Acta Cryst. (1952). 5, 599

The Crystal Structure of Copper Proline Dihydrate

By A. McL. Mathieson and H. K. Welsh*

Chemical Physics Section, Division of Industrial Chemistry, Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia

(*Received* 1 *December* 1951 *and in revised form* 28 *January* 1952)

The crystal structure of copper proline dihydrate is monoclinic, $P2_1/n$, with two molecules of $Cu(C_5H_8O_2N)_2.2H_2O$ in a cell of dimensions, $a = 5\cdot62$, $b = 17\cdot85$, $c = 7\cdot13$ Å, $\beta = 108°$. The copper atom is surrounded in a square coplanar configuration by two nitrogen and two oxygen atoms (one from each carboxyl group) at distances 1·99 and 2·03 Å respectively. On a line approximately perpendicular to this plane are two water molecules at 2·52 Å.

The atoms 1, 2, 3 and 5 (N = 1) of the pyrrolidine ring of the proline molecule are coplanar and atom 4 lies at a distance of 0·60 Å from this plane. The carboxyl group attached to atom 2 is disposed in the *trans* position with respect to atom 4. The bond lengths are in general normal, the carbon–carbon distances being slightly shorter than 1·54 Å. The copper proline dihydrate units are bound by hydrogen bonds into layers parallel to (010), the stacking of these layers being governed by van der Waals approach distances of the ring methylene groups of adjacent layers.

Introduction

A sufficient number of structure analyses of α-amino acids has been completed to give the configuration of the group, $-CH.NH_3^+.COO^-$, with reasonable accuracy (Albrecht & Corey, 1939; Donohue, 1950; Shoemaker *et al.*, 1950). The importance of further work on amino acids lies in the elucidation of the detailed configuration of the associated *R* groups. Such information will be useful in the later analysis of simple peptides and in testing proposed model structures of proteins (e.g. Pauling, 1951) for the criteria of close packing and inter-chain bonding.

Proline and hydroxyproline are the only naturally occurring amino acids in which the α-nitrogen forms part of a ring system. These compounds constitute only a small proportion by weight of the majority of proteins, but collagen and gelatine contain 13% proline and 11% hydroxy-proline residues, together forming almost one quarter of the total number of residues (Chibnall, 1946). It is therefore of importance with respect to the structure of the two proteins to obtain the detailed configuration of these compounds. A brief account of the crystal structure of L-hydroxyproline has been given by Zussman (1951), and Donohue (private communication) has completed a three-dimensional analysis of this compound.

Since Wright & Cole (1949) indicated the hydroscopic nature of L-proline, it was decided to use the copper salt of DL-proline, which is more readily handled. The heavy-atom technique on α-amino acids has been previously utilized in the analysis of nickel glycine dihydrate and in an incomplete study of copper amino-butyrate (Stosick, 1945*a*, *b*).

Cell dimensions and space-group data for copper DL-proline dihydrate have been recorded in an earlier publication (Mathieson & Dawson, 1951).

Experimental

Crystals of the copper salt of DL-proline (DL-2-pyrrolidine carboxylic acid) were prepared by the action of a solution of proline on an excess of freshly precipitated copper carbonate, the resultant filtered solution being allowed to evaporate slowly. The crystals were deep-blue plates, the main face (010) having the shape of a parallelogram of which the longer side was parallel to the *a* and the shorter parallel to the *c* axis. Many crystals were multiple, although superficially well-formed, but these were readily detected on the optical goniometer.

Unit-cell dimensions were determined from rotating-crystal photographs and reflexions indexed on equi-inclination Weissenberg photographs. The crystal system is monoclinic, unit-cell dimensions being

$$a = 5\cdot62,\ b = 17\cdot85,\ c = 7\cdot13\ \text{Å},\ \beta = 108°\ .$$

The density, measured by flotation in a chloroform–bromoform mixture, was 1·61 g.cm.$^{-3}$, the value calculated for two units of $Cu(C_5H_8O_2N)_2.2H_2O$ per cell being 1·59 g.cm.$^{-3}$. Reflexions ($h0l$) for which $h+l$ is odd, and ($0k0$) for which k is odd, are absent. The space group is therefore $P2_1/n$.

