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{Tris[2-(imidazol-2-ylmethylimino)ethyl]-methylammonium}iron(II) tris(perchlorate) dihydrate

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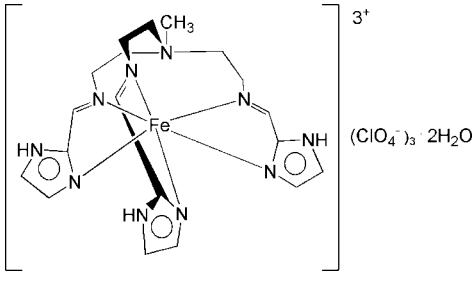
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.108; data-to-parameter ratio = 19.1.

The title complex, $[Fe(C_{19}H_{27}N_{10})](ClO_4)_3\cdot 2H_2O$, is a new polymorph of an iron(II) Schiff base complex of tris(2-aminoethyl)methylammonium with imidazole-2-carboxaldehyde. The octahedral Fe^{II} atom is bound to three facial imidazole N atoms with average $Fe-N_{imidazole}$ and $Fe-N_{imine}$ bond distances of 1.963 (5) and 1.951 (5) Å, respectively. The central N atom of the tripodal ligand is outside the bonding distance at 3.92 Å. The crystal packing is stabilized by the hydrogen-bonding interactions between the two water molecules (acceptor) and two of the three imidazole NH groups (donor). The third imidazole NH group (donor) forms a hydrogen bond to one of the three perchlorate counter-ions (acceptor).

Related literature

For the synthesis, see: Brewer *et al.* (2005). For related structures, see: Brewer *et al.* (2006, 2007).



Experimental

Crystal data

$[Fe(C_{19}H_{27}N_{10})](ClO_4)_3\cdot 2H_2O$
M_r = 785.74
Orthorhombic, *Pbca*
a = 13.9630 (18) Å
b = 11.7810 (15) Å
c = 37.182 (5) Å
V = 6116.4 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.84 mm^{-1}
T = 173 K
0.54 × 0.45 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.682, T_{max} = 0.904
65065 measured reflections
8324 independent reflections
6601 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.108
S = 1.04
8324 reflections
436 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.76 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W2···O14^{i}	0.807 (19)	2.29 (4)	2.938 (3)	138 (5)
O2W—H2W1···O14^{ii}	0.820 (17)	2.26 (2)	2.987 (3)	148 (4)
O2W—H2W2···O22^{iii}	0.821 (18)	2.07 (2)	2.861 (3)	160 (4)
N3A—H3AB···O2W	0.88	1.91	2.730 (3)	155
N3B—H3BB···O1W	0.88	1.95	2.752 (3)	152
N3C—H3CB···O14	0.88	2.05	2.907 (3)	163

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

RJB acknowleges the Laboratory for the Structure of Matter at the Naval Research Laboratory for access to their diffractometers.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2214).

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supporting information

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S1. Comment

Iron(II) and iron(III) Schiff base complexes of tris(2-aminoethyl)amine (tren) with imidazole carboxaldehyde have displayed spin crossover behavior (Brewer *et al.*, 2006). Further, it has been demonstrated that the distance between the Fe atom and the central tren-N atom, N_{ap}, is an indicator of spin-state (Brewer *et al.*, 2006). Shorter distances correlate with high spin and longer distances with low spin. Quarternization of N_{ap}, as observed in the title complex, (I), results in an elongated Fe—N_{ap} distance due to both the conformation of the N_{ap} atom (inverted away from the Fe atom) and the repulsive forces between the positively charged atoms (Brewer *et al.*, 2005). Recently, it was shown that (I), without the methyl group on N_{ap}, could serve as a bidentate hydrogen bond donor to the perchlorate anion of potassium perchlorate using the adjacent imidazole-NH and imine-CH H atoms to give supramolecular complexes (Brewer *et al.*, 2007). Since the present molecule possesses this same structural feature, the reaction of it with potassium perchlorate was investigated. The reaction did not yield the desired product but gave (I) as a polymorph (Brewer *et al.*, 2007). The structure of the iron cation differs from the original report in that the three arms of the ligand are not identical. In addition, the hydrogen bonding interactions with coordinated water and perchlorate are significantly different. Investigation of these effects on the spin crossover process and reactivity of the complex will be aided by the structural characterization of this new polymorph. In view of the importance of the spin crossover phenomenom and supramolecular systems, the present paper reports the crystal structure of (I) (Fig. 1).

The octahedral iron(II) atom is bound to three facial imidazole-N atoms whose average Fe–N bond distances for the imidazole- and imine-N atoms are 1.963 (5)Å and 1.951 (5) Å, respectively. The central N atom of the tripodal ligand is outside the bonding distance at 3.92 Å. Crystal packing is stabilized by the hydrogen bonding interactions between the two water molecules (acceptor) and two of the three imidazole NH groups (donor). The third imidazole NH group (donor) hydrogen bonds to one of the three perchlorate counterions (acceptor) (Table 1 & Fig. 2).

S2. Experimental

Complex (I) was synthesized as previously described (Brewer *et al.*, 2005) and was recrystallized from methanol solution in the presence of equimolal potassium perchlorate. The resulting crystals were analyzed by X-ray diffraction.

