The magnetic behavior is consistent with the crystal structure of α-$Cu(IO_3)_2$ (Liminga, Abrahams & Bernstein, 1975*b*), which contains isolated CuO_6 groups, the closest Cu–Cu distance (translation along the *b* axis) being 5·111 Å.

The authors (RL and SRB) wish to thank Professor I. Olovsson for the experimental facilities. They are indebted to Drs K. Nassau and S. C. Abrahams, Bell Laboratories, Murray Hill, for the donation of the crystals, and to Dr S. C. Abrahams for valuable comments on the manuscript. This work has been supported by grants from the Swedish Natural Science Research Council and the National Science Foundation (USA).

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The Crystal Structure of Antimony(III) Chloride Oxide $Sb_4O_5Cl_2$

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(*Received* 28 *January* 1978; *accepted* 21 *March* 1978)

The structure of $Sb_4O_5Cl_2$ has been reinvestigated and refined to an R of 0·031 for 1206 independent reflections. The space group is $P2_1/c$ with $a = 6{\cdot}2380\ (4)$, $b = 5{\cdot}1112\ (3)$, $c = 13{\cdot}538\ (2)$ Å, $\beta = 97{\cdot}217\ (8)°$, $Z = 2$. The main architecture of the crystal structure is the same as found by Edstrand [*Acta Chem. Scand.* (1947). **1**, 178–203] but differences were observed, especially in the Sb–O distances. There are two crystallographically independent Sb atoms, which can both be considered three-coordinated. The coordination polyhedron can be described as a distorted tetrahedron with the lone pair of electrons (E) of Sb at the fourth corner. The SbO_3E polyhedra share corners forming infinite layers parallel to the *bc* plane. The Cl atoms are situated between these layers. A short survey is given of the linking of Sb–O coordination polyhedra in some Sb^{III} compounds.

Introduction

This investigation is part of a general study of the O coordination of Sb^{III}. In order to obtain accurate Sb^{III}–O distances for the discussion of the coordination geometry, it was considered necessary to refine the structure of $Sb_4O_5Cl_2$ (Edstrand, 1947).

Experimental

$Sb_4O_5Cl_2$ was prepared according to Edstrand (1947) and colourless, transparent single crystals suitable for X-ray work were obtained.

Cell dimensions were determined by a least-squares analysis of the θ values of 59 reflections (θ: 19–26°)

Table 1. *Crystal data*

Cell parameters within brackets are those given by Edstrand (1947).

Space group	$P2_1/c$ (from systematic absences)
Unit cell: *a*	6·2380 (4) Å [6·242 Å]
b	5·1112 (3) [5·118]
c	13·538 (2) [13·53]
β	97·217 (8)° [97·27°]
V	428·22 Å^3
Z	2
D_c	4·947 g cm^{-3}
μ(Mo $K\alpha$)	122·1 cm^{-1}

Table 2. *Crystal dimensions*

Boundary planes and their distances from an internal origin

Plane	Distance (cm)
(100)	0·0045
$(\bar{1}00)$	0·0045
(001)	0·0100
$(0\bar{1}0)$	0·0100
$(01\bar{9})$	0·0105
(221)	0·0075

Table 3. *Fractional atomic coordinates for* $Sb_4O_5Cl_2$ *from Edstrand* (1947)

	x	*y*	*z*
Sb(1)	0·186 (5)	0·225 (5)	0·049 (3)
Sb(2)	0·796 (5)	0·113 (5)	0·203 (3)
Cl	0·51 (1)	0·703 (10)	0·115 (5)
O(1)	0	½	0
O(2)	0·13	0·35	0·185
O(3)	0·07	0·05	0·915

measured on an Enraf–Nonius CAD-4 computer-controlled four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation, λ(Mo $K\alpha_1$) = 0·70926 Å. Crystal data are presented in Table 1 and compared with the values given by Edstrand (1947).

