Cu–OH bonds (four from each Cu atom), as shown by the full lines in Fig. 6 (*b*) and (*e*), then it is apparent that these link one-half the Cu atoms into chains which are cross-linked into layers through the remaining Cu atoms. It will be observed that the OH groups lying between these chains have a different environment from those in the chains. An OH group in a chain is attached to 3 Cu by short Cu–OH bonds, whereas an OH group of the other type is attached to 2 Cu by short bonds and to the third by a long Cu–OH bond. The Br atoms serve to link together the two types of Cu atom, but only by secondary Cu–Br bonds (broken lines).

The crystal structure of $Cu_2Cl(OH)_3$ consists of essentially close-packed layers of the same type as in the hydroxy-bromide which are parallel to the (011) and $(0\bar{1}1)$ planes. The structure is therefore not a layer structure, for only three-quarters of the Cu atoms lie in the (011) or $(0\bar{1}1)$ layers, the remainder cross-linking the layers into a three-dimensional complex of a honeycomb type (Fig. 6 (*c*)). If the atoms in a pair of adjacent close-packed layers (the shaded circles in Fig. 6 (*c*)) are projected on to the $(0\bar{1}1)$ plane, as in Fig. 6 (*f*), the similarity of the structure to that of $Cu_2Br(OH)_3$ becomes apparent. The strong Cu–OH bonds lead to chains in the direction of the *a* axis, and these chains are cross-linked by other Cu atoms. One-half of the Cu atoms lie in the chains shown in Fig. 6 (*f*), one-quarter between the chains in the same $(0\bar{1}1)$ plane, and the remainder between the layers. Herein lies the essential difference between the structures of $Cu_2Cl(OH)_3$ and $Cu_2Br(OH)_3$. A comparison of the plans of the layers in $CuCl_2$, $Cu_2Br(OH)_3$ and $Cu_2Cl(OH)_3$ shows the strong resemblance between the crystal structures of these three compounds.

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The Crystal Structure of Hexamethylenediamine Dihydrochloride

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Hexamethylenediamine dihydrochloride is isomorphous with the dihydrobromide (monoclinic, space group $P2_1/c$). Better crystals are available, and a detailed structure analysis is given with Fourier projections along two axes mutually at right angles, from which the co-ordinates of all the atoms are obtained with an accuracy of about $\pm 0{\cdot}04$ A. In the chain molecule there is evidence of alternating long and slightly shorter bonds, but the variations are close to the experimental limits. The shorter N–Cl distances are of two types, averaging 3·11 and 3·24 A. The latter figure is close to the sum of the ionic radii, and the shorter type is probably accounted for by hydrogen bonding between the chlorine and nitrogen atoms. The compact grouping of the halogen ions in the lattice is illustrated in the diagram.

Introduction

The dihydrochloride of hexamethylenediamine is isomorphous with the dihydrobromide, described in an earlier paper (Binnie & Robertson, 1949). Well-formed crystals of the dihydrochloride are readily obtained, and it is possible to carry out a detailed structural investigation, utilizing several different zones of reflexions. Furthermore, the halogen contribution to the total scattering power of this molecule is only 33 %, and so with careful intensity estimates it should be possible to derive reasonably accurate bond-length measurements within the hexamethylenediamine chain. For the dihydrobromide molecule, with a halogen contribution of 51 %, no great accuracy was possible. The present paper gives the results of a detailed investigation of the structure of the dihydrochloride. Two projections of the structure are obtained, along axes mutually at right angles, and after refinement by the double Fourier series method these projections provide excellent resolution of all the atoms. Certain minor bond-length variations which may be significant are detected in the saturated aliphatic chain, but they are close to the limit of possible error.

Crystal data

Hexamethylenediamine dihydrochloride, $C_6H_{18}N_2Cl_2$; M, 189·1; m.p. 254·5° C.; d, calc. 1·212, found 1·216. Monoclinic prismatic, $a=4{\cdot}60\pm0{\cdot}02$, $b=14{\cdot}19\pm0{\cdot}04$, $c=15{\cdot}68\pm0{\cdot}04$ A., $\beta=90{\cdot}8°\pm0{\cdot}2°$. Absent spectra, ($h0l$) when l is odd; ($0k0$) when k is odd. Space group, C^5_{2h}–$P2_1/c$. Four molecules per unit cell. No molecular symmetry. Volume of the unit cell = 1023 A.³ Absorption coefficient for X-rays ($\lambda=1{\cdot}54$ A.), $\mu=51{\cdot}3$ cm.$^{-1}$. Total number of electrons per unit cell $=F(000)=408$.

Well-developed crystals may be obtained by slow cooling from a solution in a mixture of ethyl alcohol and water. They are generally obtained as slender colourless needles, elongated in the direction of the short a axis, the prominent faces being (011) and ($0\bar{1}1$).

Structure analysis

Projection along the a axis

The solution obtained for the dihydrobromide structure should be applicable, as a first approximation, to the principal projection of the dihydrochloride structure. For confirmation of this, a Patterson vector map representing a projection of the dihydrochloride structure on (100) was prepared from the terms $F^2(0kl)$. This is shown in Fig. 1, and it is found that the co-ordinates of the principal peaks correspond closely to those observed for the dihydrobromide. In the present case, however, there are certain additional features on the map which probably correspond to subsidiary vectors between the chlorine ions and the lighter atoms.

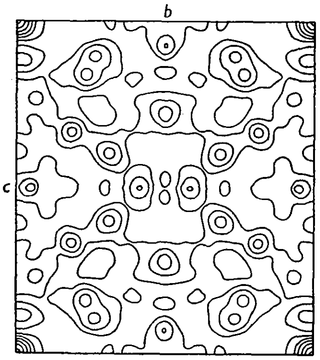

Fig. 1. Patterson projection on (100).

