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# The Crystal and Molecular Structure of Imidazolium Sulphate Dihydrate

By H. C. Freeman, F. Huq, J. M. Rosalky* and I. F. Taylor Jr†
*School of Chemistry, University of Sydney, Sydney* 2006, *Australia*

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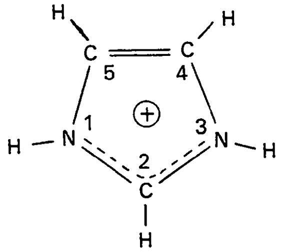
The structure of imidazolium sulphate dihydrate, $(ImH_2)_2SO_4.2H_2O$, has been determined from three-dimensional X-ray counter data and refined by the full-matrix least-squares method. The final residual $R$ is 0·042 for 1080 independent observed reflexions. The crystals are tetragonal with $a=7{\cdot}981$ (6), $c=18{\cdot}83$ (1) Å, $D_m=1{\cdot}49$ g cm$^{-3}$, $Z=4$ and space group $P4_12_12$. The imidazolium ions are joined by N–H···O hydrogen bonds to the sulphate ions (2·78 Å) and water molecules (2·73 Å). The dimensions of imidazolium ions are significantly different from those of neutral imidazole.

As Sundberg & Martin (1974) have pointed out in a recent review, the imidazolium cation



has an intrinsic interest (i) as an acid with a $pK_a$ near 7, which makes it 'an optimum catalyst and buffer' in aqueous solution, and (ii) as an aromatic cation in which the protons on N-1 and/or N-3 or C-2 can be replaced by metal ions. These properties enable the imidazole groups of histidine side chains in proteins to perform important functions in catalytic processes and as metal-binding centres.

The procedures for the isolation and crystallization of proteins frequently involve the use of concentrated ammonium sulphate solutions. The present work provides structural data for interactions which may occur between exposed imidazolium groups, sulphate ions and water molecules.

## Experimental

Imidazole (1 g) was dissolved in 1·5$M$ sulphuric acid (10 ml). The volume was reduced to 3 ml by evaporation on a water bath. Absolute ethanol (15 ml) was added to the hot solution. Colourless needle-shaped crystals formed on cooling. The product was recrystallized from methanol–ethanol to give well formed, polyhedral, deliquescent crystals.

### *Crystal data*

$C_6H_{14}N_4O_6S$, F.W. 270·3. Tetragonal, $a=7{\cdot}981(6)$, $c=18{\cdot}83(1)$ Å, $U=1199(1)$ Å$^3$, $D_m=1{\cdot}49(2)$ g cm$^{-3}$ (by flotation in $CHCl_3/CCl_4$), $D_x=1{\cdot}50$ g cm$^{-3}$ for $Z=4$. $F(000)=504$, $\mu(\text{Mo } K\alpha)=2{\cdot}95$ cm$^{-1}$, $\lambda(\text{Mo } K\alpha_1)=0{\cdot}70926$, $\lambda(\text{Mo } K\alpha_2)=0{\cdot}71354$ Å. Space group $P4_12_12$ ($P4_32_12$), determined from systematic absences ($00l$ present only for $l=4n$, $h00$ for $h=2n$).

All diffraction measurements were made on a computer-controlled Supper equi-inclination diffractometer (Freeman, Guss, Nockolds, Page & Webster, 1970). The unit-cell dimensions were fitted to the $\theta$ values of a number of high-angle reflexions. The intensity data were recorded by the $\omega$-scan procedure (Mo $K\alpha$ radiation, graphite crystal monochromator, Philips scintillation counter PW1964/10). The intensity $I$ of a reflexion with integrated peak count $P$ and background counts $B_1$ and $B_2$ was given by $I=P-(B_1+B_2)$, with variance $\sigma^2(I)=P+B_1+B_2$. Reflexions for which $I<3{\cdot}9\sigma(I)$ were considered unobservably weak.

