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Structure of Cubic Aluminate Sodalite, $Sr_8[Al_{12}O_{24}](CrO_4)_2$

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Abstract. Hexaaluminium tetrastrontium dodecaoxide chromate, $M_r = 1640{\cdot}71$, cubic, $I\bar{4}3m$ (possibly $Im\bar{3}m$), $a = 9{\cdot}427$ (2) Å, $V = 837{\cdot}8$ (5) Å^3, $Z = 1$, $D_x = 3{\cdot}25$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo } K\alpha) = 13{\cdot}28$ mm^{-1}, $F(000) = 764$, $T \sim 330$ K, final $R = 0{\cdot}045$ for 256 unique reflections. The sodalite framework is (almost) fully expanded, therefore space group $Im\bar{3}m$ is also possible. The corresponding refinement gave results almost identical with that in $I\bar{4}3m$ ($R = 0{\cdot}048$ for 149 unique reflections). The tetrahedral CrO_4 cage anion is found to be sixfold disordered; only the superposition corresponds to cubic symmetry.

Introduction. Aluminate sodalites of general composition $M_8[Al_{12}O_{24}](XO_4)_2$ with M = Ca, Sr,... and X = S, Cr, Mo, W,... have recently become the subjects of one of our research programs, mainly because they represent a new family of compounds displaying ferroic phase transitions (Depmeier, 1979, 1986). In particular, the title compound (henceforth SACR) has been shown to become ferroelectric, as well as ferroelastic, below room temperature (Setter, Mendoza-Alvarez, Depmeier & Schmid, 1984). This is one of the reasons why we concentrated our investigations on this compound; another is that it can be grown as relatively large crystals (several millimetres), in contrast to other members of the series. In order to obtain as much information as possible on SACR and in view of planned studies on its low-temperature phases, the present work was undertaken with the following main objectives: (i) to discover whether the geometry of the AlO_4 tetrahedra is in accordance with a model for sodalites which predicted a high degree of tetrahedron distortion for all aluminate sodalites (Depmeier, 1984*a*), and (ii) to determine the orientation of the CrO_4 tetrahedron within the sodalite cage. In a recent study on $Ca_8[Al_{12}O_{24}](WO_4)_2$ aluminate sodalite (Depmeier, 1984*b*), it was found that the tetrahedral cage anion takes an orientation which is not compatible with cubic symmetry. It was conjectured that this special orientation of the cage anion is responsible for the occurrence of low-symmetry phases and that the cubic–noncubic phase transitions are triggered by order–disorder processes. Thus a cubic phase would necessarily be associated with disorder of the tetrahedral cage anion (Setter & Depmeier, 1984).

Experimental. The crystal used in this study formed part of a batch of emerald-green crystals grown from a Bi_2O_3 flux and showing {110} and {211} facets. It was ground to a flattened sphere, diameter ~ 375 µm, and mounted with EPO-TEK P10 high-temperature polyimide glue. The crystal was heated to ~ 330 K by means of a hot gas stream. Data collection: Syntex $R3$ diffractometer, graphite monochromator, one hemisphere ($-14 \le h$, $k \le 14$; $0 \le l \le 14$), $(\sin\theta/\lambda)_{\max} = 0{\cdot}7035$ Å^{-1}, ω scans, scan width 0·9°, variable scan speed 2–10° min^{-1}, background measured on both sides of the reflection, total background time = 0·65 times the time used for the peaks; two standards measured every 150 reflections showed no significant change; 4643 reflections, 3344 with $I > 3\sigma(I)$; Lorentz and polarization corrections; empirical absorption correction, $\mu R = 2{\cdot}5$, maximum/minimum transmission: 0·029/0·008; $R_{\text{int}} = 0{\cdot}035$; lattice parameter from a previous Guinier photograph, 12 reflections, least squares, $10{\cdot}6 < 2\theta < 54{\cdot}0°$, 293 K, thermal expansion neglected. Structure refinement: all reflections used; started with ideal coordinates for Sr, Al, framework O (O henceforth) and Cr, CrO_4–O atoms (O11 henceforth) from difference Fourier maps; cascade least squares, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = [\sigma^2(F_o) + 0{\cdot}001F_o^2]^{-1}$; scattering factors for neutral atoms, f' and f'' from *International Tables for X-ray Crystallography* (1974); empirical isotropic extinction correction; anisotropic atomic displacement parameters for Sr, Al, Cr and O, isotropic for O11; $I\bar{4}3m$ ($Im\bar{3}m$ in parentheses): 17 (13) parameters, 256 (149) unique reflections, $R = 0{\cdot}045$ (0·048), $wR = 0{\cdot}055$ (0·064);

