numbering scheme for the molecule is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985). The packing within the crystal lattice is shown in Fig. 2 (Motherwell & Clegg, 1978). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Discussion. Although the positions of the H atoms are given considerably more accurately, uncertainties involving the non-H atoms are approximately twice as large in the neutron study (Simonsen & Mueller, 1965). The gross structure of the anion is as described for the previous determination, with octahedral coordination of the Ru centre by one OH, one NO and four NO_2 ligands. The metal and the NO and OH groups lie on a crystallographic mirror plane. The distances between the Ru and its ligating atoms are consistently longer (by an average of 0.01 Å) than the distances obtained in the neutron diffraction study. A qualitatively similar trend is observed for N—O distances. Although the six-coordination about the Na^+ ions is similar to that described in the earlier work, the distances to the apical atoms are again somewhat larger (by 0.015 – 0.025 Å) in our study: only the Na(1)—O(water) and Na(2)—O(hydroxo) distances are in good agreement.

The systematically shorter bond lengths in the neutron study do not arise from differences in the

radiation used or in the temperature at which data were collected: the discrepancies arise solely from systematic errors in the unit-cell edges ($a = 12.75$, $b = 14.52$, $c = 7.37$ Å) used in the earlier derivation. We have therefore recalculated the bond lengths for the neutron diffraction study from the published fractional coordinates, but using the accurate unit cell obtained in the course of the X-ray structure determination. The three sets of bond distances are listed for comparison in Table 2 and, although our values are significantly more accurate in respect of the non-H atoms, there are no significant differences between these and the corrected values for the neutron study.

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Structure of Ba₃(AsS₄)₂.7H₂O at 125 K

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Abstract. Diarsenic tribarium octasulfide–water (1/7) $Ba_3As_2S_8.7H_2O$, $M_r = 944.45$, monoclinic, $P2_1/n$, $a = 12.450$ (2), $b = 12.727$ (3), $c = 12.694$ (2) Å, $\beta = 97.99$ (1)°, $V = 1991.8$ (8) Å³, $Z = 4$, $D_x = 3.149$ g cm⁻³, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 99.9$ cm⁻¹, $F(000) = 1728$, $T = 125$ K, final $R = 0.019$ for 5215 unique observed reflections. The structure, known already from a room-temperature study, has been reinvestigated at 125 K in order to clarify hydrogen bonding. It consists of a framework of $BaS_4(H_2O)_4/BaS_6(H_2O)_4$ polyhedra and AsS_4 tetrahedra that is reinforced by two O—H···O, eleven O—H···S and one bifurcated O—H···S,S hydrogen bonds. Many of the hydrogen bonds are severely

bent. The O···S distances of the eight most linear respective hydrogen bonds measure on average 3.24 (8) Å, which is relatively short in comparison to a large number of other thiosalt hydrates.

Introduction. In connection with structural and spectroscopic investigations of hydrogen bonds between water molecules as donor and S atoms as acceptor (Mikenda, Mereiter & Preisinger, 1989) the crystal structure of Ba₃(AsS₄)₂.7H₂O became of interest. The structure of this salt had previously been determined at room temperature by Cordier, Schwidetzky & Schäfer (1985) and refined to $R = 0.076$ for 5904 reflections. Since no information on H-atom posi-

tions or hydrogen bonds was given, we undertook a reinvestigation. A temperature of 125 K was selected in order to obtain structure data suitable for correlation with planned low-temperature vibrational spectroscopy.

Experimental. The title compound was prepared according to Glatzel (1911) from $Ba(SH)_2$ and As_2S_5. A crystal was ground to a sphere of 0.32 mm diameter. A Philips PW1100 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation and a Leybold–Heraeus NCD2 nitrogen gas-stream cooler were used for the measurements at 125 K. Lattice parameters were determined by least-squares fit of 76 reflections with $9 < \theta < 28°$. Intensity data were collected with ω–2θ scans using a scan width of $(1.3 + 0.35\tan\theta)°$ and a scan speed of $2° \text{min}^{-1}$. Three standard reflections (600, 080, 008), monitored every 60 min, remained constant within $\pm 1.5\%$. 7166 reflections with $2 \leq \theta \leq 30°$, $-17 \leq h \leq 17$, $0 \leq k \leq 17$, $0 \leq l \leq 17$ were measured, corrected for Lorentz–polarization effects and for absorption (sphere, $\mu R = 1.6$), and averaged ($R_{int} = 0.011$ based on F) to 5798 unique reflections, 5215 of which with $F_o > 6\sigma(F_o)$ were subsequently used.

