Accordingly, the Ba atoms were distributed over four fourfold sites in $P\bar{4}3m$, and the refinement, including occupation factors, was continued; R dropped to about 0·10 for all reflexions.

Examination of the parameters at this stage showed that all, including the Ba occupation factor, were consistent, within the limits of error, with the space group $Pm3m$. Accordingly, refinement was continued in $Pm3m$, and insofar as this resulted in a slightly improved value of R (0·09) with fewer parameters, it seems likely that this is in fact the true space group.

Table 1 gives the final positional parameters, and occupancy fractions for Ba, and Table 2 some of the more important bond distances. The geometry of the framework and the coordination of the barium ions is satisfactory, and the proposed structure explains the observed chemical behaviour of the material. This, together with the reasonably good agreement between F_o and F_c,* gives confidence in the basic correctness of the structure.

Nevertheless, some unsatisfactory features remain. (1) The formula $Ba_8(AlO_2)_{12}[Al(OH)_6]_2$ has a net charge of -2, or more if the sum of the occupation fractions (which suggests that the average number of barium atoms per cell is slightly less than 8) is to be believed. The deficiency might be made good by the addition of protons, *e.g.* $Ba_8(AlO_2)_{12}[Al(OH)_5H_2O]_2$, which corresponds to the 8:7:7 mole ratio. (2) When temperature factors are allowed to refine, those of the framework oxygen atoms tend to run negative. (3) The bond lengths about the octahedral Al are not satisfactory.

Attempts to resolve these difficulties by postulating different arrangements for the cavity contents (including other sorts of disorder) all failed to produce satisfactory convergence. Coupling between the occupation factors, temperature and scale factors (even though the two first were never allowed to refine simultaneously), together with the uncertainty regarding the exact composition, certainly contributed to this, and about 100 least-squares cycles were run without further progress. In these circumstances, the presence of the heavy barium atoms is an embarrassment. The isostructural strontium compound has now been prepared in these laboratories; should it prove possible to grow reasonably sized crystals of this, it might well be a more fruitful subject for refinement.

Table 2. *Bond lengths and angles*

(*a*) The framework

Al(1)–O(1)	1·76 (2) Å	(×2)
Al(1)–O(2)	1·76 (2)	(×2)
O(1)–Al(1)–O(2)	114 (1)°	
O(1)–Al(1)–O(2)	105 (1)	(×4)
O(2)–Al(1)–O(2)	123 (1)	
Al(1)–O(1)–Al(1)	156 (1)	
Al(1)–O(2)–Al(1)	147 (1)	

(*b*) The cavity contents

Ba(1)–O(1)	2·74 (1) Å	(All ×3)
Ba(1)–O(2)	2·85 (1)	
Ba(1)–O(3)	2·94 (2)	
Ba(2)–O(1)	2·89 (1)	(All ×3)
Ba(2)–O(2)	2·68 (1)	
Ba(2)–O(4)	2·89 (1)	
Al(2)–O(3)	2·10 (3)	(Both ×6)
Al(3)–O(4)	1·77 (3)	

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* A list of observed and calculated structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30075 (2pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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Refinement of hydrogen-atom positions in L-cysteic acid.H_2O from neutron diffraction data.

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From partial three-dimensional neutron-diffraction intensity data (280 reflexions) and starting from the X-ray structure of Hendrickson & Karle [*Acta Cryst.* (1971) B**27**, 427–431], the parameters for the hydrogen atoms in L-cysteic acid. H_2O ($C_3H_7NO_5S$ H_2O) have been refined by the method of least-squares. The final R value is 0·040. The details of the hydrogen atom stereochemistry in the molecule and the hydrogen bonding are presented.

Introduction

The neutron diffraction study of L-cysteic acid monohydrate ($NH_3^+CHCH_2SO_3^-COOH.H_2O$) was undertaken as part of our current program of locating precisely the positions of hydrogen atoms in amino acids and their derivatives. The X-ray study of this amino acid has been done by Hendrickson & Karle (1971), in which the approximate positions of hydrogen atoms were determined. The orientation of the H–H vector of the water molecule was determined by El Saffar, Hendrickson & Koski (1969) using proton magnetic resonance technique.