S3. Refinement

The positional parameters of the water-bound H atoms were refined with U_{iso}(H) = 1.17–1.49U_{eq}(C,*N*); see Table 1 for distances. The remaining H atoms were included in the riding model approximation with N—H = 0.88Å and C—H = 0.95 to 0.99 Å, and with U_{iso}(H) = 1.17–1.49U_{eq}(C,*N*).

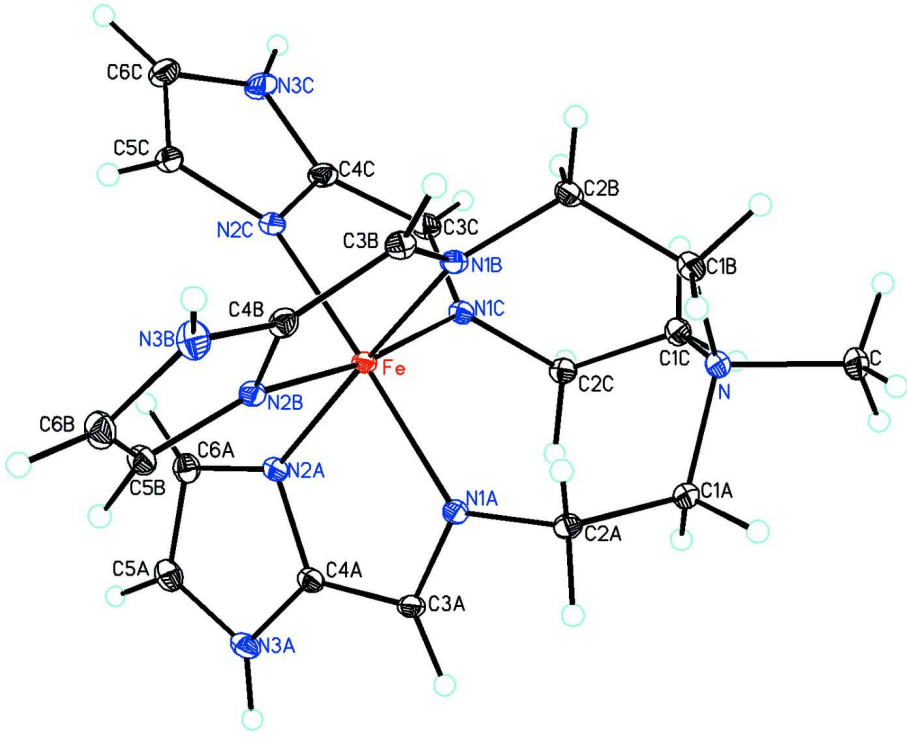

Figure 1

Molecular structure of the cation in (I), showing atom labeling and 50% probability displacement ellipsoids.

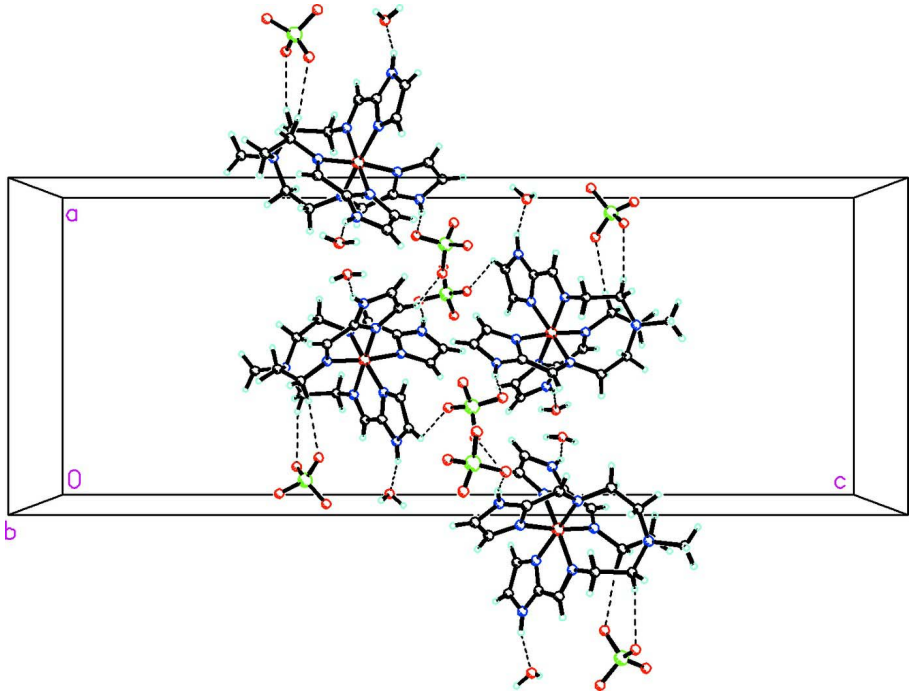

Figure 2

Partial packing diagram for (I), viewed down the *b* axis. Dashed lines indicate C–H···O (water & perchlorate) hydrogen bonds.