Initial atomic parameters of non-H atoms were taken from Cordier, Schwidetzky & Schäfer (1985) and suitably transformed (see *Discussion*). After completing preliminary refinements with anisotropic temperature factors ($wR = 0.024$) the H atoms were located from a weighted difference map. The final least-squares refinement minimized $\Sigma w(|F_o| - |F_c|)^2$ using a correction for extinction, neutral-atom scattering functions with anomalous dispersion, anisotropic temperature factors for non-H atoms, one common isotropic temperature factor for H atoms, and rigid group constraints for the H_2O molecules with fixed O—H = 0.8 Å for X-ray scattering centres, O—H = 0.96 Å for nuclear H positions, and H—O—H = 108° for both. Final residuals: $R = 0.019$, $wR = 0.021$ and $S = 1.18$ for 5215 reflections weighted by $w = 1/[\sigma^2(F_o) + 0.0001F_o^2]$ and 204 refined parameters; $R = 0.024$ for all reflections, including those below the cut-off limit. All $\Delta/\sigma < 0.01$. Final difference densities between -0.7 and 0.7 e Å^{-3} with the most prominent features near Ba atoms. Calculations were carried out with *SHELX76* (Sheldrick, 1976) and *PARST* (Nardelli, 1982); drawings were prepared with *ORTEPII* (Johnson, 1976).*

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54886 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE0081]

Discussion. Atomic parameters are presented in Table 1, selected bond lengths and angles in Table 2 and hydrogen-bond geometry in Table 3. Three important differences to the reference system of Cordier, Schwidetzky & Schäfer (1985) for the room-temperature structure have to be noted: in the present work a reduced cell with space group $P2_1/n$ and cell dimensions as given in the *Abstract* are used, atom designations are altered, and atoms are symmetry-transformed in order to assemble them in functional groups. The unit cell used by Cordier, Schwidetzky & Schäfer (1985) is unconventional and markedly oblique ($a = 12.758$, $b = 12.829$, $c = 19.077$ Å, $\beta = 139.5°$). In terms of the present cell ($P2_1/n$) it has the base vectors $\mathbf{a}$, $\mathbf{b}$, $-\mathbf{a} + \mathbf{c}$ and space group $P2_1/c$. A less oblique cell, also with space group $P2_1/c$ but with $\beta = 131.6°$, is defined by the base vectors $-\mathbf{a}$, $-\mathbf{b}$, $\mathbf{a} + \mathbf{c}$. Transformed to the present setting the room-temperature unit cell of Cordier, Schwidetzky & Schäfer (1985) has the following dimensions: $a = 12.512$ (4), $b = 12.829$ (4), $c = 12.758$ (4) Å, $\beta = 98.03$ (10)°, $V = 2028$ Å^3. Former atomic coordinates transform to the new $P2_1/n$ setting by the matrix ($\bar{1}0\bar{1}$, 010, 001).

Actual differences between the structure at room temperature (Cordier, Schwidetzky & Schäfer, 1985) and 125 K (this work) are moderate and can be briefly summarized as a decrease in the following parameters: unit-cell edge lengths 0.062, 0.102 and 0.064 Å, β by 0.04°, cell volume by 35 Å^3 (all figures referring to the $P2_1/n$ cell); atomic thermal parameters by a factor 0.55; Ba—S and Ba—O bonds by 0.022 Å on average; As—S bonds by 0.004 Å on average; hydrogen-bond distances O$\cdots$S and O$\cdots$O by 0.020 Å on average. All bond-length changes do not exceed 0.065 Å.

