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# A three-dimensional hydrogen-bonded coordination framework: *catena*-poly[[[tetraaquairon(II)]-$\mu$-4,4′-bipyridine] benzene-1,4-dicarboxylate]

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**Key indicators**

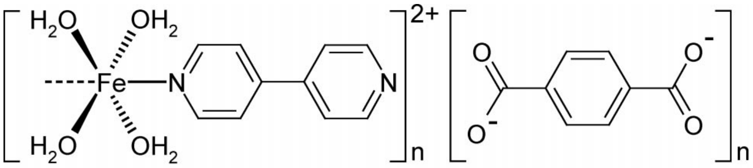
Single-crystal X-ray study
$T$ = 150 K
Mean $\sigma$(C–C) = 0.002 Å
$R$ factor = 0.023
$wR$ factor = 0.064
Data-to-parameter ratio = 14.0

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

The title compound, $\{[Fe(C_{10}H_8N_2)(H_2O)_4](C_8H_4O_4)\}_n$ or $[Fe(bpy)(H_2O)_4](bdc)$ (bpy is 4,4′-bipyridine and bdc is benzene-1,4-dicarboxylate), contains one-dimensional chains of $Fe^{II}$ centres bridged by bpy ligands along the $b$ axis, with the equatorial coordination of four water molecules completing the octahedral coordination. Non-coordinated bdc anions balance the charge of the $Fe^{II}$ centres and bridge adjacent one-dimensional chains through an extensive hydrogen-bonding network. The compound crystallizes in the centrosymmetric space group $P2/c$, which defines twofold axes along both the Fe–bpy chains and the bdc anions.

## Comment

The linking of metal ions with bridging organic ligands offers a versatile means of constructing materials with interesting structure and function (Hoskins & Robson, 1990). The linear bridging ligands 4,4′-bipyridine (bpy) and benzene-1,4-dicarboxylate (bdc) have been extensively utilized in the formation of such materials (*e.g.* Kepert & Rosseinsky, 1999; Li *et al.*, 1999). The present study of the title compound, (I), was carried out with the aim of engineering a three-dimensional coordination framework of (4,4) Fe–bpy sheets bridged by bdc pillars. However, the incorporation of coordinated water molecules resulted in a hydrogen-bonded network.



(I)

Compound (I) crystallizes in the centrosymmetric monoclinic space group $P2/c$. The primary structure consists of one-dimensional chains of $Fe^{II}$ centres bridged by bpy ligands along the $b$ axis. The equatorial coordination of four water molecules completes the mildly distorted octahedral $Fe^{II}$ coordination. The Fe—O and Fe—N bond lengths are consistent with high-spin $Fe^{II}$ (Table 1).

Non-coordinated bdc ligands balance the charge of the system and bridge adjacent one-dimensional chains through an extensive hydrogen-bonding network (Fig. 2). All water H atoms form hydrogen bonds with either of the two crystallographically distinct carboxylate O atoms (Table 2). These interactions are likely to contribute to the highly ordered nature of the structure.

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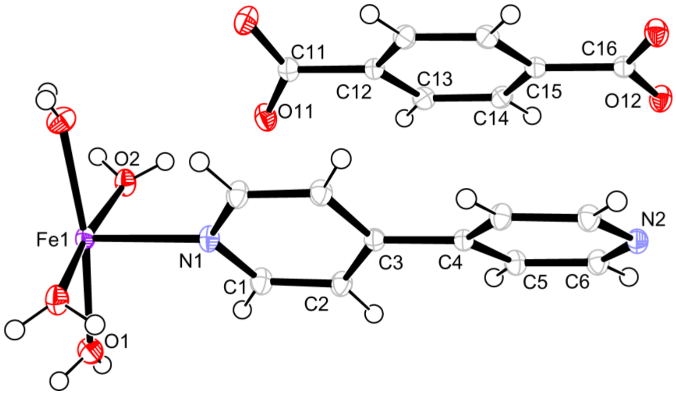

**Figure 1**
A view of the structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Unlabelled atoms are related to labelled atoms by the symmetry operation ($1 - x, y, \frac{3}{2} - z$).

