

# The Crystal Structure of 11-Amino-Undecanoic Acid Hydrobromide Hemihydrate

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11-Amino-undecanoic acid hydrobromide hemihydrate is monoclinic, space group  $A2/a$ , with eight molecules in a unit cell of dimensions  $a = 11.08$ ,  $b = 5.27$ ,  $c = 50.60$  Å,  $\beta = 90^\circ 42'$ . Electron-density projections along the  $a$  and  $b$  axes show that dimeric association between adjacent carboxyl groups occurs, and that the water molecules, situated on twofold axes, are involved in hydrogen bonding with the bromide ions and the  $\text{NH}_3^+$  groups. The average distance between two alternate carbon atoms in the hydrocarbon chain is  $2.563$  Å, and the average C-C bond length is  $1.539$  Å (estimated standard deviation  $0.013$  Å). The hydrocarbon chain is planar but the terminal nitrogen is displaced from this plane. The angle of tilt of the molecules to the (001) plane is  $41^\circ 36'$ , alternate hydrocarbon chains being inclined in opposite directions and crossing each other.

## 1. Introduction

Largely because of the difficulty of obtaining good single crystals, the number of complete structure determinations of long-chain compounds is very limited. A few  $N$ -mono- $n$ -alkyl substituted ammonium halides have been examined. The lower members of the series possess interesting high-temperature tetragonal forms which exhibit chain rotation. Of the higher members of the series, the structure of  $n$ -dodecylammonium chloride and bromide have been studied (Gordon, Stenhagen & Vand, 1953). A number of long-chain acids have been examined. A complete analysis of lauric acid has been made in two projections (Vand, Morley & Lomer, 1951), and some others, such as  $n$ -pentadecanoic acid (von Sydow, 1954), and isopalmitic acid (Stenhagen, Vand & Sim, 1952) have been studied in rather less detail.

It seemed worth while to investigate the structure of a compound which was both a long-chain carboxylic acid and an alkyl substituted ammonium halide, and with this end in view the hydrobromide and hydrochloride of 11-amino-undecanoic acid were prepared. They were found to be not isomorphous, and, as the structure of the bromide seemed the easier to determine, work has been concentrated on it.

## 2. Crystal data

11-Amino-undecanoic acid hydrobromide hemihydrate,  $\text{COOH}(\text{CH}_2)_{10}\text{NH}_3\text{Br} \cdot \frac{1}{2}\text{H}_2\text{O}$ ;  $M$ , 291.2;  $d$  (calc.) 1.309,  $d$  (found) 1.29. Monoclinic,  $a = 11.08 \pm 0.03$ ,  $b = 5.27 \pm 0.02$ ,  $c = 50.60 \pm 0.20$  Å,  $\beta = 90^\circ 42' \pm 10'$ . Space group deduced from absent reflexions either  $Aa-C_2^1$  or  $A2/a-C_2^6$ . Structure refinement confirms  $A2/a$  as true space group. Eight molecules per unit cell. Volume of unit cell,  $2954$  Å<sup>3</sup>. Absorption coefficient

for X-rays, ( $\lambda = 1.542$  Å)  $\mu = 38.6$  cm.<sup>-1</sup>. Total number of electrons per unit cell =  $F(000) = 1224$ .

Tabular crystals were obtained by crystallizing 11-amino-undecanoic acid from aqueous hydrobromic acid. The most prominent face is (001).

## 3. Experimental

Rotation, oscillation and moving-film photographic methods were used, with Cu  $K\alpha$  radiation. The cell dimensions were obtained from rotation and zero-layer-line moving-film photographs calibrated with superimposed NaCl powder lines. The intensity data used consisted of the ( $h0l$ ) and ( $0kl$ ) reflexions. The intensities were estimated visually, using the multiple-film technique to correlate strong and weak reflexions (Robertson, 1943). The structure factors were evaluated by the usual mosaic-crystal formula, and were later placed on an absolute scale by comparison with  $F_c$  values.

## 4. Structure determination

### *The b-axis projection*

Because of the short length of the  $b$  axis it seemed probable that good resolution of the atoms would be obtained in the projection along this axis. A Patterson projection was prepared and is shown in Fig. 1(a). The largest peak in the  $b$ -axis projection is due to the Br-Br vector. Structure factors were calculated for the bromide ion only, and the signs so obtained were allotted to the observed ( $h0l$ ) structure factors. Using these, an electron-density projection along the  $b$  axis was prepared and approximate coordinates for the atoms of the acid molecule were estimated. Using these coordinates, structure factors were calculated and  $F_o$  values placed on an absolute scale by comparison with  $F_c$  values.