The dimensions of the crystal chosen for intensity data collection are given in Table 2. The intensities were recorded on the diffractometer with the ω–2θ scan technique and $\Delta\omega$ = 0·8° + 0·5° tan θ. The background was measured by the moving-counter moving-crystal technique by extending the scan interval 25% on each side of the peak. A net count of 2000 for each reflection was pursued within 5 min. The scan speed was calculated from the net intensity in a fast prescan. In this way 1243 intensities in one quadrant of reciprocal space with θ = 3–30° were sampled. After every 50 reflections three control reflections were checked. Their intensities remained constant. Of the 1243 reflections 30 were considered unobserved giving net counts <10 in the prescan. Another 7 intensities were considered unobserved with $I < 3\sigma(I)$. The remaining 1206 reflections were corrected for Lorentz, polarization and absorption effects. The transmission factors were in the range 0·121–0·377.

Table 4. *Final positional parameters with their e.s.d.'s*

	x	*y*	*z*
Sb(1)	0·18703 (5)	0·22341 (6)	0·04893 (2)
Sb(2)	0·79660 (5)	0·11695 (6)	0·20157 (2)
Cl	0·5108 (3)	0·7040 (3)	0·1159 (1)
O(1)	0	½	0
O(2)	0·0743 (6)	0·3380 (8)	0·2111 (3)
O(3)	0·0707 (6)	0·0052 (8)	0·9181 (3)

Table 5. *Bond lengths* (Å) *and angles* (°)

The prime denotes atoms symmetry-related to those given in Table 4. The distances within brackets are from Edstrand (1947).

Sb(1)–O(1)	1·898 (0) [1·89]	Sb(2)–O(2)	1·960 (4) [2·03]
Sb(1)–O(3)	2·081 (4) [2·22]	Sb(2)–O(3)	2·009 (4) [2·06]
Sb(1)–O(3′)	2·141 (4) [2·06]	Sb(2)–O(2′)	2·059 (4) [2·44]
Sb(1)–O(2)	2·459 (4) [2·02]	Sb(2)–Cl	2·909 (2) [2·91]
O(1)–Sb(1)–O(3)	92·0 (1)	O(2)–Sb(2)–O(3)	94·6 (2)
O(1)–Sb(1)–O(3′)	88·3 (1)	O(2)–Sb(2)–O(2′)	94·7 (1)
O(1)–Sb(1)–O(2)	84·4 (1)	O(2)–Sb(2)–Cl	84·1 (1)
O(3)–Sb(1)–O(3′)	72·2 (2)	O(3)–Sb(2)–O(2′)	78·0 (2)
O(3)–Sb(1)–O(2)	68·0 (1)	O(3)–Sb(2)–Cl	75·3 (1)
O(3′)–Sb(1)–O(2)	139·2 (1)	O(2′)–Sb(2)–Cl	153·1 (1)

Sb(1)–O(1)–Sb(1)	180
Sb(1)–O(2)–Sb(2)	98·4 (2)
Sb(1)–O(2)–Sb(2)	124·8 (2)
Sb(2)–O(2)–Sb(2)	135·7 (2)
Sb(1)–O(3)–Sb(1)	107·8 (2)
Sb(1)–O(3)–Sb(2)	134·2 (2)
Sb(1)–O(3)–Sb(2)	114·0 (2)

Structure refinement

The coordinates given by Edstrand (1947) (Table 3) were used as a starting point for the refinement. In the least-squares calculations anisotropic thermal motions for all the atoms were assumed. Scattering factors for neutral Sb, Cl and O were taken from Doyle & Turner (1968) and the anomalous dispersion corrections from Cromer & Liberman (1970). The secondary extinction coefficient *g* (Zachariasen, 1967) was included in the refinement. The function minimized was $\sum w_i(|F_o| - |F_c|)^2$, where $w_i^{-1} = \sigma^2(|F_o|) + (0·025|F_o|)^2 + 2·0$. For 1206 reflexions the final $R = 0·031$, $R_w = 0·040$ and $S = 0·96$ {where $R = \sum(||F_o| - |F_c||)/\sum|F_o|$, $R_w = [\sum w_i(|F_o| - |F_c|)^2/\sum w_i|F_o|^2]^{1/2}$ and $S = [\sum w_i(|F_o| - |F_c|)^2/(m - n)]^{1/2}$}. The extinction coefficient refined to 8·7 (3) × 10^3.

