

Bis[di-2-pyridyl(hydroxy)methanolato- κ^3O,N,N']-
iron(III) tetrafluoroborate trihydrateDjulia Onggo,^a Aminah Mimin,^a
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Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(C-C) = 0.007$ Å
Disorder in solvent or counterion
 R factor = 0.067
 wR factor = 0.172
Data-to-parameter ratio = 12.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound, $[\text{Fe}(\text{C}_{11}\text{H}_9\text{O}_2\text{N}_2)_2]\text{BF}_4 \cdot 3\text{H}_2\text{O}$, contains two independent bis[di-2-pyridyl(hydroxy)methanolato- κ^3O,N,N']iron(III) cations, with the Fe atom occupying a special position of $\bar{1}$ symmetry in each case; the Fe atom is O,N,N' -chelated by the anionic ligands in an octahedral geometry. The cation, the disordered BF_4^- counter-ion and the uncoordinated water molecules interact *via* hydrogen bonds to form a sheet motif.

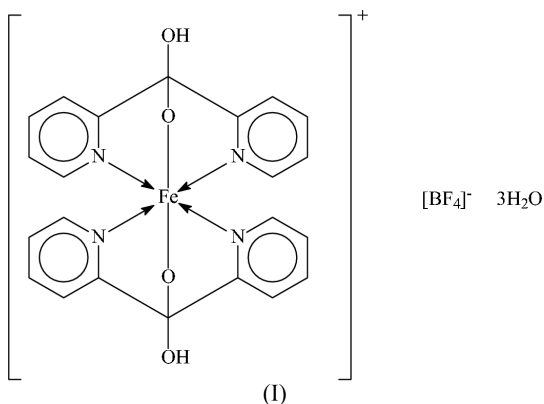
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Comment

The ketal bis(2-pyridyl)methanediol is generated *in situ* when di-2-pyridyl ketone reacts with a metal salt. Diketals can chelate to the metal atom in their neutral (doubly protonated), singly deprotonated or doubly deprotonated forms, as noted from a search of the Cambridge Structural Database (Version 5.26; Allen, 2002). For example, the diketal exists in the neutral form when di-2-pyridyl ketone is reacted with nickel and copper carboxylates (Li *et al.*, 2005*a,b*). The diketal exists in the singly deprotonated form in iron(III) nitrate monohydrate (Woltz *et al.*, 2002).



We report here another iron(III)–ketal complex, (I), with tetrafluoroborate as the counter-ion (Fig. 1). The bond distances (Table 1) and angles involving the Fe atoms are not significantly different from those reported by Woltz *et al.* (2002).

The crystal structure of (I) contains two independent bis[di-2-pyridyl(hydroxy)methanolato- κ^3O,N,N']iron(III) cations. Both are generated by inversion symmetry from the atoms of the asymmetric unit. The Fe atoms are O,N,N' -chelated by the anions in an octahedral environment. The BF_4^- counter-ion is disordered over two positions. The cations, anions and uncoordinated water molecules interact *via* $\text{O}-\text{H}\cdots\text{O}$ and $\text{O}-$

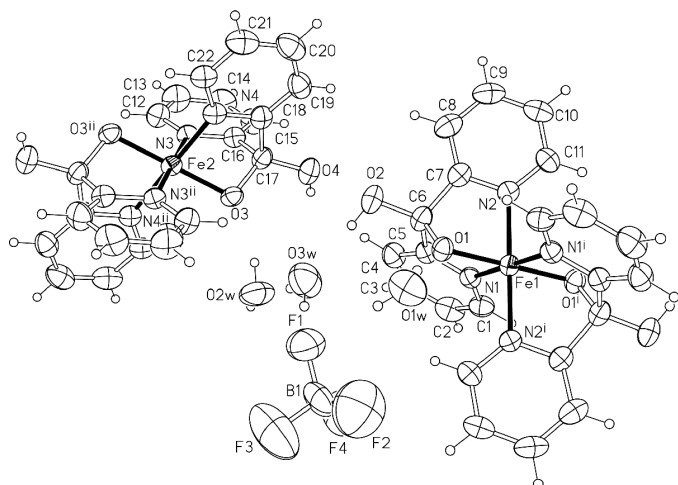


Figure 1
View of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). Only one conformation of the disordered BF_4^- species is shown. [Symmetry codes (i) $1-x, 1-y, 1-z$; (ii) $1-x, -y, -z$.]

