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Bis(dipyrido[3,2-*a*:2′,3′-*c*]phenazine)-sulfatoiron(II) monohydrate

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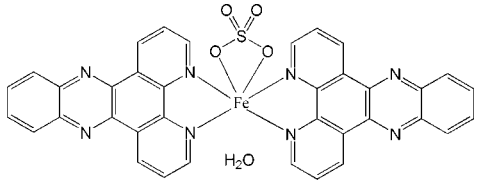
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.049; wR factor = 0.139; data-to-parameter ratio = 12.0.

In the title compound, $[Fe(SO_4)(C_{18}H_{10}N_4)_2]\cdot H_2O$, the Fe^{II} atom (site symmetry 2) is six-coodinated by four N atoms from two dipyrido[3,2-*a*:2′,3′-*c*]phenazine ligands and two O atoms from the SO_4^{2-} dianion in a distorted *cis*-FeO_2N_4 octahedral geometry. Numerous aromatic π–π stacking interactions [centroid separation = 3.671 (2)–4.090 (2) Å] and O—H···O hydrogen bonds help to stabilize the structure. The water molecule is disordered with a site-occupancy factor of 0.5.

Related literature

For related complexes containing phenanthroline-derived ligands, see: Che, Liu *et al.* (2006); Che (2006); Li *et al.* (2006). For the ligand synthesis, see: Che, Li *et al.* (2006).



Experimental

Crystal data

$[Fe(SO_4)(C_{18}H_{10}N_4)_2]\cdot H_2O$
M_r = 734.53
Monoclinic, $C2/c$
a = 17.527 (2) Å
b = 7.2674 (10) Å
c = 23.203 (3) Å
β = 94.826 (2)°
V = 2944.9 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 292 (2) K
0.32 × 0.14 × 0.09 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.895, T_{max} = 0.939
12199 measured reflections
2908 independent reflections
2041 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.139
S = 1.03
2908 reflections
242 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe—N1	2.151 (3)	Fe—O1	2.156 (3)
Fe—N2	2.185 (3)		
N1—Fe—N2	76.08 (10)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—HW11···O1	0.80 (5)	1.99 (5)	2.734 (7)	155 (7)

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: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2425).

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supplementary materials

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Bis(dipyrido[3,2-*a*:2',3'-*c*]phenazine)sulfatoiron(II) monohydrate

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Comment

Heteroaromatic N-donor chelating ligands such as 1,10-phenanthroline (phen) and its derivatives have been widely used in the construction of metal-organic complexes (Che, Liu *et al.*,2006; Li *et al.*, 2006). As a continuation of our studies, we have prepared the title compound, $FeSO_4(L)_2{\cdot}H_2O$, using the phen derivative dipyrido[3,2 − a:2',3'-c]phenazine (*L*) ligand.

In compound (I), (Fig. 1, Table 1), the Fe^{2+} atom (site symmetry 2) is six coordinated by four N atoms from two *L* ligands and two O atoms from the same SO_4^{2-} dianion in a distorted octahedral geometry. Neighbouring complexes are connected through π-π interactions between *L* ligands with a stacking distance of 3.671 (2) Å, leading to layers propagating in [101] (Fig. 2).

Experimental

Ligand *L* was synthesized according to the literature method of Che, Li *et al.* (2006). An ethanol solution (12 ml) of *L* (0.5 mmol) was added slowly to an aqueous solution (10 ml) of $FeSO_4$ (0.5 mmol). The mixture was sealed in a Teflon-lined autoclave and heated to 433 K for 4 d. Brown blocks and slabs of (I) were obtained upon cooling and opening the autoclave (65% yield based on Fe).

Refinement

The water O atom showed a large displacement parameter. Its site occupancy refined to close to 0.5 and the fit improved. Its occupancy was then fixed at 0.5. The water H atoms were located in a difference map and their positions were freely refined.

All C-bound H atoms were generated geometrically (C—H = 0.93 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}$(carrier).

Figures

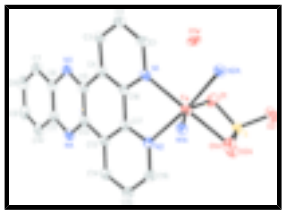

Fig. 1. View of the asymmetric unit of (I), together with further atoms to complete the Fe^{II} co-ordination sphere. Displacement ellipsoids are drawn at the 30% probability level (arbitrary spheres for the H atoms). [Symmetry code: 1 − *x*, *y*, 1/2 − *z*.]

