



Crystal structure of 4-(dimethylamino)pyridinium *cis*-diaquabis(oxalato- κ^2O,O')ferrate(III) hemihydrate

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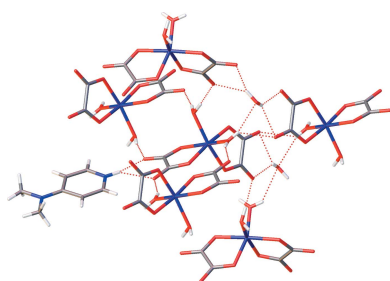
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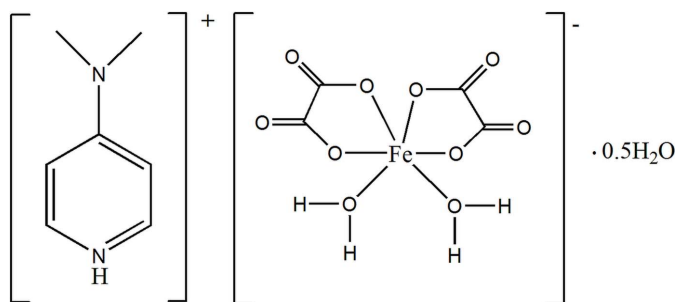
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The Fe^{III} ions in the hybrid title salt, (C₇H₁₁N₂)[Fe(C₂O₄)₂(H₂O)₂] \cdot 0.5H₂O, show a distorted octahedral coordination environment, with four O atoms from two chelating oxalate dianions and two O atoms from two *cis* aqua ligands. The average Fe–O(oxalate) bond length [2.00 (2) Å] is shorter than the average Fe–O(water) bond length [2.027 (19) Å]. The ionic components are connected *via* intermolecular N–H \cdots O and O–H \cdots O hydrogen bonds into a three-dimensional network.

1. Chemical context

Over the past years, the design and synthesis of organic–inorganic hybrid salts have attracted much attention not only because of their fascinating network topologies, but also to obtain a better understanding of the correlations between their structural and physical properties (Bloomquist *et al.*, 1981; Geiser *et al.*, 1987; Pardo *et al.*, 2012). In this context, the bis-oxalato complexes of transition metals, [M^{III}(C₂O₄)₂(H₂O)₂][−], are extremely versatile building blocks for the synthesis of organic–inorganic hybrid salts. Although several salts of general formula A[M^{III}(C₂O₄)₂(H₂O)₂] \cdot xH₂O (A⁺ = aromatic iminium cation, 0 ≤ x ≤ 1) have been explored to date (Bélombé *et al.*, 2009; Nenwa *et al.*, 2010; Chérif *et al.*, 2011; Chérif, Abdelhak *et al.*, 2012; Chérif, Zid *et al.*, 2012; Nenwa *et al.*, 2012*a,b*; Dridi *et al.*, 2013; Bebga *et al.*, 2013), the predictable and consistent formation of networks is still in its infancy. In most cases, the network topologies are influenced by the organic counter-cations, metal coordination spheres, pH values, guest molecules and the crystallization solvent. So far, most of the self-assembly processes involving anionic species, [M^{III}(C₂O₄)₂(H₂O)₂][−], and aromatic iminium cations have led to salts with *trans*-diaquabis(oxalato)metallate(III) complex anions (Bélombé *et al.*, 2009; Nenwa *et al.*, 2010, 2012*a*; Chérif, Zid *et al.*, 2012; Dridi *et al.*, 2013; Gouet *et al.*, 2013). The *cis* configuration of the complex anion [M^{III}(C₂O₄)₂(H₂O)₂][−] is less common in the literature, and has been observed in salts with 2-amino-5-chloropyridinium (Chérif, Abdelhak *et al.*, 2012) or with pyridinium (Nenwa *et al.*, 2012*b*) as aromatic iminium cations. In this work, we extend this family of salts involving the complex anion [M^{III}(C₂O₄)₂(H₂O)₂][−] in its *cis*-configuration by reporting the structural characterization of the title compound with composition (C₇H₁₁N₂)[Fe(C₂O₄)₂(H₂O)₂] \cdot 0.5H₂O.





