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Distrontium Diantimonate(V). A Rietveld Refinement of Neutron Powder Diffraction Data

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Abstract. $Sr_2Sb_2O_7$: $M_r = 530{\cdot}74$, orthorhombic, *Imma*. At $T = 300$ K: $a = 7{\cdot}4557\,(2)$, $b = 10{\cdot}3708\,(3)$, $c = 7{\cdot}6860\,(1)$ Å, $V = 594{\cdot}29\,(3)$ Å^3, $Z = 4$, $D_x = 5{\cdot}932\,(2)$ Mg m^{-3}, $\mu R = 0{\cdot}12$, $\lambda = 2{\cdot}5804\,(1)$ Å, $R_I = 1{\cdot}79$, $R_p = 3{\cdot}48$, $R_{wp} = 4{\cdot}59\%$. The structure has been refined by Rietveld analysis of neutron powder diffraction data recorded at room temperature for 85 reflections. The structure is of the weberite type.

Introduction. The antimonates $Ca_2Sb_2O_7$ and $Cd_2Sb_2O_7$ crystallize with structures of both the pyrochlore and the weberite type, but $Sr_2Sb_2O_7$ is known only as a weberite (Knop, Demazeau & Hagenmuller, 1980). In relation to $Ba_2U_2O_7$ (Cordfunke & IJdo, 1988) with weberite structure and space group *Imma*, it was desirable to know the structure of $Sr_2Sb_2O_7$ in detail. There is some confusion about the space group of $Sr_2Sb_2O_7$. Bystrom (1945) reports its space group as *Imm*2 ($a = 7{\cdot}28$, $b = 7{\cdot}44$, $c = 10{\cdot}18$ Å), Burchard & Rudorff (1979) as *Imnm*. Giuseppetti & Tadini (1978) report for the weberite Na_2MgAlF_7 the space group *Imma*. Knop, Cameron & Jochem (1982) state that descriptions of structures of weberites in space groups *Imm*2, $I2_12_12_1$ and *Imma* must be regarded as practically indistinguishable.

Experimental. AR starting materials $SrCO_3$ and Sb_2O_3 were thoroughly mixed in an agate mortar in the approximate ratios. The sample was heated in a platinum crucible at 1173 K for 7 days and at 973 K for 2 days. X-ray powder diffraction patterns were obtained using a Philips PW 1050 diffractometer. The systematic extinctions indicate space group *Imma* or *Im*2*a*. Since no single crystals were available, Rietveld's (1969) method was used for the refinement of neutron powder diffraction data; neutron powder profile recorded at the Petten High-Flux Reactor; $5 < 2\theta < 163°$ in steps of 0·1°; neutrons at 295 K from the (111) planes of a Cu crystal; pyrolytic graphite with a total thickness of 120 mm as a second-order filter; Soller slits, horizontal divergence 0·5°, placed between the reactor and the monochromator and in front of the four ^{3}He counters; sample holder ($\varnothing = 14{\cdot}46$ mm) consisted of a 'V' tube, closed with Cu plugs with 'O' rings. No

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Table 1. *Fractional atomic coordinates and thermal parameters of* $Sr_2Sb_2O_7$ *at* 300 K

	x	y	z	B (Å²)
Sr1	0	0	0	0·42 (6)
Sr2	0·25	0·25	0·75	0·59 (2)
Sb1	0·25	0·25	0·25	0·01 (5)
Sb2	0	0	0·5	0·18 (6)
O1	0	0·25	0·1538 (3)	0·32 (5)
O2	0	0·4092 (2)	0·7180 (2)	0·31 (4)
O3	0·2020 (1)	0·3809 (1)	0·4299 (1)	0·32 (4)

Table 2. *Atomic distances* (Å) *and selected angles* (°) *at* $T = 300$ K

$Sr1—O_8$ hexagonal prism		$Sr2—O_8$ square prism	
Sr1–O1	2·850 (1) 2×	Sr2–O2	2·502 (1) 4×
Sr1–O2	2·363 (2) 2×	Sr2–O3	2·8327 (8) 4×
Sr1–O3	2·5985 (8) 4×		
$Sb1—O_6$ octahedron		$Sb2—O_6$ octahedron	
Sb1–O1	2·0052 (9) 2×	Sb2–O2	1·922 (2) 2×
Sb1–O3	1·9705 (9) 4×	Sb2–O3	2·0209 (9) 4×
O1–Sb1–O1	180·	O2–Sb2–O2	180·
O3–Sb1–O3	87·09 (5)	O3–Sb2–O3	96·36 (5)
O1–Sb1–O3	95·16 (5)	O2–Sb2–O3	93·84 (5)

