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Ferrocenecarboxylic anhydride: a redetermination

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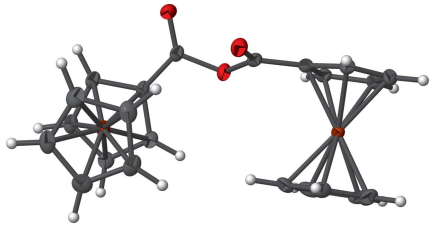
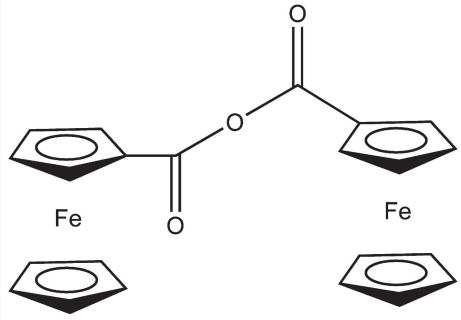
Structural data: full structural data are available from iucrdata.iucr.org

The title molecule, $[Fe_2(C_5H_5)_2(C_{12}H_8O_3)]$, briefly reported previously [Zhang (2015). Private Communication (CCDC reference 1056736). CCDC, Cambridge, England], comprises two ferrocenyl units connected by an acid anhydride bridge. Both ferrocene units have near coplanar [dihedral angles between the ring planes = 2.84 (4) and 1.74 (13)°] and eclipsed [pseudo torsion angles = 6.3 (2) and 5.1 (2)°] cyclopentadienyl (Cp) rings. A twist through the anhydride linkage results in a dihedral angle of 73.81 (8)° between the two substituted Cp rings planes. An intramolecular C—H···O hydrogen bond is also found. In the crystal, C—H···O hydrogen bonds link the molecules into a three-dimensional network.

3D view



Chemical scheme



Structure description

The title compound is a common by-product in the synthesis of ferrocenoyl chloride from ferrocene carboxylic acid (Wang & Huang, 2000). Two ferrocene units [(C101–C105)Fe1(C106–C110)] (Fc1) and [(C201–C205)Fe2(C206–C210)] (Fc2) are bridged by an acid anhydride system, with an intramolecular C206—H206···O1 hydrogen bond influencing the molecular conformation, Fig. 1. The room-temperature structure of this molecule has been deposited with the Cambridge Structural Database, CSD, (Groom *et al.*, 2016) but no details of the molecular or crystal structure were provided (Zhang, 2015). The CSD gives four other examples of Cp carboxylic anhydrides (Elschenbroich *et al.*, 1997; Siebler *et al.*, 2010; Micallef *et al.*, 2011; Liu *et al.*, 2015). The two Cp rings of each ferrocenyl group are almost eclipsed with mean C···*Cg*1*a*···*Cg*1*b*···C and C···*Cg*2*a*···*Cg*2*b*···C torsion angles of 6.3 (2) and 5.1 (2)° [*Cg*1*a*, *Cg*1*b*, *Cg*2*a* and *Cg*2*b* are the centroids of the C101–C105, C106–C110, C201–C205 and C206–C210 Cp rings respectively]. Within each ferrocenyl moiety the rings are close to coplanar, with angles between the Cp ring planes of 2.84 (4)° for Fc1 and 1.74 (13)° for Fc2. Although the two acyl carbonyls lie close to the planes of their respective Cp rings, there is a twist through

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

D—H···A	D—H	H···A	D···A	D—H···A
C206—H206···O1	0.95	2.57	3.413 (2)	149
C205—H205···O2^{i}	0.95	2.38	3.319 (2)	169
C204—H204···O1^{ii}	0.95	2.55	3.463 (2)	160
C207—H207···O2^{iii}	0.95	2.64	3.542 (2)	159

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

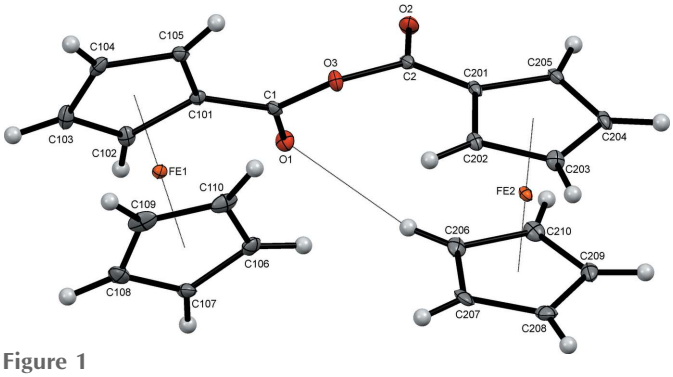


Figure 1
The structure of the title compound with ellipsoids drawn at the 50% probability level. The intramolecular C—H···O hydrogen bond is shown as a dashed line.