2. Structural commentary

The asymmetric unit of the title compound shown in Fig. 1 consists of one protonated 4-(dimethylamino)pyridine molecule ($C_7H_{11}N_2^+$), one anionic complex $[Fe(C_2O_4)_2(H_2O)_2]^-$ in a *cis*-aqua configuration and one-half solvent water molecule. Atom O3W of this water molecule of solvation lies on a crystallographic twofold rotation axis. The main geometric parameters of the ($C_7H_{11}N_2^+$) cation are in agreement with those found in a similar salt with the same cationic entity (Nenwa *et al.*, 2010). The iron(III) site in the complex anion has a distorted octahedral coordination environment built up by two O atoms (O1W, O2W) from two *cis*-aqua ligands and four O atoms (O3, O4, O5, O6) from two chelating oxalate dianions. The average $Fe-O_{(oxalate)}$ bond length [2.00 (2) Å] is shorter than the average $Fe-O_{(water)}$ bond length [2.027 (19) Å]. The bond lengths in the $[Fe(C_2O_4)_2(H_2O)_2]^-$ anion are similar to those observed in homologous compounds with a *cis*-aqua configuration of the $[M^{III}(C_2O_4)_2(H_2O)_2]^-$ anionic units (Chérif, Abdelhak *et al.*, 2012; Nenwa *et al.*, 2012*b*).

3. Supramolecular features

Within the crystal packing, the charged components are connected by an extensive hydrogen-bonding network.

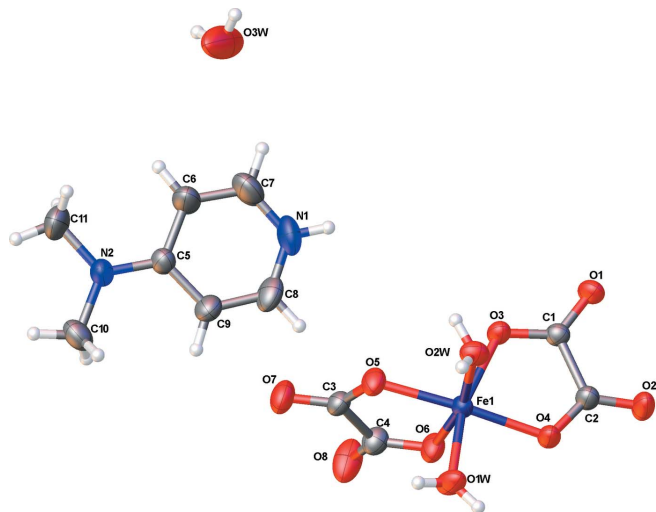


Figure 1
View of the molecular components of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1W—H1WA \cdots O2 ⁱ	0.81 (2)	1.94 (2)	2.720 (3)	162 (4)
O1W—H1WB \cdots O7 ⁱⁱ	0.77 (2)	2.40 (3)	2.988 (3)	134 (4)
O1W—H1WB \cdots O3W ⁱⁱⁱ	0.77 (2)	2.34 (3)	2.9699 (19)	139 (4)
O2W—H2WA \cdots O8 ^{iv}	0.82 (2)	1.84 (2)	2.664 (3)	176 (3)
O2W—H2WB \cdots O1 ^v	0.83 (2)	1.88 (2)	2.702 (2)	171 (3)
N1—H1 \cdots O1 ^v	0.86	2.11	2.931 (3)	160
N1—H1 \cdots O2 ^v	0.86	2.46	3.043 (3)	125
O3W—H3W \cdots O7 ^{vi}	0.84 (2)	2.36 (5)	3.040 (2)	138 (6)
O3W—H3W \cdots O8 ^{vi}	0.84 (2)	2.09 (5)	2.782 (3)	140 (6)

Symmetry codes: (i) $-x + \frac{1}{2}, -y + \frac{5}{2}, -z + \frac{1}{2}$; (ii) $-x + 1, -y + 2, -z + 1$; (iii) $x, y + 1, z$; (iv) $x - \frac{1}{2}, -y + 2, z$; (v) $-x + \frac{1}{2}, -y + \frac{3}{2}, -z + \frac{1}{2}$; (vi) $-x + 1, -y + 1, -z + 1$.

