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Tetraaquabis(4,4′-bipyridine)iron(II) pyridine-2,6-dicarboxylate tetrahydrate

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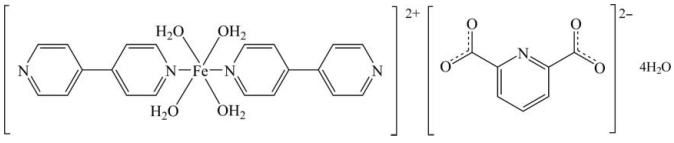
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.037; wR factor = 0.105; data-to-parameter ratio = 7.0.

The title compound, $[Fe(C_{10}H_8N_2)_2(H_2O)_4](C_7H_3NO_4)\cdot 4H_2O$, prepared by hydrothermal synthesis, contains isolated tetraaquabis(4,4′-bipyridine)iron(II) cations, comprising two 4,4′-bipyridine molecules bound to Fe^{II} in a *trans* manner. The cations lie in layers, with pyridine-2,6-dicarboxylate dianions and water molecules forming an extensive hydrogen-bond network between them. The cations exhibit noncrystallographic inversion symmetry. The crystal was a partial inversion twin.

Related literature

For literature concerning metal–organic frameworks, see: Evans, Ngo & Lin (2001); Vioux *et al.* (2004); Sanchez *et al.* (2003); Evans & Lin (2001); Jannasch (2003); Javaid *et al.* (2001); Honma *et al.* (2001); Sudik *et al.* (2005); Rowsell *et al.* (2004); Kitaura *et al.* (2004); Suzuki *et al.* (2002).



Experimental

Crystal data

$[Fe(C_{10}H_8N_2)_2(H_2O)_4](C_7H_3NO_4)\cdot 4H_2O$
M_r = 677.45
Monoclinic, Cc
a = 18.201 (1) Å
b = 6.900 (1) Å
c = 25.010 (1) Å
β = 99.40 (3)°
V = 3098.9 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.56 mm^{-1}
T = 293 (2) K
0.10 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
5104 measured reflections
3180 independent reflections
2946 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.105
S = 1.05
3180 reflections
455 parameters
26 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack (1983), with 530 Friedel pairs
Flack parameter: 0.36 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···O8^{i}	0.82 (1)	2.06 (3)	2.831 (7)	156 (6)
O1—H12···N3^{ii}	0.82 (1)	1.98 (3)	2.759 (5)	158 (6)
O2—H21···O5^{iii}	0.82 (1)	2.00 (2)	2.806 (6)	172 (5)
O2—H22···O7^{iii}	0.82 (1)	1.96 (1)	2.781 (6)	178 (8)
O3—H31···O5^{iv}	0.81 (1)	2.04 (1)	2.851 (7)	175 (7)
O3—H32···N5^{v}	0.81 (1)	1.96 (2)	2.745 (5)	162 (6)
O4—H41···O8	0.82 (1)	1.97 (1)	2.785 (5)	177 (7)
O4—H42···O11^{vi}	0.82 (1)	1.93 (2)	2.736 (5)	170 (5)
O5—H52···O6	0.82 (1)	1.93 (3)	2.739 (8)	170 (7)
O5—H51···O11^{vi}	0.82 (1)	1.94 (2)	2.742 (7)	166 (7)
O6—H62···O9	0.82 (1)	2.46 (8)	2.952 (6)	120 (7)
O6—H61···O10^{i}	0.82 (1)	2.03 (2)	2.827 (5)	164 (7)
O7—H72···O5^{vii}	0.82 (1)	2.57 (2)	3.349 (7)	159 (5)
O7—H71···O9	0.82 (1)	1.81 (2)	2.622 (6)	168 (7)
O8—H81···O7	0.82 (1)	2.01 (3)	2.817 (7)	170 (6)
O8—H82···O12^{viii}	0.81 (1)	1.97 (2)	2.768 (6)	170 (7)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXL97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXS97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); 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: BI2196).

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supplementary materials

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Tetraaquabis(4,4'-bipyridine)iron(II) pyridine-2,6-dicarboxylate tetrahydrate

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Comment

Metal-organic frameworks have applications in catalysis, optical materials, membranes, and sorption (Evans, Ngo & Lin, 2001; Vioux *et al.*, 2004; Sanchez *et al.*, 2003; Evans & Lin, 2001; Jannasch, 2003; Javaid *et al.*, 2001; Honma *et al.*, 2001; Sudik *et al.*, 2005; Rowsell *et al.*, 2004; Kitaura *et al.*, 2004; Suzuki *et al.*, 2002).

Design of metal-organic frameworks often utilizes a metal cation, metal cluster, or metal oxide substructure as a node, from which rigid or flexible multitopic organic ligands radiate to act as tethers to adjacent nodes for the bottom-up construction of complex extended architectures. While a variety of organic molecules have been investigated as potential tethers, materials incorporating multitopic carboxylates and pyridine ligands have been employed most extensively.

