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Crystal structure of (ferrocenylmethyl)dimethylammonium hydrogen oxalate

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The crystal structure of the title salt, $[Fe(C_5H_5)(C_8H_{13}N)](HC_2O_4)$, consists of discrete (ferrocenylmethyl)dimethylammonium cations and hydrogen oxalate anions. The anions are connected through a strong O—H···O hydrogen bond, forming linear chains running parallel to [100]. The cations are linked to the anions through bifurcated N—H···(O,O′) hydrogen bonds. Weak C—H···π interactions between neighbouring ferrocenyl moieties are also observed.

Keywords: crystal structure; hydrogen oxalate; (ferrocenylmethyl)dimethylammonium; bifurcated hydrogen bond; C—H···π interactions

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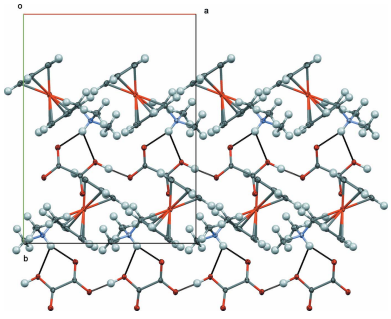
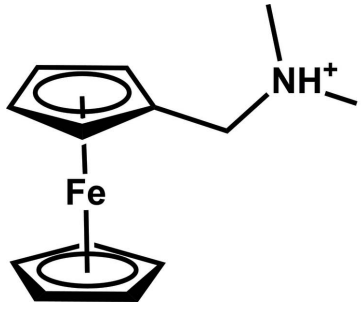
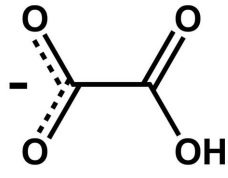
1. Chemical context

Our group has been working on the interactions between alkylammonium ions with oxalic acid, and we have recently reported the crystal structure of $(H_3C)_2NH^+\cdot HC_2O_4^-\cdot 0.5H_2C_2O_4$ (Diallo *et al.*, 2015). Numerous other reports have described crystal structures containing acidic or neutral oxalate molecules interacting with a protonated amine, see for example: Vaidhyanathan *et al.* (2002); Braga *et al.* (2013); Said *et al.* (2006); Hathwar *et al.* (2010); Matulková *et al.* (2008); Olenik *et al.* (2003); Anda *et al.* (2004). Braga *et al.* have reported several structures of columnar metallocenium sandwich compounds interacting with hydrogen oxalate (Braga *et al.*, 2002). However, none of these structures features the hydrogen oxalate anion alone. It is crystallized either with neutral oxalic acid and/or a water molecule. The crystal structure of the title salt, $[Fe(C_5H_5)(C_8H_{13}N)]^+\cdot[HC_2O_4]^-$, (I), features only the hydrogen oxalate anion. This compound was obtained when studying the interaction of (ferrocenylmethyl)dimethylamine and oxalic acid in aqueous solution.







2. Structural commentary

The asymmetric unit of (I) contains one hydrogen oxalate anion and one (ferrocenylmethyl)dimethylammonium cation (Fig. 1). As previously observed in structures featuring this