Hydrogen bonds of the type $O-H \cdots O$ involving coordinating water molecules as donor groups and auxiliary O atoms of the oxalate dianions as acceptor groups interconnect neighboring $[Fe(C_2O_4)_2(H_2O)_2]^-$ anionic units (Table 1, Fig. 2). Together with the relatively weaker $N-H \cdots O$ hydrogen bonds of the protonated imine N atoms of the 4-(dimethylamino)pyridine molecules to the oxalate dianions, a three-dimensional framework is formed (Table 1, Fig. 3).

4. Synthesis and crystallization

The salt $Fe(NO_3)_3 \cdot 6H_2O$ (1 mmol, 400 mg) was dissolved in 20 ml of water, leading to a yellowish solution. This solution was added in successive small portions in 30 ml of a mixture of oxalic acid (2 mmol, 253 mg) and 4-(dimethylamino)pyridine (1 mmol, 122 mg) with stirring at 323 K for 2 h. The resulting greenish solution was left at room temperature; crystals suitable for X-ray diffraction were obtained after two weeks upon slow evaporation.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms bonded to C and N atoms

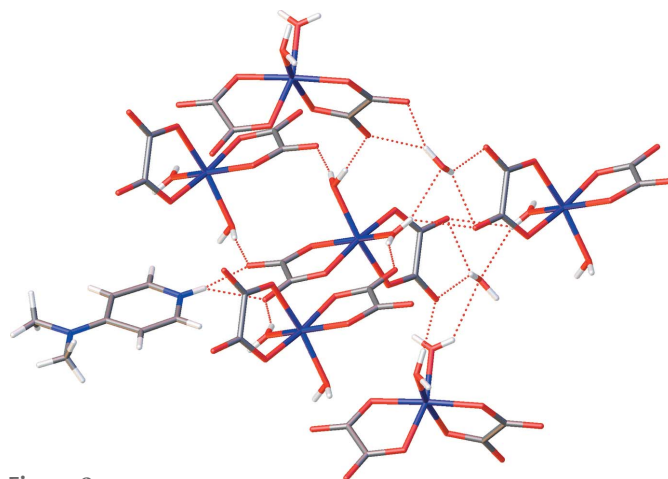


Figure 2
The environment of the $[Fe(C_2O_4)_2(H_2O)_2]^-$ octahedron. Dashed lines denote hydrogen bonds.

