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Structural Investigations on Sulfosalts from the Lengenbach, Binn Valley (Ct. Wallis). Part 2¹⁾

By W. Nowacki, Y. Iitaka, H. Bürki and V. Kunz (Bern)²⁾

With 3 figures in the text

Summary

There are two sorts of X-ray reflections of scleroclase: "strong" and "weak" ones. The strong reflections are arranged orthorhombic with $a_0' = 19,62 \pm 0,02$, $b_0' = 7,89 \pm 0,01$, $c_0' = 4,19 \pm 0,005$ Å (pseudo-cell) and space group $C_{2h}^5 - P2_1/n$, $D_{2h}^{16} - Pbnm$ or $C_{2v}^9 - Pbn2$; the weak ones show monoclinic symmetry (all inter-axial angles being 90°). As no piezoelectricity was observed and in view of the monoclinic symmetry of the true cell, we assume the space group $C_{2h}^5 - P2_1/n$ for the pseudo-cell. The true cell has the dimensions $a_0 = 3a_0' = 58,9$, $b_0 = b_0' = 7,89$, $c_0 = 11c_0' = 46,1$ Å. The complete pseudo-structure was determined (atomic coordinates see Table 2). The Pb-atoms are coordinated by $8+1=9$ S-atoms, the As-atoms by $3+2=5$ S-atoms. The structure may be described as built up by two parts: a layer like region of Pb and S // to the (100) plane and a region filled by the As-S-chains // c' . — In the true structure small deviations from the atomic positions of the pseudo structure are present. Contrary to most sulfosalts $xPbS \cdot yAs_2S_3$, scleroclase is not striated and elongated // to the chain directions (i. e. // c'), but // to the b -direction.

In the Lengenbach dolomite fine needles of a gray, sometimes reddish sulfosalt occur. Each needle consists of two species: an orthorhombic one ($a_0 = 8,38$, $b_0 = 2 \cdot 25,61$, $c_0 = 7,89$ Å) and dufrenoyite ($a_0 = 8,38$, $b_0 = 25,61$, $c_0 = 7,89$ Å, $\beta_0 = 90^\circ 25'$) in two orientations.

For Lengenbachite: $a = 35,1_3$, $b = 11,5_2$, $c = 36,9_0$ Å, $\beta_0 = 92^\circ 36'$; $C_{2h}^2 = P2_1/m$.

Hatchite has the lattice constants $a_0 = 9,27 \pm 0,09$, $b_0 = 7,81 \pm 0,08$, $c_0 = 8,01 \pm 0,07$ Å, $\alpha_0 = 66^\circ 37'$, $\beta_0 = 63^\circ 27'$, $\gamma_0 = 85^\circ 06'$ (all $\pm 14'$), $a_0 : b_0 : c_0 = 1,187 : 1 : 1,026$; space group $C_i^1 - P\bar{1}$ or $C_1^1 - P1$.

Trechmannite: a_0 (rho.) = $8,65 \pm 0,01$ Å, $\alpha_0 = 108^\circ 17' \pm 17'$, a_0 (hex.) = $14,02$, c_0 (hex.) = $9,15$ Å, $c_0/a_0 = 0,653$; space group $C_{3i}^2 - R\bar{3}$ (or $C_3^4 - R3$).

¹⁾ Part 1: W. NOWACKI und V. KUNZ, Untersuchungen an Sulfosalzen. 1. Pulverdiagramme einiger Sulfosalze aus dem Lengenbach (Binntal). *Chimia* 13 (1959), 294—297.

²⁾ Contribution No. 114b from the Abt. für Kristallographie, Mineralogisches Institut, Universität Bern.

A. The pseudo-structure of scleroclase (Y. I.)

1. Introduction

Scleroclase (of WALTERSHAUSEN, 1855, see SOLLY 1900) or sartorite (of DANA) is one of the commonest of the group of sufoarsenites of lead which have been formed in the well-known dolomite at the Lengenbach, in the Binnatal (Ct. Wallis). Notwithstanding this abundance of material, complicated morphology of the mineral has given rise to much confusion. Some historical notes of the investigation on this mineral will be briefly summarized.

In 1864, vom RATH first distinguished three distinct species among the complexly intergrown metallic minerals from Binnatal. He named them dufrenoyite, scleroclase and jordanite. Under the name scleroclase he described small, needle-shaped, prismatic crystals which were heavily striated and grooved parallel to their length. He regarded the symmetry as orthorhombic and remarked that this mineral was the same as HEUSSER's binnite and von WALTERSHAUSEN's scleroclase (RATH, 1864). Later, DANA proposed the name sartorite for the mineral by taking the name of SARTORIUS von WALTERSHAUSEN. In 1895, BAUMHAUER described the crystal as orthorhombic by adopting the vom RATH's elements. One specimen, however, displayed several new pyramidal forms, for which very complicated indices were attached. He pointed out a considerable diversity between the forms given by vom RATH and by him, especially among the macrodomes (BAUMHAUER, 1895). On the other hand, SOLLY (1903) described scleroclase as monoclinic while TRECHMANN (1906) stated that SOLLY's elements were not able to explain his own goniometric measurements, and he proposed other monoclinic parameters. TRECHMANN also noted a considerable diversity between the forms observed in his specimens.