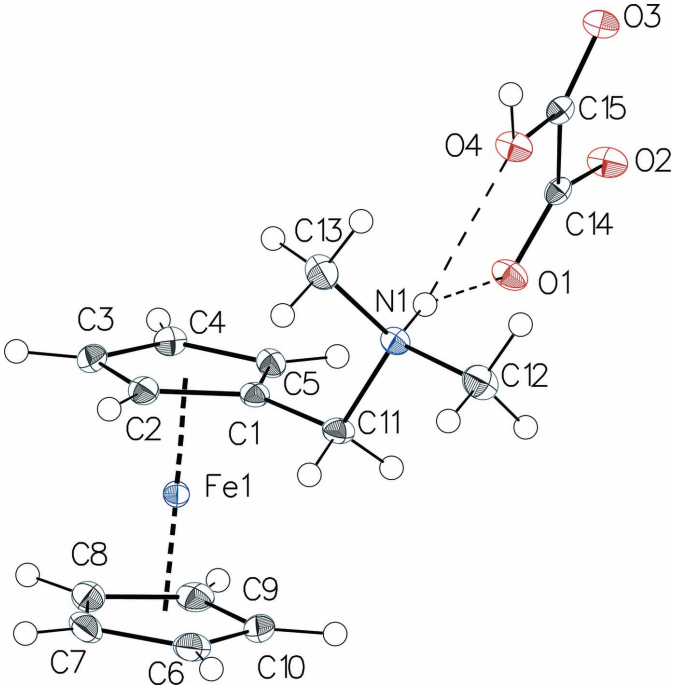


Figure 1
The molecular components in the structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radii. Fe–Cp interactions and hydrogen bonds are shown as dashed lines.

cation (Wang, 2010; Guo, 2006; Guo *et al.*, 2006*a*,*b*), the two Cp rings exhibit a nearly eclipsed conformation. They are planar and almost parallel, as demonstrated by the dihedral angle of 0.96 (5)° between their least-square planes. The Fe—C distances range from 2.0394 (10) to 2.0578 (12) Å. The Fe binding with the Cp rings is somewhat asymmetric as suggested by both the Fe···Cp plane distances [1.6601 (6) and 1.6514 (6) Å for the unsubstituted and the substituted ligand, respectively], and the Cp1–Fe–Cp2 dihedral angle of 170.96 (3)°. This behaviour was previously described as a consequence of an electron-withdrawal effect of the methyldimethylamine group that results in the less electron-rich substituted ring being slightly closer to the metal (Winter & Wolmershäuser, 1998). The oxalate anion is essentially planar and the dihedral angle between carboxylate and the carboxyl groups is only 4.6 (3)°. The C—OH bond is at 1.3052 (13) significantly longer than the other three C—O bonds with an mean of 1.24 (2) Å.

3. Supramolecular features

The hydrogen oxalate anions are held together *via* a strong intermolecular O4—H4*A*···O2 hydrogen bond, resulting in the formation of linear chains running parallel to [100] (Fig. 2). Within a chain, successive hydrogen oxalate anions are rotated by 30.89 (11)°. The cation is linked to the anionic chain through a bifurcated N1—H1···(O1,O4) hydrogen bond (Table 1). In addition to Coulomb forces and hydrogen bonds, a weak C—H···π interaction involving the centroid *Cg*2 of the Cp ligand (C6–C10; Table 1) is present and consolidates the three-dimensional supramolecular network.

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

*Cg*2 is the centroid of the Cp ligand C6–C10.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.878 (15)	1.981 (15)	2.8180 (11)	158.9 (14)
N1—H1···O4	0.878 (15)	2.346 (15)	2.8958 (11)	120.8 (11)
O4—H4*A*···O2^{i}	0.96 (2)	1.52 (2)	2.4776 (11)	174.1 (18)
C2—H2···*Cg*2^{ii}	0.935 (19)	2.743 (19)	3.6564 (13)	165.8 (13)

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

4. Database survey

A search in the Cambridge Structural database (Version 5.36 with three updates, Groom & Allen, 2014) returned only eight entries for seven independent crystal structures containing the (ferrocenylmethyl)dimethylammonium cation. These include simple salts with Cl^- (Winter & Wolmershäuser, 1998) and its hydrated form (Guo *et al.*, 2006*a*), Br^- (Wang, 2010), NO_3^- (Guo *et al.*, 2006*b*), sulfate pentahydrate (Guo, 2006), tetrachloridozincate monohydrate (Gibbons & Trotter, 1971) and a benzene solvate with dodecaborane (Yongmao *et al.*, 1983). The investigation of hydrogen-bonded hydrogen oxalate chains returned 119 unique structures of which 32 are characterized by a bifurcated hydrogen bond with an ammonium counter-cation.

