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# 4,4′-Bipyridinium(2+) hexaaquairon(II) disulfate dihydrate

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**Key indicators**

Single-crystal X-ray study
*T* = 295 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.054
*wR* factor = 0.130
Data-to-parameter ratio = 12.8

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



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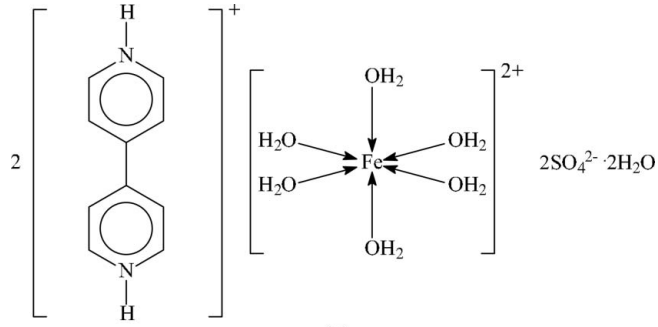
In the crystal structure of the title compound, $(C_{10}H_{10}N_2)[Fe(H_2O)_6](SO_4)_2\cdot 2H_2O$, hydrogen bonds link the 4,4′-bipyridinium cation, which lies on a special position of site symmetry 2, the hexaaquairon(II) cation, which lies on a center of symmetry, the sulfate anions and the uncoordinated water molecules into a three-dimensional network structure.

## Comment

The nature of the crystalline compounds that are isolated from the reaction of iron(II) sulfate and an aliphatic amine under hydrothermal conditions is influenced by several factors, one of which is the amine itself. The amine binds directly to iron; however, different amines direct the outcome of the reaction, and some of the reactions give rise to the formation of organic ammonium sulfates only (Fu *et al.*, 2005). A similar reaction with 4,4′-bipyridine yielded the title compound, (I) (Fig. 1), which can be regarded as a double salt of 4,4′-bipyridinium sulfate and hexaaquairon(II) sulfate.




(I)

The $C_{10}H_{10}N_2^{2+}$ and $[Fe(H_2O)_3]^{2+}$ cations, the sulfate anions and the solvent water molecules interact through hydrogen bonds (Table 2) to form a three-dimensional network. The organic cation lies on a twofold rotation axis, and the iron atom on an inversion center. The pyridine rings are twisted by 44.9 (1)° with respect to each other. There are two examples of a hexaaquairon salt having an organic molecule/ion; in hexaaquairon(II) piperazinedinium bis(hydrogenphosphate) (Abu-Shandi *et al.*, 2003) and hexaaquairon(II) dinitrate bis(hexamethylenetetramine) tetrahydrate (Zhu *et al.*, 2003), as in the title compound, the anion interacts indirectly with the metal atom through the coordinated water molecules.

## Experimental

Iron(II) sulfate heptahydrate (1.12 g, 4 mmol) was dissolved in water (5 ml) that was mixed with ethanol (2 ml). A drop of concentrated

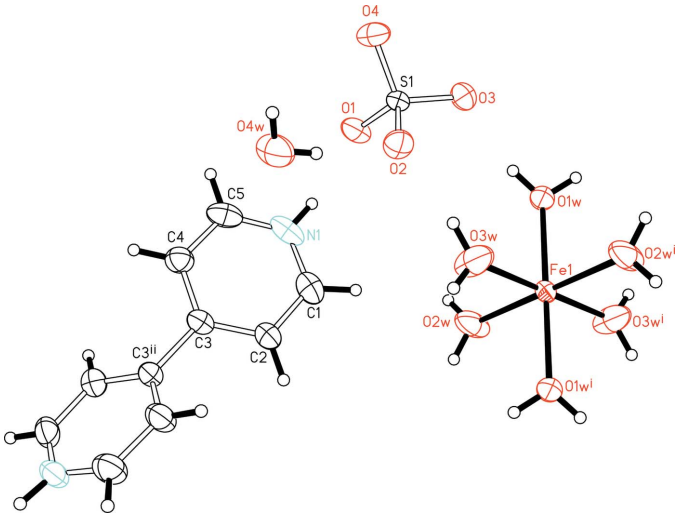



**Figure 1**
*ORTEPII* plot (Johnson, 1976) of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $\frac{1}{2}-x, \frac{3}{2}-y, 1-z$; (ii) $1-x, y, \frac{1}{2}-z$.]

