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Tris[4-(2-pyridylmethyleneamino)-phenol]iron(II) bis(perchlorate)

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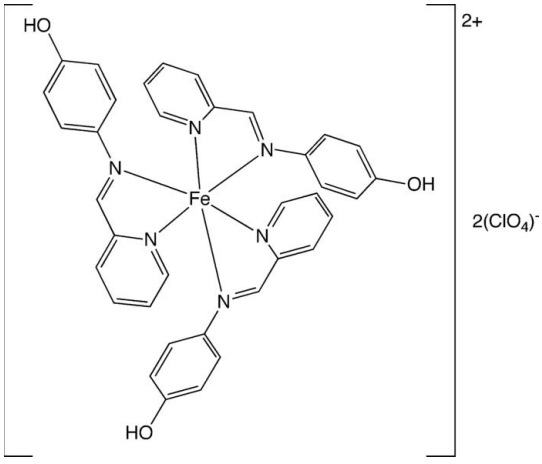
Key indicators: single-crystal X-ray study; T = 130 K; mean σ(C–C) = 0.005 Å; R factor = 0.062; wR factor = 0.115; data-to-parameter ratio = 15.6.

In the title compound, $[Fe(C_{12}H_{10}N_2O)_3](ClO_4)_2$, the metal center is coordinated by six N atoms from the three bidentate chelating ligands in a distorted octahedral coordination geometry, with overall formation of the meridional (*OC*-6-21) isomer. Intermolecular O—H···O hydrogen bonds between the hydroxyl groups of the cation and the counter-anions form an infinite one-dimensional chain in the *c*-axis direction.

Related literature

For related literature, see: Cloete & Mapolie (2006); Osman (2006); Sharma & Dubey (1994); Thankarajan & Mohanan (1986); Dash *et al.* (1983); Dhar *et al.* (2005); Golcu *et al.* (2005); Lacroxin *et al.* (2004); Shaker *et al.* (2003).



Experimental

Crystal data

$[Fe(C_{12}H_{10}N_2O)_3](ClO_4)_2$
M_r = 849.41
Monoclinic, $P2_1/c$
a = 16.201 (3) Å
b = 9.1222 (15) Å
c = 23.787 (4) Å
β = 91.089 (5)°
V = 3514.8 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 130 (2) K
0.55 × 0.07 × 0.04 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.714, T_{max} = 0.974
20409 measured reflections
7879 independent reflections
4431 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.115
S = 1.01
7879 reflections
505 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N4	1.963 (3)	Fe1—N3	1.997 (3)
Fe1—N6	1.971 (3)	Fe1—N2	2.002 (3)
Fe1—N1	1.974 (3)	Fe1—N5	2.015 (3)
N4—Fe1—N6	173.00 (12)	N1—Fe1—N2	81.04 (12)
N4—Fe1—N1	89.66 (11)	N3—Fe1—N2	172.06 (12)
N6—Fe1—N1	96.85 (12)	N4—Fe1—N5	93.27 (11)
N4—Fe1—N3	81.32 (12)	N6—Fe1—N5	80.00 (12)
N6—Fe1—N3	95.72 (11)	N1—Fe1—N5	173.71 (12)
N1—Fe1—N3	92.83 (12)	N3—Fe1—N5	82.12 (11)
N4—Fe1—N2	93.54 (11)	N2—Fe1—N5	104.31 (12)
N6—Fe1—N2	90.05 (11)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O7^{i}	0.82	2.05	2.815 (4)	155
O2—H2···O4^{ii}	0.82	1.92	2.733 (4)	174
O3—H3···O9^{iii}	0.82	1.96	2.772 (4)	172

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2231).

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supplementary materials

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Tris[4-(2-pyridylmethyleneamino)phenol]iron(II) bis(perchlorate)

M.-D. Serb, B. Calmuschi-Cula, F. Dumitru, U. Englert and C. Guran

Comment

A large number of Schiff bases and their complexes have been studied for their interesting and important properties, such as their ability to reversibly bind oxygen, their catalytic activity in the hydrogenation of olefins and their photochromic properties (Osman, 2006). The synthesis of new Schiff bases and their metal complexes are the subject of ongoing research (Golcu *et al.*, 2005; Lacroxin *et al.*, 2004; Dhar *et al.*, 2005). Similar to the ligands 2,2'-bipyridine and 1,10-phenanthroline, Schiff bases derived from 2-pyridinecarboxaldehyde provide the π-acidic α,α'-diimine fragment for metal coordination.

