Aqua(2,9-dimethyl-1,10-phenanthroline-$\kappa^2 N$,N')-dinitratoiron(II)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.038
wR factor = 0.107
Data-to-parameter ratio = 13.5

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

In the title compound, [Fe(NO₃)₂(C₁₄H₁₂N₂)(H₂O)], the Fe atom is six-coordinated in an octahedral geometry by O and N atoms. The water molecules act as donors in O—H···O hydrogen bonds, linking the molecules into chains along the c axis. The chains are connected into a three-dimensional framework by other O—H···O and C—H···O hydrogen bonds. The packing is further stabilized by C—H···π and π–π interactions.

Comment

Metal–phenanthroline complexes are good indicators for the hybridization detection of DNA in electrochemical biosensors (Wang *et al.*, 1996). In our search for new indicators (Zhang *et al.*, 2005), the title complex, (I), was synthesized. Here we report its structure.

(I)
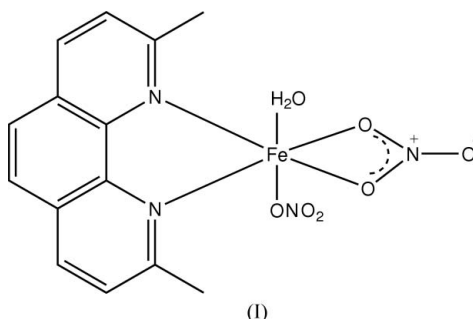
The Fe^{II} atom is six-coordinated by two N atoms from the 9,10-dimethylphenathroline ligand and four O atoms from two nitrate anions and one water molecule (Fig. 1). The geometry around the Fe atom is octahedral (Table 1). All bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

In the crystal structure, the water molecules act as donors, forming O—H···O hydrogen bonds (Table 2). These hydrogen bonds involving the nitrate anions link the molecules into chains along the c axis (Fig. 2). The chains are connected into a three-dimensional framework by other O—H···O and C—H···O hydrogen bonds (Table 2). The short $Cg5$···$Cg5^{iv}$ distance of 3.572 Å [$Cg5$ is the centroid of the C5–C8/C12/C13 ring; symmetry code: (iv) $1 - x$, $-1 - y$, $-z$] indicates π–π stacking interactions between the phenanthroline ring systems.

Experimental

To a solution of 2,9-dimethyl-1,10-phenanthroline (0.21 g, 1 mmol) in ethanol (10 ml) was added a solution of anhydrous Fe(NO₃)₂ (0.12 g, 1 mmol) in distilled water (10 ml). The mixture was stirred and reluxed for 9 h. The hot solution was then filtered into another flask
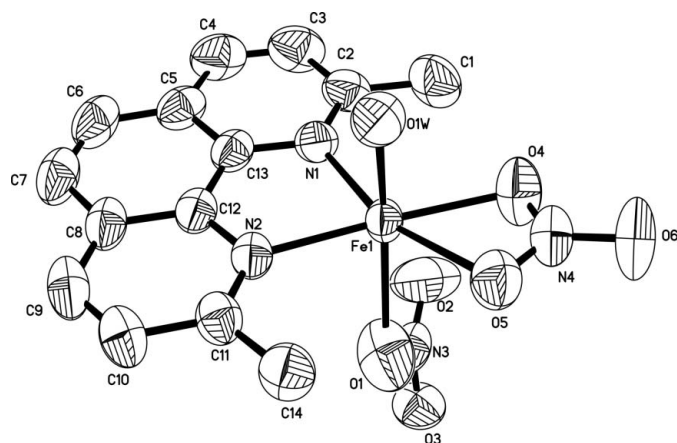
Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted.
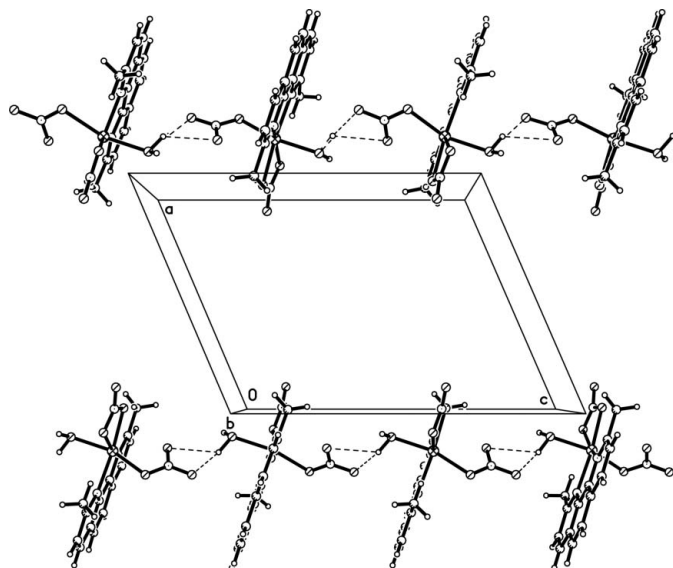

Figure 2
A view of part of the packing of (I) down the b axis, showing the chains. Intermolecular hydrogen bonds are denoted by dashed lines.

containing ethanol–water (1:2 v/v). Brown crystals of (I) appeared over a period of one week by slow evaporation at room temperature.

Crystal data

[Fe(NO$_3$)$_2$(C$_{14}$H$_{12}$N$_2$)(H$_2$O)]
M_r = 406.14
Monoclinic, $P2_1/c$
a = 11.1447 (13) Å
b = 10.8137 (13) Å
c = 15.0625 (14) Å
β = 113.306 (7)°
V = 1667.1 (3) Å^3

Z = 4
D_x = 1.618 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.95 mm^{-1}
T = 293 (2) K
Brown, plate
$0.28 \times 0.19 \times 0.09$ mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.777, T_{max} = 0.919

9395 measured reflections
3287 independent reflections
2747 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.04
3287 reflections
243 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.2884P]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected bond lengths (Å).

Fe1—O1W	2.077 (2)	Fe1—O4	2.133 (2)
Fe1—N1	2.115 (2)	Fe1—O1	2.213 (2)
Fe1—N2	2.1244 (19)	Fe1—O5	2.310 (2)

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

$Cg1$ and $Cg2$ denote the centroids of the Fe1/O4/O5/N4 and Fe1/N1/N2/C12/C13 rings, respectively.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1W—H1$W\cdots$O2^i	1.01 (4)	2.46 (4)	3.089 (4)	120 (3)
O1W—H1$W\cdots$O3^i	1.01 (4)	1.83 (4)	2.824 (3)	166 (4)
O1W—H2$W\cdots$O6^{ii}	0.84 (5)	1.96 (5)	2.780 (3)	167 (5)
C7—H7$A\cdots$O6^{iii}	0.93	2.54	3.416 (4)	158

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

C-bound H atoms were refined using a riding model, with C—H = 0.93–0.96 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The H atoms of the water molecule were located in a difference Fourier map and refined freely.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Versions 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wang, J., Cai, X., Rivas, G., Shiraishi, H., Farias, P. A. M. & Dontha, N. (1996). *Anal. Chem.* **68**, 2629–2634.
Zhang, S.-S., Niu, S.-Y., Qu, B., Jie, G.-F., Xu, H. & Ding, C.-F. (2005). *J. Inorg. Biochem.* **99**, 2340–2347.