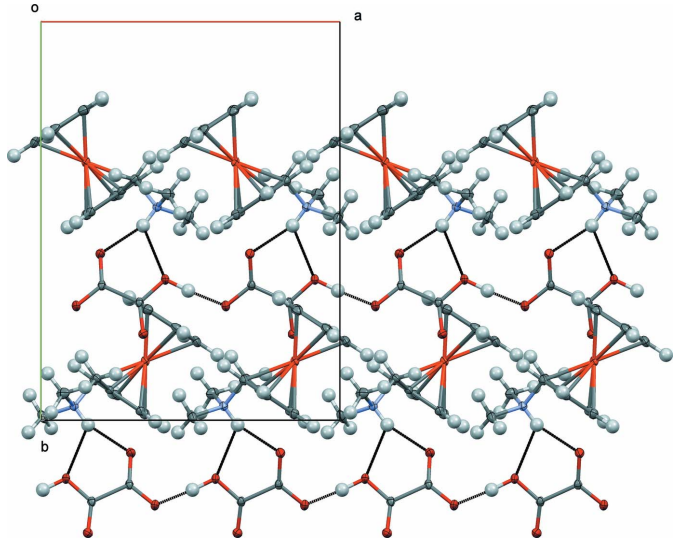


Figure 2
Partial packing diagram in the structure of the title compound viewed along [001]. The chains running along [100] as defined by the hydrogen-bonded hydrogen oxalate anions and the (ferrocenylmethyl)dimethylammonium cations linked by a bifurcated hydrogen bond are shown. Hydrogen bonds are shown as black lines.

5. Synthesis and crystallization

Crystals of the title compound were obtained by slow evaporation of an aqueous solution in which (ferrocenylmethyl)dimethylamine was mixed with oxalic acid in a 1:2 ratio.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were located in difference Fourier maps and were freely refined.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Fe(C_5H_5)(C_8H_{13}N)](C_2HO_4)$
M_r	333.16
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	100
a, *b*, *c* (Å)	11.2225 (3), 14.8991 (4), 17.2727 (5)
V (Å^3)	2888.08 (14)
Z	8
Radiation type	Ga $K\alpha$, λ = 1.34139 Å
μ (mm^{-1})	5.72
Crystal size (mm)	0.15 × 0.13 × 0.09
Data collection	
Diffractometer	Bruker Venture Metaljet
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.585, 0.752
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	56674, 3323, 3150
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.022, 0.062, 1.03
No. of reflections	3323
No. of parameters	266
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.19

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

Acknowledgements

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References

Anda, C., Llobet, A., Martell, A. E., Reibenspies, J., Berni, E. & Solans, X. (2004). *Inorg. Chem.* **43**, 2793–2802.
Braga, D., Chelazzi, L., Ciabatti, L. & Grepioni, F. (2013). *New J. Chem.* **37**, 97–104.
Braga, D., Eckert, M., Fraccastoro, M., Maini, L., Grepioni, F., Caneschi, A. & Sessoli, R. (2002). *New J. Chem.* **26**, 1280–1286.
Bruker (2014). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Diallo, W., Gueye, N., Crochet, A., Plasseraud, L. & Cattey, H. (2015). *Acta Cryst.* E**71**, 473–475.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Gibbons, C. S. & Trotter, J. (1971). *J. Chem. Soc. A*, pp. 2659–2662.
Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
Guo, H.-X. (2006). *Acta Cryst.* C**62**, m504–m506.
Guo, H.-X., Yang, L.-M. & Zhang, S.-D. (2006*a*). *Acta Cryst.* E**62**, m1338–m1339.
Guo, H.-X., Zhou, X.-J., Lin, Z.-X. & Liu, J.-M. (2006*b*). *Acta Cryst.* E**62**, m1770–m1772.
Hathwar, V. R., Pal, R. & Guru Row, T. N. (2010). *Cryst. Growth Des.* **10**, 3306–3310.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Matulková, I., Němec, I., Teubner, K., Němec, P. & Mička, Z. (2008). *J. Mol. Struct.* **873**, 46–60.
Olenik, B., Smolka, T., Boese, R. & Sustmann, R. (2003). *Cryst. Growth Des.* **3**, 183–188.
Said, F. F., Ong, T.-G., Bazinet, P., Yap, G. P. A. & Richeson, D. S. (2006). *Cryst. Growth Des.* **6**, 1848–1857.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Vaidhyanathan, R., Natarajan, S. & Rao, C. N. R. (2002). *J. Mol. Struct.* **608**, 123–133.
Wang, B. (2010). *Acta Cryst.* E**66**, m686.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Winter, R. F. & Wolmershäuser, G. (1998). *J. Organomet. Chem.* **570**, 201–218.
Yongmao, Z., Zhaoping, C., Zhiwei, C., Kezhen, P., Guomin, Z., Zhaogui, Z. & Huaxue, Z. (1983). *Chin. J. Struct. Chem.* **2**, 201–204.

