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2-Chloro-1-ferrocenylethanone

C. John McAdam and Jim Simpson*

Department of Chemistry, University of Otago, PO Box 56, Dunedin, New Zealand. *Correspondence e-mail: jsimpson@alkali.otago.ac.nz

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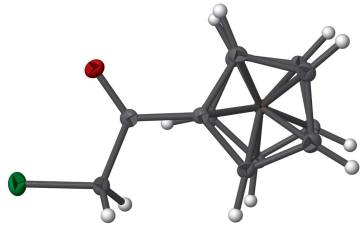
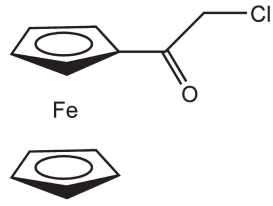
Structural data: full structural data are available from iucrdata.iucr.org

The title molecule, [Fe(C_5H_5)($C_7H_6C_lO$)], comprises a ferrocene unit with a 2-chloroethanone substituent on one of the cyclopentadienyl (Cp) rings. The two Cp rings are almost coplanar with an angle of 1.28 (1)° between them. In the crystal, C—H···Cl and C—H···O hydrogen bonds together with an edge-to-face C—H···π contact involving the unsubstituted Cp ring stack molecules along the *c*-axis direction.

3D view



Chemical scheme



Structure description

The title compound is an important synthon for the preparation of acylferrocenyl derivatives (Liu *et al.*, 2010; Bendrath *et al.*, 2011; Zheng *et al.*, (2012). The substituted Cp ring caries a 2-chloroethanone substituent, Fig. 1, and the Cp rings are slightly staggered with a mean C···*Cg*1···*Cg*2···C angle of 6.87 (19)° [*Cg*1 and *Cg*2 are the centroids of the C3–C7 and C8–C12 Cp rings, respectively]. The Cp rings are almost coplanar with an angle of 1.28 (1) ° between them, while the dihedral angle between the planar 2-chloroethanone unit (r.m.s. deviation = 0.016 Å) and the Cp ring to which it is bound is 6.07 (8)°. Bond distances and angles for the molecule are close to those found in the structure of a co-crystal of the title compound with acetylferrocene (Erben *et al.*, 2011).

In the crystal structure, the carbonyl oxygen atom, O1, acts as a bifurcated acceptor while C9 is a bifurcated donor, with C1—H1···O1, C9—H9..O1 and C9—H9···Cl1 hydrogen bonds, Table 1, forming rows of molecules along the *bc* diagonal. An additional C11—H11···Cl1 hydrogen bond together with an edge-to-face C1–H1*A*···π contact involving the unsubstituted Cp ring complete the contributions to the crystal packing, stacking molecules along the *c*-axis direction, Fig. 2.

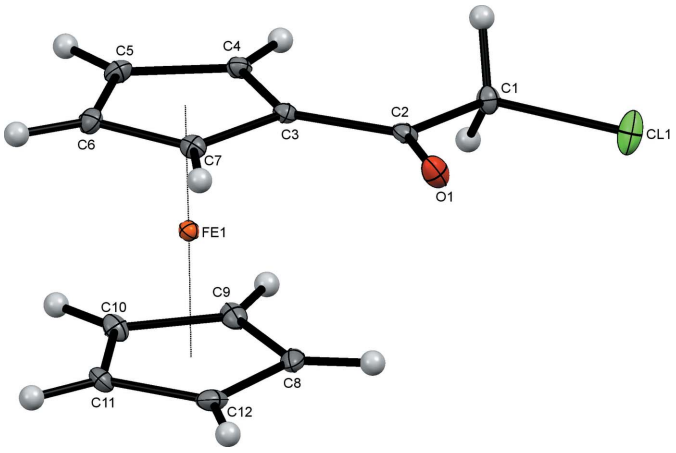


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

The title compound was synthesised by a literature method (Fang *et al.*, 2003). Crystals for the X-ray study were grown from a CH_2Cl_2 solution layered with hexane.