Intensity data were recorded on zero-layer moving films about the *a*, *b* and *c* axes, using filtered copper radiation. Packs of four films were used and the intensity of reflexions were visually estimated by comparison with a standard scale. No correction was made for absorption in the crystal. The relative structure amplitudes were later placed on an absolute scale by comparison with the calculated values. For cal-

---

* Present address: Division of Electrotechnology, National Standards Laboratory, University Grounds, Chippendale, N.S.W., Australia.

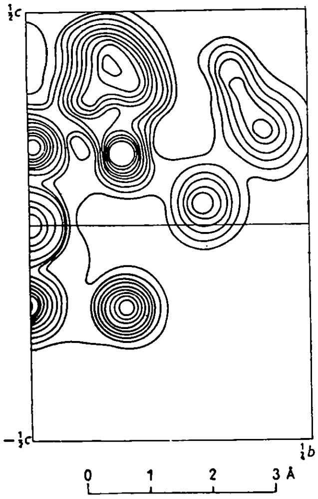

Fig. 1.

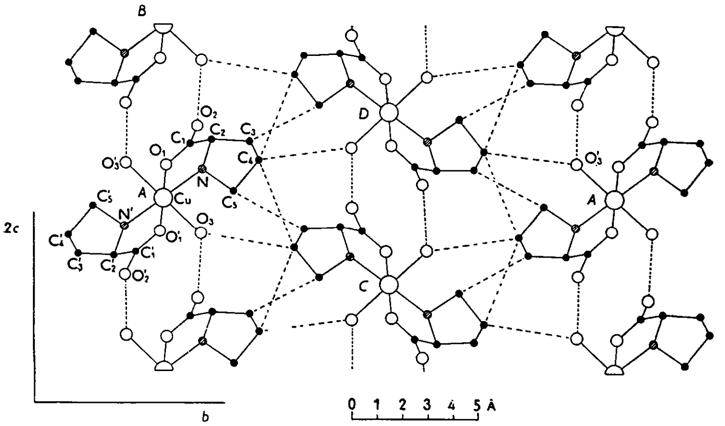

Fig. 2

Fig. 1. Final electron-density projection along the *a* axis. Contour interval: 1 e.Å^{-2} for carbon, nitrogen and oxygen atoms, beginning at the 2 e.Å^{-2} level; 10 e.Å^{-2} for the copper atom.

Fig. 2. Crystal structure of copper proline dihydrate projected down the *a* axis.

culation of the structure amplitudes, a temperature correction ($B = 2{\cdot}2 \times 10^{-16}$ cm.^{2}) was applied to the scattering factors for Cu^{2+}, O, N and C given in *Internationale Tabellen.* The Debye–Waller factor was obtained by the best fit with observed structure factors.

Fourier summations were computed by means of three-figure Beevers–Lipson strips at 6° intervals along the *b* axis and 12° intervals along the *a* and *c* axes. The contour maps of electron density were plotted by graphical interpolation.

Structure determination

For the space group, $P2_1/n$, there are four equivalent positions and, since there are only two copper atoms, these must be placed in the special positions, 0, 0, 0 and $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. Two proline molecules are associated with each copper atom lying at a centre of symmetry and it was assumed that the nitrogen atom and one oxygen atom (of the carboxyl group) of each proline molecule form a planar arrangement of bonds to the metal atom. The two water molecules were then situated above and below the planar group of bonds to provide octahedral co-ordination to the copper atom. The existence of marked cleavage in the crystal parallel to (010) suggested that the copper proline dihydrate units are bonded into layers extending parallel to (010) with only feeble van der Waals forces between layers. It was thus further assumed that the orientation of the pyrrolidine ring is approximately parallel to the *b* axis. A one-dimensional Fourier synthesis along the *b* axis was calculated, taking all terms positive. The resulting distribution was in reasonable agreement with the proposed model, from which the *z* parameter of each atom was estimated. The calculated values of (0*k*0) structure amplitudes permitted placing on an absoluet scale the $\{0kl\}$ and $\{hk0\}$ zonal reflexions and, by interpolation, the $\{h0l\}$ reflexions.

