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Poly[[[aqua(2,2-bipyridine)iron(II)]-μ_3-pyridine-3,4-dicarboxylato] monohydrate]

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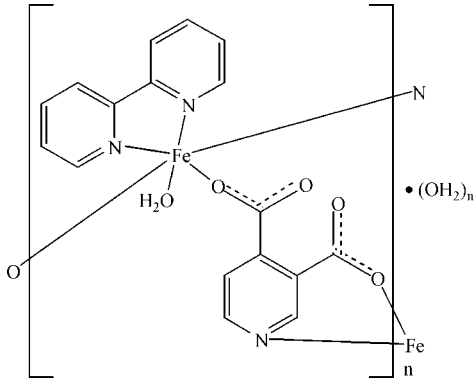
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.108; data-to-parameter ratio = 11.2.

In the title complex, $\{[Fe(C_7H_3NO_4)(C_{10}H_8N_2)(H_2O)]\cdot H_2O\}_n$, the divalent iron cation is in the centre of a distorted octahedron formed by three N and three O atoms. Two N atoms belong to the chelating 2,2-bipyridine ligand and the third to the pyridine-3,4-dicarboxylate ligand, while the O atoms belong to monodentate carboxylate groups of two different pyridine-3,4-dicarboxylate ligands and to one water molecule. The connectivity between the Fe^{II} cations and the pyridine-3,4-dicarboxylate units gives rise to corrugated layers parallel to the *ab* plane. The water molecules of crystallization are located between the layers. Medium-strong O—H···O hydrogen bonds between the water molecules and the layers stabilize the structure.



Experimental

Crystal data

$[Fe(C_7H_3NO_4)(C_{10}H_8N_2)(H_2O)]\cdot H_2O$
M_r = 413.17
Orthorhombic, *Pbca*
a = 15.7231 (13) Å
b = 12.630 (2) Å
c = 16.522 (2) Å
V = 3281.1 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.96 mm^{-1}
T = 293 (2) K
0.10 × 0.10 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.910, T_{max} = 0.910
24153 measured reflections
2870 independent reflections
2330 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.109
S = 1.00
2870 reflections
257 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

Table 1
Selected bond lengths (Å).

Fe1—O1^{i}	2.1192 (19)	Fe1—N2	2.224 (2)
Fe1—O3	2.1478 (18)	Fe1—N3	2.313 (2)
Fe1—O6	2.194 (2)	Fe1—N1^{ii}	2.361 (2)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H4W···O4^{iii}	0.824 (17)	2.02 (2)	2.827 (3)	168 (3)
O5—H3W···O2^{iv}	0.838 (17)	1.942 (17)	2.779 (3)	176 (3)
O6—H1W···O5^{v}	0.837 (17)	2.031 (18)	2.859 (3)	170 (4)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2122).

References

Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT*, *SHELXTL* (Version 5.10) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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Poly[[[aqua(2,2-bipyridine)iron(II)]-μ_3-pyridine-3,4-dicarboxylato] monohydrate]

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Comment

The molecular structure of the title compound is shown in Fig. 1. The Fe^{II} cation is in a distorted octahedral coordination by three O and three N atoms. The 2,2-bipyridine ligand provides two N atoms and the third N atom comes from the pyridine ring of the pyridine-3,4-dicarboxylate ligand. Two O atoms belong to the carboxylate groups of two different pyridine-3,4-dicarboxylate ligands and the third O atom is an aqua ligand. The Fe—O and Fe—N bond distances range from 2.1192 (19) to 2.194 (2) and from 2.224 (2) to 2.361 (2) Å, respectively. The connectivity between the Fe^{II} cations to three different pyridine-3,4-dicarboxylate ligands leads to the formation of corrugated layers parallel to the *ab* plane, as shown in Fig. 2. An additional sheet of water molecules is located between the polymeric layers. The structure is stabilized by medium-strong hydrogen bonds of the type O—H···O between the water molecules and the layers (Table 2).

Experimental

All chemicals were purchased from Acros without further purification. A mixture of $FeCl_2$ (98%, 0.5 mmol, 0.149 g), pyridine-3,4,-dicarboxylic acid (98%, 0.5 mmol, 0.162 g), and 2,2-bipyridine (98%, 0.5 mmol, 0.10 g) in a 20 ml mixture of water and ethanol (1:1) was sealed in an 30 ml Teflon-lined stainless autoclave, and kept at 423 K for 2 d. Colourless, block-shaped crystals of the title compound were obtained with an approximate yield of 20% after slowly cooling to room temperature. Anal. Calc. for $C_{17}H_{15}FeN_3O_6$: C 49.39, H 3.63, N 10.17, Fe 13.56%; Found: C 49.36, H 3.67, N 10.15, Fe 13.52%.

