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$Sr_2Fe_2F_{10}.H_2O$, the First Hydrated Strontium Iron(III) Fluoride

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Abstract

The structure of strontium iron(III) pentafluoride hemihydrate, obtained by hydrothermal growth, has been determined by single-crystal X-ray diffraction. The structure contains octahedral $[FeF_5]_n$ *trans*-vertex-sharing chains, isolated $[FeF_5(H_2O)]$ octahedra, chains of SrF_{10} polyhedra sharing two opposite faces and SrF_8 isolated polyhedra.

Comment

During the course of our investigation of the SrF_2–$0.5Fe_2O_3$–(HF and/or H_3PO_4)–H_2O system using high-temperature (T = 973 K) hydrothermal growth to synthesize new fluorophosphated compounds, the use of 11 *M* HF as a solvent led to the new fluorinated compound $Sr_2Fe_2F_{10}.H_2O$.

The structure contains two types of Fe^{III} octahedra. Each $Fe1F_6$ octahedron shares two of its opposite corners with two other $Fe1F_6$ octahedra to form parallel $[FeF_5]_n$ *trans* chains running along [100], whereas the $[Fe2F_5(H_2O)]$ octahedra are isolated. The $Fe1F_6$ octahedra are distorted, with the longest Fe1—F distances [2.008 (1) Å] involving the shared F6 atoms and the mean Fe—F distance being very close to the sum of the ionic radii (Shannon, 1976). Within these chains, the Fe—F6—Fe angle is reduced to 155.4 (2)°. The presence of a water molecule in the $[Fe2F_5(H_2O)]$ octahedra was confirmed by a valence bond analysis (Brown, 1981). A peak in a general position was observed 1.06 Å from O1 in a difference Fourier map and was assigned as an H atom.

There are two types of Sr polyhedra. The $Sr1F_{10}$ (bicapped cube) polyhedra share two opposite faces to form parallel linear chains running along [100]. Each chain is connected to one $[FeF_5]_n$ chain by one edge shared between an Sr1 polyhedron and an Fe1 octahedron. Between these double chains $Sr2F_8$ polyhedra (square antiprism) are inserted. These share two opposite corners with Fe2 octahedra to form zigzag chains along [100]. Among the hydrated metal fluorides containing $[M^{III}F_x(H_2O)_{6-x}]^{n-}$ octahedra, $Sr_2Fe_2F_{10}.H_2O$ is the first structure which exhibits octahedral $[FeF_5]_n$ *trans* chains with isolated $[FeF_5(H_2O)]$ octahedra.

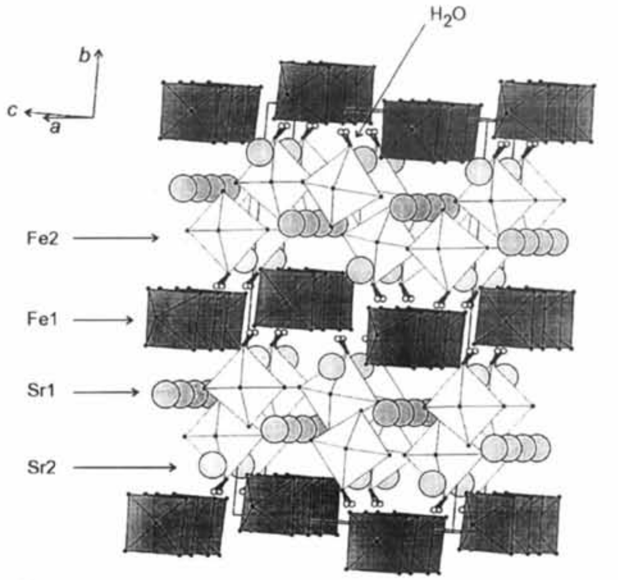

Fig. 1. A perspective view of $Sr_2Fe_2F_{10}.H_2O$, showing the $[FeF_5]_n$ chains and $[FeF_5(H_2O)]$ isolated octahedra.

Experimental

Single crystals of $Sr_2Fe_2F_{10}.H_2O$ were obtained by heating a mixture of SrF_2 (2.03 mmol), Fe_2O_3 (1.01 mmol) and 2 ml of 11 *M* HF under hydrothermal conditions (T = 973 K, P = 180 MPa). A crystal was chosen for X-ray analysis by optical examination and its quality was tested with Laue photography.

