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Peretaite, $CaSb_4O_4(OH)_2(SO_4)_2 \cdot 2H_2O$: its atomic arrangement and twinning

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Abstract

Peretaite, $CaSb_4O_4(OH)_2(SO_4)_2 \cdot 2H_2O$, crystallizes in the monoclinic space group $C2/c$, with $a = 24.665$, $b = 5.6006$, $c = 10.185$Å, $\beta = 95.98°$. The structure was determined by Patterson and direct methods and refined by least-squares technique to a final R index of 0.037. The dominant structural feature is the arrangement of the two independent Sb polyhedra into sheets parallel to (100). The Ca ion is coordinated by six O and two H_2O in a square antiprism. These polyhedra are edge- and corner-linked to SO_4 groups into chains which run parallel to the Sb–O sheets. The hydroxyl groups form hydrogen bonds between Sb–O sheets and Ca–S chains; these bonds provide for the three-dimensional cohesion of the structure. An interpretation of twinning with (100) as twin plane is given.

Introduction

Peretaite is a new sulfate of antimony and calcium found at Pereta, Tuscany, Italy. Its chemical formula, $CaSb_4O_4(OH)_2(SO_4)_2 \cdot 2H_2O$, and other mineralogical data have been reported in the preceding article (Cipriani *et al.*, 1980). It occurs as aggregates of tabular crystals, associated with quartz, pyrite, calcite, sulfur, gypsum, and other antimony minerals, stibnite, valentinite, kermesite, and klebelsbergite. Klebelsbergite and peretaite are the only two Sb sulfate minerals so far known.

This study was undertaken to solve the crystal structure of peretaite and to investigate its possible crystal-chemical relationships with the structures of klebelsbergite (Menchetti and Sabelli, 1980) and of other synthetic Sb sulfates such as the anhydrous $Sb_2O_3 \cdot 2SO_3$ (Mercier *et al.*, 1975), $Sb_2O_3 \cdot 3SO_3$ (Mercier *et al.*, 1976), $Sb_6O_7(SO_4)_2$ (Bovin, 1976), and the hydrate $Sb_2(OH)_2(SO_4)_2 \cdot 2H_2O$ (Douglade *et al.*, 1978).

Experimental

For the X-ray structure analysis a small tabular crystal (approx. 0.1 × 0.4 × 0.2 mm) was taken from an original sample of peretaite. Monoclinic symmetry and possible space groups $C2/c$ and Cc were determined by Weissenberg photographs. The centrosymmetric space group $C2/c$ was chosen for the structure determination and was subsequently found to be correct. The unit-cell dimensions, determined from 25 high-angle reflections measured on a four-circle automatic diffractometer, are $a = 24.665(4)$, $b = 5.6006(9)$, $c = 10.185(1)$Å, $\beta = 95.98(1)°$.

The sample used for intensity data collection and all other crystals tested for X-ray work were found to be twinned. Figure 1 reports the reflection population due to the whole twinned crystal and referred to reciprocal lattice levels with $k = 2n$ (levels with $k = 2n+1$ can be represented by a similar scheme with h-odd lattice points occupied instead of h-even ones). One can readily see that overlapping of points from the two individuals takes place only in the lattice rows with $l = 4n$. From the intensities of 363 pairs of well-separated reflections (with $l = 4n + 2$) the relative volumes of the two members of the twin were easily computed. The knowledge of the volume ratio so determined (A/B = 3.55±0.02) allowed the composite intensities of the overlapped reflections to be subdivided into the intensities of A and B individuals, by solving the following equations:

$$I_{hkl} = I^{A}_{hkl} + I^{B}_{h'k'l'}$$

$$I_{h'k'l'} = I^{A}_{h'k'l'} + I^{B}_{hkl}$$

where I is the observed intensity of the twinned crystal and I^A and I^B are the intensities of the two superimposed reflections; h,k,l are the Miller indices of reflections belonging to the A individual and $h' = -(h + \frac{l}{2})$, $k' = k$, $l' = l$ those of overlapped reflections of the B individual of the twin. In conclusion, according to Friedel's (1926) notation, the crystals of peretaite

