Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

{1,4-Bis[3-(3-methoxy-2-oxidobenzylideneamino)propyl]piperazine}iron(III) tetrafluoridoborate

Ray J. Butcher,[a]* Mohamad Pourian[a] and Jerry P. Jasinski[b]

[a]Department of Chemistry, Howard University, Washington, DC 20059, USA, and [b]Department of Chemistry, Keene State University, Keene, New Hampshire, USA
Correspondence e-mail: rbutcher99@yahoo.com

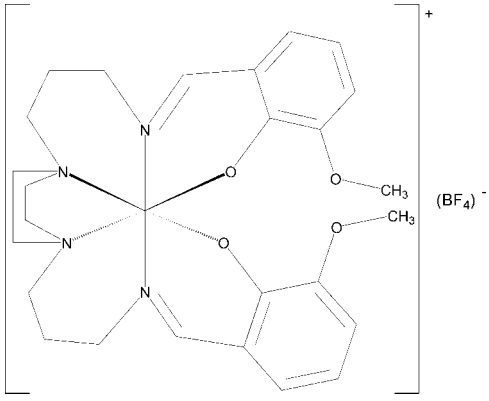
Received 8 June 2007; accepted 10 June 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; disorder in solvent or counterion; R factor = 0.053; wR factor = 0.144; data-to-parameter ratio = 12.4.

The title compound, $[Fe(C_{26}H_{34}N_4O_4)]BF_4$, shows a very distorted octahedral geometry about the Fe atom of the cation. This is due to the small bite angle of the piperazine ring, which adopts a boat conformation. The structure also displays C—H···F hydrogen bonding. Disorder with site occupancies of 0.53 (2) and 0.47 (2) is found for three of the tetrafluoridoborate F atoms.

Related literature

For related literature, see: Das Sarma & Bailar (1955); Dwyer & Lions (1947); Sinn *et al.* (1978); Yisgedu *et al.* (2007).



Experimental

Crystal data

$[Fe(C_{26}H_{34}N_4O_4)]BF_4$
M_r = 609.23
Monoclinic, $P2_1/c$
a = 18.809 (4) Å
b = 10.801 (2) Å
c = 13.559 (3) Å
β = 92.89 (3)°
V = 2751.1 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.62 mm^{-1}
T = 293 (2) K
0.48 × 0.34 × 0.28 mm

Data collection

Bruker $P4$ diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.767, T_{max} = 0.875
5070 measured reflections
4843 independent reflections
2835 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.144
S = 1.02
4843 reflections
392 parameters
19 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe—O1*A*	1.887 (3)	Fe—N1*A*	2.172 (4)
Fe—O1*B*	1.908 (3)	Fe—N2*B*	2.209 (4)
Fe—N1*B*	2.166 (4)	Fe—N2*A*	2.218 (4)
O1*A*—Fe—O1*B*	127.67 (14)	N1*B*—Fe—N2*B*	80.13 (15)
O1*A*—Fe—N1*B*	85.93 (14)	N1*A*—Fe—N2*B*	121.53 (14)
O1*B*—Fe—N1*B*	83.57 (14)	O1*A*—Fe—N2*A*	138.21 (14)
O1*A*—Fe—N1*A*	83.83 (13)	O1*B*—Fe—N2*A*	89.52 (14)
O1*B*—Fe—N1*A*	85.37 (14)	N1*B*—Fe—N2*A*	120.61 (15)
N1*B*—Fe—N1*A*	155.69 (14)	N1*A*—Fe—N2*A*	80.78 (14)
O1*A*—Fe—N2*B*	88.88 (14)	N2*B*—Fe—N2*A*	67.37 (15)
O1*B*—Fe—N2*B*	138.58 (13)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9*A*—H9*AA*···F4^{i}	0.97	2.51	3.464 (14)	170
C9*A*—H9*AA*···F2*A*^{i}	0.97	2.54	3.442 (14)	155
C12*A*—H12*B*···F4^{i}	0.97	2.46	3.275 (11)	141
C12*A*—H12*B*···F2*A*^{i}	0.97	2.30	3.252 (10)	166
C12*B*—H12*D*···F3^{ii}	0.97	2.35	3.279 (8)	160
C9*B*—H9*BB*···F3*A*^{ii}	0.97	2.21	3.119 (9)	155

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+1, -y+1, -z+1$.

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

RJB acknowledges the Laboratory for the Structure of Matter at the Naval Research Laboratory for access to their diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2037).

References

Bruker (1997). *XSCANS*. Version 2.20. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2000). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
Das Sarma, B. & Bailar, J. C. Jr (1955). *J. Am. Chem. Soc.* **77**, 5476–5480.
Dwyer, F. P. J. & Lions, F. (1947). *J. Am. Chem. Soc.* **69**, 2917–2918.

 doi:10.1107/S1600536807028413

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sinn, E., Sim, G., Dose, E. V., Tweedle, M. F. & Wilson, L. J. (1978). *J. Am. Chem. Soc.* **100**, 3375–3390.
Yisgedu, T. B., Tesema, Y. T., Gultneh, Y. & Butcher, R. J. (2007). *J. Chem. Crystallogr.* In the press.

supplementary materials

Acta Cryst. (2007). E**63**, m1913-m1914 [**doi:10.1107/S1600536807028413**]

{1,4-Bis[3-(3-methoxy-2-oxidobenzylideneamino)propyl]piperazine}iron(III) tetrafluoridoborate

R. J. Butcher, M. Pourian and J. P. Jasinski

Comment

Metal complexes of hexadentate ligands have fascinated inorganic chemists since their first report in 1947 (Dwyer & Lions, 1947). The first such report of a Fe complex of a linear FeN_4O_2 ligand (*i.e.* where the donor atoms are linked in a linear chain) derived from the Schiff base condensation of salicylaldehyde and triethylenetetraamine was in 1955 (Das Sarma & Bailar, 1955). However, this interest lapsed for several years until the discovery that some of these complexes exhibited spin-crossover magnetic behavior (Sinn *et al.*, 1978). While there have been numerous reports of complexes with a FeN_4O_2 chromophore based on Schiff base condensations of tetramines with derivatives of salicylaldehyde, there have been no reports of such complexes based on the tetramine, bis(3-aminopropyl)piperazine.