The specimen crystal was coated with shellac to protect it from atmospheric moisture. It was mounted for rotation about the $a$ axis. All the accessible reflexions (about 3500) in one octant of reciprocal space were recorded. Lorentz and polarization corrections were applied. The degree of perfection of the monochromator crystal was undetermined. Accordingly, the polarization correction applied to each intensity was the mean of the corrections appropriate for Mo $K\alpha$ radiation reflected from perfect and ideally mosaic crystals, respectively. The maximum uncertainty introduced by this procedure is about 1 %. No absorption correction was made, the linear absorption coefficient being small and the crystal approximately equidimensional. The data were placed upon a common scale, using inter-layer scale factors fitted by least squares to the symmetry-related reflexions on different layers (Rae, 1965; Rae & Blake, 1966). The final data set consisted of 1716 independent reflexions. The intensities of 636 reflexions were below the observable threshold. The standard deviations $\sigma(F)$ of the struc-

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* Present address: Research School of Chemistry, Australian National University, Canberra 2600, Australia.

† Present address: Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, U.S.A.

ture amplitudes $F$ were calculated, taking into account both the random counting errors and estimated systematic errors (Freeman & Guss, 1972). The systematic errors were expressed as functions of $|F|$ and $\sin\theta/\lambda$.

The structure was solved by standard Patterson and Fourier methods, and was refined by full-matrix least-squares calculations using the program *ORFLS* (Busing, Martin & Levy, 1962). The function minimized was $\sum w||F_o|-s|F_c||^2$. The weights were initially set at $w=1$ for all observed reflexions. Standard tabulations of atomic scattering factors were used for C,N,O,S (Cromer & Waber, 1965) and H (Stewart, Davidson & Simpson, 1965). The real and imaginary parts of the anomalous dispersion correction were applied to the scattering curve for S (Cromer, 1965).

The seven independent hydrogen atoms were located in an $(F_o-F_c)$ synthesis at an intermediate stage of the refinement. Refinement with anisotropic thermal parameters for all the non-hydrogen atoms and isotropic thermal parameters for the hydrogen atoms converged with $R=\sum||F_o|-s|F_c||/\sum|F_o|=0{\cdot}043$. When the correct weights $w=1/\sigma^2(F)$ calculated from the random and systematic errors of the observations (see above) were introduced, the residual $R$ decreased only to 0·042 but there was an immediate drop of about 30% in the estimated standard deviations of the refined variables. The value of the goodness-of-fit parameter $[\sum w||F_o|-s|F_c||^2/(m-n)]^{1/2}$ was 1·3, and there were no systematic trends in the values of $\langle w||F_o|-s|F_c||^2\rangle$ averaged over ranges of $|F|$ or $\sin\theta/\lambda$. In the final refinement cycle, no parameter changed by more than 0·1 standard deviation for a non-hydrogen atom, or more than 0·4 standard deviation for a hydrogen atom. A final $(F_o-F_c)$ synthesis showed no features of magnitude greater than 0·2 e Å$^{-3}$. The final values of the atomic positional and thermal parameters are shown in Table 1.*

## Description of the structure

The dimensions of the imidazolium cations, sulphate anions and water molecules are shown in Fig. 1, which also explains the atomic numbering scheme. The atomic labels C(1), C(2), C(3) and N(2) refer to the atoms C-2, C-4, C-5 and N-3 in the chemical convention (see p. 2833).

The imidazolium ions occupy eightfold general positions in the unit cell. They are planar within the limits of precision of the analysis. The imidazole nitrogen atoms N(1) and N(3) form strong and approximately linear N–H···O hydrogen bonds to a sulphate ion (2·78 Å) and to a water molecule (2·73 Å), respectively (Table 2). The directions of these hydrogen bonds lie at angles of 3° and 15° from the imidazolium plane.

In space group $P4_12_12$ there are eight $2_1$ axes, four being parallel to the $x$ axis and four parallel to the $y$ axis, respectively. They are related in pairs by the twofold rotation axes which run at 45° to these directions. The centres of gravity of the imidazolium rings lie close to four of the $2_1$ axes. For instance, the imidazolium ions at $x,y,z$ and $\frac{1}{2}+x,\frac{1}{2}-y,\frac{3}{4}-z$ have their centres close to the $2_1$ axis at $y=\frac{1}{4}$, $z=\frac{3}{8}$. Three other pairs of imidazolium ions are related by the $2_1$ axes at $x=\frac{1}{4}$, $z=\frac{5}{8}$; $y=\frac{3}{4}$, $z=\frac{7}{8}$; and $x=\frac{3}{4}$, $z=\frac{1}{8}$. The normals to the imidazolium planes are inclined at 23° to the