Very little effort has been spent in the preparation of Fe(II) Schiff base complexes (Thankarajan & Mohanan, 1986; Sharma & Dubey, 1994), despite their importance as complexes containing a metal in a low and potentially unstable oxidation state, as well as involving relatively unstable ligands, the Schiff bases (Shaker *et al.*, 2003). Such ligands are hydrolytically unstable but the metal ions stabilize the aldimine linkage to hydrolytic splitting (Dash *et al.*, 1983).

In this context, the title complex of Fe(II) with 2-pyridinecarboxaldehyde(*p*-hydroxyphenylimine) has been prepared and its crystal structure is reported here. The central Fe atom is coordinated by three ligand molecules through the nitrogen atoms in a bidentate manner. The coordination geometry is distorted octahedral. The complex crystallizes as the meridional isomer in the monoclinic centrosymmetric space group $P2_1/c$. The meridional isomer is associated with two different orientations of the bidentate ligands in a 2:1 distribution. These two different types of geometry are reflected in different dihedral angles between the aromatic and heteroaromatic rings. For the ligands which have the same orientation (N1···N2 and N3···N4 bidentate ligands) the dihedral angles are similar, *viz*. 36.72 (16) and 34.21 (16)°, respectively, whereas for the bidentate ligand N5···N6 with different orientation the angle is 50.30 (17)°. The Fe(II)—*N*(pyridine) distances are shorter than the Fe(II)—*N*(imine) bonds (see Table 1). Classical intermolecular O—H···O hydrogen bonds between the OH groups of the cation and the anions form an infinite one-dimensional chain in the c direction.

Experimental

Attempts to synthesize Fe(II) complexes with the Schiff base ligand 2-pyridinecarboxaldehyde(*p*-hydroxyphenylimine) in a molar ratio *M*:*L* 1:1 or 1:2 resulted in the same crystalline complex $[Fe(C_{12}H_{10}N_2O)_3](ClO_4)_2$, independent of reactant stoichiometry. The ligand 2-pyridinecarboxaldehyde(*p*-hydroxyphenylimine) was prepared following the procedure developed in the literature (Cloete & Mapolie, 2006). Stoichiometric amounts of $Fe(ClO_4)_2.xH_2O$ (0.255 g, 1 mmol) and 2-pyridinecarboxaldehyde(*p*-hydroxyphenylimine) (0.198 g, 1 mmol respectively 0.296 g, 2 mmol) were dissolved in acetonitrile (40 ml) and stirred under reflux to promote the complete formation of the purple complex, $[Fe(C_{12}H_{10}N_2O)_3](ClO_4)_2$. Single crystals suitable for X-ray diffraction were obtained by slow diffusion of diisopropyl ether into an acetonitrile solution of the complex at room temperature.

Refinement

H atoms were placed in calculated positions and refined using a riding model with C—H distances of 0.93 Å, O—H distances of 0.82 Å, U_{iso}(H) = 1.2U_{eq}(C), U_{iso}(H) = 1.5U_{eq}(O).

Figures

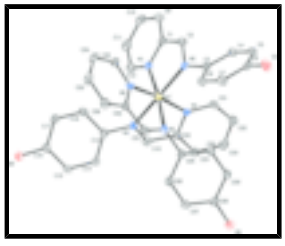


Fig. 1. Displacement ellipsoid plots (30% probability) of the cation. Hydrogen atoms and the anions have been omitted for clarity.

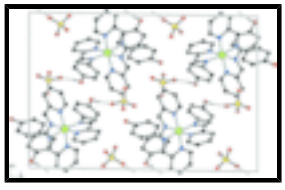


Fig. 2. The packing of the structure in the unit cell. Hydrogen bonds are indicated by dashed lines. Hydrogen atoms not involved in hydrogen bonding have been omitted.

Tris[4-(2-pyridylmethyleneamino)phenol]iron(II) bis(perchlorate)

Crystal data

$[Fe(C_{12}H_{10}N_2O)_3](ClO_4)_2$	F_{000} = 1744
M_r = 849.41	D_x = 1.605 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 16.201 (3) Å	Cell parameters from 8096 reflections
b = 9.1222 (15) Å	θ = 2.1–27.4º
c = 23.787 (4) Å	μ = 0.66 mm^{-1}
β = 91.089 (5)º	T = 130 (2) K
V = 3514.8 (10) Å^3	Rod, dark brown
Z = 4	0.55 × 0.07 × 0.04 mm

Data collection

Bruker SMART APEX CCD diffractometer	7879 independent reflections
Radiation source: fine-focus sealed tube	4431 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.081
T = 130(2) K	θ_{max} = 27.4º
ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −19→21
T_{min} = 0.714, T_{max} = 0.974	k = −11→11

20409 measured reflections

$l = -26 \rightarrow 30$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.062$

$wR(F^2) = 0.115$

$S = 1.01$

7879 reflections

505 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.034P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.60$ e Å^{-3}

$\Delta\rho_{min} = -0.53$ e Å^{-3}

Extinction correction: none

Special details

Experimental. The values of Tmin and Tmax are 0.764276 and 1.000000 from *SADABS*.