The resulting positional parameters are given in Table 4.* Selected interatomic distances and angles are listed in Table 5 with those given by Edstrand (1947).

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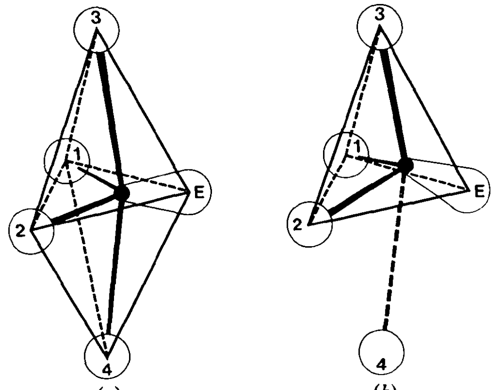
* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33526 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion

The two principal coordination polyhedra around Sb^{III} are the trigonal bipyramid where one of the equatorial corners is occupied by the lone pair of electrons (*E*) [Fig. 1(*a*)] and the tetrahedron with the lone pair at one corner [Fig. 1(*b*)]. It has been shown (Andersson, Åström, Galy & Meunier, 1973) that the stereochemically active lone pair of Sb^{III} corresponds to a volume of the same size as that of an O^{2-} ion. As shown in Fig. 1, only a slight displacement of Sb out of the equatorial plane of the trigonal bipyramid is needed to change four- to three-coordination (Galy, Meunier, Andersson & Åström, 1975). Bovin, Svensson & Särnstrand (1974) have shown that all known Sb—O polyhedra, with one exception (Poore & Russell, 1971), are identical to those in Fig. 1 or intermediate between them.



(*a*) (*b*)

Fig. 1. Coordination polyhedra of Sb^{III}. 1, 2, 3 and 4 denote O atoms and *E* denotes the lone pair of electrons of Sb^{III}. (*a*) The trigonal bipyramidal coordination polyhedron (SbO_4E). (*b*) The tetrahedral coordination polyhedron (SbO_3E) with one next-nearest neighbour (dotted line). (*a*) and (*b*) represent idealized four- and three-coordination respectively.

In $Sb_4O_5Cl_2$ the asymmetric part of the unit cell contains two Sb atoms. The distances Sb—Cl are $\geq$2·909 (2) Å. These bonds are predominantly ionic in character, since normal covalent bond lengths Sb—Cl are about 2·4 Å, *e.g.* in $SbCl_3$ (Lipka, 1977). Sb(1) can be regarded as either three- or four-coordinated by O (Fig. 2). Sb(2), however, has only three O atoms in the coordination polyhedron and the next nearest neighbour is a Cl^- ion (Fig. 3). The Sb—O distances are rather different from those given by Edstrand (1947). In Table 6 the Sb—O distances of Sb(1) and Sb(2) are compared with distances in some other Sb^{III}—O polyhedra. The compounds in the table are chosen in order to illustrate the coordination polyhedra in Fig. 1. The most representative trigonal bipyramids are placed at the top of the table and the most typical tetrahedra at the bottom, judged from the length of the fourth Sb—O distance (Sb-4). The Sb—O distances in a theoretical model of the trigonal bipyramid given by Galy *et al.* (1975) are also included.

The coordination polyhedra of Sb(1) and Sb(2) are linked forming infinite layers of the composition $(Sb_4O_5^{2+})_n$ parallel to the *bc* plane. A part of such a layer is illustrated in Fig. 4. Fig. 4(*a*) shows both Sb(1) and Sb(2) as three-coordinated. The Sb(1) polyhedra form pairs having a mutual edge [O(3) and O(3′)] with the quite short O—O distance of 2·487 (7) Å. Every pair is linked to two others by corner-sharing [O(1)] forming chains along [010]. The chains of Sb(1) polyhedra are connected in the *c* direction by the Sb(2) polyhedra by corner-sharing [O(3) or O(3′)]. The Sb(2) polyhedra are linked to each other by sharing the O(2) corner. Both Sb(1) and Sb(2) with their lone pairs are directed out from the layer. Fig. 4(*b*) shows the same part of the layer, but now Sb(1) is considered to be four-coordinated. The Cl^- ions are located between the $(Sb_4O_5^{2+})_n$ layers (Fig. 5).