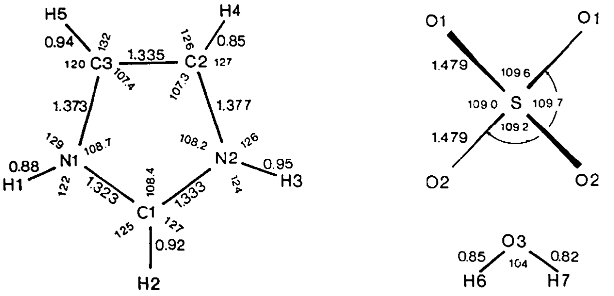

Fig. 1. Dimensions of imidazolium ions, sulphate ions and water molecule in imidazolium sulphate dihydrate. [E.s.d.'s: $d$(C–N, C–C) = 0·004, $d$(S–O) = 0·002, $d$(N–H, C–H) = 0·04, $d$(O–H) = 0·05 Å; $\theta$(O–S–O) = 0·1, $\theta$(C–N–C, C–C–N) = 0·2°]. Superscript ′ has the significance indicated in Table 2.

Table 1. *Atomic positional parameters for* $(ImH_2)_2SO_4.2H_2O$, *with anisotropic thermal parameters for non-hydrogen atoms and isotropic thermal parameters for hydrogen atoms*

Numbers in parentheses are estimated standard deviations in the units of the least significant digit of the preceding number. The form of the anisotropic temperature parameter is $\exp\left(-[h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23}]\right)\times 10^{-4}$.

| | $10^4x$ | $10^4y$ | $10^4z$ | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| S | 2263 (1) | 2263 (1) | 0 | 57 (1) | 57 (1) | 8 (0) | 3 (1) | −1 (1) | 1 (1) |
| O(1) | 2548 (2) | 467 (2) | 152 (1) | 124 (4) | 65 (2) | 16 (0) | 21 (3) | 1 (1) | 1 (1) |
| O(2) | 2773 (3) | 3262 (2) | 625 (1) | 185 (4) | 81 (3) | 15 (0) | −9 (3) | −24 (1) | −5 (1) |
| O(3) | 3030 (3) | 1636 (3) | 1926 (1) | 224 (6) | 103 (3) | 15 (1) | −31 (3) | −19 (1) | 5 (1) |
| N(1) | 1057 (4) | 3489 (3) | 3407 (1) | 117 (5) | 102 (4) | 17 (1) | −3 (3) | 13 (2) | 13 (1) |
| N(2) | 22 (3) | 2794 (4) | 4422 (1) | 119 (4) | 147 (5) | 16 (1) | 23 (4) | 11 (1) | 10 (2) |
| C(1) | 513 (4) | 4082 (4) | 4021 (2) | 128 (6) | 100 (5) | 20 (1) | 19 (4) | 2 (2) | 3 (2) |
| C(2) | 278 (4) | 1340 (4) | 4043 (2) | 117 (5) | 101 (5) | 25 (1) | −9 (4) | 17 (2) | 13 (2) |
| C(3) | 913 (4) | 1774 (4) | 3414 (2) | 126 (6) | 94 (5) | 23 (1) | −3 (4) | 19 (2) | −2 (2) |

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* A list of the observed and calculated structure amplitudes has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31090 (12 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1 (*cont.*)

| | $10^3x$ | $10^3y$ | $10^3z$ | $B$ |
|---|---|---|---|---|
| H(1) | 141 (4) | 414 (4) | 306 (2) | 0·5 (6) |
| H(2) | 48 (5) | 520 (5) | 414 (2) | 1·5 (8) |
| H(3) | −59 (4) | 290 (4) | 485 (2) | 1·6 (8) |
| H(4) | 13 (5) | 35 (5) | 419 (2) | 1·8 (8) |
| H(5) | 119 (6) | 114 (5) | 301 (2) | 2·0 (8) |
| H(6) | 281 (5) | 61 (5) | 184 (2) | 1·0 (7) |
| H(7) | 291 (5) | 210 (5) | 154 (2) | 2·8 (9) |

$2_1$ axes which intersect them, so that the angles between the normals to successive imidazolium ions along each axis are 46°. There are no contacts shorter than 3·37 Å between imidazolium ions.