Crystal data

$Sr_2Fe_2F_{10}.H_2O$	Mo $K\alpha$ radiation
M_r = 494.93	λ = 0.71073 Å
Orthorhombic	Cell parameters from 38 reflections
Cmca	θ = 14.02–15.90°
a = 7.848 (2) Å	μ = 16.163 mm^{-1}
b = 19.867 (4) Å	T = 293 (2) K
c = 10.773 (3) Å	Plate
V = 1679.7 (7) Å^3	0.190 × 0.128 × 0.048 mm
Z = 8	Colourless
D_x = 3.914 Mg m^{-3}	
D_m not measured	

Data collection

Stoe–Siemens AED-2 diffractometer	R_{int} = 0.0157
Profile data from ω/θ scans	θ_{max} = 35°
Absorption correction: Gaussian by integration	h = −12 → 12
T_{min} = 0.13, T_{max} = 0.45	k = −2 → 32
1844 measured reflections	l = 0 → 17
1741 independent reflections	3 standard reflections frequency: 60 min intensity decay: 3.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.075$
$S = 1.024$
1741 reflections
83 parameters
Only coordinates of H atom refined
$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.12$ e Å^{-3}
$\Delta\rho_{min} = -1.43$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)*

U_{iso} for H atom, $U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for all others.

	x	y	z	U_{iso}/U_{eq}
Sr1	1/4	0.28088 (2)	1/4	0.00975 (11)
Sr2	0	0.11917 (3)	0.45274 (5)	0.00814 (10)
Fe1	1/4	0.46627 (4)	1/4	0.00809 (15)
Fe2	0	0.18264 (4)	0.06392 (8)	0.0078 (2)
F1	0.1994 (3)	0.39572 (11)	0.1356 (2)	0.0137 (5)
F2	0.2403 (3)	0.18836 (11)	0.0780 (2)	0.0144 (5)
F3	0.2919 (3)	0.03352 (11)	0.1268 (3)	0.0189 (5)
F4	0	0.3010 (2)	0.3880 (3)	0.0133 (7)
F5	0	0.1800 (2)	0.2468 (4)	0.0164 (7)
F6	0	0.4704 (2)	0.2890 (4)	0.0156 (7)
F7	0	0.28067 (15)	0.1019 (3)	0.0103 (6)
O1	0	0.0841 (2)	0.0472 (4)	0.0140 (8)
H1	0.098 (3)	0.0527 (19)	0.083 (4)	0.013

Table 2. *Selected geometric parameters (Å, °)*

Sr1—F4 × 2	2.494 (2)	Fe1—F1 × 2	1.908 (2)
Sr1—F7 × 2	2.529 (2)	Fe1—F3^{iv} × 2	1.912 (2)
Sr1—F2 × 2	2.611 (2)	Fe1—F6 × 2	2.008 (1)
Sr1—F1 × 2	2.623 (2)	Fe2—F2 × 2	1.896 (3)
Sr1—F5 × 2	2.805 (3)	Fe2—F4^{v}	1.923 (4)
Sr2—F2^{i} × 2	2.480 (3)	Fe2—O1	1.965 (4)
Sr2—F3^{i} × 2	2.509 (2)	Fe2—F5	1.970 (4)
Sr2—F5	2.527 (4)	Fe2—F7	1.990 (3)
Sr2—F1^{ii} × 2	2.533 (3)	O1—H1	1.07 (3)
Sr2—F7^{iii}	2.557 (3)		
Fe1^{vi}—F6—Fe1	155.4 (2)	H1^{vii}—O1—H1	92.8 (4)

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

The maximum residual electron density was found 1.0 Å from Sr1.

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 option *PATT* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *DIAMOND* (Bergerhoff, 1996).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1176). Services for accessing these data are described at the back of the journal.

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A Tricapped Trigonal Prismatic $EuO(H_2O)_8$ Site in Trihydrogen Tris(octaaquaeuropium) Dipotassium Digermanohexatitanooctadecatungstate(14–) Tridecahydrate

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Abstract

This paper reports the first observation of a tricapped trigonal prismatic $EuO(H_2O)_8$ site containing one anion O and eight aqua O atoms in $K_2[Eu(H_2O)_8]_3H_3[(GeTi_3W_9O_{37})_2O_3].13H_2O$. Each half of the $[(GeTi_3W_9O_{37})_2O_3]^{14-}$ anion, which exhibits almost the same structure as the anion in $K_9H_5[(GeTi_3W_9O_{37})_2O_3].16H_2O$, is coordinated by either three Eu^{3+} or two K^+ atoms.

Comment

During the course of our studies of the crystal structures of photoluminescent polyoxometalloeuropates, the crystal fields of the Eu^{3+} sites in the polyoxotungstoeuropates and polyoxomolybdoeuropates have been characterized as a square antiprism and tricapped trigonal prism, respectively. $Na_9[EuW_{10}O_{36}].32H_2O$ (Sugeta & Yamase, 1993) and $K_{15}H_3[Eu_3(H_2O)_3(W_5O_{18})_3(SbW_9O_{33})].37H_2O$ (Yamase, Naruke & Sasaki, 1990) have square antiprismatic EuO_8 and $EuO_6(H_2O)_2$ sites, respectively. $(NH_4)_{12}H_2[Eu_4(H_2O)_{16}(MoO_4)(Mo_7O_{24})_4].13H_2O$ (Naruke, Ozeki & Yamase, 1991) and $Eu_2(H_2O)_{12}(Mo_8O_{27}).6H_2O$ (Yamase & Naruke, 1991) have tricapped trigonal prismatic $EuO_5(H_2O)_4$ and $EuO_3(H_2O)_6$ sites, respectively. It has recently been found that $Na_7H_{19}\{[Eu_3O(OH)_3(OH_2)_3]_2Al_2(Nb_6O_{19})_5\}$ has bi-