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This projection was further refined by evaluating Fourier difference syntheses, using  $(F_o - F_c)$  as coef-

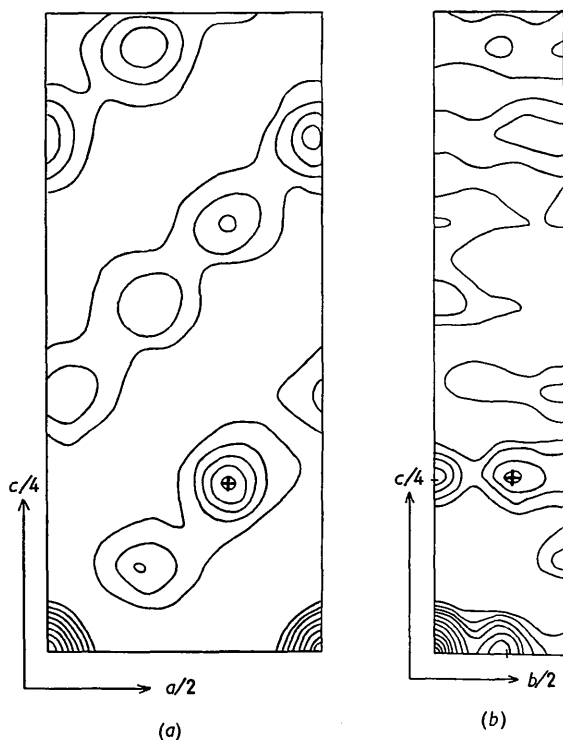


Fig. 1. (a) The  $b$ -axis Patterson projection, (b) the  $a$ -axis Patterson projection. Contours at equal, arbitrary intervals. Peak due to Br-Br vector marked by a cross.

ficients. These maps showed that the structure factor discrepancies were due to the following factors:

(a) The light atom coordinates needed adjustment.

(b) A large peak with coordinates  $x = \frac{1}{4}$ ,  $z = 0$  remained, in spite of the adjustments made to the other atoms, and obviously was due to the presence of a water molecule in the structure. At the outset of the analysis it had not been known that the crystals are hydrated.

(c) The bromide ion showed evidence of anisotropic thermal vibration, and in consequence its atomic scattering factor should be of the form

$$f = f_{\text{Br}} \exp [-\{\alpha + \beta \sin^2 (\varphi - \psi)\} \sin^2 \theta],$$

(Hughes, 1941; Cochran, 1951). In this expression  $\psi$  is the angle between the direction of maximum vibration and the  $c$  axis,  $\alpha$  and  $\beta$  are constants, and  $(2 \sin \theta, \varphi)$  are the polar coordinates of a point in the  $(h0l)$  section of the reciprocal lattice.

In order to allow for the anisotropic vibration of the bromide ion the variation of the temperature parameter  $\gamma$  with  $\varphi$  was determined graphically. The contributions of the carbon, nitrogen and oxygen atoms were subtracted from the observed structure factors to give a set of  $F_o$  values dependent only on the bro-

mine contributions. These  $F_o$  values were divided into groups with  $\varphi = 0^\circ - 15^\circ$ ,  $15^\circ - 30^\circ$ , etc. and in each group  $\gamma$  was obtained from a plot of  $\log (\Sigma |F_o| / \Sigma |G f_{\text{Br}}|)$  against  $\sin^2 \theta$ . In this expression  $G$  is the geometric part of the structure factor, and  $f_{\text{Br}}$  is the Thomas-Fermi bromine scattering factor, without temperature correction, for a neutral atom. It was found that the calculated values of  $\gamma$  could be fitted quite closely by a curve of the form

$$\gamma = 1.65 + 0.80 \sin^2 (\varphi - 154^\circ).$$

This curve and the calculated values of  $\gamma$  plotted against  $\varphi$  are compared in Fig. 2. For each reflexion

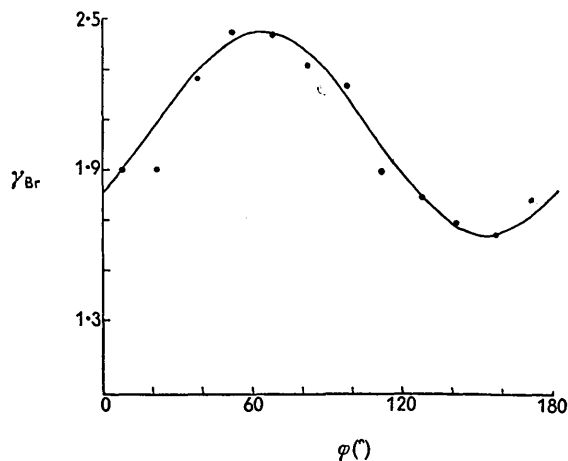


Fig. 2. Calculated values of  $\gamma$  plotted against  $\varphi$ . The curve  $\gamma = 1.65 + 0.80 \sin^2 (\varphi - 154^\circ)$  is superimposed.

the appropriate value of  $\gamma$  was obtained from this curve and substituted in  $f = f_{\text{Br}} \exp (-\gamma \sin^2 \theta)$  to obtain the anisotropic scattering factor.