The title compound, bis(2-hydroxy-3-methoxybenzyl)-3-aminopropylpiperazineiron(III) tetrafluoroborate (I), resulted from mixing iron(II) tetrafluoroborate with the product resulting from the Schiff base condensation of bis(3-aminopropyl)piperazine with 3-methoxysalicylaldehyde. The cation contains a six-coordinate FeN_4O_2 chromophore exhibiting markedly distorted octahedral geometry. This distortion is most noticeable in the fact that the largest *trans* angle is only 155.73 (14)°. The main factor in this unusual geometry is the small bite angle subtended by the central piperazine ring where the distance between the two N's is only 2.454 (5) Å. The piperazine ring has adopted a chair conformation so that both N donors can coordinate to the Fe. There are also weak C—H···F interactions between the BF_4 anion and the cation.

Experimental

To 4.0 g (20 mmol) of bis(3-aminopropyl)piperazine in 15 ml of ethanol was added drop-wise 6.0 g (40 mmol) of 3-methoxysalicylaldehyde in 10 ml of ethanol. The deep yellow solution was left to stir for half an hour and a crystalline product resulted (H_2L). To 0.85 g of H_2L dissolved in 10.0 ml of methanol was added 0.58 g of $Fe(BF_4)_2.xH_2O$. The solution became violet and a red-purple solid precipitated. This was stirred overnight, the solid filtered, washed with methanol and dried to give 1.65 g of a red powder. Crystallization was effected by slow evaporation of a DMF solution of the complex.

Refinement

The structure contains a disordered BF_4 anion with one unique F and the remaining three F atoms being disordered over two positions with multiplicities of 0.53 (2) and 0.47 (2). These were refined anisotropically in idealized geometry with the group B—F distances restrained. The H atoms were idealized with C—H distances of 0.93 (aromatic C—H), 0.96 (CH_3), and 0.97 (CH_2) Å and $U_{iso}(H) = 1.2U_{eq}(C)$ ($1.5U_{eq}(C)$ for the CH_3 protons).

Figures

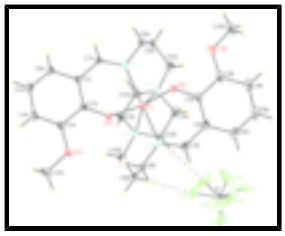


Fig. 1. The cation and anion with numbering scheme used. The disorder in the BF_4 anion is shown. Ellipsoids are drawn at the 20% probabilty level.

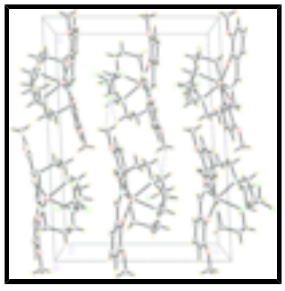


Fig. 2. The packing arrangement viewed down the *b* axis showing the strongest C—H···F interactions. Only the major component for the disordered BF_4 anion is shown.

{1,4-Bis[3-(3-methoxy-2-oxidobenzylideneamino)propyl]piperazine}iron(III) tetrafluoridoborate

Crystal data

$[Fe(C_{26}H_{34}N_4O_4)]BF_4$	F_{000} = 1268
M_r = 609.23	D_x = 1.471 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 18.809 (4) Å	Cell parameters from 45 reflections
b = 10.801 (2) Å	θ = 3.1–28.5º
c = 13.559 (3) Å	μ = 0.62 mm^{-1}
β = 92.89 (3)º	T = 293 (2) K
V = 2751.1 (10) Å^3	Prism, black
Z = 4	0.48 × 0.34 × 0.28 mm

Data collection

Bruker P4 diffractometer	R_{int} = 0.050
Radiation source: fine-focus sealed tube	θ_{max} = 25.0º
Monochromator: graphite	θ_{min} = 1.1º
T = 293(2) K	h = −22→22
ω scans	k = −12→0
Absorption correction: empirical (using intensity measurements) via ψ scan (North *et al.*, 1968)	l = 0→16
T_{min} = 0.767, T_{max} = 0.875	3 standard reflections
5070 measured reflections	every 97 reflections
4843 independent reflections	intensity decay: none
2835 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.053$	H-atom parameters constrained
$wR(F^2) = 0.144$	$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.7519P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4843 reflections	$\Delta\rho_{max} = 0.37$ e Å^{-3}
392 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
19 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
Fe	0.75426 (3)	0.23350 (6)	0.54586 (5)	0.0410 (2)	
F1	0.2266 (2)	0.1808 (4)	0.7165 (3)	0.1343 (17)	
F2	0.2255 (7)	0.2224 (13)	0.5597 (6)	0.155 (7)	0.524 (17)
F3	0.2569 (9)	0.3678 (5)	0.6634 (7)	0.163 (9)	0.524 (17)
F4	0.3229 (4)	0.2095 (8)	0.6468 (11)	0.140 (7)	0.524 (17)
F2A	0.2684 (10)	0.2029 (8)	0.5647 (6)	0.150 (10)	0.476 (17)
F3A	0.2095 (6)	0.3473 (10)	0.6327 (11)	0.135 (7)	0.476 (17)
F4A	0.3131 (6)	0.3034 (17)	0.6905 (10)	0.286 (16)	0.476 (17)
O1A	0.84521 (15)	0.1910 (3)	0.6005 (2)	0.0513 (8)	
O3A	0.98485 (17)	0.1991 (4)	0.6097 (3)	0.0761 (11)	
O1B	0.67485 (16)	0.2943 (3)	0.6135 (2)	0.0519 (8)	
O3B	0.5410 (2)	0.2911 (5)	0.6538 (3)	0.0848 (12)	
N1A	0.72422 (19)	0.0531 (3)	0.6011 (3)	0.0455 (9)	
N2A	0.6808 (2)	0.1742 (4)	0.4217 (3)	0.0530 (10)	
N1B	0.7902 (2)	0.4237 (4)	0.5574 (3)	0.0537 (10)	
N2B	0.8012 (2)	0.2548 (4)	0.4007 (3)	0.0521 (10)	
C1A	0.8439 (2)	−0.0262 (4)	0.6336 (3)	0.0472 (11)	