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.25204 (3)	0.69147 (6)	0.15599 (2)	0.02052 (14)
O1	0.44356 (17)	1.3193 (3)	0.27185 (11)	0.0430 (7)
H1	0.4309	1.3514	0.3027	0.065*
O2	0.30912 (16)	0.2248 (3)	−0.07599 (10)	0.0376 (7)
H2	0.3384	0.1595	−0.0628	0.056*
O3	0.03875 (16)	1.2193 (3)	0.01116 (10)	0.0379 (7)
H3	0.0218	1.2735	0.0359	0.057*
N1	0.22735 (17)	0.5285 (3)	0.20764 (12)	0.0243 (7)
N2	0.29799 (17)	0.7776 (3)	0.22739 (12)	0.0230 (7)
N3	0.19351 (18)	0.5951 (3)	0.09126 (11)	0.0221 (7)
N4	0.14110 (17)	0.7711 (3)	0.16764 (11)	0.0210 (7)
N5	0.27866 (17)	0.8410 (3)	0.09666 (11)	0.0211 (7)
N6	0.36340 (17)	0.6253 (3)	0.13568 (11)	0.0199 (7)
C1	0.2528 (2)	0.5509 (4)	0.26149 (15)	0.0238 (9)
C2	0.2939 (2)	0.6898 (4)	0.27008 (15)	0.0255 (8)
H2A	0.3165	0.7153	0.3049	0.031*

C3	0.3315 (2)	0.9198 (4)	0.23806 (14)	0.0225 (8)
C4	0.3158 (2)	0.9923 (4)	0.28827 (14)	0.0259 (9)
H4	0.2804	0.9502	0.3141	0.031*
C5	0.3519 (2)	1.1253 (4)	0.30015 (15)	0.0288 (9)
H5	0.3412	1.1723	0.3339	0.035*
C6	0.4038 (2)	1.1889 (4)	0.26232 (15)	0.0272 (9)
C7	0.4177 (2)	1.1200 (4)	0.21192 (15)	0.0317 (10)
H7	0.4516	1.1643	0.1858	0.038*
C8	0.3822 (2)	0.9875 (4)	0.19971 (15)	0.0277 (9)
H8	0.3922	0.9425	0.1654	0.033*
C9	0.2382 (2)	0.4512 (4)	0.30424 (15)	0.0284 (9)
H9	0.2572	0.4695	0.3407	0.034*
C10	0.1950 (2)	0.3248 (4)	0.29190 (16)	0.0335 (10)
H10	0.1854	0.2553	0.3196	0.040*
C11	0.1661 (2)	0.3035 (4)	0.23742 (16)	0.0333 (10)
H11	0.1354	0.2207	0.2280	0.040*
C12	0.1839 (2)	0.4077 (4)	0.19731 (15)	0.0282 (9)
H12	0.1642	0.3923	0.1608	0.034*
C13	0.2266 (2)	0.5021 (4)	0.04826 (14)	0.0228 (8)
C14	0.2054 (2)	0.5242 (4)	−0.00785 (14)	0.0278 (9)
H14	0.1714	0.6024	−0.0180	0.033*
C15	0.2342 (2)	0.4314 (4)	−0.04842 (15)	0.0323 (10)
H15	0.2198	0.4474	−0.0860	0.039*
C16	0.2843 (2)	0.3144 (4)	−0.03397 (15)	0.0290 (9)
C17	0.3091 (2)	0.2942 (4)	0.02191 (15)	0.0250 (9)
H17	0.3453	0.2189	0.0317	0.030*
C18	0.2791 (2)	0.3878 (4)	0.06279 (15)	0.0244 (9)
H18	0.2945	0.3736	0.1003	0.029*
C19	0.1181 (2)	0.6377 (4)	0.08495 (15)	0.0248 (9)
H19	0.0854	0.6040	0.0551	0.030*
C20	0.0859 (2)	0.7398 (4)	0.12569 (14)	0.0229 (8)
C21	0.1159 (2)	0.8625 (4)	0.20802 (14)	0.0247 (9)
H21	0.1509	0.8799	0.2387	0.030*
C22	0.0397 (2)	0.9321 (4)	0.20587 (15)	0.0280 (9)
H22	0.0245	0.9958	0.2344	0.034*
C23	−0.0129 (2)	0.9057 (4)	0.16097 (16)	0.0328 (10)
H23	−0.0628	0.9559	0.1576	0.039*
C24	0.0089 (2)	0.8037 (4)	0.12084 (15)	0.0304 (9)
H24	−0.0272	0.7790	0.0915	0.037*
C25	0.2220 (2)	0.9464 (4)	0.07386 (14)	0.0221 (8)
C26	0.1963 (2)	0.9394 (4)	0.01779 (14)	0.0271 (9)
H26	0.2202	0.8713	−0.0060	0.032*
C27	0.1365 (2)	1.0312 (4)	−0.00281 (15)	0.0297 (9)
H27	0.1200	1.0261	−0.0404	0.036*
C28	0.1004 (2)	1.1321 (4)	0.03266 (15)	0.0260 (9)
C29	0.1265 (2)	1.1410 (4)	0.08820 (14)	0.0238 (9)
H29	0.1029	1.2097	0.1119	0.029*
C30	0.1869 (2)	1.0497 (4)	0.10853 (15)	0.0241 (9)
H30	0.2044	1.0570	0.1459	0.029*