The crystal structure of $Ba_3(AsS_4)_2.7H_2O$ is built up from $BaS_4(H_2O)_4$ and $BaS_6(H_2O)_4$ polyhedra, two kinds of AsS_4 tetrahedra, and seven Ba-bonded water molecules. These constituents are linked into a complicated framework in which the coordination polyhedra of Ba are mutually connected *via* shared corners, edges and two faces, while the two independent AsS_4 tetrahedra are connected to seven and nine Ba and share edges with one and four coordination polyhedra of Ba, respectively (see Figs. 1 and 2).

The structure exhibits pseudosymmetry and derives from a B-centred prototype with space group $P2_1/m$ and the mirror planes parallel to (010) at $y = \frac{1}{4}$ and $\frac{3}{4}$. In the prototype structure the atoms Ba(1), As(1), S(1), S(2), As(2), S(5), S(6), O(7), H(13) and H(14) would be located on the mirror planes, and the atom pairs Ba(2)/Ba(3), S(3)/S(4), S(7)/S(8), O(1)/O(2), O(3)/O(4) and O(5)/O(6) at both sides of these planes. In the real structure of $Ba_3(AsS_4)_2.7H_2O$ the individual atoms deviate by up to about 1 Å from the nearest positions they could adopt in the $P2_1/m$

Table 1. Atomic coordinates and equivalent isotropic temperature factors (Å^2)

H atoms were refined as parts of rigid H$_2$O molecules with O—H = 0.96 Å and H—O—H = 108° for the nuclear positions given here. E.s.d.'s of the H$_2$O rotation angles average 3.1°. One common B_{iso} = 2.6 (3) Å^2 for all H.

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Ba(1)	0.16583 (1)	0.26266 (1)	0.12341 (1)	0.57 (1)
Ba(2)	0.24112 (1)	0.11877 (1)	0.56718 (1)	0.66 (1)
Ba(3)	0.29040 (1)	0.47737 (1)	0.59583 (1)	0.62 (1)
As(1)	0.39980 (2)	0.24553 (2)	0.86310 (2)	0.48 (1)
As(2)	0.41205 (2)	0.25821 (2)	0.37519 (2)	0.46 (1)
S(1)	0.22868 (5)	0.23960 (5)	0.87542 (5)	0.74 (2)
S(2)	0.42202 (5)	0.26982 (5)	0.69829 (5)	0.68 (2)
S(3)	0.46710 (5)	0.09491 (5)	0.91701 (5)	0.73 (2)
S(4)	0.47909 (5)	0.37480 (5)	0.95437 (5)	0.71 (2)
S(5)	0.24158 (5)	0.28241 (5)	0.38240 (5)	0.72 (2)
S(6)	0.43610 (5)	0.25704 (5)	0.21012 (5)	0.71 (2)
S(7)	0.47025 (5)	0.11722 (5)	0.45913 (5)	0.67 (2)
S(8)	0.49240 (5)	0.39172 (5)	0.45882 (5)	0.64 (2)
O(1)	0.12486 (16)	0.05739 (17)	0.23324 (17)	1.12 (4)
O(2)	0.11547 (16)	0.45101 (16)	0.22192 (16)	0.95 (4)
O(3)	0.21704 (16)	−0.04919 (17)	0.42256 (16)	0.98 (4)
O(4)	0.21560 (16)	0.56861 (16)	0.40498 (15)	0.84 (4)
O(5)	0.10036 (16)	0.05101 (16)	0.71171 (16)	0.85 (4)
O(6)	0.17508 (17)	0.47125 (17)	0.78448 (17)	1.09 (4)
O(7)	0.14144 (16)	0.31189 (17)	0.60398 (16)	0.92 (4)
H(1)	0.0972	0.0064	0.1807	
H(2)	0.0643	0.0944	0.2547	
H(3)	0.0836	0.5046	0.1743	
H(4)	0.0630	0.4306	0.2668	
H(5)	0.1522	−0.0836	0.4373	
H(6)	0.1991	−0.0080	0.3592	
H(7)	0.1479	0.6009	0.4146	
H(8)	0.2040	0.5275	0.3410	
H(9)	0.0681	−0.0044	0.6666	
H(10)	0.0445	0.1006	0.7214	
H(11)	0.1074	0.4942	0.7458	
H(12)	0.1642	0.4028	0.8130	
H(13)	0.0916	0.3230	0.5401	
H(14)	0.0999	0.2982	0.6609	