Without entering into a more detailed analysis, it may therefore be assumed that the previous solution is applicable to the present case. The y and z co-ordinates of all the atoms in the dihydrobromide structure were therefore scaled to the b and c axes of the dihydrochloride unit cell, and these values were utilized in a first calculation of the structure factors ($0kl$). The average discrepancy between the values so calculated and the observed values was about 32 %. From this starting point several successive double Fourier syntheses were carried out, until finally it was possible to include in the calculation all the observed ($0kl$) structure factors, which amount to about 75 % of those theoretically within the range of copper radiation. At this stage, the discrepancies between the calculated and observed structure factors (Table 5) were reduced to less than 16 %. The final electron-density map, which shows excellent resolution for all the atoms, is reproduced in Fig. 2, with an explanatory diagram in Fig. 3.

Projection along the b axis

Two co-ordinates for each atom are now accurately known, but there is no direct information about the third co-ordinate, x, although the general inference may be drawn that if the chain molecule consists of a regular zigzag, then the plane of the zigzag must be nearly perpendicular to the (100) plane. This feature may be utilized in formulating a trial structure for the b-axis projection. In order to obtain more direct information about the positions of the chlorine ions, however, a Patterson vector map was prepared from the terms $F^2(h0l)$, to give a projection along the b axis. The result of this calculation is shown in Fig. 4.

Attention was concentrated on the two prominent peaks marked with small crosses, as these are probably due to Cl–Cl vectors alone. The other features of the map are confused by overlapping vectors, due to the relatively small area of this projection.

The angular co-ordinates of the two prominent peaks are

	Peak 1	Peak 2
$2\pi x/a$	24°	75·5°
$2\pi z/c$	60·5°	36·5°

If the co-ordinates of the chlorine ions are

$$\mathrm{Cl}\,(1)=x_1,\ z_1;\quad \mathrm{Cl}\,(2)=x_2,\ z_2,$$

then we have the following equivalent positions in the projection on (010):

(1) x_1, z_1; (2) $-x_1, -z_1$; (3) $x_1, \frac{1}{2}+z_1$; (4) $-x_1, \frac{1}{2}-z_1$;
(5) x_2, z_2; (6) $-x_2, -z_2$; (7) $x_2, \frac{1}{2}+z_2$; (8) $-x_2, \frac{1}{2}-z_2$.

A large number of vectors may be formed between these positions, which include the following types:

(1)–(5) $x_2-x_1,\ z_2-z_1$;
(1)–(6) $x_1+x_2,\ z_1+z_2$;
(1)–(7) $x_2-x_1,\ \frac{1}{2}+z_2-z_1$;
(1)–(8) $x_1+x_2,\ -\frac{1}{2}+z_1+z_2$.

Now the z co-ordinates of the chlorine ions are known accurately from the previous projection, viz.

$$z_1=78° \quad \text{and} \quad z_2=141°,$$

in angular measure. These values may therefore be used to identify the vectors forming the two prominent peaks in the Patterson map, listed above. Thus

$$z_2-z_1=63°,$$

which is comparable with the z co-ordinates of peak (1) (60·5°). This identifies the vector as of the (1)–(5) type listed above, and hence

$$x_2-x_1=24°.$$

Also $$-\tfrac{1}{2}+z_2+z_1=39°,$$

which is comparable with the z co-ordinate of peak (2) ($36{\cdot}5^\circ$), and hence from the vector expression (1)–(8)

$$x_1+x_2=75{\cdot}5^\circ.$$

Solving for x_1 and x_2 we obtain

$$x_1=26^\circ,\quad x_2=50^\circ.$$

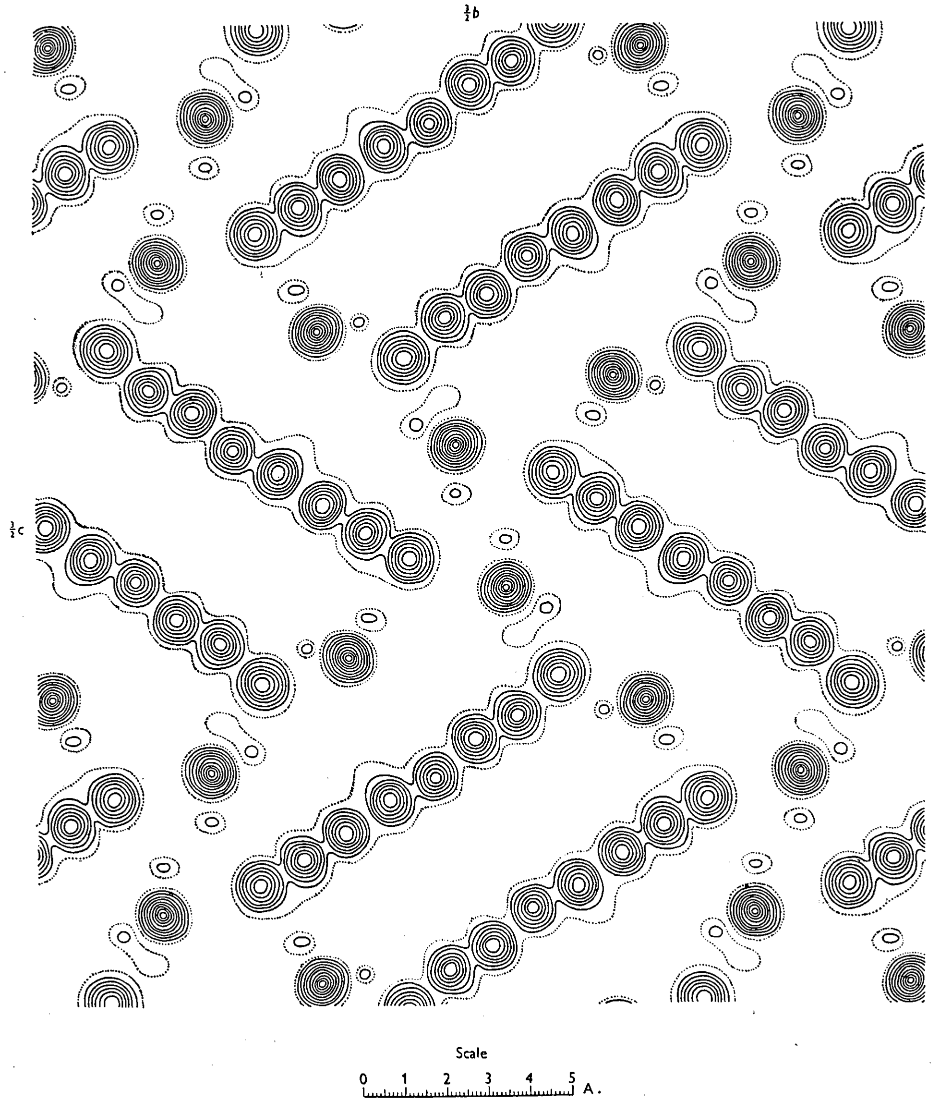

Fig. 2. Electron-density projection along a axis on (100). Contour intervals: one electron per A.^{2}, with one-electron line dotted, except on the chlorine ions, when the interval is three electrons per A.^{2}

This solution of the Patterson projection leads to the theoretical vector map illustrated in Fig. 5.