C31	0.3439 (2)	0.8096 (4)	0.06834 (14)	0.0248 (8)
H31	0.3575	0.8609	0.0360	0.030*
C32	0.3945 (2)	0.6907 (4)	0.08974 (14)	0.0223 (8)
C33	0.4093 (2)	0.5220 (4)	0.16004 (15)	0.0241 (9)
H33	0.3901	0.4780	0.1925	0.029*
C34	0.4844 (2)	0.4767 (4)	0.13958 (16)	0.0304 (9)
H34	0.5146	0.4042	0.1582	0.036*
C35	0.5140 (2)	0.5396 (4)	0.09150 (17)	0.0330 (10)
H35	0.5632	0.5079	0.0762	0.040*
C36	0.4691 (2)	0.6504 (4)	0.06662 (16)	0.0306 (9)
H36	0.4884	0.6975	0.0348	0.037*
Cl1	0.56386 (6)	0.08454 (11)	0.08604 (4)	0.0336 (3)
O4	0.59056 (18)	−0.0018 (3)	0.03976 (10)	0.0498 (8)
O5	0.48010 (18)	0.1187 (4)	0.08003 (13)	0.0798 (12)
O6	0.6107 (3)	0.2155 (4)	0.08746 (18)	0.1029 (14)
O7	0.58053 (19)	0.0088 (4)	0.13652 (11)	0.0648 (10)
Cl2	0.07519 (6)	0.85572 (12)	0.36151 (4)	0.0384 (3)
O8	0.10983 (19)	0.9829 (3)	0.33517 (11)	0.0603 (10)
O9	0.03283 (17)	0.9033 (3)	0.41089 (11)	0.0498 (8)
O10	0.1390 (2)	0.7602 (4)	0.37764 (16)	0.0819 (12)
O11	0.0186 (2)	0.7834 (4)	0.32467 (12)	0.0663 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0234 (3)	0.0188 (3)	0.0193 (3)	0.0015 (2)	−0.0003 (2)	−0.0005 (2)
O1	0.062 (2)	0.0289 (17)	0.0377 (16)	−0.0113 (15)	−0.0031 (14)	−0.0074 (14)
O2	0.0576 (19)	0.0300 (17)	0.0254 (15)	0.0163 (14)	0.0023 (13)	−0.0022 (12)
O3	0.0444 (17)	0.0399 (18)	0.0290 (15)	0.0151 (14)	−0.0073 (13)	−0.0052 (13)
N1	0.0273 (17)	0.0210 (18)	0.0245 (17)	0.0019 (14)	−0.0037 (14)	−0.0032 (14)
N2	0.0220 (16)	0.0204 (19)	0.0265 (17)	0.0042 (14)	−0.0002 (13)	0.0000 (14)
N3	0.0285 (18)	0.0171 (17)	0.0209 (16)	0.0005 (14)	0.0035 (13)	0.0002 (13)
N4	0.0256 (17)	0.0206 (18)	0.0167 (16)	−0.0016 (14)	0.0009 (13)	0.0017 (13)
N5	0.0257 (17)	0.0153 (17)	0.0221 (16)	−0.0029 (13)	−0.0021 (14)	−0.0002 (13)
N6	0.0227 (16)	0.0181 (17)	0.0189 (16)	−0.0026 (14)	−0.0018 (13)	−0.0004 (13)
C1	0.020 (2)	0.023 (2)	0.027 (2)	0.0055 (17)	−0.0035 (16)	0.0019 (18)
C2	0.029 (2)	0.025 (2)	0.022 (2)	−0.0014 (18)	−0.0028 (16)	0.0033 (18)
C3	0.022 (2)	0.019 (2)	0.026 (2)	0.0014 (17)	−0.0037 (16)	−0.0051 (17)
C4	0.030 (2)	0.026 (2)	0.022 (2)	0.0002 (18)	−0.0019 (17)	0.0018 (17)
C5	0.033 (2)	0.031 (2)	0.022 (2)	0.0036 (19)	−0.0038 (18)	−0.0052 (18)
C6	0.032 (2)	0.015 (2)	0.034 (2)	0.0023 (19)	−0.0095 (18)	−0.0030 (19)
C7	0.044 (3)	0.026 (2)	0.025 (2)	−0.008 (2)	0.0053 (19)	−0.0007 (18)
C8	0.033 (2)	0.026 (2)	0.025 (2)	0.0032 (19)	0.0020 (18)	−0.0030 (18)
C9	0.030 (2)	0.031 (2)	0.024 (2)	0.0003 (19)	−0.0033 (17)	0.0047 (18)
C10	0.037 (2)	0.024 (2)	0.039 (2)	0.004 (2)	0.000 (2)	0.0086 (19)
C11	0.036 (2)	0.022 (2)	0.042 (2)	−0.0021 (19)	0.0037 (19)	0.000 (2)
C12	0.035 (2)	0.021 (2)	0.029 (2)	−0.0057 (19)	−0.0014 (18)	0.0003 (18)
C13	0.029 (2)	0.015 (2)	0.024 (2)	−0.0001 (17)	−0.0013 (17)	−0.0008 (16)