Refinement

The H atoms of the water molecules (except for HW2) were located from difference Fourier maps and were refined with distance restraints of d(H–H) = 1.38 (2) Å and d(O–H) = 0.83 (2) Å. All other H atoms were placed in calculated positions with a C–H bond distance of 0.93 Å (O–HW2 distance of 0.82 Å) and $U_{iso}(H) = 1.2U_{eq}$ of the respective carrier atom.

Figures

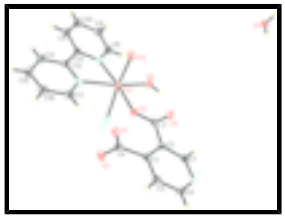

Fig. 1. A view of the coordination of the Fe^{II} cation in the structure of compound (I). Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: I) $-x + 1, -y, -z + 2$.]

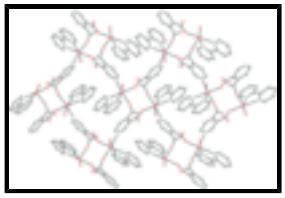

Fig. 2. two-dimensional wave-like layer of (I).

Poly[[[aqua(2,2-bipyridine)iron(II)]-μ$_3$-pyridine-3,4-dicarboxylato] monohydrate]

Crystal data

$[Fe(C_7H_3NO_4)(C_{10}H_8N_2)(H_2O)]\cdot H_2O$	F_{000} = 1696
M_r = 413.17	D_x = 1.673 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 2870 reflections
a = 15.7231 (13) Å	θ = 2.4–25.0º
b = 12.630 (2) Å	μ = 0.96 mm^{-1}
c = 16.522 (2) Å	*T* = 293 (2) K
V = 3281.1 (7) Å^3	Cube, colourless
Z = 8	0.10 × 0.10 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	2870 independent reflections
Radiation source: fine-focus sealed tube	2330 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.071
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	*h* = −18→18
T_{min} = 0.910, T_{max} = 0.910	*k* = −15→15
24153 measured reflections	*l* = −19→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.109	$w = 1/[\sigma^2(F_o^2) + (0.073P)^2 + 0.2586P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.001
2870 reflections	$\Delta\rho_{max}$ = 0.46 e Å^{-3}
257 parameters	$\Delta\rho_{min}$ = −0.68 e Å^{-3}
6 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 $F^2 > \sigma(F^2)$ 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}
Fe1	0.43846 (2)	0.18949 (3)	0.93122 (2)	0.02606 (16)
C1	0.33340 (16)	−0.0122 (2)	0.94563 (16)	0.0223 (6)
C2	0.29361 (15)	−0.10090 (19)	0.99332 (15)	0.0202 (5)
C3	0.31859 (16)	−0.1280 (2)	1.07044 (15)	0.0227 (6)
C4	0.38443 (16)	−0.0688 (2)	1.11962 (15)	0.0246 (6)
C5	0.27582 (17)	−0.2132 (2)	1.10595 (17)	0.0299 (6)
H5	0.2920	−0.2357	1.1573	0.036*
C6	0.20945 (18)	−0.2652 (2)	1.06631 (17)	0.0306 (7)
H6	0.1829	−0.3217	1.0922	0.037*
C7	0.22564 (16)	−0.1578 (2)	0.95950 (16)	0.0237 (6)
H7	0.2085	−0.1381	0.9078	0.028*
C8	0.47919 (18)	0.4002 (2)	0.82369 (17)	0.0318 (6)
H8	0.4357	0.3750	0.7907	0.038*
C9	0.52747 (19)	0.4850 (2)	0.79578 (18)	0.0346 (7)
H9	0.5158	0.5159	0.7459	0.042*
C10	0.5923 (2)	0.5219 (3)	0.84312 (19)	0.0435 (8)
H10	0.6261	0.5780	0.8260	0.052*
C11	0.6062 (2)	0.4748 (3)	0.91530 (19)	0.0414 (8)
H11	0.6506	0.4979	0.9481	0.050*
C12	0.55395 (16)	0.3913 (2)	0.94102 (16)	0.0244 (6)
C13	0.56429 (15)	0.3397 (2)	1.01905 (16)	0.0234 (6)
C14	0.61829 (18)	0.3804 (2)	1.07782 (17)	0.0325 (7)
H14	0.6500	0.4412	1.0680	0.039*
C15	0.6237 (2)	0.3293 (3)	1.14976 (19)	0.0402 (8)
H15	0.6588	0.3553	1.1905	0.048*
C16	0.5766 (2)	0.2386 (3)	1.16216 (18)	0.0404 (8)
H16	0.5805	0.2028	1.2112	0.049*
C17	0.52435 (18)	0.2010 (2)	1.10260 (18)	0.0316 (7)
H17	0.4929	0.1398	1.1117	0.038*
N1	0.18306 (13)	−0.23758 (17)	0.99403 (13)	0.0272 (5)
N2	0.51787 (13)	0.24994 (17)	1.03256 (13)	0.0240 (5)