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Table 1. *Fractional atomic coordinates* ($\times 10^4$, $\times 10^3$ *for* O11) *and equivalent isotropic displacement parameters* (Å^2 $\times 10^4$, $\times 10^3$ *for* O11)

The first line for each atom refers to $I\bar{4}3m$, the second one to $Im\bar{3}m$. E.s.d.'s are in parentheses. $U_{eq} = \frac{1}{3}$ trace **U**.

	Wyckoff notation	Site symmetry	Occupancy	x	y	z	U_{eq}(U_{iso} for O11)
Al	12(d)	$\bar{4}$	1·0	$\frac{1}{4}$	$\frac{1}{2}$	0	121 (4)
	12(d)	$\bar{4}2m$	1·0	$\frac{1}{4}$	$\frac{1}{2}$	0	125 (6)
O	24(g)	m	1·0	1595 (3)	1595 (3)	4982 (8)	235 (7)
	24(h)	mm	1·0	1593 (4)	1593 (4)	$\frac{1}{2}$	241 (10)
Sr	8(c)	$3m$	1·0	2476 (3)	2476 (3)	2476 (3)	606 (4)
	8(c)	$\bar{3}m$	1·0	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	618 (6)
Cr	2(a)	$\bar{4}3m$	1·0	0	0	0	472 (6)
	2(a)	$m\bar{3}m$	1·0	0	0	0	487 (9)
O11	24(g)	m	0·3333	625 (2)	375 (2)	464 (4)	115 (13)
	24(h)	mm	0·3333	625 (3)	375 (2)	$\frac{1}{2}$	160 (22)

maximum shift/e.s.d. after final least-squares cycle 0·06 (0·00); strongest peak in final difference Fourier map 1·4 (0·79) e Å^{-3}, 0·97 (0·69) Å apart from Sr. Refinement of the occupancy of O11 converged to 0·333.* All calculations performed with *SHELXTL* (Sheldrick, 1978) on a Nova 3 computer.

Discussion. Our first crystal of SACR, as well as the one used in this study, had an emerald colour. These crystals will subsequently be referred to as 'green crystals'. It was not until the present work was finished that new crystals, called 'yellow crystals' hereunder, were obtained by optimizing the growth conditions. Their quality seems to be superior to that of the green ones, because yellow is the usual colour of the chromate ion and only the yellow crystals allow the unambiguous detection of two structural phase transitions at ~ 287 and ~ 293 K, *e.g.* by differential scanning calorimetry or by means of optical observation under polarized light. In green crystals one of the two phase transitions is blurred or even suppressed. These findings might suggest that the transition characteristics depend quite strongly on the presence of defects, whereby a green colour indicates a higher and yellow colour a lower concentration of defects. It is also noteworthy that samples cut from the same crystal may display different transition temperatures.

We regard the publication of the results of this study on green crystals as justified, because this is the first study on a cubic aluminate sodalite and the main objectives of the work, as explained earlier, have been fully achieved. It is not to be expected that the overall structural characteristics of the cubic phase of yellow crystals will differ significantly from those found in this study. However, the supposedly subtle differences between green and yellow crystals might be quite interesting to analyse in a future study, because of the considerable attention which the role of defects on phase transitions has attracted in the last few years (*e.g.* Tolédano, 1984).

Table 2. *Important interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

	$I\bar{4}3m$	$Im\bar{3}m$
Al—O	4×1·729 (1)	4×1·728 (1)
O···O	2×3·008 (5)	2×3·004 (8)
	4×2·726 (5)	4×2·727 (8)
O—Al—O	2×120·9 (2)	2×120·7 (3)
	4×104·1 (1)	4×104·2 (2)
Al—O—Al	149·1 (2)	149·3 (3)
Tilt angle φ	0·66	0
Sr—O	3×2·638 (7)	6×2·649 (2)
	3×2·658 (7)	
Sr—O11	3×2·58 (3)	6×2·89 (2)
	3×3·20 (3)	
Cr—O11	12×1·703 (7)	12×1·66 (4)
O···O11	12×2·891 (6)	12×2·88 (4)

Table 1 lists the final atomic positional and equivalent isotropic displacement parameters and Table 2 the derived bond lengths and angles, as well as important non-bonded distances. Several descriptions and discussions of the cubic sodalite structure can be found in the literature (*e.g.* Hassan & Grundy, 1984; Depmeier, 1984*a*; Koch & Hellner, 1981, and literature cited therein).