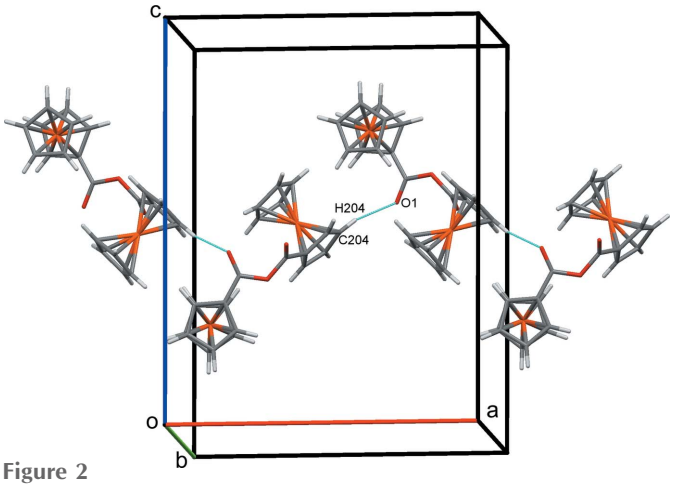


Figure 2
Zigzag chains of molecules of the title compound along the *a* axis.

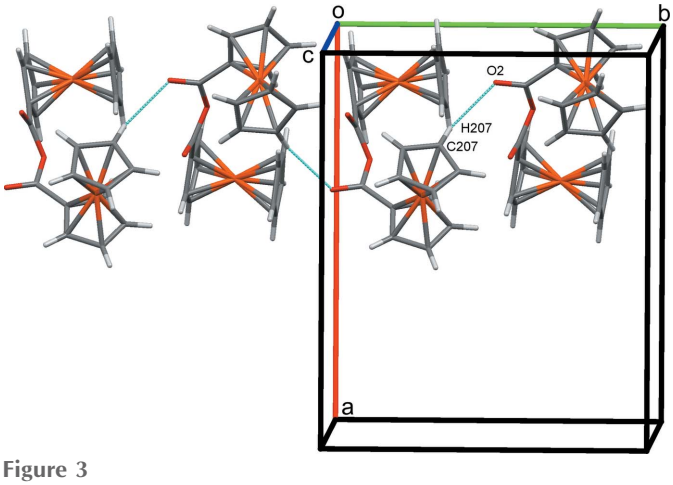


Figure 3
Chains of molecules of the title compound along the *b* axis.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Fe_2(C_5H_5)_2(C_{12}H_8O_3)]$
M_r	442.06
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	100
a, *b*, *c* (Å)	14.8773 (2), 12.2499 (2), 19.3288 (3)
V (Å^3)	3522.59 (9)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	13.38
Crystal size (mm)	0.16 × 0.13 × 0.11
Data collection	
Diffractometer	Agilent SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.267, 0.456
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13584, 3468, 3262
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.621
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.090, 0.84
No. of reflections	3468
No. of parameters	244
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.68

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS2013* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *TITAN2000* (Hunter & Simpson, 1999), *Mercury* (Macrae *et al.*, 2008), *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009), *publCIF* (Westrip, 2010) and *WinGX* (Farrugia, 2012).

the connecting O3 atom of the anhydride unit that results in a torsion angle of approximately 57.36° for O1—C1···C2—O2. Similar torsional geometry is observed in the ruthenium analogue IVOYOY (62.31°; Micallef *et al.*, 2011), the 1′,2′-bis-substituted ferrocene system KAJBUU (57.04°; Siebler *et al.*, 2010) and the vanadium compound NEWFIE (66.68°; Elschenbroich *et al.*, 1997). In the bis-ferrocenophanone structure HOVYEY (Liu *et al.*, 2015) where two ferrocene groups are constrained by two anhydride linkages, the two carbonyl groups are still inclined at an O—C···C—O torsion angle of 27.47° despite the fact that the bridged Cp rings are almost coplanar, suggesting the O—C···C—O torsion is a mechanism to relieve steric strain in the molecule.