{Tris[2-(imidazol-2-ylmethylimino)ethyl]methylammonium}iron(II) tris(perchlorate) dihydrate

Crystal data

$[Fe(C_{19}H_{27}N_{10})](ClO_4)_3 \cdot 2H_2O$
$M_r = 785.74$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 13.9630\ (18)$ Å
$b = 11.7810\ (15)$ Å
$c = 37.182\ (5)$ Å
$V = 6116.4\ (14)$ Å^3
$Z = 8$
$F(000) = 3232$
$D_x = 1.707$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9963 reflections
$\theta = 2.2–28.9°$
$\mu = 0.84$ mm^{-1}
$T = 173$ K
Chunk, dark-red
0.54 × 0.45 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.682$, $T_{max} = 0.904$
65065 measured reflections
8324 independent reflections
6601 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 29.3°$, $\theta_{min} = 1.8°$
$h = -19 \rightarrow 19$
$k = -16 \rightarrow 13$
$l = -51 \rightarrow 51$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.108$
$S = 1.04$
8324 reflections
436 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 5.7069P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.76$ e Å^{-3}
$\Delta\rho_{min} = -0.62$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe	0.542493 (19)	0.51159 (2)	0.110991 (7)	0.01627 (7)
Cl1	0.32256 (4)	0.00301 (5)	0.015096 (15)	0.02982 (12)
Cl2	0.24581 (4)	0.63667 (5)	0.208500 (15)	0.02861 (12)
Cl3	0.57715 (4)	0.00513 (5)	0.181644 (16)	0.03151 (13)

O11	0.3162 (2)	0.0909 (2)	−0.01088 (7)	0.0756 (8)
O12	0.23275 (13)	−0.05400 (18)	0.01888 (6)	0.0489 (5)
O13	0.39474 (14)	−0.07620 (18)	0.00529 (6)	0.0526 (5)
O14	0.34756 (14)	0.05377 (19)	0.04951 (5)	0.0494 (5)
O21	0.23376 (18)	0.51820 (17)	0.21519 (7)	0.0598 (6)
O22	0.19608 (18)	0.6630 (2)	0.17533 (6)	0.0610 (6)
O23	0.34507 (13)	0.66157 (19)	0.20374 (6)	0.0505 (5)
O24	0.20629 (13)	0.70357 (17)	0.23716 (5)	0.0437 (5)
O31	0.50024 (19)	−0.0216 (2)	0.15709 (7)	0.0687 (7)
O32	0.61507 (15)	−0.10022 (17)	0.19422 (8)	0.0617 (6)
O33	0.65127 (16)	0.06867 (16)	0.16416 (7)	0.0570 (6)
O34	0.53883 (17)	0.06912 (18)	0.21060 (6)	0.0576 (6)
O1W	0.96601 (19)	0.4969 (3)	0.08139 (9)	0.1023 (13)
H1W1	0.983 (4)	0.562 (3)	0.0904 (16)	0.153*
H1W2	1.003 (4)	0.489 (5)	0.0650 (12)	0.153*
O2W	0.28606 (16)	0.98531 (17)	0.12333 (6)	0.0461 (5)
H2W1	0.287 (3)	1.024 (3)	0.1051 (6)	0.069*
H2W2	0.304 (3)	1.029 (3)	0.1392 (7)	0.069*
N	0.56728 (13)	0.38916 (15)	0.20854 (4)	0.0215 (3)
N1A	0.54566 (12)	0.61049 (14)	0.15322 (4)	0.0190 (3)
N2A	0.44316 (12)	0.61938 (14)	0.09684 (5)	0.0199 (3)
N3A	0.36892 (13)	0.78081 (16)	0.10830 (5)	0.0269 (4)
H3AB	0.3517	0.8440	0.1192	0.032*
N1B	0.65376 (12)	0.42147 (14)	0.12542 (4)	0.0193 (3)
N2B	0.64328 (12)	0.60169 (14)	0.08733 (4)	0.0198 (3)
N3B	0.79608 (13)	0.61176 (17)	0.07239 (5)	0.0288 (4)
H3BB	0.8573	0.5947	0.0707	0.035*
N1C	0.45107 (12)	0.40472 (14)	0.13101 (4)	0.0192 (3)
N2C	0.52259 (12)	0.41612 (14)	0.06825 (4)	0.0198 (3)
N3C	0.44957 (15)	0.26827 (17)	0.04444 (5)	0.0304 (4)
H3CB	0.4121	0.2086	0.0422	0.036*
C	0.57789 (18)	0.3404 (2)	0.24639 (6)	0.0301 (5)
H0A	0.5769	0.2573	0.2452	0.045*
H0B	0.5248	0.3669	0.2615	0.045*
H0C	0.6388	0.3656	0.2568	0.045*
C1A	0.56992 (16)	0.51802 (17)	0.21406 (5)	0.0236 (4)
H1AA	0.5037	0.5427	0.2195	0.028*
H1AB	0.6084	0.5327	0.2359	0.028*
C2A	0.60808 (15)	0.59560 (17)	0.18461 (5)	0.0220 (4)
H2AA	0.6205	0.6712	0.1952	0.026*
H2AB	0.6702	0.5650	0.1762	0.026*
C3A	0.49249 (15)	0.69994 (17)	0.15189 (5)	0.0214 (4)
H3AA	0.4921	0.7567	0.1701	0.026*
C4A	0.43400 (14)	0.70505 (17)	0.12017 (5)	0.0209 (4)
C5A	0.33456 (16)	0.7412 (2)	0.07621 (6)	0.0301 (5)
H5AA	0.2875	0.7764	0.0615	0.036*
C6A	0.38088 (15)	0.64128 (19)	0.06928 (6)	0.0258 (4)
H6AA	0.3714	0.5947	0.0487	0.031*