Experimental

The crystal used for data collection was prismatic in shape, weight 38 mg. It was mounted with its c axis along the φ-

Table 1. *Atomic parameters in the molecule of* L-*cysteic acid*. H_2O

All parameters are multiplied by 10^4. The atomic parameters of hydrogen atoms are from the present work. The standard deviations of these parameters are the least-squares estimated standard deviations. Atomic parameters of non-hydrogen atoms are those obtained by Hendrickson & Karle after applying absorption correction to X-ray data (private communication). However, the standard deviations of these parameters are reproduced from the published X-ray work (1971).

The expression for the temperature factor is: $\exp[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S	8527 (2)	3906 (1)	−298 (3)	64 (3)	13 (0)	108 (4)	−1 (1)	9 (3)	0 (1)
O(1)	3893 (7)	3127 (2)	1789 (9)	145 (12)	17 (1)	139 (18)	−5 (3)	22 (12)	6 (4)
O(2)	3258 (7)	4194 (2)	3347 (9)	132 (12)	19 (1)	116 (18)	2 (3)	45 (12)	−6 (4)
O(3)	8843 (7)	4662 (2)	−544 (9)	159 (12)	17 (1)	194 (18)	−9 (3)	−22 (12)	−3 (4)
O(4)	8198 (7)	3726 (2)	2329 (9)	159 (12)	28 (1)	133 (18)	−12 (3)	−17 (12)	23 (4)
O(5)	9982 (7)	3501 (2)	−1571 (9)	97 (12)	31 (1)	325 (18)	20 (3)	25 (12)	−28 (4)
N	4789 (9)	4868 (3)	−569 (11)	108 (13)	18 (2)	242 (21)	16 (4)	34 (14)	27 (5)
C(1)	3838 (9)	3803 (3)	1692 (11)	46 (13)	18 (2)	156 (21)	3 (4)	−23 (14)	10 (5)
C(2)	4537 (9)	4089 (3)	−800 (11)	72 (13)	19 (2)	151 (21)	1 (4)	−8 (14)	16 (5)
C(3)	6324 (9)	3736 (3)	−1933 (11)	113 (13)	21 (2)	86 (21)	−4 (4)	−4 (14)	−4 (5)
O(*W*)	2620 (9)	2654 (3)	5911 (11)	228 (13)	19 (2)	266 (21)	10 (4)	120 (14)	11 (5)
H(1)	3371 (18)	4001 (8)	−2127 (20)	114 (25)	26 (4)	148 (46)	2 (10)	−32 (32)	2 (11)
H(2)	6094 (22)	3173 (8)	−1922 (32)	140 (27)	26 (4)	339 (63)	16 (11)	63 (47)	−40 (16)
H(3)	6572 (21)	3914 (8)	−3883 (23)	171 (31)	30 (5)	199 (52)	−24 (10)	103 (37)	12 (14)
H(4)	3444 (23)	5120 (8)	−705 (34)	171 (31)	21 (5)	444 (66)	46 (12)	−38 (49)	−24 (17)
H(5)	5360 (26)	5010 (9)	1042 (32)	269 (42)	22 (5)	358 (74)	2 (15)	−45 (53)	−9 (17)
H(6)	5586 (29)	5047 (9)	−2012 (40)	403 (60)	13 (5)	619 (103)	−17 (17)	318 (71)	50 (21)
H(7)	3347 (19)	2932 (8)	3575 (28)	156 (29)	18 (4)	155 (44)	1 (9)	1 (38)	17 (12)
H(8)	2877 (25)	2210 (9)	6559 (36)	238 (40)	18 (4)	325 (56)	−24 (11)	−27 (54)	8 (18)
H(9)	1573 (25)	2879 (10)	6856 (31)	229 (42)	34 (6)	255 (58)	3 (14)	146 (49)	−53 (18)