The a-axis projection

In the projections parallel to both the *a* and *c* axes, the copper atom does not contribute to the reflexions the sum of whose indices is odd, and these reflexions are dependent solely on the organic part of the molecule, $C_5H_8O_2N.H_2O$. Since the structure amplitudes have been placed on a roughly absolute scale, use was made of structure-factor graphs (Bragg & Lipson, 1936) rapidly to investigate proposed models. Testing the low-index reflexions combined with the known *z* parameters permitted the organic unit to be placed approximately. Calculation of the set of reflexions for which $k+l = 2n+1$ was utilized as a guide in making small parameter adjustments. When a satisfactory fit was achieved, successive Fourier syntheses were utilized to approximate more rapidly to the correct structure. The final electron-density map is shown in Fig. 1 and the corresponding diagram of the crystal structure in Fig. 2.

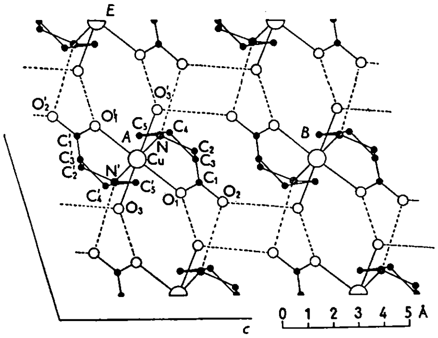

Fig. 3. Diagram of the crystal structure between the levels $+\frac{1}{4}b$ and $-\frac{1}{4}b$ projected on (010).

The b-axis projection

For the ($h0l$) reflexions, the copper atom determines the majority of signs, and the first Fourier synthesis was therefore computed with all signs positive. There is considerable overlap within the small area but the carboxyl group was located and, assuming a normal configuration, an approximate position for C_2 was derived. The information, although limited, was of use in dealing with the subsequent projection. The diagram of the crystal structure, showing only the atoms between levels $+\frac{1}{4}b$ and $-\frac{1}{4}b$, appears in Fig. 3.

The c-axis projection

From the information derived from the preceding projections, approximate siting of atoms was possible followed by small refinement to atomic parameters by means of structure-factor graphs. More precise atomic parameters were achieved by successive Fourier syntheses. The final electron-density map is shown in Fig. 4 and the corresponding diagram of the crystal structure in Fig. 5.

The atomic parameters of the assymetric unit are recorded in Table 1 and the calculated interatomic distances and angles in Table 2, the probable errors being 0·04 Å and 5° respectively. The dashed atoms are related to the undashed atoms by the centre of symmetry at the origin. Where intermolecular distances are discussed, the individual atoms are designated in brackets by a capital letter referring to the particular molecule (see Figs. 2, 3 and 5).

The comparison of observed with calculated structure amplitudes is given in Table 3, the percentage discrepancies for the three principal zones being 16·2, 19·9 and 15·8 respectively, with a weighted mean value of 16·5.

Discussion

There are two proline and two water molecules associated with each copper atom, which forms nearly square covalent bonds to the nitrogen atoms and the oxygen atoms, O_1, at distances 1·99 and 2·03 Å respectively. There are two longer bonds, 2·52 Å, to the water molecules disposed in a line approximately normal to the plane containing the short bonds. The distortion of the angle ∠ N–Cu–O_1 from the expected value 90° to the measured value 82° arises from the restricting influence of the configuration of the proline molecule. Similarly, intermolecular bonding modifies the angles ∠ O_3–Cu–N and ∠ O_3–Cu–O_1 to 93° and 79° respectively. The distorted octahedral arrangement

Table 1. *Atomic co-ordinates*

Atom	x	y	z
Cu	0	0	0
O_1	−0·211	0·004	0·185
O_2	−0·311	0·072	0·400
O_3	−0·350	0·085	−0·190
N	0·176	0·085	0·167
C_1	−0·169	0·057	0·303
C_2	0·060	0·104	0·328
C_3	0·011	0·188	0·322
C_4	0·196	0·210	0·219
C_5	0·176	0·156	0·049