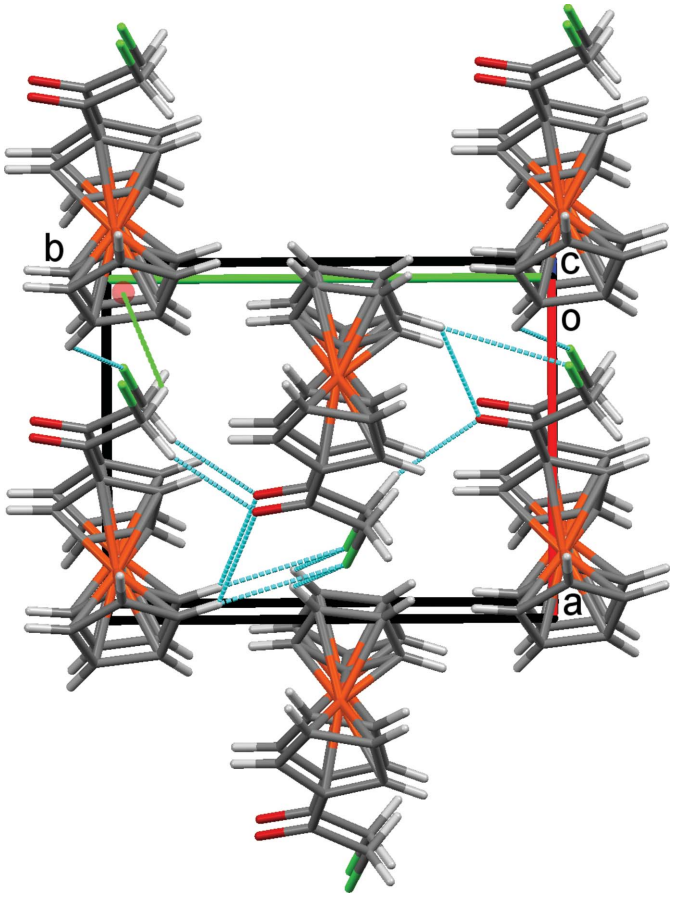


Figure 2
Crystal packing of the title compound viewed along the *c*-axis direction. A representative C—H$\cdots\pi$ contact is shown as a green dashed line. Other hydrogen bonds are drawn as blue dashed lines.

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*2 is the centroid of the C8–C12 Cp ring.

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C11—H11$\cdots$Cl1^{i}	0.95	2.88	3.4196 (19)	117
C1—H1*B*$\cdots$O1^{ii}	0.99	2.30	3.256 (2)	163
C9—H9$\cdots$Cl1^{ii}	0.95	2.95	3.841 (2)	157
C9—H9$\cdots$O1^{ii}	0.95	2.56	3.262 (2)	131
C1—H1*A*$\cdots$*Cg*2^{iii}	0.99	2.69	3.4383 (18)	132

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

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. *checkCIF* implemented in *PLATON* (Spek, 2009) signals the possibility of additional symmetry in the structure. However, the ellipsoids across the possible mirror plane are evenly sized and there is no long–short pattern of bond lengths to suggest missing symmetry. Furthermore, one ring is noticeably twisted with respect to the other, destroying mirror symmetry for the $FeCp_2$ units (ignoring substituents). Attempts at refinement in space group $P2_1/m$ were singularly unsuccessful. The structure was refined as an inversion twin with a 0.691 (12):0.309 (12) domain ratio.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Fe(C_5H_5)(C_7H_6ClO)]$
M_r	262.51
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	92
a, *b*, *c* (Å)	7.4235 (4), 9.6339 (4), 7.5209 (3)
β (°)	99.728 (2)
V (Å^3)	530.14 (4)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	1.64
Crystal size (mm)	0.32 × 0.29 × 0.11
Data collection	
Diffractometer	Bruker APEXII CCD area-detector diffractometer
Absorption correction	Multi-scan (*SADABS*; Bruker, 2011)
T_{min}, T_{max}	0.778, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9868, 3658, 3612
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.773
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.021, 0.053, 1.06
No. of reflections	3658
No. of parameters	137
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.01, −0.43
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.309 (12)

Computer programs: *APEX2* (Bruker, 2011), *SAINT* (Bruker, 2011), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *TITAN2000* (Hunter & Simpson, 1999), *Mercury* (Macrae *et al.*, 2008), *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009), *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x152177 [https://doi.org/10.1107/S241431461502177X]

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Crystal data

$[Fe(C_5H_5)(C_7H_6ClO)]$
M_r = 262.51
Monoclinic, $P2_1$
a = 7.4235 (4) Å
b = 9.6339 (4) Å
c = 7.5209 (3) Å
β = 99.728 (2)°
V = 530.14 (4) Å^3
Z = 2
$F(000)$ = 268
D_x = 1.644 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8073 reflections
θ = 2.8–33.3°
μ = 1.64 mm^{-1}
T = 92 K
Block, orange
0.32 × 0.29 × 0.11 mm

