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$NH_4Fe^{II}H(SO_4)_2\cdot 2H_2O$

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(S–O) = 0.001 Å
H-atom completeness 89%
Disorder in solvent or counterion
R factor = 0.018
wR factor = 0.049
Data-to-parameter ratio = 10.7

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

The structure of the title compound, ammonium iron(II) hydrogen bis(sulfate) dihydrate, $NH_4FeH(SO_4)_2\cdot 2H_2O$, is isotypic with three similar kröhnkite-type structures of type E. The Fe^{2+} cation is surrounded by two water molecules and four partially protonated sulfate anions. The structure is completed by ammonium ions. The Fe and the N atom are located on centres of inversion.

Comment

We report here the X-ray crystal structure analysis of $NH_4FeH(SO_4)_2\cdot 2H_2O$, (I). The Fe and the N atom are located on centres of inversion. The Fe^{2+} cation in (I) is coordinated by two water molecules and four partially protonated sulfate anions. The structure is completed by ammonium ions. (I) belongs to the group of kröhnkite-type structures of type E (Fleck, Kolitsch & Hertweck, 2002; Fleck & Kolitsch, 2003) of which three isotypic structures have already been published: $KMgH(SO_4)_2\cdot 2H_2O$ (Mačícek *et al.*, 1994), $KFeH(SO_4)_2\cdot 2H_2O$ (Fleck, Kolitsch, Hertweck, Giester *et al.*, 2002) and $KMgH(SeO_4)_2\cdot 2H_2O$ (Troyanov & Morozov, 2002).

Experimental

Crystals of the title compound were obtained from a solution of equimolar amounts of $(NH_4)_2SO_4$ and $FeSO_4\cdot 7H_2O$ in 1 ml HNO_3 and 20 ml H_2SO_4 (1 molar) at ambient temperature.

Crystal data

$FeH_9NO_{10}S_2$
M_r = 303.05
Triclinic, $P\overline{1}$
a = 4.5955 (9) Å
b = 5.8420 (12) Å
c = 8.3811 (16) Å
α = 103.678 (15)°
β = 98.069 (16)°
γ = 95.638 (16)°
V = 214.43 (8) Å^3
Z = 1
D_x = 2.347 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6924 reflections
θ = 3.6–27.4°
μ = 2.29 mm^{-1}
T = 100 (2) K
Block, colourless
0.47 × 0.22 × 0.13 mm

Data collection

Stoe IPDS II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.405, T_{max} = 0.740
3969 measured reflections
980 independent reflections
922 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.6°
$h = -5 \rightarrow 5$
$k = -7 \rightarrow 7$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.049
S = 1.12
980 reflections
92 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0271P)^2 + 0.0862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.148 (9)

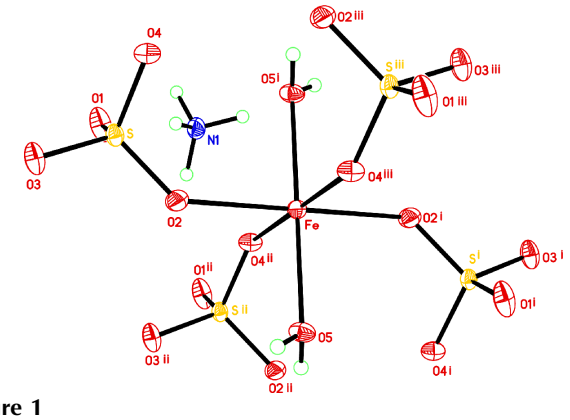

Figure 1
Perspective view of the title compound, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. Only one set of the disordered H atoms of the ammonium group is shown. Symmetry operators for generating equivalent atoms: (i) $-x, -y, -z$; (ii) $1+x, y, z$; (iii) $1-x, -y, -z$.

Table 1
Selected bond distances (Å).

Fe—O2	2.1029 (11)	S—O2	1.4670 (11)
Fe—O4^{ii}	2.1105 (11)	S—O4	1.4704 (11)
Fe—O5	2.1231 (12)	S—O1	1.5164 (11)
S—O3	1.4538 (11)		

Symmetry code: (ii) $1+x, y, z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O3^{iv}	0.70 (3)	2.07 (3)	2.7262 (16)	155 (3)
O5—H5B···O2^{v}	0.77 (3)	2.57 (3)	2.9967 (16)	117 (2)
O5—H5B···O3^{v}	0.77 (3)	2.59 (3)	3.2335 (17)	143 (2)
N1—H1A···O3^{vi}	0.908 (10)	1.995 (11)	2.9007 (12)	175 (5)
N1—H1C···O1	0.906 (10)	2.188 (14)	3.0790 (12)	168 (4)
N1—H1D···O4^{vii}	0.908 (10)	1.948 (14)	2.8433 (12)	168 (5)

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

The H atom of the partially protonated sulfate group, which has an occupancy factor of 0.5 and which is most probably bonded to O1, could not be located and was therefore omitted from the refinement. All other H atoms were refined isotropically. For the H atoms bonded to N, which are all disordered over two equally occupied sites, distance restraints were applied [N—H = 0.91 (1) Å].

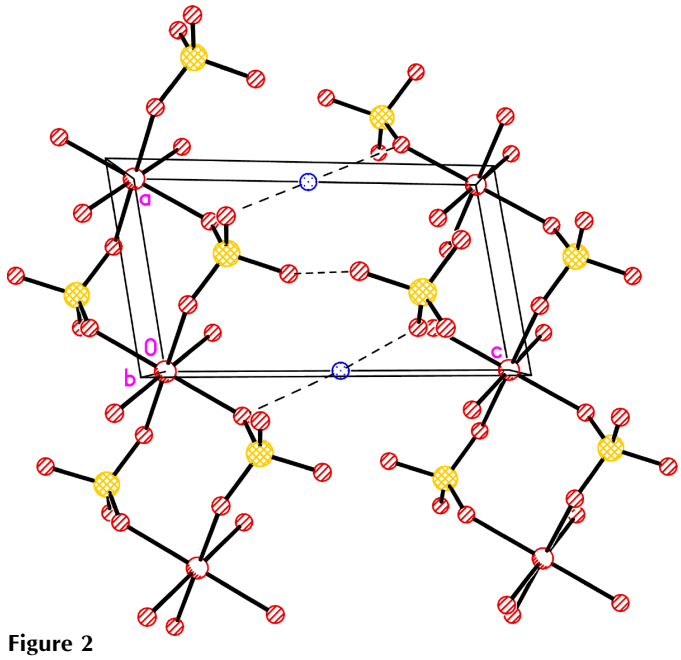

Figure 2
Packing diagram of the title compound, with an approximate view onto the *ac*-plane. Atom colours as in Fig. 1. H atoms are omitted for clarity.

Data collection: *X-Area* (Stoe & Cie, 2001); cell refinement: *X-Area*; data reduction: *X-Area*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97.

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