In the title compound, Fe^{II} is hexa-coordinated in an octahedral manner by four water molecules in the equatorial plane and two N atoms in the axial positions from two 4,4'-bipyridine molecules (Fig. 1). The Fe—N and Fe—O bond lengths are in the range 2.265 (4)–2.266 (4) and 2.161 (3)–2.202 (4) Å, respectively. The cations lie in layers in the *bc* planes. Pyridine-2,6-dicarboxylate anions and water molecules lie between these layers, forming an extensive hydrogen-bond network.

Experimental

A mixture of $FeCl_2$ (0.5 mmol), pyridine-2,6-dicarboxylic acid (0.5 mmol), NaOH (1 mmol), 4,4'-bipyridine (0.5 mmol), H_2O (8 ml) and ethanol (8 ml) were place in a 25 ml Teflon-lined stainless steel autoclave and heated at 433 K for two days. On cooling to room temperature, blue crystals were obtained with a yield of 12%. Elemental analysis calculated: C 47.83, H 5.17, N 10.33, Fe 8.27%; found: C 47.87, H 5.21, N 10.27, Fe 8.16%.

Refinement

H atoms bound to C atoms were placed geometrically and refined as riding atoms with C—H = 0.93Å and U_{iso}(H) = 1.2U_{eq}(C). H atoms of the water molecules were located from difference Fourier maps and were refined with distance restraints of O—H = 0.82 (1) Å and H···H = 1.35 (2) Å, and with U_{iso}(H) = 1.5U_{eq}(O). The $[Fe(C_{10}H_8N_2)_2(H_2O)_4]^{2+}$ cations exhibit non-crystallographic inversion symmetry. The refined Flack parameter (Flack, 1983) from 530 Freidel pairs is 0.36 (3).

Figures

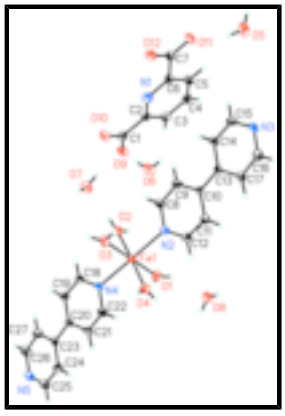


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids for non-H atoms.

Tetraaquabis(4,4'-bipyridine)iron(II) pyridine-2,6-dicarboxylate tetrahydrate

Crystal data

$[Fe(C_{10}H_8N_2)_2(H_2O)_4](C_7H_3NO_4)\cdot 4H_2O$	F_{000} = 1416
M_r = 677.45	D_x = 1.452 Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C -2yc	Cell parameters from 3290 reflections
a = 18.201 (1) Å	θ = 2.0–55.0º
b = 6.900 (1) Å	μ = 0.56 mm^{-1}
c = 25.010 (1) Å	*T* = 293 (2) K
β = 99.40 (3)º	Block, blue
V = 3098.9 (5) Å^3	0.10 × 0.10 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2946 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.018
Monochromator: graphite	θ_{max} = 25.1º
T = 293(2) K	θ_{min} = 1.7º
ω scans	*h* = −19→21
Absorption correction: none	*k* = −8→4
5104 measured reflections	*l* = −29→12
3180 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of

	independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 1.0906P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.105$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.35$ e Å^{-3}
3180 reflections	$\Delta\rho_{min} = -0.38$ e Å^{-3}
455 parameters	Extinction correction: none
26 restraints	Absolute structure: Flack (1983), with 530 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.36 (3)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2$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	0.82218 (7)	0.73736 (9)	0.62280 (5)	0.03412 (16)
C1	0.5731 (3)	0.1741 (7)	0.8192 (2)	0.0385 (11)
C2	0.5712 (5)	0.2657 (6)	0.8733 (4)	0.0335 (7)
C3	0.5690 (3)	0.4671 (6)	0.8799 (2)	0.0395 (10)
H3A	0.5680	0.5473	0.8498	0.047*
C4	0.5683 (3)	0.5490 (6)	0.9306 (2)	0.0405 (10)
H4A	0.5663	0.6829	0.9343	0.049*
C5	0.5706 (2)	0.4311 (6)	0.97538 (19)	0.0391 (10)
H5A	0.5703	0.4866	1.0092	0.047*
C6	0.5735 (3)	0.2278 (6)	0.9706 (3)	0.0342 (13)
C7	0.5791 (2)	0.0996 (6)	1.02025 (18)	0.0347 (9)
C8	0.8773 (3)	0.8254 (9)	0.7460 (2)	0.0476 (13)
H8A	0.9228	0.8122	0.7340	0.057*
C9	0.8770 (3)	0.8418 (8)	0.8006 (2)	0.0448 (12)
H9A	0.9220	0.8416	0.8244	0.054*
C10	0.8112 (3)	0.8587 (6)	0.82083 (18)	0.0279 (9)
C11	0.7468 (3)	0.8708 (8)	0.7815 (2)	0.0481 (13)
H11A	0.7007	0.8911	0.7920	0.058*
C12	0.7517 (3)	0.8526 (9)	0.7275 (2)	0.0503 (14)
H12A	0.7079	0.8582	0.7025	0.060*