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Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

(Ferrocenylmethyl)dimethylammonium hydrogen oxalate

Crystal data

$[Fe(C_5H_5)(C_8H_{13}N)](C_2HO_4)$
$M_r = 333.16$
Orthorhombic, *Pbca*
$a = 11.2225$ (3) Å
$b = 14.8991$ (4) Å
$c = 17.2727$ (5) Å
$V = 2888.08$ (14) Å^3
$Z = 8$
$F(000) = 1392$
$D_x = 1.532$ Mg m^{-3}
Ga $K\alpha$ radiation, $\lambda = 1.34139$ Å
Cell parameters from 9948 reflections
$\theta = 4.8$–$60.7°$
$\mu = 5.72$ mm^{-1}
$T = 100$ K
Block, clear light orange
0.15 × 0.13 × 0.09 mm

Data collection

Bruker Venture Metaljet diffractometer
Radiation source: Metal Jet, Gallium Liquid Metal Jet Source
Helios MX Mirror Optics monochromator
Detector resolution: 10.24 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
$T_{min} = 0.585$, $T_{max} = 0.752$
56674 measured reflections
3323 independent reflections
3150 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 60.7°$, $\theta_{min} = 4.5°$
$h = -14 \rightarrow 14$
$k = -17 \rightarrow 19$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.062$
$S = 1.03$
3323 reflections
266 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 0.8916P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Special details

Experimental. X-ray crystallographic data for I were collected from a single-crystal sample, which was mounted on a loop fiber. Data were collected using a Bruker Venture diffractometer equipped with a Photon 100 CMOS Detector, a Helios MX optics and a Kappa goniometer. The crystal-to-detector distance was 4.0 cm, and the data collection was carried out in 1024 *x* 1024 pixel mode.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.34931 (2)	0.64806 (2)	0.49469 (2)	0.01187 (7)
N1	0.11135 (8)	0.53481 (6)	0.31389 (5)	0.01286 (17)
C1	0.22887 (9)	0.57630 (6)	0.43156 (6)	0.01299 (19)
C2	0.17883 (11)	0.60032 (8)	0.50499 (6)	0.0150 (2)
C3	0.25259 (10)	0.56311 (7)	0.56398 (6)	0.0164 (2)
C4	0.34734 (9)	0.51541 (7)	0.52776 (7)	0.0161 (2)
C5	0.33275 (9)	0.52304 (7)	0.44578 (6)	0.0143 (2)
C6	0.35605 (10)	0.77834 (8)	0.45550 (7)	0.0199 (2)
C7	0.35680 (10)	0.77649 (8)	0.53827 (7)	0.0199 (2)
C8	0.45806 (10)	0.72650 (7)	0.56226 (6)	0.0193 (2)
C9	0.52010 (11)	0.69741 (8)	0.49491 (6)	0.0193 (2)
C10	0.45688 (11)	0.72947 (7)	0.42883 (6)	0.0200 (2)
C11	0.18587 (9)	0.60523 (7)	0.35384 (6)	0.0148 (2)
C12	0.07459 (11)	0.56695 (8)	0.23563 (7)	0.0209 (2)
C13	0.00554 (10)	0.50895 (8)	0.36082 (7)	0.0218 (2)
O1	0.30103 (6)	0.41777 (5)	0.27870 (5)	0.01774 (16)
O2	0.38177 (7)	0.28665 (5)	0.24336 (5)	0.01896 (17)
O3	0.15744 (6)	0.21661 (5)	0.23000 (5)	0.01636 (16)
O4	0.08414 (7)	0.35313 (5)	0.25702 (5)	0.01652 (16)
C14	0.29562 (9)	0.33826 (7)	0.25759 (6)	0.01189 (19)
C15	0.16996 (9)	0.29528 (7)	0.24647 (6)	0.01225 (19)
H11A	0.1366 (12)	0.6592 (9)	0.3575 (8)	0.015 (3)*
H5	0.3823 (13)	0.4963 (9)	0.4076 (8)	0.019 (3)*
H9	0.5900 (18)	0.6637 (12)	0.4949 (8)	0.029 (4)*
H4	0.4109 (12)	0.4842 (9)	0.5532 (8)	0.017 (3)*
H6	0.2957 (14)	0.8082 (10)	0.4249 (9)	0.025 (4)*
H1	0.1581 (12)	0.4881 (10)	0.3079 (9)	0.024 (4)*
H2	0.1122 (18)	0.6365 (12)	0.5139 (9)	0.030 (4)*
H11B	0.2507 (12)	0.6154 (9)	0.3202 (8)	0.017 (3)*
H8	0.4786 (14)	0.7118 (10)	0.6155 (9)	0.029 (4)*
H12A	0.0229 (15)	0.6170 (11)	0.2419 (9)	0.032 (4)*
H3	0.2436 (14)	0.5700 (9)	0.6199 (9)	0.027 (4)*
H13A	−0.0415 (15)	0.4691 (11)	0.3319 (9)	0.034 (4)*
H10	0.4764 (15)	0.7189 (10)	0.3752 (9)	0.032 (4)*
H13B	0.0345 (15)	0.4801 (11)	0.4091 (10)	0.033 (4)*

