Fig. 1. View of the structure at $z \sim 0.13$. The small dot is Al, the medium In; open circles are S.
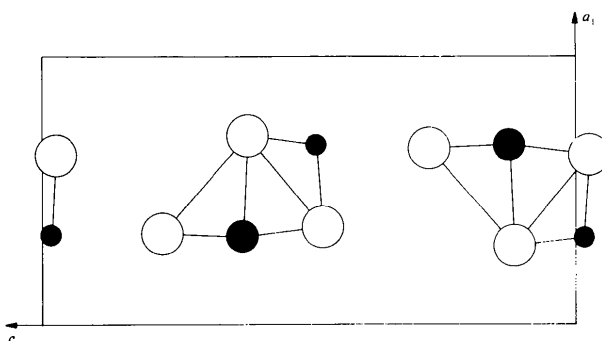

Fig. 2. Atomic positions on the $(01\overline{1}0)$ plane.

ships to the wurtzite structure type are: from ZnS to Al_2S_3 the hexagonal close packing of S is maintained but only $\frac{2}{3}$ of the corresponding tetrahedral voids are occupied by the metal; from Al_2S_3 to In_2Se_3 a strong distortion of some of the anion positions parallel to **c** is found, setting up a new fivefold coordination of one half of the voids. These are now occupied by In(1) in In_2Se_3 and by In in $AlInS_3$. The other half of the voids remain tetrahedrally coordinated and are occupied by In(2) in In_2Se_3 and Al in $AlInS_3$.

We thank Ms Dr E. Röttinger, Chemisches Laboratorium der Universität Freiburg, for the intensity collection and Ms U. Grass for drawings.

References

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A24, 321–324.

LIKFORMAN, A., CARRÉ, D. & HILLEL, R. (1978). *Acta Cryst.* B34, 1–5.

STEWART, J. M., MACHIN, P. S., DICKINSON, C., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY 76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Structure of Threadgoldite

BY PAUL PIRET, JEAN-PAUL DECLERCQ AND DOMINIQUE WAUTERS-STOOP

Laboratoire de Chimie Physique et de Crystallographie, Université de Louvain, 1 place Louis Pasteur, B-1348 Louvain-la-Neuve, Belgium

Abstract. $Al[(PO_4)_2(UO_2)_2].OH.8H_2O$ (a mineral from Kobokobo, Kivu, Zaïre), monoclinic, Cc, $a = 20.168$ (8), $b = 9.847$ (2), $c = 19.719$ (4) Å, $\beta = 110.71$ (2)°, $Z = 8$, $V = 3663$ Å³, $D_m = 3.4$, $D_c = 3.33$ Mg m⁻³; structure type: torbernite. The $[(PO_4)(UO_2)]_n^{n-}$ layers are connected principally by Al_2O_{10} groups which are composed of two octahedra.

Introduction. Threadgoldite is a mineral recently described by Deliens & Piret (1979). The results are here completed with the structure determination. The space group Cc or $C2/c$ was determined from

Weissenberg and precession photographs (systematic absences: hkl: $h + k = 2n + 1$; $h0l$: $l = 2n + 1$). However, if a few weak reflexions are neglected, the following unit cell is found: $a' = 9.530$, $b' = 9.847$, $c' = 9.860$ Å, $\beta' = 98.2°$, $V' = V/4$ with $\mathbf{a'} = \mathbf{a}/2 + \mathbf{c}/4$, $\mathbf{b'} = -\mathbf{b}$, $\mathbf{c'} = -\mathbf{c}/2$, space group $A2$, Am or $A2/m$.

Refinement of the structure with this unit cell or with the true unit cell (see *Abstract*) in space group $C2/c$ displays apparent disorder, especially for the Al_2O_{10} group. Consequently, the true space group is Cc, for which no disorder appears. Cell dimensions and intensities were measured on a Syntex $P2_1$ four-circle

Table 1. *Experimental conditions*

Radiation: Mo $K\alpha$, $\lambda = 0.71069$ Å
Graphite monochromator
ω scan: $-0.7°$ to $+0.7°$ (65 s) (background 2 × 16 s)
$2\theta_{max} = 47°$
Total number of independent reflections: 3231
Total observed $|I > 2.5\sigma(I)|$: 850
Crystal dimensions: $0.2 \times 0.04 \times 0.01$ mm
μ(Mo $K\alpha$) = 17.19 mm^{-1}

diffractometer with the experimental conditions given in Table 1. Intensities were corrected for absorption by an experimental method (Syntex, 1976). Scattering factors were those given by Cromer & Mann (1968) for neutral atoms; U was corrected for anomalous scattering (Cromer & Liberman, 1970). The structure was solved by the use of the Patterson function. Refinement by Fourier methods and least squares (full matrix) gave a final residual R of 0.076 for all observed reflections.*

