

## The Crystal Structure of $\text{InOHSO}_4(\text{H}_2\text{O})_2$

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Crystals of the basic indium sulfate  $\text{InOHSO}_4(\text{H}_2\text{O})_2$  are monoclinic with unit cell dimensions:  $a = 6.06 \text{ \AA}$ ,  $b = 7.89 \text{ \AA}$ ,  $c = 12.66 \text{ \AA}$ ,  $\beta = 107.5^\circ$ . The indium atoms are octahedrally surrounded by oxygen atoms at an average distance of  $2.16 \text{ \AA}$  with no significant differences between the In—O bond lengths. The  $\text{InO}_6$  octahedra are joined together by means of common corners, to chains running parallel to the  $b$  axis. Different indium-oxygen chains have no oxygen atoms in common but are linked together by means of hydrogen bonds to the oxygen atoms of the sulfate groups.

The preparation of a basic indium sulfate by the addition of ethanol to a solution of indium sulfate in water has been reported in the literature<sup>1,2</sup>. It is described as forming small bi-axial positive crystals, probably monoclinic<sup>1</sup>. The formula, based on indium and sulfate analysis only, has been given as  $\text{In}_2\text{O}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ <sup>1</sup> or  $2\text{InOHSO}_4 \cdot 5\text{H}_2\text{O}$ <sup>2</sup>. The structure of a basic sulfate prepared in the same way has been investigated as part of a program on structure determinations of basic salts of some trivalent metals<sup>3-5</sup>.

### EXPERIMENTAL

*Preparation of crystals.* Indium sulfate was prepared from indium metal of 99.95 % purity supplied by the Consolidated Mining and Smelting Company of Canada, Ltd. Solutions of indium sulfate with concentrations ranging from 0.05 M to 1.0 M  $\text{In}^{3+}$  were mixed with one, or two times their volume of ethanol. According to powder X-ray photographs the phases precipitated in the various solutions were identical. Good crystals were obtained from 0.05 M indium sulfate solutions when these were mixed with twice the volume of ethanol. The crystallization was very slow and several weeks were required for the formation of crystals as large as 0.1 mm in length.

*Analysis.* An air dried sample was used for the analysis. The crystals are very slightly soluble in water but dissolve readily in warm dilute acid. Indium was determined by precipitation with ammonia, igniting, and weighing as  $\text{In}_2\text{O}_3$ . Sulfate was determined as  $\text{BaSO}_4$ . The water content was obtained by Brush and Penfield's method as described by Kolthoff and Sandell<sup>6</sup>. The density was found by comparing the weight of a specimen, in benzene and in air.

The results of the analysis (Table 1) seem to favor the formula  $\text{InOHSO}_4(\text{H}_2\text{O})_2$  rather than  $\text{InOHSO}_4(\text{H}_2\text{O})_{2.5}$ . This is supported by the result of the structure determination.

Table 1.

	Found	Calculated for	
		InOHSO <sub>4</sub> (H <sub>2</sub> O) <sub>2</sub>	InOHSO <sub>4</sub> (H <sub>2</sub> O) <sub>2.5</sub>
% In <sub>2</sub> O <sub>3</sub>	52.3	52.60	50.86
% SO <sub>3</sub>	30.4	30.34	29.34
% H <sub>2</sub> O	17.5	17.07	19.80
Density	3.0 <sub>0</sub>	3.03	3.14

## UNIT CELL AND SPACE GROUP

The crystals are rod-shaped and elongated along the *b*-direction. Rotation and Weissenberg photographs around the *b* and *c* axes showed that the unit cell is monoclinic and enabled the following approximate values of the lattice parameters to be calculated:  $a = 6.1 \text{ \AA}$ ,  $b = 7.9 \text{ \AA}$ ,  $c = 12.6 \text{ \AA}$ ,  $\beta = 107^\circ$ . More precise values were determined from powder photographs (Table 2) taken with CuK $\alpha$  radiation in a Guinier focusing camera, using KCl ( $a = 6.2929 \text{ \AA}$ ) as the internal standard. The values found were:

$$a = 6.05_8 \text{ \AA}; b = 7.89_4 \text{ \AA}; c = 12.66_5 \text{ \AA}; \beta = 107.5^\circ; V = 577.6 \text{ \AA}^3.$$

The accuracy is estimated to about 0.1 %.

The density calculated on the basis of four formula weights in the unit cell is 3.03, that observed is 3.0<sub>0</sub> (Table 1).

The only systematically absent reflections are of the type  $0k0$  with  $k$  odd, which indicates the space group  $P2_1/m$  or  $P2_1$ . Since all reflections for  $k$  odd were extremely weak on the photographs, the rather few nonobserved  $0k0$  reflections might have been only accidentally absent. However, nothing during the structure determination indicated a lower space group than  $P2_1/m$  which was therefore assumed to be the correct symmetry.

Weissenberg photographs were taken around the *b* and *c* axes (layer lines 0—4 and 0,2,4, respectively) using the multiple film technique with CuK radiation. Intensities were estimated visually with the aid of a calibrated intensity strip and corrected for Lorentz and polarization factors. No correction for absorption was applied. For the photographs taken around the *b* axis, a crystal approximately 0.1 mm in length and  $0.06 \times 0.08$  mm in cross-section was used. A crystal of about the same size was used for the photographs taken around the *c*-axis.

## STRUCTURE DETERMINATION

All reflections for  $k$  odd or  $l$  odd are extremely weak and it does not seem likely that the In atoms make any contributions to the intensities of these reflections. Accordingly, it should be possible to describe the positions of the In atoms with a unit cell four times smaller than the actual unit cell. The  $x$  parameters are restricted to  $x \approx 0$  or  $x \approx \frac{1}{2}$  since the In-In peaks on the  $h0l$  Patterson projection (Fig. 1) occur at  $u = 0$ ,  $w = 0$ , and at  $u = 0$ ,  $w = \frac{1}{2}$ . Other peaks in the projection are too small to correspond to In-In vectors. In space group  $P2_1/m$  the following In positions are thus possible, if only structurally nonequivalent arrangements are included:

1.	4 In in 4(f):	$\pm(x, y, z), \pm(x, \frac{1}{2}-y, z)$	$x=0, y=0, z=1/4$
2.	2 In in 2(e):	$\pm(x, 1/4, z)$	$x=0, z=1/4$
	2 In in 2(e):		$x=0, z=3/4$
3.	2 In in 2(e):		$x=0, z=0$
	2 In in 2(e):		$x=0, z=1/2$
4.	2 In in 2(a):	$0, 0, 0; 0, \frac{1}{2}, 0$	
	2 In in 2(c):	$0, 0, \frac{1}{2}; 0, \frac{1}{2}, \frac{1}{2}$	

Table 2. Powder photograph of  $\text{InOHSO}_4(\text{H}_2\text{O})_2$ . Guinier camera.  $\text{CuK}\alpha$  radiation.