The sulphate ions have almost perfect tetrahedral symmetry (Fig. 1). The S atoms lie on fourfold special positions with point symmetry 2. Each of the symmetry-related oxygen atoms O(1) and O(1′) is hydrogen

Table 2. *Hydrogen bonds in imidazolium sulphate dihydrate*

Code for symmetry-related atoms:

| Superscript | Atom at | Superscript | Atom at |
|---|---|---|---|
| None | $x \quad y \quad z$ | iii | $\frac{1}{2}-x \quad -\frac{1}{2}+y \quad \frac{1}{4}-z$ |
| ′ | $y \quad x \quad -z$ | iv | $-\frac{1}{2}+x \quad \frac{1}{2}-y \quad \frac{3}{4}-z$ |
| ″ | $\frac{1}{2}-x \quad \frac{1}{2}+y \quad \frac{1}{4}-z$ | v | $\frac{1}{2}+x \quad \frac{1}{2}-y \quad \frac{3}{4}-z$ |

(*a*) Hydrogen-bond lengths

[E.s.d.'s: $d$(X–H), $d$(H···Y), 0·05 Å; $d$(X···Y), 0·005 Å; $\theta$(X–H···Y), 3°].

| Bond | Equivalent bond | Distances (Å) | | | Angle |
|---|---|---|---|---|---|
| $X$–H···$Y$ | $Y$···H–$X$ | $X$–H | H···$Y$ | $X$···$Y$ | $X$–H···$Y$ |
| $N(1)–H(1)\cdots O(1'')$ | $O(1)\cdots H(1^{iii})–N(1^{iii})$ | 0·88 | 1·90 | 2·78 | 173° |
| $N(2)–H(3)\cdots O(3^{iv})$ | $O(3)\cdots H(3^{v})—N(2^{v})$ | 0·95 | 1·79 | 2·73 | 170 |
| $O(3)–H(7)\cdots O(2)$ | | 0·82 | 1·96 | 2·78 | 176 |
| $O(3)–H(6)\cdots O(2^{iii})$ | $O(2)\cdots H(6'')–O(3'')$ | 0·85 | 1·93 | 2·77 | 167 |

(*b*) Angles at hydrogen-bonded atoms

[E.s.d.'s = 0·2°].

| | | | |
|---|---|---|---|
| $C(1)–N(1)\cdots O(1'')$ | 123·8° | $S——O(2)\cdots O(3)$ | 118·1° |
| $C(3)–N(1)\cdots O(1'')$ | 127·4 | $S——O(2)\cdots O(3'')$ | 115·6 |
| $C(1)–N(2)\cdots O(3^{iv})$ | 119·5 | $O(3)\cdots O(2)\cdots O(3'')$ | 120·1 |
| $C(2)–N(2)\cdots O(3^{iv})$ | 129·7 | $N(2^{v})\cdots O(3)\cdots O(2)$ | 131·8 |
| $S——O(1)\cdots N(1^{iii})$ | 118·3 | $N(2^{v})\cdots O(3)\cdots O(2^{iii})$ | 108·9 |
| | | $O(2)\cdots O(3)\cdots O(2^{iii})$ | 114·0 |