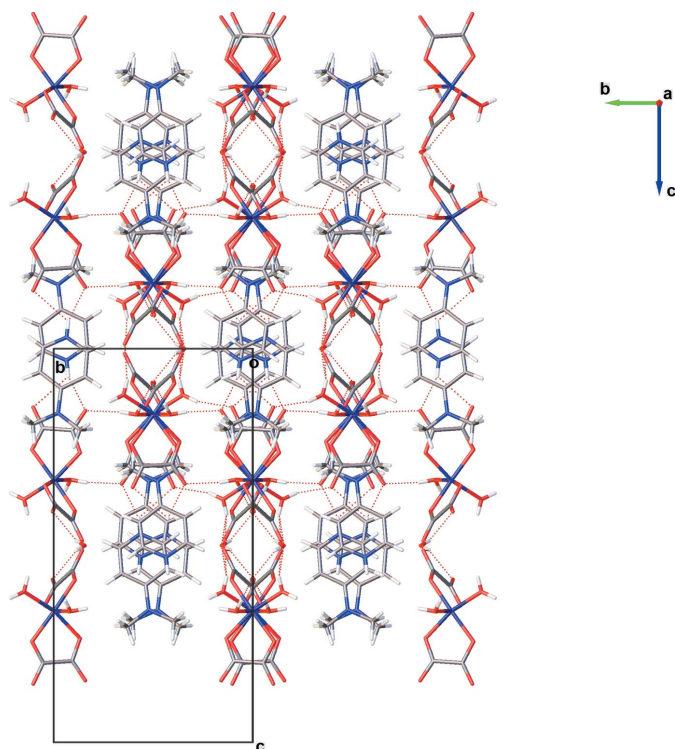


Figure 3
A (100) projection of the crystal structure of the title compound. Hydrogen bonds are shown as dashed lines.

were placed at geometrically calculated positions and refined using a riding model. C—H distances were fixed at 0.93 and 0.96 Å for aromatic and methyl C atoms, respectively. The N—H distance was fixed at 0.86 Å. The $U_{\text{iso}}(\text{H})$ values were equal to 1.2 and 1.5 times U_{eq} of the corresponding $C(sp^2)$ and $C(sp^3)$ atoms, and 1.2 times U_{eq} of the N atom. All water H atoms were located from a difference-Fourier map and refined with soft restraints on the O—H and H \cdots H distances [O—H = 0.82 (2) and H \cdots H = 1.30 (4) Å] with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

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Table 2
Experimental details.

Crystal data	
Chemical formula	$(\text{C}_7\text{H}_{11}\text{N}_2)[\text{Fe}(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_2] \cdot 0.5\text{H}_2\text{O}$
M_r	800.22
Crystal system, space group	Monoclinic, $I2/a$
Temperature (K)	296
a, b, c (Å)	14.7960 (7), 10.4422 (4), 21.7751 (10)
β (°)	108.352 (3)
V (Å ³)	3193.2 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	1.00
Crystal size (mm)	0.26 × 0.22 × 0.13
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (SADABS; Bruker, 2014)
$T_{\text{min}}, T_{\text{max}}$	0.679, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	48070, 4880, 3435
R_{int}	0.044
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.715
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.042, 0.123, 1.06
No. of reflections	4880
No. of parameters	239
No. of restraints	8
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.85, -0.58

Computer programs: APEX2 and SAINT (Bruker, 2014), SHELXS97 (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015), OLEX2 (Dolomanov *et al.*, 2009), publCIF (Westrip, 2010) and PLATON (Spek, 2009).

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Acta Cryst. (2015). E71, 934-936 [https://doi.org/10.1107/S2056989015013213]

Crystal structure of 4-(dimethylamino)pyridinium *cis*-diaquabis(oxalato- κ^2O,O')ferrate(III) hemihydrate

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Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINTE* (Bruker, 2014); data reduction: *SAINTE* (Bruker, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

4-(Dimethylamino)pyridinium *cis*-diaquabis(oxalato- κ^2O,O')ferrate(III) hemihydrate

Crystal data

(C₇H₁₁N₂)[Fe(C₂O₄)₂(H₂O)₂]·0.5H₂O

M_r = 800.22

Monoclinic, *I2/a*

a = 14.7960 (7) Å

b = 10.4422 (4) Å

c = 21.7751 (10) Å

β = 108.352 (3)°

V = 3193.2 (2) Å³

Z = 4

F(000) = 1648

D_x = 1.665 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 9923 reflections

θ = 2.2–27.3°

μ = 1.00 mm⁻¹

T = 296 K

Irregular, yellow

0.26 × 0.22 × 0.13 mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: sealed X-ray tube

φ and ω scans

Absorption correction: multi-scan
(SADABS2014; Bruker, 2014)

T_{min} = 0.679, *T_{max}* = 0.746

48070 measured reflections

4880 independent reflections

3435 reflections with *I* > 2 σ (*I*)