precautions to avoid preferred orientation were taken. Maximum absorption correction 5%, $\mu R = 0{\cdot}12$ (Weber, 1967). The background was determined from points in the diagram not containing any contribution from reflections by interpolation between these points. Statistically expected value of R_p 1·91%. The structure of the weberite Na_2MgAlF_7 (Giuseppetti & Tadini, 1978) was used as a trial model: Sr1 in 4(*a*) (0,0,0), Sr2 in 4(*d*) (0·25, 0·25, 0·75), Sb1 in 4(*c*) (0·25, 0·25, 0·25), Sb2 in 4(*b*) (0,0,0·5), O1 in 4(*e*) (0,0·25,*z*) O2 in 8(*h*) (0,*y*,*z*) and O3 in 16(*h*) (*x*,*y*,*z*). The coherent scattering lengths used were: Sr 7·02, Sb 5·64 and O 5·81 fm (Koester, Rauch, Herkens & Schroeder, 1981). 22 parameters in the refinement: one scale factor, three half-width parameters defining the Gaussian line shape of the reflections, the counter zero error, an asymmetry parameter, the unit-cell parameters, the atomic positions and the isotropic thermal parameters. The Rietveld program minimizes the function $X^2 = \sum_i w_i \times [y_i(\text{obs.}) - (1/c)y_i(\text{calc.})]^2$, where y_i(obs.) and y_i(calc.) are the observed and calculated data points, w_i is the statistical weight $[1/y_i(\text{obs.})]$ of each data point and c is the scale factor. The following R factors were calculated:

$$R_I = 100\textstyle\sum|I_i(\text{obs.}) - (1/c)I_i(\text{calc.})|/\sum I_i(\text{obs.}),$$
$$R_p = 100\textstyle\sum|y_i(\text{obs.}) - (1/c)y_i(\text{calc.})|/\sum y_i(\text{obs.}),$$
$$R_{wp} = 100[\textstyle\sum w_i|y_i(\text{obs.}) - (1/c)y_i(\text{calc.})|^2/\sum w_i|I_i(\text{obs.})|^2]^{1/2},$$

where I_i(obs.) and I_i(calc.) are the observed and calculated integrated intensities of each reflection. $\Delta/\sigma < 0{\cdot}3$ in final cycle. Largest element of the correlation matrix for structural parameters 0·72.

Discussion. Atomic parameters are given in Table 1 and selected atomic distances in Table 2. The agreement between the observed and calculated profiles is shown in Fig. 1.* Fig. 2. gives a [100] projection of the structure of $Sr_2Sb_2O_7$.

The space group *Imma* allows an adequate description of the structure. The same space group was found for $Ba_2U_2O_7$ with neutron diffraction and electron diffraction (Cordfunke & IJdo, 1988). The weberite structure consists of rows of corner-linked $Sb1—O_6$ octahedra in the [100] direction. The octahedra are

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* A table of values of y_i(obs.) and y_i(calc.) and complete lists of bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44657 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

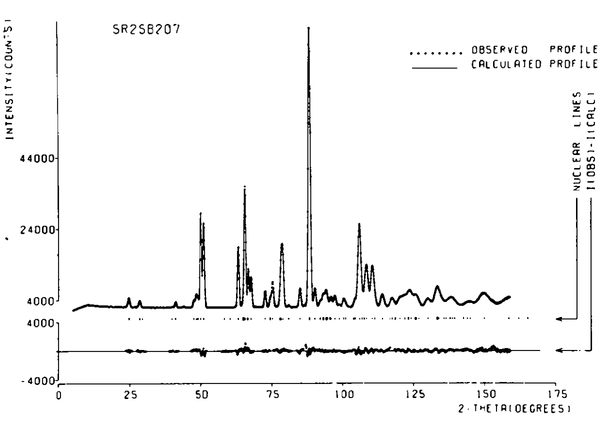

Fig. 1. Observed (dots) and calculated (full line) neutron diffraction profile of $Sr_2Sb_2O_7$ at 300 K; a difference (observed – calculated) curve appears at the bottom of the figure. Tick marks below the profile indicate the positions of the Bragg reflections included in the calculation.