With these approximate values for the co-ordinates of the chlorine ions, and with the information about the probable orientation of the chain molecule which can be derived from the a-axis projection, it proved a matter of no great difficulty to set up a trial structure for the b-axis projection. Prominent reflexions given by the $(10\bar{2})$, (206), (204), (304) and $(1{\cdot}0{\cdot}\overline{12})$ planes helped to fix the position of the trial model, and good agreements were soon obtained between the calculated and observed values of all the ($h0l$) structure factors.

Further refinement was carried out by the Fourier-series method, and the electron-density map obtained from the final synthesis is shown in Fig. 6. Although the resolution appears to be good in this picture, reference to the explanatory diagram will show that in addition to the halogens, only three atoms are separately resolved, viz. C(1), C(2) and C(3). The others occur in closely overlapping pairs. However, as the z co-ordinates of all the atoms are already known accurately from the previous projection, it is now not difficult to derive fairly accurate values for all the co-ordinates.

A further independent check was obtained by calculating the structure factors for the third principal zone, ($hk0$). A Fourier analysis for this zone was not carried out, as it was realized that no useful resolution would be obtained.

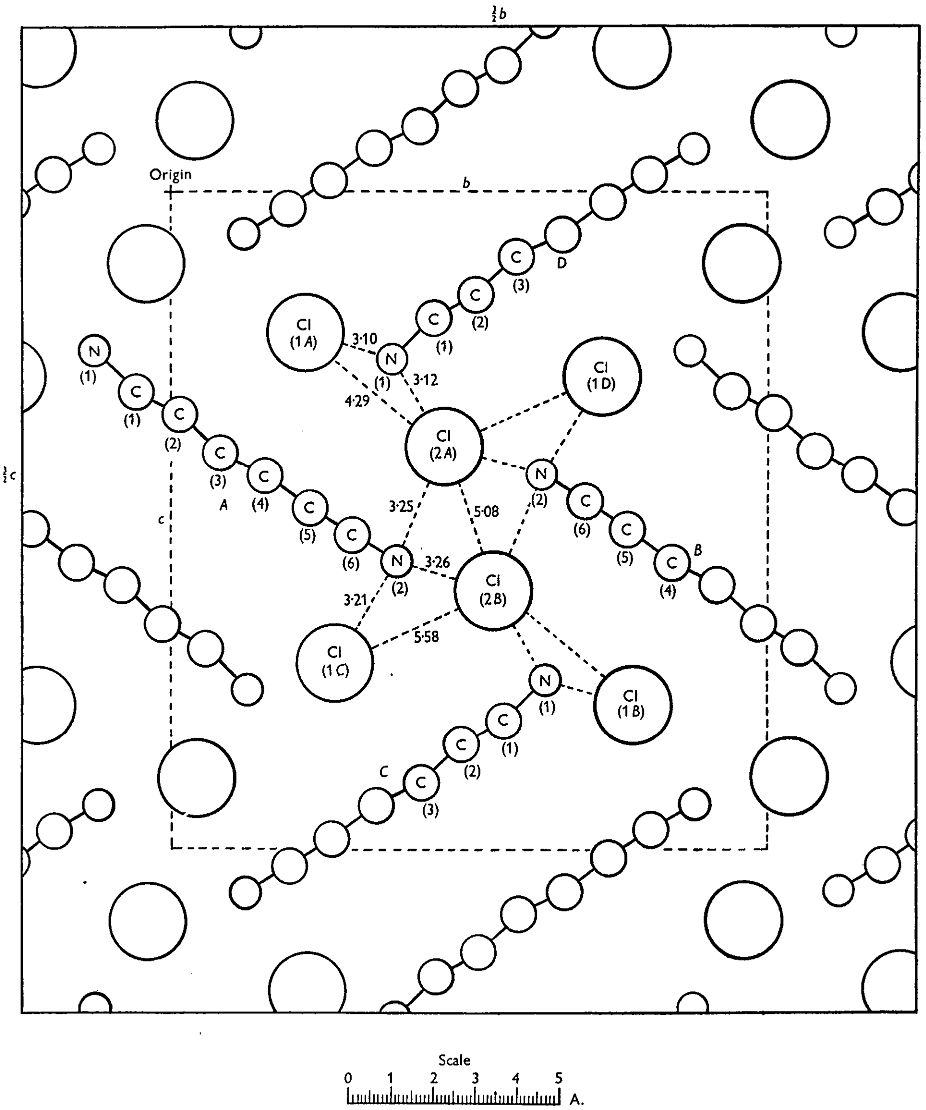

Fig. 3. Arrangement of the molecules in the *a*-axis projection.

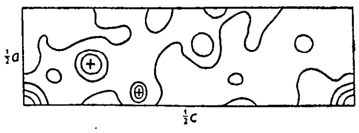

Fig. 4. Patterson projection on (010) drawn over one-quarter of the unit cell.

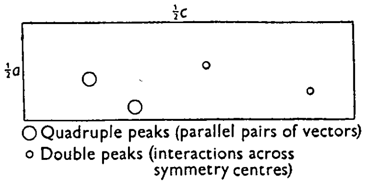

Fig. 5. Theoretical vector map for chlorine interactions, projected on (010).