R_{int} = 0.044

θ_{\max} = 30.6°, θ_{\min} = 2.8°

h = -21→21

k = -14→14

l = -31→31

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2 σ (*F*²)] = 0.042

wR(*F*²) = 0.123

S = 1.06

4880 reflections

239 parameters

8 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

w = 1/[$\sigma^2(F_o^2) + (0.0528P)^2 + 5.0524P$]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

$\Delta\rho_{\max}$ = 0.85 e Å⁻³

$\Delta\rho_{\min}$ = -0.58 e Å⁻³

Special details

Experimental. Absorption correction: SADABS-2014/3 (Bruker, 2014) was used for absorption correction. wR2(int) was 0.0678 before and 0.0491 after correction. The Ratio of minimum to maximum transmission is 0.9094. The $\lambda/2$ correction factor is 0.00150.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.33846 (2)	0.99760 (3)	0.33390 (2)	0.02994 (10)
O1	0.27198 (13)	0.84143 (16)	0.15711 (7)	0.0393 (4)
O1W	0.31148 (18)	1.14895 (18)	0.38270 (9)	0.0511 (5)
H1WA	0.287 (3)	1.216 (2)	0.3669 (16)	0.077*
H1WB	0.321 (3)	1.132 (4)	0.4186 (10)	0.077*
O2	0.23808 (16)	1.10293 (17)	0.14845 (8)	0.0524 (5)
O2W	0.21255 (12)	0.91604 (16)	0.33264 (9)	0.0381 (4)
H2WA	0.187 (2)	0.941 (3)	0.3590 (13)	0.057*
H2WB	0.223 (2)	0.8379 (17)	0.3394 (15)	0.057*
O3	0.33473 (12)	0.86776 (14)	0.26407 (7)	0.0343 (3)
O4	0.29136 (13)	1.10922 (15)	0.25610 (7)	0.0389 (4)
O5	0.39926 (12)	0.89364 (16)	0.41206 (8)	0.0398 (4)
O6	0.47565 (13)	1.04452 (18)	0.34780 (8)	0.0408 (4)
O7	0.53967 (14)	0.85689 (19)	0.48747 (9)	0.0504 (5)
O8	0.62163 (14)	1.0053 (2)	0.41370 (12)	0.0625 (6)
C1	0.29387 (15)	0.9073 (2)	0.20675 (10)	0.0293 (4)
C2	0.27184 (17)	1.0531 (2)	0.20161 (10)	0.0338 (5)
C3	0.49036 (17)	0.9055 (2)	0.43727 (11)	0.0335 (5)
C4	0.53485 (18)	0.9927 (2)	0.39683 (12)	0.0377 (5)
N1	0.34280 (18)	0.5649 (3)	0.47049 (11)	0.0564 (6)
H1	0.3107	0.5735	0.4302	0.068*
N2	0.49507 (17)	0.5221 (2)	0.66339 (10)	0.0442 (5)
C5	0.44607 (18)	0.5376 (2)	0.60033 (11)	0.0371 (5)
C6	0.4118 (3)	0.4314 (3)	0.55903 (14)	0.0609 (9)
H6	0.4241	0.3488	0.5756	0.073*
C7	0.3617 (3)	0.4484 (4)	0.49606 (15)	0.0683 (10)
H7	0.3400	0.3771	0.4700	0.082*
C8	0.3735 (2)	0.6686 (3)	0.50711 (14)	0.0518 (7)
H8	0.3599	0.7493	0.4885	0.062*
C9	0.42434 (19)	0.6587 (3)	0.57107 (13)	0.0438 (6)
H9	0.4448	0.7324	0.5955	0.053*
C10	0.5313 (2)	0.6307 (3)	0.70562 (14)	0.0521 (7)
H10A	0.5788	0.6742	0.6919	0.078*
H10B	0.5591	0.6011	0.7493	0.078*
H10C	0.4801	0.6885	0.7036	0.078*
C11	0.5175 (3)	0.3955 (3)	0.69141 (16)	0.0704 (10)