Table 2. Selected distances (Å) and angles (°)

Coordination polyhedra of Ba

Ba(1)—S(1^i)	3.361 (1)	Ba(3)—S(1^{vi})	3.369 (1)
Ba(1)—S(2^{ii})	3.330 (1)	Ba(3)—S(2)	3.281 (1)
Ba(1)—S(5)	3.301 (1)	Ba(3)—S(3^{vi})	3.521 (1)
Ba(1)—S(6)	3.393 (1)	Ba(3)—S(5)	3.662 (1)
Ba(1)—S(7^{ii})	3.347 (1)	Ba(3)—S(8)	3.428 (1)
Ba(1)—S(8^{ii})	3.411 (1)	Ba(3)—S(8^{vii})	3.330 (1)
Ba(1)—O(1)	3.038 (2)	Ba(3)—O(4)	2.731 (2)
Ba(1)—O(2)	2.814 (2)	Ba(3)—O(5^{vi})	2.788 (2)
Ba(1)—O(3^{iii})	2.905 (2)	Ba(3)—O(6)	2.959 (2)
Ba(1)—O(4^{iv})	2.925 (2)	Ba(3)—O(7)	2.818 (2)

Ba(2)—S(2)	3.240 (1)		
Ba(2)—S(4^{ii})	3.377 (1)	Mean value Ba—S	3.364 (112)
Ba(2)—S(5)	3.137 (1)	Mean value Ba—O	2.851 (85)
Ba(2)—S(7)	3.333 (1)		
Ba(2)—O(3)	2.807 (2)		
Ba(2)—O(5)	2.842 (2)		
Ba(2)—O(6^v)	2.759 (2)		
Ba(2)—O(7)	2.821 (2)		

AsS$_4$ tetrahedra

As(1)—S(1)	2.159 (1)	As(2)—S(5)	2.159 (1)
As(1)—S(2)	2.170 (1)	As(2)—S(6)	2.158 (1)
As(1)—S(3)	2.165 (1)	As(2)—S(7)	2.160 (1)
As(1)—S(4)	2.169 (1)	As(2)—S(8)	2.172 (1)
Mean value	2.166 (4)	Mean value	2.162 (6)

S(1)—As(1)—S(2)	109.5 (1)	S(5)—As(2)—S(6)	108.2 (1)
S(1)—As(1)—S(3)	106.8 (1)	S(5)—As(2)—S(7)	111.2 (1)
S(1)—As(1)—S(4)	111.6 (1)	S(5)—As(2)—S(8)	104.9 (1)
S(2)—As(1)—S(3)	109.6 (1)	S(6)—As(2)—S(7)	113.0 (1)
S(2)—As(1)—S(4)	107.3 (1)	S(6)—As(2)—S(8)	111.3 (1)
S(3)—As(1)—S(4)	112.0 (1)	S(7)—As(2)—S(8)	108.0 (1)

Short nonbonding contacts

S(3)···S(3^{viii})	3.235 (1)	S(7)···S(7^x)	3.212 (1)
S(4)···S(4^{ix})	3.406 (1)	S(8)···S(8^{vii})	2.945 (1)

Symmetry code: none x, y, z; (i) $x, y, z - 1$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (iii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iv) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (v) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$; (vi) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$; (vii) $1 - x, 1 - y, 1 - z$; (viii) $1 - x, -y, 2 - z$; (ix) $1 - x, 1 - y, 2 - z$; (x) $1 - x, -y, 1 - z$; (xi) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$.