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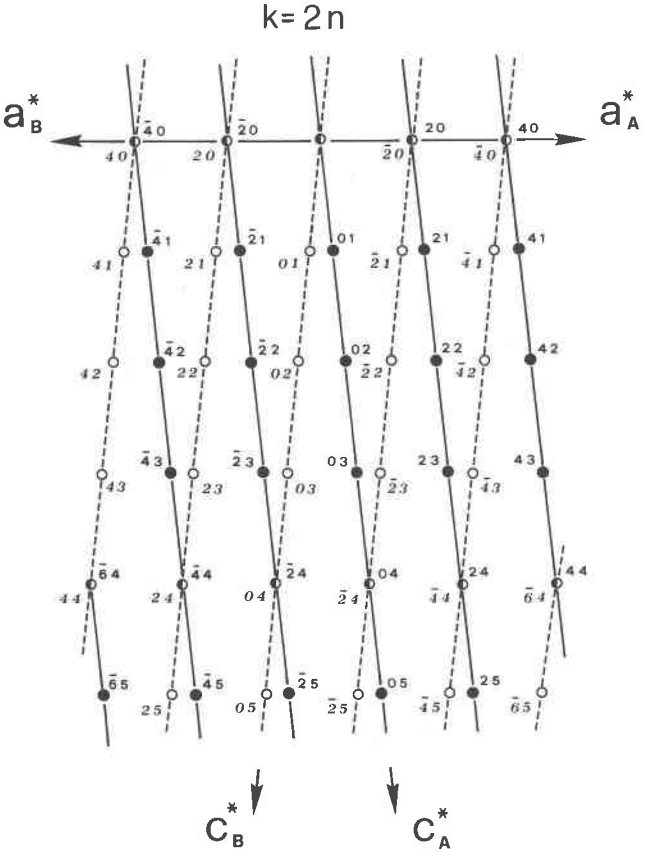

Fig. 1. A layer of the reciprocal lattice of the twinned peretaite. Numbering refers to h and l Miller indices, with $k = 2n$. The black-dotted lattice belongs to a member of the twin (A individual), the open-circled lattice to the other (B individual).

are twinned by pseudo-merohedry, with twin plane (100), twin index 4, and twin obliquity 0.05°.

Intensities were collected on a Philips PW-1100 four-circle computer-controlled diffractometer (Centro di Cristallografia Strutturale del CNR, Pavia, Italy), with graphite-monochromated Mo$K\alpha$ radiation, ω–2θ scan technique, integration width of 1.5° and scan speed of 0.1°/sec.

A total of 2044 symmetry-independent reflections were measured within the limit of $2\theta \leqslant 30°$; only 1808 reflections with $F_o > 5\sigma(F_o)$, with σ derived from counting statistics, were regarded as observed. The intensities were corrected for Lorentz and polarization factors and for absorption, the latter being carried out on the basis of the semiempirical method proposed by North *et al.* (1968).

Structure determination and refinement

The four heavy-atom positions were determined by direct methods using the computer program MULTAN (Main *et al.*, 1974). The 250 largest normalized structure amplitudes ($E > 1.52$) were selected as input for the phase determination. Because of their particular coordinates, the heavy atoms (mainly the antimony atoms) contribute, in the structure-factor calculation, almost entirely to the reflections with $l = 2n$. For this reason, in order to derive indications about the oxygen atom locations, a three-dimensional Patterson synthesis with l-odd reflections only was computed. The introduction of the oxygen positions, recognized in this map, in the structure-factor calculation allowed the l-odd reflections also to take a sign. In the next Fourier map all missing oxygen atoms were located and two cycles of isotropic full-matrix least-squares refinement led to $R = 0.07$. The observed structure factors were weighted by $1/\sigma^2(F_o)$. A difference Fourier synthesis calculated after a cycle of anisotropic refinement ($R = 0.037$) yielded the positions of hydrogen atoms. Subsequent least-squares refinement was carried out using isotropic temperature factors fixed at 4Å^2 for hydrogen atoms. Because of inconsistencies in their shifts, the hydrogen atom positions could not be refined. The last cycle achieved convergence at $R = 0.035$ for the observed reflections and $R = 0.041$ including "less thans." The scattering factors for all atoms and the anomalous dispersion coefficients for heavy atoms were taken from *International Tables for X-ray Crystallography* (1974, p. 99–101, 148–151).