Co-ordinates and dimensions

The co-ordinates assigned to the atoms from the final Fourier projections are given in Table 1. The atoms listed are those which occur in one-quarter of the unit cell, i.e. two chlorine ions and one complete chain molecule, as indicated in Fig. 3. The co-ordinates of all the other atoms may be obtained from this asymmetric unit by the operations appropriate to the space group $P2_1/c$. A centre of symmetry is chosen as origin, and the co-ordinates x, y, z are referred to the monoclinic crystal axes. Rectangular co-ordinates, x', y, z', are also listed, which refer to the a and b crystal axes, and their perpendicular c'. As β is close to 90°, $z = z'$ within the limits of measurement.

Table 1. *Co-ordinates*

Atom (cf. Fig. 3)	x (A.)	x' (A.)	y (A.)	$z=z'$ (A.)	$2\pi x/a$ (°)	$2\pi y/b$ (°)	$2\pi z/c$ (°)
Cl(1)	0·360	0·326	3·204	3·404	28·0	81·3	78·0
Cl(2)	0·590	0·529	6·520	6·116	46·0	165·5	141·1
N(1)	2·150	2·112	−1·810	3·810	168·5	−46·5	87·5
C(1)	1·520	1·473	−0·820	4·770	119·0	−20·0	110·5
C(2)	2·480	2·426	0·164	5·390	194·0	4·2	124·0
C(3)	1·720	1·657	1·148	6·270	135·1	29·2	144·1
C(4)	2·600	2·532	2·220	6·836	204·1	56·4	157·0
C(5)	1·840	1·764	3·300	7·630	144·5	84·0	175·1
C(6)	2·780	2·698	4·294	8·240	217·0	109·0	189·2
N(2)	1·940	1·852	5·392	8·856	152·1	136·8	203·3

Table 2. *Orientation*

$\chi_L = 87{\cdot}6°$ $\psi_L = 35{\cdot}2°$ $\omega_L = 54{\cdot}9°$
$\chi_M = 10{\cdot}8$ $\psi_M = 85{\cdot}9$ $\omega_M = 100{\cdot}0$
$\chi_N = 79{\cdot}5$ $\psi_N = 124{\cdot}9$ $\omega_N = 36{\cdot}9$

Table 3. *Co-ordinates referred to molecular axes*

Atom	L (A.)	M (A.)	N (A.)
N(1)	−4·429	0·244	−0·187
C(1)	−3·100	−0·480	−0·107
C(2)	−1·893	0·419	0·000
C(3)	−0·619	−0·419	−0·001
C(4)	0·619	0·419	0·001
C(5)	1·924	−0·396	−0·100
C(6)	3·123	0·486	−0·029
N(2)	4·340	−0·373	−0·323

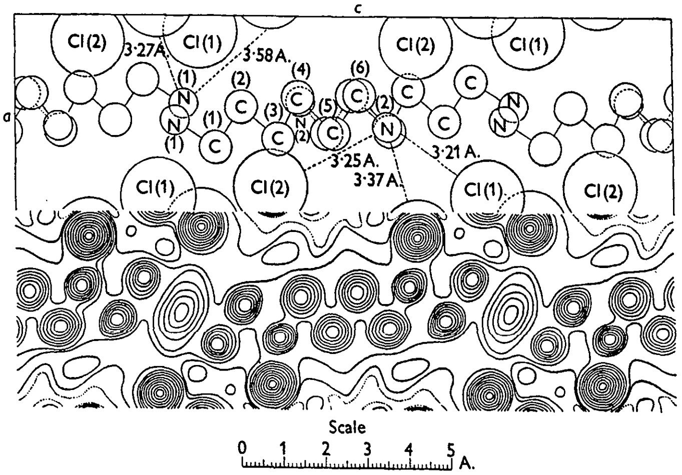

Fig. 6. Electron-density projection along the b axis on (010) showing the complete unit cell. Contour intervals: one electron per A.^{2} on single peaks, two electrons per A.^{2} on double peaks, and three electrons per A.^{2} on chlorine ions. One-electron line is dotted.

From the above co-ordinates the internal dimensions of the structure may be calculated. The details obtained for the chain molecule are given in Fig. 7, while other interionic and intermolecular distances are indicated in Figs. 3 and 6. The orientation of the chain molecule in the crystal may be expressed by giving the angles, χ_L, ψ_L, ω_L, etc., which the molecular axes L, M and N (Fig. 7) make with the a, b and c' crystal axes, c' being perpendicular to a and b. The molecular axis L passes through the mid-points of C(2)–C(3) and C(3)–C(4) and gives the direction of the carbon chain. M lies in the plane of C(2), C(3) and C(4) and is perpendicular to L. N is perpendicular to L and M. The orientation angles are collected in Table 2, and the co-ordinates of the atoms with respect to the molecular axes are given in Table 3.

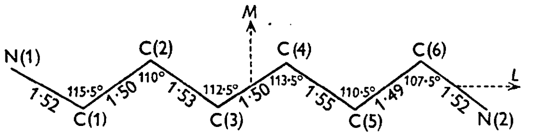

Fig. 7. Dimensions of the chain molecule.

Discussion

It is important to try and assess the limits of error in the bond-length determinations for this structure, but the task is difficult. The degree of resolution obtained in the electron-density maps is high, owing to the use of very complete Fourier series in the final summations. The relatively high atomic number of the chlorine ions will, however, tend to reduce the accuracy with which the light atoms can be placed, because a large part of the structure amplitude must on the average be due to the chlorine contribution. There is also some doubt about the x co-ordinates in the b-axis projection (Fig. 6) owing to superposition of certain atoms. In view of these factors we would estimate the limits of error in the chain bond lengths at about ±0·04 A.

It may, therefore, be unprofitable to discuss the rather interesting pattern of bond-length variations recorded in the saturated chain, because these variations may be due to errors. The length obtained for the terminal C–N bonds (equal at 1·52 A.) is slightly greater than other recent measurements, e.g. 1·465 A. in methylammonium chloride (Hughes & Lipscomb, 1946) and

1·49 A. in geranylamine hydrochloride (Jeffrey, 1945). If this bond is regarded as slightly longer than normal, then the sequence in the chain is long, short, long, short, ... throughout, the central bond, C(3)–C(4), being a short one.