Table 3. Hydrogen-bond parameters (Å,°)

O—H = 0.96 Å and H—O—H = 108° were fixed during refinement (see text and Table 1). Symmetry codes are given in Table 2. Asterisks mark bonds which are simultaneously edges of Ba coordination polyhedra.

O—H···A	H—A	O—A	O—H—A	A—O—A'
O(1)—H(1)···S(8^{iv})	2.44 (1)	3.396 (2)	173 (4)	S(8^{iv})—O(1)—S(2^{ii}) 94.1 (1)
O(1)—H(2)···S(2^{ii})*	2.51 (3)	3.331 (2)	144 (4)	
O(2)—H(3)···S(7^{iii})	2.25 (1)	3.196 (2)	169 (4)	S(7^{iii})—O(2)—S(3^{ii}) 119.0 (1)
O(2)—H(4)···S(3^{ii})	2.41 (1)	3.340 (2)	164 (4)	
O(3)—H(5)···S(4^v)	2.34 (2)	3.225 (2)	153 (4)	S(4^v)—O(3)—O(1) 107.9 (1)
O(3)—H(6)···O(1)	1.92 (1)	2.858 (3)	164 (4)	
O(4)—H(7)···S(6^{iii})*	2.66 (4)	3.269 (2)	121 (4)	S(6^{iii})—O(4)—O(2) 82.1 (1)
O(4)—H(7)···S(3^{vi})*	2.73 (3)	3.438 (2)	131 (4)	S(3^{vi})—O(4)—O(2) 109.1 (1)
O(4)—H(8)···O(2)	2.00 (2)	2.896 (3)	155 (4)	
O(5)—H(9)···S(4^v)	2.19 (1)	3.142 (2)	169 (4)	S(4^v)—O(5)—O(6^{ii}) 113.7 (1)
O(5)—H(10)···S(6^{ii})	2.25 (1)	3.184 (2)	164 (4)	
O(6)—H(11)···S(3^{vi})*	2.50 (3)	3.301 (2)	141 (4)	S(6^{xi})—O(6)—O(1) 141.3 (1)
O(6)—H(12)···S(1)	2.33 (2)	3.202 (2)	152 (4)	
O(7)—H(13)···S(3^{ii})	2.30 (2)	3.212 (2)	160 (4)	S(3^{ii})—O(7)—S(6^{xi}) 85.1 (1)
O(7)—H(14)···S(6^{xi})	2.32 (2)	3.175 (2)	147 (4)	

structure. This causes, for instance, Ba(2) and Ba(3) to show different coordination numbers, despite their pseudosymmetry relationship.

The Ba(1)S$_6$(H$_2$O)$_4$ polyhedron is distinctly mirror-symmetric and can be visualized as an elongated BaS$_4$ disphenoid [S(5)—S(6) and S(7)—S(8) edge of As(2)S$_4$] with additional two S and four H$_2$O in equatorial positions (Figs. 1 and 2). The Ba(2)S$_4$(H$_2$O)$_4$ polyhedron corresponds to a distorted square antiprism, and the Ba(3)S$_6$(H$_2$O)$_4$ polyhedron is irregular in shape (Fig. 1). The bonds to Ba (Table 2) vary in bond strength (Brown & Altermatt, 1985) from 0.09 valence units for Ba(3)—S(5) to 0.37 valence units for Ba(2)—S(5), with only small differences between S and O ligands in their mean bond strengths: Ba—S bonds average to 0.21 valence units, Ba—O bonds to 0.22. The AsS$_4$ tetrahedra are essentially regular. Their mean bond length of 2.164 (5) Å at 125 K is close to the value at room temperature (2.168 Å) and to that of Na$_3$AsS$_4$.8H$_2$O [2.162 Å according to Mereiter, Preisinger, Baumgartner, Heger, Mikenda & Steidl (1982); 2.156 Å according to Dittmar & Schäfer (1978)]. It has to be emphasized that there are four nonbonding S···S contacts between the AsS$_4$ tetrahedra of Ba$_3$-

(AsS$_4$)$_2$.7H$_2$O for which the S—S distances (Table 2) are shorter than the edge lengths of the AsS$_4$ tetrahedra [3.433 (1)–3.601 (1) Å]. Most outstanding among these is the contact S(8)···S(8), 2.945 (1) Å, which is the only one of these four that simultaneously represents a shared edge between two Ba polyhedra.