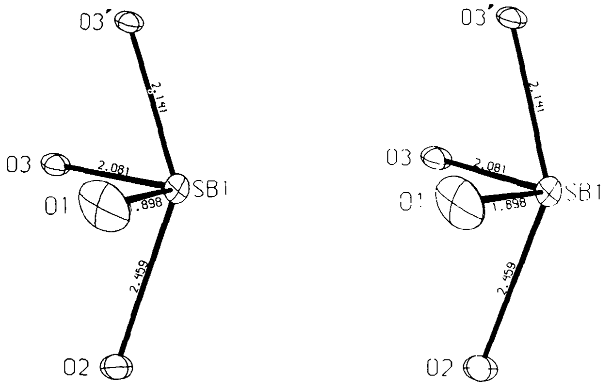


Fig. 2. Stereoview of the coordination of Sb(1) in $Sb_4O_5Cl_2$. Bond lengths (Å) are shown. The thermal vibration ellipsoids were scaled to include 50% probability.

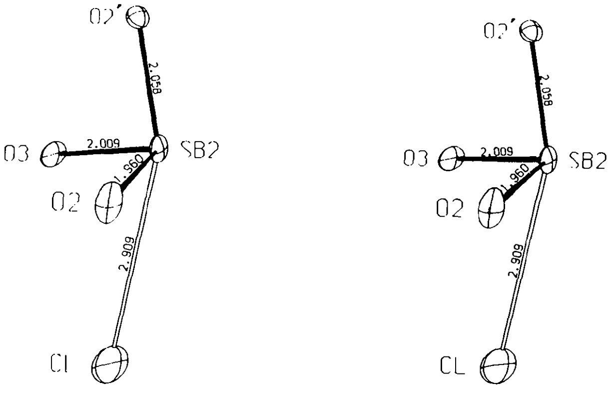


Fig. 3. Stereoview of the coordination of Sb(2) in $Sb_4O_5Cl_2$. Bond lengths (Å) are shown. The bond to the Cl^- ion is open. The thermal vibration ellipsoids were scaled to include 50% probability.

Table 6. *Bond distances* (Å) *in some* Sb^{III}—O *coordination polyhedra*

The numbering of the O atoms corresponds to Fig. 1.

	Sb-1	Sb-2	Sb-3	Sb-4	Coordination number	References
$SbPO_4$	1·983 (10)	2·035 (12)	2·181 (10)	2·181 (10)	4	Kinberger (1970)
$Na_2Sb_4O_7$	1·988 (5)	1·996 (5)	2·102 (5)	2·196 (5)	4	Maraine & Pérez (1977)
β-Sb_2O_4	2·032 (9)	2·032 (9)	2·218 (9)	2·218 (9)	4	Rogers & Skapski (1964)
$Sb(C_6H_3O_3).H_2O$	2·022 (9)	2·022 (9)	2·228 (4)	2·228 (4)	4	Aurivillius & Särnstrand (1976)
Theoretical trigonal bipyramid, SbO_4E	2·02	2·02	2·27	2·27	4	Galy *et al.* (1975)
$SbO(H_2PO_4).H_2O$	1·970 (8)	1·977 (8)	2·145 (9)	2·291 (10)	4	Särnstrand (1974)
$CaFeSbAs_2O_7$	1·962 (7)	1·974 (6)	2·121 (7)	2·335 (7)	4	Coda, Dal Negro, Sabelli & Tazzoli (1977)
$Na_2Sb_4O_7$	1·946 (5)	1·979 (5)	2·146 (5)	2·357 (5)	3–4	Maraine & Pérez (1977)
$Sb_2(SO_4)_3$	2·028 (5)	2·089 (5)	2·227 (5)	2·392 (4)	3–4	Mercier, Douglade & Bernard (1976)
$Sb_2(SO_4)_3$	2·030 (5)	2·126 (5)	2·210 (2)	2·395 (2)	3–4	Mercier, Douglade & Bernard (1976)
$Sb_4O_5Cl_2$, [Sb(1)]	1·898 (0)	2·081 (4)	2·141 (4)	2·459 (4)	3–4	This work
$Sb_4O_5Cl_2$, [Sb(2)]	1·960 (4)	2·009 (4)	2·059 (4)	2·909 (2)*	3	This work
Sb_2O_3 (cubic)	1·977 (1)	1·977 (1)	1·977 (1)	2·918 (2)	3	Svensson (1975)

*4 = Cl.