The atomic positional and thermal parameters are listed in Table 1, and a list of observed and calculated structure factors appears in Table 2.[1]

Description and discussion of the structure

There has been remarkable interest in recent years in the coordination of the Sb atom in Sb^{3+}–O compounds. For a general treatment of this matter we refer to the works by Andersson *et al.* (1973) and Galy *et al.* (1975), and also to the discussions of the structures of $Sb_6O_7(SO_4)_2$ (Bovin, 1976) and of klebelsbergite $Sb_4O_4(OH)_2SO_4$ (Menchetti and Sabelli, 1980).

Taking into account the lone pairs of electrons (E) in peretaite, the volume per oxygen and E is 17.4Å^3. This figure compares well with the values found in many Sb^{3+}–O compounds, and in this way peretaite also can be described as a structure having a close-packing of anions and lone pairs.

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[1] To receive a copy of Table 2, order Document AM-80-139 from the Business Office, Mineralogical Society of America, 2000 Florida Avenue, N.W., Washington, D.C. 20009. Please remit $1.00 in advance for the microfiche.

Table 1. Peretaite: fractional atomic coordinates and thermal parameters

Atom	x	y	z	B eq.$^+$	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Sb(1)	0.29249(2)	0.50504(7)	0.51525(4)	0.79	2(0)	72(1)	23(1)	1(0)	2(0)	16(1)
Sb(2)	0.31537(2)	0.05196(6)	0.26706(4)	0.65	3(0)	48(1)	18(1)	2(0)	2(0)	-1(1)
Ca	0.50	0.1856(3)	0.25	0.92	3(0)	83(5)	22(2)	-	1(1)	-
S	0.4201(1)	-0.0163(2)	0.5184(1)	0.94	3(0)	83(4)	23(1)	-1(1)	1(1)	4(2)
O(1)	0.3600(2)	0.0049(9)	0.5276(5)	2.00	3(1)	253(18)	49(5)	3(3)	3(2)	1(8)
O(2)	0.4296(2)	0.0463(8)	0.3829(4)	1.55	6(1)	142(15)	36(4)	1(3)	4(2)	28(6)
O(3)	0.4511(2)	0.1391(8)	0.6159(4)	1.70	8(1)	121(14)	40(5)	3(3)	-3(2)	-23(6)
O(4)	0.4378(2)	-0.2635(7)	0.5487(4)	1.52	10(1)	65(12)	33(4)	1(3)	-1(2)	7(6)
O(5)	0.4446(2)	0.5206(7)	0.3043(4)	1.30	5(1)	90(13)	39(4)	-4(2)	3(1)	-5(6)
O(6)	0.2609(2)	0.1778(7)	0.3871(4)	0.59	1(1)	68(11)	18(4)	-2(2)	4(1)	-1(5)
O(7)	0.2625(2)	0.1787(6)	0.1146(4)	0.56	2(1)	52(11)	11(3)	-3(2)	-1(1)	3(5)
O(8)	0.3394(2)	0.3860(7)	0.2667(4)	1.05	3(1)	66(11)	41(4)	-3(2)	2(1)	1(6)
H(1)	0.450	0.600	0.210	4.00						
H(2)	0.450	0.660	0.380	4.00						
H(3)	0.382	0.420	0.285	4.00						

$^+$Equivalent isotropic B (Å^2) calculated from anisotropic temperature factors, except for H(1), H(2) and H(3) where B was not refined.