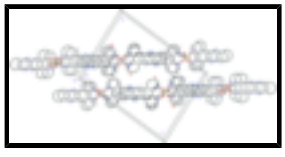

Fig. 2. View of the π-π stacking in the structure of (I) leading to chains of molecules.

Bis(dipyrido[3,2 − a:2',3'-c]phenazine)sulfatoiron(II) monohydrate

Crystal data

$[Fe(SO_4)(C_{18}H_{10}N_4)_2]\cdot H_2O$	$F_{000} = 1504$
$M_r = 734.53$	$D_x = 1.657$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 1263 reflections
$a = 17.527$ (2) Å	$\theta = 2.3$–26.0º
$b = 7.2674$ (10) Å	$\mu = 0.65$ mm^{-1}
$c = 23.203$ (3) Å	$T = 292$ (2) K
$\beta = 94.826$ (2)º	Slab, brown
$V = 2944.9$ (7) Å^3	0.32 × 0.14 × 0.09 mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	2908 independent reflections
Radiation source: fine-focus sealed tube	2041 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.061$
$T = 292$(2) K	$\theta_{max} = 26.1$º
ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -21\rightarrow21$
$T_{min} = 0.895$, $T_{max} = 0.939$	$k = -8\rightarrow8$
12199 measured reflections	$l = -28\rightarrow28$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.049$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.139$	$w = 1/[\sigma^2(F_o^2) + (0.0651P)^2 + 4.1225P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2908 reflections	$\Delta\rho_{max} = 0.55$ e Å^{-3}
242 parameters	$\Delta\rho_{min} = -0.39$ e Å^{-3}
3 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)
O1W	0.4209 (3)	0.1304 (8)	0.3969 (3)	0.0561 (17)	0.50
C1	0.4474 (2)	0.5284 (5)	0.35121 (16)	0.0351 (9)	
H1	0.3998	0.5299	0.3303	0.042*	
C2	0.4539 (2)	0.6080 (5)	0.40550 (16)	0.0374 (9)	
H2	0.4117	0.6633	0.4201	0.045*	
C3	0.52355 (19)	0.6044 (5)	0.43763 (15)	0.0335 (8)	
H3	0.5292	0.6546	0.4746	0.040*	
C4	0.58578 (19)	0.5231 (4)	0.41324 (15)	0.0291 (8)	
C5	0.66145 (19)	0.5085 (5)	0.44494 (14)	0.0296 (8)	
C6	0.7382 (2)	0.5424 (5)	0.52866 (15)	0.0335 (8)	
C7	0.7487 (2)	0.5916 (5)	0.58783 (16)	0.0432 (10)	
H7	0.7077	0.6361	0.6067	0.052*	
C8	0.8191 (3)	0.5735 (6)	0.61717 (18)	0.0490 (11)	
H8	0.8259	0.6076	0.6559	0.059*	
C9	0.8811 (2)	0.5045 (5)	0.58975 (17)	0.0467 (10)	
H9	0.9284	0.4921	0.6108	0.056*	
C10	0.8739 (2)	0.4551 (5)	0.53300 (17)	0.0436 (10)	
H10	0.9159	0.4105	0.5154	0.052*	
C11	0.8013 (2)	0.4723 (5)	0.50083 (16)	0.0343 (8)	
C12	0.72472 (19)	0.4395 (4)	0.41675 (15)	0.0292 (8)	
C13	0.71308 (19)	0.3818 (4)	0.35639 (14)	0.0284 (8)	
C14	0.77242 (19)	0.3215 (5)	0.32452 (15)	0.0341 (8)	
H14	0.8228	0.3274	0.3405	0.041*	
C15	0.7563 (2)	0.2534 (5)	0.26961 (15)	0.0353 (8)	
H15	0.7953	0.2125	0.2479	0.042*	
C16	0.6803 (2)	0.2471 (5)	0.24735 (16)	0.0362 (9)	
H16	0.6692	0.1956	0.2109	0.043*	
C17	0.63881 (18)	0.3793 (4)	0.32890 (14)	0.0281 (7)	
C18	0.57456 (18)	0.4516 (4)	0.35795 (14)	0.0266 (7)	
N1	0.50531 (15)	0.4501 (4)	0.32740 (12)	0.0297 (7)	
N2	0.62257 (15)	0.3105 (4)	0.27515 (12)	0.0307 (7)	
N3	0.66826 (17)	0.5592 (4)	0.50016 (12)	0.0339 (7)	
N4	0.79357 (16)	0.4221 (4)	0.44431 (13)	0.0329 (7)	