C2A	0.8795 (2)	0.0860 (4)	0.6198 (3)	0.0468 (11)
C3A	0.9550 (3)	0.0874 (5)	0.6274 (4)	0.0581 (13)
C31A	1.0605 (3)	0.2023 (6)	0.6043 (5)	0.094 (2)
H31D	1.0756	0.2857	0.5929	0.141*
H31E	1.0744	0.1503	0.5511	0.141*
H31F	1.0824	0.1728	0.6654	0.141*
C4A	0.9916 (3)	−0.0192 (6)	0.6512 (4)	0.0750 (17)
H4AA	1.0410	−0.0182	0.6564	0.090*
C5A	0.9553 (3)	−0.1283 (6)	0.6675 (5)	0.088 (2)
H5AA	0.9807	−0.1996	0.6846	0.105*
C6A	0.8842 (3)	−0.1322 (5)	0.6591 (4)	0.0735 (16)
H6AA	0.8610	−0.2065	0.6703	0.088*
C7A	0.7680 (2)	−0.0325 (4)	0.6278 (3)	0.0491 (12)
H7AA	0.7481	−0.1076	0.6455	0.059*
C8A	0.6483 (3)	0.0259 (5)	0.6075 (4)	0.0596 (14)
H8AA	0.6262	0.0919	0.6437	0.071*
H8AB	0.6430	−0.0503	0.6443	0.071*
C9A	0.6100 (3)	0.0128 (5)	0.5074 (4)	0.0708 (16)
H9AA	0.6322	−0.0536	0.4719	0.085*
H9AB	0.5611	−0.0115	0.5170	0.085*
C10A	0.6092 (3)	0.1273 (5)	0.4442 (4)	0.0702 (16)
H10A	0.5828	0.1097	0.3825	0.084*
H10B	0.5840	0.1922	0.4775	0.084*
C11A	0.6751 (3)	0.2905 (5)	0.3651 (4)	0.0645 (15)
H11A	0.6540	0.3545	0.4043	0.077*
H11B	0.6451	0.2784	0.3055	0.077*
C12A	0.7203 (3)	0.0841 (5)	0.3636 (4)	0.0644 (15)
H12A	0.7001	0.0804	0.2964	0.077*
H12B	0.7174	0.0022	0.3926	0.077*
C1B	0.6745 (3)	0.5121 (5)	0.5857 (4)	0.0578 (14)
C2B	0.6411 (3)	0.4020 (5)	0.6086 (3)	0.0515 (12)
C3B	0.5686 (3)	0.4036 (6)	0.6302 (4)	0.0667 (15)
C31B	0.4673 (3)	0.2838 (8)	0.6710 (5)	0.110 (3)
H31A	0.4540	0.1987	0.6786	0.166*
H31B	0.4403	0.3192	0.6160	0.166*
H31C	0.4578	0.3287	0.7301	0.166*
C4B	0.5312 (4)	0.5146 (7)	0.6261 (4)	0.089 (2)
H4BA	0.4832	0.5158	0.6398	0.107*
C5B	0.5649 (4)	0.6229 (7)	0.6019 (5)	0.096 (2)
H5BA	0.5392	0.6965	0.5982	0.115*
C6B	0.6355 (4)	0.6234 (6)	0.5835 (4)	0.0801 (18)
H6BA	0.6579	0.6976	0.5693	0.096*
C7B	0.7493 (3)	0.5168 (5)	0.5694 (4)	0.0623 (15)
H7BA	0.7700	0.5949	0.5674	0.075*
C8B	0.8657 (3)	0.4485 (5)	0.5431 (5)	0.0720 (16)
H8BA	0.8944	0.3938	0.5855	0.086*
H8BB	0.8765	0.5329	0.5630	0.086*
C9B	0.8860 (3)	0.4308 (5)	0.4384 (5)	0.0782 (17)
H9BA	0.9359	0.4522	0.4343	0.094*