C1B	0.65416 (15)	0.34079 (18)	0.18863 (6)	0.0238 (4)
H1BA	0.7086	0.3924	0.1934	0.029*
H1BB	0.6700	0.2674	0.2001	0.029*
C2B	0.65151 (15)	0.32028 (17)	0.14818 (5)	0.0221 (4)
H2BA	0.7068	0.2718	0.1417	0.026*
H2BB	0.5926	0.2771	0.1425	0.026*
C3B	0.73460 (15)	0.45056 (18)	0.11120 (5)	0.0221 (4)
H3BA	0.7922	0.4088	0.1144	0.027*
C4B	0.72866 (14)	0.55230 (18)	0.09009 (5)	0.0219 (4)
C5B	0.65706 (16)	0.69750 (18)	0.06694 (6)	0.0243 (4)
H5BA	0.6089	0.7507	0.0605	0.029*
C6B	0.75165 (17)	0.7035 (2)	0.05749 (6)	0.0300 (5)
H6BA	0.7809	0.7609	0.0433	0.036*
C1C	0.47042 (15)	0.34435 (18)	0.19572 (6)	0.0233 (4)
H1CA	0.4811	0.2670	0.1861	0.028*
H1CB	0.4291	0.3363	0.2172	0.028*
C2C	0.41328 (14)	0.40958 (18)	0.16763 (5)	0.0217 (4)
H2CA	0.3470	0.3797	0.1674	0.026*
H2CB	0.4100	0.4901	0.1751	0.026*
C3C	0.41876 (15)	0.32709 (18)	0.10959 (6)	0.0243 (4)
H3CA	0.3723	0.2723	0.1165	0.029*
C4C	0.46053 (15)	0.33235 (18)	0.07423 (6)	0.0244 (4)
C5C	0.55206 (15)	0.40500 (19)	0.03331 (6)	0.0245 (4)
H5CA	0.5964	0.4533	0.0214	0.029*
C6C	0.50736 (18)	0.3132 (2)	0.01838 (6)	0.0310 (5)
H6CA	0.5150	0.2858	−0.0055	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe	0.01808 (13)	0.01279 (13)	0.01794 (13)	−0.00035 (10)	0.00077 (10)	−0.00083 (10)
Cl1	0.0326 (3)	0.0217 (3)	0.0352 (3)	0.0011 (2)	−0.0064 (2)	−0.0012 (2)
Cl2	0.0264 (2)	0.0239 (3)	0.0356 (3)	0.00118 (19)	0.0062 (2)	−0.0030 (2)
Cl3	0.0313 (3)	0.0234 (3)	0.0399 (3)	0.0000 (2)	0.0070 (2)	−0.0013 (2)
O11	0.118 (2)	0.0463 (14)	0.0629 (15)	0.0105 (14)	−0.0153 (15)	0.0256 (12)
O12	0.0305 (9)	0.0428 (11)	0.0734 (14)	−0.0057 (8)	−0.0084 (9)	−0.0190 (10)
O13	0.0387 (11)	0.0425 (12)	0.0765 (15)	0.0111 (9)	0.0060 (10)	−0.0037 (10)
O14	0.0471 (11)	0.0592 (13)	0.0418 (11)	−0.0193 (10)	−0.0031 (9)	−0.0152 (10)
O21	0.0761 (16)	0.0259 (10)	0.0775 (16)	−0.0005 (10)	0.0343 (13)	0.0033 (10)
O22	0.0761 (16)	0.0557 (14)	0.0512 (12)	0.0259 (12)	−0.0215 (11)	−0.0124 (10)
O23	0.0311 (9)	0.0589 (13)	0.0615 (13)	−0.0122 (9)	0.0157 (9)	−0.0303 (10)
O24	0.0441 (10)	0.0425 (11)	0.0447 (10)	0.0049 (9)	0.0181 (8)	−0.0097 (8)
O31	0.0615 (15)	0.0787 (18)	0.0660 (16)	−0.0064 (13)	−0.0182 (12)	0.0026 (13)
O32	0.0452 (12)	0.0272 (10)	0.113 (2)	0.0029 (9)	−0.0029 (12)	0.0131 (11)
O33	0.0592 (13)	0.0294 (10)	0.0824 (16)	−0.0085 (9)	0.0369 (12)	−0.0004 (10)
O34	0.0787 (16)	0.0403 (12)	0.0538 (13)	−0.0004 (11)	0.0307 (11)	−0.0083 (10)
O1W	0.0430 (14)	0.185 (4)	0.079 (2)	0.0426 (19)	0.0211 (13)	0.056 (2)
O2W	0.0545 (12)	0.0397 (11)	0.0442 (11)	0.0104 (10)	−0.0002 (10)	−0.0089 (9)