H7	0.2965 (14)	0.8042 (10)	0.5702 (9)	0.028 (4)*
H13C	−0.0372 (14)	0.5622 (10)	0.3732 (9)	0.029 (4)*
H12B	0.1486 (14)	0.5830 (11)	0.2080 (10)	0.031 (4)*
H12C	0.0350 (13)	0.5191 (10)	0.2088 (8)	0.021 (3)*
H4A	0.007 (2)	0.3250 (12)	0.2542 (11)	0.049 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01085 (10)	0.01291 (10)	0.01184 (10)	−0.00209 (5)	0.00031 (5)	−0.00109 (5)
N1	0.0108 (4)	0.0125 (4)	0.0152 (4)	−0.0001 (3)	−0.0009 (3)	−0.0013 (3)
C1	0.0113 (4)	0.0122 (4)	0.0155 (5)	−0.0030 (4)	0.0000 (4)	−0.0007 (4)
C2	0.0121 (5)	0.0148 (5)	0.0182 (5)	−0.0015 (4)	0.0028 (4)	−0.0012 (4)
C3	0.0174 (5)	0.0168 (5)	0.0150 (5)	−0.0041 (4)	0.0022 (4)	0.0004 (4)
C4	0.0156 (5)	0.0150 (5)	0.0175 (5)	−0.0010 (4)	−0.0016 (4)	0.0016 (4)
C5	0.0122 (4)	0.0145 (5)	0.0160 (5)	−0.0008 (4)	0.0006 (4)	−0.0025 (4)
C6	0.0229 (6)	0.0147 (5)	0.0220 (6)	−0.0039 (4)	−0.0035 (4)	0.0015 (4)
C7	0.0211 (5)	0.0164 (5)	0.0222 (6)	−0.0040 (4)	0.0007 (4)	−0.0064 (4)
C8	0.0200 (5)	0.0206 (5)	0.0173 (5)	−0.0078 (4)	−0.0029 (4)	−0.0012 (4)
C9	0.0131 (5)	0.0184 (5)	0.0264 (6)	−0.0052 (4)	0.0008 (4)	−0.0013 (4)
C10	0.0238 (6)	0.0191 (5)	0.0170 (5)	−0.0093 (4)	0.0039 (4)	−0.0012 (4)
C11	0.0151 (5)	0.0127 (5)	0.0167 (5)	−0.0034 (4)	−0.0027 (4)	0.0002 (4)
C12	0.0258 (6)	0.0178 (5)	0.0189 (5)	0.0016 (4)	−0.0083 (5)	0.0000 (4)
C13	0.0142 (5)	0.0258 (6)	0.0254 (6)	−0.0067 (5)	0.0060 (4)	−0.0091 (5)
O1	0.0121 (3)	0.0154 (3)	0.0257 (4)	−0.0010 (3)	0.0006 (3)	−0.0054 (3)
O2	0.0099 (3)	0.0153 (4)	0.0317 (4)	0.0007 (3)	0.0018 (3)	−0.0011 (3)
O3	0.0143 (4)	0.0129 (3)	0.0219 (4)	−0.0006 (3)	−0.0025 (3)	−0.0002 (3)
O4	0.0088 (3)	0.0149 (4)	0.0258 (4)	0.0005 (3)	−0.0001 (3)	−0.0020 (3)
C14	0.0099 (4)	0.0146 (4)	0.0112 (4)	−0.0006 (4)	−0.0001 (3)	0.0024 (4)
C15	0.0106 (4)	0.0142 (4)	0.0120 (4)	−0.0001 (4)	−0.0008 (3)	0.0022 (4)