N3	0.49137 (13)	0.35332 (17)	0.89404 (13)	0.0246 (5)
O1	0.45895 (11)	−0.10617 (16)	1.12228 (12)	0.0349 (5)
O2	0.35844 (13)	0.00840 (16)	1.15898 (12)	0.0387 (5)
O3	0.38972 (11)	0.04329 (13)	0.97871 (11)	0.0269 (4)
O4	0.30668 (14)	0.00132 (15)	0.87610 (11)	0.0356 (5)
O5	0.26467 (14)	0.13856 (16)	0.26104 (13)	0.0363 (5)
O6	0.36334 (13)	0.18725 (15)	0.81936 (12)	0.0311 (5)
H2W	0.3354	0.1325	0.8173	0.047*
H4W	0.2378 (19)	0.104 (2)	0.2948 (16)	0.055 (11)*
H3W	0.2908 (19)	0.097 (2)	0.2303 (16)	0.048 (10)*
H1W	0.3316 (19)	0.2383 (15)	0.808 (2)	0.053 (11)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0263 (3)	0.0223 (3)	0.0296 (3)	−0.00241 (15)	−0.00159 (16)	0.00010 (15)
C1	0.0208 (13)	0.0166 (13)	0.0296 (15)	0.0019 (10)	0.0021 (11)	−0.0009 (11)
C2	0.0206 (12)	0.0165 (13)	0.0236 (13)	0.0008 (10)	0.0036 (10)	−0.0017 (10)
C3	0.0200 (13)	0.0213 (14)	0.0268 (14)	0.0055 (10)	0.0052 (11)	−0.0015 (10)
C4	0.0291 (15)	0.0265 (14)	0.0183 (14)	0.0011 (12)	0.0033 (11)	0.0035 (11)
C5	0.0327 (15)	0.0307 (15)	0.0263 (15)	0.0007 (12)	−0.0020 (12)	0.0066 (12)
C6	0.0307 (15)	0.0261 (15)	0.0349 (17)	−0.0076 (12)	0.0011 (12)	0.0069 (12)
C7	0.0276 (14)	0.0230 (13)	0.0205 (13)	−0.0002 (11)	0.0004 (11)	0.0006 (11)
C8	0.0327 (15)	0.0342 (16)	0.0284 (15)	−0.0051 (12)	−0.0047 (12)	0.0009 (12)
C9	0.0442 (17)	0.0329 (16)	0.0269 (16)	−0.0037 (13)	0.0011 (13)	0.0070 (12)
C10	0.0533 (19)	0.0393 (19)	0.0379 (19)	−0.0221 (15)	0.0009 (16)	0.0075 (14)
C11	0.0441 (18)	0.0443 (19)	0.0359 (18)	−0.0251 (15)	−0.0066 (14)	0.0037 (14)
C12	0.0241 (13)	0.0203 (14)	0.0288 (15)	−0.0020 (11)	0.0012 (11)	−0.0037 (11)
C13	0.0208 (13)	0.0241 (14)	0.0251 (14)	0.0009 (11)	0.0012 (11)	−0.0027 (11)
C14	0.0327 (16)	0.0292 (15)	0.0356 (17)	−0.0083 (12)	−0.0054 (13)	−0.0012 (12)
C15	0.0382 (17)	0.048 (2)	0.0342 (17)	−0.0031 (15)	−0.0108 (14)	−0.0014 (14)
C16	0.0422 (18)	0.050 (2)	0.0290 (16)	0.0025 (15)	−0.0029 (14)	0.0112 (14)
C17	0.0298 (15)	0.0318 (16)	0.0333 (16)	−0.0024 (12)	0.0021 (13)	0.0077 (12)
N1	0.0258 (12)	0.0261 (13)	0.0298 (13)	−0.0037 (10)	−0.0015 (10)	0.0004 (10)
N2	0.0230 (11)	0.0234 (12)	0.0255 (12)	−0.0004 (9)	0.0011 (9)	−0.0001 (9)
N3	0.0241 (12)	0.0252 (12)	0.0246 (12)	−0.0033 (9)	−0.0002 (9)	0.0025 (10)
O1	0.0242 (10)	0.0425 (12)	0.0380 (12)	0.0104 (9)	−0.0030 (9)	−0.0105 (9)
O2	0.0437 (12)	0.0366 (12)	0.0359 (12)	0.0109 (10)	−0.0016 (10)	−0.0147 (9)
O3	0.0289 (10)	0.0221 (10)	0.0298 (10)	−0.0060 (8)	−0.0018 (8)	0.0018 (8)
O4	0.0497 (12)	0.0317 (11)	0.0255 (11)	−0.0131 (9)	−0.0087 (9)	0.0063 (8)
O5	0.0472 (13)	0.0332 (12)	0.0285 (11)	0.0011 (10)	0.0065 (10)	0.0000 (10)
O6	0.0346 (11)	0.0268 (11)	0.0317 (11)	−0.0041 (9)	−0.0044 (9)	−0.0007 (8)