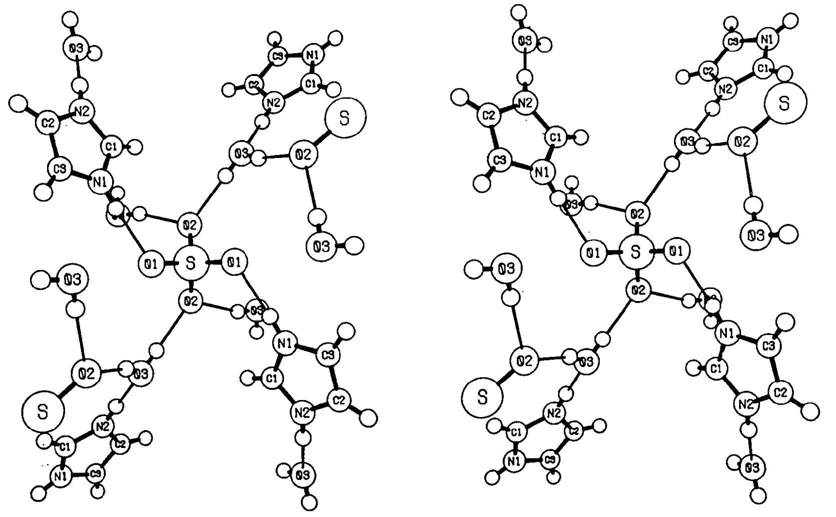

Fig. 2. Stereoscopic drawing of the imidazolium sulphate dihydrate structure, viewed from the origin along the twofold rotation axis at $x=y$, $z=0$. The $x$ and $y$ axes point away from the observer at 45° from the line of sight, and the $xy$ plane slopes upwards from left to right at 9·5° from the horizontal. The O(3)···O(2)···O(3)···O(2)···helix in the top right-hand quadrant of the drawing is generated by the $2_1$ axis at $x=\frac{1}{4}$, $z=\frac{1}{8}$, and the corresponding helix in the bottom left-hand quadrant by the $2_1$ axis at $y=\frac{1}{4}$, $z=-\frac{1}{8}$.

bonded to an imidazole nitrogen atom N(1) (see above, and Fig. 2). The other two sulphate oxygens O(2) and O(2′) are each the proton acceptor in hydrogen bonds with two water molecules O(3), (2·78, 2·77 Å) (Table 2). Successive pairs of the atoms listed in Table 2, ···O(3″)···O(2)···O(3)···O(2‴)···, are related by the $2_1$ axis at $x=\frac{1}{4}$, $z=\frac{1}{8}$. Similar helices lie along the $2_1$ axes at $y=\frac{1}{4}$, $z=\frac{7}{8}$ $(=-\frac{1}{8})$; $x=\frac{3}{4}$, $z=\frac{5}{8}$; and $y=\frac{3}{4}$, $z=\frac{3}{8}$. Thus the ···H–O–H···O(sulphate)···H–O–H···O(sulphate)··· helices are generated by those four $2_1$ axes which were not mentioned in the description of the imidazolium ion arrangement. The helix at $z=-\frac{1}{8}$ includes O(2′), the second of the two sulphate oxygens which are bonded to the S atom at $z=0$. The hydrogen-bonded helices at $z=-\frac{1}{8}$ and $z=\frac{1}{8}$ are related, like O(2′) and O(2), by the twofold axis at $z=0$.

In Table 3 we have collected the most precise structural data available for both protonated (positively charged) and unprotonated (neutral) imidazole molecules and groups. In discussing these data we shall use the abbreviations $ImH_2^+$ and ImH, respectively, and the same numbering scheme as in Fig. 1. The results of a number of less precise structure analyses of histidinium and histaminium salts have been tabulated by Yamane, Ashida & Kakudo (1973), and are not repeated here.

There is good agreement between the dimensions of the imidazolium ions in $(ImH_2)_2SO_4.2H_2O$ and in two other compounds which contain $ImH_2^+$ ions or groups. Table 3 shows that these dimensions are systematically different from those of neutral ImH molecules or groups. As already noted by Madden, McGandy & Seeman (1972), the angle N–C–N [108·5(1)°]* is equal to the two angles C–N–C [108·6(1)°] in $ImH_2^+$, but the three angles are unequal in ImH {N–C–N = 111·7(3)°, C–N(1)–C = 105·1(2)°, C–N(2)–C = 107·1(1)°}. We now find that the angles N–C–C are also equal in $ImH_2^+$ [107·1(2)°], and unequal in ImH {N(1)–C–C = 109·9(2)°, N(2)–C–C = 106·2(2)°}. The bonds N(1)–C(1) and N(2)–C(1) are structurally as well as chemically equivalent in $ImH_2^+$ [1·324(4) Å], and so also are the bonds N(1)–C(3) and N(2)–C(2) [1·374(2) Å]. In ImH, on the other hand, the bond N(1)–C(1) is significantly shorter at the 99% confidence level than N(2)–C(1) [1·324(3), 1·346(4) Å], the bond N(1)–C(3) is longer than N(2)–C(2) [1·379(2), 1·370(2) Å], and the C–C bond is longer than the corresponding bond in $ImH_2^+$ [1·359(1), 1·343(4) Å].