Table 2. *Fractional coordinates* ($\times 10^4$)

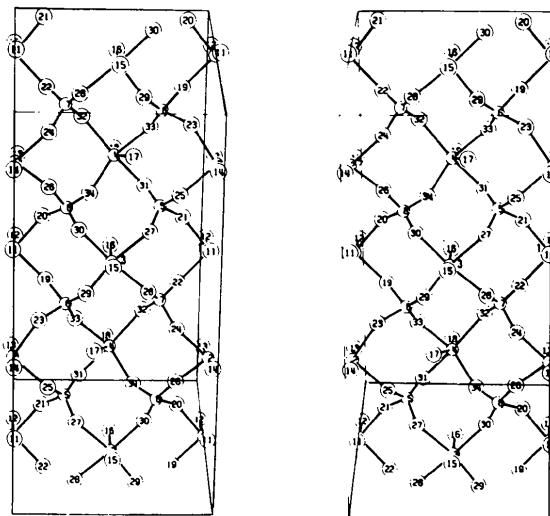|	x	y	z
U(1)	2050 (0)	3 (4)	4875 (0)
U(2)	2961 (2)	−50 (4)	2648 (2)
U(3)	2936 (2)	5068 (4)	114 (1)
U(4)	2098 (2)	4977 (4)	2359 (2)
P(5)	2479 (12)	2642 (18)	1289 (12)
P(6)	2418 (10)	2653 (18)	3692 (11)
P(7)	2474 (11)	2526 (18)	8845 (10)
P(8)	2673 (10)	2626 (20)	6337 (10)
Al(9)	−44 (11)	1546 (20)	2624 (11)
Al(10)	5002 (11)	3659 (21)	2360 (10)
O(11)	2888 (25)	−72 (65)	5378 (24)
O(12)	1047 (26)	27 (64)	4636 (26)
O(13)	1948 (25)	−149 (60)	2270 (24)
O(14)	3815 (27)	26 (66)	2938 (27)
O(15)	3817 (28)	5067 (62)	520 (27)
O(16)	1987 (24)	5067 (62)	−39 (25)
O(17)	2956 (27)	4109 (57)	2806 (27)
O(18)	1045 (27)	4872 (64)	1951 (28)
O(19)	1963 (32)	1599 (62)	4035 (31)
O(20)	1964 (31)	1416 (64)	5699 (30)
O(21)	2172 (30)	1417 (61)	835 (28)
O(22)	2079 (31)	−1576 (62)	4061 (31)
O(23)	3022 (30)	1274 (57)	3655 (27)
O(24)	2853 (29)	8304 (61)	3333 (28)
O(25)	3039 (33)	1746 (67)	1885 (31)
O(26)	3079 (30)	8288 (60)	2090 (28)
O(27)	3110 (32)	3223 (62)	1003 (32)
O(28)	3021 (32)	6791 (58)	4401 (30)
O(29)	2924 (31)	3647 (63)	4278 (30)
O(30)	2868 (30)	3251 (60)	5886 (30)
O(31)	2041 (31)	3405 (63)	1557 (29)
O(32)	2005 (30)	−3371 (60)	3291 (28)
O(33)	2030 (32)	3176 (63)	3058 (32)
O(34)	2317 (28)	3794 (57)	6496 (27)
O(35)	−481 (27)	103 (66)	1933 (29)
O(36)	428 (29)	−375 (54)	3059 (30)
O(37)	−692 (27)	−2093 (55)	2730 (26)
O(38)	4366 (28)	3670 (55)	8123 (28)
O(39)	643 (28)	2295 (55)	3587 (26)
O(40)	660 (28)	1743 (57)	2134 (27)
O(41)	5545 (29)	7921 (59)	7981 (27)
O(42)	5815 (28)	6591 (55)	6945 (28)
O(43)	4435 (28)	2058 (58)	7273 (26)
O(44)	4438 (28)	7798 (57)	6565 (27)
O(45)	4681 (25)	−6 (65)	1403 (24)
O(46)	4181 (27)	467 (55)	4787 (28)
O(47)	653 (28)	1536 (56)	820 (27)
O(48)	735 (26)	4933 (66)	5217 (25)
O(49)	603 (30)	2542 (58)	5786 (29)
O(50)	228 (26)	4775 (56)	8788 (25)
O(51)	4288 (27)	2621 (56)	9479 (28)
O(52)	4482 (30)	2785 (57)	4403 (28)