Geometric parameters (Å, °)

Fe1—O1^{i}	2.1192 (19)	C9—C10	1.367 (4)
Fe1—O3	2.1478 (18)	C9—H9	0.9300
Fe1—O6	2.194 (2)	C10—C11	1.350 (4)

Fe1—N2	2.224 (2)	C10—H10	0.9300
Fe1—N3	2.313 (2)	C11—C12	1.404 (4)
Fe1—N1^{ii}	2.361 (2)	C11—H11	0.9300
C1—O4	1.235 (3)	C12—N3	1.342 (3)
C1—O3	1.255 (3)	C12—C13	1.454 (4)
C1—C2	1.505 (4)	C13—N2	1.367 (3)
C2—C3	1.377 (4)	C13—C14	1.388 (4)
C2—C7	1.404 (4)	C14—C15	1.355 (4)
C3—C5	1.397 (4)	C14—H14	0.9300
C3—C4	1.514 (4)	C15—C16	1.380 (5)
C4—O2	1.241 (3)	C15—H15	0.9300
C4—O1	1.264 (3)	C16—C17	1.367 (4)
C5—C6	1.397 (4)	C16—H16	0.9300
C5—H5	0.9300	C17—N2	1.316 (4)
C6—N1	1.312 (3)	C17—H17	0.9300
C6—H6	0.9300	N1—Fe1^{iii}	2.361 (2)
C7—N1	1.337 (3)	O1—Fe1^{i}	2.1192 (18)
C7—H7	0.9300	O5—H4W	0.824 (17)
C8—N3	1.318 (4)	O5—H3W	0.838 (17)
C8—C9	1.391 (4)	O6—H2W	0.8200
C8—H8	0.9300	O6—H1W	0.837 (17)
O1^{i}—Fe1—O3	89.83 (8)	C8—C9—H9	120.7
O1^{i}—Fe1—O6	92.98 (8)	C11—C10—C9	118.4 (3)
O3—Fe1—O6	96.01 (7)	C11—C10—H10	120.8
O1^{i}—Fe1—N2	93.28 (8)	C9—C10—H10	120.8
O3—Fe1—N2	102.73 (8)	C10—C11—C12	120.2 (3)
O6—Fe1—N2	160.24 (8)	C10—C11—H11	119.9
O1^{i}—Fe1—N3	93.42 (8)	C12—C11—H11	119.9
O3—Fe1—N3	173.94 (8)	N3—C12—C11	121.5 (3)
O6—Fe1—N3	88.94 (7)	N3—C12—C13	115.8 (2)
N2—Fe1—N3	72.00 (8)	C11—C12—C13	122.7 (2)
O1^{i}—Fe1—N1^{ii}	173.18 (8)	N2—C13—C14	121.3 (2)
O3—Fe1—N1^{ii}	83.46 (7)	N2—C13—C12	117.2 (2)
O6—Fe1—N1^{ii}	86.54 (8)	C14—C13—C12	121.5 (3)
N2—Fe1—N1^{ii}	89.40 (8)	C15—C14—C13	118.4 (3)
N3—Fe1—N1^{ii}	93.37 (8)	C15—C14—H14	120.8
O4—C1—O3	124.6 (2)	C13—C14—H14	120.8
O4—C1—C2	116.7 (2)	C14—C15—C16	119.5 (3)
O3—C1—C2	118.8 (2)	C14—C15—H15	120.3
C3—C2—C7	117.3 (2)	C16—C15—H15	120.3
C3—C2—C1	123.4 (2)	C17—C16—C15	120.3 (3)
C7—C2—C1	119.3 (2)	C17—C16—H16	119.9
C2—C3—C5	116.3 (2)	C15—C16—H16	119.9
C2—C3—C4	124.7 (2)	N2—C17—C16	121.1 (3)
C5—C3—C4	118.9 (2)	N2—C17—H17	119.5
O2—C4—O1	125.5 (3)	C16—C17—H17	119.5

