$2\theta_{\min} = 10.00$, $2\theta_{\max} = 119.98°$
Increment in $2\theta = 0.03°$
Profile function: pseudo-Voigt function
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Gd1	0.19320 (14)	0.10615 (14)	0.44679 (9)	0.0008 (4)
Gd2	0	0.3958 (2)	0.4029 (9)	0.0008
Ga	0	0.1947 (4)	0	0.0003 (13)
O1	0.155 (2)	0.2512 (14)	0.183 (3)	0.004 (3)
O2	0.338 (2)	0.013 (2)	0.219 (5)	0.004
O3	0	0.049 (2)	0.167 (5)	0.004
O4	0	0.202 (2)	0.661 (4)	0.004

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

Gd1—O2	2.08 (2)	Gd2—O1	2.46 (2)
Gd1—O1	2.21 (2)	Gd2—O1$^{\rm vi}$	2.46 (2)
Gd1—O4	2.36 (2)	Gd2—O2$^{\rm ii}$	2.49 (2)
Gd1—O2$^{\rm i}$	2.39 (2)	Gd2—O2$^{\rm vii}$	2.49 (2)
Gd1—O3	2.41 (2)	Gd2—O4	2.60 (2)
Gd1—O1$^{\rm ii}$	2.48 (2)	Ga—O1	1.83 (2)
Gd1—O3$^{\rm iii}$	2.74 (2)	Ga—O1$^{\rm vi}$	1.83 (2)
Gd2—O2$^{\rm iv}$	2.21 (2)	Ga—O4$^{\rm viii}$	1.86 (2)
Gd2—O2$^{\rm v}$	2.21 (2)	Ga—O3	1.89 (2)
O1—Ga—O4$^{\rm viii}$	122.1 (7)	O4$^{\rm viii}$—Ga—O3	121.8 (12)
O1—Ga—O3	92.2 (8)	O4$^{\rm viii}$—Ga—O1$^{\rm vi}$	122.1 (7)
O1—Ga—O1$^{\rm vi}$	99.0 (12)	O3—Ga—O1$^{\rm vi}$	92.2 (8)

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

The possible space groups were *Cmc*2₁, *C*2*cm* and *Cmcm* from the systematic extinction conditions. The structure could be solved only in *Cmc*2₁ by direct methods. During the Rietveld refinement, a preferred orientation correction was carried out using the March–Dollase function (Dollase, 1986). The adjustable parameter of the function was refined to be 0.956 (2) for a (100) preferred orientation of plate crystals. The isotropic displacement parameters were constrained to be the same for the Gd atoms and for the O atoms. The atomic parameters were standardized using the program *STRUCTURE TIDY* (Gelato & Parthé, 1987).

Data collection: *RISM* (Rigaku Corporation, 1995). Program(s) used to solve structure: *EXPO* (Altomare *et al.*, 1998). Program(s) used to refine structure: *RIETAN*97 (Izumi, 1997). Molecular graphics: *ATOMS* (Dowty, 1998).

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$SrFeP_2O_7$, a new homologue in the AMP_2O_7 series

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Abstract

Strontium iron diphosphate, $SrFeP_2O_7$, was obtained from hydrothermal synthesis and its structure determined by single-crystal X-ray diffraction analysis. The main crystallographic feature is the 4+1 square-pyramidal coordination polyhedron around the Fe atom. The structure is built up from corner-sharing [FeO_5] pyramids and [P_2O_7] bitetrahedral units. Such a connection gives rise to a three-dimensional framework,

with Sr^{2+} cations inserted in channels composed of five-membered rings running along [100].