H9BB	0.8587	0.4884	0.3967	0.094*
C10B	0.8747 (3)	0.3013 (5)	0.3972 (4)	0.0702 (16)
H10C	0.8883	0.3005	0.3291	0.084*
H10D	0.9062	0.2449	0.4340	0.084*
C11B	0.7971 (3)	0.1263 (5)	0.3642 (4)	0.0606 (14)
H11C	0.8266	0.0729	0.4068	0.073*
H11D	0.8142	0.1220	0.2980	0.073*
C12B	0.7496 (3)	0.3288 (5)	0.3385 (4)	0.0646 (15)
H12C	0.7562	0.3131	0.2691	0.078*
H12D	0.7568	0.4165	0.3510	0.078*
B	0.2557 (3)	0.2526 (5)	0.6480 (4)	0.0686 (17)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe	0.0385 (3)	0.0446 (4)	0.0395 (3)	0.0059 (3)	−0.0019 (2)	−0.0004 (3)
F1	0.174 (5)	0.114 (3)	0.118 (3)	0.046 (3)	0.038 (3)	0.024 (3)
F2	0.161 (11)	0.201 (17)	0.097 (8)	−0.024 (10)	−0.040 (7)	0.008 (8)
F3	0.36 (3)	0.044 (5)	0.079 (6)	0.052 (9)	−0.016 (12)	−0.017 (4)
F4	0.063 (6)	0.096 (7)	0.26 (2)	−0.002 (4)	0.031 (7)	0.004 (8)
F2A	0.34 (3)	0.045 (6)	0.064 (7)	0.048 (10)	0.025 (10)	−0.016 (4)
F3A	0.120 (10)	0.113 (10)	0.173 (15)	0.054 (7)	0.019 (8)	0.049 (10)
F4A	0.068 (9)	0.56 (5)	0.221 (16)	−0.011 (16)	−0.022 (9)	−0.16 (2)
O1A	0.0378 (17)	0.0477 (19)	0.067 (2)	0.0017 (15)	−0.0096 (15)	0.0077 (16)
O3A	0.038 (2)	0.080 (3)	0.109 (3)	−0.0077 (18)	−0.0039 (19)	−0.001 (2)
O1B	0.0497 (19)	0.054 (2)	0.0520 (19)	0.0101 (16)	0.0054 (15)	0.0015 (15)
O3B	0.051 (2)	0.121 (4)	0.083 (3)	0.021 (2)	0.014 (2)	0.007 (3)
N1A	0.040 (2)	0.050 (2)	0.047 (2)	0.0032 (18)	−0.0024 (18)	0.0022 (19)
N2A	0.057 (3)	0.058 (3)	0.043 (2)	0.016 (2)	−0.0102 (19)	−0.005 (2)
N1B	0.057 (3)	0.045 (2)	0.059 (3)	0.004 (2)	0.000 (2)	−0.001 (2)
N2B	0.059 (2)	0.051 (2)	0.047 (2)	0.018 (2)	0.0114 (18)	0.005 (2)
C1A	0.042 (3)	0.053 (3)	0.047 (3)	0.010 (2)	0.006 (2)	0.010 (2)
C2A	0.043 (3)	0.059 (3)	0.039 (3)	0.005 (2)	0.000 (2)	0.003 (2)
C3A	0.042 (3)	0.075 (4)	0.057 (3)	0.005 (3)	0.001 (2)	0.007 (3)
C31A	0.047 (3)	0.115 (5)	0.119 (5)	−0.013 (3)	0.000 (3)	−0.010 (4)
C4A	0.040 (3)	0.106 (5)	0.079 (4)	0.023 (3)	0.006 (3)	0.024 (4)
C5A	0.069 (4)	0.085 (5)	0.110 (5)	0.035 (4)	0.020 (4)	0.039 (4)
C6A	0.061 (4)	0.067 (4)	0.095 (4)	0.018 (3)	0.019 (3)	0.021 (3)
C7A	0.051 (3)	0.048 (3)	0.049 (3)	−0.001 (2)	0.003 (2)	0.005 (2)
C8A	0.044 (3)	0.062 (3)	0.073 (4)	−0.008 (2)	0.004 (3)	0.005 (3)
C9A	0.045 (3)	0.076 (4)	0.090 (4)	−0.013 (3)	−0.007 (3)	−0.007 (3)
C10A	0.054 (3)	0.083 (4)	0.071 (4)	0.009 (3)	−0.025 (3)	−0.014 (3)
C11A	0.082 (4)	0.064 (4)	0.045 (3)	0.030 (3)	−0.013 (3)	0.000 (3)
C12A	0.079 (4)	0.063 (3)	0.051 (3)	0.017 (3)	−0.005 (3)	−0.010 (3)
C1B	0.075 (4)	0.050 (3)	0.047 (3)	0.019 (3)	−0.010 (3)	−0.011 (2)
C2B	0.053 (3)	0.065 (3)	0.036 (3)	0.021 (3)	−0.004 (2)	−0.009 (2)
C3B	0.057 (3)	0.094 (5)	0.049 (3)	0.028 (3)	−0.003 (3)	−0.008 (3)
C31B	0.059 (4)	0.184 (8)	0.089 (5)	0.020 (5)	0.012 (3)	0.030 (5)

C4B	0.079 (5)	0.126 (6)	0.062 (4)	0.056 (5)	0.001 (3)	−0.008 (4)
C5B	0.108 (6)	0.092 (5)	0.087 (5)	0.060 (5)	−0.007 (4)	−0.018 (4)
C6B	0.094 (5)	0.065 (4)	0.081 (4)	0.032 (3)	−0.007 (4)	−0.016 (3)
C7B	0.078 (4)	0.044 (3)	0.064 (4)	0.008 (3)	−0.011 (3)	−0.012 (3)
C8B	0.061 (4)	0.052 (3)	0.103 (5)	−0.008 (3)	0.006 (3)	0.001 (3)
C9B	0.062 (4)	0.077 (4)	0.097 (5)	−0.004 (3)	0.018 (3)	0.022 (4)
C10B	0.069 (4)	0.075 (4)	0.069 (4)	0.008 (3)	0.025 (3)	0.011 (3)
C11B	0.078 (4)	0.056 (3)	0.049 (3)	0.023 (3)	0.013 (3)	−0.002 (3)
C12B	0.088 (4)	0.063 (3)	0.043 (3)	0.022 (3)	−0.001 (3)	0.005 (3)
B	0.068 (4)	0.066 (5)	0.073 (4)	0.011 (4)	0.007 (3)	−0.005 (4)