When all these factors had been allowed for, the percentage discrepancy  $(100 \Sigma |F_o - F_c| / \Sigma |F_o|)$  was reduced to 15.0 for the 249 observed reflexions in this zone.

At this stage another electron-density projection along the  $b$  axis was prepared, using the final signs which had been obtained for the structure factors. This is shown in Fig. 3(a).

#### The $a$ -axis projection

A Patterson projection along the  $a$  axis was prepared (Fig. 1(b)) and the peaks due to Br-Br vectors were located. In the  $b$ -axis projection there had been an ambiguity in the choice of origin of the unit cell because of the halving of the cell in both the  $a$  and  $c$  directions, but calculation of  $(0kl)$  structure factors for the bromide ions only, using the two possible  $z$  values, and comparison with the observed structure factors for planes with  $l$  odd, served to decide between the two alternatives.

Using the signs of the bromide ion contributions, an electron-density projection along the  $a$  axis was

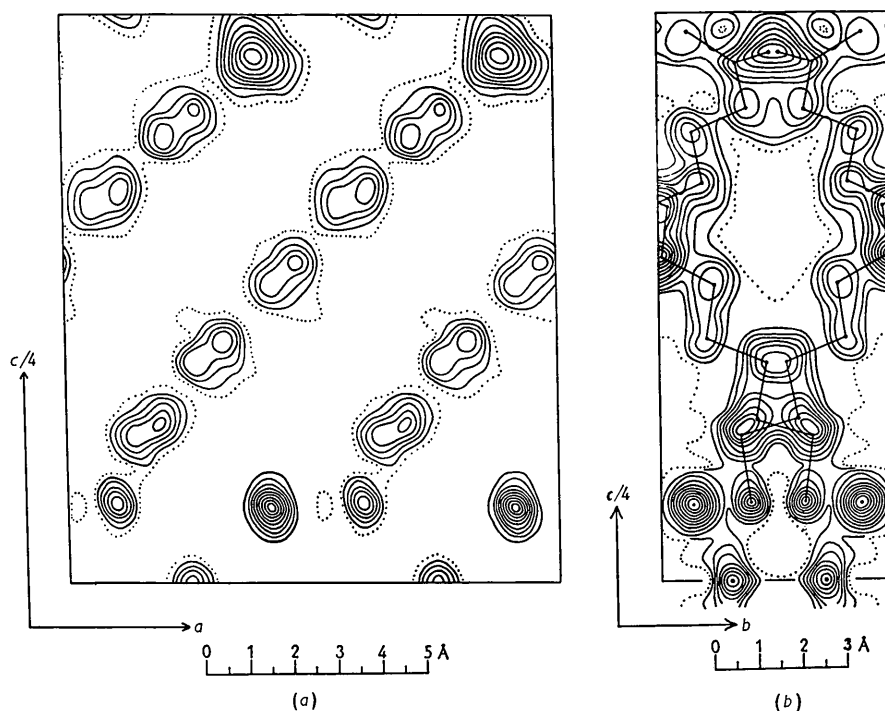


Fig. 3. Electron-density projection (a) along the  $b$  axis, (b) along the  $a$  axis. Contours around the carbon, nitrogen and oxygen atoms at intervals of  $1 \text{ e.}\text{\AA}^{-2}$ , starting at the two-electron line, which is dotted. Contours around the bromide ion at intervals of  $5 \text{ e.}\text{\AA}^{-2}$ .

computed from the observed  $(0kl)$  structure factors. From this projection coordinates for the light atoms were chosen, and the refinement was continued by Fourier and difference Fourier syntheses. The final electron-density projection is shown in Fig. 3(b). The final percentage discrepancy for this zone is 13.9 for the 108 observed reflexions. Observed and calculated structure factors are listed in Table 4 for both the  $(h0l)$  and  $(0kl)$  zones.

### 5. Coordinates, molecular dimensions and estimates of accuracy

The final coordinates for the atoms are shown in Table 1, and the various interatomic distances and angles calculated from these coordinates are listed in Table 2.

The standard deviation of a C-C bond was estimated by the method of Cruickshank (Cruickshank, 1949, 1954; Ahmed & Cruickshank, 1953) to be  $0.040 \text{ \AA}$ .