Form of anisotropic temperature factors (x10^4): $\exp -(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)$

Selected interatomic distances and angles are reported in Table 3. In peretaite there are two crystallographically distinct Sb polyhedra. The Sb(1) polyhedron (of SbO_4E type) shows a trigonal bipyramidal arrangement with one of the equatorial corners occupied by the lone pair *E*. The two axial Sb–O distances are somewhat different (2.206 and 2.336Å), but their mean value of 2.271Å, as well as the values of 98.0° and 141.0° (equatorial and axial angles) correspond well to 2.27Å, 92.2°, and 151.5°, the respective values of the theoretically derived model (Galy *et al.*, 1975).

On the contrary, the Sb(2) polyhedron is of the SbO_3E type and can be described as a tetrahedron with the lone pair at one corner and O(6), O(7), and O(8) on the others. As one can see in Table 1, both Sb–O and O–O distances show a narrow range of variation, as well as O–Sb–O and O–O–O angles; this constancy agrees with a tetrahedral approximation of the polyhedron.

Each Sb(1) polyhedron shares edges with two symmetry-equivalent Sb(1) polyhedra (through the symmetry centers at 1/4, 1/4, 1/2 and 1/4, 3/4, 1/2), to form edge-by-edge chains running in the *b* direction. The oxygens of Sb(2) polyhedron are: O(6) belonging to a chain, O(7) of the adjacent chain, and the hydroxyl oxygen O(8). In this way Sb(1) chains result cross-linked by the Sb(2) polyhedra, to make dense Sb–O sheets. These sheets, two per unit cell and parallel to (100), account for the platy habit of the crystals. A view of the structure looking along the sheets is shown in Figure 2.

The calcium atom, on the two-fold axis, binds three pairs of sulfate oxygens and one pair of water oxygen, to form a distorted square antiprism (Fig. 2). Ca–O distances range from 2.419 to 2.508Å. Each Ca polyhedron is connected to four surrounding SO_4 groups by edge and corner sharing. This linkage produces Ca–S zig-zag chains running in the *c* direction (Fig. 3). Connections between Ca–S are provided by H-bonds, with O(5) water oxygen acting as donor and O(3) and O(4) as acceptors.

The formula unit of peretaite can be regarded as the summation of a formula unit of klebelsbergite

Table 3. Peretaite: selected interatomic distances (Å) and angles (°)

Sb(1)	
Sb(1)-O(6,3)	2.011(4)
Sb(1)-O(7,5)	2.040(4)
Sb(1)-O(7,6)	2.206(4)
Sb(1)-O(6)	2.336(4)
average	2.148
Sb(1)-O(8,6)	2.766(4)◆
Sb(1)-O(8)	2.967(5)◆
O(6)-O(6,3)	2.547(6)
O(6)-O(7,5)	2.863(5)
O(6)-O(7,6)	4.283(6)
O(6,3)-O(7,5)	3.056(6)
O(6,3)-O(7,6)	2.854(5)
O(7,5)-O(7,6)	2.484(5)
O(6)-Sb(1)-O(6,3)	71.3(1)
O(6)-Sb(1)-O(7,5)	81.4(1)
O(6)-Sb(1)-O(7,6)	141.0(1)
O(6,3)-Sb(1)-O(7,5)	98.0(2)
O(6,3)-Sb(1)-O(7,6)	85.0(2)
O(7,5)-Sb(1)-O(7,6)	71.5(1)

Sb(2)	
Sb(2)-O(8)	1.963(4)
Sb(2)-O(6)	2.035(4)
Sb(2)-O(7)	2.048(4)
average	2.015
Sb(2)-O(1)	2.778(5)◆
Sb(2)-O(1,8)	2.796(5)◆
Sb(2)-O(2)	2.938(5)◆
O(8)-O(6)	2.666(6)
O(8)-O(7)	2.594(6)
O(6)-O(7)	2.779(5)
O(8)-Sb(2)-O(6)	83.6(2)
O(8)-Sb(2)-O(7)	80.6(2)
O(6)-Sb(2)-O(7)	85.8(2)
O(7)-O(6)-O(8)	56.9(1)
O(6)-O(7)-O(8)	59.3(1)
O(6)-O(8)-O(7)	63.8(2)