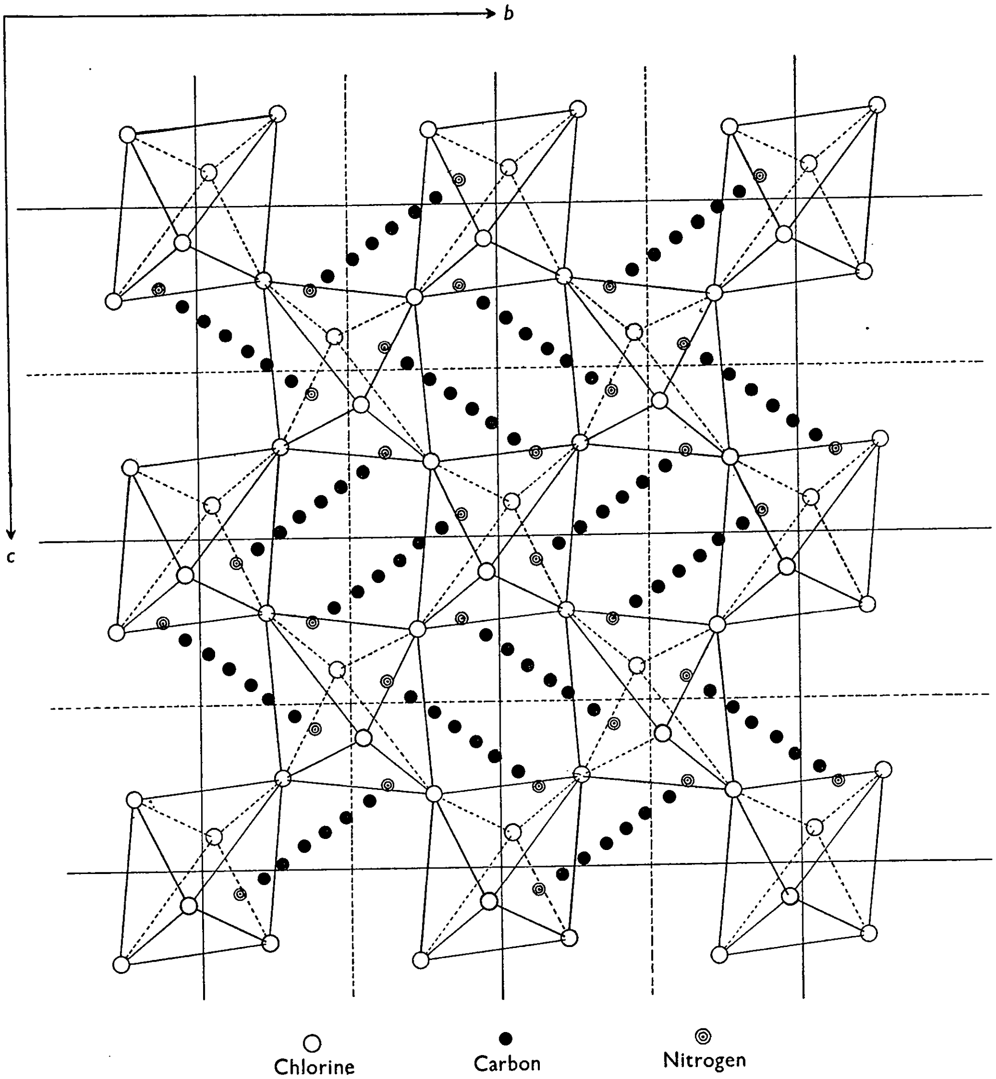

Fig. 8. Arrangement of halogen ions with respect to the chain molecules, viewed along the *a* axis.

The geometry of the chain molecule approximates closely to that of a centrosymmetrical co-planar structure, with both the terminal nitrogen atoms slightly removed from this plane in the same direction, so that the bonds N(1)–C(1) and N(2)–C(6) make angles of about 7° and 10° with the plane of the zigzag chain. This deviation is probably real and may be required in order to achieve a better fitting of the nitrogen atoms into the ionic lattice.

The arrangement of the chlorine ions is illustrated in Fig. 3 and on a smaller scale in Fig. 8. A different arrangement of the chlorine ions exists at the two ends of the chain molecule. This is best seen from Fig. 3. The nitrogen atom N(1) is closely associated with two chlorine ions at distances of 3·10 and 3·12 A., and with two other chlorine ions one translation farther along the *a* axis at distances of 3·27 and 3·58 A. (compare Fig. 6). The nitrogen atom N(2) is associated with three chlorine ions at distances of 3·25, 3·26 and 3·21 A. (average, 3·24 A.) and with a fourth chlorine ion farther along the *a* axis at 3·37 A.

There are thus two fairly distinct types of N–Cl distance in this structure, averaging 3·11 and 3·24 A. The latter value corresponds closely to the sum of the ionic radius for chlorine, taken as 1·81 A. (Pauling, 1940), and that for the ammonium ion, taken as 1·40 A. (Hughes & Lipscomb, 1946), this sum being 3·21 A. This value is also in good agreement with the values of 3·24 and 3·17 A. found in geranylamine hydrochloride (Jeffrey, 1945).

The shorter distances of 3·10 and 3·12 A. are less than the sum of the ionic radii, and they correspond closely to the N–Cl distances found by Donohue & Lipscomb (1947) in hydrazinium dichloride. The existence of hydrogen bonds between the chlorine and the nitrogen atoms is likely to be the explanation of these shorter distances.

The shortest Cl–Cl distance is about 4·3 A., between Cl(1) and Cl(2). The compact grouping of the chlorine ions in the lattice is illustrated in Fig. 8, where the chlorine ions in each unit cell are seen to form an array of distorted octahedra with common vertices.

The principal halogen–nitrogen and halogen–halogen distances are summarized in Table 4, where the numbering of the atoms corresponds to Fig. 3 and Table 1 and the letters *A*–*D* refer to equivalent positions in the space group, as follows:

$$A\colon x, y, z; \qquad C\colon x, \tfrac{1}{2}-y, \tfrac{1}{2}+z;$$
$$B\colon -x, -y, -z; \qquad D\colon -x, \tfrac{1}{2}+y, \tfrac{1}{2}-z.$$

Primed letters indicate an atom one translation along the *a* axis, above or below the atom shown in Fig. 3.