The highest possible symmetry for a sodalite framework is $Im\bar{3}m$ which is, of course, a centrosymmetric space group. This symmetry requires the framework to be in its fully expanded state, *i.e.* the so-called tilt angle has to be 0°. Even the slightest deviation from this value destroys the centre of symmetry and the space group becomes $I\bar{4}3m$ (in 1:1 aluminosilicate sodalites, ordering of Al and Si would further reduce the symmetry to $P\bar{4}3n$). The framework O atom thereby passes smoothly from $x, x, 0$ [24(h) in $Im\bar{3}m$] to x, x, z [24(g) in $I\bar{4}3m$]. In connection with the interpretation of the domain pattern in the low-temperature phases, it should also be noted that this transition destroys the mirror plane perpendicular to ⟨100⟩.

Now, it has been argued (Depmeier, 1984*a*) that within the aluminosilicate series of sodalites (frame-

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* Lists of structure factors and anisotropic displacement parameters for $I\bar{4}3m$ and $Im\bar{3}m$ have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44234 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

work composition $[Al_{12-n}Si_nO_{24}]^{(12-n)-}$, $0 \leq n \leq 12$) only the Al-rich members of the series, and in particular the pure aluminate sodalites, should be capable of taking a very low, or even a zero, value for the tilt angle. This arises because only these sodalites are able to undergo the corresponding necessary distortion of the $(Al,Si)O_4$ tetrahedra constituting the framework. Hence, in the whole series only very Al-rich and pure aluminate sodalites should have the possibility of becoming centrosymmetric. It is also important to note that the non-framework atoms, too, have the possibility of gradually passing from $Im\bar{3}m$ to $I\bar{4}3m$ (Sr: $\frac{1}{4}, \frac{1}{4}, \frac{1}{4} \rightarrow x, x, z$; O11 (disordered): $x, x, 0 \rightarrow x, x, z$) and that Al and Cr do not change their positions during such a transition. Thus, there is a possibility for certain sodalites to be or to become centrosymmetric, provided they have a suitable framework composition. However, as far as we know, until now only one sodalite-type structure has been described in a centrosymmetric space group, *viz* 'barium aluminate hydrate' (Ahmed, Dent Glasser & King, 1973) of poorly defined composition.

Our refinement started in $I\bar{4}3m$ and it became quickly evident that SACR is almost, or actually, in its fully expanded state; hence, either $Im\bar{3}m$ or $I\bar{4}3m$ could be a suitable description. We have tried to draw upon several criteria in order to make a choice between these two possibilities, but, unfortunately, none gave an unambiguous answer. Therefore, we must state that actually we do not know which of the two possible space groups is the correct one. However, we hope that planned further experiments will help in eludicating this problem. For the following brief crystal-chemical discussion the question is not essential and we have chosen arbitrarily the results of the $I\bar{4}3m$ refinement as a basis for the discussion.

The strong angular distortion of the AlO_4 tetrahedron is indeed as expected [$\alpha = 120{\cdot}9\,(2)$, $\alpha' = 104{\cdot}1\,(1)°$, $d_1 = 3{\cdot}008\,(5)$, $d_2 = 2{\cdot}726\,(5)$, $l = 1{\cdot}729\,(1)$ Å; *cf.* Depmeier (1984*a*)] and the values describing the conformation of the framework (tilt angle $\varphi = 0{\cdot}7°$) and that of the oxygen four-rings [mean dihedral angle $\omega = 1{\cdot}3°$, conformational shear angle $\tau = 0°$ (Depmeier, 1983)] are well in accordance with a cubic, almost fully expanded, sodalite framework. The Al—O—Al angle depends on both tetrahedron distortion and framework conformation, and its observed value [$\gamma = 149{\cdot}1\,(2)°$] is in very good agreement with the calculated one [equation (15) of Depmeier (1984*a*)].