O1	0.48704 (15)	0.0364 (3)	0.29847 (10)	0.0432 (7)	
O2	0.43230 (14)	−0.2049 (4)	0.23570 (12)	0.0496 (7)	
S	0.5000	−0.09498 (18)	0.2500	0.0333 (3)	
Fe	0.5000	0.28602 (10)	0.2500	0.0322 (2)	
HW11	0.431 (4)	0.077 (10)	0.3686 (18)	0.048*	0.50
HW12	0.453 (3)	0.123 (12)	0.423 (2)	0.048*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1W	0.071 (4)	0.044 (3)	0.060 (4)	−0.009 (3)	0.051 (3)	−0.010 (3)
C1	0.0228 (18)	0.042 (2)	0.040 (2)	0.0028 (16)	0.0006 (16)	0.0013 (17)
C2	0.029 (2)	0.040 (2)	0.044 (2)	0.0051 (16)	0.0048 (16)	−0.0009 (17)
C3	0.032 (2)	0.037 (2)	0.0314 (19)	0.0018 (16)	0.0037 (15)	−0.0036 (16)
C4	0.0275 (18)	0.0286 (18)	0.0309 (19)	−0.0020 (14)	0.0014 (15)	0.0025 (15)
C5	0.0313 (19)	0.0279 (18)	0.0291 (19)	−0.0007 (15)	0.0001 (15)	0.0025 (15)
C6	0.039 (2)	0.0312 (19)	0.029 (2)	−0.0063 (16)	−0.0053 (16)	0.0051 (15)
C7	0.053 (3)	0.043 (2)	0.033 (2)	−0.0086 (19)	−0.0005 (18)	0.0003 (17)
C8	0.064 (3)	0.046 (2)	0.035 (2)	−0.012 (2)	−0.010 (2)	0.0043 (18)
C9	0.051 (3)	0.043 (2)	0.043 (2)	−0.008 (2)	−0.018 (2)	0.0045 (19)
C10	0.040 (2)	0.040 (2)	0.048 (3)	−0.0026 (18)	−0.0105 (19)	0.0044 (18)
C11	0.034 (2)	0.0282 (18)	0.038 (2)	−0.0047 (15)	−0.0089 (17)	0.0030 (15)
C12	0.0266 (18)	0.0287 (19)	0.0316 (19)	0.0002 (14)	−0.0027 (14)	0.0033 (14)
C13	0.0253 (18)	0.0291 (18)	0.0303 (18)	0.0019 (14)	−0.0004 (14)	0.0025 (14)
C14	0.0212 (18)	0.040 (2)	0.039 (2)	0.0000 (15)	−0.0042 (15)	0.0029 (17)
C15	0.0297 (19)	0.042 (2)	0.034 (2)	0.0035 (16)	0.0033 (15)	−0.0022 (16)
C16	0.033 (2)	0.046 (2)	0.030 (2)	0.0032 (17)	0.0012 (15)	−0.0040 (16)
C17	0.0257 (17)	0.0286 (18)	0.0300 (18)	0.0018 (14)	0.0012 (14)	0.0032 (14)
C18	0.0246 (17)	0.0288 (18)	0.0256 (18)	0.0003 (14)	−0.0018 (14)	0.0041 (14)
N1	0.0219 (15)	0.0324 (16)	0.0343 (16)	0.0004 (12)	−0.0008 (12)	0.0039 (12)
N2	0.0269 (15)	0.0392 (17)	0.0256 (15)	0.0010 (13)	0.0006 (12)	−0.0016 (13)
N3	0.0358 (17)	0.0357 (17)	0.0297 (16)	−0.0034 (13)	−0.0005 (13)	−0.0005 (13)
N4	0.0270 (16)	0.0347 (17)	0.0359 (17)	−0.0026 (13)	−0.0043 (13)	−0.0007 (13)
O1	0.0521 (17)	0.0475 (16)	0.0298 (14)	0.0019 (13)	0.0023 (12)	−0.0019 (12)
O2	0.0301 (15)	0.0503 (17)	0.0663 (19)	−0.0089 (13)	−0.0086 (13)	−0.0017 (14)
S	0.0253 (6)	0.0395 (7)	0.0341 (7)	0.000	−0.0036 (5)	0.000
Fe	0.0237 (4)	0.0425 (5)	0.0294 (4)	0.000	−0.0037 (3)	0.000