Table 4. *Principal* N–Cl *and* Cl–Cl *distances*

N (1*D*)–Cl (1*A*) = 3·10 A.
N (1*D*)–Cl (2*A*) = 3·12 A.
N (1*D*)–Cl (1*A*′) = 3·27 A.
N (1*D*)–Cl (2*A*′) = 3·58 A.
N (2*A*)–Cl (2*A*) = 3·25 A.
N (2*A*)–Cl (2*B*) = 3·26 A.
N (2*A*)–Cl (1*C*) = 3·21 A.
N (2*A*)–Cl (2*A*′) = 4·42 A.
N (2*A*)–Cl (2*B*′) = 3·37 A.
N (2*A*)–Cl (1*C*′) = 4·17 A.
Cl (1*A*)–Cl (1*A*′) = 4·60 A.*
Cl (1*A*)–Cl (2*A*) = 4·29 A.
Cl (1*D*)–Cl (2*A*) = 5·58 A.
Cl (1*C*)–Cl (2*A*) = 5·77 A.
Cl (2*B*)–Cl (2*A*) = 5·08 A.

* *a* translation.

Experimental

The dihydrochloride was prepared by saturation of a methanolic solution of hexamethylenediamine with dry hydrochloric acid gas (Braun & Müller, 1905). The slender needle crystals obtained were recrystallized from a mixture of ethyl alcohol and water in the proportion of 20 to 1. Density measurements were made by flotation of the crystals in a mixture of carbon tetrachloride and benzene.

All the X-ray work was carried out with Cu $K\alpha$ radiation, $\lambda = 1{\cdot}54$ A. Rotation, oscillation and moving-film photographs were used. The intensity estimates were made on moving-film exposures of the equatorial layer lines for crystals rotated about the principal axes. Small crystal specimens were used, completely bathed in a uniform X-ray beam. The cross-sections of the crystals, normal to the rotation axes, were 0·18 by 0·18 mm., 0·26 by 0·28 mm., and 0·53 by 0·56 mm., for the (0*kl*), (*h*0*l*) and (*hk*0) zones respectively. In view of these uniform dimensions, absorption corrections were not applied. The multiple-film technique (Robertson, 1943) was used to correlate the strong and weak reflexions over a range of about 1500 to 1, the estimations being made visually. The values of the structure amplitude were derived by the usual formulae (mosaic crystal) and are listed in Table 5. The absolute scale was derived by correlation with the *F* values calculated from the final co-ordinates.

The Fourier syntheses were carried out by the usual methods, the axial subdivisions being $\frac{1}{30}a = 0{\cdot}153$ A., $\frac{1}{60}b = 0{\cdot}236$ A., and $\frac{1}{60}c = 0{\cdot}261$ A. For the final summations, three-figure methods were used (Robertson, 1948*a*).

The structure factors were recalculated from the final co-ordinates with the results shown in Table 5. The average discrepancies, expressed as the sum of all the discrepancies divided by the total of the measured structure factors (absent reflexions being omitted from the count), are 15·9 % for the (0*kl*) zone, 16·3 % for the (*h*0*l*) zone, and 18·2 % for the (*hk*0) zone, the overall value being 16·6 %. In these calculations the atomic scattering curves for the chlorine ions and for carbon were taken from the *International Tables for the Determination of Crystal Structure*, and corrected for temperature according to the Debye-Waller formula, the value of the constant *B* in the factor $\exp[-B(\sin\theta/\lambda)^2]$ being taken as $2{\cdot}0 \times 10^{-16}$.

The nitrogen atoms were treated as carbon, the same scattering curve being employed.

In conclusion, one of us (W. P. B.) desires to express his thanks to Messrs Imperial Chemical Industries Ltd. for a grant which enabled him to take part in this work, and for a generous supply of hexamethylenediamine. He is also indebted to the Department of Scientific and Industrial Research for a further grant during a later period of the work.

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Table 5. *Measured and calculated values of the structure factor*