$hkl$	$10^4 \sin^2\theta$ calc.	$10^4 \sin^2\theta$ obs.	$I$ obs.
002	163	165	m
100	178	179	m
10 $\bar{2}$	238	239	s
012	258	259	vw
110	273	275	vw
020	381	381	m
102	443	445	m
022	544	545	s
120	559	559	s
10 $\bar{4}$	624	625	w
004	651	653	vwv
20 $\bar{2}$	669	671	vw
200	711	716	vw
11 $\bar{4}$	719		
014	746	748	vw
210	806	805	vw
122	824	825	w
10 $\bar{5}$	939	947	w
211	949		
20 $\bar{4}$	952		
12 $\bar{4}$	1005	1005	w
024	1032	1034	vw
104	1033		
11 $\bar{5}$	1034		
22 $\bar{2}$	1050	1050	vw
123	1078	1084	vw
202	1079		
220	1092	1096	vw
13 $\bar{2}$	1095		
22 $\bar{4}$	1333	1334	w
10 $\bar{6}$	1335		
124	1414	1418	vw
222	1460	1464	vw
006	1464		
040	1523	1522	w
016	1560	1562	vw
20 $\bar{6}$	1561		
300	1600	1601	vw
31 $\bar{3}$	1601		
304	1637	1638	vw
042	1686	1684	vw
140	1701	1701	vw
12 $\bar{6}$	1716	1717	vw
14 $\bar{2}$	1762	1759	vw
322	1837	1839	vw
026	1845		
22 $\bar{6}$	1942	1941	vw
14 $\bar{2}$	1966	1966	w
324	2018	2017	vw

The distance, 3.95 Å, between the mirror planes of the unit cell is too small for the sulfate groups to have positions between these planes. The sulfur atoms must therefore occupy two twofold positions, 2(*e*), on the mirror planes. The perpendicular distance from the S atom to one of the edges of the SO<sub>4</sub> tetrahedron should be about 0.8 Å<sup>13</sup>. In a Patterson projection along the *b* axis, that is perpendicular to the mirror planes, an In-S vector should then always be accompanied by a peak of two overlapping In-O vectors of approximately the same height and at a distance of about 0.8 Å.

The Patterson projection,  $P(uvw)$ , calculated from the  $h0l$  reflections,\* is shown in Fig. 1. The four alternative sets of In positions all result in identical In-In vectors corresponding to the high peaks at  $u = 0$ ,  $w = 1/2$ , and at the origin.  $P(uvw)$  also shows four double peaks which, according to the discussion above, probably represent vectors between In and atoms of the sulfate groups.

Assuming the parameters of the sulfur atoms, S<sub>1</sub> and S<sub>2</sub>, to be  $x_1, z_1$  and  $x_2, z_2$ , respectively, the following In-S vectors should appear on the  $h0l$  projection if the 1st or 2nd alternative sets of In positions are applicable:

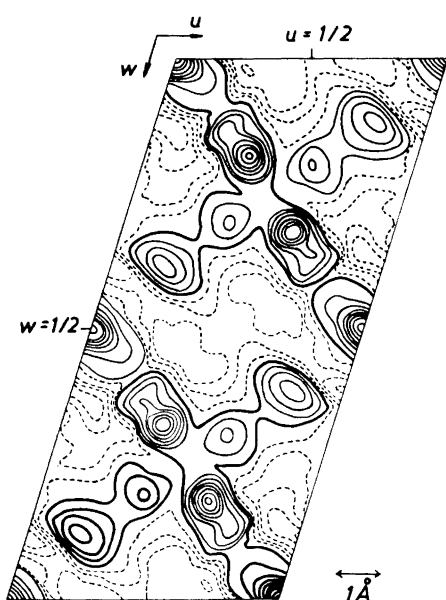


Fig. 1. Patterson projection along the *b*-axis. Dashed lines indicate negative regions. In the peaks at (0,0) and (0, 1/2) only every 5th contour has been drawn. The four double peaks with the approximate positions  $\pm [0.38, 0.18]$  and  $\pm [0.38, 0.68]$  contain the In-S vectors.

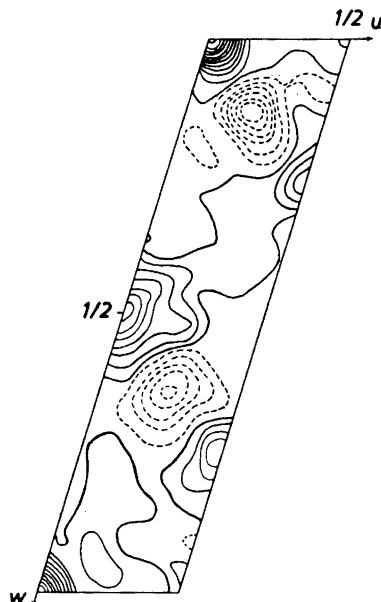


Fig. 2. The generalized Patterson projection  $P_1(uw)$ . Dashed lines indicate negative regions.

\* All calculations were carried out on the "Besk" computer with the use of programs designed for the calculation of structure factors<sup>7</sup> and electron densities.<sup>8</sup>

$$\begin{array}{ll} \pm[\bar{x}_1, 1/4 - z_1] & \pm[\bar{x}_2, 1/4 - z_2] \\ \pm[x_1, 1/4 + z_1] & \pm[x_2, 1/4 + z_2] \end{array}$$

For the 3rd and 4th alternatives the corresponding vectors are:

$$\begin{array}{ll} \pm[x_1, z_1] & \pm[x_2, z_2] \\ \pm[x_1, 1/2 + z_1] & \pm[x_2, 1/2 + z_2] \end{array}$$

The presence of only four double peaks in  $P(uvw)$  within the unit cell instead of the expected eight, and the heights of these peaks compared to the height of the origin peak, indicate the presence of overlapping In—S vectors. This can be explained by assuming:  $x_2 \approx x_1$  and  $z_2 \approx 1/2 + z_1$ . The most likely positions of the In—S vectors are:

$$\pm [0.38, 0.68_5] \text{ and } \pm [0.38, 0.18_5]$$

The  $y$  parameters of the corresponding sulfur atoms are  $1/4$  or  $3/4$ . For geometrical reasons they must be the same and can be chosen to be  $1/4$  since  $y = 3/4$  would result in an equivalent structure.

In a generalized Patterson projection, calculated from the  $h1l$  reflections to which the In atoms do not contribute, the following sulfur-sulfur vectors should then be present:

$$\begin{array}{ll} u = 0.24, w = 0.13 & \text{weight: } -2 \\ u = 0, w = 0.50 & \text{» : } +2 \\ u = 0.24, w = 0.63 & \text{» : } -2 \end{array}$$

As is shown on Fig. 2 the calculated vectors correspond to the three largest peaks of  $P_1(uw)$ , thus confirming the correctness of the assumed arrangement of sulfur atoms.

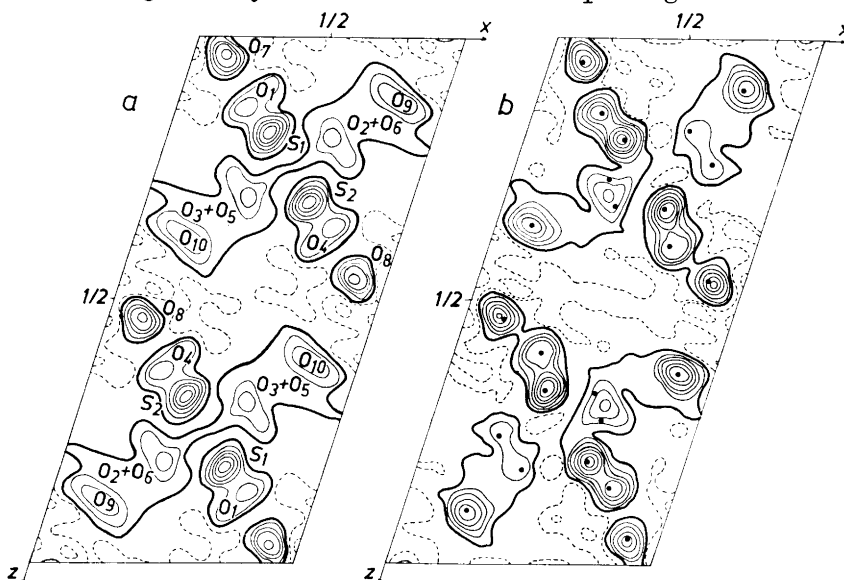
The In contributions determine the signs of the structure factors of those  $h0l$  reflections for which  $l$  is even, and a Fourier projection with the In atoms subtracted was calculated using these reflections. The resulting Fourier map (Fig. 3a) gives only an average of the electron density projections of the two halves of the unit cell, but because it contains all but the very weakest reflections it still shows the main features of the structure.