Geometric parameters (Å, °)

Fe—O1A	1.887 (3)	C7A—H7AA	0.9300
Fe—O1B	1.908 (3)	C8A—C9A	1.511 (7)
Fe—N1B	2.166 (4)	C8A—H8AA	0.9700
Fe—N1A	2.172 (4)	C8A—H8AB	0.9700
Fe—N2B	2.209 (4)	C9A—C10A	1.504 (7)
Fe—N2A	2.218 (4)	C9A—H9AA	0.9700
F1—B	1.347 (6)	C9A—H9AB	0.9700
F2—B	1.339 (8)	C10A—H10A	0.9700
F3—B	1.261 (7)	C10A—H10B	0.9700
F4—B	1.349 (7)	C11A—C12B	1.522 (7)
F2A—B	1.284 (7)	C11A—H11A	0.9700
F3A—B	1.351 (8)	C11A—H11B	0.9700
F4A—B	1.317 (8)	C12A—C11B	1.514 (7)
O1A—C2A	1.324 (5)	C12A—H12A	0.9700
O3A—C3A	1.357 (6)	C12A—H12B	0.9700
O3A—C31A	1.429 (6)	C1B—C2B	1.388 (7)
O1B—C2B	1.325 (5)	C1B—C6B	1.408 (7)
O3B—C3B	1.365 (7)	C1B—C7B	1.435 (7)
O3B—C31B	1.420 (6)	C2B—C3B	1.409 (7)
N1A—C7A	1.278 (5)	C3B—C4B	1.390 (8)
N1A—C8A	1.466 (5)	C31B—H31A	0.9600
N2A—C11A	1.474 (6)	C31B—H31B	0.9600
N2A—C12A	1.476 (6)	C31B—H31C	0.9600
N2A—C10A	1.484 (6)	C4B—C5B	1.378 (10)
N1B—C7B	1.281 (6)	C4B—H4BA	0.9300
N1B—C8B	1.467 (6)	C5B—C6B	1.363 (9)
N2B—C11B	1.474 (6)	C5B—H5BA	0.9300
N2B—C10B	1.475 (6)	C6B—H6BA	0.9300
N2B—C12B	1.486 (6)	C7B—H7BA	0.9300
C1A—C2A	1.402 (6)	C8B—C9B	1.501 (8)
C1A—C6A	1.408 (7)	C8B—H8BA	0.9700
C1A—C7A	1.428 (6)	C8B—H8BB	0.9700
C2A—C3A	1.419 (6)	C9B—C10B	1.517 (8)
C3A—C4A	1.370 (7)	C9B—H9BA	0.9700
C31A—H31D	0.9600	C9B—H9BB	0.9700
C31A—H31E	0.9600	C10B—H10C	0.9700

C31A—H31F	0.9600	C10B—H10D	0.9700
C4A—C5A	1.384 (8)	C11B—H11C	0.9700
C4A—H4AA	0.9300	C11B—H11D	0.9700
C5A—C6A	1.337 (7)	C12B—H12C	0.9700
C5A—H5AA	0.9300	C12B—H12D	0.9700
C6A—H6AA	0.9300		
O1A—Fe—O1B	127.67 (14)	N2A—C11A—C12B	108.1 (4)
O1A—Fe—N1B	85.93 (14)	N2A—C11A—H11A	110.1
O1B—Fe—N1B	83.57 (14)	C12B—C11A—H11A	110.1
O1A—Fe—N1A	83.83 (13)	N2A—C11A—H11B	110.1
O1B—Fe—N1A	85.37 (14)	C12B—C11A—H11B	110.1
N1B—Fe—N1A	155.69 (14)	H11A—C11A—H11B	108.4
O1A—Fe—N2B	88.88 (14)	N2A—C12A—C11B	107.7 (4)
O1B—Fe—N2B	138.58 (13)	N2A—C12A—H12A	110.2
N1B—Fe—N2B	80.13 (15)	C11B—C12A—H12A	110.2
N1A—Fe—N2B	121.53 (14)	N2A—C12A—H12B	110.2
O1A—Fe—N2A	138.21 (14)	C11B—C12A—H12B	110.2
O1B—Fe—N2A	89.52 (14)	H12A—C12A—H12B	108.5
N1B—Fe—N2A	120.61 (15)	C2B—C1B—C6B	119.7 (6)
N1A—Fe—N2A	80.78 (14)	C2B—C1B—C7B	121.5 (4)
N2B—Fe—N2A	67.37 (15)	C6B—C1B—C7B	118.7 (5)
C2A—O1A—Fe	135.2 (3)	O1B—C2B—C1B	122.9 (5)
C3A—O3A—C31A	116.8 (4)	O1B—C2B—C3B	117.8 (5)
C2B—O1B—Fe	131.5 (3)	C1B—C2B—C3B	119.3 (5)
C3B—O3B—C31B	118.3 (5)	O3B—C3B—C4B	125.4 (6)
C7A—N1A—C8A	117.0 (4)	O3B—C3B—C2B	114.9 (5)
C7A—N1A—Fe	124.9 (3)	C4B—C3B—C2B	119.7 (6)
C8A—N1A—Fe	118.1 (3)	O3B—C31B—H31A	109.5
C11A—N2A—C12A	108.0 (4)	O3B—C31B—H31B	109.5
C11A—N2A—C10A	110.8 (4)	H31A—C31B—H31B	109.5
C12A—N2A—C10A	111.8 (4)	O3B—C31B—H31C	109.5
C11A—N2A—Fe	100.1 (3)	H31A—C31B—H31C	109.5
C12A—N2A—Fe	106.5 (3)	H31B—C31B—H31C	109.5
C10A—N2A—Fe	118.6 (3)	C5B—C4B—C3B	120.2 (6)
C7B—N1B—C8B	117.8 (4)	C5B—C4B—H4BA	119.9
C7B—N1B—Fe	124.4 (4)	C3B—C4B—H4BA	119.9
C8B—N1B—Fe	117.6 (3)	C6B—C5B—C4B	120.8 (6)
C11B—N2B—C10B	110.1 (4)	C6B—C5B—H5BA	119.6
C11B—N2B—C12B	107.1 (4)	C4B—C5B—H5BA	119.6
C10B—N2B—C12B	112.6 (4)	C5B—C6B—C1B	120.3 (6)
C11B—N2B—Fe	100.6 (3)	C5B—C6B—H6BA	119.9
C10B—N2B—Fe	118.8 (3)	C1B—C6B—H6BA	119.9
C12B—N2B—Fe	106.5 (3)	N1B—C7B—C1B	126.3 (5)
C2A—C1A—C6A	118.7 (4)	N1B—C7B—H7BA	116.9
C2A—C1A—C7A	121.2 (4)	C1B—C7B—H7BA	116.9
C6A—C1A—C7A	120.0 (5)	N1B—C8B—C9B	113.4 (5)
O1A—C2A—C1A	122.3 (4)	N1B—C8B—H8BA	108.9
O1A—C2A—C3A	118.8 (4)	C9B—C8B—H8BA	108.9
C1A—C2A—C3A	118.9 (4)	N1B—C8B—H8BB	108.9

