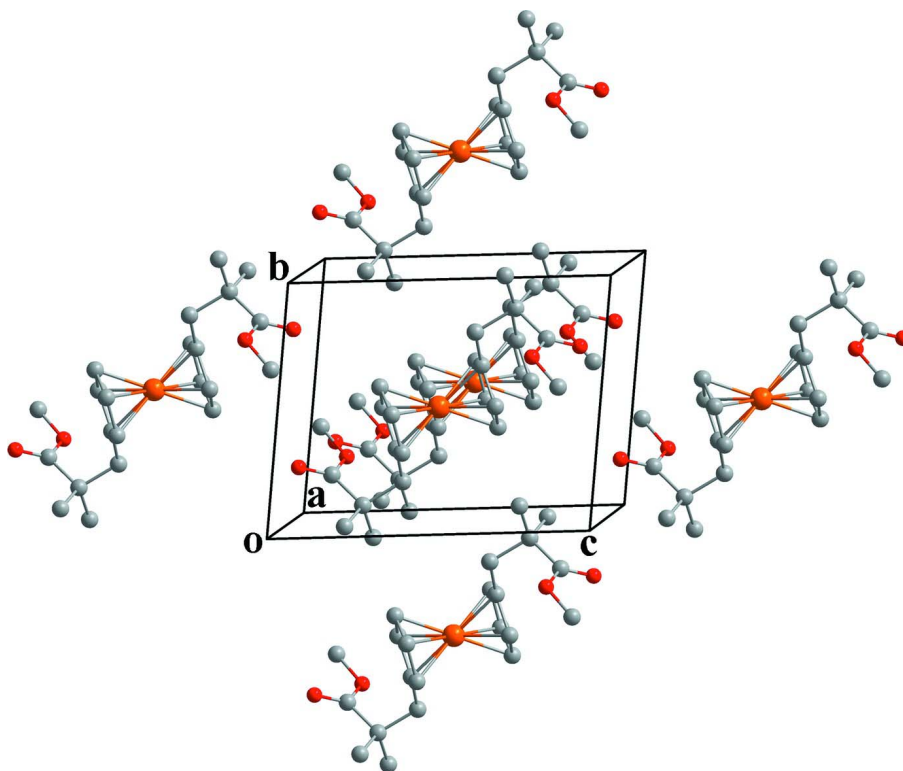


Figure 2

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

### 1,1'-Bis(2-methoxycarbonyl-2-methylpropyl)ferrocene

#### Crystal data

[Fe(C<sub>11</sub>H<sub>15</sub>O<sub>2</sub>)<sub>2</sub>]

$M_r = 414.31$

Triclinic,  $P\bar{1}$

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<sup>3</sup>

$Z = 1$

$F(000) = 220$

$D_x = 1.323$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1589 reflections

$\theta = 3.3$ – $28.2$ °

$\mu = 0.75$  mm<sup>-1</sup>

$T = 296$  K

Block, yellow

$0.15 \times 0.12 \times 0.12$  mm

#### Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  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  $I > 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 25.0$ °,  $\theta_{\min} = 2.0$ °

$h = -7 \rightarrow 5$

$k = -9 \rightarrow 9$

$l = -12 \rightarrow 12$

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  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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, °)*

Fe1—C2 <sup>i</sup>	2.043 (2)	C6—C7	1.554 (3)
Fe1—C2	2.043 (2)	C6—H6A	0.9700
Fe1—C4 <sup>i</sup>	2.044 (2)	C6—H6B	0.9700
Fe1—C4	2.044 (2)	C4—C3	1.418 (3)
Fe1—C3 <sup>i</sup>	2.044 (2)	C4—H4A	0.9800
Fe1—C3	2.044 (2)	C7—C11	1.522 (3)
Fe1—C1 <sup>i</sup>	2.0445 (19)	C7—C10	1.536 (3)
Fe1—C1	2.0445 (19)	C11—H11A	0.9600
Fe1—C5 <sup>i</sup>	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 <sup>i</sup> —Fe1—C2	180.0	C2—C1—C5	108.24 (18)
C2 <sup>i</sup> —Fe1—C4 <sup>i</sup>	67.98 (9)	C2—C1—Fe1	69.61 (10)
C2—Fe1—C4 <sup>i</sup>	112.02 (9)	C5—C1—Fe1	69.95 (10)