In 1919, SMITH and SOLLY again investigated scleroclase extensively including TRECHMANN's crystals and they arrived at the conclusion that the crystals are built up by three crystal lattices, one being monoclinic and the two others triclinic. They also proposed the name sartorite- α for TRECHMANN's crystals which exhibited some discordance with other crystals.

The main morphological difficulties are:

1. Chemical analysis gives always nearly the same composition i. e. $\text{PbS} \cdot \text{As}_2\text{S}_3$.
2. Some faces have almost "irrational" indices when referred to a simple lattice. The crystal system was described as orthorhombic or monoclinic or even triclinic in order to fit better with the observed angles.
3. Diversity between the forms appeared in different crystals and also between the measured angles for different crystals.

It is very interesting that as TRECHMANN has already pointed out these difficulties are always observed among the planes which make large angles to the *b*-axis (of our notation).

An X-ray investigation on scleroclase was first made by BANNISTER, PABST and VAUX (1939). By taking X-ray rotation photographs they recognized many

weak reflections which form a 3×20 greater monoclinic true-cell compared with the smaller orthorhombic pseudo-cell formed by the strong reflections. They examined several crystals by X-ray rotation method, including TRECHMANN's specimens which were named sartorite- α , but they could not find neither a difference between these specimens nor X-ray evidence of twinning in scleroclase. They also tried to explain the complicated morphology of this mineral by reference to its pseudo- and true-crystal lattice, and found that even those faces which have almost "irrational" indices when referred to the pseudo-lattice, can be given simpler indices by adopting the true-lattice.

We now have investigated the crystal structure of scleroclase by X-ray methods and also recognize a super-structure which form a 3×11 times greater cell as compared with the sub-cell.

2. Experimental

One of the crystals from the Lengenbach was used for the X-ray investigation. Small fragments of the specimen were ground into spheres by BOND's method (BOND, 1951) so that the correction for X-ray absorption can be easily applied. We got several spherical specimens of which two specimens with the diameter 0,15 mm and 0,20 mm were used for the c -axis and the b -axis integrated Weissenberg photographs with Cu-radiation. The linear absorption coefficient of the crystal for CuK_α -radiation is $\mu = 768 \text{ cm}^{-1}$ (this means that a plate 0,1 mm thick will reduce the intensity 1 to $e^{-7,68} = 0,0005!$). With spherical crystals it is also easier to compare the intensities of symmetrical equivalent reflections. The intensities of nearly 800 three-dimensional reflections were measured from the integrated Weissenberg diagrams with the help of a photometer (Joyce-Loebl-double beam automatic recording Microdensitometer Mark III). Beside these photographs, oscillation, Weissenberg and precession photographs around the three crystallographic axes were taken.

Absorption correction factors for the spherical crystals were referred to the International Tables for X-ray Crystallography vol. II, p. 302 (1959). The factors indeed were ranged between 600 at $\theta = 0^\circ$ and 20,6 at $\theta = 90^\circ$.

3. Superlattice reflections

The X-ray photographs showed the following peculiar features:

1. Beside the well-marked "strong" reflections there are many "weak" superlattice reflections. The "strong" reflections are arranged orthorhombic with cell dimensions $a' = 19,62 \pm 0,02$, $b' = 7,89 \pm 0,01$, $c' = 4,19 \pm 0,005 \text{ \AA}$ (pseudo-cell), while the "weak" reflections show

monoclinic symmetry. All interaxial angles are, however, exactly 90° . These observations are in agreement with that of BANNISTER et al. (1939) ($a_0' = 19.46$, $b_0' = 7.79$, $c_0' = 4.17$ Å, space group $D_2^4 - P2_12_12_1$; true cell $a_0 = 3a_0'$, $b_0 = b_0'$, $c_0 = 20c_0'$). However, missing spectra found for the "strong" reflections are different, namely in the $(h0l)$ planes only the reflections having the indices $h + l = 2n$, and in the $(0kl)$ and $(0k0)$ planes only $k = 2n$ were observed. This leads to the space groups $C_{2v}^9 - Pbn2$, $D_{2h}^{16} - Pbnm$ or $C_{2h}^5 - P2_1/n$ instead of $D_2^4 - P2_12_12_1$ reported by them. As no piezoelectric effect could be observed, we assume the structure to be centrosymmetrical ($P2_1/n$ or $Pbnm$).