Fig. 1. Stereoscopic drawing (Johnson, 1965) of the layer (**b** across, **c** down). Limits for x: 0 to $\frac{1}{2}$ (box) and 0.1 to 0.4 (atoms). Limits for y and z: 0 to 1 (box) and −0.1 to 1.1 (atoms).
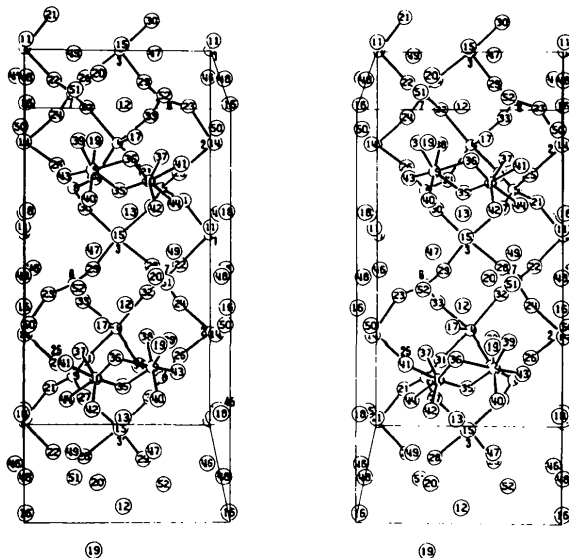

Fig. 2. Stereoscopic drawing (Johnson, 1965) of the interlayer structure (**b** across, **c** down). Limits for x: $\frac{1}{4}$ to $\frac{3}{4}$ (box) and 0.2 to 0.7 (atoms). Limits for y and z: 0 to 1 (box) and −0.1 to 1.1 (atoms).

Table 3. *Interatomic distances* (Å)

Standard deviations are about 0·055 Å for U—O, 0·06 Å for Al—O and P—O and 0·08 Å for O—O.

In the layer (d < 2·80 Å)

U(1)—O(11)	1·63	U(2)—O(13)	1·91	U(3)—O(15)	1·67	U(4)—O(17)	1·85
—O(12)	1·91	—O(14)	1·61	—O(16)	1·83	—O(18)	1·99
—O(19)	2·25	—O(23)	2·34	—O(27)	2·46	—O(31)	2·19
—O(20)	2·19	—O(24)	2·17	—O(28)	2·35	—O(32)	2·51
—O(21)	2·30	—O(25)	2·36	—O(29)	2·07	—O(33)	2·28
—O(22)	2·25	—O(26)	2·03	—O(30)	2·29	—O(34)	2·26
P(5)—O(21)	1·50	P(6)—O(19)	1·68	P(7)—O(22)	1·39	P(8)—O(20)	1·94
—O(25)	1·58	—O(23)	1·84	—O(24)	1·68	—O(26)	1·68
—O(27)	1·66	—O(29)	1·58	—O(28)	1·42	—O(30)	1·25
—O(31)	1·40	—O(33)	1·32	—O(32)	1·43	—O(34)	1·45
O(11)—O(20)	2·62	O(16)—O(30)	2·63	O(21)—O(27)	2·53	O(24)—O(32)	2·36
—O(21)	2·36	O(17)—O(31)	2·59	—O(31)	2·49	O(25)—O(27)	2·31
O(12)—O(20)	2·63	—O(33)	2·29	O(22)—O(24)	2·47	—O(31)	2·49
O(13)—O(24)	2·71	O(19)—O(23)	2·52	—O(28)	2·40	O(26)—O(30)	2·72
O(14)—O(23)	2·77	—O(29)	2·72	—O(32)	2·30	—O(34)	2·58
—O(25)	2·71	—O(33)	2·51	O(23)—O(29)	2·68	O(28)—O(32)	2·42
—O(26)	2·49	O(20)—O(30)	2·50	—O(33)	2·69	O(29)—O(33)	2·49
O(15)—O(27)	2·68	—O(34)	2·77	O(24)—O(26)	2·65	O(30)—O(34)	1·98
—O(29)	2·78	O(21)—O(25)	2·21	—O(28)	2·50		

