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

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Ferric sulfate reacts with 1,6-diaminohexane in the presence of acid under hydrothermal conditions to form the title compound, $(C_6H_{18}N_2)[Fe(OH)(SO_4)_2]\cdot H_2O$. The six-coordinate ferrate trianion exists as a μ_2-hydroxodi-μ_2-sulfate-bridged chain that propagates along the *b* axis of the monoclinic unit cell. The dication and uncoordinated water molecule connect the polyanionic chain into a three-dimensional network structure. The asymmetric unit contains two Fe ions, each on a center of symmetry.

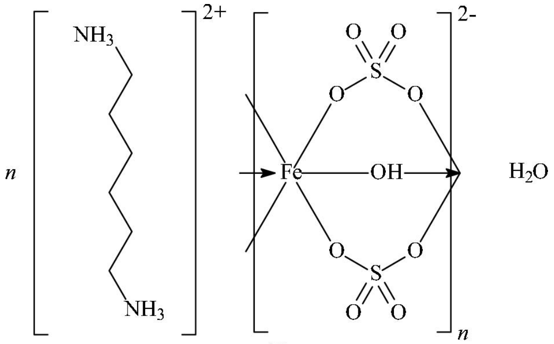
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.100
Data-to-parameter ratio = 16.8

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

Comment

Ferric sulfate reacts with diethylenetriamine to yield *catena*-poly[diethylenetriaminium [ferrate(III)-tri-μ-sulfato-κ^6O:O'] monohydrate] (Fu *et al.*, 2005). The use of 1,6-hexanediamine in place of the triamine afforded the title compound, (I) (Fig. 1), which has a hydroxo group in the anion. The anion exists as a polyanionic chain whose Fe atoms are bridged by the hydroxo and sulfate groups in an all-*trans* octahedral environment (Fig. 2). The two independent Fe atoms lie on special positions of $\overline{1}$ site symmetry. The hydroxo group serves the same purpose as the bridging fluoride group in diethylenetriammonium fluorodisulfatoferrate(II), bis(guanidinium) fluorodisulfatoferrate(III) and piperazinium trifluorosulfatoferrate(III) (Paul *et al.*, 2003). In the title compound, the trication and uncoordinated water molecule connect the polyanionic chains into a three-dimensional network structure through hydrogen bonds (Table 2).



(I)

Experimental

Ferric sulfate nonahydrate (0.28 g, 0.5 mmol), 1,6-diaminohexane (0.48 g, 0.30 mmol), concentrated sulfuric acid (0.29 ml), water (7.2 ml) and ethanol (5 ml) were placed in a Teflon-lined stainless steel bomb. The bomb was heated in an autoclave at 383 K for 2 d and then cooled to room temperature to furnish crystals of (I)

Crystal data

$(C_6H_{18}N_2)[Fe(OH)(SO_4)_2]\cdot H_2O$
$M_r = 401.22$
Monoclinic, $P2_1/n$
$a = 8.7918$ (7) Å
$b = 6.9963$ (5) Å
$c = 25.032$ (1) Å
$\beta = 90.289$ (2)°
$V = 1539.68$ (18) Å^3
$Z = 4$
$D_x = 1.731$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2196 reflections
$\theta = 2.5$–28.3°
$\mu = 1.30$ mm^{-1}
$T = 295$ (2) K
Block, colorless
0.35 × 0.12 × 0.12 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.640$, $T_{\max} = 0.860$
8058 measured reflections
3439 independent reflections
2483 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 27.5°$
$h = -4 \rightarrow 11$
$k = -8 \rightarrow 9$
$l = -31 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.100$
$S = 0.99$
3439 reflections
205 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Extinction correction: none

Table 1
Selected geometric parameters (Å, °).

Fe1—O1	2.008 (2)	Fe2—O2	2.030 (2)
Fe1—O6	2.003 (2)	Fe2—O5	2.031 (2)
Fe1—O9	1.962 (2)	Fe2—O9	1.959 (2)
O1—Fe1—O1$^{\rm i}$	180	O2—Fe2—O2$^{\rm ii}$	180
O1—Fe1—O6	90.98 (7)	O2—Fe2—O5	90.43 (7)
O1—Fe1—O6$^{\rm i}$	89.02 (7)	O2—Fe2—O5$^{\rm ii}$	89.57 (6)
O1—Fe1—O9	89.58 (7)	O2—Fe2—O9	92.79 (7)
O1—Fe1—O9$^{\rm i}$	90.42 (7)	O2—Fe2—O9$^{\rm ii}$	87.21 (7)
O6—Fe1—O6$^{\rm i}$	180	O5—Fe2—O5$^{\rm ii}$	180
O6—Fe1—O9	92.32 (7)	O5—Fe2—O9	88.95 (7)
O6—Fe1—O9$^{\rm i}$	87.68 (7)	O5—Fe2—O9$^{\rm ii}$	91.05 (7)
O9—Fe1—O9$^{\rm i}$	180	O9—Fe2—O9$^{\rm ii}$	180