C2 <sup>i</sup> —Fe1—C4	112.02 (9)	C2—C1—H1A	125.9
C2—Fe1—C4	67.98 (9)	C5—C1—H1A	125.9
C4 <sup>i</sup> —Fe1—C4	180.0	Fe1—C1—H1A	125.9
C2 <sup>i</sup> —Fe1—C3 <sup>i</sup>	40.08 (9)	C5—C6—C7	113.97 (15)
C2—Fe1—C3 <sup>i</sup>	139.92 (9)	C5—C6—H6A	108.8
C4 <sup>i</sup> —Fe1—C3 <sup>i</sup>	40.61 (8)	C7—C6—H6A	108.8
C4—Fe1—C3 <sup>i</sup>	139.39 (8)	C5—C6—H6B	108.8
C2 <sup>i</sup> —Fe1—C3	139.92 (9)	C7—C6—H6B	108.8
C2—Fe1—C3	40.08 (9)	H6A—C6—H6B	107.7
C4 <sup>i</sup> —Fe1—C3	139.39 (8)	C3—C4—C5	108.21 (18)
C4—Fe1—C3	40.61 (8)	C3—C4—Fe1	69.70 (12)
C3 <sup>i</sup> —Fe1—C3	180.0	C5—C4—Fe1	69.92 (10)
C2 <sup>i</sup> —Fe1—C1 <sup>i</sup>	40.65 (8)	C3—C4—H4A	125.9
C2—Fe1—C1 <sup>i</sup>	139.35 (8)	C5—C4—H4A	125.9
C4 <sup>i</sup> —Fe1—C1 <sup>i</sup>	68.18 (8)	Fe1—C4—H4A	125.9
C4—Fe1—C1 <sup>i</sup>	111.82 (8)	C11—C7—C8	110.12 (17)
C3 <sup>i</sup> —Fe1—C1 <sup>i</sup>	68.00 (9)	C11—C7—C10	109.95 (17)
C3—Fe1—C1 <sup>i</sup>	112.00 (9)	C8—C7—C10	108.85 (16)
C2 <sup>i</sup> —Fe1—C1	139.35 (8)	C11—C7—C6	110.52 (16)
C2—Fe1—C1	40.65 (8)	C8—C7—C6	108.53 (15)
C4 <sup>i</sup> —Fe1—C1	111.82 (8)	C10—C7—C6	108.84 (16)
C4—Fe1—C1	68.18 (8)	C7—C11—H11A	109.5
C3 <sup>i</sup> —Fe1—C1	112.00 (9)	C7—C11—H11B	109.5
C3—Fe1—C1	68.00 (9)	H11A—C11—H11B	109.5
C1 <sup>i</sup> —Fe1—C1	180.0	C7—C11—H11C	109.5
C2 <sup>i</sup> —Fe1—C5 <sup>i</sup>	68.48 (8)	H11A—C11—H11C	109.5
C2—Fe1—C5 <sup>i</sup>	111.52 (8)	H11B—C11—H11C	109.5
C4 <sup>i</sup> —Fe1—C5 <sup>i</sup>	40.80 (8)	C7—C10—H10A	109.5
C4—Fe1—C5 <sup>i</sup>	139.20 (8)	C7—C10—H10B	109.5
C3 <sup>i</sup> —Fe1—C5 <sup>i</sup>	68.51 (8)	H10A—C10—H10B	109.5
C3—Fe1—C5 <sup>i</sup>	111.49 (8)	C7—C10—H10C	109.5
C1 <sup>i</sup> —Fe1—C5 <sup>i</sup>	40.67 (7)	H10A—C10—H10C	109.5
C1—Fe1—C5 <sup>i</sup>	139.33 (7)	H10B—C10—H10C	109.5
C2 <sup>i</sup> —Fe1—C5	111.52 (8)	C3—C2—C1	108.31 (18)
C2—Fe1—C5	68.48 (8)	C3—C2—Fe1	69.99 (12)
C4 <sup>i</sup> —Fe1—C5	139.20 (8)	C1—C2—Fe1	69.73 (11)
C4—Fe1—C5	40.80 (8)	C3—C2—H2A	125.8
C3 <sup>i</sup> —Fe1—C5	111.49 (8)	C1—C2—H2A	125.8
C3—Fe1—C5	68.51 (8)	Fe1—C2—H2A	125.8
C1 <sup>i</sup> —Fe1—C5	139.33 (7)	O1—C9—H9A	109.5
C1—Fe1—C5	40.67 (7)	O1—C9—H9B	109.5
C5 <sup>i</sup> —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)

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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)		

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