In Al₂O₁₀ groups (d < 2·80 Å)

Al(9)—O(35)	1·95	Al(9)—O(39)	2·05	Al(10)—O(35)	1·76	Al(10)—O(41)	2·04
—O(36)	2·16	—O(40)	1·99	—O(36)	1·65	—O(42)	2·09
—O(38)	1·80	—O(43)	1·72	—O(37)	1·94	—O(44)	2·13
O(35)—O(36)	2·37	O(36)—O(37)	2·71	O(37)—O(41)	2·50	O(38)—O(43)	2·35
—O(37)	2·79	—O(38)	2·76	—O(44)	2·50	O(41)—O(42)	2·64
—O(40)	2·72	—O(41)	2·53	O(38)—O(39)	2·59		

Between atoms implicated in hydrogen bonds (d < 3·30 Å)

O(11)—O(46)	3·26	O(17)—O(37)	3·03	O(26)—O(38)	3·30	O(42)—O(49)	2·37
O(12)—O(36)	2·94	O(18)—O(40)	3·23	—O(43)	2·65	O(44)—O(45)	2·28
—O(39)	2·96	—O(45)	2·58	O(27)—O(44)	2·70	—O(49)	3·24
—O(47)	3·13	—O(48)	3·26	O(28)—O(51)	2·57	O(45)—O(46)	3·02
O(13)—O(40)	3·13	O(19)—O(39)	2·58	O(29)—O(52)	3·18	O(46)—O(48)	2·99
—O(42)	2·57	O(20)—O(49)	3·02	O(31)—O(47)	3·24	—O(51)	3·12
O(14)—O(37)	3·08	O(21)—O(47)	3·06	O(32)—O(41)	2·82	—O(52)	2·54
—O(43)	2·94	O(23)—O(46)	2·72	O(37)—O(52)	3·19	O(47)—O(52)	3·03
—O(50)	2·76	—O(52)	3·16	O(38)—O(51)	2·92	O(48)—O(49)	2·66
O(15)—O(35)	2·64	O(24)—O(51)	3·11	O(39)—O(50)	3·07	—O(50)	2·65
—O(43)	2·81	O(25)—O(37)	2·76	O(40)—O(47)	2·59	O(49)—O(51)	2·98
O(16)—O(48)	2·75	—O(44)	3·13	O(41)—O(50)	2·65		

Calculations were performed with *SHELX* (Sheldrick, 1976). The final coordinates are given in Table 2. The (non-significant) temperature factors B after group refinement are, for U 0·69, for P 0·16, for Al 0·16 and for O 1·13 Å².

Discussion. The unit cell and the structure are related to those of minerals of the torbernite group; for example autunite and sabugalite: autunite: $Ca[(PO_4)_2(UO_2)_2] \cdot {\sim}10H_2O$ (Beintema, 1938), $a = b = 7·00$, $c = 20·67$ Å, $\alpha = \beta = \gamma = 90°$; sabugalite: $(AlH)_{0·5}[(PO_4)_2(UO_2)_2] \cdot {\sim}8H_2O$ (Frondel, 1951), $a = b = 6·96$, $c = 19·3$ Å, $\alpha = \beta = \gamma = 90°$; threadgoldite: $Al[(PO_4)_2(UO_2)_2] \cdot OH \cdot 8H_2O$ (this work), $b/\sqrt{2} =$

6·963, $c/(2\sqrt{2}) = 6·972$, $a = 20·168$ Å, $\alpha = \gamma = 90°$, $\beta = 110·71°$.

The structure of threadgoldite consists of $[(PO_4)(UO_2)]_n^{n-}$ layers parallel to **b** and **c** (Fig. 1). The configuration around U is a tetragonal bipyramid. The mean U—O distance in UO_2 is the normal value, 1·80 Å. The mean distance between U and O of the base of the pyramid is 2·27 Å, shorter than the corresponding distances in the pentagonal bipyramid (2·36 Å) and hexagonal bipyramid (2·51 Å) of phurcalite (Piret & Declercq, 1978) and phuralumite (Piret, Piret-Meunier & Declercq, 1979). The mean P—O distance in the PO_4 anions is 1·55 Å. The layers are connected by two Al atoms, two OH ions [probably O(35) and O(36)] and sixteen H_2O molecules. Each Al is surrounded by six O

atoms. The two octahedra share one edge, forming an Al_2O_{10} group (Fig. 2). The mean Al—O distance is 1·94 Å. Principal distances are given in Table 3.