N	0.0263 (8)	0.0185 (8)	0.0196 (8)	−0.0007 (7)	0.0005 (7)	0.0020 (6)
N1A	0.0221 (8)	0.0159 (8)	0.0190 (8)	−0.0029 (6)	0.0008 (6)	0.0002 (6)
N2A	0.0207 (8)	0.0163 (8)	0.0227 (8)	0.0005 (6)	0.0009 (6)	0.0007 (6)
N3A	0.0275 (9)	0.0192 (9)	0.0340 (10)	0.0054 (7)	0.0034 (8)	0.0025 (7)
N1B	0.0221 (8)	0.0155 (8)	0.0203 (8)	0.0013 (6)	0.0020 (6)	−0.0021 (6)
N2B	0.0230 (8)	0.0168 (8)	0.0195 (8)	−0.0023 (6)	0.0008 (6)	−0.0013 (6)
N3B	0.0237 (9)	0.0324 (10)	0.0304 (9)	−0.0048 (8)	0.0048 (7)	0.0049 (8)
N1C	0.0198 (8)	0.0161 (8)	0.0218 (8)	−0.0002 (6)	0.0013 (6)	0.0000 (6)
N2C	0.0227 (8)	0.0158 (8)	0.0209 (8)	0.0024 (6)	0.0001 (6)	−0.0013 (6)
N3C	0.0373 (10)	0.0250 (10)	0.0288 (9)	−0.0055 (8)	−0.0040 (8)	−0.0109 (8)
C	0.0424 (13)	0.0268 (11)	0.0212 (10)	−0.0012 (10)	−0.0011 (9)	0.0066 (8)
C1A	0.0323 (10)	0.0193 (10)	0.0193 (9)	−0.0004 (8)	−0.0019 (8)	−0.0014 (7)
C2A	0.0267 (10)	0.0177 (9)	0.0217 (9)	−0.0037 (8)	−0.0044 (8)	−0.0026 (7)
C3A	0.0268 (10)	0.0150 (9)	0.0224 (9)	−0.0010 (7)	0.0047 (8)	−0.0023 (7)
C4A	0.0212 (9)	0.0170 (9)	0.0243 (10)	0.0005 (7)	0.0040 (7)	0.0015 (7)
C5A	0.0277 (11)	0.0297 (12)	0.0329 (12)	0.0038 (9)	−0.0022 (9)	0.0069 (9)
C6A	0.0246 (10)	0.0276 (11)	0.0251 (10)	−0.0010 (8)	−0.0022 (8)	0.0025 (8)
C1B	0.0246 (10)	0.0214 (10)	0.0254 (10)	0.0031 (8)	−0.0014 (8)	0.0038 (8)
C2B	0.0254 (10)	0.0163 (9)	0.0245 (10)	0.0025 (8)	0.0021 (8)	0.0021 (7)
C3B	0.0213 (9)	0.0220 (10)	0.0231 (9)	0.0014 (8)	0.0009 (8)	−0.0022 (8)
C4B	0.0218 (9)	0.0231 (10)	0.0209 (9)	−0.0026 (8)	0.0033 (7)	−0.0012 (8)
C5B	0.0301 (10)	0.0191 (10)	0.0236 (10)	−0.0033 (8)	−0.0012 (8)	0.0022 (8)
C6B	0.0344 (12)	0.0275 (12)	0.0279 (10)	−0.0087 (9)	0.0032 (9)	0.0049 (9)
C1C	0.0250 (10)	0.0211 (10)	0.0238 (10)	−0.0045 (8)	0.0026 (8)	0.0025 (8)
C2C	0.0199 (9)	0.0213 (10)	0.0241 (10)	−0.0008 (8)	0.0039 (8)	−0.0003 (8)
C3C	0.0240 (10)	0.0195 (10)	0.0293 (10)	−0.0046 (8)	0.0004 (8)	−0.0019 (8)
C4C	0.0273 (10)	0.0197 (10)	0.0261 (10)	−0.0011 (8)	−0.0039 (8)	−0.0056 (8)
C5C	0.0292 (10)	0.0229 (10)	0.0213 (9)	0.0046 (8)	−0.0007 (8)	−0.0019 (8)
C6C	0.0384 (12)	0.0309 (12)	0.0236 (10)	0.0044 (10)	−0.0027 (9)	−0.0080 (9)

Geometric parameters (Å, º)

Fe—N1C	1.9414 (17)	N1C—C3C	1.294 (3)
Fe—N2A	1.9528 (17)	N1C—C2C	1.461 (3)
Fe—N1A	1.9559 (17)	N2C—C4C	1.332 (3)
Fe—N1B	1.9567 (17)	N2C—C5C	1.369 (3)
Fe—N2C	1.9665 (17)	N3C—C4C	1.349 (3)
Fe—N2B	1.9701 (17)	N3C—C6C	1.368 (3)
Cl1—O11	1.418 (2)	N3C—H3CB	0.8800
Cl1—O13	1.421 (2)	C—H0A	0.9800
Cl1—O12	1.4295 (19)	C—H0B	0.9800
Cl1—O14	1.4548 (19)	C—H0C	0.9800
Cl2—O23	1.4277 (18)	C1A—C2A	1.523 (3)
Cl2—O21	1.428 (2)	C1A—H1AA	0.9900
Cl2—O24	1.4357 (18)	C1A—H1AB	0.9900
Cl2—O22	1.449 (2)	C2A—H2AA	0.9900
Cl3—O34	1.419 (2)	C2A—H2AB	0.9900
Cl3—O32	1.428 (2)	C3A—C4A	1.436 (3)