Geometric parameters (Å, °)

O1W—HW11	0.79 (2)	C11—N4	1.357 (4)
O1W—HW12	0.80 (2)	C12—N4	1.323 (4)
C1—N1	1.324 (4)	C12—C13	1.460 (5)
C1—C2	1.382 (5)	C13—C14	1.396 (5)
C1—H1	0.9300	C13—C17	1.401 (4)
C2—C3	1.377 (5)	C14—C15	1.374 (5)
C2—H2	0.9300	C14—H14	0.9300
C3—C4	1.401 (5)	C15—C16	1.389 (5)
C3—H3	0.9300	C15—H15	0.9300

C4—C18	1.383 (5)	C16—N2	1.327 (4)
C4—C5	1.465 (5)	C16—H16	0.9300
C5—N3	1.329 (4)	C17—N2	1.352 (4)
C5—C12	1.425 (5)	C17—C18	1.458 (4)
C6—N3	1.348 (4)	C18—N1	1.353 (4)
C6—C7	1.416 (5)	O1—S	1.507 (3)
C6—C11	1.422 (5)	O2—S	1.446 (2)
C7—C8	1.364 (5)	S—O2^{i}	1.446 (3)
C7—H7	0.9300	S—O1^{i}	1.507 (3)
C8—C9	1.399 (6)	Fe—N1	2.151 (3)
C8—H8	0.9300	Fe—N2	2.185 (3)
C9—C10	1.361 (5)	Fe—O1	2.156 (3)
C9—H9	0.9300	Fe—N1^{i}	2.151 (3)
C10—C11	1.425 (5)	Fe—O1^{i}	2.156 (3)
C10—H10	0.9300	Fe—N2^{i}	2.185 (3)
HW11—O1W—HW12	114 (4)	C14—C15—C16	118.2 (3)
N1—C1—C2	123.6 (3)	C14—C15—H15	120.9
N1—C1—H1	118.2	C16—C15—H15	120.9
C2—C1—H1	118.2	N2—C16—C15	123.7 (3)
C3—C2—C1	119.1 (3)	N2—C16—H16	118.2
C3—C2—H2	120.5	C15—C16—H16	118.2
C1—C2—H2	120.5	N2—C17—C13	122.7 (3)
C2—C3—C4	118.3 (3)	N2—C17—C18	116.7 (3)
C2—C3—H3	120.8	C13—C17—C18	120.6 (3)
C4—C3—H3	120.8	N1—C18—C4	122.7 (3)
C18—C4—C3	118.6 (3)	N1—C18—C17	116.6 (3)
C18—C4—C5	119.4 (3)	C4—C18—C17	120.6 (3)
C3—C4—C5	122.0 (3)	C1—N1—C18	117.6 (3)
N3—C5—C12	122.0 (3)	C1—N1—Fe	127.3 (2)
N3—C5—C4	117.9 (3)	C18—N1—Fe	114.6 (2)
C12—C5—C4	120.1 (3)	C16—N2—C17	117.8 (3)
N3—C6—C7	119.4 (4)	C16—N2—Fe	127.9 (2)
N3—C6—C11	121.3 (3)	C17—N2—Fe	113.7 (2)
C7—C6—C11	119.2 (3)	C5—N3—C6	116.9 (3)
C8—C7—C6	119.9 (4)	C12—N4—C11	117.0 (3)
C8—C7—H7	120.1	S—O1—Fe	96.58 (13)
C6—C7—H7	120.1	O2—S—O2^{i}	112.9 (2)
C7—C8—C9	120.8 (4)	O2—S—O1	110.43 (15)
C7—C8—H8	119.6	O2^{i}—S—O1	110.55 (15)
C9—C8—H8	119.6	O2—S—O1^{i}	110.55 (15)
C10—C9—C8	121.5 (4)	O2^{i}—S—O1^{i}	110.43 (15)
C10—C9—H9	119.3	O1—S—O1^{i}	101.4 (2)
C8—C9—H9	119.3	N1—Fe—N1^{i}	112.66 (15)
C9—C10—C11	119.4 (4)	N1—Fe—O1^{i}	154.52 (10)
C9—C10—H10	120.3	N1^{i}—Fe—O1^{i}	91.72 (10)
C11—C10—H10	120.3	N1—Fe—O1	91.72 (10)