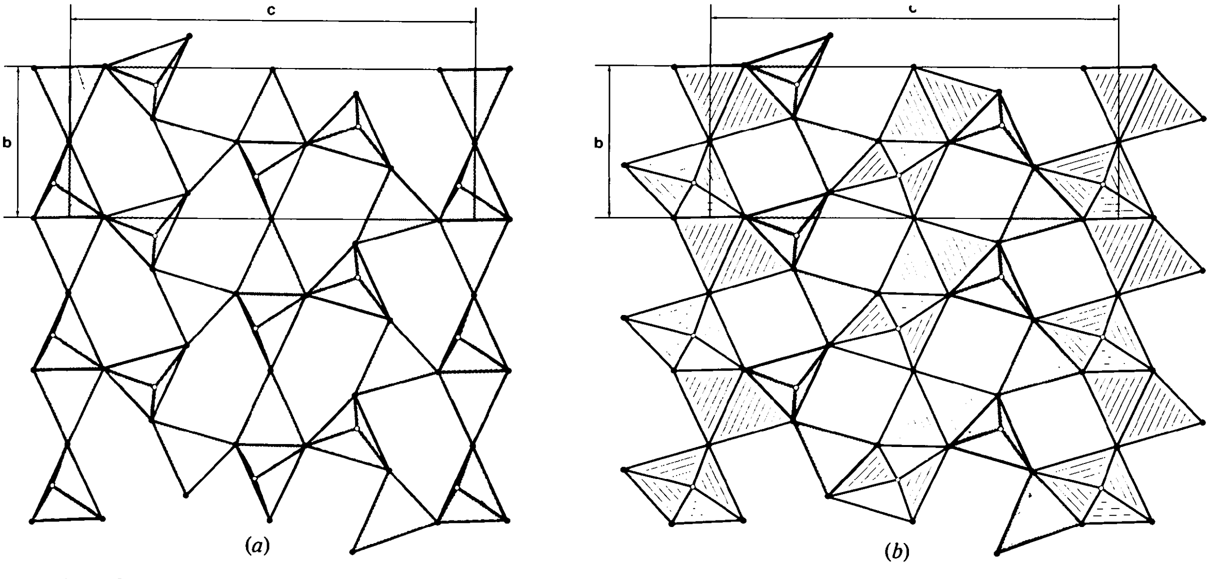


Fig. 4. The $(Sb_4O_5^{2+})_n$ layer of linked coordination polyhedra in $Sb_4O_5Cl_2$. In (*a*) all Sb atoms are shown as three-coordinated. In (*b*), however, Sb(1) is considered to be four-coordinated (striped polyhedra) and Sb(2) three-coordinated (shaded polyhedra). Small open circles denote Sb and filled circles, O atoms.

The linking of trigonal bipyramids (SbO_4E) and tetrahedra (SbO_3E) is realized in several ways in different structures. In $SbPO_4$ (Kinberger, 1970), however, the Sb—O coordination polyhedra are separated from each other and two-dimensional nets are formed by corner-sharing with phosphate tetrahedra.

Corner-sharing of SbO_3E tetrahedra is present in orthorhombic and cubic Sb_2O_3 (Svensson, 1974, 1975). In the cubic phase four tetrahedra form Sb_4O_6 molecules. Orthorhombic Sb_2O_3 consists of double infinite chains of tetrahedra [Fig. 6(*a*)].