Cl3—O33	1.4330 (19)	C3A—H3AA	0.9500
Cl3—O31	1.444 (2)	C5A—C6A	1.368 (3)
O1W—H1W1	0.866 (19)	C5A—H5AA	0.9500
O1W—H1W2	0.807 (19)	C6A—H6AA	0.9500
O2W—H2W1	0.820 (17)	C1B—C2B	1.524 (3)
O2W—H2W2	0.821 (18)	C1B—H1BA	0.9900
N—C	1.527 (3)	C1B—H1BB	0.9900
N—C1C	1.528 (3)	C2B—H2BA	0.9900
N—C1B	1.531 (3)	C2B—H2BB	0.9900
N—C1A	1.532 (3)	C3B—C4B	1.435 (3)
N1A—C3A	1.290 (3)	C3B—H3BA	0.9500
N1A—C2A	1.467 (2)	C5B—C6B	1.369 (3)
N2A—C4A	1.337 (3)	C5B—H5BA	0.9500
N2A—C6A	1.368 (3)	C6B—H6BA	0.9500
N3A—C4A	1.348 (3)	C1C—C2C	1.523 (3)
N3A—C5A	1.368 (3)	C1C—H1CA	0.9900
N3A—H3AB	0.8800	C1C—H1CB	0.9900
N1B—C3B	1.293 (3)	C2C—H2CA	0.9900
N1B—C2B	1.462 (3)	C2C—H2CB	0.9900
N2B—C4B	1.330 (3)	C3C—C4C	1.440 (3)
N2B—C5B	1.373 (3)	C3C—H3CA	0.9500
N3B—C4B	1.345 (3)	C5C—C6C	1.367 (3)
N3B—C6B	1.364 (3)	C5C—H5CA	0.9500
N3B—H3BB	0.8800	C6C—H6CA	0.9500

N1C—Fe—N2A	93.33 (7)	H0B—C—H0C	109.5
N1C—Fe—N1A	95.36 (7)	C2A—C1A—N	120.44 (17)
N2A—Fe—N1A	81.09 (7)	C2A—C1A—H1AA	107.2
N1C—Fe—N1B	93.73 (7)	N—C1A—H1AA	107.2
N2A—Fe—N1B	172.17 (7)	C2A—C1A—H1AB	107.2
N1A—Fe—N1B	94.88 (7)	N—C1A—H1AB	107.2
N1C—Fe—N2C	81.14 (7)	H1AA—C1A—H1AB	106.8
N2A—Fe—N2C	93.08 (7)	N1A—C2A—C1A	115.85 (17)
N1A—Fe—N2C	173.05 (7)	N1A—C2A—H2AA	108.3
N1B—Fe—N2C	91.34 (7)	C1A—C2A—H2AA	108.3
N1C—Fe—N2B	172.05 (7)	N1A—C2A—H2AB	108.3
N2A—Fe—N2B	92.10 (7)	C1A—C2A—H2AB	108.3
N1A—Fe—N2B	91.22 (7)	H2AA—C2A—H2AB	107.4
N1B—Fe—N2B	81.23 (7)	N1A—C3A—C4A	113.15 (18)
N2C—Fe—N2B	92.77 (7)	N1A—C3A—H3AA	123.4
O11—Cl1—O13	110.43 (16)	C4A—C3A—H3AA	123.4
O11—Cl1—O12	110.79 (16)	N2A—C4A—N3A	110.61 (18)
O13—Cl1—O12	109.80 (12)	N2A—C4A—C3A	116.54 (18)
O11—Cl1—O14	108.29 (15)	N3A—C4A—C3A	132.85 (19)
O13—Cl1—O14	108.99 (13)	C6A—C5A—N3A	106.98 (19)
O12—Cl1—O14	108.48 (12)	C6A—C5A—H5AA	126.5
O23—Cl2—O21	109.69 (14)	N3A—C5A—H5AA	126.5
O23—Cl2—O24	110.63 (11)	C5A—C6A—N2A	108.8 (2)