C13	0.8081 (2)	0.8626 (6)	0.87930 (18)	0.0287 (10)
C14	0.8730 (3)	0.8650 (7)	0.91726 (18)	0.0379 (11)
H14A	0.9194	0.8654	0.9062	0.045*
C15	0.8673 (3)	0.8668 (7)	0.9720 (2)	0.0397 (11)
H15A	0.9111	0.8685	0.9971	0.048*
C16	0.7424 (3)	0.8637 (7)	0.9536 (2)	0.0450 (12)
H16A	0.6970	0.8625	0.9661	0.054*
C17	0.7409 (3)	0.8628 (7)	0.8989 (2)	0.0402 (11)
H17A	0.6958	0.8623	0.8752	0.048*
C18	0.8956 (3)	0.6415 (7)	0.51671 (19)	0.0387 (11)
H18A	0.9401	0.6401	0.5410	0.046*
C19	0.8987 (3)	0.6324 (8)	0.46210 (18)	0.0414 (12)
H19A	0.9443	0.6245	0.4501	0.050*
C20	0.8331 (3)	0.6353 (6)	0.42539 (18)	0.0285 (10)
C21	0.7673 (3)	0.6404 (7)	0.4459 (2)	0.0418 (12)
H21A	0.7218	0.6382	0.4227	0.050*
C22	0.7698 (3)	0.6488 (8)	0.5007 (2)	0.0457 (12)
H22A	0.7247	0.6521	0.5137	0.055*
C23	0.8328 (3)	0.6379 (6)	0.36586 (18)	0.0294 (10)
C24	0.7669 (3)	0.6306 (8)	0.3286 (2)	0.0478 (13)
H24A	0.7214	0.6219	0.3408	0.057*
C25	0.7689 (3)	0.6362 (8)	0.2741 (2)	0.0482 (13)
H25A	0.7239	0.6311	0.2502	0.058*
C26	0.8947 (4)	0.6555 (9)	0.2887 (2)	0.0576 (15)
H26A	0.9394	0.6641	0.2753	0.069*
C27	0.8981 (3)	0.6507 (8)	0.3441 (2)	0.0467 (13)
H27A	0.9439	0.6560	0.3668	0.056*
N1	0.5734 (3)	0.1479 (6)	0.91981 (19)	0.0466 (10)
N2	0.8150 (2)	0.8275 (5)	0.70892 (16)	0.0345 (9)
N3	0.8037 (3)	0.8662 (5)	0.99043 (16)	0.0383 (10)
N4	0.8316 (2)	0.6524 (5)	0.53671 (15)	0.0322 (9)
N5	0.8321 (3)	0.6486 (5)	0.25322 (17)	0.0424 (10)
O1	0.7848 (2)	1.0205 (5)	0.59307 (15)	0.0469 (9)
H11	0.757 (3)	1.098 (6)	0.605 (2)	0.070*
H12	0.799 (3)	1.074 (7)	0.5671 (17)	0.070*
O2	0.9377 (2)	0.8418 (5)	0.63036 (16)	0.0457 (8)
H21	0.947 (3)	0.957 (2)	0.631 (3)	0.069*
H22	0.974 (2)	0.785 (6)	0.646 (3)	0.069*
O3	0.8599 (2)	0.4528 (5)	0.65227 (14)	0.0477 (9)
H31	0.894 (2)	0.384 (8)	0.646 (2)	0.072*
H32	0.856 (3)	0.445 (8)	0.6841 (8)	0.072*
O4	0.70693 (19)	0.6295 (5)	0.61352 (15)	0.0407 (8)
H41	0.693 (3)	0.516 (2)	0.611 (3)	0.061*
H42	0.672 (2)	0.697 (6)	0.600 (3)	0.061*
O5	0.4859 (3)	0.7286 (6)	0.6359 (2)	0.0577 (14)
H51	0.511 (3)	0.738 (9)	0.612 (2)	0.087*
H52	0.514 (3)	0.724 (11)	0.6650 (14)	0.087*
O6	0.5641 (3)	0.6916 (7)	0.73867 (17)	0.0714 (11)
H61	0.569 (4)	0.761 (9)	0.7655 (19)	0.107*

