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catena-Poly[[bis(ethane-1,2-diaminium)[ferrate(III)-μ-hydroxo-κ^2O:O-di-μ-sulfato-κ^4O:O']] sulfate monohydrate]

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The reaction of ferric sulfate with ethylenediamine in the presence of acid under hydrothermal conditions results in the title compound, $(C_2H_{10}N_2)_2[Fe^{III}(OH)(SO_4)_2](SO_4)\cdot H_2O$. The six-coordinate ferrate dianion participates in a μ_2-hydroxo-di-μ_2-sulfate-bridged chain. The component species interact with each other by an extensive network of O—H$\cdots$O and N—H$\cdots$O hydrogen bonds. The two independent Fe^{III} atoms lie on sites of $\overline{1}$ symmetry.

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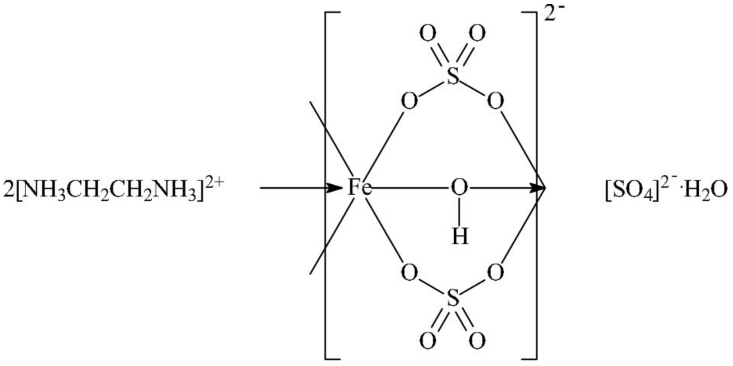
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.028
wR factor = 0.081
Data-to-parameter ratio = 11.2

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

Comment

The structure reported here, (I), is a continuation of our studies on the reactions of ferric sulfate and diamines under hydrothermal conditions: 1,6-hexanediamine yielded $(C_6H_{18}N_2)[Fe(OH)(SO_4)_2]\cdot H_2O$ (Fu *et al.*, 2005*a*) and diethylenetriamine yielded $(C_4H_{16}N_3)[Fe(SO_4)_3]\cdot H_2O$ (Fu *et al.*, 2005*b*). However, diethylenetriamine and triethylenetetraamine gave only the organic ammonium sulfates (Fu *et al.*, 2005*c*,*d*).



(I)

The iron-containing anion in (I) (Fig. 1) is a polyanionic $[Fe(OH)(SO_4)_2]_n$ chain, the Fe^{III} atoms of which are bridged by the hydroxo and sulfate groups in an all-*trans* octahedral environment. There are two independent Fe atoms, both of which lie on special positions of site symmetry $\overline{1}$. The formula unit features a free sulfate group; this is linked to the polyanionic chain (Fig. 2), the cations and non-coordinated water molecules, resulting in a three-dimensional hydrogen-bonded network (Table 2).

Experimental

Ferric sulfate nonahydrate (0.28 g, 0.5 mmol) was dissolved in a water–ethanol mixture (9 ml, 2:1 v/v). Concentrated sulfuric acid solution (0.16 ml, 3 mmol) was added, followed by ethylenediamine (0.18 ml, 3 mmol). The mixture was stirred briefly to form a homogeneous gel; the gel was transferred into a 15 ml Teflon-lined Parr bomb which was then at heated 383 K for 48 h. The solid product consisted of yellow crystals of (I) that were isolated in about 80% yield (based on Fe).

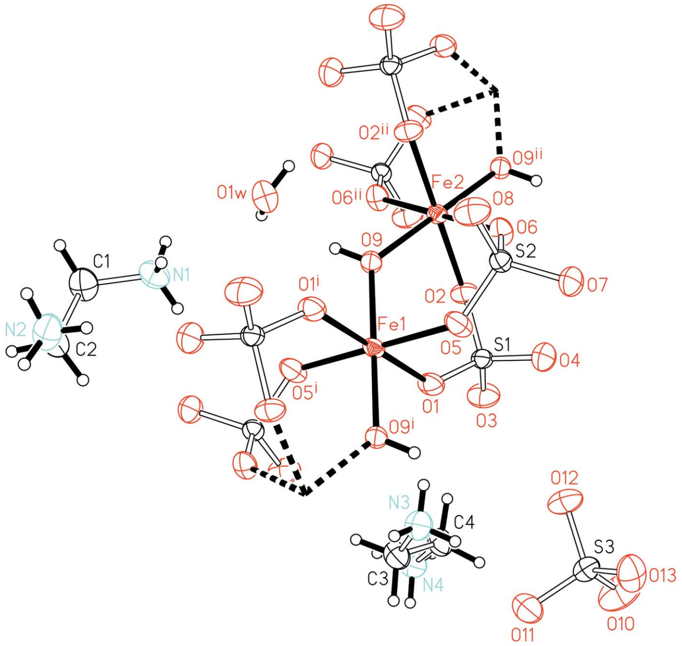


Figure 1
View of (I) showing 70% displacement ellipsoids (arbitrary spheres for the H atoms). The dashed lines indicate bonds to adjacent Fe atoms in the polymeric chain. [Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $-x, 1 - y, 1 - z$.]