Since only reflections for  $l$  even have been used, false centers of symmetry have been introduced at  $x = 1/2, z = 1/4$  and at  $x = 1/2, z = 3/4$  with the result that the two crystallographically non-equivalent sulfate groups of the unit cell are related by centers of symmetry in the Fourier map. The peak at  $x \approx 0.29, z \approx 0.12$ , which is not well resolved from the peak of the sulfur atom, gives the positions of those oxygen atoms of the sulfate groups ( $\text{O}_1$  and  $\text{O}_4$ ), which lie outside the mirror planes. The rather extended peak at  $x \approx 0.37, z \approx 0.30$  at a distance of roughly  $1.5 \text{ \AA}$  from the sulfur peak, indicates the positions of those oxygen atoms of the sulfate groups ( $\text{O}_2, \text{O}_3, \text{O}_5$ , and  $\text{O}_6$ ) which lie on the mirror planes. The projection shows only two more peaks which probably indicate the positions of the twelve remaining oxygen atoms of the unit cell: ( $\text{O}_7, \text{O}_8, \text{O}_9, \text{O}_{10}$ ). Approximate values of the corresponding  $y$  parameters can be found from geometrical considerations since the available space is restricted by the presence of mirror planes and by oxygen atoms of the sulfate groups for which approximate positions are already known. The oxygen atom corresponding to the peak at  $x = 0.12, z = 0.05$  must then be on the

mirror plane since its distance to  $O_1$  and  $O_4$  would otherwise be much too short. It therefore corresponds to four oxygen atoms occupying two twofold positions ( $O_7$  and  $O_8$ ). The rather extended peak at  $x \approx 0.81$ ,  $z \approx 0.12$  must correspond to oxygen atoms outside the mirror planes since they would otherwise occupy positions too close to one of the atoms  $O_2$ ,  $O_3$ ,  $O_5$ , or  $O_6$ . This peak must then represent eight oxygen atoms in two fourfold positions ( $O_9$  and  $O_{10}$ ).

The positions of the atoms  $O_7$  and  $O_8$  eliminate the 2nd and 3rd alternative sets of In positions, that is with the In atoms on the mirror planes, since they would lead to too short In—O distances (about 0.8 Å). The remaining two alternatives both correspond to a structural arrangement consisting of  $\text{InO}_6$  octahedra joined together by means of common corners to chains running parallel to the  $b$  axis. Of the oxygen atoms of the sulfate groups only the atoms outside the mirror planes ( $O_1$  and  $O_4$ ) belong to the coordination polyhedra of the In atoms.  $O_7$  and  $O_8$  (on the mirror planes) are in contact with two In atoms each and the remaining eight oxygen atoms of the unit cell ( $O_9$  and  $O_{10}$ ; outside the mirror planes) are each in contact with one In atom.

The shapes of the peaks in Fig. 3a suggest that the presence of the very weak reflections for  $l$  odd results from small displacements of the sulfate groups and of  $O_9$  and  $O_{10}$ . Since the peaks corresponding to the sulfur atoms do not seem to be deformed the displacements of the  $\text{SO}_4$  tetrahedra were assumed to correspond to small rotations around the sulfur atoms. The resulting trial structures, corresponding to the two remaining alternatives for the In positions, are shown in Fig. 4. They were the results of attempts to get the best fit both



*Fig. 3a.* Electron density projection along the  $b$ -axis. Only structure factors for  $l = 2n$  have been included and the contribution from the indium atoms has been subtracted. *Fig. 3b.* Final electron density projection along the  $b$ -axis with all structure factors included and with the contribution from the indium atoms subtracted. The final positions of the sulfur and oxygen atoms are marked by dots.

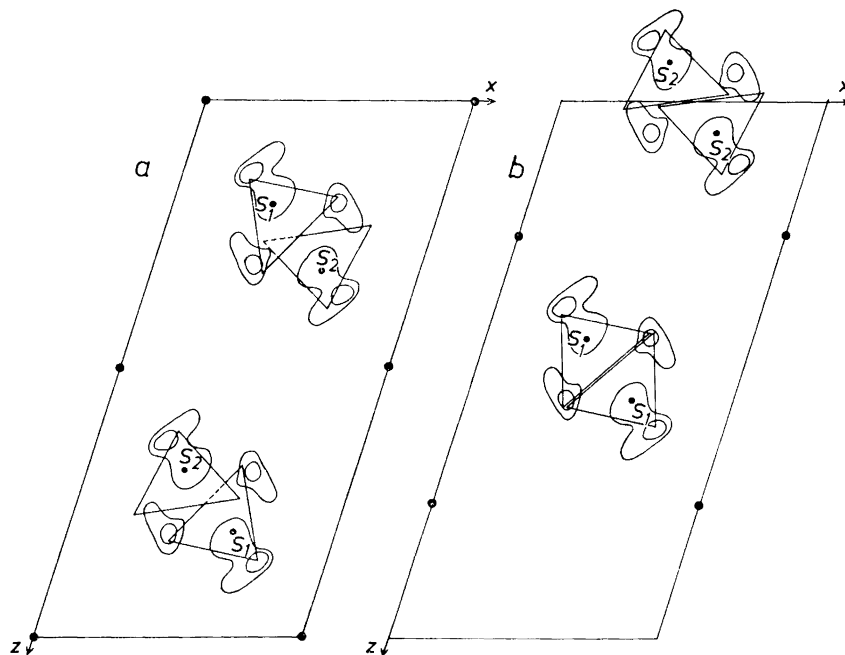


Fig. 4. The two trial structures assumed to explain the presence at the weak reflections for  $l$  odd. Black dots indicate In atoms. The sulfate groups are represented by triangles. Some of the contours from the electron density projection in Fig. 3 a are also shown in the figures:  
 a) In atoms assumed to be at centers of symmetry  
 b) In atoms assumed to occupy a fourfold position with  $x = 0$ ,  $y = 0$ ,  $z = \frac{1}{4}$ .

to the peaks of the electron density projections and to the expected In—O distance of about 2.1 Å, without making any changes in the assumed sulfur positions. If the angles of rotation of the  $\text{SO}_4$  tetrahedra around the sulfur atoms are the same for the two trial structures, they both give the same absolute values for those structure factors for which  $l$  is even and differ only for  $l$  odd.