Table 2. *Interatomic distances and bond angles*

(*a*)	Cu–O_1	2·03 Å	∠ N–Cu–O_1	82°
	Cu–N	1·99 Å	O_3–Cu–O_1	79°
	Cu–O_3	2·52 Å	O_3–Cu–N	93°
	C_1–O_1	1·24 Å	O_1–C_1–O_2	122°
	C_1–O_2	1·24 Å	C_2–C_1–O_1	120°
	C_1–C_2	1·50 Å	C_2–C_1–O_2	118°
	C_2–C_3	1·52 Å	C_1–C_2–C_3	112°
	C_3–C_4	1·50 Å	C_1–C_2–N	108°
	C_4–C_5	1·52 Å	C_2–C_3–C_4	97°
	C_5–N	1·53 Å	C_3–C_4–C_5	109°
	N–C_2	1·52 Å	C_4–C_5–N	96°
			C_5–N–C_2	108°
			N–C_2–C_3	108°
(*b*)	N(*A*)–O_2(*E*)	2·86 Å	Cu–N–C_2	112°
	O_3'(*A*)–O_2'(*B*)	3·00 Å	Cu–N–C_5	113°
	O_3'(*A*)–O_1(*E*)	2·94 Å	Cu–O_1–C_1	116°
(*c*)	C_4(*A*)–C_4'(*C*)	4·06 Å		
	C_5(*A*)–C_3'(*C*)	3·96 Å		
	O_3(*A*)–C_4'(*C*)	3·77 Å		

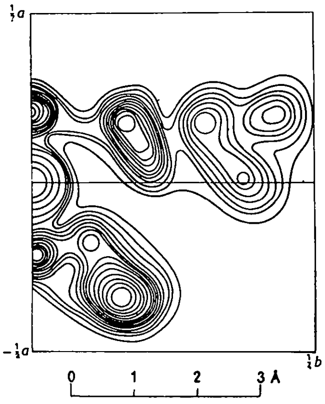

Fig. 4.

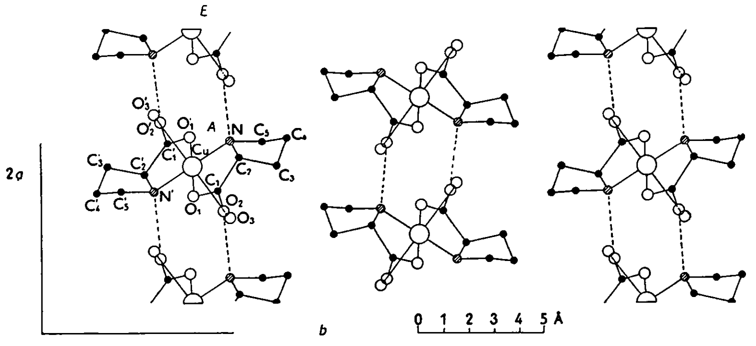

Fig. 5.

Fig. 4. Final electron-density projection along the *c* axis. Contour intervals as in Fig. 1.
Fig. 5. Diagram of the crystal structure viewed down the *c* axis.