2. There are two intermediate layers of the "weak" reflections between the rows of constant h in the pseudo-cell and ten layers between the rows of constant l . Thus the dimensions of the true-cell are $a = 3 \times 19.62 = 58.9$, $b = 7.89$, $c = 11 \times 4.19 = 46.1$ Å.
3. The intensity distribution for the pseudo-cell is almost exactly orthorhombic. Any serious intensity difference between a set of (hkl) , $(\bar{h}kl)$ and $(h\bar{k}l)$ planes could not be observed. On the other hand, the intensity distribution for the true-cell is monoclinic and the details of it among weaker reflections look like different for different specimens.
4. As has already been reported by BANNISTER et al. (1939), there is no X-ray evidence for morphological twinning. This is in contrast to the older morphological statements.

4. Space group considerations

Laue photographs taken with incident Mo-radiation parallel to the a -axis also exhibit orthorhombic symmetry for the "strong" reflections. For the "weak" superlattice reflections, however, only the (010) symmetry plane is demonstrated, which indicates monoclinic symmetry. Together with the fact that no piezoelectric effect could be observed for the crystal and no regularity between the indices of missing spectra could be found for the "weak" reflections, we may choose the space group of the true-cell of scleroclase either as $C_{2h}^3 - P2_1/m$ or $C_{2h}^1 - P2/m$ if there is no extinction in the $(0k0)$ planes.

For the pseudo-cell, X-ray photographs indicate the orthorhombic space groups $C_{2v}^9 - Pbn2$ or $D_{2h}^{16} - Pbnm$, general diffraction unit no. 12d according to NOWACKI (1952). ($h0l$ only with $h + l = 2n$, $0kl$ with $k = 2n$, $h00$ with $h = 2n$, $0k0$ with $k = 2n$, $00l$ with $l = 2n$ present.) A "general

diffraction unit" is defined as the totality of all space groups having the same set of missing spectra regardless of the Laue symmetry; in the paper a determinative table of all 62 general diffraction units is given, based only on missing spectra. But as has been turned out by the recent structure determination of the pseudo-cell, such an orthorhombic symmetry is really caused by a special arrangement of the atoms in the crystal. That is all atoms are situated with the z parameters of $\frac{1}{4}$ or $\frac{3}{4}$ just on the mirror planes.

In view of the monoclinic symmetry of the true-cell, we take the space group $C_{2h}^5 - P2_1/n$ for the pseudo-cell by making allowances for the non-symmetric electron density distribution over the mirror planes (001).

There are 4 formula units of PbAs_2S_4 contained in a pseudo-cell (3,96 if we take the observed density 5,05 gr/cm³), while in a true-cell 132 formula units are involved.

5. Determination of the pseudo-structure

After correcting for absorption and Lorentz-polarization factors the structure factors were placed on an absolute basis by WILSON's method. The temperature factors determined by this method were surprisingly large, namely $B = 4,8 \text{ \AA}^2$ for the $(hk0)$ and $B = 4,2 \text{ \AA}^2$ for the $(h0l)$ planes. We can hardly think that these values indicate violent thermal vibrations of the atoms in the crystal, but it might be caused by a certain kind of disorder.

To find the position of the heavy atoms a Patterson projection along the c -axis was calculated. An analysis of the Patterson projection gave the x - and y -parameters of the lead atom which yielded most of the signs of the structure amplitudes. Successive refinement by the c -axis Fourier projections made it possible to locate all of the lighter atoms, arsenic and sulfur, and the reliability factor became 26% which was further dropped to 22% when anisotropy for the lead atom was taking into account.

For the $(h0l)$ projection, the orthorhombic intensity distribution of the "strong" reflections limits the z -parameters of the atoms in the pseudo-cell to either of the following values, 0, $\frac{1}{4}$, $\frac{2}{4}$, $\frac{3}{4}$. A crystal chemical consideration led to a plausible arrangement of the atoms which gave the reliability factor 29%. Several repeated cycles of refinement by Fourier and difference Fourier method dropped this factor down to 16%

if an anisotropic temperature factor for the lead atom was taken into account.

For both ($hk0$) and ($h0l$) projections, the temperature factor was surprisingly large. Moreover, the difference Fourier maps demonstrated still a clear indication of anisotropy in electron distribution around the arsenic and sulfur atoms. At the present stage, the individual temperature factors and the corresponding root mean square displacement of the thermal vibrations are as follows.

Table 1. *Individual temperature factors and their corresponding root mean square displacement*

	$// a$ $B (\text{\AA})$ r. m. s. Displacements (\AA)		$// b$ $B (\text{\AA})$ r. m. s. \AA .		$// c$ $B (\text{\AA})$ r. m. s. \AA .	
Pb	6,7	0,29	4,5	0,24	3,6	0,22
As ₁	7,4	0,31	3,5	0,21	7,4	0,31
As ₂	7,4	0,31	4,5	0,24	7,4	0,31
S	4,5	0,24	3,0	0,20	4,5	0,24

If we adopt these values, the agreement between the observed and calculated structure factors over all nearly 800 independent three-dimensional reflections becomes rather satisfactory and the reliability factor of 21% is obtained.