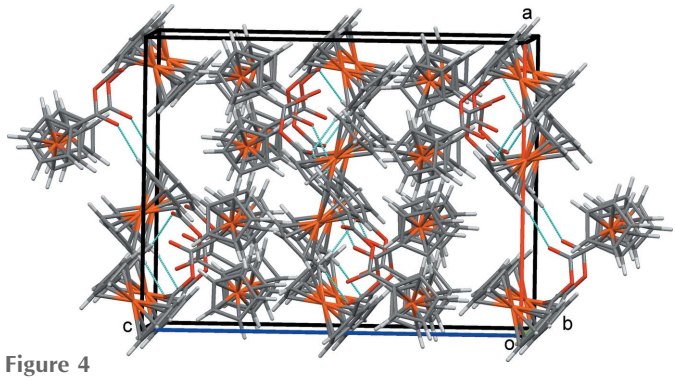


Figure 4
Overall packing of the title compound viewed along the *b*-axis direction.

In the crystal, C204—H204···O1 hydrogen bonds (Table 1) generate zigzag chains along the *a* axis, Fig. 2. The carbonyl oxygen atom O2 acts as a dual acceptor with C207—H207···O2 hydrogen bonds forming chains along the *b* axis Fig. 3, and C205—H205···O2-generated inversion dimers linking the two chain motifs in the third dimension with molecules stacked along the *b* axis in such a way that one ferrocene unit of each individual molecule lies approximately parallel to the *b*-axis direction while the other lies in the *ac* plane, Fig. 4.

Synthesis and crystallization

The title compound was obtained as a by-product in the synthesis of ferrocenoyl chloride from the reaction of ferrocene carboxylic acid with triphosgene and 4-dimethylaminopyridine (Wang & Huang, 2000). Orange block-shaped crystals were obtained from diffusion of a dichloromethane solution layered with petroleum ether.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161769 [https://doi.org/10.1107/S2414314616017697]

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Ferrocenecarboxylic anhydride

Crystal data

$[Fe_2(C_5H_5)_2(C_{12}H_8O_3)]$
M_r = 442.06
Orthorhombic, *Pbca*
a = 14.8773 (2) Å
b = 12.2499 (2) Å
c = 19.3288 (3) Å
V = 3522.59 (9) Å^3
Z = 8
F(000) = 1808
D_x = 1.667 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 7707 reflections
θ = 4.6–73.5°
μ = 13.38 mm^{-1}
T = 100 K
Block, orange
0.16 × 0.13 × 0.11 mm

Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: SuperNova (Cu) X-ray Source
Detector resolution: 5.1725 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlis PRO; Agilent, 2014)
T_{min} = 0.267, T_{max} = 0.456
13584 measured reflections
3468 independent reflections
3262 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 73.3°, θ_{min} = 4.6°
h = −17→18
k = −14→8
l = −20→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.090
S = 0.84
3468 reflections
244 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.089P)^2 + 0.2107P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.12604 (2)	0.19991 (2)	0.26009 (2)	0.01531 (11)
Fe2	0.39445 (2)	0.28549 (2)	0.51371 (2)	0.01595 (11)
O2	0.38674 (9)	0.00699 (11)	0.43945 (7)	0.0246 (3)
O1	0.19839 (8)	0.05176 (10)	0.42555 (6)	0.0226 (3)
C2	0.37846 (11)	0.09637 (16)	0.41529 (9)	0.0188 (4)
C205	0.50220 (13)	0.19334 (16)	0.48270 (9)	0.0204 (4)
H205	0.5261	0.1314	0.5059	0.024*
O3	0.31555 (8)	0.11814 (10)	0.36295 (6)	0.0206 (3)
C107	0.06717 (12)	0.32095 (15)	0.31823 (10)	0.0229 (4)
H107	0.0316	0.3117	0.3586	0.027*
C102	0.08858 (13)	0.05045 (14)	0.29736 (9)	0.0210 (4)
H102	0.0477	0.0366	0.3341	0.025*
C202	0.41558 (13)	0.30383 (14)	0.41050 (9)	0.0191 (4)
H202	0.3724	0.3276	0.3776	0.023*
C101	0.18384 (12)	0.06756 (14)	0.30387 (9)	0.0179 (3)
C201	0.43179 (12)	0.19276 (15)	0.43170 (9)	0.0184 (4)
C105	0.21968 (13)	0.08585 (14)	0.23576 (8)	0.0202 (4)
H105	0.2808	0.0999	0.2245	0.024*
C210	0.34157 (13)	0.21774 (16)	0.60180 (10)	0.0243 (4)
H210	0.3607	0.1521	0.6234	0.029*
C106	0.16221 (13)	0.33524 (15)	0.31630 (10)	0.0236 (4)
H106	0.2013	0.3371	0.3552	0.028*
C204	0.52947 (13)	0.30364 (16)	0.49200 (10)	0.0218 (4)
H204	0.5754	0.3283	0.5224	0.026*
C208	0.32878 (12)	0.39904 (16)	0.57213 (10)	0.0259 (4)
H208	0.3380	0.4757	0.5705	0.031*
C103	0.06679 (14)	0.05807 (15)	0.22591 (10)	0.0250 (4)
H103	0.0084	0.0504	0.2066	0.030*
C1	0.23007 (11)	0.07514 (14)	0.37007 (8)	0.0178 (3)
C209	0.37555 (12)	0.3243 (2)	0.61544 (10)	0.0258 (4)
H209	0.4214	0.3422	0.6477	0.031*
C104	0.14701 (15)	0.07904 (14)	0.18836 (9)	0.0238 (4)
H104	0.1513	0.0872	0.1396	0.029*
C108	0.03498 (15)	0.32293 (18)	0.24893 (11)	0.0292 (4)
H108	−0.0259	0.3148	0.2350	0.035*
C206	0.27372 (13)	0.22702 (17)	0.55000 (10)	0.0245 (4)
H206	0.2398	0.1686	0.5310	0.029*
C109	0.10961 (16)	0.33925 (17)	0.20395 (11)	0.0314 (5)
H109	0.1073	0.3445	0.1549	0.038*
C110	0.18818 (16)	0.34619 (15)	0.24563 (11)	0.0290 (4)
H110	0.2478	0.3564	0.2292	0.035*
C207	0.26578 (12)	0.33924 (17)	0.53162 (9)	0.0245 (4)
H207	0.2257	0.3689	0.4983	0.029*
C203	0.47615 (12)	0.37101 (15)	0.44800 (9)	0.0216 (4)
H203	0.4805	0.4482	0.4444	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01694 (18)	0.01469 (19)	0.01431 (17)	0.00019 (9)	−0.00108 (10)	0.00082 (9)
Fe2	0.01265 (17)	0.02041 (18)	0.01477 (17)	0.00060 (9)	0.00069 (10)	0.00078 (9)
O2	0.0259 (7)	0.0202 (7)	0.0279 (7)	0.0041 (5)	−0.0058 (5)	−0.0001 (5)
O1	0.0233 (6)	0.0270 (6)	0.0175 (6)	−0.0009 (5)	0.0002 (5)	0.0032 (5)
C2	0.0168 (8)	0.0242 (9)	0.0156 (8)	0.0040 (6)	−0.0003 (6)	−0.0007 (7)
C205	0.0141 (8)	0.0273 (9)	0.0196 (9)	0.0040 (7)	0.0001 (7)	0.0006 (7)
O3	0.0179 (6)	0.0256 (6)	0.0183 (6)	−0.0024 (5)	−0.0030 (5)	0.0034 (5)
C107	0.0228 (9)	0.0209 (8)	0.0250 (9)	0.0061 (7)	0.0027 (7)	−0.0039 (7)
C102	0.0228 (8)	0.0173 (8)	0.0228 (8)	−0.0032 (7)	−0.0034 (7)	0.0037 (7)
C202	0.0173 (8)	0.0258 (9)	0.0143 (8)	0.0001 (7)	0.0022 (7)	0.0032 (6)
C101	0.0203 (8)	0.0157 (8)	0.0177 (8)	0.0009 (6)	−0.0011 (6)	0.0000 (6)
C201	0.0143 (8)	0.0267 (9)	0.0142 (8)	0.0021 (7)	0.0004 (7)	−0.0001 (6)
C105	0.0227 (9)	0.0181 (8)	0.0199 (9)	0.0042 (7)	0.0007 (7)	−0.0012 (6)
C210	0.0232 (10)	0.0329 (10)	0.0167 (8)	0.0012 (7)	0.0046 (7)	0.0044 (7)
C106	0.0273 (10)	0.0168 (8)	0.0266 (9)	−0.0019 (7)	−0.0004 (7)	−0.0060 (7)
C204	0.0129 (8)	0.0317 (10)	0.0208 (9)	−0.0015 (7)	0.0009 (7)	−0.0008 (7)
C208	0.0231 (9)	0.0276 (10)	0.0269 (9)	0.0046 (7)	0.0076 (7)	−0.0054 (8)
C103	0.0294 (10)	0.0195 (9)	0.0262 (9)	−0.0053 (7)	−0.0096 (8)	0.0020 (7)
C1	0.0164 (8)	0.0168 (7)	0.0204 (8)	0.0030 (6)	−0.0009 (6)	0.0011 (6)
C209	0.0192 (9)	0.0403 (11)	0.0180 (9)	−0.0006 (8)	0.0020 (7)	−0.0062 (8)
C104	0.0363 (10)	0.0175 (8)	0.0175 (8)	0.0009 (8)	−0.0036 (7)	−0.0035 (7)
C108	0.0314 (11)	0.0258 (9)	0.0304 (9)	0.0134 (9)	−0.0067 (8)	−0.0020 (8)
C206	0.0168 (8)	0.0350 (10)	0.0216 (8)	−0.0047 (8)	0.0050 (7)	−0.0013 (8)
C109	0.0537 (13)	0.0175 (10)	0.0230 (9)	0.0068 (9)	0.0014 (9)	0.0066 (8)
C110	0.0376 (11)	0.0170 (9)	0.0324 (10)	−0.0059 (8)	0.0119 (9)	−0.0026 (8)
C207	0.0136 (8)	0.0368 (10)	0.0232 (8)	0.0068 (8)	0.0032 (7)	0.0005 (8)
C203	0.0196 (8)	0.0251 (9)	0.0200 (8)	−0.0034 (7)	0.0036 (7)	0.0030 (7)