From a difference electron density map the CrO_4 O atoms (O11) were found on x, x, z (0·375, 0·375, 0·536). It was one of the main objectives of this study to locate this cage anion. This finding corresponds to what was found in $Ca_8[Al_{12}O_{24}](WO_4)_2$ (Depmeier, 1984*b*) and is in accordance with a model for cubic aluminate sodalites (Setter & Depmeier, 1984). In $I\bar{4}3m$ the equivalent positions of 0·375, 0·375, 0·536 occupy the vertices of a cuboctahedron, centred at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$, which is distorted towards a truncated tetrahedron (*cf.* Fig. 1), *i.e.* four tetrahedrally arranged triangles are larger than the remaining four. (This distortion is due to the deviation of z from 0·5; in $Im\bar{3}m$, with $x, x, \frac{1}{2}$, the corresponding cuboctahedron would be regular.) The cuboctahedron can be understood as representing six superposed CrO_4 tetrahedra. Each of these is oriented in such a way that one of its $\bar{4}$ axes points along one of the $\langle 100 \rangle$ directions, but it is rotated about this axis by $\sim \pm 45°$ (Depmeier, 1984*b*). As such, the threefold axes of the CrO_4 tetrahedron (in its various orientations) and those of the framework no longer coincide.

Therefore, an ordered arrangement of CrO_4 destroys the cubic symmetry. Conversely, one can state that the fact that SACR is cubic in its high-temperature phase results from disorder of the cage anions. Each vertex of the cuboctahedron (O11) belongs to two different tetrahedron orientations; thus the occupancy of O11 is 0·3333. It is not currently known whether the disorder is dynamic or static in nature, as elastic diffraction experiments are not sensitive to this question; suitable experiments using different methods are planned. The Cr—O11 distance of 1·703 (7) Å is slightly longer than the 'best estimates' for the Cr—O bond length in a CrO_4 tetrahedron [1·670 Å (McGinnety, 1972)]. This observation reflects the fact that the use of a cuboctahedron to mimic a sixfold-disordered tetrahedron is geometrically not the best choice. A better one, however, using more realistic disorder models, would necessarily be at the expense of an increased number of variables in the least-squares procedure. The planned structure determinations of the low-temperature phases of SACR

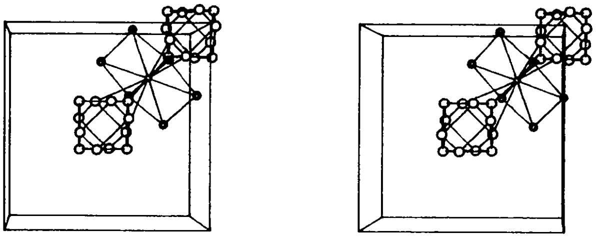

Fig. 1. Stereoplot of part of the structure of SACR in $I\bar{4}3m$. The outlines of the unit cell are shown as well as two cuboctahedra centred on 0,0,0 and $\frac{1}{2},\frac{1}{2},\frac{1}{2}$, each representing six disordered CrO_4 groups. An Sr atom at $\frac{1}{4},\frac{1}{4},\frac{1}{4}$ (small circle) is coordinated by a large triangle of the cuboctahedron in the centre and by a small triangle of the cuboctahedron in 0,0,0. The small triangle is called 'the co-antistar', the large one the 'co-star'. Both cuboctahedra are located in two adjacent sodalite cages and the coordination sphere of Sr is completed by a six-membered ring of framework oxygens (●) which is common to both cages. The ring has the shape of a flat trigonal antiprism. The triangles called 'star' and 'antistar' in the text have the same orientation as the corresponding 'co-star' and 'co-antistar'. The Cr atoms and the greater part of the framework have been omitted for the sake of clarity.

are expected to provide more realistic Cr—O bond lengths.

The O atoms of the framework are also at *x, x, z* (0·160, 0·160, 0·498) and they again form a cuboctahedron, centred at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$, which envelops the O11 cuboctahedron and has the same orientation. Thus, each O11 atom points, more or less directly, at a framework O atom [distance 2·891 (6) Å] and thereby exerts a repulsive force on it. If the cage anions are fully disordered, as in our case, the resulting forces add up to zero and the sodalite framework remains undistorted; if, on the other hand, the cage anions are ordered, then the resulting forces may have a non-zero value and a deformation of the framework with consequent possible lowering of the symmetry results [see, as an example, Depmeier (1984*b*)]. The deformation of the framework may become noticeable in the lattice parameters and the spontaneous deformation as a macroscopic order parameter of a paraelastic–ferroelastic phase transition can be determined.