The type of parallel stacking of imidazolium ions which occurs in $(ImH_2)H_2PO_4$ (Blessing & McGandy, 1972) is not found in the present structure. The orientations of the $ImH_2^+$ ions appear to be determined by the hydrogen bonds which link the $ImH_2^+$ ions to the framework of sulphate ions and water molecules. These hydrogen bonds are approximately linear (Table 2). The N(imidazole)–H···O(water) bond length, 2·73 Å, is in the range attributed to strong N–H···O interactions (Hamilton & Ibers, 1968). The formation of strong N–H···O bonds by $ImH_2^+$ groups has been observed previously in structures where the proton

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* A value in square brackets is the unbiased mean $\bar{x}$, of the relevant quantities $x_i$ in Table 3, followed by the standard deviation $s=[\sum_{i=1}^{n}(x_i-\bar{x})^2/n(n-1)]^{1/2}$.

Table 3. *The dimensions of protonated and neutral imidazole molecules and groups*

The numbering scheme is the same as in Fig. 1. In the neutral imidazole molecules and groups the ‘pyridine’ nitrogen is at N(1) and the ‘pyrrole’ nitrogen at N(2). The point of attachment to the side chains in histidine and histamine is assumed to be at C(3).

(*a*) Bond lengths (in Å)

| | Protonated imidazole | | | Neutral imidazole | | |
|---|---|---|---|---|---|---|
| Compound | *A* | *B* | *C* | *D* | *E* | *F* |
| N(1)–C(1) | 1·323 | 1·332 | 1·320 (4) | 1·326 | 1·319 | 1·327 |
| N(2)–C(1) | 1·333 | 1·314 | | 1·349 | 1·350 | 1·339 |
| N(1)–C(3) | 1·373 | 1·379 | 1·367 (12) | 1·378 | 1·377 | 1·382 |
| N(2)–C(2) | 1·377 | 1·373 | | 1·369 | 1·367 | 1·374 |
| C(2)–C(3) | 1·335 | 1·349 | 1·345 (7) | 1·358 | 1·359 | 1·361 |
| Mean e.s.d. | 0·004 | 0·004 | * | 0·005 | 0·005 | 0·003 |

(*b*) Bond angles (°)

| | *A* | *B* | *C* | *D* | *E* | *F* |
|---|---|---|---|---|---|---|
| N(1)–C(1)–N(2) | 108·4 | 108·5 | 108·7 (3) | 111·3 | 111·7 | 112·2 |
| C(1)–N(1)–C(3) | 108·7 | 108·6 | 108·6 (6) | 105·4 | 105·0 | 104·9 |
| C(1)–N(2)–C(2) | 108·2 | 109·1 | | 107·2 | 107·2 | 106·9 |
| N(1)–C(3)–C(2) | 107·4 | 106·5 | 107·0 (3) | 109·8 | 110·2 | 109·6 |
| N(2)–C(2)–C(3) | 107·3 | 107·2 | | 106·3 | 105·9 | 106·4 |
| Mean e.s.d. | 0·2 | 0·2 | * | 0·3 | 0·3 | 0·2 |

Compounds and references: $(ImH_2)^+$ = imidazolium, $(HsnH)^{2+}$ = histaminium. (*A*) $(ImH_2)SO_4.2H_2O$ (present work). (*B*) (HsnH) $(H_2PO_4)_2.H_2O$ (Veidis, Palenik, Schaffrin & Trotter, 1969). (*C*) $(ImH_2)H_2PO_4$ (Blessing & McGandy, 1972). (*D*) Imidazole, X-ray data (Martínez-Carrera, 1966). (*E*) Imidazole, neutron data (Bell & Freeman, 1974, unpublished work). (*F*) L-Histidine (Madden, McGandy & Seeman, 1972).

