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Bis(di-2-pyridylmethanediol-κ^2N,N')iron(III) nitrate dihydrate

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The crystal structure of the title compound, $[Fe(C_{11}H_{10}N_2O_2)_2]NO_3{\cdot}2H_2O$, is the first reported iron(III) complex containing the hydrated form of di-2-pyridyl ketone (dpk-hydrate) as a ligand. Fe atoms reside on inversion centers in the triclinic cell and each cation has a distorted octahedral environment, which is typical of bis(dpk-hydrate)–transition metal complexes.

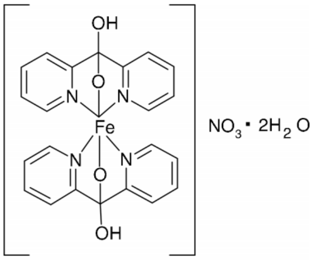
Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.108
Data-to-parameter ratio = 14.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

An *ORTEP*-3 (Farrugia, 1997) diagram of the two molecules, half of each of which constitue the asymmetric unit of the title compound, (I), is shown in Fig. 1, and selected bond lengths and angles are listed in Table 1. Molecules of (I) are centrosymmetric, as Fe atoms reside on inversion centers. The cations of (I) form discrete hydrogen bonds with nearby water molecules of hydration (Table 2). Both cations have a distorted octahedral geometry, with the off-axis coordination of ligand O atoms at an angle of 8.3° with respect to the axis that would normally be orthogonal to the plane generated by the coordinated N atoms in a non-distorted octahedral complex. The cation of (I) is similar in structure to several other known transition metal complexes of tridentate bis(dpk-hydrate). For example, the octahedral distortion in (I) is similar in magnitude to the off-axis angle of 8.0° reported for the isomorphous complex $[Co^{III}(dpk\text{-}hydrate)_2]NO_3{\cdot}2H_2O$ (Sommerer *et al.*, 1993). Previous studies on bis(dpk-hydrate) complexes of chromium(III) and rubidium(II) show greater distortions of the octahedral environment; chromium(III) has an off-axis distortion of 12.5°, whereas the angle for the rubidium(II) complex is 15.0° (Sommerer *et al.*, 1990). The greatest distortions from octahedral geometry come with the bis(dpk-hydrate) complexes of copper(II) and nickel(II), whose ligand O atoms retain their H atoms; the off-axis distortions are about 25 and 41°, respectively, for these molecules (Wang *et al.*, 1986).



(I)

Experimental

The title compound, (I), was prepared by combining 0.237 g (0.587 mmol) of ferric nitrate nonahydrate with 0.249 g (1.35 mmol)

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of di-2-pyridyl ketone in 30 ml of H_2O. The resulting mixture was filtered, and slow evaporation over 12 h of the orange filtrate afforded clear orange–yellow crystals suitable for X-ray analysis.

Crystal data

$[Fe(C_{11}H_{10}N_2O_2)_2]NO_3·2H_2O$
$M_r = 556.30$
Triclinic, $P\overline{1}$
$a = 8.7813$ (10) Å
$b = 8.8586$ (10) Å
$c = 16.1136$ (18) Å
$\alpha = 103.703$ (2)°
$\beta = 105.009$ (2)°
$\gamma = 94.870$ (2)°
$V = 1161.9$ (2) Å^3

$Z = 2$
$D_x = 1.579$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7293 reflections
$\theta = 2.4$–28.2°
$\mu = 0.71$ mm^{-1}
$T = 153$ (2) K
Parallelepiped, orange–yellow
0.35 × 0.13 × 0.11 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*, Bruker, 1997)
$T_{min} = 0.899$, $T_{max} = 1.000$
9820 measured reflections

5174 independent reflections
4609 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 28.3°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.108$
$S = 1.02$
5174 reflections
353 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.7918P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.041$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—O1	1.8598 (15)	Fe2—O3	1.8639 (14)
Fe1—N2	1.9491 (17)	Fe2—N4	1.9470 (17)
Fe1—N1	1.9499 (17)	Fe2—N3	1.9517 (18)
O1—Fe1—N2	82.70 (7)	O3—Fe2—N4	82.45 (7)
O1—Fe1—N1	82.59 (7)	O3—Fe2—N3	82.95 (7)
N2—Fe1—N1	87.26 (7)	N4—Fe2—N3	87.10 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O8	0.84	1.76	2.604 (2)	178
O4—H4B···O9	0.84	1.82	2.660 (2)	174

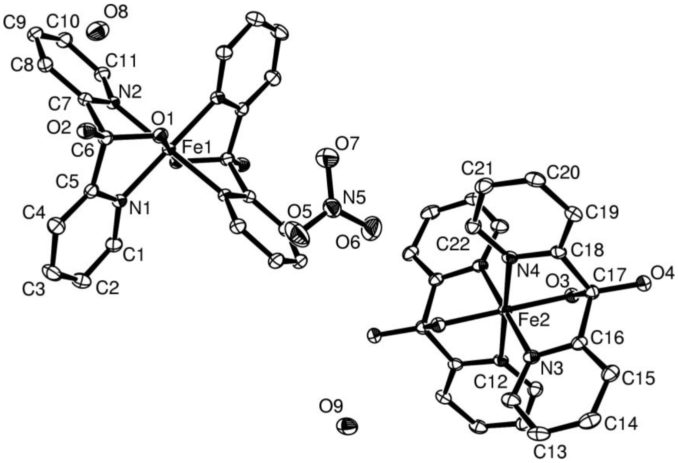

Figure 1
A view of the asymmetric unit of (I), together with the inversion-related second half of each cation. Displacement ellipsoids are drawn at the 50% probability level (*ORTEP*-3; Farrugia, 1997).

Water H atoms were located in difference maps and were refined freely. All other H atoms were placed geometrically and refined with a riding model.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

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