Geometric parameters (Å, º)

Fe1—C101	2.0210 (17)	C102—C101	1.438 (2)
Fe1—C105	2.0283 (18)	C102—H102	0.9500
Fe1—C110	2.0355 (19)	C202—C203	1.419 (3)
Fe1—C109	2.037 (2)	C202—C201	1.441 (2)
Fe1—C108	2.038 (2)	C202—H202	0.9500
Fe1—C102	2.0449 (18)	C101—C105	1.438 (2)
Fe1—C104	2.0523 (18)	C101—C1	1.456 (2)
Fe1—C106	2.0538 (18)	C105—C104	1.420 (3)
Fe1—C107	2.0563 (18)	C105—H105	0.9500
Fe1—C103	2.0573 (19)	C210—C209	1.424 (3)
Fe2—C201	2.0277 (18)	C210—C206	1.426 (3)
Fe2—C202	2.0320 (18)	C210—H210	0.9500
Fe2—C208	2.0407 (18)	C106—C110	1.426 (3)
Fe2—C209	2.0423 (19)	C106—H106	0.9500
Fe2—C203	2.0465 (17)	C204—C203	1.426 (3)

Fe2—C205	2.0501 (19)	C204—H204	0.9500
Fe2—C210	2.0510 (18)	C208—C209	1.422 (3)
Fe2—C207	2.0538 (18)	C208—C207	1.424 (3)
Fe2—C206	2.0570 (19)	C208—H208	0.9500
Fe2—C204	2.064 (2)	C103—C104	1.420 (3)
O2—C2	1.197 (2)	C103—H103	0.9500
O1—C1	1.206 (2)	C209—H209	0.9500
C2—O3	1.404 (2)	C104—H104	0.9500
C2—C201	1.457 (3)	C108—C109	1.424 (3)
C205—C204	1.422 (3)	C108—H108	0.9500
C205—C201	1.438 (2)	C206—C207	1.425 (3)
C205—H205	0.9500	C206—H206	0.9500
O3—C1	1.383 (2)	C109—C110	1.422 (3)
C107—C108	1.423 (3)	C109—H109	0.9500
C107—C106	1.425 (3)	C110—H110	0.9500
C107—H107	0.9500	C207—H207	0.9500
C102—C103	1.422 (3)	C203—H203	0.9500