References

BEINTEMA, J. (1938). Recl Trav. Chim. Pays-Bas, 57, 155–170.
CROMER, D. T. & LIBERMAN, D. (1970). J. Chem. Phys. 53, 1891–1898.
CROMER, D. T. & MANN, J. B. (1968). Acta Cryst. A24, 321–324.
DELIENS, M. & PIRET, P. (1979). Bull. Minéral. 102, 338–341.
FRONDEL, C. (1951). Am. Mineral. 36, 671–680.
JOHNSON, C. K. (1965). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
PIRET, P. & DECLERCQ, J. P. (1978). Acta Cryst. B34, 1677–1679.
PIRET, P., PIRET-MEUNIER, J. & DECLERCQ, J. P. (1979). Acta Cryst. B35, 1880–1882.
SHELDRICK, G. M. (1976). SHELX. Program for crystal structure determination. Univ. of Cambridge, England.
SYNTEX (1976). XTL/E-XTL. Structure Determination System Operation Manual. Syntex Analytical Instruments Inc., 10040 Bubb Road, Cupertino, California 95014, USA.

An X-ray Structure Redetermination of Antimony Trichloride

By ANNEGRET LIPKA*

Institut für Anorganische Chemie und Strukturchemie, Universität Düsseldorf, Universitätsstrasse 1, 4000 Düsseldorf, Federal Republic of Germany

Abstract. $SbCl_3$, orthorhombic, Pnma, $a = 8·111$ (2), $b = 9·419$ (1), $c = 6·313$ (1) Å, $Z = 4$, $D_x = 3·14$ Mg m^{-3}. The structure has been redetermined from counter data; with 963 independent observed reflections the final R is 0·045. The main improvements and results of this more accurate study are (1) the space group, and thus the mirror symmetry of the $SbCl_3$ molecules, was confirmed, (2) the structure was refined with anisotropic thermal parameters, (3) Sb—Cl lengths differ significantly due to intermolecular interactions, (4) intermolecular contacts and the resulting eightfold coordination of the Sb atom are discussed.

Introduction. $SbCl_3$ molecules have been found as units of structures in adducts of $SbCl_3$ with different donor systems (Hulme & Scruton, 1968; Hulme, Mullen & Scruton, 1969; Hulme & Szymański, 1969; Hulme & Mullen, 1976; Demaldé, Mangia, Nardelli, Pelizzi & Vidoni Tani, 1972; Lipka & Mootz, 1978; Lipka, 1978).

Comparison with the structure of $SbCl_3$ as determined by Lindqvist & Niggli (1956) showed that the

shapes of the molecules are similar, but with regard to intermolecular contacts the coordination polyhedra are different. However, the accuracy of the above-mentioned work based on refinement by difference Fourier techniques was not sufficient to allow further discussion of structural details. The equivalence of the Sb—Cl lengths was just within large e.s.d.'s and the mirror symmetry of the molecules resulting from the adopted space group Pbmn was questionable, for accurate X-ray (Nyburg, Ozin & Szymański, 1971) and neutron (Bartl, 1975) structure determinations had shown that the isostructural $BiCl_3$ crystallizes in the noncentrosymmetric space group $Pn2_1a$. Therefore a complete X-ray structure redetermination of $SbCl_3$ was undertaken.

$SbCl_3$ p.a. was purified by reductive distillation over Sb under a nitrogen atmosphere. A bipyramidal-shaped single crystal grown from the melt was selected in a dry box under an argon atmosphere and sealed in a Lindemann-glass capillary ($\varnothing = 0·3$ mm). Photographs showed systematic absences $0kl$ for $k + l$ odd and $hk0$ for h odd, indicative of the space groups Pnma and $Pn2_1a$ (conventional setting $Pna2_1$). Cell dimensions were obtained by least-squares refinement from angular measurements of 15 reflections in the range $36 < 2\theta <$

* Present address: Laboratoire de Cristallographie aux Rayons X, Université de Genève, 24 quai Ernest Ansermet, CH-1211 Genève 4, Switzerland.