Table 2. *Bond distances and angles for the hydrogen atoms only**

Distances		Angles	
C(2)—H(1)	1·084 (13) Å	H(1)–C(2)–N	107·0 (0·9)°
C(3)—H(2)	1·082 (16)	H(1)–C(2)–C(1)	105·9 (0·8)
C(3)—H(3)	1·102 (14)	H(1)–C(2)–C(3)	106·2 (0·8)
N—H(4)	1·051 (17)	H(2)–C(3)–H(3)	109·4 (1·3)
N—H(5)	0·980 (18)	H(2)–C(3)–C(2)	108·2 (1·0)
N—H(6)	1·003 (21)	H(2)–C(3)–S	107·6 (0·9)
O(1)—H(7)	1·085 (16)	H(3)–C(3)–C(2)	111·1 (0·9)
O(*W*)–H(8)	0·930 (18)	H(3)–C(3)–S	105·5 (0·9)
O(*W*)–H(9)	0·980 (18)	H(4)–N—H(5)	106·9 (1·5)
H(8)—H(9)	1·569 (25)	H(4)–N—H(6)	106·3 (1·5)
Torsion angles†		H(5)–N—H(6)	110·5 (1·6)
φ_1 C(1)–C(2)–N–H(5)	40·5° (1·4°)	H(4)–N—C(2)	110·0 (1·0)
φ^2 C(1)–C(2)–N–H(6)	164·4° (1·3°)	H(5)–N—C(2)	113·1 (1·2)
φ^3 C(1)–C(2)–N–H(4)	−79·0° (1·2°)	H(6)–N—C(2)	109·7 (1·1)
		C(1)–O(1)—H(7)	111·5 (0·9)
		H(8)–O(*W*)—H(9)	110·4 (1·6)
		H(8)–O(*W*)···H(7)	125·9 (1·4)
		H(9)–O(*W*)···H(7)	122·6 (1·3)

* The errors in parentheses have been calculated from the standard deviations of the coordinates given in Table 1 and include standard deviations of the unit-cell constants.

† Names and signs of these are in accordance with the recommendations made by the IUPAC–IUB Commission on Biochemical Nomenclature (1970).

Table 3. *Hydrogen bond data in* L-*cysteic acid*. H_2O

Bond	Symmetry operation on acceptor atom			$X\cdots Y$	X-H	H$\cdots Y$	∠H–$X\cdots Y$
N—H(4)···O(2)	$\frac{1}{2}-x$	$1-y$	$-\frac{1}{2}+z$	2·824 (8) Å	1·051 (17) Å	1·829 (16) Å	15·0 (0·9)°
N—H(6)···O(3)	$1\frac{1}{2}-x$	$1-y$	$-\frac{1}{2}+z$	2·943 (8)	1·003 (21)	1·993 (22)	15·3 (1·2)
N—H(5)···O(3)	$1\frac{1}{2}-x$	$1-y$	$\frac{1}{2}+z$	2·967 (8)	0·980 (18)	1·993 (18)	5·2 (1·1)
O(1)—H(7)···O(*W*)	x	y	z	2·524 (8)	1·085 (16)	1·439 (16)	0·9 (0·8)
O(*W*)–H(8)···O(4)	$-\frac{1}{2}+x$	$\frac{1}{2}-y$	$1-z$	2·814 (7)	0·930 (18)	1·887 (17)	3·8 (1·1)
O(*W*)–H(9)···O(5)	$-1+x$	y	$1+z$	2·780 (8)	0·980 (18)	1·821 (18)	9·7 (1·1)