O3A—C3A—C4A	125.6 (5)	C9B—C8B—H8BB	108.9
O3A—C3A—C2A	114.6 (4)	H8BA—C8B—H8BB	107.7
C4A—C3A—C2A	119.8 (5)	C8B—C9B—C10B	115.3 (5)
O3A—C31A—H31D	109.5	C8B—C9B—H9BA	108.5
O3A—C31A—H31E	109.5	C10B—C9B—H9BA	108.5
H31D—C31A—H31E	109.5	C8B—C9B—H9BB	108.5
O3A—C31A—H31F	109.5	C10B—C9B—H9BB	108.5
H31D—C31A—H31F	109.5	H9BA—C9B—H9BB	107.5
H31E—C31A—H31F	109.5	N2B—C10B—C9B	114.6 (4)
C3A—C4A—C5A	120.5 (5)	N2B—C10B—H10C	108.6
C3A—C4A—H4AA	119.8	C9B—C10B—H10C	108.6
C5A—C4A—H4AA	119.8	N2B—C10B—H10D	108.6
C6A—C5A—C4A	120.8 (5)	C9B—C10B—H10D	108.6
C6A—C5A—H5AA	119.6	H10C—C10B—H10D	107.6
C4A—C5A—H5AA	119.6	N2B—C11B—C12A	108.6 (4)
C5A—C6A—C1A	121.3 (6)	N2B—C11B—H11C	110.0
C5A—C6A—H6AA	119.3	C12A—C11B—H11C	110.0
C1A—C6A—H6AA	119.3	N2B—C11B—H11D	110.0
N1A—C7A—C1A	127.5 (4)	C12A—C11B—H11D	110.0
N1A—C7A—H7AA	116.2	H11C—C11B—H11D	108.3
C1A—C7A—H7AA	116.2	N2B—C12B—C11A	107.6 (4)
N1A—C8A—C9A	112.8 (4)	N2B—C12B—H12C	110.2
N1A—C8A—H8AA	109.0	C11A—C12B—H12C	110.2
C9A—C8A—H8AA	109.0	N2B—C12B—H12D	110.2
N1A—C8A—H8AB	109.0	C11A—C12B—H12D	110.2
C9A—C8A—H8AB	109.0	H12C—C12B—H12D	108.5
H8AA—C8A—H8AB	107.8	F2A—B—F4A	111.9 (8)
C10A—C9A—C8A	115.1 (4)	F3—B—F2	113.1 (7)
C10A—C9A—H9AA	108.5	F3—B—F1	117.3 (6)
C8A—C9A—H9AA	108.5	F2A—B—F1	117.8 (6)
C10A—C9A—H9AB	108.5	F4A—B—F1	106.9 (7)
C8A—C9A—H9AB	108.5	F2—B—F1	107.9 (6)
H9AA—C9A—H9AB	107.5	F3—B—F4	109.4 (7)
N2A—C10A—C9A	114.5 (4)	F2—B—F4	105.1 (7)
N2A—C10A—H10A	108.6	F1—B—F4	102.9 (6)
C9A—C10A—H10A	108.6	F2A—B—F3A	109.1 (7)
N2A—C10A—H10B	108.6	F4A—B—F3A	105.0 (7)
C9A—C10A—H10B	108.6	F1—B—F3A	105.2 (6)
H10A—C10A—H10B	107.6		
O1B—Fe—O1A—C2A	105.8 (4)	C31A—O3A—C3A—C4A	7.7 (8)
N1B—Fe—O1A—C2A	−175.6 (4)	C31A—O3A—C3A—C2A	−172.5 (5)
N1A—Fe—O1A—C2A	26.5 (4)	O1A—C2A—C3A—O3A	−2.5 (7)
N2B—Fe—O1A—C2A	−95.4 (4)	C1A—C2A—C3A—O3A	178.0 (4)
N2A—Fe—O1A—C2A	−42.2 (5)	O1A—C2A—C3A—C4A	177.4 (5)
O1A—Fe—O1B—C2B	115.0 (4)	C1A—C2A—C3A—C4A	−2.2 (7)
N1B—Fe—O1B—C2B	35.3 (4)	O3A—C3A—C4A—C5A	−179.9 (5)
N1A—Fe—O1B—C2B	−166.4 (4)	C2A—C3A—C4A—C5A	0.2 (9)
N2B—Fe—O1B—C2B	−31.9 (5)	C3A—C4A—C5A—C6A	0.9 (10)
N2A—Fe—O1B—C2B	−85.6 (4)	C4A—C5A—C6A—C1A	−0.1 (10)