	Ca	
2	Ca-O(5)	2.419(5)
2	Ca-O(2)	2.440(5)
2	Ca-O(4)	2.468(4)
2	Ca-O(3,2)	2.508(5)
	average	2.459
	O(5)-Ca-O(2)	70.7(2)
	O(5)-Ca-O(4,2)	89.4(2)
	O(5)-Ca-O(3,2)	133.6(1)
	O(5)-Ca-O(2,4)	145.8(2)
	O(5)-Ca-O(4,8)	74.7(1)
	O(5)-Ca-O(3,8)	115.8(2)
	O(5)-Ca-O(5,4)	78.3(2)
	O(2)-Ca-O(4,2)	90.7(2)
	O(2)-Ca-O(3,2)	78.2(2)
	O(2)-Ca-O(2,4)	142.7(2)
	O(2)-Ca-O(4,8)	95.7(2)
	O(2)-Ca-O(3,8)	74.9(2)
	O(4,4)-Ca-O(3,2)	56.9(1)
	O(4,4)-Ca-O(4,8)	159.6(2)
	O(4,4)-Ca-O(3,8)	143.4(2)
	O(3,4)-Ca-O(3,8)	87.0(2)

S	
S-O(2)	1.466(5)
S-O(3)	1.473(5)
S-O(4)	1.475(4)
S-O(1)	1.500(5)
average	1.479
O(2)-O(3)	2.433(6)
O(2)-O(4)	2.415(6)
O(2)-O(1)	2.387(7)
O(3)-O(4)	2.396(6)
O(3)-O(1)	2.449(7)
O(4)-O(1)	2.429(7)
O(2)-S-O(3)	111.8(3)
O(2)-S-O(4)	110.4(3)
O(2)-S-O(1)	107.2(3)
O(3)-S-O(4)	107.0(3)
O(3)-S-O(1)	111.0(3)
O(4)-S-O(1)	109.5(3)

H(1)-O(5)-H(2) water	
O(5)-O(3,7)	2.721(6)
O(5)-H(1)	1.08
H(1)...O(3,7)	1.75
O(5)-O(4,1)	2.789(6)
O(5)-H(2)	1.10
H(2)...O(4,1)	1.83
O(3,7)-O(5)-O(4,1)	109.8(2)
H(1)-O(5)-H(2)	108
O(5)-H(1)...O(3,7)	147
O(5)-H(2)...O(4,1)	144

O(8)-H(3) hydroxyl	
O(8)-O(5)	2.691(6)
O(8)-H(3)	1.07
H(3)...O(5)	1.64
O(8)-H(3)...O(5)	170

Cation-cation distances (<3.8 Å)	
Sb(1)-Sb(1,9)	3.447(2)
Sb(1)-Sb(1,3)	3.538(2)
Sb(1)-Sb(2,6)	3.571(2)
Sb(1)-Sb(2,3)	3.650(2)
Sb(1)-Sb(2)	3.997(2)
Sb(1)-Sb(2,5)	3.718(2)
Sb(2)-S	3.463(2)
Ca-S(,2)	3.063(2)
Ca-S	3.709(2)

The equivalent positions (referred to Table 1) are designated by the second number in parentheses and are (1) = x,1+y,z; (2) = 1-x,-y,1-z; (3) = 1/2-x,1/2-y,1-z; (4) = 1-x,y,1/2-z; (5) = 1/2-x,1/2+y,1/2-z; (6) = x,1-y,1/2+z; (7) = x,1-y,-1/2+z; (8) = x,-y,-1/2+z; (9) = 1/2-x,3/2-y,1-z.