N4—C11—C6	121.1 (3)	$N1^{i}$—Fe—O1	154.52 (10)
N4—C11—C10	119.7 (4)	$O1^{i}$—Fe—O1	65.47 (13)
C6—C11—C10	119.2 (3)	N1—Fe—$N2^{i}$	98.64 (10)
N4—C12—C5	121.7 (3)	$N1^{i}$—Fe—$N2^{i}$	76.08 (10)
N4—C12—C13	119.0 (3)	$O1^{i}$—Fe—$N2^{i}$	94.22 (10)
C5—C12—C13	119.3 (3)	O1—Fe—$N2^{i}$	93.64 (10)
C14—C13—C17	117.4 (3)	N1—Fe—N2	76.08 (10)
C14—C13—C12	123.2 (3)	$N1^{i}$—Fe—N2	98.64 (10)
C17—C13—C12	119.3 (3)	$O1^{i}$—Fe—N2	93.64 (10)
C15—C14—C13	120.0 (3)	O1—Fe—N2	94.22 (10)
C15—C14—H14	120.0	$N2^{i}$—Fe—N2	170.66 (15)
C13—C14—H14	120.0		

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—HW11···O1	0.80 (5)	1.99 (5)	2.734 (7)	155 (7)

Fig. 1

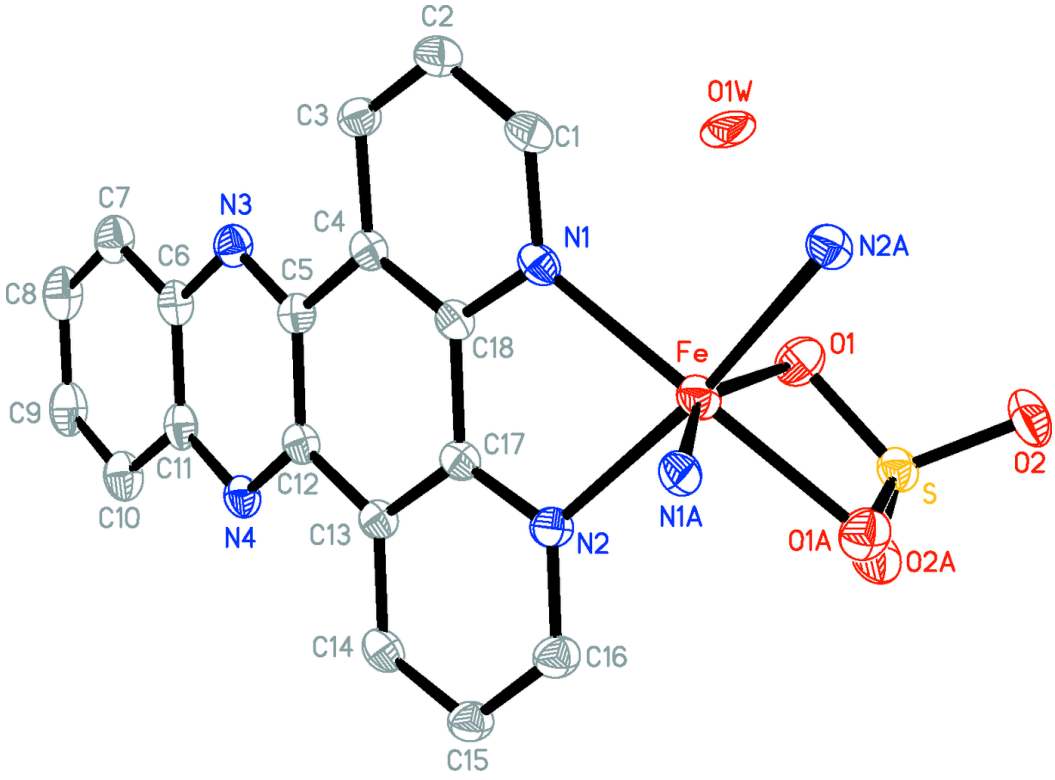
C2
C3
C1
O1W
C7
N3
C4
N1
N2A
C6
C5
C8
C18
Fe
O1
C9
C17
O2
C11
C12
S
C10
N4
C13
N1A
N2
O1A
O2A
C14
C16
C15

Fig. 2