hkl	$F_{\text{meas.}}$	$F_{\text{calc.}}$
100	39	−33
200	45·5	+49·5
300	13·5	−15·5
400	41·5	−38·5
500	11	−12
020	< 2	− 2·5
040	47·5	+56·5
060	23·5	−24
080	6·5	+ 4
0,10,0	26	−24·5
0,12,0	29	−28
0,14,0	< 6	− 1
0,16,0	10	−12·5
0,18,0	8·5	+ 9
002	11	−12
004	13	+ 8
006	48	−43·5
008	20·5	+15·5
0,0,10	34	+36·5
0,0,12	19·5	−17·5
0,0,14	< 6	− 2·5
0,0,16	8	−12·5
0,0,18	15	+20·5
0,0,20	< 2·5	+ 4
110	30·5	−34·5
120	10·5	−12
130	< 3	− 1
140	56	+59
150	43·5	−45
160	26	−23
170	5·5	+ 2·5
180	15·5	−11
190	19	−18
1,10,0	20·5	−16
1,11,0	7·5	− 7
1,12,0	19·5	−17·5
1,13,0	< 5·5	+ 0·5
1,14,0	7·5	− 8
1,15,0	< 5	− 0·5
1,16,0	18	−20
1,17,0	12·5	+12
1,18,0	3	+ 8
210	48	−44
220	29	−25
230	< 4	+ 3·5
240	11·5	+ 9
250	37·5	−43
260	6·5	+ 1·5
270	42	−39
280	20	+20
290	26	−23·5
2,10,0	5·5	+ 3
2,11,0	8	−10
2,12,0	< 5·5	− 3
2,13,0	13·5	+13
2,14,0	7	+ 7
2,15,0	< 4·5	+ 2
2,16,0	< 3·5	− 3
2,17,0	12·5	+18
310	32·5	−32
320	23·5	−21
330	17	+10
340	8·5	− 5
350	41·5	−45·5
360	18·5	−14·5
370	8	+11·5
380	5·5	− 7
390	14	−14
3,10,0	9·5	+ 8·5
3,11,0	< 5·5	− 3·5
3,12,0	10·5	+12
3,13,0	8	+ 6·5
3,14,0	10	+ 9
3,15,0	< 3·5	+ 1
410	22·5	−20·5
420	8·5	− 5·5
430	16	+19·5
440	11·5	−17·5
450	< 5·5	− 3
460	16	+11·5
470	< 5·5	+ 2·5
480	9	+13
490	< 5	− 3·5
4,10,0	13·5	+11·5
4,11,0	< 4·5	− 2
4,12,0	13	+12
4,13,0	< 3	+ 1
510	7	− 3·5
520	< 5	− 5
530	14	+17·5
540	15	−14
550	< 4·5	− 0·5
560	< 4·5	+ 3
570	11·5	+14
580	6	− 2
590	4·5	+ 7·5
5,10,0	< 2	+ 8·5
1,0,18	18	+18·5
1,0,16	22·5	−25·5
1,0,14	6	+ 3
1,0,12	6	+ 9
1,0,10	46	+43·5
108	11	+10
106	53	−56
104	9	+ 7·5
102	24·5	−19·5
10$\bar{2}$	105	−96
10$\bar{4}$	45	−38·5
10$\bar{6}$	22	+16
10$\bar{8}$	14·5	+16·5
1,0,$\overline{10}$	29·5	+29·5
1,0,$\overline{12}$	73·5	−70
1,0,$\overline{14}$	< 6	+ 6·5
1,0,$\overline{16}$	< 5·5	− 7·5
1,0,$\overline{18}$	16·5	+19·5
2,0,18	7·5	+11
2,0,16	22	−27
2,0,14	< 6	+ 1·5
2,0,12	< 6	+ 4
2,0,10	17	−24·5
208	< 5	+ 2
206	68·5	−65·5
204	66·5	+61·5
202	11	+ 7
20$\bar{2}$	46	−35·5
20$\bar{4}$	24	−20·5
20$\bar{6}$	37·5	+42
20$\bar{8}$	< 5·5	− 0·5
2,0,$\overline{10}$	< 6	+12
2,0,$\overline{12}$	< 6	− 5
2,0,$\overline{14}$	23·5	+24
2,0,$\overline{16}$	12·5	+14
2,0,$\overline{18}$	3·5	+ 2·5
3,0,16	7	− 9
3,0,14	9	− 9·5
3,0,12	33·5	+38
3,0,10	10·5	+ 9
308	< 6	− 2
306	31·5	−24·5
304	56	+46·5
302	< 5	− 0·5
30$\bar{2}$	< 5	− 3
30$\bar{4}$	5·5	− 7·5
30$\bar{6}$	50·5	+52·5
30$\bar{8}$	29	−28
3,0,$\overline{10}$	< 6	− 0·5
3,0,$\overline{12}$	35	−44
3,0,$\overline{14}$	< 5	+ 5
3,0,$\overline{16}$	6·5	+ 6·5
4,0,14	< 3·5	+ 1·5
4,0,12	4·5	+ 6·5
4,0,10	38	−46·5
408	6	+ 4·5
406	6	+ 0·5
404	19	+23·5
402	8·5	− 7·5
40$\bar{2}$	10·5	−11·5
40$\bar{4}$	< 6	− 1
40$\bar{6}$	24	+21·5
40$\bar{8}$	20	−22·5
4,0,$\overline{10}$	< 5·5	− 2·5
4,0,$\overline{12}$	8	+13·5
4,0,$\overline{14}$	9·5	+15·5
5,0,10	9·5	−15
508	7·5	+12
506	< 5	+ 4
504	5	+ 5
502	< 5·5	− 3·5
50$\bar{2}$	42·5	+47
50$\bar{4}$	5	+ 2·5
50$\bar{6}$	< 4·5	+10·5
50$\bar{8}$	12	−19
5,0,$\overline{10}$	< 3	− 1·5
011	32	+28
012	2	− 3·5
013	14	+ 7·5
014	53	+49
015	43·5	−38·5
016	56	−55·5
017	4	− 5
018	20·5	+21·5
019	34	+37
0,1,10	18·5	−20
0,1,11	5·5	− 4
0,1,12	18	+17
0,1,13	16·5	−15
0,1,14	< 6	− 0·5
0,1,15	8·5	−10·5
0,1,16	10	− 9·5
0,1,17	12	+13
0,1,18	6·5	+10·5
0,1,19	< 4	+ 5
0,1,20	< 2·5	− 4
021	8·5	+ 3
022	10·5	+13
023	67	−53·5
024	118	−111
025	29·5	−28·5
026	9	− 8
027	23·5	−15·5
028	12	+ 8
029	11·5	+ 7·5
0,2,10	5	+ 3
0,2,11	9·5	+ 8·5
0,2,12	8	+11·5
0,2,13	13	+14
0,2,14	34	−33·5
0,2,15	14·5	−18
0,2,16	16	+16
0,2,17	< 5	+ 1
0,2,18	< 4·5	+ 3·5
0,2,19	< 3·5	− 8
031	7	+10
032	68	+60·5
033	14	+12·5
034	15·5	−12
035	65·5	−60·5
036	5·5	− 4·5
037	16·5	+12
038	47	−50
039	11·5	+11·5
0,3,10	32·5	+38
0,3,11	16	+15·5
0,3,12	< 5·5	+ 1·5
0,3,13	20·5	−18·5