◆Largest Sb-O distances (<3 Å).

with a formula unit of gypsum. Each calcium atom in gypsum, as in peretaite, is surrounded by one pair of water molecules and by three pairs of oxygen atoms belonging to SO_4 groups. Moreover the *a* axis of gypsum (5.670Å) is comparable with the *b* axis of peretaite (5.6006Å). However, there are no other similarities between the structures of these two minerals. In gypsum (Cole and Lancucki, 1974) the Ca polyhedra are directly and strictly linked to each other to form chains, connected in their turn by SO_4 groups to form sheets. In peretaite on the contrary the Ca polyhedra are isolated and form chains by the connection with SO_4 tetrahedra.

The sulfate group shows the usual tetrahedral configuration, slightly distorted. The longest S–O distance involves O(1), which is further "bonded" twice

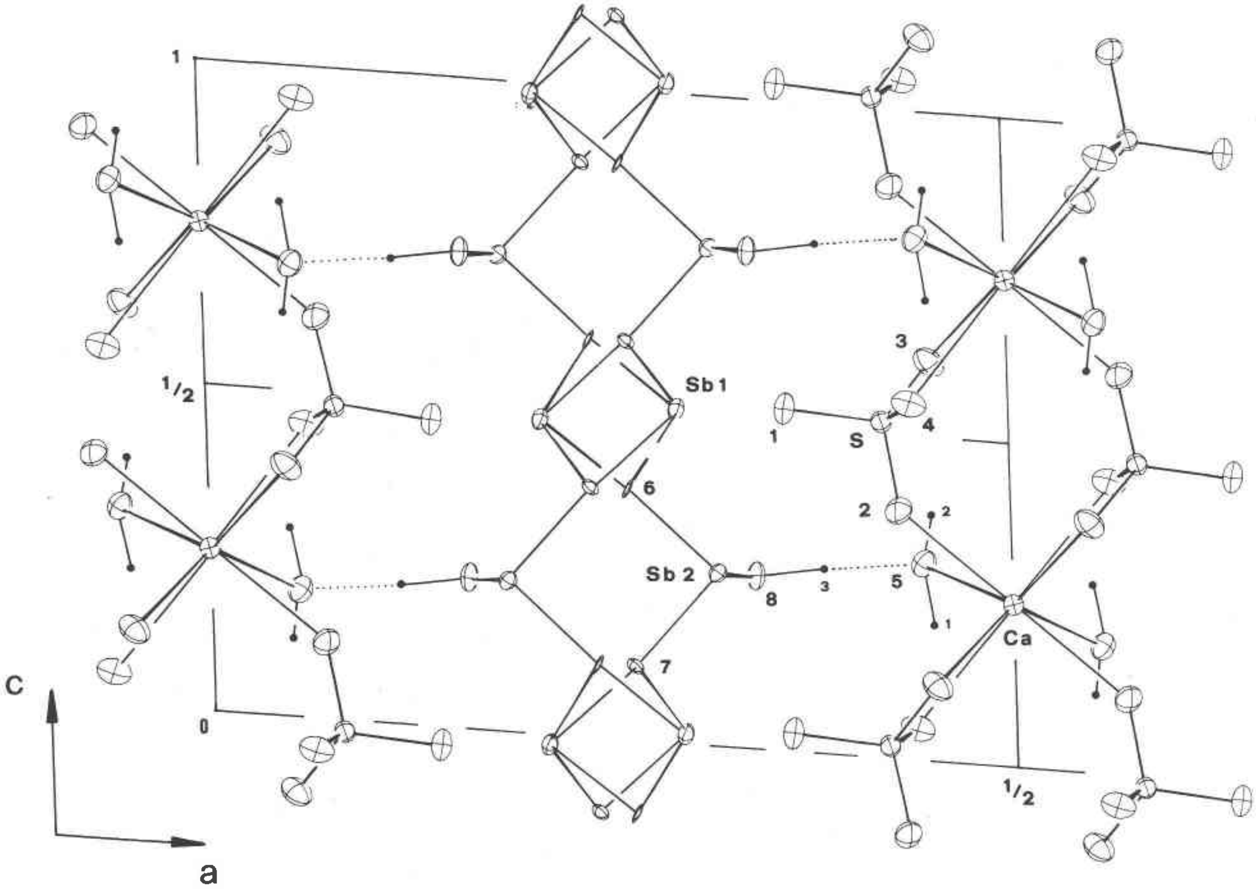

Fig. 2. The structure of peretaite projected down the *b* axis. Thermal ellipsoids are scaled at the 50% probability.

to the Sb(2) atom. Since these Sb(2)–O(1) distances (2.778 and 2.796Å) are to be considered very weak bonds, a shorter S–O(1) distance would be expected. The charge deficiency of O(1) is clearly shown by the electrostatic valence balance (Table 4), computed according to Brown and Kun Wu (1976). A similar imbalance was also observed for the sulfate oxygens of klebelsbergite.

As already said, the hydrogen atoms of the water molecule make two H-bonds among the Ca polyhedra. The water oxygen O(5) in turn is the recipient of a third H-bond from the donor O(8). The hydroxyl oxygen belongs to the Sb–O sheet, and its role is