Data collection

Bruker APEXII CCD area detector diffractometer
Radiation source: fine-focus sealed tube
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2011)
T_{min} = 0.778, T_{max} = 1.000
9868 measured reflections
3658 independent reflections
3612 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 33.3°, θ_{min} = 3.5°
h = −11→7
k = −14→14
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.053
S = 1.06
3658 reflections
137 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 0.0787P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.01 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.309 (12)

Special details

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

Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.77444 (7)	0.45989 (5)	0.70617 (6)	0.02778 (11)
C1	0.6570 (2)	0.41602 (18)	0.4880 (2)	0.0159 (3)
H1A	0.7421	0.3671	0.4211	0.019*
H1B	0.5559	0.3516	0.4999	0.019*
C2	0.5800 (2)	0.54234 (17)	0.3809 (2)	0.0134 (3)
O1	0.60745 (19)	0.66056 (14)	0.4372 (2)	0.0205 (3)
C3	0.4737 (2)	0.51284 (16)	0.2015 (2)	0.0119 (3)
C4	0.4327 (2)	0.37918 (18)	0.1191 (2)	0.0142 (3)
H4	0.4760	0.2920	0.1676	0.017*
C5	0.3146 (3)	0.4020 (2)	−0.0494 (3)	0.0167 (3)
H5	0.2670	0.3323	−0.1338	0.020*
C6	0.2806 (3)	0.5478 (2)	−0.0689 (3)	0.0161 (3)
H6	0.2057	0.5913	−0.1681	0.019*
C7	0.3778 (2)	0.61677 (18)	0.0852 (2)	0.0140 (3)
H7	0.3790	0.7139	0.1072	0.017*
Fe1	0.20089 (3)	0.47611 (2)	0.16401 (2)	0.01033 (6)
C8	0.1248 (2)	0.4631 (3)	0.4129 (2)	0.0198 (3)
H8	0.2041	0.4677	0.5260	0.024*
C9	0.0737 (3)	0.3400 (2)	0.3121 (3)	0.0180 (3)
H9	0.1123	0.2483	0.3461	0.022*
C10	−0.0457 (3)	0.3791 (2)	0.1508 (3)	0.0162 (3)
H10	−0.1001	0.3177	0.0582	0.019*
C11	−0.0692 (3)	0.5260 (2)	0.1523 (2)	0.0154 (3)
H11	−0.1421	0.5795	0.0612	0.018*
C12	0.0364 (3)	0.5785 (2)	0.3151 (3)	0.0196 (4)
H12	0.0460	0.6729	0.3517	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0403 (3)	0.0232 (2)	0.01616 (16)	−0.0061 (2)	−0.00591 (16)	0.00289 (16)
C1	0.0159 (8)	0.0150 (7)	0.0158 (7)	−0.0018 (6)	0.0000 (6)	−0.0003 (6)
C2	0.0079 (7)	0.0149 (7)	0.0176 (7)	−0.0005 (5)	0.0026 (5)	−0.0010 (5)
O1	0.0180 (6)	0.0147 (6)	0.0266 (6)	0.0006 (5)	−0.0027 (5)	−0.0044 (5)
C3	0.0095 (7)	0.0117 (6)	0.0146 (6)	−0.0003 (5)	0.0026 (5)	−0.0005 (5)
C4	0.0133 (7)	0.0135 (7)	0.0161 (7)	0.0021 (6)	0.0036 (6)	−0.0021 (6)
C5	0.0193 (9)	0.0171 (8)	0.0146 (8)	0.0004 (6)	0.0054 (6)	−0.0033 (6)
C6	0.0179 (9)	0.0178 (8)	0.0129 (7)	0.0009 (6)	0.0039 (6)	0.0019 (6)
C7	0.0134 (7)	0.0127 (7)	0.0164 (7)	−0.0005 (6)	0.0037 (6)	0.0022 (5)
Fe1	0.00899 (9)	0.01107 (9)	0.01090 (9)	−0.00018 (9)	0.00159 (6)	−0.00057 (8)
C8	0.0130 (6)	0.0346 (10)	0.0122 (6)	−0.0008 (8)	0.0035 (5)	−0.0002 (8)
C9	0.0137 (8)	0.0218 (8)	0.0187 (8)	−0.0011 (7)	0.0036 (6)	0.0071 (7)
C10	0.0122 (8)	0.0152 (7)	0.0206 (8)	−0.0031 (6)	0.0012 (6)	0.0016 (6)
C11	0.0092 (7)	0.0175 (7)	0.0188 (7)	0.0016 (6)	0.0007 (6)	0.0012 (6)
C12	0.0156 (9)	0.0218 (8)	0.0234 (9)	−0.0003 (7)	0.0091 (7)	−0.0071 (7)