Table 3. *Comparison of observed and calculated structure amplitudes*

hkl	F_o	F_c
020	106	113
040	29	28
060	4	8
080	4	6
0,10,0	61	61
0,12,0	58	57
0,14,0	34	43
0,16,0	13	17
0,18,0	14	10
0,20,0	15	13
0,22,0	13	8
011	61	58
021	70	−73
031	35	45
041	5	−3
051	57	55
061	30	−29
071	65	60
081	13	8
091	38	37
0,10,1	8	8
0,11,1	32	29
0,12,1	< 4	−2
0,13,1	38	34
0,14,1	< 6	6
0,15,1	15	14
0,16,1	< 7	−3
0,17,1	6	8
0,18,1	< 6	−1
0,19,1	15	13
0,20,1	< 5	−5
0,21,1	10	10
0,22,1	< 4	−4
0,23,1	6	10
012	14	18
022	19	23
032	40	35
042	21	26
052	20	20
062	66	50
072	< 4	−3
082	36	37
092	13	−21
0,10,2	< 5	−1
0,11,2	< 5	−7
0,12,2	12	18
0,13,2	< 6	−2
0,14,2	15	14
0,15,2	10	−8
0,16,2	13	12
0,17,2	< 6	4
0,18,2	14	13
0,19,2	< 5	1
0,20,2	8	6
0,21,2	< 4	1
0,22,2	< 3	4
013	18	20
023	26	−17
033	10	11
043	20	−18
053	26	31
063	10	9
073	29	29
083	3	−1
093	4	5
0,10,3	15	16
0,11,3	11	7
0,12,3	4	7
0,13,3	< 4	1
0,14,3	7	−6
0,15,3	4	10
0,16,3	< 6	−4
0,17,3	16	18
0,18,3	< 5	−2
0,19,3	14	12
0,20,3	< 4	2
0,21,3	12	8
014	6	−12
024	21	14
034	10	−7
044	35	32
054	7	9
064	36	37
074	< 5	−1
084	25	26
094	< 5	2
0,10,4	17	16
0,11,4	< 6	6
0,12,4	6	9
0,13,4	< 6	−4
0,14,4	13	13
0,15,4	3	6
0,16,4	12	8
0,17,4	< 5	4
0,18,4	12	11
0,19,4	< 4	3
0,20,4	12	10
015	31	42
025	4	4
035	27	29
045	7	9
055	18	16
065	< 6	−1
075	15	11
085	7	−6
095	21	23
0,10,5	< 6	−2
0,11,5	24	23
0,12,5	4	3
0,13,5	25	26
0,14,5	< 6	0
0,15,5	14	15
0,16,5	< 5	−1
0,17,5	4	3
0,18,5	< 4	3
016	< 6	−2
026	18	22
036	9	6
046	17	13
056	7	4
066	10	13
076	6	−2
086	14	14
096	< 6	−3
0,10,6	20	21
0,11,6	< 6	0
0,12,6	18	16
0,13,6	< 5	2
0,14,6	7	6
0,15,6	< 4	−6
0,16,6	5	7
017	9	14
027	< 6	−4
037	10	15
047	< 6	4
057	5	7
067	< 5	1
077	4	7
087	< 5	−3
097	3	5
0,10,7	< 4	−1
0,11,7	4	7
0,12,7	< 4	−4
0,13,7	< 3	7
018	< 1	0
028	< 4	4
038	3	6
048	6	9
058	< 4	−2
068	4	5
078	< 4	−1
088	4	8
$\bar{7}01$	< 6	10
$\bar{5}01$	26	27
$\bar{3}01$	22	26
$\bar{1}01$	60	62
101	32	38
301	15	−11
501	24	30
$\bar{6}02$	< 10	5
$\bar{4}02$	17	22
$\bar{2}02$	36	37
002	25	−20
202	26	−20
402	24	21
602	< 7	13
$\bar{7}03$	< 7	16
$\bar{5}03$	14	12
$\bar{3}03$	41	45
$\bar{1}03$	47	48
103	58	57
303	26	30
503	< 9	6
$\bar{6}04$	11	13
$\bar{4}04$	28	28
$\bar{2}04$	25	38
004	19	19
204	23	17
404	< 9	9
$\bar{7}05$	< 5	3
$\bar{5}05$	14	16
$\bar{3}05$	23	17
$\bar{1}05$	9	18
105	< 10	7
305	16	22
$\bar{6}06$	< 7	16
$\bar{4}06$	13	10
006	25	35
$\bar{2}06$	15	26
206	< 9	14
$\bar{5}07$	< 8	4
$\bar{3}07$	15	20
$\bar{1}07$	17	23
107	< 9	5
$\bar{4}08$	< 7	6
$\bar{2}08$	16	13
008	< 4	−3

Table 3 (*cont.*)

hkl	F_o	F_c
110	81	79
120	5	4
130	18	26
140	64	58
150	72	63
160	16	15
170	46	44
180	< 3	-1
190	43	38
1,10,0	11	-15
1,11,0	38	32
1,12,0	< 4	-7
1,13,0	24	14
1,14,0	14	-3
1,15,0	16	18
1,16,0	< 5	4
1,17,0	17	18
1,18,0	12	13
1,19,0	16	19
1,20,0	< 4	1
1,21,0	13	15
1,22,0	< 3	4
200	16	-12
210	47	-41
220	< 3	5
230	52	-39
240	24	26
250	8	-6
260	50	43
270	22	18
280	35	34
290	26	30
2,10,0	25	25
2,11,0	12	7
2,12,0	10	6
2,13,0	14	-8
2,14,0	< 5	-4
2,15,0	15	-16
2,16,0	16	17
2,17,0	< 4	-2
2,18,0	11	12
2,19,0	4	3
2,20,0	11	11
2,21,0	6	4
310	28	28
320	6	-2
330	21	24
340	6	-8
350	12	9
360	< 4	2
370	11	8
380	< 4	-1
390	21	19
3,10,0	5	-1
3,11,0	25	30
3,12,0	< 5	-5
3,13,0	13	13
3,14,0	< 5	1
3,15,0	14	13
3,16,0	6	2
3,17,0	14	13
3,18,0	< 4	-4
3,19,0	< 4	0
3,20,0	< 3	-3
400	21	23
410	13	14
420	20	23
430	13	10
440	32	34
450	10	5
460	38	37
470	< 5	-3
480	25	24
490	8	-13
4,10,0	21	24
4,11,0	6	11
4,12,0	15	13
4,13,0	< 5	-2
4,14,0	15	13
4,15,0	18	13
4,16,0	17	18
4,17,0	8	4
4,18,0	12	13
510	25	27
520	8	-8
530	16	18
540	8	-10
550	27	30
560	< 2	-2
570	16	20
580	6	7
590	16	20
5,10,0	6	4
5,11,0	13	17
5,12,0	< 4	-1
5,13,0	14	13
5,14,0	8	2
5,15,0	12	12
5,16,0	< 2	2
600	24	29
610	4	-5
620	16	16
630	< 4	-1
640	9	9
650	< 4	0
660	< 4	6
670	< 4	0
680	< 4	2
690	< 3	4
6,10,0	6	10
6,11,0	< 3	0
710	< 2	5
720	< 2	-2
730	< 2	6