Structure factors were calculated for the  $h1l$  and  $h3l$  reflections for the two trial structures. The  $y$  parameters of  $\text{O}_1$  and  $\text{O}_4$  were determined by assuming the O—O distance in a  $\text{SO}_4$  tetrahedron to be 2.4 Å<sup>13</sup>. The oxygen atoms  $\text{O}_9$  and  $\text{O}_{10}$  were not included in the calculations. Their contributions to the  $h1l$  and  $h3l$  reflections should be small since they have positions close to the planes  $y = 0$  and  $y = 1/2$ . Only the structure corresponding to the 4th alternative, that is with the indium atoms at centers of symmetry, gave a reasonable agreement for structure factors for  $l$  odd.

Most of the signs of the  $h1l$  and  $h3l$  structure factors could then be determined, and more accurate parameters were obtained from the generalized electron density projections  $S_3(xz) + S_1(xz)$ , to which atoms on the mirror planes do not contribute, and  $S_3(xz) - S_1(xz)$ , which to a large extent eliminates

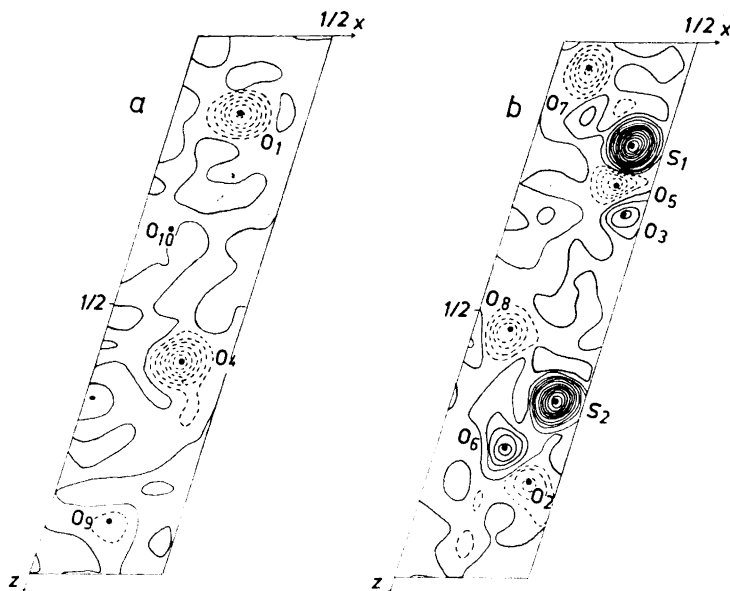


Fig. 5a. The Fourier summation  $S_3(x,z) + S_1(x,z)$ .  
 Fig. 5b. The Fourier summation  $S_3(x,z) - S_1(x,z)$ .

the contributions from atoms outside the mirror planes relative to those on these planes (Fig. 5).  $S_1(xz)$  and  $S_3(xz)$  represent the Fourier summations:

$$S_1(xz) = \sum_h \sum_l F(h1l) \sin 2\pi(hx + lz)$$

$$S_3(xz) = \sum_h \sum_l F(h3l) \sin 2\pi(hx + lz)$$

The parameters obtained were used for determining the signs of  $h0l$  reflections with  $l$  odd. From  $h0l$  electron density projections the positions of  $O_9$  and  $O_{10}$  could then be determined (Fig 3b).

#### REFINEMENT AND ACCURACY OF THE PARAMETERS

The parameters obtained from the electron density projections were refined by means of difference maps. The refinement was carried out from the  $h1l$  and the  $h3l$  reflections independently. The positions of the sulfur atoms and the oxygen atoms in fourfold positions were also refined in the same way from the  $h0l$  data. A single isotropic temperature factor of  $1.6 \text{ \AA}^2$ , determined by a comparison of observed and calculated structure factors, was used for all the atoms. Atomic scattering factors were calculated according to the analytical expressions given by Vand, Eiland and Pepinsky<sup>9</sup>.

The  $y$  parameters of  $O_9$  and  $O_{10}$  were determined from the heights of the corresponding peaks on the generalized  $h3l$  electron density projection. The



height of the peak at the  $\text{O}_9$  position corresponds to  $y = -0.010$ . Since no peak occurs at the  $\text{O}_{10}$  position the corresponding parameter should be close to  $y = 0$  (Fig. 5a). Accurate values of the  $y$  parameters of  $\text{O}_1$  and  $\text{O}_4$  cannot be obtained from the  $h1l$  or  $h3l$  generalized projections since the corresponding peak heights are rather insensitive to a change of  $y$  parameters; they were therefore calculated assuming the  $\text{O}_1-\text{O}_1$  and  $\text{O}_4-\text{O}_4$  distances to be the same as the average value of the final  $\text{O}_2-\text{O}_3$  and  $\text{O}_5-\text{O}_6$  distances, which are 2.38 and 2.39 Å respectively. The  $y$  parameters obtained in this way are consistent with the  $h1l$  and  $h3l$  difference maps, giving no peaks of significant heights at the  $\text{O}_1$  and  $\text{O}_4$  positions.

The final parameters (Table 3) were calculated by taking the average of the values obtained in the independent refinements of the  $h0l$ ,  $h1l$ , and  $h3l$  data. Calculated and observed structure factors for the zones  $h0l$ ,  $h1l$ ,  $h2l$ ,  $h3l$ , and  $hk0$  are given in Table 5. If only observed reflections are included the overall  $R$  factor  $\left[ R = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|} \right]$  is 0.127. For those 205 observed reflections to which In contributes,  $R = 0.125$  (with In contributions only  $R = 0.23$ ). For the 171 reflections without In contributions,  $R = 0.132$ . The  $R$  value is 0.164 for the 103 reflections with  $l$  odd which were observed. The higher  $R$  value for these reflections may probably be explained in part by the high percentage of very weak reflections for which intensities are difficult to estimate accurately.

The standard deviations of the positional parameters were calculated according to the formula given by Cruickshank<sup>10</sup>. For the  $x$  and  $z$  parameters the average between the results obtained from the  $h1l$  and  $h3l$  data was 0.01<sub>1</sub> Å for the sulfur atoms and 0.02<sub>5</sub> Å for the oxygen atoms except  $\text{O}_9$  and  $\text{O}_{10}$ . The standard deviations of the  $x$  and  $z$  parameters of  $\text{O}_9$  and  $\text{O}_{10}$ , calculated from the  $h0l$  data, were 0.04 Å. A value of 0.03<sub>5</sub> Å was obtained for the  $y$  parameters of  $\text{O}_9$  and  $\text{O}_{10}$  from an estimate of the standard deviation in the electron density of the final  $h3l$  difference map.

Since the parameters were refined from the  $h1l$  and the  $h3l$  data independently, a standard deviation may also be calculated from a comparison between the refined parameters obtained from the  $h1l$  and the  $h3l$  data, respectively. This gives a value of 0.03<sub>1</sub> Å for the oxygen atoms ( $\text{O}_1$  to  $\text{O}_8$ ), which is nearly the same as the value given above.

These values lead to standard deviations of 0.01 to 0.04 Å in the In—O distances and of about 0.03 Å in S—O distances within the  $\text{SO}_4$  tetrahedra. If the  $\text{SO}_4$  groups are assumed to be regular tetrahedra the variation of the S—O distances ( $\text{S}_1-\text{O}_2$ ,  $\text{S}_1-\text{O}_3$ ,  $\text{S}_2-\text{O}_5$ ,  $\text{S}_2-\text{O}_6$ ) around the calculated mean (1.48 Å) corresponds to a standard deviation of 0.02<sub>8</sub> Å, which gives some support for the correctness of the values calculated above. The standard deviations in O—O distances are generally less than 0.05 Å.

#### DISCUSSION OF THE STRUCTURE

Interatomic distances are listed in Table 4. Projections of the structure along the  $a$  and  $b$  axes are shown in Figs. 6 and 7.

Table 3. Final parameters.

		<i>x</i>	<i>y</i>	<i>z</i>
In:	2 In <sub>1</sub> in 2( <i>a</i> )	0	0	0
	2 In <sub>2</sub> in 2( <i>c</i> )	0	0	1/2
S:	2 S <sub>1</sub> in 2( <i>e</i> )	0.378	1/4	0.197
	2 S <sub>2</sub> in 2( <i>e</i> )	0.610	3/4	0.326
O:	4 O <sub>1</sub> in 4( <i>f</i> )	0.258	0.099	0.146
	2 O <sub>2</sub> in 2( <i>e</i> )	0.608	1/4	0.180
	2 O <sub>3</sub> in 2( <i>e</i> )	0.422	1/4	0.321
	4 O <sub>4</sub> in 4( <i>f</i> )	0.676	0.599	0.397
	4 O <sub>5</sub> in 2( <i>e</i> )	0.364	3/4	0.270
	2 O <sub>6</sub> in 2( <i>e</i> )	0.736	3/4	0.242
OH:	2 O <sub>7</sub> in 2( <i>e</i> )	0.121	3/4	0.050
	2 O <sub>8</sub> in 2( <i>e</i> )	0.142	3/4	0.540
H <sub>2</sub> O:	4 O <sub>9</sub> in 4( <i>f</i> )	0.762	0.990	0.100
	4 O <sub>10</sub> in 4( <i>f</i> )	0.867	0.000	0.640

Indium is octahedrally surrounded by six oxygen atoms at an average distance of 2.16 Å, with no significant differences between the indium-oxygen bond lengths. The InO<sub>6</sub> octahedra are joined together by means of common corners, in chains running parallel to the *b* axis. The unit cell contains two crystallographically non-equivalent chains of InO<sub>6</sub> octahedra; they are, however, both built up in the same way. No oxygen atoms are shared between two chains.

Table 4. Interatomic distances.

Within the InO<sub>6</sub> octahedra:

In <sub>1</sub> —2O <sub>7</sub> : 2.14 Å	In <sub>2</sub> —2O <sub>8</sub> : 2.15 Å
—2O <sub>9</sub> : 2.19	—2O <sub>10</sub> : 2.16
—2O <sub>1</sub> : 2.18	—2O <sub>4</sub> : 2.15
O <sub>7</sub> —O <sub>9</sub> : 3.09 Å	O <sub>8</sub> —O <sub>10</sub> : 3.09 Å
O <sub>7</sub> —O <sub>9</sub> : 3.02	O <sub>8</sub> —O <sub>10</sub> : 3.00
O <sub>1</sub> —O <sub>9</sub> : 3.16	O <sub>4</sub> —O <sub>10</sub> : 3.05
O <sub>1</sub> —O <sub>9</sub> : 3.01	O <sub>4</sub> —O <sub>10</sub> : 3.04
O <sub>1</sub> —O <sub>7</sub> : 3.03	O <sub>4</sub> —O <sub>8</sub> : 2.99
O <sub>1</sub> —O <sub>7</sub> : 3.07	O <sub>4</sub> —O <sub>8</sub> : 3.09

Within the SO<sub>4</sub> tetrahedra:

S <sub>1</sub> —2O <sub>1</sub> : 1.44 Å	S <sub>2</sub> —2O <sub>4</sub> : 1.47 Å
—O <sub>2</sub> : 1.47	—O <sub>5</sub> : 1.45
—O <sub>3</sub> : 1.51	—O <sub>6</sub> : 1.48
O <sub>2</sub> —O <sub>3</sub> : 2.38 Å	O <sub>5</sub> —O <sub>6</sub> : 2.39 Å
O <sub>1</sub> —O <sub>2</sub> : 2.36	O <sub>4</sub> —O <sub>5</sub> : 2.40
O <sub>1</sub> —O <sub>3</sub> : 2.45	O <sub>4</sub> —O <sub>6</sub> : 2.42
(O <sub>1</sub> —O <sub>1</sub> : 2.38)	(O <sub>4</sub> —O <sub>4</sub> : 2.38)

Short O—O distances:

O <sub>7</sub> —O <sub>5</sub> : 2.73 Å	O <sub>8</sub> —O <sub>3</sub> : 2.70 Å
O <sub>9</sub> —O <sub>2</sub> : 2.58	O <sub>10</sub> —O <sub>3</sub> : 2.78
O <sub>9</sub> —O <sub>6</sub> : 2.65	O <sub>10</sub> —O <sub>5</sub> : 2.85

Other O—O distances < 3.5 Å

O <sub>1</sub> —O <sub>5</sub> : 3.14 Å	O <sub>3</sub> —O <sub>4</sub> : 3.16 Å
O <sub>1</sub> —O <sub>9</sub> : 3.39	O <sub>4</sub> —O <sub>10</sub> : 3.27
O <sub>1</sub> —O <sub>10</sub> : 3.12	O <sub>6</sub> —O <sub>10</sub> : 3.12
O <sub>2</sub> —O <sub>8</sub> : 3.42	O <sub>9</sub> —O <sub>9</sub> : 3.41
	O <sub>9</sub> —O <sub>10</sub> : 3.37

Table 5. Observed and calculated structure factors.

$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$
000	—	484	$\bar{7}04$	56	63	307	15	24
1	124	132	$\bar{6}$	48	51	4	<13	8
2	85	91	$\bar{5}$	69	77	5	<10	-11
3	142	145	$\bar{4}$	97	98			
4	58	63	$\bar{3}$	138	142	$\bar{7}08$	45	40
5	84	92	$\bar{2}$	243	207	$\bar{6}$	56	72
6	55	50	$\bar{1}$	188	155	$\bar{5}$	58	67
7	46	52	0	108	100	$\bar{4}$	116	109
			1	147	143	$\bar{3}$	87	71
$\bar{7}01$	<9	-8	2	114	119	$\bar{2}$	96	85
$\bar{6}$	<13	-17	3	77	90	$\bar{1}$	146	149
$\bar{5}$	<13	-3	4	62	74	0	88	94
$\bar{4}$	18	23	5	68	76	1	82	99
$\bar{3}$	12	17	6	69	71	2	61	61
$\bar{2}$	<7	-4				3	61	77
$\bar{1}$	14	9	$\bar{7}05$	<10	0	4	97	83
0	—	-6	$\bar{6}$	24	31	5	33	25
1	<5	1	$\bar{5}$	16	-21			
2	38	-36	$\bar{4}$	20	-19	$\bar{7}09$	<8	-2
3	19	-20	$\bar{3}$	49	49	$\bar{6}$	18	-18
4	28	28	$\bar{2}$	37	-31	$\bar{5}$	17	17
5	<13	11	$\bar{1}$	51	-38	$\bar{4}$	<13	6
6	<13	-10	0	8	4	$\bar{3}$	28	-19
7	<8	-3	1	22	18	$\bar{2}$	26	13
			2	24	29	$\bar{1}$	13	15
$\bar{7}02$	45	55	3	<13	-4	0	<13	5
$\bar{6}$	71	83	4	<14	-14	1	14	-19
$\bar{5}$	47	54	5	<13	2	2	<13	-6
$\bar{4}$	113	138	6	<8	-2	3	<13	1
$\bar{3}$	98	96				4	<10	-1
$\bar{2}$	131	107	$\bar{7}06$	38	48			
$\bar{1}$	217	268	$\bar{6}$	49	57			
0	120	126	$\bar{5}$	79	84	$\bar{7}010$	26	39
1	218	203	$\bar{4}$	56	54	$\bar{6}$	48	52
2	157	179	$\bar{3}$	152	132	$\bar{5}$	85	88
3	73	86	$\bar{2}$	192	185	$\bar{4}$	74	72
4	82	90	$\bar{1}$	143	127	$\bar{3}$	98	96
5	57	65	0	153	145	$\bar{2}$	104	95
6	56	62	1	88	88	$\bar{1}$	69	58
7	39	49	2	81	96	0	81	85
			3	63	66	1	59	66
			4	62	61	2	54	64
$\bar{7}03$	<10	1	5	88	80	3	88	78
$\bar{6}$	<13	-7	6	27	37	4	41	47
$\bar{5}$	<13	5						
$\bar{4}$	30	-31	$\bar{7}07$	<9	0	$\bar{6}011$	11	16
$\bar{3}$	<9	-6	$\bar{6}$	<13	-7	$\bar{5}$	<13	-1
$\bar{2}$	75	73	$\bar{5}$	<13	8	$\bar{4}$	<13	-13
$\bar{1}$	40	-27	$\bar{4}$	37	35	$\bar{3}$	<13	5
0	11	5	$\bar{3}$	15	-15	$\bar{2}$	27	-14
1	30	30	$\bar{2}$	20	-14	$\bar{1}$	<13	3
2	20	-9	$\bar{1}$	21	20	0	<13	1
3	<11	-8	0	15	-16	1	<13	12
4	13	-17	1	24	-28	2	<13	11
5	<13	-8	2	<12	-5	3	<11	-16
6	<12	14						