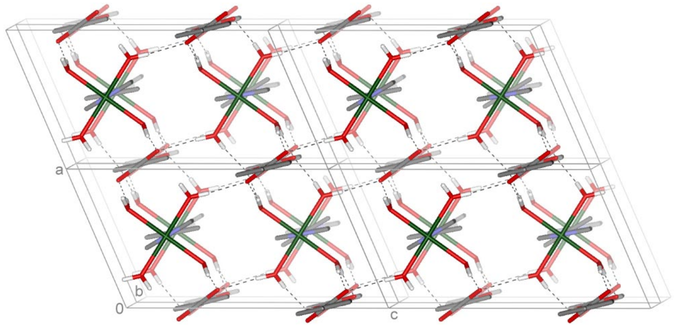

**Figure 2**
The crystal packing of (I) viewed down the *b* axis, showing the extensive hydrogen-bonding network.

## Experimental

$[Fe(bpy)(H_2O)_4](bdc)$, (I), was synthesized by the slow diffusion of aqueous solutions of $Fe(ClO_4)_2$ (31 mg, 0.12 mmol) and a mixture of bpy (37 mg, 0.24 mmol) and $Na_2bdc$ (25 mg, 0.12 mmol) in an H-shaped tube. Orange block-shaped single crystals of (I) grew after approximately one month.

*Crystal data*

$[Fe(C_{10}H_8N_2)(H_2O)_4](C_8H_4O_4)$
$M_r = 448.21$
Monoclinic, $P2/c$
$a = 6.9541$ (12) Å
$b = 11.465$ (2) Å
$c = 12.059$ (2) Å
$\beta = 113.547$ (3)°
$V = 881.4$ (3) Å$^3$
$Z = 2$

$D_x = 1.689$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 889 reflections
$\theta = 3.2$–27.9°
$\mu = 0.91$ mm$^{-1}$
$T = 150$ (2) K
Block, orange
0.50 × 0.37 × 0.22 mm

*Data collection*

Bruker SMART 1000 CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan, (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.733$, $T_{max} = 0.819$
8043 measured reflections

2075 independent reflections
2005 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 28.0°$
$h = -9 \rightarrow 8$
$k = -15 \rightarrow 15$
$l = -15 \rightarrow 15$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.065$
$S = 1.13$
2075 reflections
148 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0212P)^2 + 0.3552P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å$^{-3}$
$\Delta\rho_{min} = -0.44$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Fe1—O1 | 2.1311 (10) | Fe1—N1 | 2.1495 (15) |
| Fe1—O2 | 2.1375 (10) | Fe1—N2$^{i}$ | 2.2081 (14) |
| | | | |
| O1—Fe1—O1$^{ii}$ | 171.39 (5) | O2—Fe1—N1 | 86.32 (2) |
| O1—Fe1—O2$^{ii}$ | 86.77 (4) | O1—Fe1—N2$^{i}$ | 85.69 (3) |
| O1—Fe1—O2 | 93.79 (4) | O2—Fe1—N2$^{i}$ | 93.68 (2) |
| O2$^{ii}$—Fe1—O2 | 172.65 (5) | N1—Fe1—N2$^{i}$ | 180.0 |
| O1—Fe1—N1 | 94.31 (3) | | |

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, y, -z + \frac{3}{2}$.

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| O1—H1$A\cdots$O12$^{iii}$ | 0.797 (13) | 2.074 (14) | 2.8578 (13) | 167.7 (17) |
| O1—H1$B\cdots$O11$^{iv}$ | 0.813 (13) | 1.910 (14) | 2.7204 (14) | 174.3 (17) |
| O2—H2$A\cdots$O11$^{v}$ | 0.796 (13) | 1.965 (13) | 2.7570 (13) | 174.0 (17) |
| O2—H2$B\cdots$O12 | 0.816 (13) | 1.907 (13) | 2.7220 (13) | 177.2 (18) |

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

All H atoms were modelled with fixed isotropic parameters of $1.2U_{eq}$ (aromatic) or $1.5U_{eq}$ (water) of their parent atom. Aromatic H-atom sites were constrained to geometrically idealized positions, with C—H distances of 0.95 Å. Water H atoms were located from a difference map and the O—H bond lengths were restrained to be approximately equal.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT-Plus* (Bruker, 1997); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997) and *WebLab ViewerPro3.7* (Accelrys, 2001); software used to prepare material for publication: *XCIF* in *SHELXTL*.

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