It should be noticed, as described earlier, that the statistical treatment of the intensities had also given a large overall temperature factor. As the structure so far discussed is the average structure over the 11×3 sub-cells, we may dare to suppose that at least most part of the temperature factors derived from the pseudo-cell have no real meaning, but are indicating statistical displacement of the atoms from their mean positions.

Table 2. *The atomic coordinates*

	x'	y'	z'
Pb	0,1947	0,0849	0,2500
As ₁	0,1290	0,5030	0,7500
As ₂	0,0040	0,8020	0,2500
S ₁	0,2210	0,3630	0,7500
S ₂	0,1680	0,6810	0,2500
S ₃	0,0780	0,9750	0,7500
S ₄	0,0480	0,3410	0,2500

6. Discussion of the pseudo-structure

Some of the interatomic distances calculated from the coordinates given in Table 2 are listed in Table 3. These values agree well with the values found in the previously determined structures of sulfosalts, although some of them are slightly shorter or longer than usual. The coordination around the lead atom is somewhat different from other sulfosalts in which lead atoms are normally surrounded by six to eight neighbouring sulfur atoms. In scleroclase, the lead atom is surrounded by nine nearest sulfur atoms although one of them is situated at a longer distance of 3,52 Å from the lead. This kind of coordination is, however, really formed in the structure of halides of lead such as PbCl_2 and PbBr_2 .

Table 3. *Coordination for Pb and As₁, As₂*

Pb:	2,80 (S ₂) + 3,08 (2 S ₁) + 3,19 (2 S ₁) + 3,22 (2 S ₃) + 3,23 (S ₂) + 3,52 (S ₄) = 8 + 1
As ₁ :	2,12 (S ₁) + 2,63 (2 S ₂) + 2,92 (2 S ₄) = 3 + 2
As ₂ :	2,38 (S ₃) + 2,59 (2 S ₄) + 2,89 (2 S ₃) = 3 + 2

The coordination around the arsenic atoms are also listed in Table 3. There are five immediate neighbours of sulfur atoms around the arsenic atoms, of which three are situated at distances of about 2,5 Å. The arsenic and sulfur atoms thus form an AsS_3 -group of trigonal-pyramidal shape.

The structure projected along the *c* and *b* axis is shown in Fig. 1. It may be described as if it is composed of two parts. One of them is the layer like region of the lead and sulfur atoms extended parallel to the (100) plane, and the other part is the region filled by the chains of arsenic and sulfur atoms. The reported good cleavage parallel to the (100) plane of scleroclase can be explained as the result of the structural nature described above. There are two crystallographically independent arsenic atoms in a pseudo-cell, each constituting two kinds of $(\text{AsS}_2)_n$ chains parallel to the *c*-axis by forming trigonal-pyramids of AsS_3 and by sharing their corners. These chains offer their sulfur atoms around the lead atoms. In view of the fact that the present pseudo-structure is the average structure over the 33 sub-cells of the true structure, it is not certain whether these chains are extended infinitely along the *c*-axis, forming strong bonds between the arsenic and sulfur atoms. If account is taken of the three structural features described below, it might be found that the chains are broken into pieces which have (at the present stage) unknown length.

1. The length of the *c*-axis is too large to assume that the chains are continued throughout the crystal. As a fact, some rather longer bond distances between the arsenic and sulfur atoms are observed in the average structure (such as 2,63 Å and 2,59 Å).
2. In contrast to most of the sulfosalts in which the elongation and the striation are both directed along the direction of the chains, which may be regarded as the direction of the strongest binding forces,