C101—Fe1—C105	41.60 (7)	C103—C102—H102	126.3
C101—Fe1—C110	124.78 (8)	C101—C102—H102	126.3
C105—Fe1—C110	105.23 (8)	Fe1—C102—H102	126.7
C101—Fe1—C109	161.12 (8)	C203—C202—C201	107.21 (16)
C105—Fe1—C109	122.41 (8)	C203—C202—Fe2	70.18 (10)
C110—Fe1—C109	40.88 (9)	C201—C202—Fe2	69.04 (10)
C101—Fe1—C108	156.99 (8)	C203—C202—H202	126.4
C105—Fe1—C108	160.30 (8)	C201—C202—H202	126.4
C110—Fe1—C108	68.68 (9)	Fe2—C202—H202	126.0
C109—Fe1—C108	40.92 (9)	C105—C101—C102	107.93 (15)
C101—Fe1—C102	41.42 (7)	C105—C101—C1	128.30 (16)
C105—Fe1—C102	69.64 (8)	C102—C101—C1	123.49 (15)
C110—Fe1—C102	163.80 (8)	C105—C101—Fe1	69.47 (10)
C109—Fe1—C102	154.83 (9)	C102—C101—Fe1	70.18 (10)
C108—Fe1—C102	121.21 (9)	C1—C101—Fe1	121.20 (12)
C101—Fe1—C104	68.87 (7)	C205—C201—C202	108.18 (16)
C105—Fe1—C104	40.71 (7)	C205—C201—C2	123.34 (16)
C110—Fe1—C104	118.25 (8)	C202—C201—C2	127.72 (16)
C109—Fe1—C104	105.24 (8)	C205—C201—Fe2	70.18 (10)
C108—Fe1—C104	124.27 (8)	C202—C201—Fe2	69.36 (10)
C102—Fe1—C104	68.50 (8)	C2—C201—Fe2	118.35 (12)
C101—Fe1—C106	108.33 (8)	C104—C105—C101	107.42 (16)
C105—Fe1—C106	119.92 (7)	C104—C105—Fe1	70.55 (10)
C110—Fe1—C106	40.81 (7)	C101—C105—Fe1	68.93 (10)
C109—Fe1—C106	68.71 (9)	C104—C105—H105	126.3
C108—Fe1—C106	68.49 (8)	C101—C105—H105	126.3
C102—Fe1—C106	127.42 (8)	Fe1—C105—H105	125.8
C104—Fe1—C106	154.15 (8)	C209—C210—C206	107.95 (17)
C101—Fe1—C107	122.06 (7)	C209—C210—Fe2	69.31 (11)
C105—Fe1—C107	156.34 (7)	C206—C210—Fe2	69.91 (10)

C110—Fe1—C107	68.51 (8)	C209—C210—H210	126.0
C109—Fe1—C107	68.65 (8)	C206—C210—H210	126.0
C108—Fe1—C107	40.67 (8)	Fe2—C210—H210	126.3
C102—Fe1—C107	109.69 (7)	C107—C106—C110	107.78 (17)
C104—Fe1—C107	162.58 (8)	C107—C106—Fe1	69.80 (10)
C106—Fe1—C107	40.58 (7)	C110—C106—Fe1	68.91 (10)
C101—Fe1—C103	68.85 (7)	C107—C106—H106	126.1
C105—Fe1—C103	68.78 (7)	C110—C106—H106	126.1
C110—Fe1—C103	153.38 (8)	Fe1—C106—H106	126.7
C109—Fe1—C103	119.02 (8)	C205—C204—C203	108.41 (17)
C108—Fe1—C103	107.80 (9)	C205—C204—Fe2	69.25 (11)
C102—Fe1—C103	40.55 (7)	C203—C204—Fe2	69.04 (11)
C104—Fe1—C103	40.44 (8)	C205—C204—H204	125.8
C106—Fe1—C103	164.54 (8)	C203—C204—H204	125.8
C107—Fe1—C103	127.01 (8)	Fe2—C204—H204	127.5
C201—Fe2—C202	41.59 (7)	C209—C208—C207	108.32 (18)
C201—Fe2—C208	161.01 (8)	C209—C208—Fe2	69.67 (11)
C202—Fe2—C208	122.82 (8)	C207—C208—Fe2	70.14 (10)
C201—Fe2—C209	157.03 (8)	C209—C208—H208	125.8
C202—Fe2—C209	160.18 (9)	C207—C208—H208	125.8
C208—Fe2—C209	40.78 (8)	Fe2—C208—H208	125.9
C201—Fe2—C203	68.83 (7)	C104—C103—C102	108.46 (17)
C202—Fe2—C203	40.73 (7)	C104—C103—Fe1	69.59 (10)
C208—Fe2—C203	106.19 (8)	C102—C103—Fe1	69.25 (10)
C209—Fe2—C203	124.07 (8)	C104—C103—H103	125.8
C201—Fe2—C205	41.30 (7)	C102—C103—H103	125.8
C202—Fe2—C205	69.69 (7)	Fe1—C103—H103	127.0
C208—Fe2—C205	155.70 (8)	O1—C1—O3	122.57 (15)
C209—Fe2—C205	121.15 (8)	O1—C1—C101	125.59 (16)
C203—Fe2—C205	68.66 (8)	O3—C1—C101	111.78 (14)
C201—Fe2—C210	121.83 (8)	C208—C209—C210	107.90 (17)
C202—Fe2—C210	156.79 (8)	C208—C209—Fe2	69.55 (11)
C208—Fe2—C210	68.46 (8)	C210—C209—Fe2	69.97 (11)
C209—Fe2—C210	40.72 (8)	C208—C209—H209	126.0
C203—Fe2—C210	161.84 (8)	C210—C209—H209	126.0
C205—Fe2—C210	108.65 (8)	Fe2—C209—H209	126.0
C201—Fe2—C207	124.52 (8)	C105—C104—C103	108.71 (16)
C202—Fe2—C207	105.92 (8)	C105—C104—Fe1	68.74 (10)
C208—Fe2—C207	40.71 (8)	C103—C104—Fe1	69.97 (11)
C209—Fe2—C207	68.58 (7)	C105—C104—H104	125.6
C203—Fe2—C207	119.61 (8)	C103—C104—H104	125.6
C205—Fe2—C207	162.67 (8)	Fe1—C104—H104	127.2
C210—Fe2—C207	68.42 (8)	C107—C108—C109	108.34 (18)
C201—Fe2—C206	108.10 (8)	C107—C108—Fe1	70.36 (11)
C202—Fe2—C206	120.55 (8)	C109—C108—Fe1	69.52 (11)
C208—Fe2—C206	68.32 (8)	C107—C108—H108	125.8
C209—Fe2—C206	68.44 (8)	C109—C108—H108	125.8
C203—Fe2—C206	155.30 (8)	Fe1—C108—H108	125.9