Assuming that the hydrocarbon chain is a periodic structure, so that the coordinates of the even and odd carbon atoms are fitted by a linear equation, and that the atomic coordinates can all be given equal weight, a least-squares calculation was made of the components of the average vector between alternate carbon atoms. The values found are given in Table 3. From these components the modulus of the vector  $s$  between alternate carbon atoms is found to be  $2.563 \text{ \AA}$ .

The average C-C bond length is  $1.539 \text{ \AA}$ , with a standard deviation of  $0.040/\sqrt{10} = 0.0126 \text{ \AA}$ . From this bond length and the value of  $s$ , the average inter-bond angle is calculated to be  $112^\circ 44'$ .

Table 1. *Coordinates*

Atom	$x/a$	$y/b$	$z/c$
N	0.103	1.623	0.0342
C <sub>11</sub>	0.130	1.660	0.0630
C <sub>10</sub>	0.197	1.410	0.0700
C <sub>9</sub>	0.270	1.450	0.0950
C <sub>8</sub>	0.340	1.200	0.1050
C <sub>7</sub>	0.417	1.238	0.1300
C <sub>6</sub>	0.486	1.008	0.1400
C <sub>5</sub>	0.556	1.055	0.1650
C <sub>4</sub>	0.626	0.813	0.1730
C <sub>3</sub>	0.705	0.864	0.1970
C <sub>2</sub>	0.780	0.625	0.2050
C <sub>1</sub>	0.860	0.667	0.2290
O <sub>1</sub>	0.942	0.520	0.2333
O <sub>2</sub>	0.860	0.870	0.2400
H <sub>2</sub> O	0.250	0.300	0
Br	-0.087	0.140	0.0328

The difference,  $\Delta l$ , between the length of each C-C bond and the expected value of  $1.545 \text{ \AA}$  was used to confirm the correctness of the estimate of the C-C bond standard deviation. It is to be expected that  $\{\Delta l^2\}^{\frac{1}{2}}$  should give a good estimate of  $\sigma(l)$ , for there are ten independent values of  $\Delta l$ . The value found in this way is  $0.030 \text{ \AA}$ , which is rather smaller than the value of  $0.040 \text{ \AA}$  deduced by Cruickshank's method.

Table 2. *Interatomic distances and angles*

The letters associated with some of the atoms refer to the following equivalent positions:

$b: \bar{x}, \bar{y}, \bar{z}; c: \frac{1}{2}+x, \bar{y}, z; d: \bar{x}, \frac{1}{2}-y, \frac{1}{2}-z; e: \frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z; f: \frac{1}{2}-x, y, \bar{z}.$

Intramolecular distances		Intermolecular distances		Interbond angles	
N-C <sub>11</sub>	1.50 Å	N-Br	3.30, 3.44 Å	C-C-C	112° 44'
C <sub>11</sub> -C <sub>10</sub>	1.56	N-Br (b)	3.62	O <sub>1</sub> -C <sub>1</sub> -O <sub>2</sub>	119°
C <sub>10</sub> -C <sub>9</sub>	1.52	N-Br (c)	3.66	C <sub>2</sub> -C <sub>1</sub> -O <sub>1</sub>	119°
C <sub>9</sub> -C <sub>8</sub>	1.61	N-H <sub>2</sub> O	2.92	C <sub>2</sub> -C <sub>1</sub> -O <sub>2</sub>	120°
C <sub>8</sub> -C <sub>7</sub>	1.54	H <sub>2</sub> O-Br (c)	3.38, 3.84	C <sub>1</sub> -O <sub>1</sub> -O <sub>2</sub> (d)	125°
C <sub>7</sub> -C <sub>6</sub>	1.52	H <sub>2</sub> O-Br	4.17	C <sub>1</sub> -O <sub>2</sub> -O <sub>1</sub> (d)	116°
C <sub>6</sub> -C <sub>5</sub>	1.51	Br-Br (b)	4.11	C <sub>11</sub> -N-Br	104°, 92°
C <sub>5</sub> -C <sub>4</sub>	1.55	N-N (b)	4.34	C <sub>11</sub> -N-Br (b)	151°
C <sub>4</sub> -C <sub>3</sub>	1.52	C <sub>2</sub> -C <sub>5</sub> (c)	4.04	C <sub>11</sub> -N-H <sub>2</sub> O	123°
C <sub>3</sub> -C <sub>2</sub>	1.56	C <sub>4</sub> -C <sub>7</sub> (c)	3.90	Br (c)-H <sub>2</sub> O-Br (b)	93°
C <sub>2</sub> -C <sub>1</sub>	1.52	C <sub>5</sub> -C <sub>9</sub> (c)	4.57	N-H <sub>2</sub> O-N (f)	109°
C <sub>1</sub> -O <sub>1</sub>	1.22	C <sub>8</sub> -C <sub>11</sub> (c)	3.92		
C <sub>1</sub> -O <sub>2</sub>	1.21	C <sub>10</sub> -Br (c)	3.86		
		C <sub>2</sub> -C <sub>6</sub> (c)	4.45		
		C <sub>9</sub> -O <sub>2</sub> (e)	3.46		
		C <sub>1</sub> -O <sub>2</sub> (d)	3.48		
		C <sub>11</sub> -H <sub>2</sub> O	3.94		
		C <sub>11</sub> -Br	3.80, 3.95		
		O <sub>1</sub> -C <sub>3</sub> (c)	4.00		
		O <sub>1</sub> -O <sub>2</sub> (d)	2.64		
		O <sub>2</sub> -O <sub>2</sub> (e)	3.73		