H11A	0.4600	0.3467	0.6833	0.106*
H11B	0.5480	0.4030	0.7372	0.106*
H11C	0.5596	0.3530	0.6723	0.106*
O3W	0.2500	0.1564 (4)	0.5000	0.117 (2)
H3W	0.302 (3)	0.115 (5)	0.512 (3)	0.176*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.03909 (18)	0.02585 (16)	0.02228 (15)	−0.00039 (12)	0.00595 (11)	−0.00107 (11)
O1	0.0583 (11)	0.0306 (8)	0.0283 (8)	−0.0001 (7)	0.0126 (7)	−0.0071 (6)
O1W	0.0945 (16)	0.0285 (9)	0.0335 (9)	0.0046 (9)	0.0247 (10)	−0.0040 (7)
O2	0.0879 (15)	0.0359 (9)	0.0250 (8)	0.0134 (9)	0.0059 (9)	0.0014 (7)
O2W	0.0401 (9)	0.0323 (8)	0.0426 (9)	0.0015 (7)	0.0141 (7)	−0.0069 (7)
O3	0.0490 (10)	0.0253 (7)	0.0270 (7)	0.0055 (6)	0.0099 (7)	−0.0007 (6)
O4	0.0615 (11)	0.0244 (8)	0.0252 (7)	0.0070 (7)	0.0058 (7)	−0.0026 (6)
O5	0.0411 (9)	0.0398 (9)	0.0349 (8)	−0.0073 (7)	0.0070 (7)	0.0109 (7)
O6	0.0430 (9)	0.0422 (9)	0.0349 (9)	−0.0037 (7)	0.0089 (7)	0.0125 (7)
O7	0.0505 (11)	0.0505 (11)	0.0431 (10)	0.0023 (8)	0.0047 (8)	0.0186 (8)
O8	0.0355 (10)	0.0863 (17)	0.0660 (14)	0.0012 (10)	0.0163 (9)	0.0341 (12)
C1	0.0349 (11)	0.0266 (10)	0.0281 (10)	0.0003 (8)	0.0121 (8)	−0.0021 (8)
C2	0.0442 (12)	0.0283 (10)	0.0273 (10)	0.0042 (9)	0.0088 (9)	−0.0016 (8)
C3	0.0412 (12)	0.0280 (10)	0.0308 (10)	0.0008 (9)	0.0106 (9)	0.0051 (8)
C4	0.0377 (12)	0.0379 (12)	0.0390 (12)	0.0001 (9)	0.0140 (10)	0.0058 (10)
N1	0.0565 (14)	0.0824 (19)	0.0260 (10)	0.0018 (13)	0.0071 (10)	0.0052 (11)
N2	0.0548 (13)	0.0377 (11)	0.0308 (10)	0.0045 (9)	−0.0001 (9)	−0.0004 (8)
C5	0.0457 (13)	0.0341 (11)	0.0293 (11)	0.0038 (10)	0.0086 (9)	−0.0016 (9)
C6	0.090 (2)	0.0377 (15)	0.0408 (14)	0.0106 (14)	0.0006 (14)	−0.0075 (12)
C7	0.088 (3)	0.063 (2)	0.0414 (16)	0.0035 (18)	0.0019 (15)	−0.0201 (15)
C8	0.0521 (16)	0.0561 (17)	0.0468 (15)	−0.0012 (13)	0.0152 (12)	0.0233 (13)
C9	0.0515 (15)	0.0350 (13)	0.0418 (13)	−0.0050 (10)	0.0101 (11)	0.0070 (10)
C10	0.0519 (16)	0.0573 (17)	0.0395 (13)	−0.0063 (13)	0.0032 (11)	−0.0116 (12)
C11	0.087 (2)	0.0517 (18)	0.0514 (17)	0.0136 (17)	−0.0089 (16)	0.0126 (14)
O3W	0.076 (3)	0.057 (2)	0.154 (4)	0.000	−0.056 (3)	0.000

