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The Crystal Structure of $AlSeCl_7$

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Solid $AlSeCl_7$ appears as irregular blocks of yellow triclinic crystals. The space group is $P\bar{1}$. The lattice constants are $a=9{\cdot}87$, $b=8{\cdot}27$, $c=9{\cdot}83$ Å, $\alpha=139{\cdot}9$, $\beta=94{\cdot}8$, $\gamma=93{\cdot}8°$ and $Z=2$. The structure, solved by minimum functions applied to photographic data (Mo radiation), is built up from $AlCl_4$ tetrahedra and $SeCl_6$ octahedra. The tetrahedra (Al–Cl = 2·13 Å) are quite regular but the octahedra are distorted and contain three long and three short Se–Cl distances (3·04 and 2·11 Å). The chlorine atoms contributing to the long Se–Cl distances are shared between Al and Se. The structure consists approximately of $AlCl_4^-$ and $SeCl_3^+$ ions.

Introduction

A number of compounds with composition $M^{n+}Cl_n.XCl_4$ (X being S, Se or Te) were prepared and investigated by Groeneveld (1953). For the complex with M = Al and X = Se he found an Al/Se ratio of 1:1 and not a ratio of 2:1 as reported by Weber (1858) and Lenher & Kao (1926). In view of the chlorine donating properties of $SeCl_4$ it can be anticipated that the title compound consists of $AlCl_4^-$ and $SeCl_3^+$ ions. Aluminum compounds $AlBr_3$, $Al(CH_3)_3$ and $Al(CH_3)_2X$, X being Br or Cl, form dimeric molecules in the solid and vapour state, in which the aluminum atoms are tetrahedrally coordinated by Br, Cl or CH_3 groups (Renes & MacGillavry, 1945; Palmer & Elliot, 1938; Brockway & Davidson, 1941). Gaseous aluminum trichloride also consists of double molecules, Al_2Cl_6 (Palmer & Elliot, 1938). In the solid state, however, it forms cubic close-packed layers of Cl ions with Al ions in octahedral coordination (Ketelaar, MacGillavry & Renes, 1947). In view of the fluctuating coordinating properties of aluminum with respect to chlorine it was deemed necessary to investigate the crystal structure of $AlSeCl_7$.

Experimental

$AlSeCl_7$ was obtained by boiling a solution of 1·5 mol $AlCl_3$ and 1 mol $SeCl_4$ in SO_2Cl_2. After cooling, pale yellow crystals appeared which were filtered in the absence of moisture. The product was washed with SO_2Cl_2 and dried *in vacuo*. The crystals were of irregular shape. Owing to their extreme sensitivity to moisture several crystals were used during the investigation.

Unit-cell dimensions were derived from equi-inclination Weissenberg photographs about axes a' (zero, first and second layer) and c' (zero layer) using unfiltered copper radiation ($\lambda_{K\alpha_1}=1{\cdot}54051$, $\lambda_{K\alpha_2}=1{\cdot}54433$, $\lambda_{K\beta}=1{\cdot}39217$ Å). The photographs were superposed with aluminum powder lines ($a=4{\cdot}0492$ Å at 20°C). The measured interplanar spacings $d(hkl)$ were determined with a linear least-squares program resulting in the following parameters:

$a'=14{\cdot}50$ Å	$\alpha'=123{\cdot}4°$
$b'=6{\cdot}37$	$\beta'=42{\cdot}7$
$c'=9{\cdot}83$	$\gamma'=121{\cdot}3$.

The Delaunay reduced cell (*International Tables for X-ray Crystallography*, 1952) was obtained by the transformation $1\ 0\ \bar{1}/0\ 1\ 1/0\ 0\ \bar{1}$ resulting in a unit cell with

$a=9{\cdot}87$ Å	$\alpha=139{\cdot}9°$
$b=8{\cdot}27$	$\beta=94{\cdot}8$
$c=9{\cdot}83$	$\gamma=93{\cdot}8$.

This cell is identical with the Bravais reduced cell (Kennard, Speakman & Donnay, 1967). The least-squares program produces estimated standard devia-

tions in six quantities, a^{*2}, b^{*2}, $a^*b^* \cos \gamma^*$, *etc.* However, the repeat distances and angles of the real lattice are functions of the corresponding quantities in reciprocal space. For this reason it was impossible to calculate standard deviations with this program in the linear version.