H62	0.597 (3)	0.610 (9)	0.744 (3)	0.107*
O7	0.5641 (3)	0.1491 (5)	0.68152 (16)	0.0554 (10)
H71	0.571 (4)	0.180 (8)	0.7137 (8)	0.083*
H72	0.543 (4)	0.044 (5)	0.679 (2)	0.083*
O8	0.6593 (3)	0.2454 (5)	0.6074 (2)	0.0487 (13)
H81	0.636 (3)	0.220 (9)	0.6318 (17)	0.073*
H82	0.635 (3)	0.209 (9)	0.5790 (13)	0.073*
O9	0.5726 (3)	0.2898 (6)	0.77972 (17)	0.0533 (11)
O10	0.5739 (2)	−0.0063 (5)	0.81586 (14)	0.0472 (8)
O11	0.5811 (2)	0.1826 (5)	1.06512 (14)	0.0470 (9)
O12	0.5839 (2)	−0.0786 (4)	1.01353 (13)	0.0453 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0355 (3)	0.0432 (3)	0.0237 (2)	0.0010 (3)	0.00518 (18)	0.0011 (3)
C1	0.028 (2)	0.049 (3)	0.038 (3)	0.005 (2)	0.0020 (19)	−0.001 (2)
C2	0.0201 (14)	0.0445 (18)	0.0342 (17)	0.002 (2)	−0.0004 (12)	0.004 (2)
C3	0.034 (2)	0.0435 (19)	0.039 (3)	0.002 (2)	−0.0002 (19)	0.003 (2)
C4	0.040 (2)	0.032 (2)	0.048 (3)	0.0044 (19)	0.002 (2)	−0.003 (2)
C5	0.036 (2)	0.044 (2)	0.037 (2)	−0.001 (2)	0.0022 (19)	−0.012 (2)
C6	0.026 (2)	0.041 (2)	0.036 (3)	−0.0011 (19)	0.006 (2)	−0.007 (2)
C7	0.028 (2)	0.045 (2)	0.032 (2)	−0.0034 (19)	0.0064 (17)	−0.0061 (19)
C8	0.030 (3)	0.087 (4)	0.027 (3)	−0.003 (3)	0.010 (2)	−0.007 (3)
C9	0.024 (2)	0.081 (4)	0.027 (3)	−0.002 (2)	−0.002 (2)	−0.005 (2)
C10	0.030 (2)	0.0301 (19)	0.023 (2)	0.0014 (17)	0.0050 (19)	−0.0006 (17)
C11	0.029 (3)	0.081 (3)	0.036 (3)	0.007 (2)	0.012 (2)	−0.009 (3)
C12	0.039 (3)	0.081 (4)	0.029 (3)	0.014 (3)	0.001 (2)	−0.005 (2)
C13	0.033 (3)	0.0258 (18)	0.026 (2)	0.0020 (17)	0.003 (2)	−0.0030 (16)
C14	0.035 (3)	0.059 (3)	0.020 (2)	−0.001 (2)	0.006 (2)	−0.003 (2)
C15	0.037 (3)	0.051 (2)	0.028 (3)	−0.004 (2)	−0.006 (2)	−0.003 (2)
C16	0.044 (3)	0.059 (3)	0.035 (3)	0.002 (2)	0.016 (2)	0.000 (2)
C17	0.035 (3)	0.057 (3)	0.030 (3)	0.003 (2)	0.009 (2)	−0.004 (2)
C18	0.030 (3)	0.060 (3)	0.025 (2)	0.008 (2)	0.000 (2)	−0.005 (2)
C19	0.033 (3)	0.070 (3)	0.021 (2)	0.004 (2)	0.003 (2)	−0.003 (2)
C20	0.035 (3)	0.0285 (18)	0.022 (2)	0.0008 (18)	0.0047 (18)	−0.0012 (16)
C21	0.030 (3)	0.070 (3)	0.025 (2)	−0.007 (2)	0.003 (2)	−0.006 (2)
C22	0.033 (3)	0.074 (4)	0.031 (3)	−0.008 (3)	0.008 (2)	−0.007 (2)
C23	0.034 (3)	0.0307 (19)	0.025 (2)	0.0023 (18)	0.0105 (19)	−0.0012 (17)
C24	0.034 (3)	0.075 (3)	0.035 (3)	0.002 (2)	0.008 (2)	0.000 (2)
C25	0.047 (3)	0.071 (3)	0.028 (3)	0.003 (3)	0.007 (2)	0.001 (2)
C26	0.054 (4)	0.090 (4)	0.034 (3)	−0.008 (3)	0.022 (3)	−0.008 (3)
C27	0.034 (3)	0.074 (3)	0.033 (3)	−0.005 (2)	0.006 (2)	−0.005 (2)
N1	0.036 (2)	0.056 (2)	0.048 (3)	0.003 (2)	0.0065 (19)	−0.002 (2)
N2	0.035 (2)	0.042 (2)	0.027 (2)	0.0029 (17)	0.0048 (17)	−0.0016 (16)
N3	0.050 (3)	0.044 (2)	0.023 (2)	0.0024 (18)	0.0102 (18)	−0.0008 (16)
N4	0.033 (2)	0.041 (2)	0.022 (2)	−0.0026 (16)	0.0033 (17)	−0.0015 (16)
N5	0.054 (3)	0.046 (2)	0.028 (2)	−0.005 (2)	0.0100 (19)	−0.0045 (18)