C205—Fe2—C206	126.21 (8)	C207—C206—C210	108.09 (17)
C210—Fe2—C206	40.63 (8)	C207—C206—Fe2	69.60 (10)
C207—Fe2—C206	40.56 (8)	C210—C206—Fe2	69.46 (11)
C201—Fe2—C204	68.58 (7)	C207—C206—H206	126.0
C202—Fe2—C204	68.78 (7)	C210—C206—H206	126.0
C208—Fe2—C204	120.33 (8)	Fe2—C206—H206	126.6
C209—Fe2—C204	107.74 (7)	C110—C109—C108	107.65 (18)
C203—Fe2—C204	40.60 (7)	C110—C109—Fe1	69.49 (11)
C205—Fe2—C204	40.44 (7)	C108—C109—Fe1	69.56 (11)
C210—Fe2—C204	125.85 (8)	C110—C109—H109	126.2
C207—Fe2—C204	155.07 (8)	C108—C109—H109	126.2
C206—Fe2—C204	163.06 (8)	Fe1—C109—H109	126.3
O2—C2—O3	121.58 (17)	C109—C110—C106	108.33 (18)
O2—C2—C201	126.89 (16)	C109—C110—Fe1	69.63 (12)
O3—C2—C201	111.46 (15)	C106—C110—Fe1	70.28 (11)
C204—C205—C201	107.41 (16)	C109—C110—H110	125.8
C204—C205—Fe2	70.30 (11)	C106—C110—H110	125.8
C201—C205—Fe2	68.51 (10)	Fe1—C110—H110	125.8
C204—C205—H205	126.3	C208—C207—C206	107.73 (16)
C201—C205—H205	126.3	C208—C207—Fe2	69.15 (10)
Fe2—C205—H205	126.4	C206—C207—Fe2	69.84 (10)
C1—O3—C2	117.96 (13)	C208—C207—H207	126.1
C108—C107—C106	107.89 (17)	C206—C207—H207	126.1
C108—C107—Fe1	68.97 (11)	Fe2—C207—H207	126.4
C106—C107—Fe1	69.62 (10)	C202—C203—C204	108.79 (17)
C108—C107—H107	126.1	C202—C203—Fe2	69.09 (10)
C106—C107—H107	126.1	C204—C203—Fe2	70.36 (10)
Fe1—C107—H107	126.9	C202—C203—H203	125.6
C103—C102—C101	107.47 (16)	C204—C203—H203	125.6
C103—C102—Fe1	70.20 (10)	Fe2—C203—H203	126.5
C101—C102—Fe1	68.40 (9)		