Table 3. *Components of the average vector s between two alternate carbon atoms*

Coordinate	Fractional component	Rectangular component (Å)
<i>x</i>	0.1454	1.612
<i>y</i>	-0.1965	-1.036
<i>z</i>	0.03363	1.702

## 6. Discussion of structure and molecular dimensions

The C-C bond lengths appear at first sight to fall into two series, one having an average value of 1.560 Å and the other 1.519 Å. However, the standard deviation of each value is  $0.040/\sqrt{5} = 0.018$  Å, and the total standard deviation of the difference,  $\sigma(t)$ , is  $0.018/\{2(1-\cos 112^\circ 44')\} = 0.030$  Å.  $\Delta/\sigma(t)$  has a value of 1.33, and consequently the difference cannot be regarded as significant. The average C-C bond length of 1.539 Å, with standard deviation of 0.012<sub>6</sub> Å, does not differ significantly from the accepted value of 1.545 Å.

For a hydrocarbon chain in which the average carbon bond length is 1.545 Å and the interbond angle tetrahedral, a value of *s* equal to 2.522 Å is to be expected. The distance of 2.563 Å found in this analysis is 0.041 Å greater. This difference is significant, for  $\Delta/\sigma(t) = 3.26$ . Similar enlargements of this distance have been found in strontium laurate (Morley & Vand, 1949) with *s* = 2.610 Å, and potassium caprate (Vand, Lomer & Lang, 1949) with *s* = 2.598 Å. The value found in lauric acid (Vand, Morley & Lomer, 1951), however, is 2.521 Å. The enlargement of *s* in the present case from 2.522 to 2.563 Å is connected with an enlargement of the interbond angle from the normal tetrahedral value of 109° 28' to 112° 44'. This ex-

pansion of the interbond angle is more likely to be caused by intermolecular rather than by intramolecular forces, though further clarification of this point must await structure analyses of other derivatives of 11-amino-undecanoic acid.

It was found that the coordinates, expressed in Ångström units, of the atoms C<sub>1</sub>, C<sub>2</sub>, ..., C<sub>11</sub> of the hydrocarbon chain could be fitted to an equation of the form

$$X = AY + BZ + C.$$

*A*, *B* and *C* were determined by the method of least squares to be -0.3394, 0.7413 and 1.9964 respectively. The average displacement of a carbon atom from this plane is 0.050 Å and the largest (C<sub>1</sub>) is 0.106 Å, so that atoms C<sub>1</sub>, C<sub>2</sub>, ..., C<sub>11</sub> may be regarded as coplanar, and the deviations of these atoms from the common plane are due solely to experimental errors. The terminal nitrogen atom, however, departs significantly from this plane, the displacement being 0.593 Å. The carboxyl group also appears to be twisted out of the plane of the hydrocarbon chain, but the magnitude of this effect is rather uncertain since the coordinates of the oxygen atoms are less accurate than those of the carbon atoms, owing to the considerable lack of resolution, in both projections, of the atoms of the carboxyl group.

The two molecules which are connected by hydrogen bonding between carboxyl groups (O...O separation 2.64 Å) are not coplanar, the perpendicular distance between the two planes being 1.62 Å. In this respect the structure is similar to that of pimelic acid (MacGillavry, Hoogschagen & Sixma, 1948), and contrasts with carboxylic acids of the benzene series in which the two molecules constituting the dimer are very nearly coplanar (Cochran, 1953; Sim, Robertson &



Goodwin, 1955). The length of the hydrogen bonds between the carboxyl groups, 2.64 Å, is normal for this type of hydrogen bonding, where values ranging from 2.53 Å in furoic acid (Goodwin & Thomson, 1954) to 2.69 Å in glutaric acid (Morrison & Robertson, 1949) are to be found.

