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Refinement of ferrous sulfate heptahydrate (melanterite) with low-temperature CCD data

Frank R. Fronczek,* Sibrina N. Collins and Julia Y. Chan

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA

Correspondence e-mail: fronz@chxray1.chem.lsu.edu

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Refinement of the title compound, $FeSO_4·7H_2O$, with CCD data at 120 K has led to a fivefold increase in precision over the previously reported structure based on film data. The H atoms have been located and refined. Two independent octahedral $[Fe(OH_2)_6]^{2+}$ cations lie on inversion centers, while one water molecule is uncoordinated. Fe—O distances are in the range 2.0795 (9)–2.1873 (9) Å.

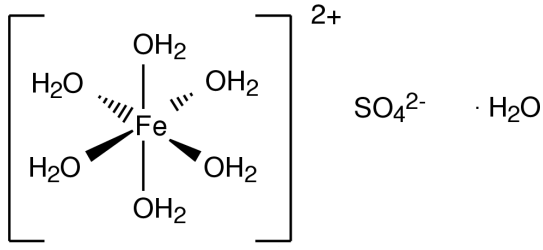
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(S–O) = 0.0008 Å
R factor = 0.029
wR factor = 0.076
Data-to-parameter ratio = 25.7

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

Comment

While attempting to prepare mixed-metal complexes of cysteine, we encountered crystals of the title compound. We discovered that the best available structure determination was based on 738 film data (Baur, 1964). We report herein the refinement of the structure using modern data-collection techniques. The excellent structure determination reported by Baur is confirmed, including the asymmetric pattern of Fe—O distances in the two centrosymmetric $[Fe(OH_2)_6]^{2+}$ ions [2.068 (5)–2.188 (5) Å (Baur, 1964) and 2.0795 (9)–2.1873 (9) Å from our data]. From the film data, the H-atom positions were not directly obtained, but were placed from geometric hydrogen-bonding considerations. We confirm Baur's placement of the H atoms except for that involved in the bifurcated hydrogen bond involving uncoordinated water O7*W* as donor, and O3 and O6*W* as acceptors (Table 2). Placement of that H atom had been considered ambiguous, and the bifurcated interaction which we observe was mentioned as a possibility by Baur (1964), who adequately described both the structure of the individual ions and the packing.



(I)

Experimental

The crystal used for data collection was taken from a commercial sample (J. T. Baker Chemical Co., lot 302120) and was reduced in size by partial dissolution using water.

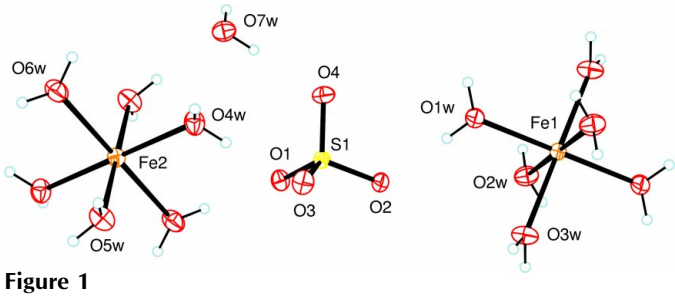


Figure 1
A view of ferrous sulfate heptahydrate with the numbering scheme and ellipsoids at the 70% probability level.

Crystal data

$[Fe(H_2O)_6](SO_4)\cdot H_2O$
$M_r = 278.02$
Monoclinic, $P2_1/c$
$a = 13.9969\ (3)$ Å
$b = 6.4803\ (2)$ Å
$c = 11.0211\ (2)$ Å
$\beta = 105.5959\ (11)^\circ$
$V = 962.85\ (4)$ Å^3
$Z = 4$
$D_x = 1.918$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8709 reflections
$\theta = 2.5$–36.3°
$\mu = 1.82$ mm^{-1}
$T = 120$ K
Fragment, pale blue–green
$0.25 \times 0.20 \times 0.17$ mm

Data collection

KappaCCD diffractometer (with Oxford Cryosystems Cryostream cooler)
ω scans with κ offsets
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.688$, $T_{max} = 0.734$
14 982 measured reflections
4580 independent reflections
3686 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 36.3^\circ$
$h = -23 \rightarrow 22$
$k = -10 \rightarrow 10$
$l = -17 \rightarrow 18$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.076$
$S = 1.04$
4580 reflections
178 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0305P)^2 + 0.2242P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.64$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0140 (11)

Table 1
Selected geometric parameters (Å).

Fe1—O1*W*	2.0795 (9)	Fe2—O6*W*	2.1873 (9)
Fe1—O2*W*	2.1474 (9)	S1—O1	1.4864 (9)
Fe1—O3*W*	2.1287 (9)	S1—O2	1.4822 (8)
Fe2—O4*W*	2.1034 (9)	S1—O3	1.4738 (8)
Fe2—O5*W*	2.0992 (9)	S1—O4	1.4813 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11···O1^{i}	0.82 (2)	1.93 (2)	2.7394 (12)	169 (2)
O1*W*—H12···O2	0.81 (2)	1.91 (2)	2.7208 (13)	177 (2)
O2*W*—H22···O2^{ii}	0.85 (2)	1.95 (2)	2.7879 (12)	167 (2)
O2*W*—H24···O4^{iii}	0.82 (2)	2.09 (2)	2.8972 (12)	166 (2)
O3*W*—H31···O1^{iv}	0.856 (19)	1.995 (19)	2.8426 (12)	170.4 (18)
O3*W*—H32···O2^{iii}	0.85 (2)	2.04 (2)	2.8734 (12)	164.5 (19)
O4*W*—H41···O3	0.82 (2)	1.97 (2)	2.7915 (12)	174.2 (18)
O4*W*—H47···O7*W*^{v}	0.80 (2)	1.92 (2)	2.7175 (13)	172.8 (19)
O5*W*—H54···O4^{v}	0.89 (2)	2.03 (2)	2.9167 (13)	173.9 (18)
O5*W*—H57···O7*W*^{vi}	0.79 (2)	1.91 (2)	2.7052 (13)	177 (2)
O6*W*—H61···O1^{vi}	0.87 (2)	1.96 (2)	2.8058 (12)	166.0 (19)
O6*W*—H63···O3^{vii}	0.90 (2)	1.87 (2)	2.7708 (12)	174.3 (19)
O7*W*—H74···O4	0.81 (2)	1.99 (2)	2.7712 (13)	162 (2)
O7*W*—H76···O3^{viii}	0.82 (2)	2.47 (2)	3.0242 (12)	126.0 (18)
O7*W*—H76···O6*W*^{ix}	0.82 (2)	2.30 (2)	2.9633 (13)	138.4 (18)

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

The coordinates of Baur (1964) were used as an initial refinement model, with some atoms moved to form connected sets. H atoms were located from difference maps and were individually refined. O—H distances are in the range 0.79 (2)–0.90 (2) Å, and U_{iso} values for H atoms are in the range 0.028 (5)–0.047 (6) Å^2.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; structure solution: coordinates of Baur (1964); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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