Comment

The investigation of the SrF_2–$\frac{1}{2}Fe_2O_3$–(HF and/or H_3PO_4)–H_2O system using high-temperature hydrothermal growth was carried out with a double aim: (i) the synthesis of new fluorophosphated compounds and (ii) the understanding of the role of solvent through the P:F ratio within resulting fluorophosphates. The use of a 7 *M* H_3PO_4 solvent led to the compound $SrFeP_2O_7$, which is a new homologue in the AMP_2O_7 series. This compound results from the reduction of Fe^{III} to Fe^{II} and has been confirmed by a bond-valence analysis (Brown, 1981). The hydrothermal medium became the reductor. Such an observation has already been made (Kuznetsov, 1968) and can be explained by traces of water outside the platinum tube, between the tube and the steel lining of the autoclave. Metallic iron from steel is oxidized with hydrogen production. The gas migrates through the platinum and reacts with the elements inside the tube. The water can be brought in by moist nitrogen, which applies the external counter-pressure. $SrFeP_2O_7$ is isostructural with $BaCuP_2O_7$ (Riou & Goreaud, 1990) and $SrCoP_2O_7$ (Riou & Raveau, 1991), and derives from α-$Ca_2P_2O_7$ (Calvo, 1968). The structure is built up from two kinds of fundamental units: (i) $[FeO_5]$ square pyramids and (ii) $[P_2O_7]$ bitetrahedra which share corners to give rise to a three-dimensional framework. The Sr^{2+} cations are inserted inside channels composed of five-membered rings running along [100] (as shown in Fig. 1) and adopt an eightfold coordination polyhedron, with distances ranging from 2.53 to 2.78 Å. Fe atoms are located in the vicinity of the centre of the square pyramid, with mean Fe—O distances very close to the sum of the ionic radii (Shannon, 1976).

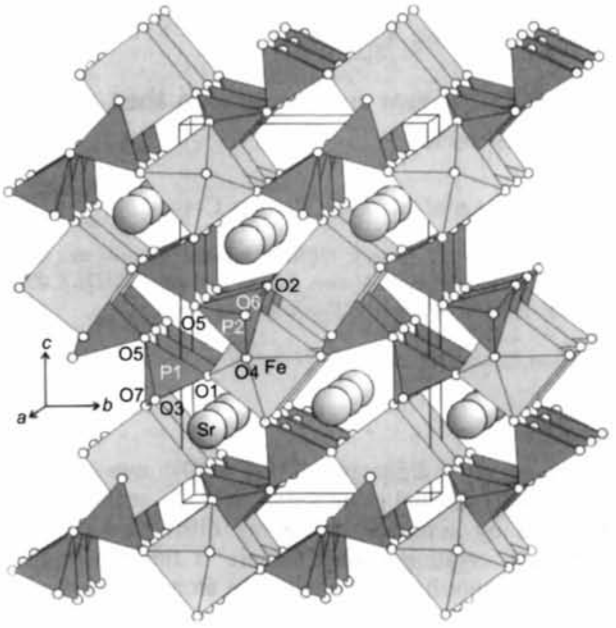

Fig. 1. Perspective view of $SrFeP_2O_7$ along [100] showing the Sr^{2+} cations inserted in channels composed of five-membered rings.

The $[P_2O_7]$ bitetrahedra are characterized by a longer bridging P—O distance: 1.604 (2) Å *versus* an average of 1.523 (2) Å for the other bonds. At the final stage of refinement, the maximum residual electron density of 1.99 e Å^{-3} was found 0.7 Å from the Sr atom. The displacement ellipsoids in the asymmetric unit are shown in Fig. 2.

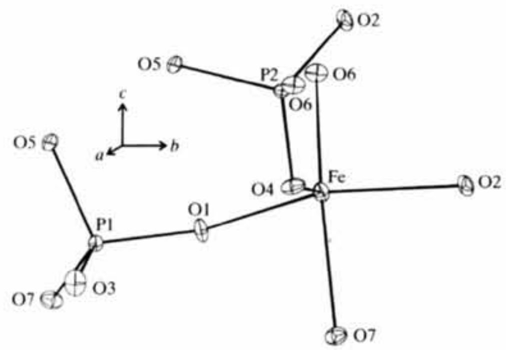

Fig. 2. Displacement ellipsoid plot (50% probability) of the title compound.

Experimental

Single crystals of $SrFeP_2O_7$ were obtained by hydrothermal growth starting from the reactants SrF_2 and Fe_2O_3 in the molar ratio 2:1 in an aqueous solution of phosphoric acid (7 *M*). The mixture was sealed in a platinum tube (filling level 40%) and heated at 956 K for 24 h. The maximum pressure reached was 186 MPa. The resulting product was filtered off, washed with distilled water and dried in air at room temperature. The main by-product was unreacted SrF_2.

Crystal data

$SrFeP_2O_7$
M_r = 317.41
Monoclinic
$P2_1/n$
a = 5.370 (2) Å
b = 8.268 (4) Å
c = 12.693 (3) Å
β = 90.37 (2)°
V = 563.5 (4) Å^3
Z = 4
D_x = 3.741 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 34 reflections
θ = 14–16°
μ = 12.59 mm^{-1}
T = 291 (2) K
Platelet
0.114 × 0.104 × 0.095 mm
Translucent light pink

Data collection

Stoe Siemens AED-2 diffractometer

$R_{\rm int}$ = 0.035
$\theta_{\rm max}$ = 34.97°

Profile data from ω/θ scans
Absorption correction: none
4171 measured reflections
2471 independent reflections
1922 reflections with $I > 2\sigma(I)$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 13$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 60 min
intensity decay: 4.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.090$
$S = 1.018$
2471 reflections
101 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.988$ e Å^{-3}
$\Delta\rho_{\min} = -1.928$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0288 (16)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sr1—O3^{i}	2.533 (2)	Fe1—O4	2.113 (2)
Sr1—O3	2.559 (2)	Fe1—O7	2.199 (2)
Sr1—O7	2.566 (2)	P1—O3^{vi}	1.513 (2)
Sr1—O2^{ii}	2.637 (2)	P1—O1^{vii}	1.515 (2)
Sr1—O6^{iii}	2.648 (2)	P1—O7	1.529 (2)
Sr1—O1^{iv}	2.657 (3)	P1—O5^{iii}	1.605 (2)
Sr1—O4	2.722 (2)	P2—O4	1.523 (2)
Sr1—O7^{i}	2.776 (2)	P2—O6	1.524 (2)
Fe1—O1	1.988 (2)	P2—O2^{viii}	1.536 (2)
Fe1—O2^{v}	2.049 (2)	P2—O5	1.604 (2)
Fe1—O6^{vi}	2.113 (2)		
O3^{vi}—P1—O1^{vii}	113.78 (13)	O4—P2—O2^{viii}	111.98 (12)
O3^{vi}—P1—O7	112.94 (13)	O6—P2—O2^{viii}	111.02 (12)
O1^{vii}—P1—O7	108.35 (13)	O4—P2—O5	103.91 (12)
O3^{vi}—P1—O5^{iii}	107.80 (13)	O6—P2—O5	108.02 (12)
O1^{vii}—P1—O5^{iii}	105.71 (12)	O2^{viii}—P2—O5	106.41 (12)
O7—P1—O5^{iii}	107.86 (12)	P2—O5—P1^{ix}	127.39 (14)
O4—P2—O6	114.82 (12)		

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

Absorption corrections were attempted (Gaussian method), but they did not improve the results of the refinement. The rather complex shape of the crystal makes a good numerical correction difficult to obtain. Thus, the results are based on observed structure factors with no absorption correction. Only about half the reflections in the *hkl* range indicated could be measured, but all independent reflections were measured at least once.

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 (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: BR1225). Services for accessing these data are described at the back of the journal.

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Ta_5Sb_4

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Abstract

The title compound, pentatantalum tetraantimonide, was obtained as a by-product of the reaction between Ba and Sb mixed in a 1:2 molar ratio using tantalum as a reaction container. This compound adopts the Ti_5Te_4 structure type, which consists of chains of vertex-sharing Ta_6Sb_8 face-capped octahedral clusters. The resulting formulation is $Ta_4Ta_{2/2}Sb_{8/2}$. Distance ranges are: Ta—Ta 2.930 (1)–3.301 (1) and Ta—Sb 2.775 (2)–3.074 (1) Å; there are no bonding Sb···Sb distances. Closer inspection reveals that the Ta substructure also resembles chains cut out from *bcc* packing.

Comment

During investigations on the binary barium–antimony system using sealed tantalum containers for their preparation, the compound Ta_5Sb_4 was obtained as a side product. Previously, this compound was prepared by direct reaction between the elements and characterized by X-ray powder diffraction (Boller & Nowotny, 1964; Furuseth *et al.*, 1965). Ta_5Sb_4 is isostructural with tetragonal Ti_5Te_4 (Grønvold *et al.*, 1961), which adopts the space group *I*4/*m*. Unit-cell parameters for Ta_5Sb_4 have been determined to be $a = 10.2357 (14)$ and $c = 3.5425 (7)$ Å. The shortest interatomic distances

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