* E.s.d.'s for compound *C* are in parentheses.

acceptors in the bonds were phosphate oxygens (Veidis, Palenik, Schaffrin & Trotter, 1969; Blessing & McGandy, 1972).

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# Die Kristallstruktur von α-Tetrasilbertelluriddinitrat

Von E. Schultze-Rhonhof*

*Chemisches Institut der Universität Bonn, Bonn, Deutschland* (*BRD*)

(*Eingegangen am* 28. *Juli* 1974; *angenommen am* 3. *September* 1974)

The crystal structure of $\alpha$-$Ag_4Te(NO_3)_2$ has been determined from powder data (final $R$ value 10·5 %). The compound is cubic, space group $Pa3$ ($T_h^6$, No. 205) with 4 formula units per unit cell; $a = 8{\cdot}173$ (4) Å. The structure can be compared with that of $\alpha$-$Ag_2Te$. The tellurium atoms form a body-centred cell. The silver atoms and the nitrate groups are statistically distributed.

## Einleitung

Tempert man gepulverte und in ein Markröhrchen eingeschmolzene Kristalle von $\gamma$-$Ag_4Te(NO_3)_2$ sechs Monate bei 100 °C, dann wandeln sie sich über die $\beta$-Modifikation in $\alpha$-$Ag_4Te(NO_3)_2$ um (Schultze-Rhonhof & Bergerhoff, 1966). Die Verbindung wurde seinerzeit anhand einer Debije–Scherrer-Aufnahme, auf der zehn Linien erkennbar waren, charakterisiert und mit einer Gitterkonstanten $a = 4{,}07$ (2) Å indiziert (der in der Arbeit von 1966 angegebene Wert beruht auf einem Rechenfehler). Diese Indizierung führt aber zu Widersprüchen zwischen Dichte und Zellinhalt; die Elementarzelle enthält nur eine halbe Formeleinheit. Daher wurde auf länger belichteten Aufnahmen nach Hinweisen auf eine grössere Zelle gesucht. In der Tat fanden sich weitere 18 schwächere Linien, die auf eine Zelle mit doppelter Gitterkonstante führen.

Als Ausgangsmaterial für die Darstellung von $\alpha$-$Ag_4Te(NO_3)_2$ standen nur *ca* 2 mg $\gamma$-$Ag_4Te(NO_3)_2$ zur Verfügung. Leider gelang es nicht, Einkristalle zu gewinnen. Daher mussten die Gitterparameter der Substanz und die Intensitäten ihrer Reflexe aus Pulveraufnahmen bestimmt werden (Cu $K\alpha$-Strahlung, $\lambda = 1{,}5418$ Å; Kameradurchmesser = 57,3 mm, Filmanordnung nach Straumanis & Ieviņš (1936), $d$-Werte beobachtet: $5{,}67 \geq d \geq 0{,}7864$ Å). Auch der Versuch einer Strukturaufklärung musste mit Pulverdaten unternommen werden.

Die 28 auf den Aufnahmen beobachteten Linien liessen sich kubisch indizieren. Die Extrapolation nach $\frac{1}{2}(\cos^2\theta/\sin\theta + \cos^2\theta/\theta)$ (Azároff & Buerger, 1958) ergab $a = 8{,}173$ (4) Å. Daraus errechnet sich mit $n = 4$ und $M = 683{,}10$ die Röntgendichte $D_x = 8{,}31$ (1) g cm$^{-3}$. (Wegen der geringen Substanzmenge konnte die Dichte $D_o$ von $\alpha$-$Ag_4Te(NO_3)_2$ nicht gemessen werden; als Vergleichswert diente die Dichte der $\gamma$-Modifikation: $D_x(\gamma) = 7{,}2$ g cm$^{-3}$.) Die Intensitäten wurden mit einem Mikrodensitometer gemessen. Gerechnet wurde auf einer IBM 7090 mit der Programmkette BN-X-64 (Schultze-Rhonhof, 1966), die mit Ausnahme eines für Pulveraufnahmen verwendbaren Absorptionsprogrammes alle notwendigen Programme enthält. Wegen des

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* Neue Adresse: D53 Bonn, Amsterdamer Str. 15, Deutschland.