Geometric parameters (Å, º)

Fe1—C1	2.0394 (10)	C6—C7	1.4298 (19)
Fe1—C2	2.0490 (12)	C6—C10	1.4223 (17)
Fe1—C3	2.0524 (10)	C6—H6	0.967 (16)
Fe1—C4	2.0574 (11)	C7—C8	1.4206 (17)
Fe1—C5	2.0538 (11)	C7—H7	0.965 (16)
Fe1—C6	2.0570 (12)	C8—C9	1.4233 (16)
Fe1—C7	2.0578 (12)	C8—H8	0.974 (16)
Fe1—C8	2.0536 (11)	C9—C10	1.4263 (16)
Fe1—C9	2.0528 (12)	C9—H9	0.93 (2)
Fe1—C10	2.0549 (11)	C10—H10	0.965 (16)
N1—C11	1.5087 (12)	C11—H11A	0.978 (13)
N1—C12	1.4923 (13)	C11—H11B	0.943 (14)
N1—C13	1.4885 (13)	C12—H12A	0.951 (17)
N1—H1	0.878 (15)	C12—H12B	0.987 (17)
C1—C2	1.4324 (14)	C12—H12C	0.959 (15)

C1—C5	1.4316 (14)	C13—H13A	0.939 (17)
C1—C11	1.4902 (14)	C13—H13B	0.992 (17)
C2—C3	1.4251 (15)	C13—H13C	0.951 (15)
C2—H2	0.935 (19)	O1—C14	1.2410 (12)
C3—C4	1.4237 (15)	O2—C14	1.2595 (13)
C3—H3	0.976 (15)	O3—C15	1.2144 (14)
C4—C5	1.4299 (15)	O4—C15	1.3052 (13)
C4—H4	0.958 (14)	O4—H4A	0.96 (2)
C5—H5	0.950 (15)	C14—C15	1.5607 (14)

