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catena-Poly[piperazinium(2+) [ferrate(III)-μ-hydroxo-κ^2O:O-di-μ-sulfato-κ^4O:O'] monohydrate]

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In the crystal structure of the title compound, $\{(C_4H_{12}N_2)[Fe(OH)(SO_4)_2]\cdot H_2O\}_n$, the anion exists as a linear chain that propagates in a zigzag manner along the *c* axis of the monoclinic unit cell. The organic cations, the polyanions and non-coordinated water molecules interact through O—H···O and N—H···O hydrogen bonds, forming a three-dimensional network.

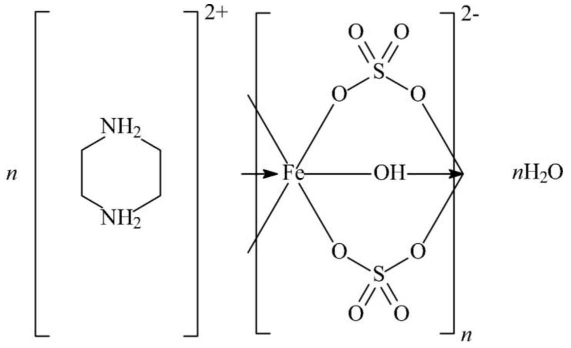
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.039
wR factor = 0.098
Data-to-parameter ratio = 13.7

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

Comment

This report describes our continuing studies on the reactions of ferrous sulfate and piperazine under hydrothermal conditions. We have previously described a piperizinium salt of a polyanionic hydroxodisulfatoferrate(III) chain, accompanied by two water molecules of crystallization (Fu *et al.*, 2005). Mixing the reagents in a different order before hydrothermal treatment afforded the corresponding monohydrate, (I) (Fig. 1).



(I)

The $[Fe(OH)(SO_4)_2]_\infty$ chain adopts a zigzag motif (Fig. 2), and the organic cation and non-coordinated water molecule link the chains into a three-dimensional architecture through N—H···O and O—H···O hydrogen bonds (Table 2). The principal features, *e.g.* bond lengths and repeat distance, of the chain are similar to those already reported (Fu *et al.*, 2005). However, the monohydrate is somewhat more compact, as noted by its density (2.052 Mg m^{-3}) compared with that of the dihydrate (1.972 Mg m^{-3}).

Experimental

Ferrous sulfate heptahydrate (0.28 g, 1 mmol) was dissolved in water (5 ml). Concentrated sulfuric acid (0.06 ml, 1 mmol) was added, followed by piperazine (0.09 g, 1 mmol) and ethanol (5 ml). The mixture was stirred briefly to form a homogeneous gel; the gel was transferred into a 15 ml Teflon-lined Parr bomb that was then heated 383 K for 72 h. The bomb was cooled and opened to reveal yellow crystals of (I) in about 70% yield with respect to Fe.

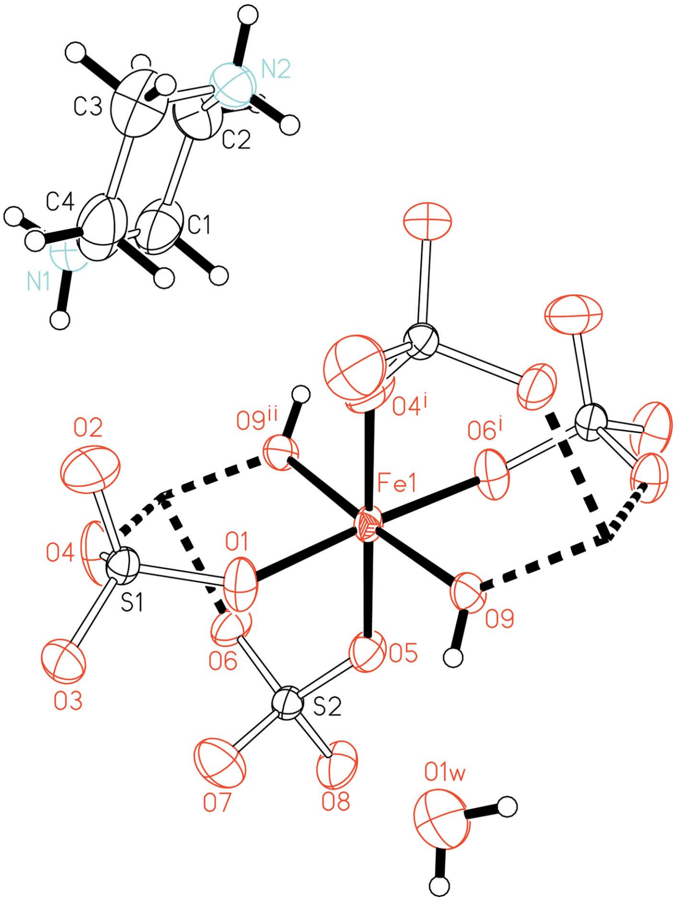


Figure 1
A view of (I), showing 70% displacement ellipsoids (arbitrary spheres for the H atoms). Symmetry codes are given in Table 1.