Table 5 (*cont.*)

hkl	$F_{\text{meas.}}$	$F_{\text{calc.}}$
0,3,14	< 6	+ 8·5
0,3,15	< 6	− 1·5
0,3,16	16	−16·5
0,3,17	12	+14
0,3,18	9	−14·5
0,3,19	< 3·5	+ 6
041	74	+63
042	27	−27
043	11	+11·5
044	3·5	− 1
045	8·5	+15·5
046	24	+19·5
047	10·5	− 7·5
048	17	+18·5
049	5	− 6
0,4,10	30	+30·5
0,4,11	35·5	+36
0,4,12	20	−22
0,4,13	< 6	− 4·5
0,4,14	< 6	+ 7·5
0,4,15	< 6	− 2·5
0,4,16	17·5	−22·5
0,4,17	7	− 6·5
0,4,18	11	+13·5
0,4,19	8	+11·5
051	9·0	+13
052	27·5	+28
053	16	−16
054	52	+49·5
055	9	+ 8·5
056	68	−62
057	17	−20
058	8·5	+ 8·5
059	35·5	+38
0,5,10	< 5·5	− 0·5
0,5,11	< 5·5	− 8·5
0,5,12	33	+33
0,5,13	13·5	−13
0,5,14	< 6	+ 3
0,5,15	8	−10
0,5,16	15	−14·5
0,5,17	6·5	− 7
0,5,18	< 4	+ 1
0,5,19	< 3	+ 6
061	10·5	− 8
062	18·5	+19·5
063	70	+67·5
064	18	−18·5
065	24·5	−26·5
066	8	+ 6·5
067	23·5	−22·5
068	5	− 3
069	21·5	−18
0,6,10	25	−23·5
0,6,11	10	+ 5·5
0,6,12	10·5	+10·5
0,6,13	< 6	+ 7·5
0,6,14	< 6	− 6·5
0,6,15	31·5	−30·5
0,6,16	7	+ 8
0,6,17	< 4·5	+ 1
0,6,18	< 3·5	− 4
071	9·5	−10·5
072	47·5	+43
073	17	+17·5
074	11	+ 7
075	18·5	−17·5
076	18	−16
077	17·5	+16
078	30·5	−30·5
079	11	−12·5
0,7,10	5·5	+ 9
0,7,11	20·5	+20·5
0,7,12	< 6	− 4
0,7,13	< 6	− 7
0,7,14	10	− 8
0,7,15	< 5·5	+ 2
0,7,16	12	−13·5
0,7,17	< 4	+ 6·5
0,7,18	< 3	− 5
081	53·5	+50·5
082	18	−14·5
083	< 5	+ 1
084	25·5	+23
085	11	+10
086	< 5	− 1·5
087	30·5	−28
088	5·5	− 3
089	< 5·5	− 6·5
0,8,10	23·5	−19·5
0,8,11	41·5	+42
0,8,12	8·5	+ 8·5
0,8,13	< 6	+ 2·5
0,8,14	15·5	+18·5
0,8,15	< 5	+ 4·5
0,8,16	< 4·5	+ 0·5
0,8,17	10·5	−16·5
0,8,18	< 2·5	+ 3
091	< 5	− 2
092	22	+20·5
093	9	− 9
094	10·5	+10·5
095	33·5	+36
096	9·5	−12
097	45·5	−44
098	8	+ 6
099	< 6	+ 0·5
0,9,10	13·5	+12
0,9,11	17	−17
0,9,12	11·5	+12·5
0,9,13	12·5	+12
0,9,14	< 5	+ 3
0,9,15	9·5	+12
0,9,16	5·5	− 6·5
0,9,17	4	− 4
0,10,1	12	−14
0,10,2	< 5·5	+ 1·5
0,10,3	31	+30
0,10,4	12·5	+13
0,10,5	11	−11
0,10,6	13	+13·5
0,10,7	29	−24
0,10,8	6	− 6·5
0,10,9	6	+ 5·5
0,10,10	17	−16·5
0,10,11	< 6	− 1·5
0,10,12	8	+ 5·5
0,10,13	13·5	+22
0,10,14	6·5	+ 9
0,10,15	14·5	−23
0,10,16	5·5	− 9·5
0,11,1	19·5	−20·5
0,11,2	< 5·5	+ 1
0,11,3	36·5	+31·5
0,11,4	20	−16
0,11,5	16·5	+12·5
0,11,6	14·5	−15
0,11,7	22	+20·5
0,11,8	< 6	− 3
0,11,9	29	−32
0,11,10	< 6	+ 1·5
0,11,11	8	+11
0,11,12	7·5	+ 5
0,11,13	< 5	+ 0·5
0,11,14	< 4	− 1·5
0,11,15	8	+12·5
0,12,1	14·5	+14
0,12,2	< 6	+ 5·5
0,12,3	10	−10
0,12,4	6	+ 4·5
0,12,5	10·5	+12
0,12,6	15·5	+ 9
0,12,7	8·5	− 7·5
0,12,8	13	−11
0,12,9	< 5·5	− 5·5
0,12,10	9·5	−10·5
0,12,11	10·5	+11·5
0,12,12	10·5	+ 9·5
0,12,13	9·5	−11
0,12,14	8	+11
0,12,15	< 2·5	+10·5
0,13,1	34	−30·5
0,13,2	8·5	+10
0,13,3	6	− 8
0,13,4	6	− 7
0,13,5	29	+32
0,13,6	11·5	+12
0,13,7	14	−15
0,13,8	< 5·5	− 1
0,13,9	< 5·5	− 0·5
0,13,10	< 5	+ 0·5
0,13,11	10	−13·5
0,13,12	< 4	− 1
0,13,13	6	+10
0,13,14	< 2·5	+ 0·5
0,14,1	28·5	−30·5
0,14,2	11·5	−14
0,14,3	8	+ 8·5
0,14,4	16	+17·5
0,14,5	< 5·5	− 3·5
0,14,6	5·5	+ 4
0,14,7	7·5	+ 9·5
0,14,8	10	−10·5
0,14,9	8	+ 5
0,14,10	< 4·5	− 6·5
0,14,11	5·5	− 9·5
0,14,12	< 3	+ 1
0,15,1	< 5·5	− 4
0,15,2	< 5·5	− 0·5
0,15,3	15	+22·5
0,15,4	7·5	+ 7
0,15,5	5	+ 5
0,15,6	< 5	+ 2
0,15,7	6·5	+ 9·5
0,15,8	10·5	+13
0,15,9	12·5	−16·5
0,15,10	10·5	−16·5
0,15,11	< 2·5	+ 2
0,16,1	< 5	− 2·5
0,16,2	< 5	+ 3·5
0,16,3	15	−19
0,16,4	< 4·5	+ 5·5
0,16,5	8·5	+12·5
0,16,6	6	+ 9
0,16,7	6·5	+ 7
0,16,8	< 3·5	− 3·5
0,17,3	< 4	− 2
0,17,4	11	−18·5
0,17,5	< 3	− 0·5