O1	0.061 (2)	0.0487 (18)	0.0351 (19)	0.0179 (17)	0.0201 (17)	0.0103 (15)
O2	0.037 (2)	0.0534 (18)	0.047 (2)	−0.0058 (16)	0.0062 (17)	−0.0034 (17)
O3	0.069 (3)	0.0472 (18)	0.0299 (17)	0.0214 (17)	0.0172 (17)	0.0102 (15)
O4	0.036 (2)	0.0444 (16)	0.0406 (19)	−0.0041 (14)	0.0036 (16)	0.0007 (15)
O5	0.054 (3)	0.067 (3)	0.058 (3)	0.008 (2)	0.026 (3)	0.014 (2)
O6	0.083 (3)	0.084 (3)	0.046 (2)	−0.006 (3)	0.010 (2)	−0.011 (2)
O7	0.070 (3)	0.053 (2)	0.045 (2)	−0.009 (2)	0.013 (2)	−0.0043 (18)
O8	0.050 (3)	0.054 (3)	0.042 (3)	−0.0061 (16)	0.010 (2)	−0.0065 (16)
O9	0.067 (3)	0.065 (2)	0.028 (2)	0.006 (2)	0.007 (2)	0.0021 (19)
O10	0.055 (2)	0.0498 (19)	0.0355 (17)	0.0017 (16)	0.0048 (15)	−0.0096 (15)
O11	0.052 (2)	0.059 (2)	0.0322 (19)	−0.0061 (18)	0.0110 (16)	−0.0121 (16)
O12	0.057 (2)	0.0426 (17)	0.0350 (17)	0.0006 (16)	0.0052 (15)	−0.0001 (14)

Geometric parameters (Å, °)

Fe1—O1	2.161 (3)	C16—H16A	0.930
Fe1—O3	2.169 (3)	C17—H17A	0.930
Fe1—O2	2.202 (4)	C18—N4	1.342 (6)
Fe1—O4	2.202 (4)	C18—C19	1.377 (7)
Fe1—N4	2.265 (4)	C18—H18A	0.930
Fe1—N2	2.266 (4)	C19—C20	1.381 (6)
C1—O10	1.248 (6)	C19—H19A	0.930
C1—O9	1.268 (7)	C20—C21	1.379 (7)
C1—C2	1.499 (10)	C20—C23	1.488 (6)
C2—C3	1.401 (6)	C21—C22	1.366 (7)
C2—N1	1.414 (9)	C21—H21A	0.930
C3—C4	1.390 (7)	C22—N4	1.321 (6)
C3—H3A	0.930	C22—H22A	0.930
C4—C5	1.380 (7)	C23—C27	1.389 (7)
C4—H4A	0.930	C23—C24	1.394 (7)
C5—C6	1.410 (6)	C24—C25	1.371 (7)
C5—H5A	0.930	C24—H24A	0.930
C6—N1	1.385 (7)	C25—N5	1.341 (7)
C6—C7	1.514 (7)	C25—H25A	0.930
C7—O12	1.246 (5)	C26—N5	1.327 (8)
C7—O11	1.255 (5)	C26—C27	1.378 (8)
C8—N2	1.342 (6)	C26—H26A	0.930
C8—C9	1.371 (7)	C27—H27A	0.930
C8—H8A	0.930	O1—H11	0.82 (1)
C9—C10	1.379 (7)	O1—H12	0.82 (1)
C9—H9A	0.930	O2—H21	0.82 (1)
C10—C11	1.405 (7)	O2—H22	0.82 (1)
C10—C13	1.473 (6)	O3—H31	0.81 (1)
C11—C12	1.373 (7)	O3—H32	0.81 (1)
C11—H11A	0.930	O4—H41	0.82 (1)
C12—N2	1.323 (7)	O4—H42	0.82 (1)
C12—H12A	0.930	O5—H51	0.82 (1)
C13—C17	1.389 (7)	O5—H52	0.82 (1)
C13—C14	1.391 (6)	O6—H61	0.82 (1)