$\text{H}\cdots\text{F}$ hydrogen bonds (Table 2), resulting in a layered structure.

Experimental

Iron(II) tetrafluoroborate hexahydrate (0.17 g, 0.5 mmol) was dissolved in methanol (8 ml) under an argon atmosphere. To the solution was added di-2-pyridyl ketone (0.18 g, 1.0 mmol) dissolved in ethanol (8 ml). The solvent was allowed to evaporate from the mixture for more than a week in air to afford orange crystals of (I).

Crystal data

$[\text{Fe}(\text{C}_{11}\text{H}_9\text{O}_2\text{N}_2)_2]\text{BF}_4\cdot 3\text{H}_2\text{O}$
 $M_r = 599.11$
 Triclinic, $P\bar{1}$
 $a = 8.9669$ (6) Å
 $b = 9.0527$ (6) Å
 $c = 16.295$ (1) Å
 $\alpha = 86.608$ (1)°
 $\beta = 78.840$ (1)°
 $\gamma = 86.593$ (1)°
 $V = 1293.9$ (2) Å³

$Z = 2$
 $D_x = 1.538$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3106 reflections
 $\theta = 2.3\text{--}26.2^\circ$
 $\mu = 0.66$ mm⁻¹
 $T = 298$ (2) K
 Block, orange
 $0.23 \times 0.10 \times 0.07$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.793$, $T_{\max} = 0.955$
 13700 measured reflections

5202 independent reflections
 4175 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\max} = 26.3^\circ$
 $h = -11 \rightarrow 11$
 $k = -11 \rightarrow 11$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.172$
 $S = 1.14$
 5202 reflections
 418 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2 + 0.9114P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.77$ e Å⁻³
 $\Delta\rho_{\min} = -0.38$ e Å⁻³

Table 1
Selected bond lengths (Å).

Fe1—O1	1.858 (3)	Fe2—O3	1.856 (3)
Fe1—N1	1.962 (3)	Fe2—N3	1.963 (3)
Fe1—N2	1.969 (3)	Fe2—N4	1.969 (3)

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

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
O2—H2o···O3w	0.85 (1)	1.86 (1)	2.708 (5)	178 (5)
O4—H4o···O1w ⁱⁱⁱ	0.85 (1)	1.77 (1)	2.614 (5)	174 (5)
O1w—H1w1···O1	0.85 (1)	1.84 (2)	2.676 (4)	167 (6)
O1w—H1w2···F1	0.85 (1)	2.03 (1)	2.881 (9)	177 (6)
O1w—H1w2···F2'	0.85 (1)	2.22 (5)	2.92 (1)	139 (6)
O2w—H2w1···O3	0.85 (1)	1.89 (2)	2.727 (5)	165 (6)
O2w—H2w2···F2 ⁱⁱⁱ	0.85 (1)	2.07 (4)	2.84 (1)	150 (6)
O2w—H2w2···F3 ⁱⁱⁱ	0.85 (1)	2.09 (2)	2.94 (1)	175 (7)
O3w—H3w1···F1	0.85 (1)	2.18 (3)	2.964 (9)	154 (5)
O3w—H3w1···F1'	0.85 (1)	2.28 (4)	3.06 (2)	153 (7)
O3w—H3w2···O2w	0.85 (1)	1.96 (1)	2.795 (6)	171 (6)

Symmetry code: (iii) $x, y-1, z$.

The BF_4^- counter-ion is disordered over two positions. The fractional site occupancies of the two overlapping conformations could not be refined and they are assumed to be equal. The B—F distances were restrained to 1.37 (1) Å and the F···F distances to 2.24 (1) Å. The displacement parameters for B1 and B1' were constrained to be equal; additionally, the anisotropic displacement parameters of the F atoms were restrained to be approximately isotropic. The hydroxy and water H atoms were located in difference Fourier maps, and refined with distance restraints of O—H = 0.85 (1) Å and H···H = 1.39 (1) Å. The other H atoms were positioned geometrically and refined as riding on their carrier atoms. The constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ was applied in all cases.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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