C1—Fe1—C2	41.02 (4)	C4—C3—H3	124.4 (9)
C1—Fe1—C3	68.77 (4)	Fe1—C4—H4	125.8 (8)
C1—Fe1—C4	68.76 (4)	C3—C4—Fe1	69.54 (6)
C1—Fe1—C5	40.94 (4)	C3—C4—C5	108.05 (9)
C1—Fe1—C6	110.06 (4)	C3—C4—H4	126.7 (8)
C1—Fe1—C7	135.24 (4)	C5—C4—Fe1	69.51 (6)
C1—Fe1—C8	174.93 (4)	C5—C4—H4	125.3 (8)
C1—Fe1—C9	143.86 (4)	Fe1—C5—H5	127.8 (8)
C1—Fe1—C10	113.75 (4)	C1—C5—Fe1	68.99 (6)
C2—Fe1—C3	40.66 (4)	C1—C5—H5	126.2 (9)
C2—Fe1—C4	68.43 (4)	C4—C5—Fe1	69.78 (6)
C2—Fe1—C5	68.67 (4)	C4—C5—C1	107.89 (9)
C2—Fe1—C6	112.98 (5)	C4—C5—H5	125.9 (9)
C2—Fe1—C7	109.22 (4)	Fe1—C6—H6	126.0 (9)
C2—Fe1—C8	134.68 (4)	C7—C6—Fe1	69.70 (6)
C2—Fe1—C9	174.88 (4)	C7—C6—H6	124.0 (9)
C2—Fe1—C10	143.28 (5)	C10—C6—Fe1	69.68 (6)
C3—Fe1—C4	40.54 (4)	C10—C6—C7	108.02 (10)
C3—Fe1—C5	68.45 (4)	C10—C6—H6	128.0 (9)
C3—Fe1—C6	142.49 (5)	Fe1—C7—H7	125.3 (9)
C3—Fe1—C7	112.44 (5)	C6—C7—Fe1	69.64 (6)
C3—Fe1—C8	109.50 (4)	C6—C7—H7	124.0 (9)
C3—Fe1—C9	135.53 (5)	C8—C7—Fe1	69.63 (6)
C3—Fe1—C10	175.95 (5)	C8—C7—C6	107.85 (10)
C4—Fe1—C7	142.38 (5)	C8—C7—H7	128.2 (9)
C5—Fe1—C4	40.71 (4)	Fe1—C8—H8	123.4 (9)
C5—Fe1—C6	136.37 (5)	C7—C8—Fe1	69.95 (6)
C5—Fe1—C7	175.92 (5)	C7—C8—C9	108.21 (10)
C5—Fe1—C10	111.15 (4)	C7—C8—H8	125.7 (9)
C6—Fe1—C4	176.56 (5)	C9—C8—Fe1	69.69 (6)
C6—Fe1—C7	40.67 (5)	C9—C8—H8	126.0 (9)
C8—Fe1—C4	113.28 (5)	Fe1—C9—H9	126.3 (11)
C8—Fe1—C5	143.48 (5)	C8—C9—Fe1	69.75 (6)
C8—Fe1—C6	68.18 (5)	C8—C9—C10	107.99 (11)
C8—Fe1—C7	40.43 (5)	C8—C9—H9	125.2 (9)
C8—Fe1—C10	68.26 (5)	C10—C9—Fe1	69.76 (6)
C9—Fe1—C4	110.71 (5)	C10—C9—H9	126.8 (9)
C9—Fe1—C5	114.21 (4)	Fe1—C10—H10	124.6 (9)

C9—Fe1—C6	68.18 (5)	C6—C10—Fe1	69.84 (6)
C9—Fe1—C7	68.17 (5)	C6—C10—C9	107.94 (10)
C9—Fe1—C8	40.56 (4)	C6—C10—H10	125.1 (10)
C9—Fe1—C10	40.64 (5)	C9—C10—Fe1	69.60 (6)
C10—Fe1—C4	136.64 (5)	C9—C10—H10	126.9 (10)
C10—Fe1—C6	40.47 (5)	N1—C11—H11A	106.7 (8)
C10—Fe1—C7	68.26 (5)	N1—C11—H11B	104.9 (8)
C11—N1—H1	105.9 (9)	C1—C11—N1	112.98 (8)
C12—N1—C11	110.15 (8)	C1—C11—H11A	111.3 (9)
C12—N1—H1	108.2 (10)	C1—C11—H11B	110.6 (8)
C13—N1—C11	111.91 (8)	H11A—C11—H11B	110.1 (11)
C13—N1—C12	110.84 (9)	N1—C12—H12A	108.5 (9)
C13—N1—H1	109.6 (10)	N1—C12—H12B	106.5 (10)
C2—C1—Fe1	69.85 (6)	N1—C12—H12C	109.1 (8)
C2—C1—C11	126.76 (9)	H12A—C12—H12B	112.3 (14)
C5—C1—Fe1	70.07 (6)	H12A—C12—H12C	110.8 (13)
C5—C1—C2	107.80 (9)	H12B—C12—H12C	109.7 (13)
C5—C1—C11	125.35 (9)	N1—C13—H13A	108.8 (10)
C11—C1—Fe1	123.00 (7)	N1—C13—H13B	107.9 (10)
Fe1—C2—H2	124.2 (11)	N1—C13—H13C	108.0 (9)
C1—C2—Fe1	69.13 (6)	H13A—C13—H13B	111.0 (13)
C1—C2—H2	127.2 (10)	H13A—C13—H13C	111.3 (13)
C3—C2—Fe1	69.80 (6)	H13B—C13—H13C	109.7 (12)
C3—C2—C1	107.95 (10)	C15—O4—H4A	111.6 (11)
C3—C2—H2	124.8 (10)	O1—C14—O2	127.06 (10)
Fe1—C3—H3	124.5 (9)	O1—C14—C15	118.16 (9)
C2—C3—Fe1	69.54 (6)	O2—C14—C15	114.78 (8)
C2—C3—H3	127.3 (9)	O3—C15—O4	125.78 (9)
C4—C3—Fe1	69.92 (6)	O3—C15—C14	121.97 (9)
C4—C3—C2	108.29 (10)	O4—C15—C14	112.25 (8)

