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A three-dimensional hydrogen-bonded network of diaquabis(2,5-di-3-pyridyl-1,3,4-oxadiazole)dithiocyanatoiron(II)

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The molecular structure of the neutral mononuclear complex [Fe(NCS)$_2$(3-bpo)$_2$(H$_2$O)$_2$], in which 3-bpo denotes 2,5-di-3-pyridyl-1,3,4-oxadiazole ($C_{12}H_8N_4O$), is centrosymmetric, with Fe^{II} located on an inversion center. This complex is octahedral, and the Fe^{II} atom is *trans* coordinated by two monodentate NSC$^-$ ligands, two monodentate 3-bpo ligands and two water molecules. Each complex is linked to four others, forming a (011) sheet, by O—H···N hydrogen bonding. Weak C—H···S interactions link each (011) sheet to the two adjacent sheets, hence forming a three-dimensional array. There is also some slipped π stacking within each sheet.

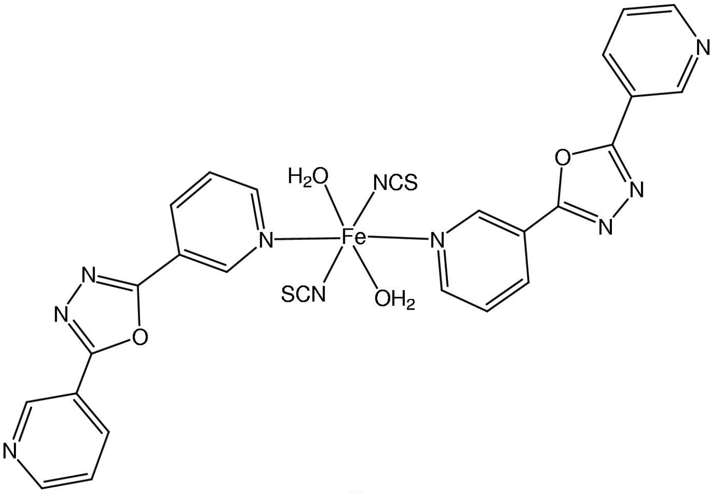
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.093
Data-to-parameter ratio = 12.5

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

Comment

This work is part of our continuing interest in metal-containing assemblies based on dipyridyl ligands containing the oxadiazole spacer, such as 2,5-di-4-pyridyl-1,3,4-oxadiazole (4-bpo) and its 3,3′-*N*-donor analog (3-bpo), *via* coordination, hydrogen-bonded and/or other intermolecular cooperative interactions (Du, Zhao & Guo, 2005, and references therein; Du & Zhao, 2004). Very recently, we have described an Mn^{II}–3-bpo–NCS mononuclear complex, [Mn(NCS)$_2$(3-bpo)$_2$(H$_2$O)$_2$], with a three-dimensional hydrogen-bonded network (Du, Jiang & Zhao, 2005), and we report here the isostructural Fe^{II} compound, namely the title complex, (I).



(I)

In the molecular structure (Fig. 1) of the neutral mononuclear complex (I), each Fe^{II} atom, located on an inversion center, is six-coordinated by two monodentate NSC$^-$ anions and two water molecules, forming the equatorial plane, and

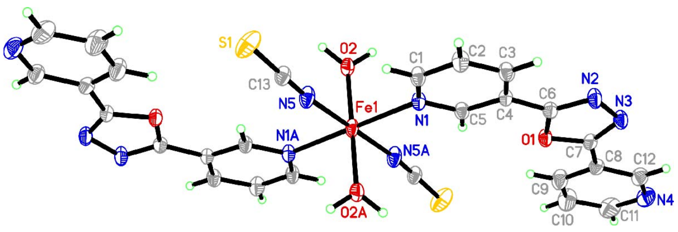


Figure 1
The title complex, with atom labeling of the asymmetric unit and the Fe^{II} coordination sphere, shown with 30% probability displacement ellipsoids. [Symmetry code: (*A*) $1 - x, 1 - y, 1 - z$.]

two pyridyl N atoms of a pair of monodentate 3-bpo ligands in the axial positions, displaying an FeN_4O_2 octahedral geometry. The metal–ligand distances within the octahedral framework are close to values reported for related $[Fe(NCS)_2(L)_2(H_2O)_2]$ complexes (Long *et al.*, 1983; Cingi *et al.*, 1986; Noro *et al.*, 1999). The 3-bpo ligand is not planar; the pyridyl rings make dihedral angles of 6.8 (2) and 10.0 (2)° with the central oxadiazole system, and the dihedral angle between them is 5.0 (2)°.

The 3-bpo molecule takes the unusual *trans* configuration with respect to the two terminal pyridyl groups (Du & Zhao, 2003), in order to favor the hydrogen bond between the uncoordinated 3-pyridyl N atom and the aqua ligand. Each water molecule also generates a hydrogen bond with one N atom of the oxadiazole group. Thus, each complex is linked to four others, forming a (011) sheet, by O—H···N hydrogen bonds. Weak C—H···S interactions link each (011) sheet to the two adjacent sheets, hence forming a three-dimensional array (Table 1). There is also some slipped π stacking within each sheet. The centroid-to-centroid and centroid-to-plane distances between two neighboring almost antiparallel pyridyl rings are 3.87 and 3.50/3.42 Å, respectively.