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y+2, -z+1$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H9o···O1w	0.83 (1)	1.91 (1)	2.726 (3)	172 (2)
O1w—H1w1···O3$^{\rm iii}$	0.84 (1)	2.07 (2)	2.826 (3)	150 (3)
O1w—H1w2···O8$^{\rm ii}$	0.84 (1)	1.93 (2)	2.740 (3)	162 (4)
N1—H1n1···O4	0.86	2.04	2.848 (3)	157
N1—H1n2···O7$^{\rm i}$	0.86	2.09	2.902 (3)	157
N1—H1n3···O1w	0.86	2.06	2.916 (4)	171
N2—H2n1···O8$^{\rm iv}$	0.86	1.92	2.767 (3)	170
N2—H2n2···O9$^{\rm v}$	0.86	2.37	3.107 (3)	143
N2—H2n3···O3$^{\rm vi}$	0.86	2.18	3.028 (3)	169

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

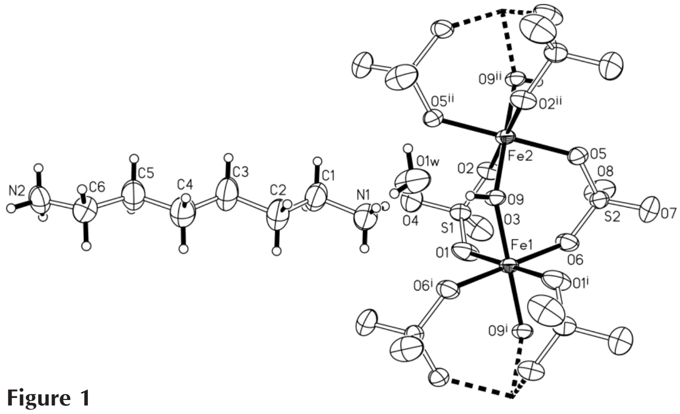


Figure 1
ORTEPII (Johnson, 1976) plot illustrating the coordination geometry of the Fe atom in $[C_6H_{18}N_2][Fe(OH)(SO_4)_2]\cdot H_2O$. Displacement ellipsoids are drawn at the 70% probability level and H atoms are drawn as spheres of arbitrary radii. Symmetry codes are those used in Table 1.

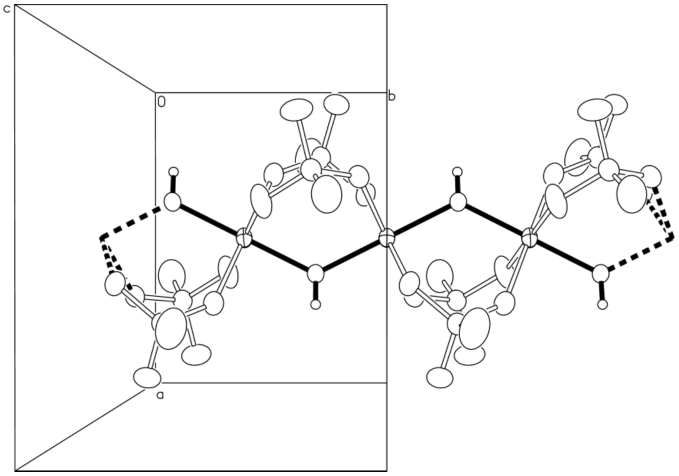


Figure 2
ORTEPII (Johnson, 1976) plot of the polycationic $[Fe(OH)(SO_4)_2]$ chain.

The C- and N-bound H atoms were placed at calculated positions (C—H = 0.97 Å and N—H = 0.86 Å) and were included in the refinements in the riding model approximation, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C,N). The water H atoms were located and refined with distance restraints of O—H = 0.85 (1) Å and H···H = 1.39 (1) Å; their displacement parameters were also refined.

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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