Single infinite corner-shared chains of SbO_4E polyhedra are found in $SbO(H_2PO_4).H_2O$ (Särnstrand, 1974) and in $Sb(C_6H_3O_3).H_2O$ (Aurivillius & Särnstrand, 1976), where the linking is accomplished by equatorial and axial O atoms respectively [Fig. 6(*b*,*c*)].

In the low-temperature phase of SbOF (Åström & Andersson, 1973) there are infinite zig-zag chains of SbO_3FE polyhedra, where every O is shared among

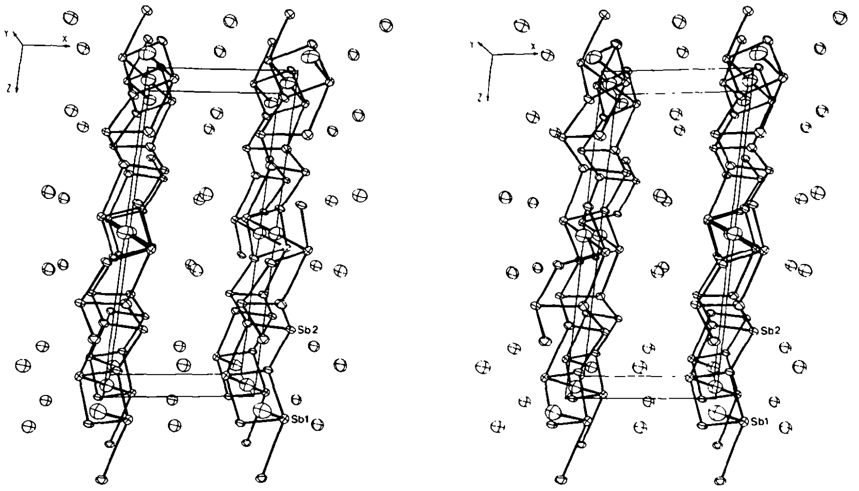


Fig. 5. Stereoscopic drawing of the structure of $Sb_4O_5Cl_2$. Within the $(Sb_4O_5^{2+})_n$ layer the Sb—O bonds are drawn. The Cl^- ions are situated between the layers. One Sb(1) and one Sb(2) are labeled. The unit cell is shown.

three Sb and the F atoms are unshared and equatorial [Fig. 6(*d*)].

β-Sb_2O_4 (Rogers & Skapski, 1964) and $SbNbO_4$ (Skapski & Rogers, 1965), isotypic with α-Sb_2O_4, are composed of infinite chains of edge-shared SbO_4E polyhedra, where each O is shared between two polyhedra [Fig. 6(*e*)]. These chains are separated by sheets of corner-shared octahedra containing Sb^V and Nb^V respectively. Such chains as in Fig. 6(*e*) are linked by corner-sharing through SbO_3E tetrahedra to form layers in $Sb_4O_4(OH)_2(NO_3)_2$ (Bovin, 1974*a*).

There are several compounds, *e.g.* $Sb_4O_4(OH)_2(NO_3)_2$ (Bovin, 1974*a*) and $Sb_4O_5(OH)ClO_4.\frac{1}{2}H_2O$ (Bovin, 1974*b*), in which the coordination polyhedra form two-dimensional nets as in the title structure, but hitherto no structures with three-dimensional networks of Sb^{III} coordination polyhedra have been reported. This is presumably due to the lone pairs of Sb^{III}, which prevent linking in three dimensions.

The author thanks Professor Bengt Aurivillius for valuable discussions, Dr Karin Aurivillius for constructive criticism of the manuscript and Mr Lars Thell for his skilled technical assistance.

This investigation is a part of a research project financially supported by the Swedish Natural Science Research Council.