The type of packing of the hydrocarbon chains is of the unusual crossed-chain type found for form A of potassium caprate (Vand *et al.*, 1949). Normally hydrocarbon chains pack together with their chain axes parallel, and the majority of structures which have been found to have hydrocarbon chains crossing each other are ionic in type. The inclination of the chain axes to the (001) plane was calculated from the components of the vector between alternate carbon atoms. We obtain

$$\sin \tau = 1.702/2.563 = 0.6640,$$

whence  $\tau = 41^\circ 36'$ . This value is considerably smaller than that found for any known form of straight-chain fatty acid, and is very close to the value of  $43^\circ\text{--}45^\circ$  found for the branched chain fatty acid, isopalmitic acid (Stenhagen *et al.*, 1952). The effective cross-section of the molecules at right angles to the chain axis was calculated from the expression

$$S = \frac{1}{2}ab \sin \tau$$

and a value of  $19.39 \text{ \AA}^2$  was obtained. This value agrees with similar determinations made on other long-chain compounds (Lingafelter & Jensen, 1950). It is slightly larger than the value of  $18.43 \text{ \AA}^2$  found for *n*-hexatriacontane at  $20^\circ \text{ C.}$  (Vand, 1953).

The terminal  $\text{NH}_3^+$  group forms three hydrogen bonds, two to bromide ions of lengths 3.30 and 3.44 Å, and one to a water molecule of length 2.92 Å. These bonds are directed approximately tetrahedrally, the angles which the  $\text{C}_{11}\text{--N}$  bond makes with the hydrogen bonds being  $104^\circ$ ,  $92^\circ$  and  $123^\circ$  respectively. The water molecules, situated on twofold axes, are involved in four hydrogen bonds, two to  $\text{NH}_3^+$  groups and two to bromide ions. These bonds are also directed nearly tetrahedrally, the angles  $\text{Br--H}_2\text{O--Br}$  and  $\text{N--H}_2\text{O--N}$  being  $93^\circ$  and  $109^\circ$  respectively. The bonds from the water molecule to the bromide ions are 3.38 Å in length, and presumably the hydrogen atoms of the water molecule are directed towards the bromide ions.

A section in the plane of the water molecules, parallel to (001), showing the arrangement of the water molecules,  $\text{NH}_3^+$  and  $\text{Br}^-$  ions is shown in Fig. 4(a). The  $\text{NH}_3^+$  and  $\text{Br}^-$  ions are at heights of 1.73 and 1.66 Å respectively, both above and below the plane of the water molecules. There is an almost planar, distorted hexagonal arrangement of alternate  $\text{Br}^-$  and  $\text{NH}_3^+$  ions arranged in two sheets, one above and the other below the plane of the water molecules, with the  $\text{Br}^-$  ions of the lower sheet fitting approximately below the  $\text{NH}_3^+$  ions of the upper sheet. The separation of the two sheets is 3.4 Å. The water molecules pack into the channels which run vertically through this hexagonal network. Fig. 4(b) illustrates this arrangement.

While the surroundings of a water molecule may be regarded as symmetrical, having  $\text{NH}_3^+$  and  $\text{Br}^-$  ions both above and below, the surroundings of a  $\text{Br}^-$  or

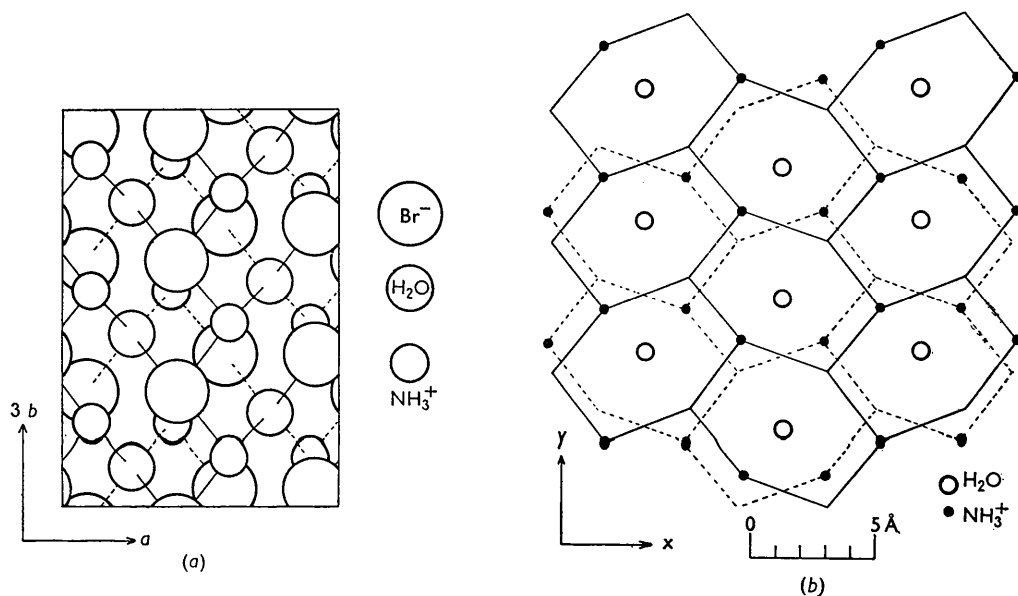


Fig. 4. (a) Section through the plane of the water molecules, parallel to (001), showing the packing of the  $\text{NH}_3^+$  and  $\text{Br}^-$  ions around the water molecules. Full lines represent hydrogen bonds above the plane of the water molecules, while broken lines represent hydrogen bonds below this plane.