of short and long bonds around the copper atom is in agreement with the stereochemistry of divalent copper observed in other compounds (Wells, 1951).

In the proline molecule, the atoms C_2, C_3, N and C_5 of the pyrrolidine ring are coplanar within 0·06 Å, while C_4 lies at a distance of 0·60 Å from this plane. The carboxyl group $C_1O_1O_2$ is situated in the *trans* position to C_4 with respect to the plane of the ring. The carbon–oxygen distances are 1·24 Å. The mean carbon–carbon bond length, 1·51 Å, is slightly smaller than the normal value, 1·54 Å. The carbon–nitrogen distance of 1·52 Å is normal (Donohue, 1950; Shoemaker *et al.*, 1950), the nitrogen atom lying at a distance of 0·21 Å from the plane through C_2, C_1, O_1 and O_2, a value similar to that observed in nickel glycine dihydrate, 0·27 Å. The remaining bond angles within the pyrrolidine ring, shown in Table 2, are tetrahedral with the exception of $\angle\, C_4–C_5–N$ and $\angle\, C_4–C_3–C_2$ which are unusually small, 96° and 97° respectively. This diminution of the bond angle is correlated with the large displacement of C_4, 0·60 Å, from the main plane of the ring. For L-hydroxyproline a smaller value of 0·48 Å is quoted by Donohue (private communication), but it may be that the strong intermolecular hydrogen bonding by the hydroxyl group in hydroxyproline produces this difference.

The copper proline dihydrate units are maintained in a stable layer structure parallel to (010) by a hydrogen bond $N(A)–O_2(E)$, 2·86 Å, and by weaker hydrogen bonds, $O_3'(A)–O_1(E)$, 2·94 Å, and $O_3'(A)–O_2'(B)$, 3·00 Å (Figs. 3 and 5). If one hydrogen of the water molecule $O_3'(A)$ is directed towards $O_1(E)$ and the other towards $O_2'(B)$, Fig. 3, then one 'lone pair' of electrons (Fowler & Bernal, 1933*a*, *b*) is directed towards the copper atom and the other towards $C_4(D)$, Fig. 2. The stacking of the layers parallel to the *b* axis is governed by the close approaches, $O_3(A)–C_4'(C)$, 3·77 Å, $C_4(A)–C_4'(C)$, 4·06 Å, and $C_5(A)–C_3'(F)$, 3·96 Å, shown in Fig. 2.

References

Albrecht, G. A. & Corey, R. B. (1939). *J. Amer. Chem. Soc.* **61**, 1087.

Bragg, W. L. & Lipson, H. (1936). *Z. Krystallogr.* **95**, 323.

Chibnall, A. C. (1946). *J. Int. Soc. Leath. Chem.* **30**, 1.

Donohue, J. (1950). *J. Amer. Chem. Soc.* **72**, 949.

Fowler, R. H. & Bernal, J. D. (1933*a*). *Trans. Faraday Soc.* **29**, 1049.