Assuming a close-packed array of atoms, a chlorine atom with a radius of 1·8 Å occupies a volume of 33·05 Å^3. Accordingly, the maximum number of chlorine atoms in the unit cell of $AlSeCl_7$ cannot be larger than $V/33{\cdot}05 = 15{\cdot}2$. There are clearly two units of $AlSeCl_7$ in the chosen cell. With $Z=2$ the calculated density is 2·33 g.cm^{-3}.

Non-integrated equi-inclination Weissenberg photographs (multiple-film technique) of the layers $l=0, -1, \ldots -5$ and zero-layer photographs about [021] and [012] were taken at room temperature with Zr-filtered Mo radiation ($\lambda = 0{\cdot}7107$ Å). The intensities of 1381 reflexions were estimated visually and reduced to structure factor moduli in the usual way. An absorption correction was applied assuming a cylindrical cross-section for the crystals used [μ(Mo $K\alpha$) = 57 cm^{-1}]. The structure factors of the various levels were put on a single scale by hand.

Determination of the structure and refinement

As a first step an unsharpened Patterson function was calculated which contained a large extended peak at a distance of 6·8 Å from the origin. It was anticipated that this peak, with a height about 0·4 times the value of the origin peak, should be attributed to several interactions of the kind Se–Se and Cl–Cl. The next class of peaks with heights about 0·1 times the value of the origin peak could be explained as being interactions of the mixed type Se–Cl. However, it was not possible to find a proper solution on the basis of the centric space group $P\bar{1}$.

The $hk0$ reflexions were subjected to the $N(z)$ test of Howells, Phillips & Rogers (1950). The computer-calculated and plotted curve (Fig. 1) clearly indicates that the structure is acentric and the space group is $P1$.

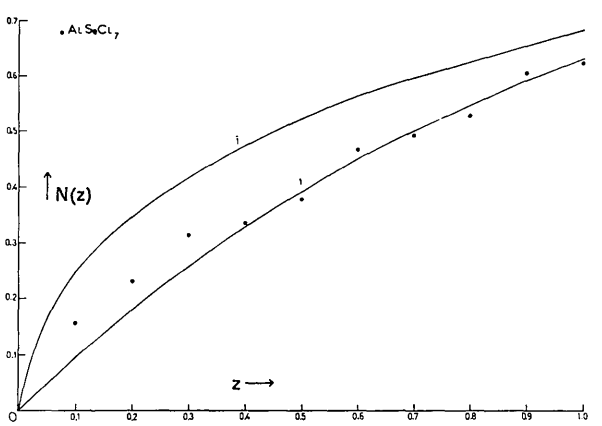


Fig. 1. The intensity distribution of reflexions $hk0$ in $AlSeCl_7$.

Taking into account the ionic radii of Se^{4+} (0·5 Å) and of Cl^- (1·8 Å) we arrive at a shortest Se–Cl distance of approximately 2·3 Å. A careful inspection of spheres with a radius of 2·3 Å about the origin and about the next-largest peak indicates that there are two candidates for single Se–Cl vectors representing a possible image of a part of the structure. A minimum function (Buerger, 1959) was therefore calculated by shifting over these two vectors. A model on spokes was constructed from the 31 highest peaks of this superposition function. We could distinguish two octahedra of chlorine atoms, the centres of which are separated by a vector of 6·8 Å (see Fig. 2) corresponding with the highest peak in the original Patterson function. The two octahedra are more or less oriented in the same direction, explaining why many overlapping interactions contribute to the predominant peak. At this stage two selenium and twelve chlorine atoms were located.

The positions of these atoms were refined with a least-squares method (block-diagonal approximation). Dirac–Slater scattering factors calculated by Cromer & Waber (1965) were used for selenium and chlorine and afterwards also for aluminum atoms. This model refined to a conventional R index of 36·6 % and resulted in a three-dimensional Fourier synthesis in which the two remaining chlorine atoms were found. It was now possible to distinguish more or less close-packed layers of halogen atoms.

Since the refinement with the block-diagonal approximation was slow, we shifted to a full-matrix refinement in which Se(1) was fixed at the origin. The justification for the application of this procedure to an acentric structure was the presence of strong interactions of the kind $\frac{\delta F}{\delta x_i} \cdot \frac{\delta F}{\delta x_j}, \frac{\delta F}{\delta x_i} \cdot \frac{\delta F}{\delta y_j}$, *etc.* contributing to the matrix of normal equations (Srinivasan, 1961). For example, we afterwards observed interactions $\frac{\delta F}{\delta x_3} \cdot \frac{\delta F}{\delta x_7}, \frac{\delta F}{\delta y_3} \cdot \frac{\delta F}{\delta z_7}$ [Cl(3) and Cl(7)] amounting to $-0{\cdot}28 \times 10^3$ and $-0{\cdot}18 \times 10^3$. The elements $\left(\frac{\delta F}{\delta x_3}\right)^2$ and $\left(\frac{\delta F}{\delta x_7}\right)^2$ were $0{\cdot}38 \times 10^4$ and $0{\cdot}36 \times 10^4$ respectively.

After four cycles with an overall temperature factor the reliability index dropped to 26·2 %. Attempts to locate the aluminum atoms failed at this stage. The refinement was continued with individual isotropic B values and the R index decreased to 21·7 %. It was decided to calculate a difference Fourier synthesis; the aluminum atoms could then be distinguished. After three more cycles with isotropic B values the agreement index dropped to 16·1 %. Up to this stage unitary weights were used. Analysis of the residuals for different groupings of F values suggested the weighting scheme $w = 1/a + F_{obs} + bF_{obs}^2$ with $a=2{\cdot}0$ and $b=0{\cdot}0755$; for $F_{obs} \leq 6{\cdot}0$ the value $F_{obs} = 6{\cdot}0$ was used. The smallest estimated intensity value (before reduction) was 1·0. The non-observed reflexions were given this intensity

values resulting in slightly varying $F=(1\times(\mathrm{Lp})^{-1}\times$ absorption correction)$^{1/2}$ after reduction. Only if $F_{calc}>F$ were these reflexions included in the refinement with an F_{obs} value of $0{\cdot}7\times F$ and a constant weight of 0·05. The refinement with isotropic B values was continued over 4 cycles and R decreased to 12·3% (observed reflexions only). The largest errors, probably due to inefficient absorption correction and/or extinction effects, were observed for the 14 strongest reflexions (Table 1). These reflexions were discarded during the following calculations. A difference electron density map was computed, which gave a clear indication of the anisotropic thermal motion for nearly all the atoms. Since most difficulty was encountered in the location of the aluminum atom we checked that no disordering of these atoms was present in this map.

Table 1. *Discarded structure factors*

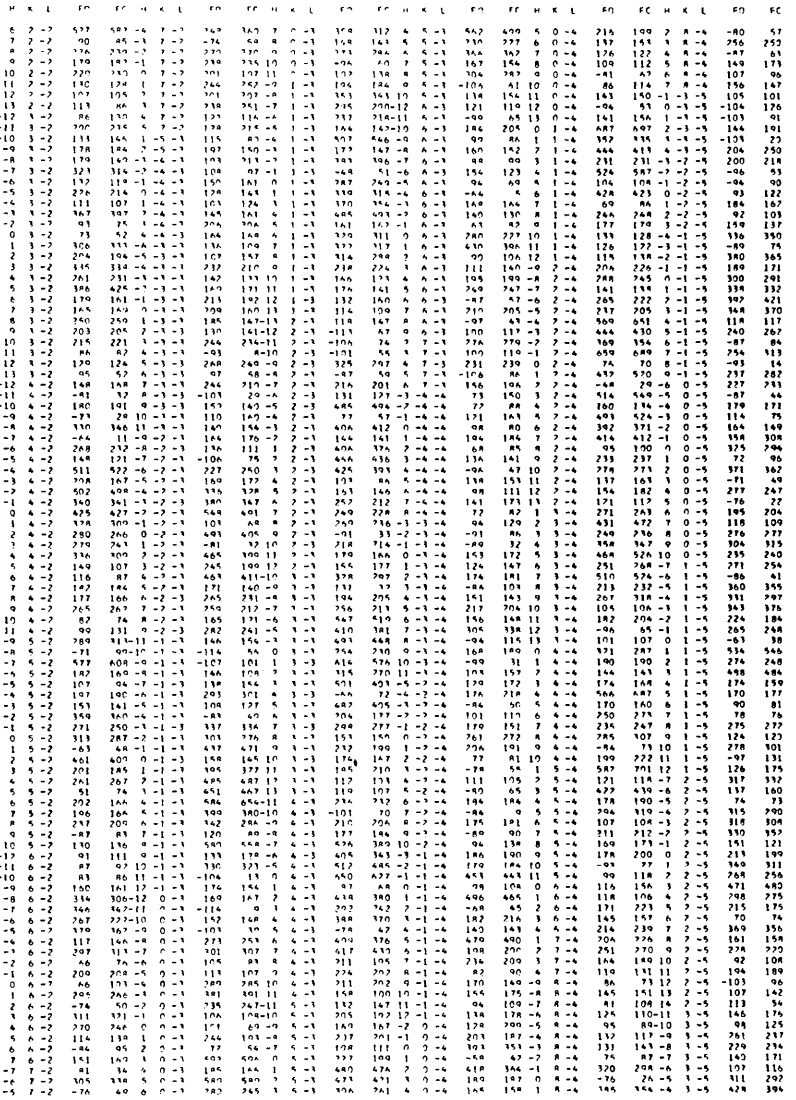
h	k	l	F_{obs}	F_{calc}
2	0	0	83·7	102·4
−3	1	0	110·8	109·4
−6	2	0	114·2	96·7
3	−2	−1	107·8	153·4
0	−1	−1	76·1	107·8
−3	0	−1	110·1	169·4
2	3	−1	98·5	139·3
5	1	−2	125·4	147·5
2	2	−2	92·2	133·0
−1	3	−2	162·4	169·7
2	1	−3	139·1	162·5
−4	3	−3	118·0	146·7
4	3	−5	88·1	71·9
1	4	−5	106·5	122·6

The final anisotropic refinement was carried out over two cycles. The matrix to the normal equations was

Table 2. *Calculated and observed structure factors on an absolute scale* (×10).

The non-observed reflexions are indicated by a minus sign.

Table 2 (*cont.*)



divided into 20 blocks: one 54 × 54 block for the positional parameters of all atoms and eighteen 6 × 6 blocks for the individual thermal parameters and one 2 × 2 block for the scale and overall B factor. This anisotropic refinement hardly affected the positional parameters but significantly decreased (~20%) the standard de-

viations. The final R value is 10·9% (including all reflexions) and 10·1% (excluding non-observed reflexions).

The structure factors are listed in Table 2, the positional parameters and their standard deviations in Table 3, and the vibrational parameters U_{ij} in Table 4.

Structures with space group $P1$ are quite rare and have three degrees of freedom, *i.e.* one atom can be fixed at will in the unit cell on $x_0y_0z_0$. As a result of the least-squares refinement this atom has zero standard deviations in its positional parameters if one refines with a full matrix. This situation is less paradoxical if one remembers that the reflexion intensities are functions of distances and not of positions. The standard deviations in the distance are independent of the choice of origin.

Discussion of the structure

A projection of the structure along the direction **a*** on (100) is presented in Fig. 2. The average values of machine-calculated standard deviations are 0·013 Å for Se–Cl, 0·016 Å for Al–Cl and 0·018 Å for Cl–Cl distances, the influence of errors in the unit-cell parameters being negligible. However, no estimate of systematic errors is incorporated in these standard deviations.

A careful study of adjoining unit cells (not shown in Fig. 2) reveals that the chlorine atoms are stacked in cubic close-packed layers, and suggests a partial ionic character for the structure. The selenium atoms are coordinated octahedrally by six chlorine atoms. The octahedra are distorted in such a way (Table 5) that there are three short Se–Cl distances (average value ~2·1 Å) and three large Se–Cl distances (average value ~3·0 Å). The symmetry is approximately C_{3v}. The selenium atom is located on the line connecting the centres of gravity of the two kinds of chlorine atoms (Fig. 3). The interatomic Cl–Cl distances are approximately 3·2 Å in the small triangle and approximately 4·0 Å in the large triangle of chlorine atoms.

Each Al atom is surrounded tetrahedrally by 4 Cl atoms at distances of 2·1 Å. These tetrahedra are quite regular (Table 5). The atoms Cl(13) and Cl(14) are only coordinated to Al, the atoms Cl(1), Cl(4), Cl(5), Cl(7), Cl(8) and Cl(11) only to Se; Cl(2), Cl(3), Cl(6), Cl(9),

Table 3. *Atomic parameters (in fractions of cell edges) and their standard deviations* (Å)

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
Se(1)	0·0	0·0	0·0	0·0	0·0	0·0
Se(2)	0·4763	0·0037	0·3436	0·0060	0·7871	0·0076
Cl(1)	0·0480	0·0131	0·6668	0·0187	0·8784	0·0236
Cl(2)	0·1320	0·0107	0·8490	0·0174	0·6557	0·0194
Cl(3)	0·9963	0·0129	0·4714	0·0170	0·1280	0·0192
Cl(4)	0·9270	0·0119	0·1580	0·0201	0·2658	0·0232
Cl(5)	0·8059	0·0119	0·8348	0·0214	0·7848	0·0257
Cl(6)	0·2965	0·0097	0·3041	0·0165	0·2878	0·0206
Cl(7)	0·4905	0·0116	0·9846	0·0160	0·6428	0·0222
Cl(8)	0·3671	0·0115	0·4890	0·0188	0·0278	0·0226
Cl(9)	0·5768	0·0108	0·1601	0·0174	0·4109	0·0194
Cl(10)	0·4418	0·0121	0·7835	0·0146	0·8794	0·0173
Cl(11)	0·6807	0·0098	0·5491	0·0165	0·9802	0·0195
Cl(12)	0·2040	0·0097	0·0746	0·0194	0·4805	0·0233
Cl(13)	0·8713	0·0097	0·0237	0·0160	0·5443	0·0212
Cl(14)	0·6278	0·0109	0·4212	0·0179	0·2595	0·0218
Al(1)	0·0460	0·0095	0·8565	0·0162	0·4558	0·0200
Al(2)	0·4838	0·0104	0·1670	0·0168	0·2080	0·0205

Table 4. *Vibrational parameters* U_{ij} (Å²) *in the temperature factor* $\exp[-2\pi^2\sum_{ij}U_{ij}a_i^*a_j^*h_ih_j]$

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
Se(1)	0·0200	0·0206	0·0228	0·0051	0·0334	0·0058
Se(2)	0·0209	0·0182	0·0274	0·0047	0·0315	0·0071
Cl(1)	0·0635	0·0372	0·0659	0·0330	0·0697	0·0277
Cl(2)	0·0450	0·0508	0·0316	0·0411	0·0692	0·0369
Cl(3)	0·0677	0·0322	0·0199	0·0345	0·0265	0·0016
Cl(4)	0·0494	0·0570	0·0553	0·0413	0·0799	0·0515
Cl(5)	0·0332	0·0592	0·0710	−0·0069	0·0901	−0·0111
Cl(6)	0·0221	0·0356	0·0472	0·0172	0·0511	0·0129
Cl(7)	0·0505	0·0283	0·0634	0·0232	0·0575	0·0193
Cl(8)	0·0403	0·0529	0·0589	0·0240	0·0768	0·0392
Cl(9)	0·0419	0·0446	0·0331	0·0429	0·0568	0·0276
Cl(10)	0·0652	0·0258	0·0127	0·0129	0·0206	0·0002
Cl(11)	0·0284	0·0394	0·0394	−0·0085	0·0601	−0·0124
Cl(12)	0·0253	0·0617	0·0658	−0·0270	0·1097	−0·0150
Cl(13)	0·0309	0·0325	0·0598	0·0337	0·0576	0·0278
Cl(14)	0·0385	0·0435	0·0552	−0·0005	0·0813	0·0073
Al(1)	0·0199	0·0245	0·0373	0·0102	0·0462	−0·0021
Al(2)	0·0238	0·0272	0·0282	0·0153	0·0436	0·0164

Cl(10) and Cl(12) are shared by tetrahedra and octahedra. It can be seen that the shared chlorine atoms are at larger distances from the selenium atoms.

In view of the observed coordination and distances we may conclude that the structure is composed of $AlCl_4^-$ ions and $SeCl_3^+$ ions, in agreement with the do-

Table 5. *Bond lengths* (Å) *and decimal angles in* $AlSeCl_7$

Se(1) octahedron		Se(2) octahedron	
Se(1)–Cl(1)	2·11	Se(2)—Cl(7)	2·12
–Cl(2)	3·03	—Cl(8)	2·11
–Cl(3)	3·05	—Cl(9)	3·03
–Cl(4)	2·07	—Cl(10)	3·05
–Cl(5)	2·13	—Cl(11)	2·13
–Cl(6)	3·11	—Cl(12)	2·97
Cl(1)–Se(1)–Cl(2)	86·7	Cl(7)—Se(2)–Cl(8)	100·0
–Cl(3)	165·0	–Cl(9)	88·1
–Cl(4)	99·3	–Cl(10)	165·8
–Cl(5)	99·4	–Cl(11)	98·9
–Cl(6)	90·2	–Cl(12)	85·9
Cl(2)–Se(1)–Cl(3)	79·8	Cl(8)—Se(2)–Cl(9)	166·3
–Cl(4)	171·4	–Cl(10)	90·9
–Cl(5)	85·9	–Cl(11)	99·3
–Cl(6)	84·8	–Cl(12)	88·1
Cl(3)–Se(1)–Cl(4)	93·3	Cl(9)—Se(2)–Cl(10)	79·5
–Cl(5)	86·6	–Cl(11)	90·3
–Cl(6)	81·8	–Cl(12)	81·4
Cl(4)–Se(1)–Cl(5)	99·1	Cl(10)–Se(2)–Cl(11)	88·2
–Cl(6)	89·0	–Cl(12)	85·4
Cl(5)–Se(1)–Cl(6)	166·3	Cl(11)–Se(2)–Cl(12)	170·2
Al(1) tetrahedron		Al(2) tetrahedron	
Al(1)—Cl(2)	2·12	Al(2)—Cl(6)	2·12
—Cl(3)	2·14	—Cl(9)	2·17
—Cl(12)	2·15	—Cl(10)	2·14
—Cl(13)	2·09	—Cl(14)	2·13
Cl(2)—Al(1)–Cl(3)	108·3	Cl(6)—Al(2)–Cl(9)	112·0
–Cl(12)	107·5	–Cl(10)	109·2
–Cl(13)	113·1	–Cl(14)	108·3
Cl(3)—Al(1)–Cl(12)	108·2	Cl(9)—Al(2)–Cl(10)	107·9
–Cl(13)	110·6	–Cl(14)	107·5
Cl(12)–Al(1)–Cl(13)	109·0	Cl(10)–Al(2)–Cl(14)	112·0