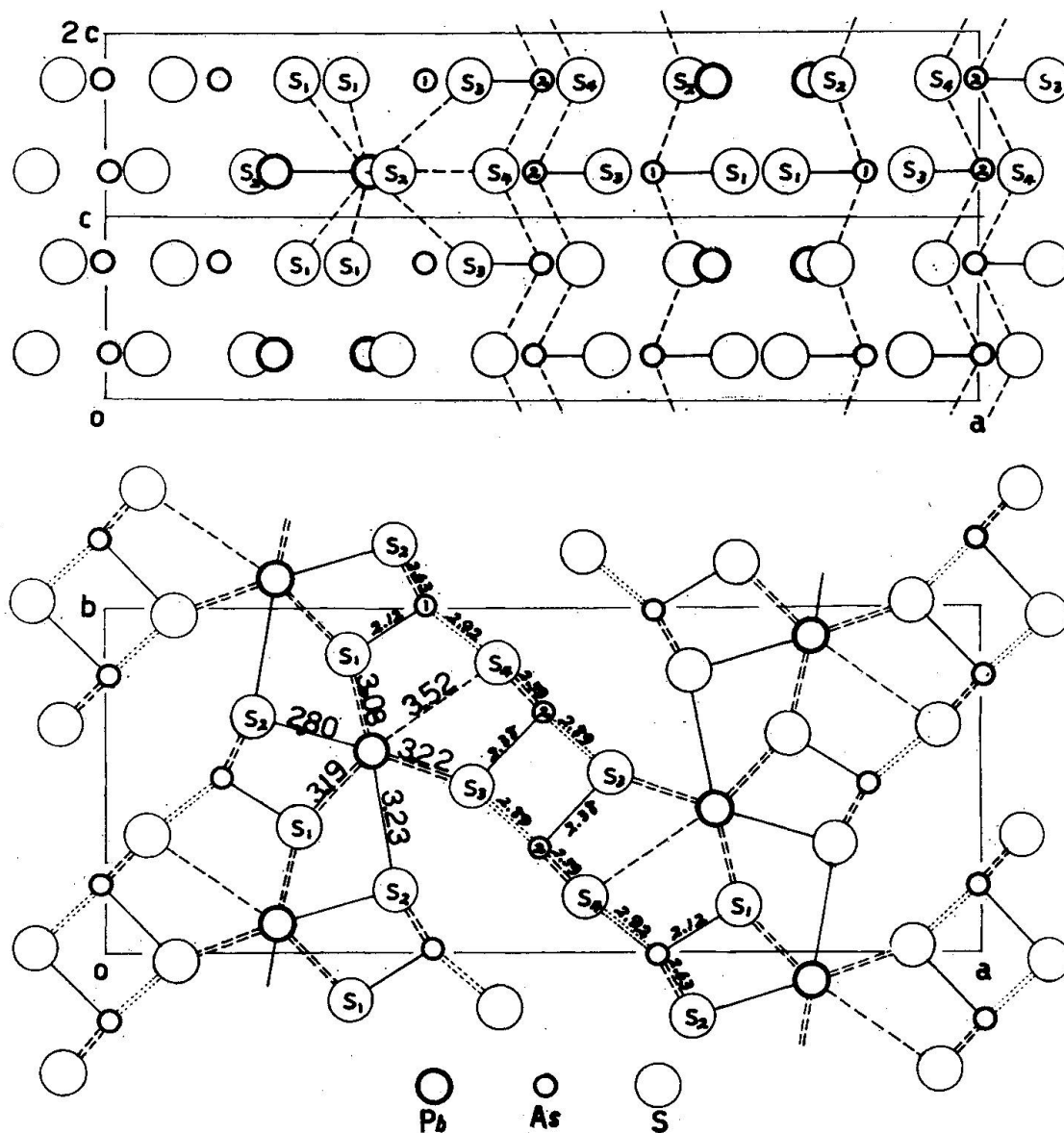


Fig. 1. Projection of the pseudo-structure of scleroclase on the (010) = *ac*-plane (above) and on the (001) = *ab*-plane (below), with indication of the coordination and interatomic distances.

scleroclase crystals are elongated and striated along the b -axis, but not along the c -axis.

3. The difference Fourier projection along the b -axis indicated a marked elongation of the electron density distribution of the sulfur atoms along the c -axis. The fact infers that in the real structure some of the sulfur atoms are displaced from their mean position either upwards or downwards along the chain direction. Namely, some of the arsenic and sulfur distances may be shorter than those observed in the average structure, while others may be too long to assume that strong bonding exists between these arsenic and sulfur atoms.

It is interesting to see that in this scleroclase structure the ionic radius and the packing sequence of the lead atom in the c -axis direction seems to be too large to fit the sulfoarsenite chains along the c -axis. In berthierite (BUERGER and HAHN, 1955) which has a very similar chemical formula (FeSb_2S_4) and a similar crystal structure, the iron atoms are just fitted in the structure forming a regular octahedral coordination without breaking the chains of sulfoarsenite "molecules".

7. Relations between structure and morphology

As mentioned in the Introduction several crystallographic elements have been given to scleroclase.

VOM RATH (1864) and BAUMHAUER (1895) (see also BADER, 1934) considered scleroclase to be orthorhombic with $(a:b:c)_{RB} = 0,539 : 1 : 0,619$ which is in accordance with the ratio from the pseudo-cell $a'_0 : b'_0 : c'_0 = 4 \times 0,621_8 : 1 : 0,531_1$ after the transformation $a'_0 = 4c_{RB}$, $b'_0 = b_{RB}$, $c'_0 = a_{RB}$. According to TRECHMANN (1906) scleroclase is monoclinic with $a:b:c = 1,2755 : 1 : 1,1949$, $\beta = 102^\circ 12'$. These elements also fit the pseudo-cell by the transformation $4\mathbf{a}_T + \mathbf{c}_T = \mathbf{a}'_0$, $\mathbf{c}_T = \mathbf{c}'_0$. The elements calculated from the pseudo-cell are in fair agreement $1,271_2 : 1 : 1,062_0$, $\beta = 102^\circ 03'$.

SMITH and SOLLY (1919) introduced several systems of elements, which show no relation to the structure and which — in the past — have complicated unnecessarily the discussion of the morphology of scleroclase. They also believed in a triclinic variety of scleroclase, which they called sartorite — α ; but according to BANNISTER et al. (1939) this sartorite — α (crystal Nos. 1 and 2) is identical with scleroclase (sartorite) (crystal No. 5).