(b) Representation of the double ionic layer. The corners of the hexagonal network which are dotted are occupied by  $\text{NH}_3^+$  ions; those not dotted, by  $\text{Br}^-$  ions. Ions above the plane of the water molecules are joined by full lines, those below by broken lines.

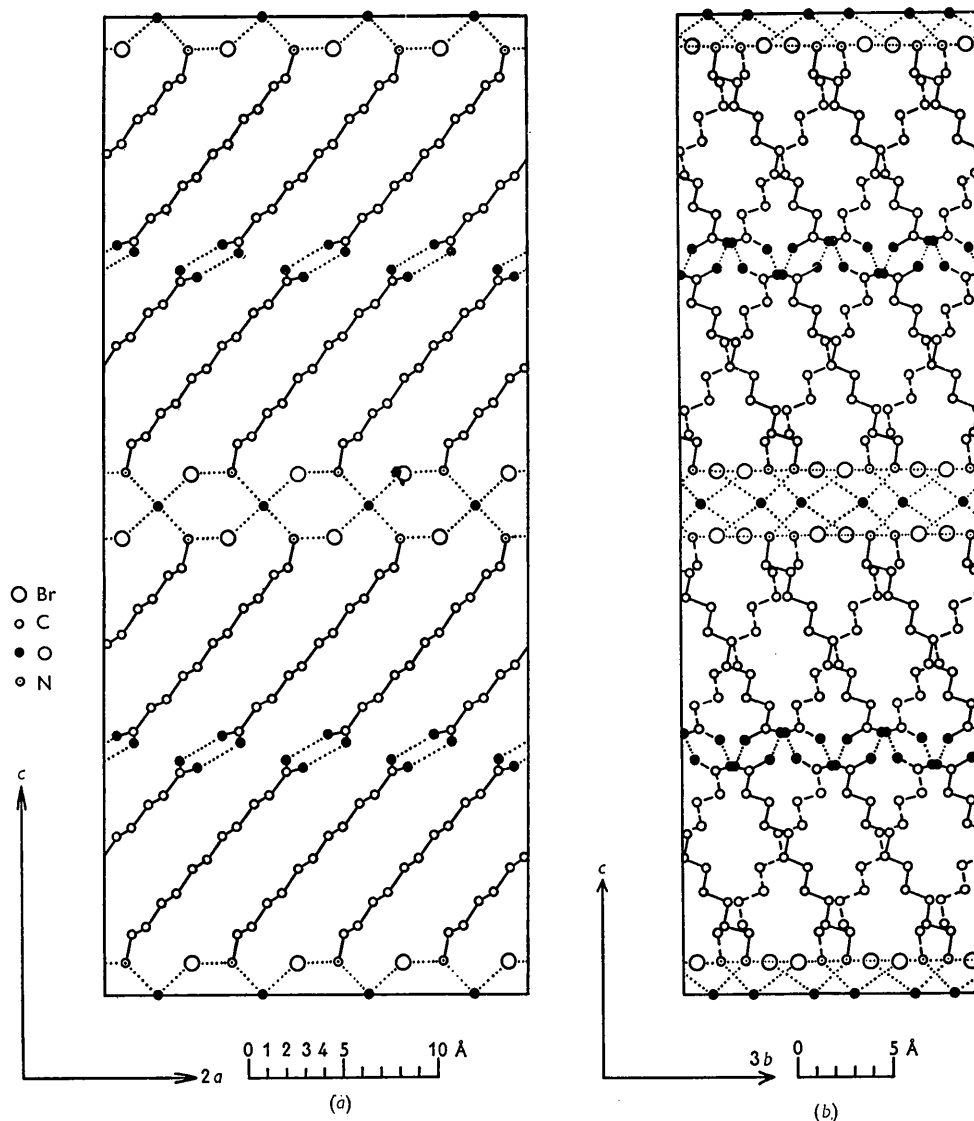


Fig. 5. (a) Arrangement of molecules in (010) projection. Dotted lines represent hydrogen bonding. Hydrogen atoms are not shown.