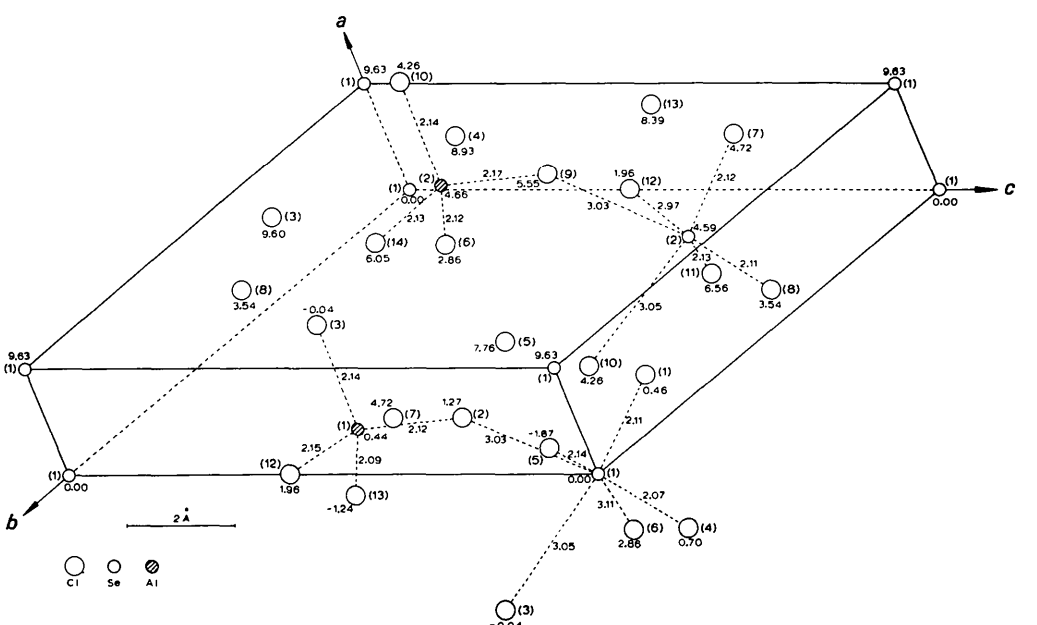


Fig. 2. Projection of the structure of $AlSeCl_7$ along the direction **a*** on a plane parallel to (100). The heights (Å) of the atoms above this plane are indicated. The numbers in brackets indicate the atoms. The remaining numbers (Å) refer to bond lengths.

nating properties of $SeCl_4$ (Groeneveld, 1953). The observed Al–Cl distances (average 2·13 Å) in this structure agree very well with those (~2·14 Å) found in $AlCl_4^-$ ions in the compound $Mg(CH_3CN)_6(AlCl_4)_2$ (Stork-Blaisse & Romers, 1971). Al is coordinated octahedrally in $AlCl_3$ and accordingly the Al–Cl distances are larger (~2·31 Å) (Ketelaar *et al.* 1947).

All calculations were performed on the IBM 360/50 computer with programs developed by Mrs E. W. M. Rutten (Fourier and least-squares) and by Mr R. A. G. de Graaff (geometry). The authors thank Dr Groeneveld for his supply of crystals. They are indebted to Mr R. Ch. D. E. Hasekamp and Mr J. C. Portheine for the production of the diffraction photographs.

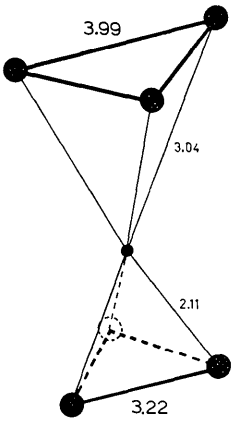


Fig. 3. Idealized deformed octahedron with C_{3v} symmetry. The distances shown (Å) are mean values of the two octahedra.

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The Crystal and Molecular Structure of a Sulphur-Containing Antibiotic, 'vD844', 5-Oxo-6-*N*-methylformylamino-4,5-dihydro-1,2-dithiolo[4,3-*b*] pyrrole

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The antibiotic 5-oxo-6-*N*-methylformylamino-4,5-dihydro-1,2-dithiolo[4,3-*b*]pyrrole, $C_7H_6N_2O_2S_2$, crystallizes in the monoclinic space group $P2_1/c$ with four molecules in a unit cell of dimensions $a=14{\cdot}22$, $b=3{\cdot}788$, $c=19{\cdot}06$ Å; $\beta=116{\cdot}4°$. The unit cell also contains about 3 molecules of water. The structure was solved from two- and three-dimensional Patterson syntheses and refined by the full-matrix least-squares method, producing a final R value of 0·105. It shows some disorder, caused partly by the water molecules and partly by the presence of two different conformations of the vD844 molecule in the crystal. The ring system is approximately planar and the side chain is twisted about 42° out of this plane, the angle being approximately the same in both of the conformations of the vD844 molecule. N–H---O hydrogen bonds of length 2·83 Å connect the vD844 molecules in pairs. The positions of the water molecules are not well defined; the molecular packing is very compact and leaves sufficient space for only about three of any four equivalent positions to be occupied. The average distance found between the oxygen atoms of two water molecules is 2·34 Å.

Introduction

A compound with anti-bacterial activity, and called vD844, has been separated from the culture fluid of an unidentified *Streptomyces* species isolated from a soil sample collected near Copenhagen. It is strongly active against *Neisseria* and certain other gram-negative organisms, but it is rather toxic. Chemical investiga-