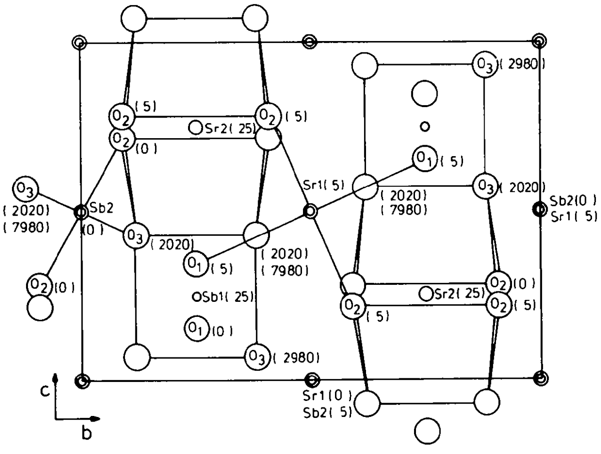

Fig. 2. The projection of the structure of $Sr_2Sb_2O_7$ along the [100] axis.

tilted 21·6 (1)° around an axis parallel to [010]. Between these rows lie rows of Sr2—O_8 square prisms linked with two vertices to each other and with two vertices to two Sb1—O_6 octahedra, forming slabs perpendicular to [010]. This part of the structure is basically the same as parts of the structure of La_3NbO_7 (Rossell, 1979) and pyrochlore. The difference is in the arrangement of the ions between these slabs as was described by Rossell (1979). The Sb2—O_6 octahedra lie between the above-mentioned slabs and share four corners with Sb1—O_6 octahedra and two corners with Sr2—O_8 square prisms. The Sr1—O_8 hexagonal prisms share four vertices with Sb1—O_6 octahedra and two vertices parallel to (100) with the Sb2—O_6 octahedra (Fig. 2). The difference from the pyrochlore structure can be described as follows. In the pyrochlore structure all large cations have a cubic 8 coordination. In the weberite structure these ions occur in one square and one hexagonal biprism. The way the octahedra are connected is also different. In the pyrochlore structure all the octahedra are connected to six identical octahedra. In the weberite structure one type (Sb1) is connected to six octahedra, the other (Sb2) is connected to four octahedra.

The weberite structure does not exist for M^{5+} = Nb and Ta but it does exist for the larger U^{5+} ion (Cordfunke & IJdo, 1988).

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Synthesis and Structure of Pentaamminephosphatocobalt(III)–Silver Nitrate–Water (1/3/1) (I) and Pentaamminephosphatocobalt(III)–Water (1/2) (II)

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Abstract. (I) $[CoPO_4(NH_3)_5].3AgNO_3.H_2O$, M_r = 766·70, monoclinic, $P2_1/c$, a = 6·800 (1), b = 9·344 (1), c = 27·087 (3) Å, β = 93·66 (1)°, V = 1717·4 (3) Å^3, Z = 4, D_m = 2·87, D_x = 2·965 g cm^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 44·4 cm^{-1}, $F(000)$ = 1472, T = 296 (2) K. (II) $[CoPO_4(NH_3)_5].2H_2O$, M_r = 275·08, monoclinic, $P2_1/c$, a = 6·401 (1), b = 11·048 (1), c = 13·839 (2) Å, β = 104·55 (1)°, V = 947·2 (2) Å^3, Z = 4, D_m = 1·91, D_x = 1·930 g cm^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 20·7 cm^{-1}, $F(000)$ = 576, T = 296 (2) K. Final R values 0·0282 (I) and 0·0209 (II) for 2075 and 1425 observed reflections with $F_o \geq 6{\cdot}0\sigma(F_o)$. Co is hexacoordinate in both structures with an octahedral geometry. Two of the three independent Ag atoms have 2+2 coordination and form centrosymmetric dimers with an $(AgO_2P)_2$ core, while the third Ag atom is pentacoordinate with a distorted tetragonal pyramidal geometry.

Introduction. Metal ions coordinated to the phosphate groups of nucleotides promote, in living cells, phosphate ester hydrolysis and maintain polynucleotide structure (Spiro, 1980). The complex between Mg^{2+} and ATP is a necessary cofactor for phosphoryl and nucleotidyl transfer enzymes. There are several examples of structurally known metal-ion nucleotide complexes, such as Co^{2+}, Zn^{2+}, Hg^{2+}, Cu^{2+}, Cd^{2+}, Pt^{2+} strongly bonded to the phosphate group (Spiro, 1980),

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