axis of the paper-tape controlled automatic neutron diffractometer '3D-FAD' (Momin, Sequeira & Chidambaram, 1969). θ–2θ step-scan mode (0·1° step in 2θ) was employed and the scanning range varied from 5 to 6°. The wavelength of the incident beam was 1·178 Å. The setting angles were calculated using the orthorhombic cell constants as determined by Hendrickson & Karle (1971): $a = 6{\cdot}927$ (1), $b = 19{\cdot}027$ (3) and $c = 5{\cdot}305$ (1) Å ($Z = 4$, calculated density $= 1{\cdot}77$ g cm^{-3}). The space group absences for $P2_12_12_1$ were confirmed. After the neutron intensities of 280 independent* reflexions out of 970 possible reflexions, up to $\sin\theta/\lambda = 0{\cdot}65$ Å^{-1}, had been measured, the crystal was unfortunately destroyed in an accident. Since we did not have another good crystal we decided to use the available data and refine only the hydrogen atom parameters rather than the complete structure. The integrated intensities of these reflexions were reduced to F_o^2's with the program *DATARED* (Srikanta & Sequeira, 1967, unpublished) and absorption corrections were applied ($\mu_{\text{measured}} = 2{\cdot}23$ cm^{-1}). The range of transmission factors was 0·610 to 0·680. Starting from the structure given by Hendrickson & Karle (1971), the positional and anisotropic thermal parameters of the hydrogen atoms, the scale factor, the overall temperature factor† and an extinction parameter which corrects for extinction by Zachariasen's method (1967) – in all 84 parameters – were subjected to full-matrix least-squares refinement using 280 F_o^2's and the program *XFLS* (Busing, Martin & Levy, 1962). The heavy atom parameters were fixed at the values obtained from absorption corrected X-ray data (Hendrickson & Karle, private communication). The neutron scattering factors (in units of 10^{-12} cm) used were: S, 0·2847; N, 0·940; C, 0·6626; O, 0·575 and H, −0·3723 (Shull, 1971). In the initial stages, weights based on counting statistics were used but in the final stages these were determined from a $|\Delta F^2|$ *vs.* $|F_c|^2$ plot to be $\omega^{-1/2} = 0{\cdot}39 + 0{\cdot}045\ |F_c|^2$ for $|F_c|^2 \leq 26$ and $\omega^{-1/2} = 1{\cdot}12 + 0{\cdot}009\ |F_c|^2$ for $|F_c|^2 > 26$. The refinement converged at the R value $[= (\sum|F_o - |F_c||)/\sum F_o]$ of 0·040 and a weighted R value $\{= [\sum\omega|F_o^2 - |F_c|^2|^2/\sum\omega F_o^4]^{1/2}\}$ of 0·077. The final nuclear parameters for all the atoms are given in Table 1.† The value of the extinction parameter $G (= \gamma^*\lambda^{-1})$ was found to be $0{\cdot}136 \times 10^4$, which corresponds to an equivalent mosaic spread of 43 sec of arc. The value obtained for the overall temperature factor‡ was −0·21 Å^2.

The bond distances and angles involving the hydrogen atoms are given in Table 2, and the hydrogen bond data are presented in Table 3. As expected, these values are now more reasonable than those from the X-ray study. A stereoscopic drawing of the molecule is shown in Fig. 1. One of the principal axes of the thermal ellipsoid of atom H(6) is unusually short (0·054 Å). This may be ascribed to the limited data used for refinement.

The hydrogen bonding scheme described by Hendrickson & Karle (1971) is confirmed. The water molecule has approximately trigonal planar coordination. It acts as donor for two hydrogen bonds to two cysteic acid molecules related by the 2_1 screw axis and acceptor for a hydrogen bond from the carboxylic oxygen O(1). The sum of the three angles, H(7)···O(W)–H(8), H(7)···O(W)–H(9) and H(8)–O(W)–H(9) is 359·0°. The lone pair coordination of the water molecule is thus of type F (Chidambaram, Sequeira & Sikka, 1964). The value of 110·5° for the HOH angle is higher than the vapour value 104·5°, which is not unexpected for the trigonally coordinated water molecule (Sikka & Chidambaram, 1969; Coppens & Sabine, 1969). The orientation of the p–p vector as determined in this study is in agreement with that determined by El Saffar *et al.* (1969), using proton magnetic resonance. The p–p vector subtends angles of 35·2 (0·9) and 84·3 (0·9)° with the [010] and [001] axes compared with the p.m.r. values of 36 (1) and 87 (1)°. The H–H distance is 1·569 (0·02) compared with 1·59 (0·01) Å. The short hydrogen bond O(1)–H(7)···O(W) of length 2·524 Å is almost linear; correspondingly the O–H distance has lengthened to 1·085 Å.

We are very grateful to Professor Z. M. El Saffar for kindly supplying the crystal. Thanks are also due to Shri S. N. Momin and Shri H. Rajagopal for technical assistance. We are also grateful to Dr W. A. Hendrickson and

* These were distributed as 71 *hk*0, 61 *hk*1, 60 *hk*2, 37 *hk*3, 30 *hk*4, 14 *hk*5 and 7 *hk*6.

† The overall temperature factor was varied partly to compensate for any possible systematic deviations between neutron and X-ray thermal parameters of non-hydrogen atoms. In L-lysine HCl $2H_2O$, for example, it was found that the equivalent isotropic temperature factors of the non-hydrogen atoms as determined by X-rays were on the average 0·91 Å^2 higher than those determined from the neutron data (Bugayong, Sequeira & Chidambaram, 1972).