O1A—Fe—N1A—C7A	−16.0 (4)	C2A—C1A—C6A—C5A	−1.8 (8)
O1B—Fe—N1A—C7A	−144.7 (4)	C7A—C1A—C6A—C5A	−178.3 (5)
N1B—Fe—N1A—C7A	−81.6 (5)	C8A—N1A—C7A—C1A	−176.2 (5)
N2B—Fe—N1A—C7A	68.9 (4)	Fe—N1A—C7A—C1A	4.6 (7)
N2A—Fe—N1A—C7A	125.0 (4)	C2A—C1A—C7A—N1A	7.4 (8)
O1A—Fe—N1A—C8A	164.8 (3)	C6A—C1A—C7A—N1A	−176.2 (5)
O1B—Fe—N1A—C8A	36.1 (3)	C7A—N1A—C8A—C9A	−110.1 (5)
N1B—Fe—N1A—C8A	99.2 (5)	Fe—N1A—C8A—C9A	69.2 (5)
N2B—Fe—N1A—C8A	−110.3 (3)	N1A—C8A—C9A—C10A	−62.7 (6)
N2A—Fe—N1A—C8A	−54.2 (3)	C11A—N2A—C10A—C9A	−176.4 (4)
O1A—Fe—N2A—C11A	−119.4 (3)	C12A—N2A—C10A—C9A	63.1 (6)
O1B—Fe—N2A—C11A	85.3 (3)	Fe—N2A—C10A—C9A	−61.5 (5)
N1B—Fe—N2A—C11A	3.1 (3)	C8A—C9A—C10A—N2A	59.4 (7)
N1A—Fe—N2A—C11A	170.7 (3)	C12A—N2A—C11A—C12B	−53.5 (5)
N2B—Fe—N2A—C11A	−59.3 (3)	C10A—N2A—C11A—C12B	−176.2 (4)
O1A—Fe—N2A—C12A	−7.0 (4)	Fe—N2A—C11A—C12B	57.7 (4)
O1B—Fe—N2A—C12A	−162.3 (3)	C11A—N2A—C12A—C11B	70.4 (5)
N1B—Fe—N2A—C12A	115.5 (3)	C10A—N2A—C12A—C11B	−167.5 (4)
N1A—Fe—N2A—C12A	−76.9 (3)	Fe—N2A—C12A—C11B	−36.4 (4)
N2B—Fe—N2A—C12A	53.1 (3)	Fe—O1B—C2B—C1B	−29.8 (6)
O1A—Fe—N2A—C10A	120.0 (3)	Fe—O1B—C2B—C3B	151.8 (3)
O1B—Fe—N2A—C10A	−35.2 (3)	C6B—C1B—C2B—O1B	−179.0 (4)
N1B—Fe—N2A—C10A	−117.4 (3)	C7B—C1B—C2B—O1B	−2.5 (7)
N1A—Fe—N2A—C10A	50.2 (3)	C6B—C1B—C2B—C3B	−0.6 (7)
N2B—Fe—N2A—C10A	−179.8 (4)	C7B—C1B—C2B—C3B	175.9 (5)
O1A—Fe—N1B—C7B	−151.1 (4)	C31B—O3B—C3B—C4B	3.4 (8)
O1B—Fe—N1B—C7B	−22.4 (4)	C31B—O3B—C3B—C2B	−176.5 (5)
N1A—Fe—N1B—C7B	−85.9 (5)	O1B—C2B—C3B—O3B	−0.2 (7)
N2B—Fe—N1B—C7B	119.4 (4)	C1B—C2B—C3B—O3B	−178.7 (4)
N2A—Fe—N1B—C7B	63.2 (4)	O1B—C2B—C3B—C4B	179.9 (4)
O1A—Fe—N1B—C8B	33.7 (4)	C1B—C2B—C3B—C4B	1.4 (7)
O1B—Fe—N1B—C8B	162.4 (4)	O3B—C3B—C4B—C5B	179.6 (6)
N1A—Fe—N1B—C8B	98.9 (5)	C2B—C3B—C4B—C5B	−0.6 (9)
N2B—Fe—N1B—C8B	−55.8 (4)	C3B—C4B—C5B—C6B	−1.2 (10)
N2A—Fe—N1B—C8B	−112.0 (4)	C4B—C5B—C6B—C1B	2.1 (10)
O1A—Fe—N2B—C11B	86.3 (3)	C2B—C1B—C6B—C5B	−1.2 (8)
O1B—Fe—N2B—C11B	−119.3 (3)	C7B—C1B—C6B—C5B	−177.8 (5)
N1B—Fe—N2B—C11B	172.4 (3)	C8B—N1B—C7B—C1B	−179.2 (5)
N1A—Fe—N2B—C11B	4.3 (3)	Fe—N1B—C7B—C1B	5.7 (7)
N2A—Fe—N2B—C11B	−58.4 (3)	C2B—C1B—C7B—N1B	12.4 (8)
O1A—Fe—N2B—C10B	−33.8 (3)	C6B—C1B—C7B—N1B	−171.1 (5)
O1B—Fe—N2B—C10B	120.6 (3)	C7B—N1B—C8B—C9B	−105.6 (5)
N1B—Fe—N2B—C10B	52.3 (3)	Fe—N1B—C8B—C9B	69.9 (5)
N1A—Fe—N2B—C10B	−115.8 (3)	N1B—C8B—C9B—C10B	−60.6 (7)
N2A—Fe—N2B—C10B	−178.5 (4)	C11B—N2B—C10B—C9B	−176.9 (4)
O1A—Fe—N2B—C12B	−162.1 (3)	C12B—N2B—C10B—C9B	63.6 (6)
O1B—Fe—N2B—C12B	−7.7 (4)	Fe—N2B—C10B—C9B	−61.7 (5)
N1B—Fe—N2B—C12B	−76.0 (3)	C8B—C9B—C10B—N2B	56.9 (7)
N1A—Fe—N2B—C12B	115.9 (3)	C10B—N2B—C11B—C12A	−177.0 (4)