O2—C2—O3—C1	44.3 (2)	C105—C101—C1—O3	8.2 (2)
C201—C2—O3—C1	−138.50 (15)	C102—C101—C1—O3	−164.96 (15)
C103—C102—C101—C105	0.1 (2)	Fe1—C101—C1—O3	−79.25 (17)
Fe1—C102—C101—C105	−59.44 (12)	C207—C208—C209—C210	0.0 (2)
C103—C102—C101—C1	174.50 (16)	Fe2—C208—C209—C210	−59.69 (13)
Fe1—C102—C101—C1	114.94 (16)	C207—C208—C209—Fe2	59.71 (12)
C103—C102—C101—Fe1	59.56 (12)	C206—C210—C209—C208	0.0 (2)
C204—C205—C201—C202	−0.6 (2)	Fe2—C210—C209—C208	59.43 (13)
Fe2—C205—C201—C202	59.16 (12)	C206—C210—C209—Fe2	−59.45 (13)
C204—C205—C201—C2	−171.34 (17)	C101—C105—C104—C103	0.62 (19)
Fe2—C205—C201—C2	−111.54 (17)	Fe1—C105—C104—C103	−58.68 (13)
C204—C205—C201—Fe2	−59.80 (13)	C101—C105—C104—Fe1	59.31 (11)
C203—C202—C201—C205	0.4 (2)	C102—C103—C104—C105	−0.6 (2)
Fe2—C202—C201—C205	−59.67 (12)	Fe1—C103—C104—C105	57.93 (12)
C203—C202—C201—C2	170.58 (17)	C102—C103—C104—Fe1	−58.49 (13)
Fe2—C202—C201—C2	110.50 (18)	C106—C107—C108—C109	−0.4 (2)

C203—C202—C201—Fe2	60.09 (12)	Fe1—C107—C108—C109	−59.36 (14)
O2—C2—C201—C205	−2.1 (3)	C106—C107—C108—Fe1	58.92 (12)
O3—C2—C201—C205	−179.04 (15)	C209—C210—C206—C207	0.0 (2)
O2—C2—C201—C202	−170.88 (18)	Fe2—C210—C206—C207	−59.07 (12)
O3—C2—C201—C202	12.2 (2)	C209—C210—C206—Fe2	59.07 (13)
O2—C2—C201—Fe2	−86.0 (2)	C107—C108—C109—C110	0.6 (2)
O3—C2—C201—Fe2	97.05 (15)	Fe1—C108—C109—C110	−59.28 (14)
C102—C101—C105—C104	−0.45 (19)	C107—C108—C109—Fe1	59.88 (14)
C1—C101—C105—C104	−174.49 (17)	C108—C109—C110—C106	−0.5 (2)
Fe1—C101—C105—C104	−60.34 (12)	Fe1—C109—C110—C106	−59.86 (14)
C102—C101—C105—Fe1	59.89 (12)	C108—C109—C110—Fe1	59.32 (14)
C1—C101—C105—Fe1	−114.15 (18)	C107—C106—C110—C109	0.3 (2)
C108—C107—C106—C110	0.1 (2)	Fe1—C106—C110—C109	59.46 (14)
Fe1—C107—C106—C110	58.62 (13)	C107—C106—C110—Fe1	−59.18 (13)
C108—C107—C106—Fe1	−58.52 (13)	C209—C208—C207—C206	0.0 (2)
C201—C205—C204—C203	0.6 (2)	Fe2—C208—C207—C206	59.41 (12)
Fe2—C205—C204—C203	−58.05 (13)	C209—C208—C207—Fe2	−59.42 (12)
C201—C205—C204—Fe2	58.67 (13)	C210—C206—C207—C208	0.0 (2)
C101—C102—C103—C104	0.3 (2)	Fe2—C206—C207—C208	−58.97 (12)
Fe1—C102—C103—C104	58.69 (13)	C210—C206—C207—Fe2	58.98 (13)
C101—C102—C103—Fe1	−58.43 (12)	C201—C202—C203—C204	0.0 (2)
C2—O3—C1—O1	24.4 (2)	Fe2—C202—C203—C204	59.33 (13)
C2—O3—C1—C101	−158.36 (15)	C201—C202—C203—Fe2	−59.36 (12)
C105—C101—C1—O1	−174.66 (17)	C205—C204—C203—C202	−0.4 (2)
C102—C101—C1—O1	12.1 (3)	Fe2—C204—C203—C202	−58.55 (13)
Fe1—C101—C1—O1	97.86 (19)	C205—C204—C203—Fe2	58.18 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C206—H206···O1	0.95	2.57	3.413 (2)	149
C205—H205···O2^{i}	0.95	2.38	3.319 (2)	169
C204—H204···O1^{ii}	0.95	2.55	3.463 (2)	160
C207—H207···O2^{iii}	0.95	2.64	3.542 (2)	159

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