† Structure factor tables may be obtained from the authors on request.

‡ To calculate structure factors for neutron diffraction, the temperature factors for the non-hydrogen atoms have to be decreased by this value of the overall temperature factor.

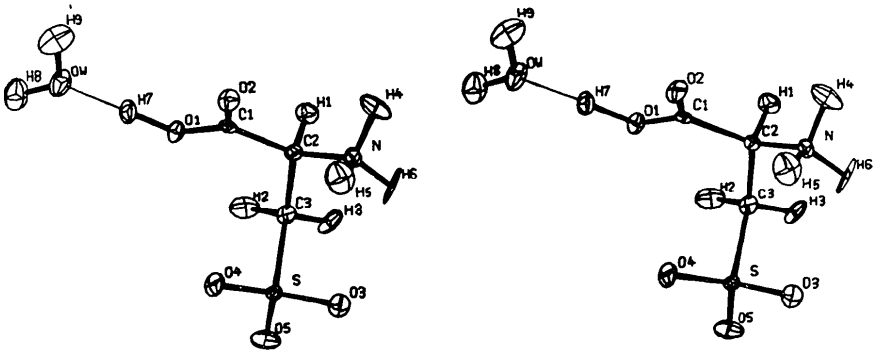

Fig. 1. Stereoscopic drawing of L-cysteic acid molecule. The figure was drawn by the computer program *ORTEP* (Johnson, 1965).

Dr J. Karle for supplying new heavy-atom parameters based on the absorption-correction data.

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The crystal structure of D-iso-ascorbic acid. Errata.

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Typographical errors in several numerical results in the paper by Azarnia, Berman & Rosenstein [*Acta Cryst.* (1972). B**28**, 2157–2161] are corrected.

The x parameter of O(6) in Table 1 should be 0·1292 (6), not 0·2192 (6). In Table 3 the hydrogen-bonding distance $d(jk)$ of H(O2) → O(6a) should be 1·76 Å, not 176· Å, and the torsion angle O(6)–C(6)–C(5)–O(5) should be 67·2°, not 70·7° (p. 2161, first line).

We are indebted to Dr David L. Hughes for informing us of these errors.

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Acta Cryst. (1973). B**29**, 1170

The crystal structure of β-tantalum.

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The X-ray powder pattern of β-tantalum has been indexed in terms of a tetragonal unit cell with $a = 10{\cdot}194$ and $c = 5{\cdot}313$ Å. β-Tantalum appears to be isomorphous with β-uranium.

In recent years a second crystalline modification of elemental tantalum has been reported in addition to the body centred cubic form (Read & Altman, 1965; Mills, 1966).

The new phase, which is now generally referred to as β-tantalum exhibits rather different electrical properties from the cubic variety and is thus of some concern to those employing tantalum films in microcircuits (Westwood, 1970).

Previously β-tantalum was prepared during sputtering experiments and the diffraction data available have in some cases suffered from the effect of preferred orientation. In other cases the material was not single phase. X-ray powder patterns were indexed in terms of a tetragonal unit cell, initially having $a = 5{\cdot}34$, $c = 9{\cdot}94$ Å (Read & Altman, 1965) and later $a = 5{\cdot}32$, $c = 9{\cdot}92$ Å (Mills, 1966).

The electron diffraction powder pattern of β-tantalum has recently been indexed with the tetragonal parameters $a = 10{\cdot}29$, $c = 9{\cdot}2$ Å (Das, 1972).

β-Tantalum has now been prepared as a single phase by electrodeposition from a molten fluoride bath at 800 °C. The X-ray powder pattern has been recorded using a Nonius Guinier camera calibrated against a silicon standard ($a = 5{\cdot}4307$ Å) and re-indexed as shown in Table 1. The unit cell is indeed tetragonal but the parameters, which have been derived with the aid of a least-squares refinement program (Marples & Shaw, 1966), are $a = 10{\cdot}194$, $c = 5{\cdot}313$ Å.

The intensities of the powder lines were recorded using a microdensitometer and are also given in Table 1. The general form of these intensities is remarkably similar to