(b) Arrangement of molecules in (100) projection. Molecules on a lower relative level are indicated by broken lines. Hydrogen bonding is indicated by dotted lines. Hydrogen atoms are not shown.

$\text{NH}_3^+$  ion cannot be so regarded. Each  $\text{Br}^-$  ion is surrounded by three  $\text{NH}_3^+$  ions (at 3.30, 3.44 and 3.66 Å), the four ions being approximately in the one plane, and by three water molecules (at 3.38, 3.84 and 4.17 Å) in a plane 1.7 Å below. These six neighbours form a distorted octahedron. Below the face formed by the water molecules there is a fourth  $\text{NH}_3^+$  ion (at 3.62 Å), so that each  $\text{Br}^-$  ion has an unsymmetrical arrangement of four oppositely charged  $\text{NH}_3^+$  ions to one side of it. The environment of an  $\text{NH}_3^+$  ion is similar.

The approach distances between carbon atoms of neighbouring chains are approximately 4 Å, and correspond to normal van der Waals interactions. Some of the shorter of these distances are listed in Table 2.

The arrangements of the molecules in the (010) and (100) projections are indicated in Fig. 5.

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### Short Communications

*Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.*

*Acta Cryst.* (1955). **8**, 840

**Some lattice constants.** By J. W. MENARY, *Research Department, African Explosives and Chemical Industries Limited, P. O. Northrand, Transvaal, South Africa*

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During analytical work involving the identification of crystalline compounds by X-ray diffraction analysis, powder patterns were obtained with a Berthold diffractometer for compounds of high purity (generally AR grade). Since the diffractometer measures spacings with high precision, accurate lattice constants were calculated from the data available for crystals having orthorhombic or higher symmetry. These figures are reported in Table 1;

powder data are being submitted to the A.S.T.M. for prospective inclusion in their *Index of Diffraction Patterns*. Some idea of the accuracy attained may be seen where standard deviations are reported.

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Table 1

Compound	Temperature (°C.)	System*	Lattice constants
Ca(NO <sub>3</sub> ) <sub>2</sub>	24	C	$a = 7.590 \pm 0.006 \text{ \AA}$
Pb(NO <sub>3</sub> ) <sub>2</sub>	26	C	$a = 7.853 \pm 0.002 \text{ \AA}$
FeS <sub>2</sub> (pyrite)	26	C	$a = 5.417 \pm 0.002 \text{ \AA}$
KAl(SO <sub>4</sub> ) <sub>2</sub> · 12H <sub>2</sub> O	24	C	$a = 12.164 \pm 0.006 \text{ \AA}$
(NH <sub>4</sub> )Al(SO <sub>4</sub> ) <sub>2</sub> · 12H <sub>2</sub> O	24	C	$a = 12.241 \pm 0.006 \text{ \AA}$
KCr(SO <sub>4</sub> ) <sub>2</sub> · 12H <sub>2</sub> O	24	C	$a = 12.204 \pm 0.007 \text{ \AA}$
NaCr(SO <sub>4</sub> ) <sub>2</sub> · 12H <sub>2</sub> O	23	C	$a = 12.12 \pm 0.08 \text{ \AA} \dagger$
(NH <sub>4</sub> )Fe(SO <sub>4</sub> ) <sub>2</sub> · 12H <sub>2</sub> O	24	C	$a = 12.324 \pm 0.007 \text{ \AA}$
β-Hexachlorocyclohexane	24	C	$a = 10.078 \pm 0.007 \text{ \AA}$
(NH <sub>4</sub> )H <sub>2</sub> PO <sub>4</sub>	24	T	$a = 7.510 \pm 0.003, c = 7.564 \pm 0.008 \text{ \AA}$
NaNO <sub>3</sub>	24	R	$a = 6.326 \text{ \AA}, \alpha = 47^\circ 17'$
KBrO <sub>3</sub>	23	R	$a = 4.413 \text{ \AA}, \alpha = 85^\circ 48'$
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	23	O	$a = 6.000 \pm 0.005, b = 10.66 \pm 0.02, c = 7.794 \pm 0.006 \text{ \AA}$
(NH <sub>4</sub> )NO <sub>3</sub> IV	24	O	$a = 5.757, b = 5.451, c = 4.935 \text{ \AA}$
(NH <sub>4</sub> )NO <sub>3</sub> III	>32	O	$a = 7.18, b = 7.71, c = 5.83 \text{ \AA}$
AgNO <sub>3</sub>	23	O	$a = 6.994 \pm 0.004, b = 7.330 \pm 0.004, c = 10.13 \pm 0.01 \text{ \AA}$

\* C = cubic, T = tetragonal, R = rhombohedral, O = orthorhombic.

† From low-angle lines only.