particularly relevant for the cohesion of the structure. In fact this H-bond is the only "strong" bond between Sb–O sheets and Ca–S chains. The antimony–oxygen interaction due to Sb(2)–O very weak bonds (2.778, 2.796, and 2.938Å) and some van der Waals interactions further contribute to hold the structure together.

This kind of three-dimensional connection, based on two-dimensional Sb–O elements, is different from that found in the structures of the known Sb sulfates. The structure of $Sb_2O_3 \cdot 2SO_3$ (Mercier *et al.*, 1975) consists of molecular units linked to each other by van der Waals interactions and can then be considered as a molecular structure. The atomic arrangement of $Sb_2(OH)_2(SO_4)_2 \cdot 2H_2O$ (Douglade *et al.*, 1978) is also organized in "molecular units," but the linkage among them is assured by H-bonds. The structures of $Sb_2O_3 \cdot 3SO_3$ (Mercier *et al.*, 1976) and $Sb_6O_7(SO_4)_2$ (Bovin, 1976), on the contrary, are characterized by one-dimensional elements: a sort of double chain built by both SbO_3 and SO_4 polyhedra in the first instance and a "cylindrical unit" of SbO_3 polyhedra in the second. These one-dimensional elements are held together by Sb–O weak interactions and by van der Waals forces. As in peretaite, the arrangement of Sb–O polyedra in klebelsbergite takes place according to two-dimensional elements; the sheets of klebelsbergite however are built in a different way and their connection is made directly by

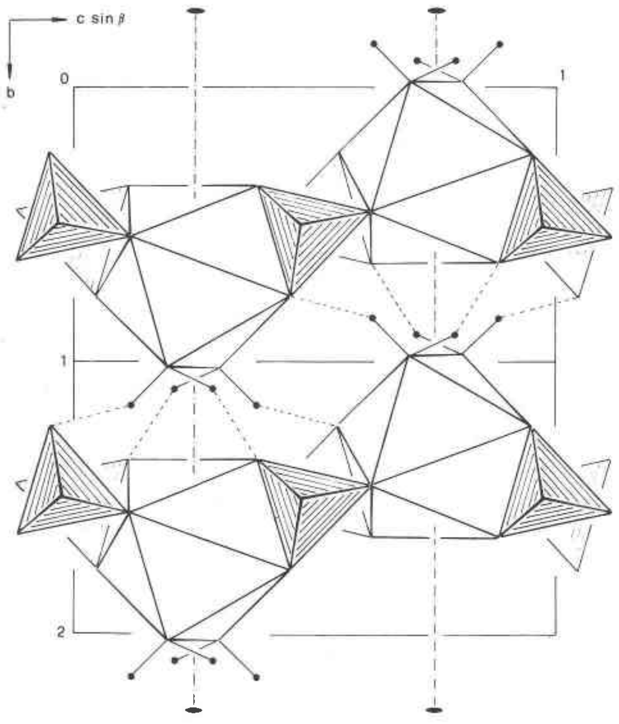

Fig. 3. A projection of peretaite down the *a* axis. Zig-zag Ca–S chains running in the *c* direction are shown.