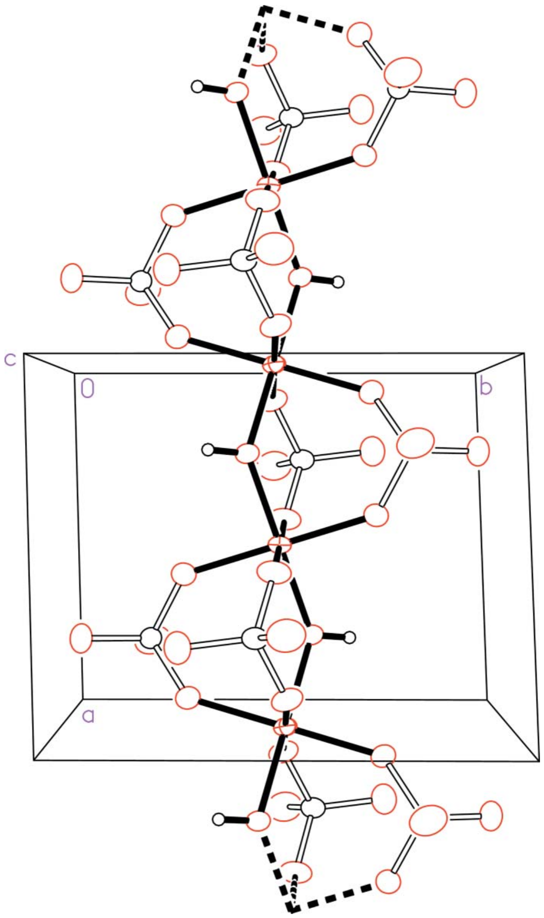


Figure 2
View of a fragment of the polyanionic $[Fe(OH)(SO_4)_2]$ chain in (I).

Crystal data

$(C_2H_{10}N_2)_2[Fe(OH)(SO_4)_2]$-$(SO_4)\cdot H_2O$
$M_r = 503.29$
Triclinic, $P\overline{1}$
$a = 7.1352\ (4)$ Å
$b = 8.7869\ (5)$ Å
$c = 13.1860\ (7)$ Å
$\alpha = 88.659\ (1)^\circ$
$\beta = 85.782\ (1)^\circ$
$\gamma = 88.145\ (1)^\circ$
$V = 823.86\ (8)$ Å^3

$Z = 2$
$D_x = 2.029$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4504 reflections
$\theta = 2.3$–28.4°
$\mu = 1.38$ mm^{-1}
$T = 295\ (2)$ K
Needle, yellow
$0.25 \times 0.06 \times 0.04$ mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.795$, $T_{max} = 0.947$
9220 measured reflections

3695 independent reflections
3324 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.5^\circ$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.081$
$S = 1.04$
3695 reflections
330 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2 + 0.2749P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Fe1—O1	2.094 (1)	Fe2—O2	2.044 (1)
Fe1—O5	1.983 (1)	Fe2—O6	1.998 (1)
Fe1—O9	1.951 (1)	Fe2—O9	1.962 (1)

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H9o···O1w	0.84 (1)	1.94 (1)	2.757 (2)	164 (2)
O1W—H1w1···O4^{i}	0.84 (1)	2.18 (1)	2.988 (2)	163 (3)
O1W—H1w2···O7^{ii}	0.84 (1)	1.99 (1)	2.790 (2)	159 (3)
N1—H1n1···O12^{iii}	0.85 (1)	2.14 (2)	2.912 (3)	151 (3)
N1—H1n2···O8^{ii}	0.85 (1)	2.32 (3)	2.966 (3)	132 (3)
N1—H1n3···O7^{iii}	0.86 (1)	2.17 (2)	2.967 (3)	153 (3)
N1—H1n2···O1w	0.85 (1)	2.30 (3)	2.923 (3)	130 (3)
N2—H2n1···O4^{iii}	0.86 (1)	2.04 (1)	2.847 (3)	158 (3)
N2—H2n2···O13^{iv}	0.85 (1)	2.05 (2)	2.840 (3)	155 (3)
N2—H2n3···O10^{v}	0.85 (1)	1.88 (1)	2.714 (3)	165 (3)
N3—H3n1···O11	0.85 (1)	2.02 (1)	2.842 (3)	161 (3)
N3—H3n2···O2^{vi}	0.85 (1)	2.07 (1)	2.912 (2)	174 (3)
N3—H3n3···O1	0.85 (1)	2.12 (1)	2.964 (2)	176 (3)
N4—H4n1···O11^{vii}	0.86 (1)	1.97 (1)	2.811 (3)	168 (3)
N4—H4n2···O12^{viii}	0.86 (1)	1.96 (1)	2.807 (3)	169 (3)
N4—H4n3···O13^{ii}	0.86 (1)	1.99 (1)	2.834 (3)	165 (3)

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

Acta Cryst. (2005). E**61**, m1478–m1480 Fu *et al.* • $(C_2H_{10}N_2)_2[Fe(OH)(SO_4)_2](SO_4)\cdot H_2O$ **m1479**

H atoms were located in difference maps and refined with distance restraints [O—H = N—H = 0.85 (1) and C—H = 0.95 (1) Å]. The $U_{\rm iso}$ 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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