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Iron(II) hydrazinium sulfate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(N–N) = 0.004 Å
R factor = 0.032
wR factor = 0.076
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.

The title compound, poly[[dihydraziniumiron(II)]-di-μ-sulfato-κ^4O:O'], $[Fe(SO_4)_2(N_2H_5)_2]_n$, contains fairly regular *trans*-FeN_2O_4 octahedra. The Fe atoms (site symmetry $\overline{1}$) are bridged by pairs of sulfate groups into infinite [100] chains, which are cross-linked by a network of N—H$\cdots$O hydrogen bonds. $Fe(N_2H_5)_2(SO_4)_2$ is isostructural with its zinc, chromium(II) and cadmium-containing analogues.

Comment

The divalent-metal–hydrazinium sulfates of general formula $M(N_2H_5)_2(SO_4)_2$, where M = Cr, Mn, Fe, Co, Ni, Cu, Zn and Cd can be readily prepared by reacting a salt of the respective metal with hydrazinium sulfate in dilute sulfuric acid (Hand & Prout, 1966), although this method usually results in a microcrystalline product. Recently, we described the single-crystal structure of $Cd(N_2H_5)_2(SO_4)_2$ (Srinivasan *et al.*, 2006) and we now report the isostructural title compound, (I), $Fe(N_2H_5)_2(SO_4)_2$. The compounds $Zn(N_2H_5)_2(SO_4)_2$ (Prout & Powell, 1961) and $Cr(N_2H_5)_2(SO_4)_2$ (Parkins *et al.*, 2001) also share the same stucture.

Compound (I) contains *trans*-FeN_2O_4 octahedra (Fig. 1), in which the N atom is part of a hydrazinium ($N_2H_5^+$) cation. The Fe atoms (site symmetry $\overline{1}$) are connected by pairs of sulfate groups into infinite chains that propagate in [100]. The intrachain Fe$\cdots$Fe separation in (I) is equal to the a unit-cell dimension, *i.e.* 5.3306 (3) Å. The two distinct Fe—O bond lengths in (I) are similar (Table 1) and do not show the gross differences seen in the chromium and zinc analogues.

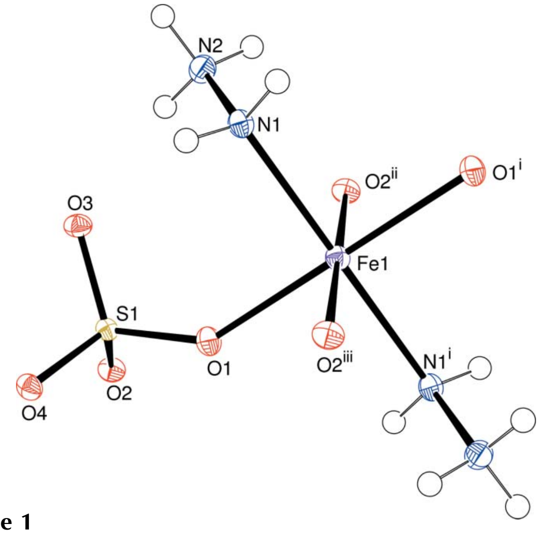

Figure 1
The asymmetric unit of (I) expanded to show the iron coordination (50% displacement ellipsoids; arbitrary spheres for the H atoms). Symmetry codes: (i) $-x, -y, -z$; (ii) $x - 1, y, z$; (iii) $1 - x, -y, -z$.

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The iron–sulfate chains in (I) are cross-linked by N—H···O hydrogen bonds (Table 2), resulting in the same hydrogen-bonding network seen in the other analogues noted above. A well defined trifurcated N2—H3*C*···(O,O,O) interaction occurs (mean bond angle about H3*C* = 107.3°).

Experimental

The reaction of hydrazine monohydrate ($N_2H_4\cdot H_2O$; 0.50 g, 10 mmol) and ethyl bromoacetate (1.671 g, 10 mmol) in 5 ml of dry ethanol resulted in the formation of a white solid containing hydrazinium bromide and ethyl hydrazinoacetate, as reported earlier (Srinivasan *et al.*, 2006). This white solid (0.236 g) was dissolved in water (30 ml) and mixed with an aqueous solution (30 ml) of $FeSO_4\cdot 7H_2O$ (0.278 g, 1 mmol) and a few drops of conc. H_2SO_4. The resulting clear solution, with a pH of 2, was concentrated over a water bath to 20 ml and kept for crystallization at room temperature. After three days, many block-shaped light-green crystals of (I) had formed. These were recovered by filtration, washed with cold water and dried in air.

Crystal data

$[Fe(SO_4)_2(N_2H_5)_2]$
$M_r = 314.08$
Triclinic, $P\overline{1}$
$a = 5.3306\ (3)$ Å
$b = 5.8205\ (3)$ Å
$c = 7.3835\ (4)$ Å
$\alpha = 92.034\ (3)^\circ$
$\beta = 103.313\ (3)^\circ$
$\gamma = 99.237\ (3)^\circ$
$V = 219.41\ (2)$ Å^3
$Z = 1$
$D_x = 2.377$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.23$ mm^{-1}
$T = 120\ (2)$ K
Lath, pale green
0.05 × 0.02 × 0.01 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min} = 0.897$, $T_{max} = 0.978$
3957 measured reflections
1004 independent reflections
911 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 28.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.076$
$S = 1.12$
1004 reflections
90 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0127P)^2 + 0.6538P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.58$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—O1	2.109 (2)	Fe1—N1	2.184 (2)
Fe1—O2^{i}	2.147 (2)		
S1—O1—Fe1	142.94 (13)	S1—O2—Fe1^{ii}	128.85 (12)

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

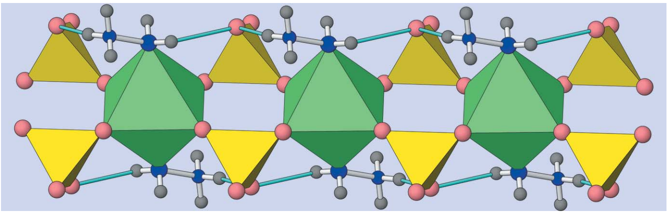

Figure 2
Polyhedral view of a fragment of the chain structure of (I). Colour key: Fe octahedra green, S tetrahedra yellow, O pink, N blue, H grey. The H···O portions of the hydrogen bonds are coloured light blue.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O3^{iii}	0.82 (4)	2.37 (4)	3.070 (3)	143 (3)
N1—H1*B*···O4^{iv}	0.82 (4)	2.12 (4)	2.867 (3)	151 (4)
N2—H2*A*···O4^{iii}	0.88 (4)	1.96 (4)	2.799 (3)	160 (4)
N2—H2*B*···O3	0.80 (4)	2.02 (4)	2.769 (4)	156 (4)
N2—H2*C*···O2^{v}	0.82 (4)	2.51 (4)	2.849 (3)	106 (3)
N2—H2*C*···O2^{iv}	0.82 (4)	2.32 (4)	3.011 (4)	141 (4)
N2—H2*C*···O1^{vi}	0.82 (4)	2.45 (4)	3.073 (3)	133 (4)

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

The H atoms were located in difference maps and their positions and U_{iso} values were freely refined.

Data collection: Collect (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor 1997) & *SORTAV* (Blessing 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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