Crystal data

$(C_4H_{12}N_2)[Fe(OH)(SO_4)_2]\cdot H_2O$
$M_r = 371.15$
Monoclinic, $P2_1/c$
$a = 9.2570$ (6) Å
$b = 18.318$ (1) Å
$c = 7.1562$ (4) Å
$\beta = 98.094$ (1)°
$V = 1201.4$ (1) Å^3
$Z = 4$
$D_x = 2.052$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3698 reflections
$\theta = 2.2$–28.0°
$\mu = 1.66$ mm^{-1}
$T = 295$ (2) K
Rod, yellow
0.26 × 0.07 × 0.06 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.776$, $T_{\max} = 0.907$
13166 measured reflections
2743 independent reflections
2452 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$
$\theta_{\max} = 27.5°$
$h = -11 \rightarrow 11$
$k = -23 \rightarrow 23$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.098$
$S = 1.07$
2743 reflections
200 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 1.0669P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.59$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}

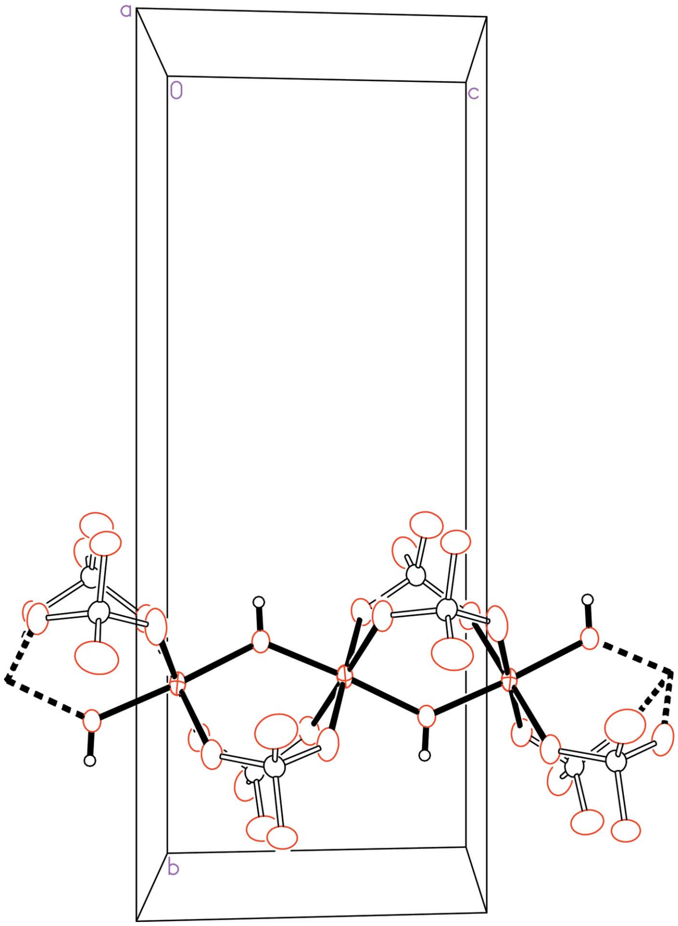


Figure 2
Detail of (I), showing the polyanionic $[Fe(OH)(SO_4)_2]_\infty$ chain.

Table 1
Selected geometric parameters (Å, °).

Fe1—O1	2.014 (2)	Fe1—O6^{i}	2.048 (2)
Fe1—O4^{i}	1.999 (2)	Fe1—O9	1.960 (2)
Fe1—O5	2.023 (2)	Fe1—O9^{ii}	1.977 (2)
S1—O1—Fe1	135.1 (1)	S2—O6—Fe1^{ii}	135.0 (1)
S1—O4—Fe1^{ii}	139.4 (1)	Fe1—O9—Fe1^{i}	130.8 (1)
S2—O5—Fe1	133.4 (1)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H9o···O1w	0.84 (1)	2.16 (1)	2.971 (3)	164 (3)
O1w—H1w1···O7^{iii}	0.85 (1)	2.16 (2)	2.955 (4)	155 (4)
O1w—H1w2···O8^{iv}	0.85 (1)	2.19 (2)	2.974 (4)	153 (4)
N1—H1n1···O2	0.85 (1)	1.86 (1)	2.666 (4)	158 (3)
N1—H1n2···O8^{v}	0.85 (1)	1.93 (1)	2.783 (4)	173 (3)
N2—H2n1···O7^{vi}	0.85 (1)	1.97 (2)	2.775 (3)	157 (3)
N2—H2n2···O3^{i}	0.85 (1)	2.02 (1)	2.844 (3)	162 (3)

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

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The carbon-bound H atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding with the constraint $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(carrier). The ammonium, water and hydroxy H atoms were located in a difference Fourier map, and were refined with distance restraints of N—H = O—H = 0.85 (1) Å and H···H = 1.39 (1) Å; the H-atom $U_{\rm iso}$ values 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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