$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$
$\bar{6}012$	38	44	311	20	22	$\bar{2}15$	26	-20
$\bar{5}$	49	42	4	<14	3	$\bar{1}$	13	7
$\bar{4}$	95	79	5	<15	-9	0	27	29
$\bar{3}$	116	96	6	<14	4	1	13	-16
$\bar{2}$	70	58	7	<9	-1	2	<12	-8
$\bar{1}$	83	65				3	<14	8
0	59	71	$\bar{7}12$	19	18	4	<15	-3
1	49	47	$\bar{6}$	<15	3	5	<14	4
2	57	62	$\bar{5}$	17	-11	6	<9	-6
3	41	41	$\bar{4}$	25	21			
			$\bar{3}$	49	-50	$\bar{7}16$	<11	1
			$\bar{2}$	16	14	$\bar{6}$	<15	12
$\bar{6}013$	<7	-3	$\bar{1}$	11	4	$\bar{5}$	24	-24
$\bar{5}$	<10	-4	0	55	-57	$\bar{4}$	<14	2
$\bar{4}$	16	13	1	82	82	$\bar{3}$	<12	4
$\bar{3}$	13	-5	2	11	16	$\bar{2}$	27	-32
$\bar{2}$	<13	1	3	<13	-19	$\bar{1}$	39	39
$\bar{1}$	23	-25	4	<14	5	0	<11	6
0	<12	-4	5	20	-13	1	<12	11
1	26	28	6	<13	3	2	<13	7
2	<9	-10	7	<7	-3	3	30	-32
						4	20	17
						5	<13	-10
$\bar{5}014$	39	42	$\bar{7}13$	<11	-2			
$\bar{4}$	77	61	$\bar{6}$	<15	5	$\bar{7}17$	<11	2
$\bar{3}$	69	45	$\bar{5}$	<15	-2	$\bar{6}$	<14	-13
$\bar{2}$	72	61	$\bar{4}$	25	-26	$\bar{5}$	<15	9
$\bar{1}$	73	61	$\bar{3}$	34	32	$\bar{4}$	<14	6
0	52	39	$\bar{2}$	<8	-7	$\bar{3}$	27	-26
1	59	65	$\bar{1}$	<7	-5	$\bar{2}$	34	32
			0	<7	3	$\bar{1}$	<12	12
$\bar{4}015$	<10	-13	1	<8	-8	0	27	-33
$\bar{3}$	<10	11	2	18	25	1	13	17
$\bar{2}$	<9	0	3	11	-16	2	<14	-6
$\bar{1}$	<9	-3	4	<13	1	3	<15	3
			5	<15	2	4	<14	0
$\bar{3}016$	29	37	6	<12	1	5	<11	-6
$\bar{2}$	38	43						
			$\bar{7}14$	<12	-4	$\bar{7}18$	<10	4
010	-	-	$\bar{6}$	<15	-5	$\bar{6}$	<14	-6
1	46	-50	$\bar{5}$	23	25	$\bar{5}$	<15	2
2	61	65	$\bar{4}$	39	-39	$\bar{4}$	<15	-6
3	<11	6	$\bar{3}$	<11	2	$\bar{3}$	24	-23
4	<13	-1	$\bar{2}$	<9	7	$\bar{2}$	32	32
5	<15	-9	$\bar{1}$	43	-44	$\bar{1}$	<13	3
6	<14	-11	0	69	66	0	<13	9
7	<10	9	1	<10	9	1	<14	3
			2	<11	-9	2	21	-20
$\bar{7}11$	<11	-1	3	15	14	3	21	16
$\bar{6}$	<15	4	4	25	-26	4	18	-15
$\bar{5}$	<15	-4	5	<15	8	5	<9	-8
$\bar{4}$	<13	0	6	<11	-4			
$\bar{3}$	<11	-9				$\bar{6}19$	<13	6
$\bar{2}$	<8	6	$\bar{7}15$	<11	5	$\bar{5}$	<15	6
$\bar{1}$	<6	2	$\bar{6}$	<15	-6	$\bar{4}$	15	-20
0	<4	0	$\bar{5}$	19	-18	$\bar{3}$	32	31
1	<6	13	$\bar{4}$	27	25	$\bar{2}$	<14	13
2	25	-28	$\bar{3}$	<12	-1	$\bar{1}$	<14	0