O2—C4—C3	116.4 (2)	C6—N1—C7	115.5 (2)
O1—C4—C3	117.9 (2)	C6—N1—Fe1^{iii}	123.53 (18)
C6—C5—C3	121.7 (3)	C7—N1—Fe1^{iii}	120.76 (17)
C6—C5—H5	119.2	C17—N2—C13	119.5 (2)
C3—C5—H5	119.2	C17—N2—Fe1	122.96 (19)
N1—C6—C5	122.5 (3)	C13—N2—Fe1	117.49 (17)
N1—C6—H6	118.7	C8—N3—C12	117.1 (2)
C5—C6—H6	118.7	C8—N3—Fe1	125.69 (18)
N1—C7—C2	126.7 (2)	C12—N3—Fe1	115.46 (17)
N1—C7—H7	116.7	C4—O1—Fe1^{i}	151.22 (18)
C2—C7—H7	116.7	C1—O3—Fe1	124.97 (17)
N3—C8—C9	124.0 (3)	H4W—O5—H3W	109 (2)
N3—C8—H8	118.0	Fe1—O6—H2W	109.5
C9—C8—H8	118.0	Fe1—O6—H1W	120 (2)
C10—C9—C8	118.7 (3)	H2W—O6—H1W	108.7
C10—C9—H9	120.7		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H4W···O4^{iv}	0.824 (17)	2.02 (2)	2.827 (3)	168 (3)
O5—H3W···O2^{v}	0.838 (17)	1.942 (17)	2.779 (3)	176 (3)
O6—H1W···O5^{vi}	0.837 (17)	2.031 (18)	2.859 (3)	170 (4)

Symmetry codes: (iv) −*x*+1/2, −*y*, *z*−1/2; (v) *x*, *y*, *z*−1; (vi) *x*, −*y*+1/2, *z*+1/2.

Fig. 1

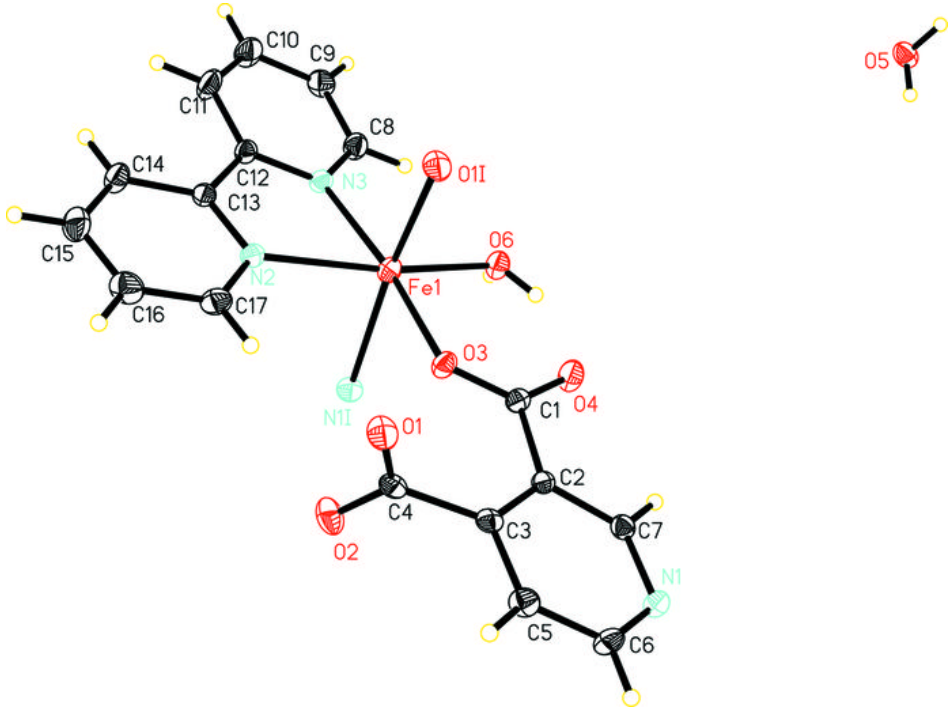
C10
C9
C11
C8
C14
C12
N3
O1I
C13
C15
N2
Fe1
O6
C16
C17
O3
N1I
C1
O4
O1
C2
C4
C3
C7
O2
N1
C5
C6
O5

Fig. 2