Geometric parameters (Å, °)

Fe1—O1W	2.0133 (18)	N1—C7	1.330 (5)
Fe1—O2W	2.0407 (18)	N1—C8	1.337 (4)
Fe1—O3	2.0250 (15)	N2—C5	1.345 (3)
Fe1—O4	1.9927 (16)	N2—C10	1.452 (3)
Fe1—O5	1.9799 (16)	N2—C11	1.450 (4)
Fe1—O6	2.0163 (18)	C5—C6	1.417 (4)
O1—C1	1.235 (3)	C5—C9	1.407 (3)
O1W—H1WA	0.809 (17)	C6—H6	0.9300
O1W—H1WB	0.771 (17)	C6—C7	1.349 (4)
O2—C2	1.223 (3)	C7—H7	0.9300
O2W—H2WA	0.824 (17)	C8—H8	0.9300

O2W—H2WB	0.834 (17)	C8—C9	1.363 (4)
O3—C1	1.272 (3)	C9—H9	0.9300
O4—C2	1.272 (3)	C10—H10A	0.9600
O5—C3	1.291 (3)	C10—H10B	0.9600
O6—C4	1.269 (3)	C10—H10C	0.9600
O7—C3	1.218 (3)	C11—H11A	0.9600
O8—C4	1.226 (3)	C11—H11B	0.9600
C1—C2	1.554 (3)	C11—H11C	0.9600
C3—C4	1.550 (3)	O3W—H3W	0.842 (19)
N1—H1	0.8600		
O1W—Fe1—O2W	90.16 (8)	O8—C4—O6	125.9 (2)
O1W—Fe1—O3	163.51 (8)	O8—C4—C3	119.0 (2)
O1W—Fe1—O6	95.03 (9)	C7—N1—H1	119.9
O3—Fe1—O2W	84.35 (7)	C7—N1—C8	120.2 (2)
O4—Fe1—O1W	85.14 (7)	C8—N1—H1	119.9
O4—Fe1—O2W	99.16 (8)	C5—N2—C10	121.7 (2)
O4—Fe1—O3	80.42 (6)	C5—N2—C11	121.2 (2)
O4—Fe1—O6	92.95 (7)	C11—N2—C10	117.1 (2)
O5—Fe1—O1W	95.09 (8)	N2—C5—C6	121.6 (2)
O5—Fe1—O2W	87.05 (7)	N2—C5—C9	122.9 (2)
O5—Fe1—O3	100.13 (7)	C9—C5—C6	115.5 (2)
O5—Fe1—O4	173.78 (7)	C5—C6—H6	119.5
O5—Fe1—O6	80.84 (7)	C7—C6—C5	121.0 (3)
O6—Fe1—O2W	167.19 (7)	C7—C6—H6	119.5
O6—Fe1—O3	93.64 (7)	N1—C7—C6	121.4 (3)
Fe1—O1W—H1WA	126 (3)	N1—C7—H7	119.3
Fe1—O1W—H1WB	110 (3)	C6—C7—H7	119.3
H1WA—O1W—H1WB	123 (3)	N1—C8—H8	119.2
Fe1—O2W—H2WA	118 (2)	N1—C8—C9	121.5 (3)
Fe1—O2W—H2WB	107 (2)	C9—C8—H8	119.2
H2WA—O2W—H2WB	107 (3)	C5—C9—H9	119.8
C1—O3—Fe1	114.30 (13)	C8—C9—C5	120.3 (3)
C2—O4—Fe1	116.08 (14)	C8—C9—H9	119.8
C3—O5—Fe1	116.33 (14)	N2—C10—H10A	109.5
C4—O6—Fe1	114.72 (15)	N2—C10—H10B	109.5
O1—C1—O3	126.2 (2)	N2—C10—H10C	109.5
O1—C1—C2	119.43 (19)	H10A—C10—H10B	109.5
O3—C1—C2	114.39 (17)	H10A—C10—H10C	109.5
O2—C2—O4	126.3 (2)	H10B—C10—H10C	109.5
O2—C2—C1	119.91 (19)	N2—C11—H11A	109.5
O4—C2—C1	113.77 (18)	N2—C11—H11B	109.5
O5—C3—C4	112.84 (19)	N2—C11—H11C	109.5
O7—C3—O5	126.3 (2)	H11A—C11—H11B	109.5
O7—C3—C4	120.9 (2)	H11A—C11—H11C	109.5
O6—C4—C3	115.1 (2)	H11B—C11—H11C	109.5
Fe1—O3—C1—O1	-170.06 (19)	O7—C3—C4—O6	-175.6 (2)