C14	0.038 (2)	0.021 (2)	0.024 (2)	0.0073 (19)	−0.0034 (18)	0.0003 (17)
C15	0.046 (3)	0.034 (3)	0.017 (2)	0.011 (2)	−0.0044 (18)	−0.0031 (18)
C16	0.040 (2)	0.023 (2)	0.024 (2)	0.005 (2)	0.0053 (18)	−0.0031 (18)
C17	0.026 (2)	0.017 (2)	0.031 (2)	0.0009 (17)	−0.0008 (17)	0.0012 (18)
C18	0.033 (2)	0.020 (2)	0.020 (2)	−0.0081 (18)	−0.0031 (17)	0.0005 (17)
C19	0.026 (2)	0.023 (2)	0.025 (2)	−0.0071 (18)	−0.0004 (17)	0.0001 (17)
C20	0.026 (2)	0.019 (2)	0.024 (2)	−0.0005 (17)	0.0029 (17)	0.0037 (16)
C21	0.031 (2)	0.027 (2)	0.0156 (19)	0.0036 (18)	0.0017 (16)	0.0018 (17)
C22	0.032 (2)	0.030 (2)	0.022 (2)	0.0020 (19)	0.0089 (18)	−0.0002 (18)
C23	0.026 (2)	0.039 (3)	0.034 (2)	0.011 (2)	0.0085 (19)	0.007 (2)
C24	0.027 (2)	0.039 (3)	0.025 (2)	0.001 (2)	−0.0051 (17)	0.000 (2)
C25	0.026 (2)	0.014 (2)	0.026 (2)	−0.0007 (16)	−0.0010 (17)	0.0020 (16)
C26	0.037 (2)	0.020 (2)	0.024 (2)	0.0028 (18)	0.0030 (18)	0.0002 (17)
C27	0.040 (2)	0.026 (2)	0.023 (2)	0.0027 (19)	−0.0034 (18)	−0.0015 (18)
C28	0.027 (2)	0.021 (2)	0.029 (2)	0.0016 (18)	−0.0024 (17)	0.0016 (18)
C29	0.027 (2)	0.018 (2)	0.027 (2)	−0.0040 (17)	0.0034 (17)	−0.0047 (17)
C30	0.027 (2)	0.021 (2)	0.024 (2)	−0.0014 (17)	−0.0031 (17)	−0.0017 (17)
C31	0.029 (2)	0.024 (2)	0.0212 (19)	−0.0061 (19)	0.0044 (17)	−0.0025 (18)
C32	0.0245 (19)	0.017 (2)	0.025 (2)	0.0007 (17)	−0.0034 (16)	−0.0037 (17)
C33	0.026 (2)	0.019 (2)	0.027 (2)	0.0009 (17)	−0.0038 (17)	0.0027 (17)
C34	0.027 (2)	0.019 (2)	0.045 (3)	0.0058 (18)	−0.0073 (19)	0.0015 (19)
C35	0.025 (2)	0.024 (2)	0.051 (3)	0.0050 (19)	0.0047 (19)	−0.004 (2)
C36	0.031 (2)	0.029 (2)	0.032 (2)	−0.0013 (19)	0.0080 (18)	−0.0022 (19)
Cl1	0.0392 (6)	0.0342 (6)	0.0273 (5)	0.0127 (5)	0.0016 (4)	−0.0004 (5)
O4	0.078 (2)	0.0455 (19)	0.0267 (16)	0.0280 (17)	0.0097 (15)	−0.0017 (14)
O5	0.046 (2)	0.139 (4)	0.054 (2)	0.047 (2)	−0.0139 (17)	−0.024 (2)
O6	0.124 (4)	0.036 (2)	0.149 (4)	−0.007 (2)	0.015 (3)	−0.023 (2)
O7	0.074 (2)	0.089 (3)	0.0317 (18)	0.036 (2)	0.0095 (16)	0.0198 (18)
Cl2	0.0365 (6)	0.0503 (7)	0.0283 (6)	−0.0131 (5)	−0.0019 (5)	−0.0016 (5)
O8	0.078 (2)	0.069 (2)	0.0346 (17)	−0.0477 (19)	0.0098 (16)	−0.0038 (16)
O9	0.056 (2)	0.048 (2)	0.0459 (18)	−0.0090 (16)	0.0241 (16)	0.0002 (15)
O10	0.064 (2)	0.084 (3)	0.097 (3)	0.037 (2)	−0.015 (2)	−0.024 (2)
O11	0.077 (2)	0.083 (3)	0.0391 (18)	−0.052 (2)	−0.0175 (16)	0.0084 (17)