Fe1—C1—C2—C3	−59.20 (8)	C5—C1—C11—N1	83.77 (12)
Fe1—C1—C5—C4	59.14 (7)	C6—C7—C8—Fe1	−59.36 (7)
Fe1—C1—C11—N1	171.61 (7)	C6—C7—C8—C9	0.04 (12)
Fe1—C2—C3—C4	−59.38 (7)	C7—C6—C10—Fe1	59.37 (7)
Fe1—C3—C4—C5	−59.03 (7)	C7—C6—C10—C9	−0.03 (12)
Fe1—C4—C5—C1	−58.64 (7)	C7—C8—C9—Fe1	−59.56 (8)
Fe1—C6—C7—C8	59.36 (7)	C7—C8—C9—C10	−0.06 (13)
Fe1—C6—C10—C9	−59.40 (8)	C8—C9—C10—Fe1	−59.50 (8)
Fe1—C7—C8—C9	59.40 (8)	C8—C9—C10—C6	0.05 (13)
Fe1—C8—C9—C10	59.50 (8)	C10—C6—C7—Fe1	−59.36 (7)
Fe1—C9—C10—C6	59.55 (8)	C10—C6—C7—C8	0.00 (11)
C1—C2—C3—Fe1	58.79 (8)	C11—C1—C2—Fe1	−116.75 (10)
C1—C2—C3—C4	−0.59 (13)	C11—C1—C2—C3	−175.95 (9)
C2—C1—C5—Fe1	−59.91 (7)	C11—C1—C5—Fe1	116.95 (10)
C2—C1—C5—C4	−0.78 (11)	C11—C1—C5—C4	176.08 (9)
C2—C1—C11—N1	−99.97 (12)	C12—N1—C11—C1	−178.23 (9)
C2—C3—C4—Fe1	59.15 (8)	C13—N1—C11—C1	57.99 (12)

C2—C3—C4—C5	0.11 (12)	O1—C14—C15—O3	−175.95 (10)
C3—C4—C5—Fe1	59.05 (7)	O1—C14—C15—O4	3.94 (13)
C3—C4—C5—C1	0.41 (11)	O2—C14—C15—O3	4.30 (14)
C5—C1—C2—Fe1	60.05 (7)	O2—C14—C15—O4	−175.81 (9)
C5—C1—C2—C3	0.85 (12)		

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the Cp ligand C6–C10.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.878 (15)	1.981 (15)	2.8180 (11)	158.9 (14)
N1—H1···O4	0.878 (15)	2.346 (15)	2.8958 (11)	120.8 (11)
C11—H11*B*···O3^{i}	0.943 (14)	2.401 (14)	3.2282 (13)	146.3 (11)
C12—H12*A*···O3^{ii}	0.951 (17)	2.556 (17)	3.4792 (14)	163.8 (13)
C13—H13*A*···O4	0.939 (17)	2.578 (16)	3.0631 (14)	112.5 (12)
O4—H4*A*···O2^{iii}	0.96 (2)	1.52 (2)	2.4776 (11)	174.1 (18)
C2—H2···*Cg*2^{iv}	0.935 (19)	2.743 (19)	3.6564 (13)	165.8 (13)

Symmetry codes: (i) −*x*+1/2, *y*+1/2, *z*; (ii) −*x*, *y*+1/2, −*z*+1/2; (iii) *x*−1/2, *y*, −*z*+1/2; (iv) *x*−1/2, −*y*+3/2, −*z*+1.