O21—Cl2—O24	111.22 (13)	C5A—C6A—H6AA	125.6
O23—Cl2—O22	108.41 (15)	N2A—C6A—H6AA	125.6
O21—Cl2—O22	107.53 (15)	C2B—C1B—N	121.13 (17)
O24—Cl2—O22	109.27 (13)	C2B—C1B—H1BA	107.0
O34—Cl3—O32	110.68 (15)	N—C1B—H1BA	107.0
O34—Cl3—O33	109.81 (13)	C2B—C1B—H1BB	107.0
O32—Cl3—O33	109.56 (13)	N—C1B—H1BB	107.0
O34—Cl3—O31	108.37 (16)	H1BA—C1B—H1BB	106.8
O32—Cl3—O31	107.02 (15)	N1B—C2B—C1B	116.20 (17)
O33—Cl3—O31	111.37 (16)	N1B—C2B—H2BA	108.2
H1W1—O1W—H1W2	103 (3)	C1B—C2B—H2BA	108.2
H2W1—O2W—H2W2	104 (2)	N1B—C2B—H2BB	108.2
C—N—C1C	104.07 (16)	C1B—C2B—H2BB	108.2
C—N—C1B	103.21 (16)	H2BA—C2B—H2BB	107.4
C1C—N—C1B	114.95 (16)	N1B—C3B—C4B	113.24 (18)
C—N—C1A	104.30 (16)	N1B—C3B—H3BA	123.4
C1C—N—C1A	113.92 (16)	C4B—C3B—H3BA	123.4
C1B—N—C1A	114.49 (16)	N2B—C4B—N3B	111.19 (19)
C3A—N1A—C2A	118.04 (17)	N2B—C4B—C3B	117.34 (18)
C3A—N1A—Fe	116.28 (14)	N3B—C4B—C3B	131.4 (2)
C2A—N1A—Fe	125.51 (13)	C6B—C5B—N2B	108.64 (19)
C4A—N2A—C6A	106.42 (17)	C6B—C5B—H5BA	125.7
C4A—N2A—Fe	112.58 (14)	N2B—C5B—H5BA	125.7
C6A—N2A—Fe	140.88 (15)	N3B—C6B—C5B	107.06 (19)
C4A—N3A—C5A	107.23 (18)	N3B—C6B—H6BA	126.5
C4A—N3A—H3AB	126.4	C5B—C6B—H6BA	126.5
C5A—N3A—H3AB	126.4	C2C—C1C—N	120.22 (17)
C3B—N1B—C2B	118.14 (17)	C2C—C1C—H1CA	107.3
C3B—N1B—Fe	115.93 (14)	N—C1C—H1CA	107.3
C2B—N1B—Fe	125.72 (13)	C2C—C1C—H1CB	107.3
C4B—N2B—C5B	106.05 (17)	N—C1C—H1CB	107.3
C4B—N2B—Fe	111.71 (13)	H1CA—C1C—H1CB	106.9
C5B—N2B—Fe	142.13 (15)	N1C—C2C—C1C	115.48 (17)
C4B—N3B—C6B	107.04 (19)	N1C—C2C—H2CA	108.4
C4B—N3B—H3BB	126.5	C1C—C2C—H2CA	108.4
C6B—N3B—H3BB	126.5	N1C—C2C—H2CB	108.4
C3C—N1C—C2C	118.37 (17)	C1C—C2C—H2CB	108.4
C3C—N1C—Fe	116.85 (14)	H2CA—C2C—H2CB	107.5
C2C—N1C—Fe	124.70 (13)	N1C—C3C—C4C	112.98 (18)
C4C—N2C—C5C	106.43 (17)	N1C—C3C—H3CA	123.5
C4C—N2C—Fe	112.39 (14)	C4C—C3C—H3CA	123.5
C5C—N2C—Fe	141.17 (15)	N2C—C4C—N3C	110.58 (19)
C4C—N3C—C6C	107.37 (19)	N2C—C4C—C3C	116.60 (18)
C4C—N3C—H3CB	126.3	N3C—C4C—C3C	132.8 (2)
C6C—N3C—H3CB	126.3	C6C—C5C—N2C	108.9 (2)
N—C—H0A	109.5	C6C—C5C—H5CA	125.5
N—C—H0B	109.5	N2C—C5C—H5CA	125.5
H0A—C—H0B	109.5	C5C—C6C—N3C	106.71 (19)