C14—C15	1.390 (7)	O6—H62	0.82 (1)
C14—H14A	0.930	O7—H71	0.82 (1)
C15—N3	1.314 (7)	O7—H72	0.82 (1)
C15—H15A	0.930	O8—H81	0.82 (1)
C16—N3	1.326 (7)	O8—H82	0.81 (1)
C16—C17	1.366 (7)		
O1—Fe1—O3	179.73 (19)	N3—C16—H16A	117.5
O1—Fe1—O2	88.66 (15)	C17—C16—H16A	117.5
O3—Fe1—O2	91.28 (16)	C16—C17—C13	118.6 (5)
O1—Fe1—O4	91.61 (15)	C16—C17—H17A	120.7
O3—Fe1—O4	88.44 (15)	C13—C17—H17A	120.7
O2—Fe1—O4	178.69 (18)	N4—C18—C19	123.4 (4)
O1—Fe1—N4	88.59 (14)	N4—C18—H18A	118.3
O3—Fe1—N4	91.14 (13)	C19—C18—H18A	118.3
O2—Fe1—N4	86.94 (15)	C18—C19—C20	119.2 (5)
O4—Fe1—N4	91.79 (15)	C18—C19—H19A	120.4
O1—Fe1—N2	90.87 (14)	C20—C19—H19A	120.4
O3—Fe1—N2	89.40 (14)	C21—C20—C19	117.5 (4)
O2—Fe1—N2	91.78 (16)	C21—C20—C23	120.7 (4)
O4—Fe1—N2	89.50 (15)	C19—C20—C23	121.8 (4)
N4—Fe1—N2	178.62 (18)	C22—C21—C20	119.1 (5)
O10—C1—O9	125.1 (5)	C22—C21—H21A	120.5
O10—C1—C2	118.9 (5)	C20—C21—H21A	120.5
O9—C1—C2	116.0 (4)	N4—C22—C21	124.7 (5)
C3—C2—N1	118.1 (7)	N4—C22—H22A	117.7
C3—C2—C1	122.0 (6)	C21—C22—H22A	117.7
N1—C2—C1	119.9 (4)	C27—C23—C24	116.0 (5)
C4—C3—C2	121.0 (6)	C27—C23—C20	121.9 (4)
C4—C3—H3A	119.5	C24—C23—C20	122.1 (4)
C2—C3—H3A	119.5	C25—C24—C23	120.3 (5)
C5—C4—C3	119.9 (4)	C25—C24—H24A	119.8
C5—C4—H4A	120.1	C23—C24—H24A	119.8
C3—C4—H4A	120.1	N5—C25—C24	123.5 (5)
C4—C5—C6	121.0 (5)	N5—C25—H25A	118.3
C4—C5—H5A	119.5	C24—C25—H25A	118.3
C6—C5—H5A	119.5	N5—C26—C27	124.4 (6)
N1—C6—C5	118.6 (5)	N5—C26—H26A	117.8
N1—C6—C7	120.7 (4)	C27—C26—H26A	117.8
C5—C6—C7	120.7 (5)	C26—C27—C23	119.7 (5)
O12—C7—O11	125.3 (4)	C26—C27—H27A	120.2
O12—C7—C6	117.5 (4)	C23—C27—H27A	120.2
O11—C7—C6	117.1 (4)	C6—N1—C2	121.5 (4)
N2—C8—C9	123.2 (5)	C12—N2—C8	116.4 (4)
N2—C8—H8A	118.4	C12—N2—Fe1	124.0 (3)
C9—C8—H8A	118.4	C8—N2—Fe1	118.7 (3)
C8—C9—C10	121.1 (5)	C15—N3—C16	116.6 (4)
C8—C9—H9A	119.4	C22—N4—C18	116.1 (4)
C10—C9—H9A	119.4	C22—N4—Fe1	117.6 (3)
C9—C10—C11	115.1 (4)	C18—N4—Fe1	125.0 (3)