$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$
019	<14	10	421	<17	18	225	<15	12
1	22	-22	5	<18	8	3	<17	14
2	20	18	6	<16	1	4	<17	-10
3	<15	-9				5	<17	-13
4	<12	-1	$\bar{7}22$	45	55			
			$\bar{6}$	48	50	$\bar{7}26$	45	57
$\bar{6}110$	<12	0	$\bar{5}$	84	112	$\bar{6}$	61	68
$\bar{5}$	<14	-9	$\bar{4}$	81	98	$\bar{5}$	45	64
$\bar{4}$	<15	-10	$\bar{3}$	133	141	$\bar{4}$	96	98
$\bar{3}$	19	22	$\bar{2}$	121	105	$\bar{3}$	85	79
$\bar{2}$	<14	-6	$\bar{1}$	72	53	$\bar{2}$	133	118
$\bar{1}$	<14	5	0	220	217	$\bar{1}$	200	151
0	<15	12	1	141	133	0	92	93
1	15	-14	2	119	145	1	137	138
2	<14	5	3	132	148	2	79	83
3	<13	-10	4	60	71	3	76	80
4	<9	-7	5	80	80	4	87	79
			6	57	51	5	41	33
$\bar{6}111$	<11	0	$\bar{7}23$	<13	3			
$\bar{5}$	<13	-9	$\bar{6}$	<18	14	$\bar{7}27$	<12	-7
$\bar{4}$	14	17	$\bar{5}$	23	-24	$\bar{6}$	<17	4
$\bar{3}$	<14	-10	$\bar{4}$	23	-20	$\bar{5}$	<18	3
$\bar{2}$	<15	15	$\bar{3}$	17	23	$\bar{4}$	<18	-2
$\bar{1}$	<15	-10	$\bar{2}$	<17	-5	$\bar{3}$	<16	12
0	<15	-13	$\bar{1}$	<9	5	$\bar{2}$	<14	-9
1	20	20	0	<9	5	$\bar{1}$	<14	3
2	14	-14	1	29	30	0	<14	1
3	<11	1	2	<13	4	1	19	-28
			3	28	-30	2	<17	8
$\bar{6}112$	<10	-6	4	<18	-9	3	<18	11
$\bar{5}$	<13	-3	5	<18	9	4	<18	2
$\bar{4}$	<14	10	6	<14	6	5	<12	6
$\bar{3}$	<15	-5						
$\bar{2}$	<15	3	$\bar{7}24$	35	46	$\bar{7}28$	36	48
$\bar{1}$	18	16	$\bar{6}$	64	79	$\bar{6}$	51	61
0	<15	-11	$\bar{5}$	67	74	$\bar{5}$	87	104
1	<14	-1	$\bar{4}$	88	101	$\bar{4}$	47	47
			$\bar{3}$	141	136	$\bar{3}$	83	73
020	94	110	$\bar{2}$	195	142	$\bar{2}$	111	94
1	244	224	$\bar{1}$	153	114	$\bar{1}$	81	82
2	111	108	0	91	73	0	139	142
3	74	88	1	113	125	1	79	83
4	121	137	2	140	163	2	76	90
5	48	53	3	73	86	3	87	83
6	63	67	4	84	76	4	45	39
7	42	49	5	89	80	5	41	43
			6	35	41			
$\bar{6}21$	<17	-16	$\bar{7}25$	<15	16	$\bar{7}29$	<10	-11
$\bar{5}$	<18	10	$\bar{6}$	<18	4	$\bar{6}$	<16	-4
$\bar{4}$	<16	15	$\bar{5}$	<18	6	$\bar{5}$	<18	-6
$\bar{3}$	<13	-1	$\bar{4}$	<16	-12	$\bar{4}$	<19	5
$\bar{2}$	<10	9	$\bar{3}$	23	-26	$\bar{3}$	<18	-9
$\bar{1}$	12	-7	$\bar{2}$	15	18	$\bar{2}$	<18	0
0	<6	7	$\bar{1}$	28	-22	$\bar{1}$	45	38
1	<9	-16	0	<11	-7	0	<18	-4
2	35	-36	1	24	21	1	<18	-13
3	<14	-1						

$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$
229	<18	-8	330	22	23	$\bar{3}35$	<11	4
3	<18	-3	4	18	-16	$\bar{2}$	21	-22
4	<18	6	5	<14	3	$\bar{1}$	<9	8
			6	18	15	0	<9	0
$\bar{7}210$	23	40				1	<10	-1
$\bar{6}$	52	63	$\bar{6}31$	<12	8	2	<12	4
$\bar{5}$	52	55	$\bar{5}$	19	16	3	<13	-7
$\bar{4}$	97	91	$\bar{4}$	21	23	4	<13	3
$\bar{3}$	84	73	$\bar{3}$	<11	5			
$\bar{2}$	65	53	$\bar{2}$	17	20	$\bar{5}36$	22	25
$\bar{1}$	100	96	$\bar{1}$	8	-8	4	<14	-4
0	76	82	0	<4	-3	$\bar{3}$	<13	2
1	67	77	1	12	-17	$\bar{2}$	19	18
2	73	71	2	<10	15	$\bar{1}$	47	-41
3	43	53	3	<12	2	0	22	22
4	41	52	4	<14	-5	1	<15	-14
			5	<14	-7	2	31	-29
$\bar{6}211$	19	-16	6	<12	5	3	43	43
$\bar{5}$	<16	-1						
$\bar{4}$	<17	2	$\bar{7}32$	16	-15	$\bar{4}37$	<14	0
$\bar{3}$	<18	-13	$\bar{6}$	<11	-4	$\bar{3}$	<13	-7
$\bar{2}$	22	13	$\bar{5}$	17	17	$\bar{2}$	21	-16
$\bar{1}$	<18	-13	$\bar{4}$	19	-24	$\bar{1}$	20	20
0	<18	11	$\bar{3}$	24	22	0	<12	3
1	<18	21	$\bar{2}$	15	20	1	20	-25
2	<18	-12	$\bar{1}$	16	13	2	19	21
3	<18	-6	0	10	-15			
			1	48	-48	$\bar{3}38$	28	23
$\bar{6}212$	31	38	2	21	26	$\bar{2}$	23	-26
$\bar{5}$	47	45	3	<12	-7	$\bar{1}$	<13	7
$\bar{4}$	75	62	4	<14	-14	0	<13	-9
$\bar{3}$	85	70	5	29	25	1	23	-21
$\bar{2}$	76	81				2	28	28
$\bar{1}$	72	66	$\bar{5}33$	17	-18			
0	61	62	$\bar{4}$	19	17	$\bar{3}39$	24	-29
1	57	74	$\bar{3}$	<11	9	$\bar{2}$	38	40
2	37	40	$\bar{2}$	15	-9	$\bar{1}$	<14	-12
3	28	37	$\bar{1}$	27	-27	0	28	-28
			0	15	19	1	34	35
$\bar{5}213$	<17	0	1	<9	13	2	<13	-11
$\bar{4}$	<16	-8	2	16	-23			
$\bar{3}$	27	23	3	<13	12	$\bar{3}310$	15	-15
$\bar{2}$	19	-12	4	<14	-8	$\bar{2}$	<14	1
$\bar{1}$	<16	-10				$\bar{1}$	<14	-2
0	<16	5	$\bar{5}34$	19	-23	0	20	-16
1	<17	-7	$\bar{4}$	32	31	1	19	13
			$\bar{3}$	<12	10	2	<12	4
$\bar{5}214$	37	41	$\bar{2}$	<10	5			
$\bar{4}$	52	38	$\bar{1}$	<9	-6	$\bar{3}311$	17	24
$\bar{3}$	80	58	0	59	-49	$\bar{2}$	<14	-19
$\bar{2}$	71	55	1	41	40	$\bar{1}$	<14	-8
$\bar{1}$	63	58	2	17	-13	0	25	26
0	57	66	3	32	-36	1	<13	-10
1	57	38	4	44	43	2	<12	-1
030	-	-						
1	28	-29	$\bar{5}35$	<14	4	040	223	218
2	29	-28	$\bar{4}$	<13	7	1	90	107

$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$	$hkl$	$F_o$	$F_c$
240]	97	112	060	42	40	080	142	117
3	78	89	1	112	123	1	75	53
4	33	42	2	78	86	2	78	53
5	77	88	3	46	49	3	76	59
6	41	43	4	86	87	4	35	30
			5	48	44			
050	—	—				090	—	—
1	17	-15	070	—	—	1	<12	-2
2	24	31	1	<16	18	2	<11	12
3	<15	-4	2	27	-28	3	<9	-5
4	<15	5	3	<14	2			
5	<14	-6	4	<13	3	0100	40	37
6	<10	-10	5	<14	3	1	50	55