The orthorhombic indices given by VOM RATH and BAUMHAUER can be transformed into our pseudo-cell or our true-cell. In some cases the indices become simpler, in others not. It is quite clear, that the morphology of a crystal with such a complicated true-cell also must be a complicated one. In addition the superstructure seems not to be always the same; this may explain why SOLLY and SMITH believed to have to introduce different elements for different crystals or parts of the same crystal. The different elements are shown in Fig. 2.

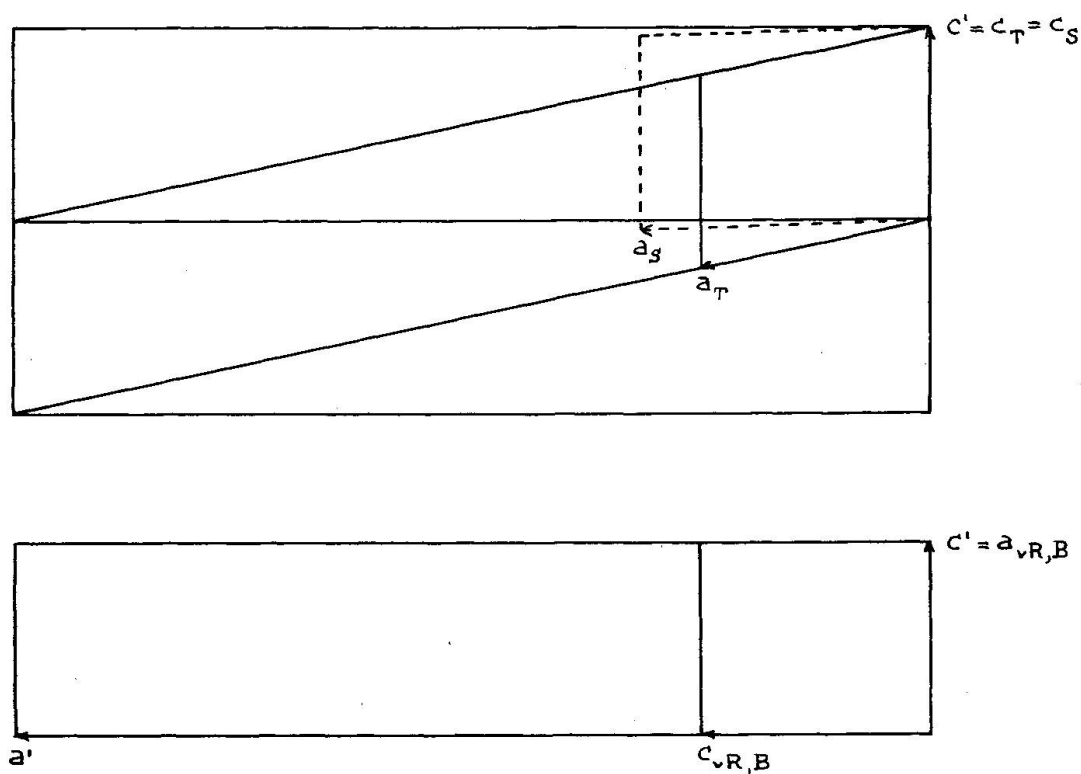


Fig. 2. Relation of the structural pseudocell (a' , c') to the elements used by VOM RATH-BAUMHAUER (vR , B), TRECHMANN (T) and SMITH-SOLLY (S) (see text).

B. Comparison of some sulfosalts with the composition $x\text{PbS} \cdot y\text{As}_2\text{S}_3$ (Y. I., V. K.)

In Table 4 the data for five sulfosalts of the composition $x\text{PbS} \cdot y\text{As}_2\text{S}_3$ are given. It is seen that the structural ratio $a_0 : b_0 : c_0$ and β_0 agrees very well with the morphological data (for scleroclase see section A; for dufrenoyite the morphological ratio may be written $2 \times 0,325_5 : 1 : 2 \times 0,306_3$). It was found that baumhauerite shows a strong piezoelectric