means of SO_4 groups. In addition to the above sulfates, many other Sb synthetic compounds are structurally known. Some of these, such as the chloride oxide $Sb_4O_5Cl_2$ (Särnstrand, 1978), show Sb^{3+}–O polyhedra arranged in two-dimensional nets, as in the peretaite and klebelsbergite structures.

Table 4. Peretaite: electrostatic valence balance

Atom	Sb(1)	Sb(2)	Ca	S	H(1)	H(2)	H(3)	Sum
O(1)		0.20 0.19		1.40				1.79
O(2)		0.15	0.26	1.56				1.97
O(3)			0.22	1.52	0.22			1.96
O(4)			0.25	1.52		0.20		1.97
O(5)			0.27		0.78	0.80	0.23	2.08
O(6)	0.85 0.43	0.78						2.06
O(7)	0.80 0.56	0.76						2.12
O(8)	0.21 0.15	0.92					0.77	2.05
Sum	3.00	3.00	1.00 (x2)	6.00	1.00	1.00	1.00	

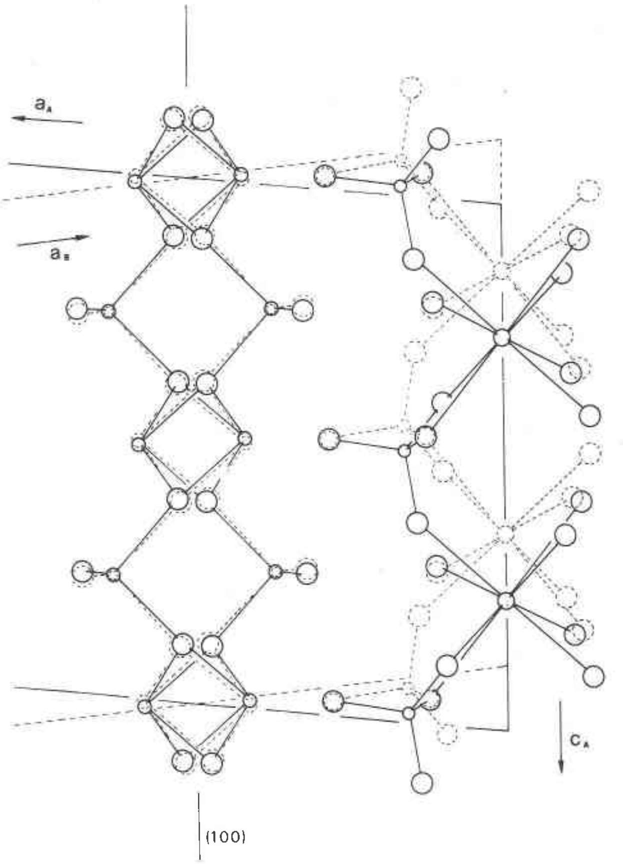

Fig. 4. A view of twinned peretaite: the A and B individuals of the twin are drawn with solid and dashed lines respectively. The unit cells are also outlined.

As concerns the change of the atomic arrangement responsible for the twinning, it seems clear that the (100) twin plane pre-exists as a "pseudo" symmetry operator in the Sb–O sheet, owing to the particular coordinates of atoms involved. These "rigid" structural slabs of an individual of the twin are practically interchangeable with those of its mate. As Figure 4 illustrates, the real change in the atomic arrangement takes place in the "soft" part of the structure, starting from the SO_4 tetrahedron and affecting mainly the Ca polyhedra.

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