C9—C10—C13	122.9 (4)	C26—N5—C25	116.2 (5)
C11—C10—C13	122.0 (4)	Fe1—O1—H11	130 (4)
C12—C11—C10	120.1 (5)	Fe1—O1—H12	124 (4)
C12—C11—H11A	119.9	H11—O1—H12	106.3 (18)
C10—C11—H11A	119.9	Fe1—O2—H21	121 (4)
N2—C12—C11	123.9 (5)	Fe1—O2—H22	125 (4)
N2—C12—H12A	118.1	H21—O2—H22	107.8 (19)
C11—C12—H12A	118.1	Fe1—O3—H31	132 (4)
C17—C13—C14	117.3 (4)	Fe1—O3—H32	109 (4)
C17—C13—C10	122.0 (4)	H31—O3—H32	109.4 (19)
C14—C13—C10	120.7 (4)	Fe1—O4—H41	127 (4)
C15—C14—C13	118.7 (5)	Fe1—O4—H42	121 (4)
C15—C14—H14A	120.6	H41—O4—H42	107.8 (19)
C13—C14—H14A	120.6	H51—O5—H52	108.4 (19)
N3—C15—C14	123.9 (5)	H61—O6—H62	107.2 (19)
N3—C15—H15A	118.1	H71—O7—H72	106.9 (19)
C14—C15—H15A	118.1	H81—O8—H82	108.6 (19)
N3—C16—C17	125.0 (5)		
O10—C1—C2—C3	−179.0 (7)	C19—C20—C23—C24	176.6 (4)
O9—C1—C2—C3	0.1 (10)	C27—C23—C24—C25	0.0 (8)
O10—C1—C2—N1	2.5 (10)	C20—C23—C24—C25	179.0 (4)
O9—C1—C2—N1	−178.5 (6)	C23—C24—C25—N5	0.0 (9)
N1—C2—C3—C4	−0.6 (11)	N5—C26—C27—C23	0.0 (9)
C1—C2—C3—C4	−179.2 (6)	C24—C23—C27—C26	0.0 (7)
C2—C3—C4—C5	0.7 (9)	C20—C23—C27—C26	−179.0 (5)
C3—C4—C5—C6	−0.2 (8)	C5—C6—N1—C2	0.5 (9)
C4—C5—C6—N1	−0.4 (8)	C7—C6—N1—C2	−177.4 (6)
C4—C5—C6—C7	177.5 (4)	C3—C2—N1—C6	−0.1 (11)
N1—C6—C7—O12	0.2 (7)	C1—C2—N1—C6	178.6 (5)
C5—C6—C7—O12	−177.7 (5)	C11—C12—N2—C8	−1.8 (8)
N1—C6—C7—O11	177.9 (5)	C11—C12—N2—Fe1	167.1 (4)
C5—C6—C7—O11	0.0 (7)	C9—C8—N2—C12	2.0 (8)
N2—C8—C9—C10	1.0 (9)	C9—C8—N2—Fe1	−167.5 (5)
C8—C9—C10—C11	−4.1 (8)	O1—Fe1—N2—C12	72.5 (4)
C8—C9—C10—C13	175.5 (5)	O3—Fe1—N2—C12	−107.6 (4)
C9—C10—C11—C12	4.3 (8)	O2—Fe1—N2—C12	161.2 (4)
C13—C10—C11—C12	−175.3 (5)	O4—Fe1—N2—C12	−19.1 (4)
C10—C11—C12—N2	−1.5 (9)	O1—Fe1—N2—C8	−118.9 (4)
C9—C10—C13—C17	−174.3 (4)	O3—Fe1—N2—C8	61.1 (4)
C11—C10—C13—C17	5.2 (7)	O2—Fe1—N2—C8	−30.2 (4)
C9—C10—C13—C14	5.3 (7)	O4—Fe1—N2—C8	149.5 (4)
C11—C10—C13—C14	−175.1 (4)	C14—C15—N3—C16	0.0 (7)
C17—C13—C14—C15	0.2 (7)	C17—C16—N3—C15	−0.3 (8)
C10—C13—C14—C15	−179.4 (4)	C21—C22—N4—C18	−1.9 (8)
C13—C14—C15—N3	0.0 (7)	C21—C22—N4—Fe1	165.8 (4)
N3—C16—C17—C13	0.6 (8)	C19—C18—N4—C22	1.8 (7)
C14—C13—C17—C16	−0.6 (7)	C19—C18—N4—Fe1	−164.9 (4)
C10—C13—C17—C16	179.1 (4)	O1—Fe1—N4—C22	−63.8 (4)
N4—C18—C19—C20	0.3 (8)	O3—Fe1—N4—C22	116.3 (4)