Geometric parameters (Å, º)

Cl1—C1	1.7741 (17)	C7—Fe1	2.0434 (17)
C1—C2	1.517 (2)	C7—H7	0.9500
C1—H1A	0.9900	Fe1—C10	2.0426 (18)
C1—H1B	0.9900	Fe1—C8	2.0487 (15)
C2—O1	1.220 (2)	Fe1—C11	2.0491 (19)
C2—C3	1.471 (2)	Fe1—C9	2.0508 (19)
C3—C7	1.437 (2)	Fe1—C12	2.0555 (19)
C3—C4	1.439 (2)	C8—C9	1.424 (3)
C3—Fe1	2.0284 (16)	C8—C12	1.430 (3)
C4—C5	1.430 (3)	C8—H8	0.9500
C4—Fe1	2.0357 (17)	C9—C10	1.427 (3)
C4—H4	0.9500	C9—H9	0.9500
C5—C6	1.431 (2)	C10—C11	1.426 (3)
C5—Fe1	2.0631 (19)	C10—H10	0.9500
C5—H5	0.9500	C11—C12	1.430 (3)
C6—C7	1.421 (3)	C11—H11	0.9500
C6—Fe1	2.0604 (19)	C12—H12	0.9500
C6—H6	0.9500		
C2—C1—Cl1	112.47 (12)	C7—Fe1—C9	163.52 (7)
C2—C1—H1A	109.1	C8—Fe1—C9	40.65 (9)
Cl1—C1—H1A	109.1	C11—Fe1—C9	68.67 (8)
C2—C1—H1B	109.1	C3—Fe1—C12	120.81 (7)
Cl1—C1—H1B	109.1	C4—Fe1—C12	155.48 (8)
H1A—C1—H1B	107.8	C10—Fe1—C12	68.61 (8)
O1—C2—C3	122.05 (16)	C7—Fe1—C12	108.26 (8)
O1—C2—C1	122.61 (16)	C8—Fe1—C12	40.77 (9)
C3—C2—C1	115.32 (14)	C11—Fe1—C12	40.79 (8)
C7—C3—C4	108.32 (14)	C9—Fe1—C12	68.62 (9)
C7—C3—C2	123.89 (14)	C3—Fe1—C6	68.65 (7)
C4—C3—C2	127.55 (15)	C4—Fe1—C6	68.92 (8)
C7—C3—Fe1	69.90 (10)	C10—Fe1—C6	119.73 (8)
C4—C3—Fe1	69.53 (9)	C7—Fe1—C6	40.53 (7)
C2—C3—Fe1	121.95 (11)	C8—Fe1—C6	163.83 (8)
C5—C4—C3	107.29 (15)	C11—Fe1—C6	107.73 (8)
C5—C4—Fe1	70.61 (11)	C9—Fe1—C6	154.25 (8)
C3—C4—Fe1	68.99 (9)	C12—Fe1—C6	126.31 (9)
C5—C4—H4	126.4	C3—Fe1—C5	68.78 (7)
C3—C4—H4	126.4	C4—Fe1—C5	40.84 (8)
Fe1—C4—H4	125.6	C10—Fe1—C5	106.55 (8)
C4—C5—C6	108.22 (17)	C7—Fe1—C5	68.67 (7)
C4—C5—Fe1	68.55 (10)	C8—Fe1—C5	154.42 (9)
C6—C5—Fe1	69.60 (12)	C11—Fe1—C5	125.02 (8)
C4—C5—H5	125.9	C9—Fe1—C5	119.18 (8)
C6—C5—H5	125.9	C12—Fe1—C5	162.89 (8)
Fe1—C5—H5	127.5	C6—Fe1—C5	40.60 (7)