Geometric parameters (Å, °)

Fe1—N4	1.963 (3)	C14—C15	1.372 (5)
Fe1—N6	1.971 (3)	C14—H14	0.9300
Fe1—N1	1.974 (3)	C15—C16	1.380 (5)
Fe1—N3	1.997 (3)	C15—H15	0.9300
Fe1—N2	2.002 (3)	C16—C17	1.394 (5)
Fe1—N5	2.015 (3)	C17—C18	1.388 (5)
O1—C6	1.370 (4)	C17—H17	0.9300
O1—H1	0.8200	C18—H18	0.9300
O2—C16	1.358 (4)	C19—C20	1.449 (5)
O2—H2	0.8200	C19—H19	0.9300
O3—C28	1.369 (4)	C20—C24	1.378 (5)
O3—H3	0.8200	C21—C22	1.388 (5)
N1—C12	1.328 (4)	C21—H21	0.9300

N1—C1	1.354 (4)	C22—C23	1.375 (5)
N2—C2	1.296 (4)	C22—H22	0.9300
N2—C3	1.426 (4)	C23—C24	1.384 (5)
N3—C19	1.288 (4)	C23—H23	0.9300
N3—C13	1.440 (4)	C24—H24	0.9300
N4—C21	1.341 (4)	C25—C30	1.382 (5)
N4—C20	1.358 (4)	C25—C26	1.391 (5)
N5—C31	1.296 (4)	C26—C27	1.365 (5)
N5—C25	1.429 (4)	C26—H26	0.9300
N6—C33	1.327 (4)	C27—C28	1.385 (5)
N6—C32	1.351 (4)	C27—H27	0.9300
C1—C9	1.388 (5)	C28—C29	1.382 (5)
C1—C2	1.444 (5)	C29—C30	1.366 (5)
C2—H2A	0.9300	C29—H29	0.9300
C3—C8	1.385 (5)	C30—H30	0.9300
C3—C4	1.393 (5)	C31—C32	1.446 (5)
C4—C5	1.374 (5)	C31—H31	0.9300
C4—H4	0.9300	C32—C36	1.387 (5)
C5—C6	1.371 (5)	C33—C34	1.382 (5)
C5—H5	0.9300	C33—H33	0.9300
C6—C7	1.376 (5)	C34—C35	1.374 (5)
C7—C8	1.368 (5)	C34—H34	0.9300
C7—H7	0.9300	C35—C36	1.372 (5)
C8—H8	0.9300	C35—H35	0.9300
C9—C10	1.378 (5)	C36—H36	0.9300
C9—H9	0.9300	Cl1—O5	1.397 (3)
C10—C11	1.383 (5)	Cl1—O7	1.407 (3)
C10—H10	0.9300	Cl1—O6	1.416 (4)
C11—C12	1.381 (5)	Cl1—O4	1.427 (3)
C11—H11	0.9300	Cl2—O10	1.400 (3)
C12—H12	0.9300	Cl2—O11	1.418 (3)
C13—C18	1.385 (5)	Cl2—O8	1.438 (3)
C13—C14	1.387 (5)	Cl2—O9	1.439 (3)
N4—Fe1—N6	173.00 (12)	C14—C15—C16	120.6 (3)
N4—Fe1—N1	89.66 (11)	C14—C15—H15	119.7
N6—Fe1—N1	96.85 (12)	C16—C15—H15	119.7
N4—Fe1—N3	81.32 (12)	O2—C16—C15	117.6 (3)
N6—Fe1—N3	95.72 (11)	O2—C16—C17	122.6 (3)
N1—Fe1—N3	92.83 (12)	C15—C16—C17	119.8 (3)
N4—Fe1—N2	93.54 (11)	C18—C17—C16	119.2 (3)
N6—Fe1—N2	90.05 (11)	C18—C17—H17	120.4
N1—Fe1—N2	81.04 (12)	C16—C17—H17	120.4
N3—Fe1—N2	172.06 (12)	C13—C18—C17	120.6 (3)
N4—Fe1—N5	93.27 (11)	C13—C18—H18	119.7
N6—Fe1—N5	80.00 (12)	C17—C18—H18	119.7
N1—Fe1—N5	173.71 (12)	N3—C19—C20	118.0 (3)
N3—Fe1—N5	82.12 (11)	N3—C19—H19	121.0
N2—Fe1—N5	104.31 (12)	C20—C19—H19	121.0
C6—O1—H1	109.5	N4—C20—C24	123.7 (3)