Experimental

$Fe(ClO_4)_2\cdot6H_2O$ (0.1 mmol), 3-bpo (0.1 mmol) and NH_4SCN (0.1 mmol) were mixed in a CH_3CN/H_2O (20 ml, 1:1) solution with refluxing for *ca* 20 min. The resulting solution was filtered and left to stand at room temperature. Red prismatic crystals suitable for X-ray analysis were obtained in 50% yield by slow evaporation of the solvent over a period of 1 week. Analysis calculated for $C_{26}H_{20}FeN_{10}O_4S_2$: C 47.57, H 3.07, N 21.34%; found: C 47.55, H 2.98, N 21.00%. IR (KBr pellet, cm^{-1}): 3235 (*b*), 3070 (*m*), 2079 (*vs*), 1663 (*m*), 1606 (*vs*), 1587 (*s*), 1551 (*m*), 1485 (*s*), 1464 (*s*), 1436 (*s*), 1417 (*s*), 1337 (*m*), 1276 (*m*), 1196 (*m*), 1128 (*m*), 1116 (*m*), 1087 (*s*), 1042 (*s*), 1028 (*s*), 997 (*w*), 965 (*m*), 823 (*s*), 733 (*s*), 699 (*vs*), 636 (*m*).

Crystal data

$[Fe(C_{12}H_8N_4O)_2(NCS)_2(H_2O)_2]$
$M_r = 656.49$
Triclinic, $P\bar{1}$
$a = 8.159$ (7) Å
$b = 8.712$ (7) Å
$c = 10.531$ (9) Å
$\alpha = 82.385$ (13)°
$\beta = 77.494$ (13)°
$\gamma = 80.064$ (14)°
$V = 716.3$ (10) $Å^3$

$Z = 1$
$D_x = 1.522$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 697 reflections
$\theta = 2.4$–26.0°
$\mu = 0.73$ mm^{-1}
$T = 293$ (2) K
Prism, red
0.20 × 0.18 × 0.14 mm

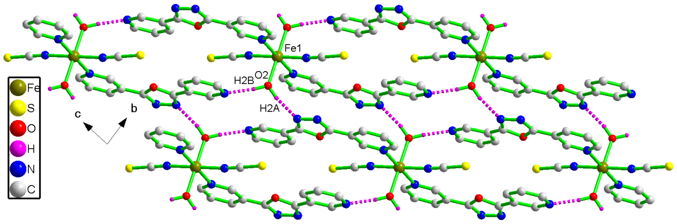


Figure 2
The two-dimensional supramolecular structure of the title complex, formed *via* O—H···N hydrogen-bonds between water and 3-bpo (indicated by purple dashed lines).

Data collection

Bruker APEX II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.810$, $T_{max} = 0.903$
3735 measured reflections

2517 independent reflections
1851 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 6$
$k = -10 \rightarrow 9$
$l = -12 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.093$
$S = 1.02$
2517 reflections
202 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 0.299P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e $Å^{-3}$
$\Delta\rho_{min} = -0.31$ e $Å^{-3}$

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N3^{i}	0.84 (1)	1.98 (1)	2.807 (4)	167 (3)
O2—H2*B*···N4^{ii}	0.84 (1)	1.95 (1)	2.775 (3)	168 (3)
C10—H10···S1^{iii}	0.93	2.83	3.575 (4)	138
C9—H9···S1^{iv}	0.93	2.85	3.625 (4)	141

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

H atoms were located in difference maps, but those attached to C atoms were introduced in calculated positions and treated as riding, with C—H = 0.93Å. H atoms of water molecules were refined using restraints [O—H = 0.85 (1) Å and H···H = 1.39 (2) Å]. All H atoms were allocated displacement parameters related to those of their parent atoms [$U_{iso}(H) = 1.2U_{eq}(C,O)$].

Data collection: *APEX2* (Bruker, 2003); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001) and *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *SHELXTL*.

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References

Brandenburg, K. & Berndt, M. (1999). *DIAMOND*. Version 2.1c. Crystal Impact GbR, Bonn, Germany.

Bruker (2001). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2003). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cingi, M. B., Lanfredi, A. M. M., Tiripicchio, A., Cornelissen, J. P., Haasnot, J. G. & Reedijk, J. (1986). *Acta Cryst.* C**42**, 1296–1298.

Du, M., Jiang, X.-J. & Zhao, X.-J. (2005). *Acta Cryst.* E**61**, m485–m487.

Du, M. & Zhao, X.-J. (2003). *J. Mol. Struct.* **655**, 191–197.

Du, M. & Zhao, X.-J. (2004). *J. Mol. Struct.* **694**, 235–240.

Du, M., Zhao, X.-J. & Guo, J. H. (2005). *Inorg. Chem. Commun.* **8**, 1–5.

Long, G. L., Galeazzi, G., Russo, U., Valle, G. & Calogero, S. (1983). *Inorg. Chem.* **22**, 507–510.

Noro, S., Kondo, M., Ishii, T., Kitagawa, S. & Matsuzaka, H. (1999). *J. Chem. Soc. Dalton Trans.* pp. 1569–1574.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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