N2A—Fe—N2B—C12B	53.2 (3)	C12B—N2B—C11B—C12A	−54.2 (5)
Fe—O1A—C2A—C1A	−23.4 (7)	Fe—N2B—C11B—C12A	56.9 (4)
Fe—O1A—C2A—C3A	157.1 (3)	N2A—C12A—C11B—N2B	−13.5 (5)
C6A—C1A—C2A—O1A	−176.6 (4)	C11B—N2B—C12B—C11A	70.9 (5)
C7A—C1A—C2A—O1A	−0.2 (7)	C10B—N2B—C12B—C11A	−168.0 (4)
C6A—C1A—C2A—C3A	2.9 (7)	Fe—N2B—C12B—C11A	−36.1 (5)
C7A—C1A—C2A—C3A	179.4 (4)	N2A—C11A—C12B—N2B	−14.6 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9A—H9AA···F4^{i}	0.97	2.51	3.464 (14)	170
C9A—H9AA···F2A^{i}	0.97	2.54	3.442 (14)	155
C12A—H12B···F4^{i}	0.97	2.46	3.275 (11)	141
C12A—H12B···F2A^{i}	0.97	2.30	3.252 (10)	166
C12B—H12D···F3^{ii}	0.97	2.35	3.279 (8)	160
C9B—H9BB···F3A^{ii}	0.97	2.21	3.119 (9)	155

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

Fig. 1

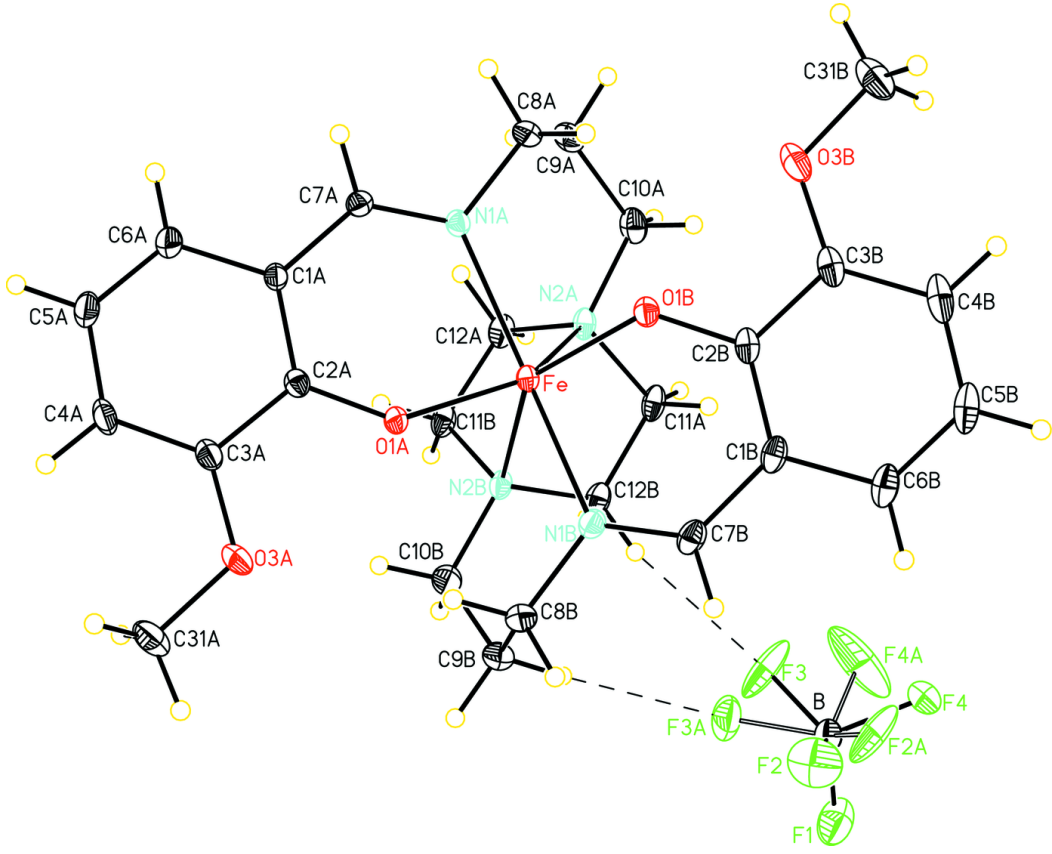

C31B
C8A
C9A
O3B
C10A
C7A
N1A
C6A
C1A
C3B
C4B
N2A
O1B
C5A
C12A
C2B
C2A
Fe
C5B
C4A
C11B
C11A
O1A
C1B
C3A
N2B
C12B
C6B
N1B
C7B
O3A
C10B
C8B
C31A
C9B
F4A
F3
F4
B
F3A
F2A
F2
F1

Fig. 2