For a discussion of the coordination of Sr by O atoms, see Figs. 1 and 2. Sr is situated almost at $\frac{1}{4}, \frac{1}{4}, \frac{1}{4}$, which means it is very close to the centre of the six-rings of the sodalite framework. The O atoms of the six-rings form a trigonal antiprism, which is regular in the fully expanded state, but distorted with one larger and one smaller triangle, when the framework is partially collapsed. The partial collapse of the framework is at least partially triggered by the requirements of the cage cation, Sr in our case, to make good bonds with the O atoms of the framework. In order to do so, Sr has to leave $\frac{1}{4}, \frac{1}{4}, \frac{1}{4}$ by moving along $\langle 111 \rangle$, thereby approaching three of the O atoms of the trigonal antiprism. These three O atoms form an equilateral triangle (in the cubic case) and are natural candidates for bonding to the cage cation for all degrees of partial collapse. In our case the three bonds are 2·638 (7) Å. We wish to call the triangle formed by these O atoms the 'star' with respect to Sr. Similarly, the inversely oriented triangle of the remaining three O atoms is called the 'antistar'. Sr—O distances involving the O atoms of the antistar are longer than the corresponding ones of the star; for the almost fully expanded framework of SACR, however, the difference is very small [2·658 (7) *vs* 2·638 (7) Å]. It is now an interesting general observation that, in order to make *shorter bonds* with the star, the Sr has to move in the direction of the antistar; *i.e.* its vertical distance from the *plane* of the star is *longer* than the corresponding one from the antistar (Fig. 2). As an aside we note that O atoms which are part of the antistar with respect to a given cage cation belong, by symmetry, to the star of another cage cation.

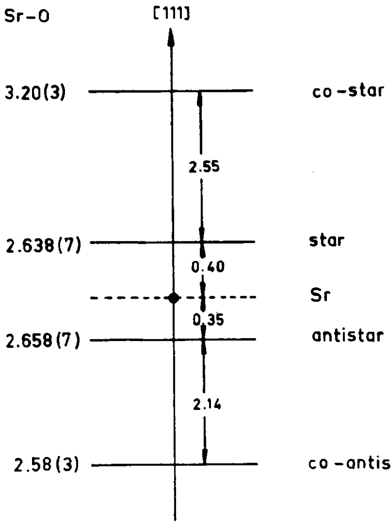

Fig. 2. Coordination characteristics along $\langle 111 \rangle$ for Sr in SACR, $I\bar{4}3m$ refinement; Sr—O distances are given on the left, distances between planes on the right-hand side. All distances are in Å. 'Star', 'co-star', 'antistar' and 'co-antistar' are as shown in Fig. 1.

The coordination sphere of the Sr atoms is completed by O atoms of the disordered CrO_4 groups. Equilateral triangles of the corresponding cuboctahedra are considered. Fig. 1 shows two of them in contact with the Sr, a smaller one and a larger one. The O atoms of the smaller triangle have quite short [2·58 (3) Å] distances from Sr. The orientation of the triangle corresponds to that of the antistar and, therefore, we wish to call this arrangement of three O11 the 'co-antistar' (co = cuboctahedron). Similarly, the larger 'co-star' has the orientation of the star and the distances of its O atoms from Sr [3·20 (3) Å] indicate very weak, if any, Sr—O bonds.

In the cubic phase, star, antistar, co-star and co-antistar define planes perpendicular to $\langle 111 \rangle$. They lend themselves to a convenient method of displaying the essential aspects of the coordination sphere around the cage cation. The corresponding graph for SACR (in $I\bar{4}3m$) is shown in Fig. 2.

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Structure of Godlevskite, Ni_9S_8

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Abstract. $(Ni_{8\cdot7}Fe_{0\cdot3})S_8$, $M_r = 784{\cdot}1$, orthorhombic, $C222$, $a = 9{\cdot}3359\,(7)$, $b = 11{\cdot}2185\,(10)$, $c = 9{\cdot}4300\,(6)$ Å, $V = 987{\cdot}7$ Å^3, $Z = 4$, $D_x = 5{\cdot}273$ g cm^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 180{\cdot}06$ cm^{-1}, $F(000) = 1518$, room temperature, final $R = 0{\cdot}041$ for 1743 unique reflections. The crystal structure of godlevskite is based on a distorted cubic close-packed array of 32 S atoms per unit cell, with 20 Ni atoms in tetrahedral coordination and 16 in square-pyramidal coordination. The ideal stoichiometry is established as Ni_9S_8. NiS_4 tetrahedra form clusters of three-membered chains and five-membered crosses, and NiS_5 square pyramids form fourfold clusters and chains. The structure contains elements of the structures of the related phases α-Ni_7S_6, millerite (NiS) and pentlandite [$(Fe,Ni)_9S_8$].