C16—O2—H2	109.5	N4—C20—C19	112.7 (3)
C28—O3—H3	109.5	C24—C20—C19	123.6 (3)
C12—N1—C1	116.8 (3)	N4—C21—C22	122.7 (3)
C12—N1—Fe1	128.5 (2)	N4—C21—H21	118.6
C1—N1—Fe1	114.4 (2)	C22—C21—H21	118.6
C2—N2—C3	116.6 (3)	C23—C22—C21	119.1 (3)
C2—N2—Fe1	113.5 (2)	C23—C22—H22	120.4
C3—N2—Fe1	129.9 (2)	C21—C22—H22	120.4
C19—N3—C13	117.4 (3)	C22—C23—C24	119.3 (3)
C19—N3—Fe1	113.1 (2)	C22—C23—H23	120.3
C13—N3—Fe1	129.0 (2)	C24—C23—H23	120.3
C21—N4—C20	116.8 (3)	C20—C24—C23	118.0 (3)
C21—N4—Fe1	128.5 (2)	C20—C24—H24	121.0
C20—N4—Fe1	114.3 (2)	C23—C24—H24	121.0
C31—N5—C25	118.4 (3)	C30—C25—C26	118.9 (3)
C31—N5—Fe1	113.7 (2)	C30—C25—N5	120.1 (3)
C25—N5—Fe1	125.2 (2)	C26—C25—N5	120.8 (3)
C33—N6—C32	116.9 (3)	C27—C26—C25	120.9 (3)
C33—N6—Fe1	128.2 (2)	C27—C26—H26	119.5
C32—N6—Fe1	114.9 (2)	C25—C26—H26	119.5
N1—C1—C9	122.8 (3)	C26—C27—C28	119.6 (3)
N1—C1—C2	113.4 (3)	C26—C27—H27	120.2
C9—C1—C2	123.8 (3)	C28—C27—H27	120.2
N2—C2—C1	117.5 (3)	O3—C28—C29	122.1 (3)
N2—C2—H2A	121.2	O3—C28—C27	118.2 (3)
C1—C2—H2A	121.2	C29—C28—C27	119.8 (3)
C8—C3—C4	118.2 (3)	C30—C29—C28	120.4 (3)
C8—C3—N2	121.1 (3)	C30—C29—H29	119.8
C4—C3—N2	120.6 (3)	C28—C29—H29	119.8
C5—C4—C3	120.8 (4)	C29—C30—C25	120.3 (3)
C5—C4—H4	119.6	C29—C30—H30	119.8
C3—C4—H4	119.6	C25—C30—H30	119.8
C6—C5—C4	120.1 (4)	N5—C31—C32	116.4 (3)
C6—C5—H5	119.9	N5—C31—H31	121.8
C4—C5—H5	119.9	C32—C31—H31	121.8
O1—C6—C5	123.4 (3)	N6—C32—C36	123.0 (3)
O1—C6—C7	117.2 (3)	N6—C32—C31	113.5 (3)
C5—C6—C7	119.4 (4)	C36—C32—C31	123.5 (3)
C8—C7—C6	120.9 (4)	N6—C33—C34	123.3 (3)
C8—C7—H7	119.5	N6—C33—H33	118.4
C6—C7—H7	119.5	C34—C33—H33	118.4
C7—C8—C3	120.4 (3)	C35—C34—C33	119.4 (4)
C7—C8—H8	119.8	C35—C34—H34	120.3
C3—C8—H8	119.8	C33—C34—H34	120.3
C10—C9—C1	119.0 (3)	C36—C35—C34	118.5 (4)
C10—C9—H9	120.5	C36—C35—H35	120.8
C1—C9—H9	120.5	C34—C35—H35	120.8
C9—C10—C11	118.6 (4)	C35—C36—C32	118.9 (4)
C9—C10—H10	120.7	C35—C36—H36	120.6