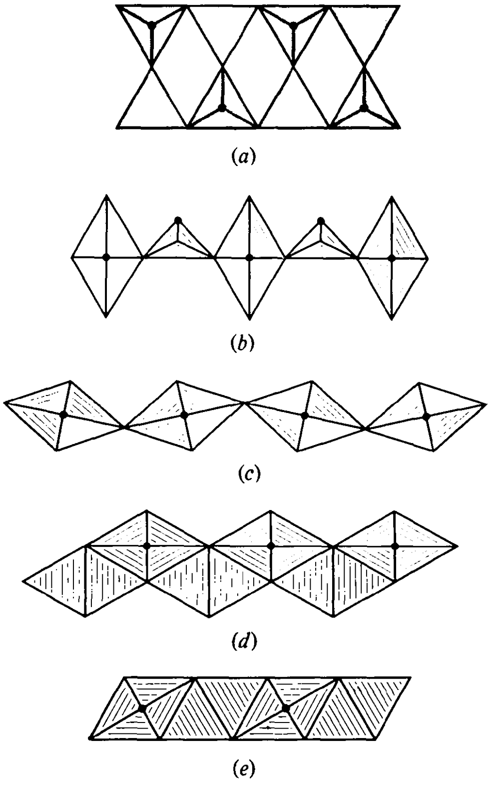


Fig. 6. The linking of coordination polyhedra in some Sb^{III} compounds. Tetrahedra (SbO_3E) are shaded and trigonal bipyramids are striped. (*a*) Orthorhombic Sb_2O_3 (Svensson, 1974); (*b*) $SbO(H_2PO_4).H_2O$ (Särnstrand, 1974); (*c*) $Sb(C_6H_3O_3).H_2O$ (Aurivillius & Särnstrand, 1976); (*d*) *L*-SbOF (Åström & Andersson, 1973); (*e*) β-Sb_2O_4 (Rogers & Skapski, 1964) and $SbNbO_4$ (Skapski & Rogers, 1965).

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The Crystal Structure of Aluminite

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(*Received* 5 *January* 1978; *accepted* 11 *April* 1978)

The crystal structure of aluminite, $Al_2(OH)_4(SO_4).7H_2O$, has been determined and refined from 1737 single-crystal diffractometer data by means of direct methods and least-squares refinement with anisotropic thermal parameters (isotropic for hydrogen atoms), giving $R = 0{\cdot}035$. Hydrogen atoms were located on a ΔF map. Lattice constants determined on the same diffractometer are $a = 7{\cdot}440\,(1)$, $b = 15{\cdot}583\,(2)$, $c = 11{\cdot}700\,(2)$ Å, $\beta = 110{\cdot}18\,(2)°$; space group $P2_1/c$; $Z = 4$. An important structural feature is the presence of the complex ion $[Al_4(OH)_8(H_2O)_6]^{4+}$ consisting of four edge-sharing Al octahedra polymerized in chains. These chains, running in the *a* direction, are connected to the SO_4 tetrahedra by a three-dimensional hydrogen-bonding system. In the asymmetric unit there are seven water molecules, four of which lie among the chains and tetrahedra as free water and three are coordinated, together with the hydroxyls, to the Al atoms as ligand waters. Hence the crystal chemical formula of aluminite is best written $[Al_2(OH)_4(H_2O)_3](SO_4).4H_2O$.

Introduction

Aluminite, $Al_2(OH)_4(SO_4).7H_2O$, is one of four well defined compounds (in addition to the alunites) belonging to the basic aluminium sulphates group. The other three are basaluminite, hydrobasaluminite (Hollingworth & Bannister, 1950) and meta-aluminite (Frondel, 1968). Other less known minerals, close in composition to these compounds, are winebergite, felsöbanyite, lapparentite and paraluminite. These basic aluminium sulphates have probably formed by the reaction of acid sulphate solutions, derived from the oxidation of pyrites, with marl or clay minerals at quite ordinary or moderate temperatures. From the study of the system Al_2O_3–SO_3–H_2O at 25 °C (Basset & Goodwin, 1949) it appears that in the phase diagram the region of existence of most basic aluminium sulphates occupies a small area in the neighbourhood of the water corner. In this area there are also the phases hydrargillite and bayerite, which are built up of layers of $Al(OH)_6$ octahedra: moreover sheets having hydrargillite structure are present in kaolinite. On this basis Bassett & Goodwin (1949) inferred that aluminite and basaluminite, frequently associated in their occurrence, might also show layer-lattice structures. From the present investigation it appears that this inference is not true, at least with regard to the structure of aluminite. The main feature of the crystal structure of aluminite is