C7—C6—C5	108.61 (18)	C9—C8—C12	108.40 (15)
C7—C6—Fe1	69.09 (10)	C9—C8—Fe1	69.75 (10)
C5—C6—Fe1	69.80 (12)	C12—C8—Fe1	69.87 (10)
C7—C6—H6	125.7	C9—C8—H8	125.8
C5—C6—H6	125.7	C12—C8—H8	125.8
Fe1—C6—H6	127.0	Fe1—C8—H8	126.2
C6—C7—C3	107.55 (15)	C8—C9—C10	107.72 (17)
C6—C7—Fe1	70.38 (11)	C8—C9—Fe1	69.60 (10)
C3—C7—Fe1	68.78 (9)	C10—C9—Fe1	69.30 (10)
C6—C7—H7	126.2	C8—C9—H9	126.1
C3—C7—H7	126.2	C10—C9—H9	126.1
Fe1—C7—H7	126.2	Fe1—C9—H9	126.5
C3—Fe1—C4	41.48 (7)	C11—C10—C9	108.32 (18)
C3—Fe1—C10	162.20 (7)	C11—C10—Fe1	69.85 (12)
C4—Fe1—C10	123.89 (8)	C9—C10—Fe1	69.91 (11)
C3—Fe1—C7	41.32 (7)	C11—C10—H10	125.8
C4—Fe1—C7	69.72 (8)	C9—C10—H10	125.8
C10—Fe1—C7	154.67 (7)	Fe1—C10—H10	126.0
C3—Fe1—C8	107.88 (7)	C10—C11—C12	107.91 (17)
C4—Fe1—C8	119.67 (8)	C10—C11—Fe1	69.36 (11)
C10—Fe1—C8	68.48 (8)	C12—C11—Fe1	69.85 (11)
C7—Fe1—C8	126.57 (8)	C10—C11—H11	126.0
C3—Fe1—C11	155.78 (8)	C12—C11—H11	126.0
C4—Fe1—C11	161.59 (7)	Fe1—C11—H11	126.3
C10—Fe1—C11	40.79 (9)	C8—C12—C11	107.64 (17)
C7—Fe1—C11	120.28 (7)	C8—C12—Fe1	69.36 (10)
C8—Fe1—C11	68.57 (7)	C11—C12—Fe1	69.37 (11)
C3—Fe1—C9	125.21 (7)	C8—C12—H12	126.2
C4—Fe1—C9	106.05 (8)	C11—C12—H12	126.2
C10—Fe1—C9	40.79 (7)	Fe1—C12—H12	126.7

Cl1—C1—C2—O1	4.3 (2)	C5—C6—C7—Fe1	−58.76 (16)
Cl1—C1—C2—C3	−177.38 (12)	C4—C3—C7—C6	−0.9 (2)
O1—C2—C3—C7	−6.7 (3)	C2—C3—C7—C6	−175.62 (16)
C1—C2—C3—C7	174.94 (16)	Fe1—C3—C7—C6	−59.96 (13)
O1—C2—C3—C4	179.58 (18)	C4—C3—C7—Fe1	59.10 (12)
C1—C2—C3—C4	1.2 (2)	C2—C3—C7—Fe1	−115.66 (16)
O1—C2—C3—Fe1	−92.74 (19)	C12—C8—C9—C10	0.4 (2)
C1—C2—C3—Fe1	88.90 (16)	Fe1—C8—C9—C10	−59.01 (13)
C7—C3—C4—C5	1.19 (19)	C12—C8—C9—Fe1	59.38 (13)
C2—C3—C4—C5	175.70 (17)	C8—C9—C10—C11	−0.3 (2)
Fe1—C3—C4—C5	60.52 (13)	Fe1—C9—C10—C11	−59.50 (14)
C7—C3—C4—Fe1	−59.33 (12)	C8—C9—C10—Fe1	59.20 (12)
C2—C3—C4—Fe1	115.18 (17)	C9—C10—C11—C12	0.1 (2)
C3—C4—C5—C6	−1.1 (2)	Fe1—C10—C11—C12	−59.42 (14)
Fe1—C4—C5—C6	58.42 (16)	C9—C10—C11—Fe1	59.54 (13)
C3—C4—C5—Fe1	−59.49 (12)	C9—C8—C12—C11	−0.3 (2)
C4—C5—C6—C7	0.5 (3)	Fe1—C8—C12—C11	59.01 (13)

Fe1—C5—C6—C7	58.33 (15)	C9—C8—C12—Fe1	−59.31 (12)
C4—C5—C6—Fe1	−57.78 (14)	C10—C11—C12—C8	0.1 (2)
C5—C6—C7—C3	0.2 (2)	Fe1—C11—C12—C8	−59.01 (13)
Fe1—C6—C7—C3	58.95 (12)	C10—C11—C12—Fe1	59.12 (14)

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C8–C12 Cp ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···Cl1^{i}	0.95	2.88	3.4196 (19)	117
C1—H1*B*···O1^{ii}	0.99	2.30	3.256 (2)	163
C9—H9···Cl1^{ii}	0.95	2.95	3.841 (2)	157
C9—H9···O1^{ii}	0.95	2.56	3.262 (2)	131
C1—H1*A*···*Cg*2^{iii}	0.99	2.69	3.4383 (18)	132

Symmetry codes: (i) *x*−1, *y*, *z*−1; (ii) −*x*+1, *y*−1/2, −*z*+1; (iii) *x*+1, *y*, *z*.