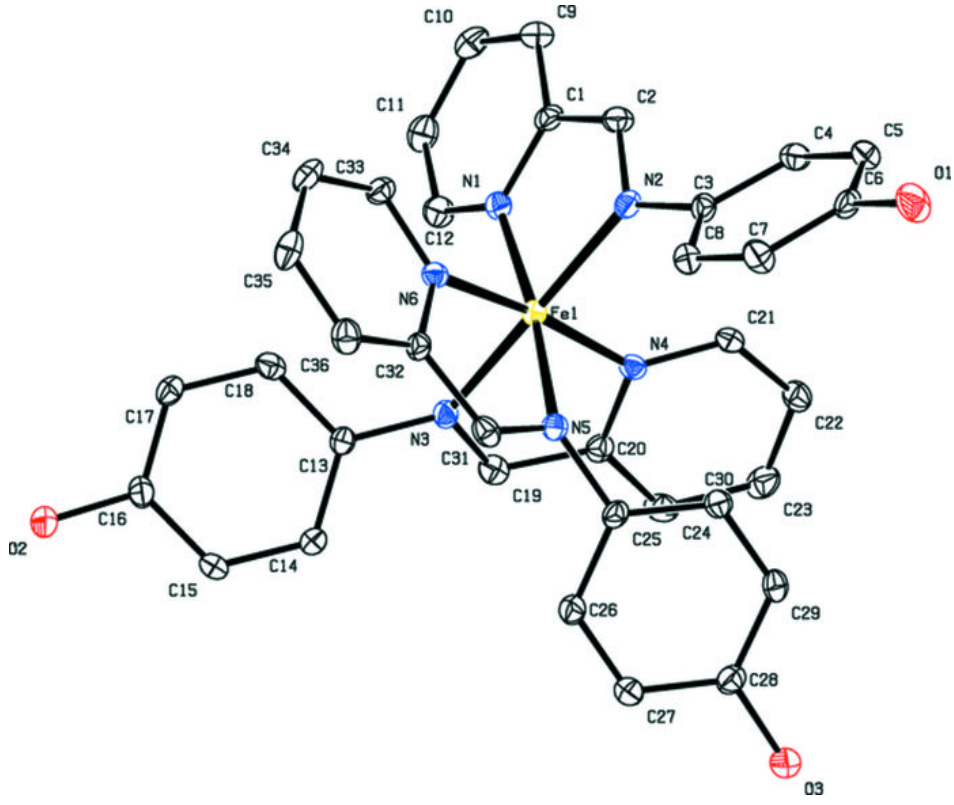
C11—C10—H10	120.7	C32—C36—H36	120.6
C12—C11—C10	118.7 (4)	O5—Cl1—O7	111.5 (2)
C12—C11—H11	120.7	O5—Cl1—O6	109.5 (2)
C10—C11—H11	120.7	O7—Cl1—O6	107.4 (2)
N1—C12—C11	124.1 (4)	O5—Cl1—O4	110.62 (19)
N1—C12—H12	118.0	O7—Cl1—O4	109.36 (17)
C11—C12—H12	118.0	O6—Cl1—O4	108.3 (2)
C18—C13—C14	119.3 (3)	O10—Cl2—O11	110.2 (2)
C18—C13—N3	120.1 (3)	O10—Cl2—O8	109.2 (2)
C14—C13—N3	120.6 (3)	O11—Cl2—O8	111.05 (18)
C15—C14—C13	120.4 (3)	O10—Cl2—O9	109.0 (2)
C15—C14—H14	119.8	O11—Cl2—O9	109.40 (19)
C13—C14—H14	119.8	O8—Cl2—O9	107.96 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O7^{i}	0.82	2.05	2.815 (4)	155
O2—H2···O4^{ii}	0.82	1.92	2.733 (4)	174
O3—H3···O9^{iii}	0.82	1.96	2.772 (4)	172

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

Fig. 1


C10
C9
C11
C1
C2
C34
C33
C4
C5
N1
N2
C3
C6
O1
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C7
C35
N6
Fe1
C21
N4
C36
C32
C18
C17
C22
N3
N5
C20
C13
C31
C19
C30
C23
C16
C25
C24
O2
C14
C15
C26
C29
C28
C27
O3

Fig. 2