Fe1—O3—C1—C2	10.4 (2)	O7—C3—C4—O8	5.0 (4)
Fe1—O4—C2—O2	176.4 (2)	N1—C8—C9—C5	0.1 (4)
Fe1—O4—C2—C1	-3.0 (3)	N2—C5—C6—C7	179.1 (3)
Fe1—O5—C3—O7	174.7 (2)	N2—C5—C9—C8	-179.2 (3)
Fe1—O5—C3—C4	-4.7 (3)	C5—C6—C7—N1	0.1 (6)
Fe1—O6—C4—O8	178.3 (2)	C6—C5—C9—C8	0.0 (4)
Fe1—O6—C4—C3	-1.1 (3)	C7—N1—C8—C9	-0.2 (5)
O1—C1—C2—O2	-4.1 (4)	C8—N1—C7—C6	0.1 (5)
O1—C1—C2—O4	175.3 (2)	C9—C5—C6—C7	-0.2 (5)
O3—C1—C2—O2	175.4 (2)	C10—N2—C5—C6	179.2 (3)
O3—C1—C2—O4	-5.2 (3)	C10—N2—C5—C9	-1.6 (4)
O5—C3—C4—O6	3.8 (3)	C11—N2—C5—C6	1.9 (5)
O5—C3—C4—O8	-175.6 (2)	C11—N2—C5—C9	-178.8 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1 <i>W</i> —H1 <i>WA</i> ...O2 ⁱ	0.81 (2)	1.94 (2)	2.720 (3)	162 (4)
O1 <i>W</i> —H1 <i>WB</i> ...O7 ⁱⁱ	0.77 (2)	2.40 (3)	2.988 (3)	134 (4)
O1 <i>W</i> —H1 <i>WB</i> ...O3 <i>W</i> ⁱⁱⁱ	0.77 (2)	2.34 (3)	2.9699 (19)	139 (4)
O2 <i>W</i> —H2 <i>WA</i> ...O8 ^{iv}	0.82 (2)	1.84 (2)	2.664 (3)	176 (3)
O2 <i>W</i> —H2 <i>WB</i> ...O1 ^v	0.83 (2)	1.88 (2)	2.702 (2)	171 (3)
N1—H1...O1 ^v	0.86	2.11	2.931 (3)	160
N1—H1...O2 ^v	0.86	2.46	3.043 (3)	125
O3 <i>W</i> —H3 <i>W</i> ...O7 ^{vi}	0.84 (2)	2.36 (5)	3.040 (2)	138 (6)
O3 <i>W</i> —H3 <i>W</i> ...O8 ^{vi}	0.84 (2)	2.09 (5)	2.782 (3)	140 (6)

Symmetry codes: (i) $-x+1/2, -y+5/2, -z+1/2$; (ii) $-x+1, -y+2, -z+1$; (iii) $x, y+1, z$; (iv) $x-1/2, -y+2, z$; (v) $-x+1/2, -y+3/2, -z+1/2$; (vi) $-x+1, -y+1, -z+1$.