FOWLER, R. H. & BERNAL, J. D. (1933*b*). *J. Chem. Phys.* **1**, 515.
Internationale Tabellen zur Bestimmung von Kristallstrukturen (1935). Berlin: Borntraeger.
MATHIESON, A. McL. & DAWSON, B. (1951). *Acta Cryst.* **4**, 475.
PAULING, L. (1951). *Proc. Nat. Acad. Sci., Wash.* **37**, 235.
SHOEMAKER, D. P., DONOHUE, J., SCHOMAKER, V. & COREY, R. B. (1950). *J. Amer. Chem. Soc.* **72**, 2328.
STOSICK, A. J. (1945*a*). *J. Amer. Chem. Soc.* **67**, 362.
STOSICK, A. J. (1945*b*). *J. Amer. Chem. Soc.* **67**, 365.
WELLS, A. F. (1951). *Acta Cryst.* **4**, 200.
WRIGHT, B. A. & COLE, P. A. (1949). *Acta Cryst.* **2**, 129.
ZUSSMAN, J. (1951). *Acta Cryst.* **4**, 72.

Acta Cryst. (1952). **5**, 604

The Structure of Mercuric Amidobromide

By LIES NIJSSEN AND WILLIAM N. LIPSCOMB

School of Chemistry, University of Minnesota, Minneapolis 14, Minnesota, U.S.A.

(*Received* 1 *May* 1952)

The structure of $HgNH_2Br$, similar to that of $HgNH_2Cl$, consists of infinite chains of alternating Hg and NH_2^+ with linear bonds about Hg and tetrahedral bonds about N. The unit cell is orthorhombic, C_{2v}^1–$P2mm$, with $a = 5{\cdot}439$, $b = 4{\cdot}487$ and $c = 6{\cdot}761$ Å. The Hg–N bond distance is 2·07 Å. The N–H ··· Br distance is 3·36 Å, based on the assumption that the Br ··· Br distance is 3·90 Å.

Because the structure of orthorhombic $HgNH_2Cl$ was determined from powder photographs only (Lipscomb, 1951*b*), and because of the relatively small scattering of N and Cl as compared with Hg, we felt that an investigation of $HgNH_2Br$ should be made. The results of this investigation, which is part of more general chemical studies of the reactions of Hg(*I*) compounds with ammonia, show that $HgNH_2Br$ has the same structure as that proposed for $HgNH_2Cl$. In addition, values have been determined for the Hg–N bond distance, and for the N–H ··· Br hydrogen-bond distance.

A sample of $HgNH_2Br$ was prepared by the reaction of 150 ml. of 14N aqueous ammonia with 2 g. of Hg_2Br_2. The diffraction pattern from this powdered sample was taken by means of a General Electric XRD-3 unit with the use of a Geiger counter for recording the diffracted Cu $K\alpha$ radiation. Integrated intensities were measured from the tracing by the usual method.

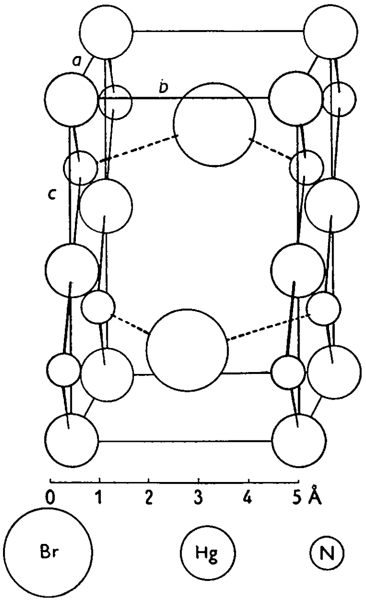

Fig. 1. The structure of $HgNH_2Br$.

The diffraction pattern was satisfactorily indexed on an orthorhombic unit cell with

$$a = 5{\cdot}439,\ b = 4{\cdot}487 \quad \text{and} \quad c = 6{\cdot}761 \text{ Å},$$

all $\pm 0{\cdot}005$ Å ($\lambda = 1{\cdot}5418$ Å). Comparison of these unit-cell dimensions with those of $HgNH_2Cl$ confirms the proposed atomic arrangement (Fig. 1) in a striking way. The values for $HgNH_2Br$ are greater by 0·27 Å for *a*, 0·13 Å for *b*, and 0·07 Å for *c* than those for $HgNH_2Cl$. It should be noted that binding in the *a* direction is weakest, being chiefly dispersion forces, binding in the *b* direction is by hydrogen bonding, while binding in the *c* direction occurs through the relatively strong covalent bonds.

Comparison of the observed and calculated spacings and intensities is shown in Table 1. Calculated intensities include the contributions of all atoms. The usual Lorentz, polarization and multiplicity factors have been included. Atomic positions and parameters are: