

SHORT-FORMAT PAPERS

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Room-Temperature Structure of Iron(II) Hexafluorostannate(IV) Hexahydrate

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Abstract. $\text{FeSnF}_6 \cdot 6\text{H}_2\text{O}$, $M_r = 397.42$, rhombohedral, $R\bar{3}$, $a = 9.826$ (2), $c = 10.106$ (2) Å (hexagonal cell, $\gamma = 120^\circ$), $V = 845.0$ (5) Å³, $Z = 3$, $D_x = 2.344$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 3.64$ mm⁻¹, $F(000) = 570$, $T = 293$ K, $R = 0.029$, 1067 unique reflections, growth from a solution of elementary fluorides by slow evaporation. H atoms of water molecules in the isolated $\text{Fe}(\text{H}_2\text{O})_6$ octahedra point towards the fluoride anions of neighbouring SnF_6 octahedra. The Sn—F and Fe—O distances are 1.945 (2) and 2.116 (2) Å respectively.

Experimental. Growth from a solution prepared by reaction of 40% HF with Fe metal and addition of SnF_2 with Fe/Sn = 1/2, slow evaporation in air atmosphere and oxidation of Sn^{2+} . Platelet crystal $0.05 \times 0.15 \times 0.24$ mm limited by (1210), (1010) and (0001) faces respectively. Data collected on a Siemens AED2 four-circle diffractometer; ω -2 θ step scan mode in N steps of 0.035° , $38 \leq N \leq 45$, time per step: min. 1 s, max. 4 s; aperture $D = 3.5$ mm. Lattice constants based on 30 reflections measured in double-step scan mode at $\pm(2\theta = 30^\circ)$; absorption correction by the Gauss method, $A_{\text{max}} = 0.85$, $A_{\text{min}} = 0.57$. Intensity measurement to $2\theta \leq 80^\circ$ within range $0 \leq |h| \leq 15$, $0 \leq |k| \leq 15$, $0 \leq l \leq 18$. Standard reflections 402, 150, 344, intensity variation 1.5%, 2328 reflections measured, 1067 independent reflections used for refinements [$|F| > 6\sigma(|F|)$], $R_{\text{int}} = 0.017$.

Structure solved by isotypy with $\text{NiSnCl}_6 \cdot 6\text{H}_2\text{O}$. Isotropic thermal motion of H atoms fixed to 6.0 Å². F magnitudes used in least-squares refinements: 30 parameters refined; mean $\Delta/\sigma = 0.04$, max. $\Delta/\sigma = 0.05$ for non-H atoms, max. $\Delta/\sigma = 0.35$ for H atoms; secondary-extinction factor: $x = 0.1(1) \times 10^{-7}$; atomic scattering factors for Fe^{2+} , Sn^{4+} , F^- , O, H from *International Tables for X-ray Crystallography*

(1974, Vol. IV); calculations with *SHELX76* (Sheldrick, 1976). Max. and min. heights in final difference F map: $+0.9$ and -1.0 e Å⁻³. $R = 0.029$, $wR = 0.030$; $w = 1.55/[\sigma^2(|F_o|) + 0.00003F_o^2]$.

Related literature. All the compounds of the series $M^{II}\text{Sn}^{IV}\text{F}_6 \cdot 6\text{H}_2\text{O}$ are thought to be isotypic with $\text{NiSnCl}_6 \cdot 6\text{H}_2\text{O}$ ($R\bar{3}$) (Hassel, 1927; Pauling, 1930). The same structure was observed for many other hexafluoride hexahydrates, such as $M\text{SiF}_6 \cdot 6\text{H}_2\text{O}$ with $M = \text{Co}, \text{Ni}, \text{Zn}$ (Kodera, Torii, Osaki & Watanabe, 1972; Ray, Zalkin & Templeton, 1973). However, $\text{FeSiF}_6 \cdot 6\text{H}_2\text{O}$ was described differently: $R\bar{3}m$ (Hamilton, 1962; Syoyama & Osaki, 1972) or close to $P\bar{3}m1$ (Jehanno & Varret, 1975). More recently, $\text{FeSiF}_6 \cdot 6\text{H}_2\text{O}$ was described as a periodic antiphase structure built on ordered monoclinic cells, $P2_1/c$ (Chevrier, Hardy & Jehanno, 1981). Recently, we have found evidence (Calage, Tortevois & Varret, 1990) from ⁵⁷Fe and ¹¹⁹Sn Mössbauer spectroscopy of a phase transition in $\text{FeSnF}_6 \cdot 6\text{H}_2\text{O}$ at $T = 65$ K, whose many aspects are reminiscent of the $P\bar{3}m1 \rightarrow P2_1/c$ transition observed in $\text{FeSiF}_6 \cdot 6\text{H}_2\text{O}$ (Chappert, Jehanno & Varret, 1977). In order to compare the phase transitions in fluorosilicates and fluorostannates with the space-group transformations, we have undertaken this structure determination at room temperature. It is to be noted that the plane symmetry in $P\bar{3}m1$ implies a statistical disorder of mirror-related octahedron rotations and this feature is lost in the $R\bar{3}$ space group.

The final atomic coordinates and equivalent isotropic thermal parameters‡ are given in Table 1. Selected distances and angles are listed in Table 2.

‡ Lists of structure factors, anisotropic thermal parameters and atomic coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52861 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Coordinates and isotropic temperature factors (\AA^2) in $\text{FeSnF}_6 \cdot 6\text{H}_2\text{O}$ (e.s.d.'s in parentheses)

	x	y	z	B
Fe	0	0	$\frac{1}{2}$	1.99 (2)*
Sn	0	0	0	1.67 (1)*
F	0.1460 (2)	0.1718 (2)	0.1126 (2)	4.2 (1)*
O	0.1840 (3)	0.1701 (3)	0.3813 (2)	4.8 (1)*
H(1)	0.310 (5)	0.223 (5)	0.411 (4)	6.0
H(2)	0.161 (6)	0.151 (5)	0.271 (4)	6.0

* Equivalent isotropic temperature factor, defined as $B_{\text{eq}} = [4/3] \sum_i \beta_i a_i^2$, (Hamilton, 1959).

Table 2. Selected bond lengths (\AA) and angles ($^\circ$) in $\text{FeSnF}_6 \cdot 6\text{H}_2\text{O}$ (e.s.d.'s in parentheses)

6 \times Fe—O	2.116 (2)	O—H(2)	1.14 (4)
6 \times Sn—F	1.945 (2)	F...H(1)	1.61 (5)
O—H(1)	1.12 (5)	F...H(2)	1.63 (4)
O—H(1)...F	176 (4)	Fe—O—H(2)	114 (2)
O—H(2)...F	165 (4)	O—Fe—O	89.96 (8)
H(1)—O—H(2)	115 (4)	F—Sn—F	89.21 (8)
Fe—O—H(1)	121 (2)		

Unlike $\text{FeSiF}_6 \cdot 6\text{H}_2\text{O}$, no extra reflection, signifying a lowering of symmetry from $R\bar{3}$, can be seen in $\text{FeSnF}_6 \cdot 6(\text{H}_2\text{O})$.

The structure is built up from isolated $\text{Fe}(\text{H}_2\text{O})_6$ and SnF_6 octahedra. The stability of the structure is ensured by hydrogen bonds: H atoms of water molecules point towards fluoride anions of neighbouring octahedra (Fig. 1 and Table 2). The mean Sn—F distance [1.945 (2) \AA] is close to the sum of the ionic radii of Sn^{4+} (0.69 \AA) and F^- (1.285 \AA).

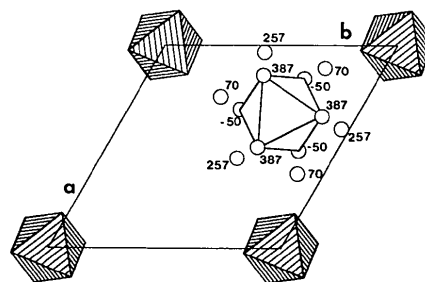


Fig. 1. [0001] projection of $\text{FeSnF}_6 \cdot 6\text{H}_2\text{O}$. SnF_6 octahedra (at $z = 0$) are shaded; heights of the H atoms of the water molecules in the $\text{Fe}(\text{H}_2\text{O})_6$ octahedra (at $z = 1/6$) are multiplied by 10^3 .

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Aluminium Trimagnesium Diplatinum

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Abstract. AlMg_3Pt_2 , $M_r = 490.10$, cubic, $Fd\bar{3}m$, $a = 11.802$ (1) \AA , $V = 1643.9$ (4) \AA^3 , $Z = 16$, $D_x = 7.92$ Mg m^{-3} , $\lambda(\text{Cu K}\alpha) = 1.5418$ \AA , $\mu = 133.0$ mm^{-1} , $F(000) = 3280$, $T = 296$ K. The structure was refined to $R = 0.017$, $wR = 0.025$ for 78 absorption-corrected reflections. AlMg_3Pt_2 crystallizes as an ordered ternary variant of the NiTi_2 structure.

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Experimental. The alloy was synthesized by reaction of a stoichiometric mixture of the elements in a modified Belt-type high-pressure apparatus (Range & Leeb, 1975). This method avoids the vaporization losses of Al and Mg inevitably found in normal-pressure experiments and allows for exact control of composition. From the reaction product obtained at 40 kbar ($\equiv 4$ GPa) and 1675 K (BN capsule, reaction time 30 min, subsequent slow cooling to 1275 K and quenching to ambient conditions) a crystal fragment