Table 4. Comparison of some sulfosalts $xPbS \cdot yAs_2S_3$

	a_0	b_0	c_0	β_0	$a_0 : b_0 : c_0$
Scleroklase ($x=y=1$) (pseudo-cell)	19,62 $\pm 0,02$	7,89 $\pm 0,01$	4,19 Å $\pm 0,005$	90°	2,487 : 1 : 0,531 ₁
Baumhauerite	22,8 ₉	8,37 ₀	7,92 ₀	97° 18'*)	2,735 : 1 : 0,946 ₂
Rathite I	25,10 $\pm 0,09$	7,93 $\pm 0,02_5$	8,42 $\pm 0,02_4$	98° 20' $\pm 12'$	3,165 : 1 : 1,062
Dufrenoy-site	8,37 $\pm 0,04$	25,74 $\pm 0,05$	7,90 $\pm 0,01$	90° 21' $\pm 12'$	0,325 ₂ : 1 : 0,306 ₉
Jordanite	8,96 $\pm 0,04$	31,92 $\pm 0,01$	8,45 $\pm 0,03$	117° 50' $\pm 10'$	0,280 ₇ : 1 : 0,264 ₇
	$a : b : c$	β	SG	PE	Striation
Scleroklase (pseudo-cell)	0,539 : 1 : 0,619**)	90°	$C_{2h}^5-P2_1/n$	—	// b_0 , structural chains // c_0 $= \frac{1}{2} \cdot 8,38 \text{ Å}$
Baumhauerite	2,737 : 1 : 0,947 ₂	97° 17'	C_1^1-P1	+	also // $b_0=8,37$
Rathite I	3,154 ₄ : 1 : 1,069 ₈	98° 43 $\frac{1}{2}'$	$C_{2h}^5-P2_1/a$	—	// $c_0=8,42$
Dufrenoy-site	0,651 ₀ : 1 : 0,612 ₆	90° 33 $\frac{1}{2}'$	$C_{2h}^2-P2_1/m$	—	// $a_0=8,37$
Jordanite	0,279 ₄ : 1 : 0,265 ₅	117° 47'	$C_{2h}^2-P2_1/m$	—	also // $c_0=8,45$
SG = space group, PE = piezoelectricity, *) $\alpha=\gamma=90^\circ$, **) BADER (1934)					

effect. Its space group therefore must be C_1^1-P1 and not $C_i^1-P\bar{1}$. The observed negative effect for scleroklase, rathite and jordanite is in agreement with the given space groups.

In the structure of scleroklase As-S-chains are running // to the period of $\sim \frac{1}{2} \cdot 8,4 \text{ Å}$. Normally the striation of the crystal would be // to this direction. This is the case for baumhauerite, rathite, dufrenoy-site and jordanite, but not for scleroklase, where the striation is // b_0 and the chains are // c_0 .

C. Fibrous sulfosalt (H. B.)

In the Lengenbach dolomite fine needles of a gray, sometimes reddish sulfosalt occur (see Fig. 16 in NOWACKI, 1960a). Each needle consists of two (or one and two) species: an orthorhombic (with $a_0=8,38$, $b_0=2 \times 25,61$, $c_0=7,89 \text{ Å}$; only missing spectra $h+k=\text{odd}$: space groups $C_{2v}^{11}-Cmm2$, $C_{2v}^{14}-Cm2m-C2mm$, D_2^6-C222 , $D_{2h}^{19}-Cmmm$; the spots with $h=\text{odd}$ are weaker than those with $h=\text{even}$ and somewhat diffuse)

and two monoclinic (with $a_0 = 8,38$, $b_0 = 25,61$, $c_0 = 7,89$ Å, $\beta_0 = 90^\circ 25'$; for (hkl) or $(h0l)$ no reflections are missing, $(0k0)$ spots coinciding with those of the orthorhombic species, space groups $C_s^1 - Pm$, $C_2^1 - P2$, $C_2^2 - P2_1$, $C_{2h}^1 - P2/m$, $C_{2h}^2 - P2_1/m$) (needle axis $\parallel a_0$, Fig. 3). The whole mass of a needle is distributed about equally over the three species. Each species only shows *one* orientation within a given crystal. The cells of the two monoclinic species are symmetrical to a plane \perp to the needle axis. The structures of the two monoclinic species may be either identical or enantiomorphous. The monoclinic species is identical with dufrenoyite [$a_0 = 8,37$, $b_0 = 25,74$, $c_0 = 7,90$ Å, $\beta_0 = 90^\circ 21'$; $C_{2h}^2 - P2_1/m$]; the lattice constants also show a similarity with one form of rathite (I) [LE BIHAN (1959), $a_0 = 8,43$, $b_0 = 25,80$, $c_0 = 7,91$, $\beta_0 = 90^\circ$; $C_2^2 - P2_1$].

On the same sample of dolomite there are other needles which only consist of the two monoclinic species (V.K.).