N—C—H0C	109.5	C5C—C6C—H6CA	126.6
H0A—C—H0C	109.5	N3C—C6C—H6CA	126.6

N1C—Fe—N1A—C3A	−98.03 (15)	Fe—N1A—C3A—C4A	4.9 (2)
N2A—Fe—N1A—C3A	−5.49 (15)	C6A—N2A—C4A—N3A	−0.5 (2)
N1B—Fe—N1A—C3A	167.75 (15)	Fe—N2A—C4A—N3A	176.30 (13)
N2B—Fe—N1A—C3A	86.44 (15)	C6A—N2A—C4A—C3A	179.43 (18)
N2A—Fe—N1A—C2A	179.28 (16)	Fe—N2A—C4A—C3A	−3.7 (2)
N1B—Fe—N1A—C2A	−7.49 (16)	C5A—N3A—C4A—N2A	0.6 (2)
N2B—Fe—N1A—C2A	−88.79 (16)	C5A—N3A—C4A—C3A	−179.4 (2)
N1C—Fe—N2A—C4A	99.71 (14)	N1A—C3A—C4A—N2A	−0.7 (3)
N1A—Fe—N2A—C4A	4.80 (14)	N1A—C3A—C4A—N3A	179.3 (2)
N2C—Fe—N2A—C4A	−178.99 (14)	C4A—N3A—C5A—C6A	−0.4 (2)
N2B—Fe—N2A—C4A	−86.10 (14)	N3A—C5A—C6A—N2A	0.0 (2)
N1C—Fe—N2A—C6A	−85.1 (2)	C4A—N2A—C6A—C5A	0.3 (2)
N1A—Fe—N2A—C6A	180.0 (2)	Fe—N2A—C6A—C5A	−175.06 (17)
N2C—Fe—N2A—C6A	−3.8 (2)	C—N—C1B—C2B	151.74 (19)
N2B—Fe—N2A—C6A	89.1 (2)	C1C—N—C1B—C2B	39.1 (3)
N1C—Fe—N1B—C3B	167.02 (15)	C1A—N—C1B—C2B	−95.6 (2)
N1A—Fe—N1B—C3B	−97.27 (15)	C3B—N1B—C2B—C1B	99.6 (2)
N2C—Fe—N1B—C3B	85.81 (15)	Fe—N1B—C2B—C1B	−85.8 (2)
N2B—Fe—N1B—C3B	−6.79 (15)	N—C1B—C2B—N1B	72.2 (2)
N1C—Fe—N1B—C2B	−7.71 (16)	C2B—N1B—C3B—C4B	−178.72 (17)
N1A—Fe—N1B—C2B	88.00 (16)	Fe—N1B—C3B—C4B	6.1 (2)
N2C—Fe—N1B—C2B	−88.92 (16)	C5B—N2B—C4B—N3B	−0.2 (2)
N2B—Fe—N1B—C2B	178.48 (16)	Fe—N2B—C4B—N3B	177.05 (14)
N2A—Fe—N2B—C4B	−178.30 (14)	C5B—N2B—C4B—C3B	178.36 (18)
N1A—Fe—N2B—C4B	100.57 (14)	Fe—N2B—C4B—C3B	−4.4 (2)
N1B—Fe—N2B—C4B	5.82 (14)	C6B—N3B—C4B—N2B	−0.1 (2)
N2C—Fe—N2B—C4B	−85.11 (14)	C6B—N3B—C4B—C3B	−178.4 (2)
N2A—Fe—N2B—C5B	−2.7 (2)	N1B—C3B—C4B—N2B	−1.0 (3)
N1A—Fe—N2B—C5B	−83.8 (2)	N1B—C3B—C4B—N3B	177.2 (2)
N1B—Fe—N2B—C5B	−178.5 (2)	C4B—N2B—C5B—C6B	0.4 (2)
N2C—Fe—N2B—C5B	90.5 (2)	Fe—N2B—C5B—C6B	−175.37 (17)
N2A—Fe—N1C—C3C	91.28 (16)	C4B—N3B—C6B—C5B	0.4 (3)
N1A—Fe—N1C—C3C	172.62 (16)	N2B—C5B—C6B—N3B	−0.5 (3)
N1B—Fe—N1C—C3C	−92.11 (16)	C—N—C1C—C2C	153.57 (18)
N2C—Fe—N1C—C3C	−1.33 (15)	C1B—N—C1C—C2C	−94.3 (2)
N2A—Fe—N1C—C2C	−85.53 (16)	C1A—N—C1C—C2C	40.6 (2)
N1A—Fe—N1C—C2C	−4.18 (16)	C3C—N1C—C2C—C1C	93.2 (2)
N1B—Fe—N1C—C2C	91.09 (16)	Fe—N1C—C2C—C1C	−90.1 (2)
N2C—Fe—N1C—C2C	−178.13 (16)	N—C1C—C2C—N1C	72.7 (2)
N1C—Fe—N2C—C4C	0.15 (14)	C2C—N1C—C3C—C4C	179.14 (18)
N2A—Fe—N2C—C4C	−92.75 (15)	Fe—N1C—C3C—C4C	2.1 (2)
N1B—Fe—N2C—C4C	93.72 (15)	C5C—N2C—C4C—N3C	0.1 (2)
N2B—Fe—N2C—C4C	175.00 (15)	Fe—N2C—C4C—N3C	−179.40 (14)
N2A—Fe—N2C—C5C	88.0 (2)	C5C—N2C—C4C—C3C	−179.55 (18)
N1B—Fe—N2C—C5C	−85.6 (2)	Fe—N2C—C4C—C3C	0.9 (2)

N2B—Fe—N2C—C5C	−4.3 (2)	C6C—N3C—C4C—N2C	0.1 (3)
C—N—C1A—C2A	150.83 (19)	C6C—N3C—C4C—C3C	179.7 (2)
C1C—N—C1A—C2A	−96.4 (2)	N1C—C3C—C4C—N2C	−2.0 (3)
C1B—N—C1A—C2A	38.8 (3)	N1C—C3C—C4C—N3C	178.4 (2)
C3A—N1A—C2A—C1A	98.1 (2)	C4C—N2C—C5C—C6C	−0.3 (2)
Fe—N1A—C2A—C1A	−86.8 (2)	Fe—N2C—C5C—C6C	178.98 (18)
N—C1A—C2A—N1A	73.3 (2)	N2C—C5C—C6C—N3C	0.4 (3)
C2A—N1A—C3A—C4A	−179.54 (17)	C4C—N3C—C6C—C5C	−0.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*2···O14^{i}	0.81 (2)	2.29 (4)	2.938 (3)	138 (5)
O2*W*—H2*W*1···O14^{ii}	0.82 (2)	2.26 (2)	2.987 (3)	148 (4)
O2*W*—H2*W*2···O22^{iii}	0.82 (2)	2.07 (2)	2.861 (3)	160 (4)
N3*A*—H3*AB*···O2*W*	0.88	1.91	2.730 (3)	155
N3*B*—H3*BB*···O1*W*	0.88	1.95	2.752 (3)	152
N3*C*—H3*CB*···O14	0.88	2.05	2.907 (3)	163

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