Introduction. Godlevskite (Ni_9S_8) was originally described from the Noril'sk and Talnakh Cu–Ni sulfide deposits in northern Siberia (Kulagov, Evstigneeva & Yushko-Zakharova, 1969), and was subsequently reported in a nickel sulfide ore from the Texmont mine, Ontario (Naldrett, Gasparrini, Buchan & Muir, 1972). Both of these studies recognized godlevskite as a metal-excess nickel sulfide mineral equivalent to the synthetic low-temperature Ni-excess phase previously referred to as 'β-Ni_7S_6' (*e.g.* Kullerud & Yund, 1962). Lundqvist (1947) and Kullerud & Yund (1962) did not index the powder pattern of 'β-Ni_7S_6', but Lundqvist noted that the possibility of a degenerate pentlandite structure for the phase could not be excluded. Kulagov *et al.* (1969) reported that the X-ray single-crystal and powder diffraction patterns of godlevskite were consistent with the space groups *C*222, *Cmm*2, *Amm*2, and *Cmmm*, with $a = 9{\cdot}18$, $b = 11{\cdot}29$, $c = 9{\cdot}47$ Å.

In the present study, godlevskite is shown to have a crystal structure based on a distorted cubic close-packed array of S atoms, with 20 Ni atoms per unit cell in tetrahedral coordination and 16 in square-pyramidal coordination. The ideal stoichiometry is established as Ni_9S_8. Other aspects of godlevskite and synthetic Ni_9S_8 will be discussed elsewhere (Fleet, 1988).

Experimental. The godlevskite specimen was from the Noril'sk Cu–Ni sulfide deposit. It occurs as twinned crystals enclosed by chalcopyrite ($CuFeS_2$) and intergrown with bornite (Cu_5FeS_4) and millerite (NiS) (Kulagov *et al.*, 1969). The crystal was rectangular in shape with approximate dimensions $0{\cdot}10 \times 0{\cdot}12 \times 0{\cdot}13$ mm and a calculated volume of $0{\cdot}17 \times 10^{-2}$ mm^3. It was removed from a polished thin section, and size-reduced with 600 abrasive paper. A preliminary X-ray precession study confirmed that the diffraction pattern was consistent with space groups *C*222, *Cmm*2, *Amm*2, *Cmmm* and, possibly, $C222_1$, and revealed very weak reflections of a second phase (chalcopyrite?) and weak twin-related reflections [twin plane, (101)]. Enraf–Nonius CAD-4*F* diffractometer, graphite-monochromatized Mo *Kα* radiation. 20 reflections in 2θ range 53·5–60·8° for cell parameters. Data collected by θ–2θ scan; scan angle $(2\theta) = 2{\cdot}4°$, with dispersion correction. 4387 *hkl*, $-hkl$ reflections permitted by space group *C*222 out to $2\theta = 90°$ measured, but structure refinement limited to data with $2\theta \le 80°$; $-16 \le h \le 16$, $0 \le k \le 20$, $0 \le l \le 17$. Standard reflections 044, $04\bar{4}$, 800; $R_{\mathrm{int}} = 0{\cdot}02$, no significant decline in intensity. Background, Lorentz, polarization and absorption corrections applied; transmission factors (by Gaussian integration with a $12 \times 12 \times 12$ grid) varied from 0·136 for 020 to 0·258 for $\bar{6}$,20,5. 1743 unique reflections. 389 reflections considered unobserved $[I \le 3\sigma(I)]$; $\sigma(I) = [I_m + 0{\cdot}002^2(I_m - B)^2 + 0{\cdot}005^2(I - I_m)^2]^{1/2}$; I_m, measured intensity and B, background. Data set corrected for interference due to 2·4% (by diffraction volume) of a twin component, following measurements on 30 strongest reflections with $(h + l) \ne 2n$.

Crystal-structure analysis was complicated by pseudosymmetry, space-group ambiguity and poor diffraction quality of both synthetic (Fleet, 1988) and

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