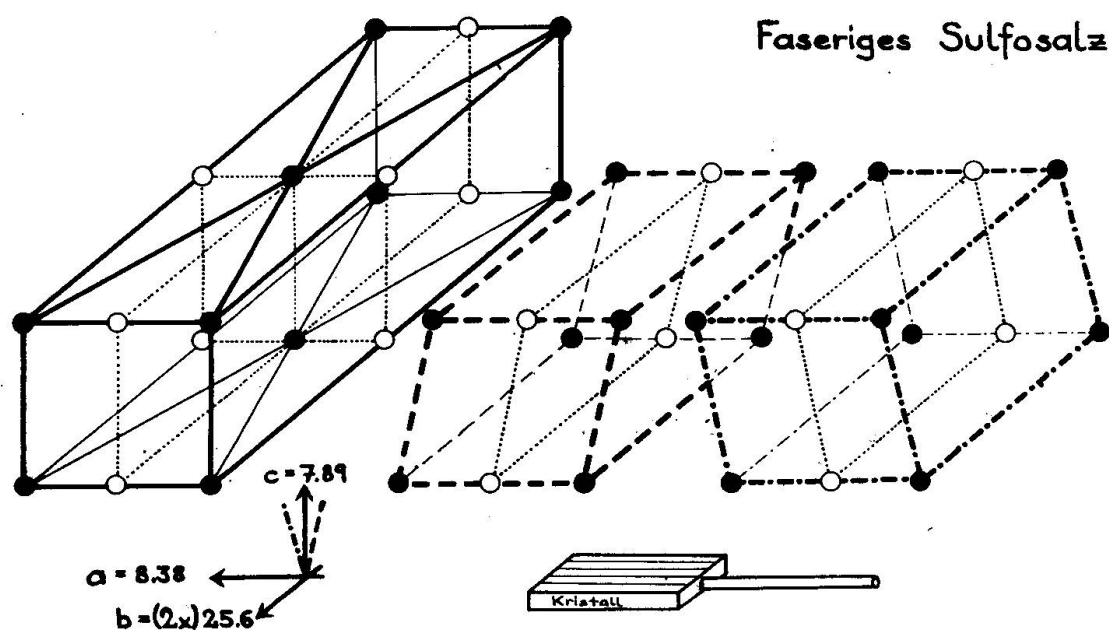


Fig. 3. Orthorhombic and monoclinic parts of fibrous sulfosalt. The monoclinic part is identical with dufrenoyite.

D. Lengenbachite (Y. I.)

Oscillation, Weissenberg, precession and Laue photographs yielded the data: $a_0 = 35,1_3$, $b_0 = 11,5_2$, $c_0 = 36,9_0$ Å, $\beta_0 = 92^\circ 36'$, space group $C_{2h}^2 - P2_1/m$; the sheets are $\parallel (001)$; no piezoelectric effect observed.

E. Hatchite and Trechmannite (V. K.)

When looking through the minerals found during the summer 1960 for the "Bernische Arbeitsgemeinschaft Lengenbach" (NOWACKI, 1960a), ST. GRAESER observed some very minute crystals of an unusual habit, which turned out by X-rays to be hatchite. The crystals show a bright lead-grey colour, but are dark-red in transparent light at edges or corners. A small crystal of about 1/10 mm in length, the habit of which has a certain similarity with Fig. 1 in the paper of SOLLY and SMITH (1912), was X-rayed and yielded the lattice constants $a_0 = 9,27 \pm 0,09$, $b_0 = 7,81 \pm 0,08$, $c_0 = 8,01 \pm 0,07$ Å, $\alpha_0 = 113^\circ 23'$, $\beta_0 = 116^\circ 33'$, $\gamma_0 = 85^\circ 06'$ (all $\pm 14'$), $a_0 : b_0 : c_0 = 1,187 : 1 : 1,026$, which can be compared with the data of SOLLY and SMITH $a' : b' : c' = 0,9787 : 1 : 1,1575$, $\alpha' = 116^\circ 53\frac{1}{2}'$, $\beta' = 85^\circ 12'$, $\gamma' = 113^\circ 44\frac{1}{2}'$, or — after the transformation $a' \rightarrow b$, $b' \rightarrow c$, $c' \rightarrow a$, $\alpha' \rightarrow \beta$, $\beta' \rightarrow \gamma$, $\gamma' \rightarrow \alpha$ — $a : b : c = 1,183 : 1 : 1,022$, $\alpha = 113^\circ 44\frac{1}{2}'$, $\beta = 116^\circ 53'$, $\gamma = 85^\circ 12'$, which is in perfect agreement with the X-ray data; space group $C_i^1 - P\bar{1}$ or $C_1^1 - P1$. The (true) reduced cell has $a_0 = 9,27 \pm 0,09$, $b_0 = 7,81 \pm 0,08$, $c_0 = 8,01 \pm 0,07$ Å, $\alpha_0 = 66^\circ 37'$, $\beta_0 = 63^\circ 27'$, $\gamma_0 = 85^\circ 06'$ (type I, all angles acute).

For trechmannite the data are: a_0 (rho.) = $8,65 \pm 0,01$ Å, $\alpha_0 = 108^\circ 17'$, a_0 (hex.) = $14,02$, c_0 (hex.) = $9,15$ Å, $c_0/a_0 = 0,653$, morphological (SOLLY 1905, SMITH and PRIOR 1907) $c/a = 0,6530$; space group $C_{3i}^2 - R\bar{3}$ (or $C_3^4 - R3$); colour wine-red, brighter than hutchinsonite, diamond brilliancy (NOWACKI and KUNZ 1961).

The piezoelectric tests were performed by Y. TAKEUCHI of Tokyo University with the apparatus constructed by IITAKA (1953).

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