sulfuric acid (0.06 ml, 1 mmol) was added, followed by hydrogen peroxide (0.15 ml, 4 mmol) and 4,4′-bipyridine (0.10 g, 0.5 mmol). The mixture was then stirred quickly to form a gel. The gel was then placed in a 15 ml Teflon-lined Parr bomb, which was heated at 383 K for 2 d. A few colorless crystals were formed and these were separated by hand (in about 3% yield).

*Crystal data*

$(C_{10}H_{10}N_2)[Fe(H_2O)_6](SO_4)_2\cdot 2H_2O$
$M_r = 550.30$
Monoclinic, $C2/c$
$a = 17.543$ (1) Å
$b = 9.0676$ (6) Å
$c = 13.7592$ (9) Å
$\beta = 96.670$ (1)°
$V = 2173.9$ (2) Å$^3$
$Z = 4$
$D_x = 1.681$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3298 reflections
$\theta = 2.4$–26.5°
$\mu = 0.97$ mm$^{-1}$
$T = 295$ (2) K
Block, colorless
0.18 × 0.18 × 0.16 mm

*Data collection*

Bruker APEX area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.750$, $T_{max} = 0.861$
11660 measured reflections
2482 independent reflections
2310 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 27.5°$
$h = -22 \rightarrow 22$
$k = -11 \rightarrow 11$
$l = -17 \rightarrow 17$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.130$
$S = 1.21$
2482 reflections
194 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 2.4914P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å$^{-3}$
$\Delta\rho_{min} = -0.88$ e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| Fe1—O1*w* | 2.107 (2) | Fe1—O3*w* | 2.132 (2) |
| Fe1—O2*w* | 2.103 (2) | | |
| O1*w*—Fe1—O2*w* | 93.6 (1) | O1*w*—Fe1—O3*w*$^{i}$ | 90.8 (1) |
| O1*w*—Fe1—O2*w*$^{i}$ | 86.4 (1) | O2*w*—Fe1—O3*w* | 91.5 (1) |
| O1*w*—Fe1—O3*w* | 89.2 (1) | O2*w*—Fe1—O3*w*$^{i}$ | 88.6 (1) |

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

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*w*—H1*w*1···O3 | 0.86 (1) | 1.86 (2) | 2.692 (3) | 164 (3) |
| O1*w*—H1*w*2···O4$^{iii}$ | 0.85 (1) | 1.88 (1) | 2.736 (3) | 178 (3) |
| O2*w*—H2*w*1···O1$^{iv}$ | 0.85 (1) | 1.86 (1) | 2.705 (3) | 176 (4) |
| O2*w*—H2*w*2···O3$^{v}$ | 0.85 (1) | 1.93 (1) | 2.783 (3) | 177 (3) |
| O3*w*—H3*w*1···O2 | 0.85 (1) | 1.92 (1) | 2.762 (3) | 173 (6) |
| O3*w*—H3*w*2···O4*w*$^{vi}$ | 0.85 (1) | 1.86 (1) | 2.703 (4) | 172 (4) |
| O4*w*—H4*w*1···O2 | 0.85 (1) | 1.96 (2) | 2.792 (3) | 168 (5) |
| O4*w*—H4*w*2···O4$^{vii}$ | 0.85 (1) | 1.89 (1) | 2.720 (3) | 168 (4) |
| N1—H1*n*···O1 | 0.85 (1) | 1.86 (2) | 2.680 (3) | 162 (6) |

Symmetry codes: (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (v) $x, -y+1, z-\frac{1}{2}$; (vi) $-x+1, -y+1, -z+1$; (vii) $-x+1, y, -z+\frac{3}{2}$.

H atoms were located in difference Fourier maps and were refined with distance restraints of O—H = N—H = 0.85 (1) Å and C—H = 0.95 (1) Å; their displacement parameters were refined freely.

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: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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