The hydrogen atoms of the unit cell may be approximately located from considerations of the sum of electrostatic bond strengths for the various oxygen atoms and the short O—O distances listed in Table 4. The number of short O—O contacts is the same as the number of hydrogen atoms and may probably be taken as an indication of the presence of hydrogen bonds.  $O_7$  and  $O_8$ , which

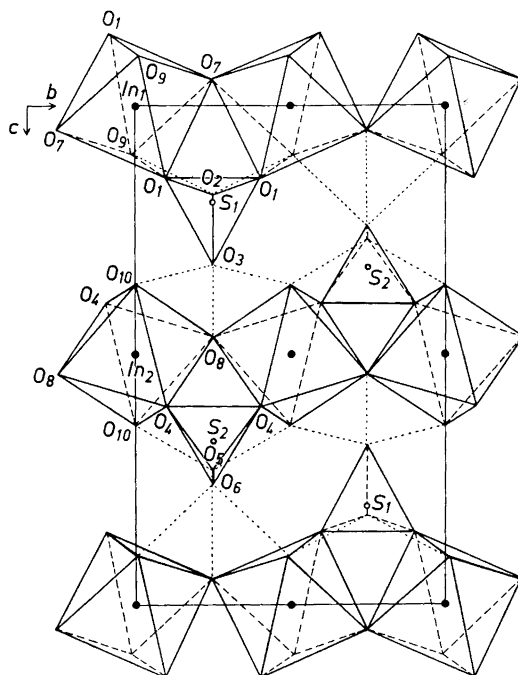


Fig. 6. Projection of the structure along the  $a$ -axis. Dotted lines represent short O—O distances which probably indicate hydrogen bonds. Filled circles represent indium atoms. At the lower left hand corner one of the dotted lines connects  $O_7$  with  $O_5$  and not with  $O_6$  as the drawing seems to indicate.

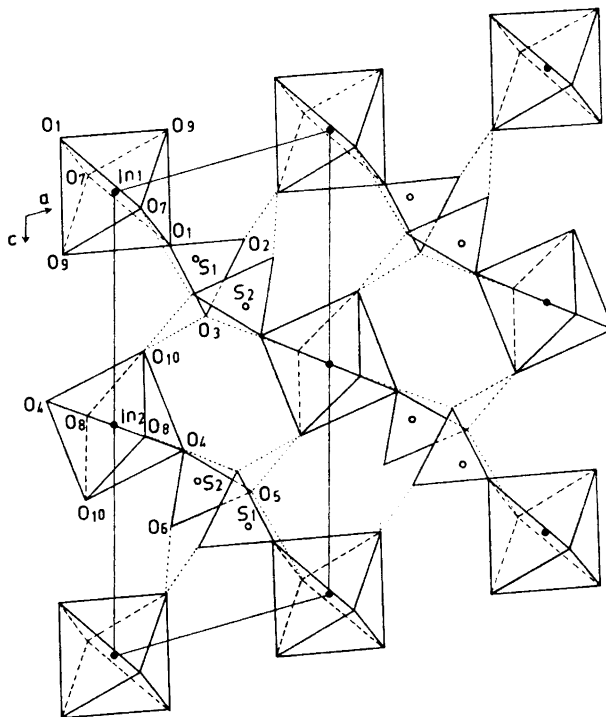


Fig. 7. Projection of the structure along the  $b$ -axis. Filled circles indicate indium atoms. Dotted lines represent short O—O distances.

are both in contact with two indium atoms and therefore probably associated with one hydrogen atom, are each involved in one short O—O distance.  $O_9$  and  $O_{10}$ , which are both in contact with only one In atom and therefore are likely to be  $H_2O$  molecules, are each involved in two short O—O distances. All the hydrogen bonds are formed to those oxygen atoms of the sulfate groups which are not in contact with indium atoms. The  $H_2O$  molecules,  $O_9$ , of the  $In_1$  chains (see Figs. 6 and 7) are bonded to  $O_2$  and  $O_6$ ; the bond lengths are 2.58 and 2.65 Å, with estimated standard deviations slightly less than 0.05 Å, and the angle  $O_2-O_9-O_6$  is  $102^\circ$ . The  $H_2O$  molecules,  $O_{10}$ , of the  $In_2$  chains are bonded to  $O_3$  and  $O_5$  with bond lengths of 2.78 and 2.85 Å; estimated standard deviations are about 0.05 Å and the angle  $O_3-O_{10}-O_5$  is  $91^\circ$ .  $O_3$  and  $O_5$  are also bonded to the OH groups,  $O_7$  and  $O_8$ . The  $H_2O$  molecules of the  $In_2$  chains are therefore bonded to those oxygen atoms of the sulfate groups which are also involved in hydrogen bonds to the OH groups,  $O_7$  and  $O_8$ . The  $H_2O$  molecules of the  $In_1$  chains, on the other hand, are bonded to oxygen atoms ( $O_2$  and  $O_6$ ) which do not form other hydrogen bonds. This is the main difference between the two kinds of indium-oxygen chains in the unit cell. The  $O_{10}-O_3$  and  $O_{10}-O_5$  hydrogen bonds are longer than the bonds  $O_9-O_2$



and  $\text{O}_9-\text{O}_6$ , and in view of the standard deviations this difference may have some significance.

The indium atoms within one of the chains of  $\text{InO}_6$  octahedra can also be described as being joined together by means of single hydroxo bridges ( $\text{O}_7$  and  $\text{O}_8$ ). The corresponding In—O distances are the most accurately known of the indium-oxygen bond lengths in the structure; they are for  $\text{In}_1-\text{O}_7$  2.14 Å, and for  $\text{In}_2-\text{O}_8$  2.15 Å, with estimated standard deviations of about 0.01 Å. The shortest In—In distance within a chain is 3.95 Å, which is equal to one half of the length of the  $b$  axis. The  $\text{O}_7-\text{In}_1-\text{O}_7$  and  $\text{O}_8-\text{In}_1-\text{O}_8$  angles are exactly  $180^\circ$ , as required by considerations of symmetry. The corresponding angles at the oxygen atoms are about  $134^\circ$  for both  $\text{In}_1-\text{O}_7-\text{In}_1$  and  $\text{In}_2-\text{O}_8-\text{In}_2$ . These angles can be compared with those found in  $\text{TlOHSO}_4(\text{H}_2\text{O})_{2.5}^3$ , which contains  $\text{TlO}_6$  chains very similar to the  $\text{InO}_6$  chains in the structure under consideration. The corresponding O—Tl—O angle, which is not determined by symmetry, is very nearly  $180^\circ$ . The average value of the two Tl—O—Tl angles, which do not differ significantly, is  $134^\circ$ , that is the same as in the indium salt.

In—O—In chains may also be distinguished in the three dimensional framework of  $\text{InO}_2\text{F}_4$  octahedra in the structure of  $\text{InOHF}_2$ , in which the In—In distances are 3.87 Å, the In—O distances are 2.08 Å, and the angles O—In—O and In—O—In are  $180^\circ$  and  $137^\circ$ , respectively<sup>11</sup>. In the cubic  $\text{In}(\text{OH})_3$ <sup>12</sup> the  $\text{InO}_6$  octahedra seem to share corners with six other octahedra; the In—In distance is 3.96 Å, which is equal to one half of the unit cell edge. These distances are similar to those found in the present structure determination.

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