The water molecules $H_2O(1)$ and $H_2O(2)$ are each bonded at their lone-pair sides to one Ba and one hydrogen-bond-donating water molecule. The remaining five molecules bridge pairs of Ba atoms with Ba—O—Ba angles of 138.5 (1)° for $H_2O(3)$ and about 110° for the rest. All water molecules donate pairs of hydrogen bonds, which can be subdivided into two O—H···O, eleven O—H···S and one bifurcated O—H···S,S type bonds. Geometric data

Fig. 1. $Ba_3(AsS_4)_2.7H_2O$ in a view approximately down **a***. Ba atoms are dotted and located at $0 < x < \frac{1}{2}$. Only atom designation numbers of Ba, As, S and O are given.

Fig. 2. Stereoview of the structure down **b**.

are provided in Table 3. It can be noted that many of the hydrogen bonds are unusually severely bent, *e.g.* O(6)—H(11)···S(3), O(1)—H(2)···S(2) or O(7)—H(14)···S(3). An analysis of the H_2O environments revealed two plausible reasons for this feature: (i) the ligands around the water molecules deviate more or less severely from a regular tetrahedral arrangement; (ii) the water molecules obviously adopt orientations that maximize distances to the ligands at their lone-pair sides – two Ba or one Ba + one H_2O – rather than orientations that make their hydrogen-bond angles straight. Examples for these features can be found in Fig. 1, for instance $H_2O(7)$. The eight most nearly linear O—H···S bonds with bond angles larger than 150° exhibit O···S (H···S) distances of 3.142–3.396 (2.199–2.441 Å), the mean values being 3.24 (8) [2.31 (8) Å]. A comparison with the O···S distance *versus* abundancy histogram of Mikenda, Mereiter & Preisinger (1989) shows that these eight bonds belong to the shortest ones observed so far in thiosalt hydrates. This relative shortness of the O—H···S bonds in $Ba_3(AsS_4)_2.7H_2O$ is most likely the result of inductive effects of the Ba—O bonds, *i.e.* caused by cation effects.

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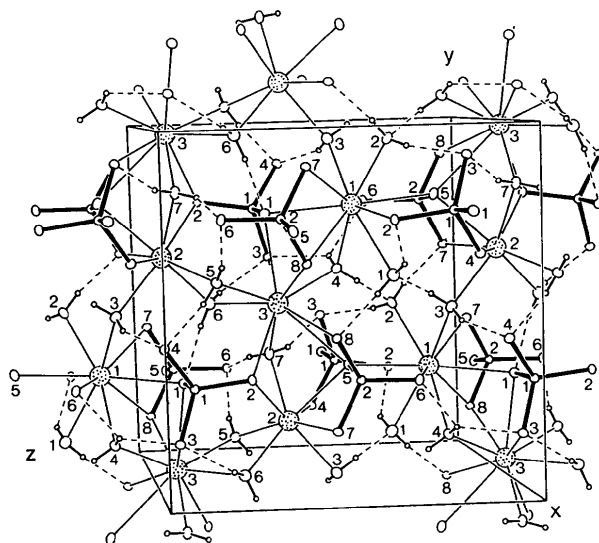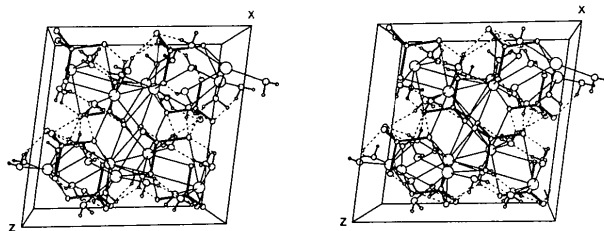SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.