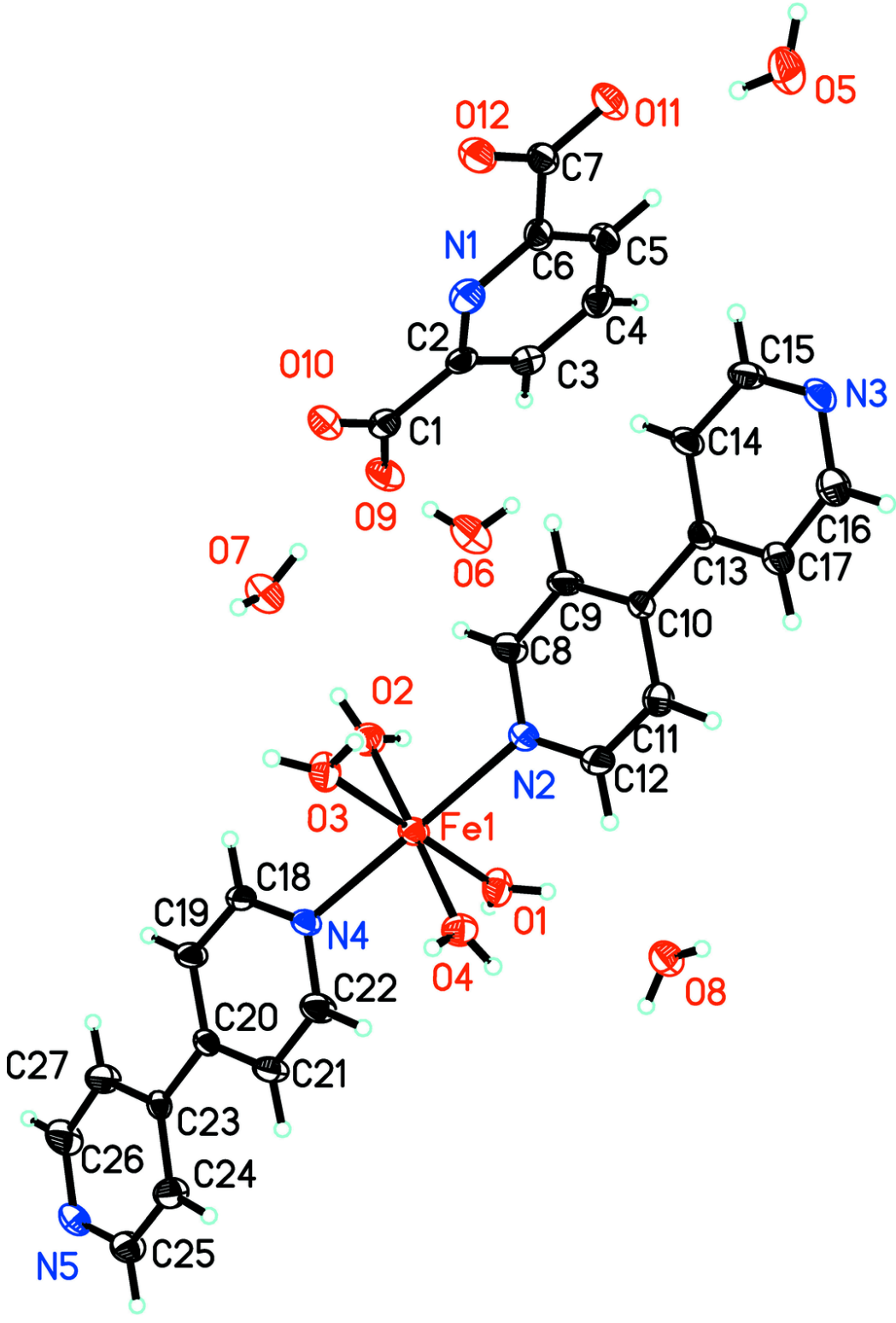
C18—C19—C20—C21	−2.2 (7)	O2—Fe1—N4—C22	−152.5 (4)
C18—C19—C20—C23	176.3 (4)	O4—Fe1—N4—C22	27.8 (4)
C19—C20—C21—C22	2.1 (7)	O1—Fe1—N4—C18	102.7 (4)
C23—C20—C21—C22	−176.4 (4)	O3—Fe1—N4—C18	−77.2 (4)
C20—C21—C22—N4	0.0 (8)	O2—Fe1—N4—C18	14.0 (4)
C21—C20—C23—C27	174.0 (4)	O4—Fe1—N4—C18	−165.7 (4)
C19—C20—C23—C27	−4.4 (7)	C27—C26—N5—C25	0.0 (9)
C21—C20—C23—C24	−5.0 (7)	C24—C25—N5—C26	0.0 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H11···O8^{i}	0.82 (1)	2.06 (3)	2.831 (7)	156 (6)
O1—H12···N3^{ii}	0.82 (1)	1.98 (3)	2.759 (5)	158 (6)
O2—H21···O5^{iii}	0.82 (1)	2.00 (2)	2.806 (6)	172 (5)
O2—H22···O7^{iii}	0.82 (1)	1.96 (1)	2.781 (6)	178 (8)
O3—H31···O5^{iv}	0.81 (1)	2.04 (1)	2.851 (7)	175 (7)
O3—H32···N5^{v}	0.81 (1)	1.96 (2)	2.745 (5)	162 (6)
O4—H41···O8	0.82 (1)	1.97 (1)	2.785 (5)	177 (7)
O4—H42···O11^{vi}	0.82 (1)	1.93 (2)	2.736 (5)	170 (5)
O5—H52···O6	0.82 (1)	1.93 (3)	2.739 (8)	170 (7)
O5—H51···O11^{vi}	0.82 (1)	1.94 (2)	2.742 (7)	166 (7)
O6—H62···O9	0.82 (1)	2.46 (8)	2.952 (6)	120 (7)
O6—H61···O10^{i}	0.82 (1)	2.03 (2)	2.827 (5)	164 (7)
O7—H72···O5^{vii}	0.82 (1)	2.57 (2)	3.349 (7)	159 (5)
O7—H71···O9	0.82 (1)	1.81 (2)	2.622 (6)	168 (7)
O8—H81···O7	0.82 (1)	2.01 (3)	2.817 (7)	170 (6)
O8—H82···O12^{viii}	0.81 (1)	1.97 (2)	2.768 (6)	170 (7)

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

Fig. 1


O5
O12
O11
C7
N1
C6
C5
C4
C2
C3
O10
C1
C15
N3
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O9
O6
O7
C16
C13
C17
C9
C10
C8
O2
C11
N2
C12
O3
Fe1
C18
O1
C19
N4
O4
O8
C22
C20
C27
C21
C23
C26
C24
N5
C25