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The Crystal Structure of Pyrazoline Hydrochloride

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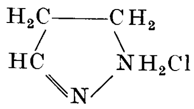
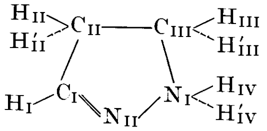
Pyrazoline hydrochloride, $C_3H_7N_2Cl$, crystallizes in the orthorhombic space group D_{2h}^{16}–*Pnma* with unit-cell dimensions

$$a = 10{\cdot}43,\ b = 5{\cdot}71,\ c = 8{\cdot}99\ \text{Å},$$

and with $Z = 4$. The structure has been determined from (010) projections and refined by Booth three-dimensional differential syntheses using $h0l$, $h1l$ and $h2l$ reflections. The organic ring lies in a mirror plane, with bond distances indicating a remarkable localization of the C = N double bond. The whole crystal structure is determined by the packing of Cl^-'s, which form a zig-zag chain around a 2_1 axis parallel to [010]. Each organic cation is bonded, through its NH_2^+ group, to two Cl^- ions.

Pyrazoline hydrochloride





crystallizes from anhydrous alcoholic solutions in colourless prisms elongated along the *b* axis. The crystals are hygroscopic, and so the one used for X-ray analysis was sealed in a Lindemann capillary. The unit-cell dimensions and space group were determined from rotation and Weissenberg photographs (Cu $K\alpha$ radiation) with the crystal rotating about the *b* axis. The following data were obtained:

$C_3H_7N_2Cl$; $M = 106{\cdot}6$; m.p. = 128 °C.

Orthorhombic,

$$a = 10{\cdot}43 \pm 0{\cdot}01,\ b = 5{\cdot}71 \pm 0{\cdot}01,\ c = 8{\cdot}99 \pm 0{\cdot}01\ \text{Å}.$$

Volume of the unit cell = 535·4 Å^3.
Four molecules per unit cell.
Calculated density = 1·32 g.cm.$^{-3}$.
$F(000) = 224$.
Absent spectra: $0kl$ when $k+l$ is odd, and $hk0$ when h is odd.
Space group: D_{2h}^{16}–*Pnma* or C_{2v}^{9}–$Pn2_1a$.

For the structural analysis the $h0l$, $h1l$ and $h2l$ reflections only were considered, their intensities being determined photometrically from integrated and non-integrated Weissenberg photographs (multiple film technique). 86 independent $h0l$ reflections, 71$h1l$ and 64$h2l$ were observed, representing 72, 61 and 59% respectively of the possible number observable.

To correct for absorption, the sample was treated as cylindrical ($\bar{r} = 0{\cdot}02$ cm., $\mu = 50{\cdot}4$ cm.$^{-1}$ for Cu $K\alpha$ radiation). The structure amplitudes were derived by the usual formulae, the absolute scale for each layer being established first by Wilson's method, then by correlation with the calculated values.

Structure analysis and refinement

As a starting hypothesis it was assumed that the space group was *Pnma*. This choice implies the planarity of the organic ring which must lie in the fourfold (*c*) positions (International Tables nomenclature) on the mirror planes. The Cl's also lie on these positions; they cannot occupy the fourfold (*a*) or (*b*) positions (on the centres of symmetry) because of steric hindrance. From these assumptions there were no overlappings in the (010) projection, so that the 'heavy-atom method' could be used succesfully.

The sharpened $P(U, W)$ Patterson projection (Fig. 1) shows the rotation peak *C* and the reflection peaks *A* and *B* due to the Cl–Cl interactions. The signs of the contributions of Cl atoms alone were applied to the structure factors $F(h0l)$ and the resultant first $\varrho(X, Z)$ projection showed all the atoms of the pentagonal ring well resolved.

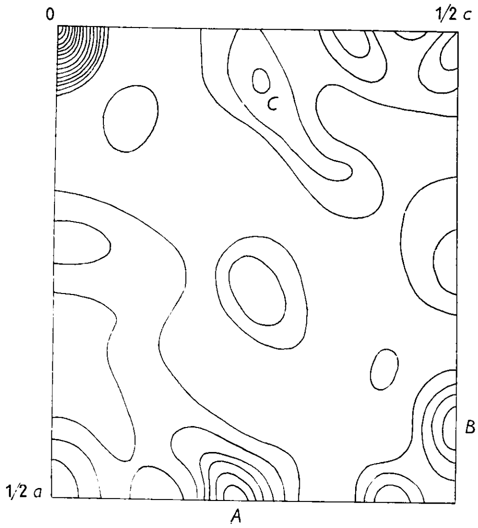


Fig. 1. Sharpened $P(U, W)$ projection Contours at arbitrary intervals.

Table 1. *Observed and calculated structure factors*

A minus sign for $|F_o|$ means 'less than'

h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c
0 0 2	30.2	-33.3	6 0 10	1.1-	0.0	2 1 4	4.9	-4.4	9 1 1	5.2	5.4	4 2 3	13.9	11.2
4	34.4	-37.6	7 0 1	3.0	-3.0	5	23.0	-20.8	2	3.5	4.5	4	22.7	21.0
6	4.2	4.9	2	7.0	-6.8	6	2.9-	0.0	3	3.3-	-3.0	5	21.6	-20.0
8	16.7	17.9	3	10.0	10.3	7	3.2-	-6.0	4	3.2-	0.8	6	3.5-	-3.6
10	9.7	-9.4	4	2.1-	2.2	8	5.0	5.2	5	3.1-	-0.3	7	5.1	-6.3
1 0 1	9.7	10.3	5	2.2-	-1.7	9	8.7	9.2	6	2.9-	2.9	8	9.8	-10.1
2	27.3	-29.8	6	15.3	15.2	10	5.2	-4.7	7	2.4-	4.2	9	7.9	8.2
3	41.2	-47.1	7	2.0-	-1.0	11	2.0	-2.1	8	2.9	-3.2	10	2.2-	1.1
4	17.1	-16.0	8	6.8	-6.4	3 1 1	17.2	16.9	10 1 0	3.3-	4.0	5 2 1	2.7-	-1.1
5	10.4	7.6	9	2.5	-2.7	2	36.3	35.0	1	5.4	-6.4	2	22.1	22.3
6	14.0	14.3	8 0 0	9.6	-7.2	3	37.9	-37.1	2	3.2-	0.5	3	6.0	-4.7
7	9.9	9.3	1	9.9	-10.0	4	11.8	-10.8	3	6.3	-6.6	4	3.3	0.4
8	7.4	-7.0	2	9.9	8.7	5	7.7	-7.5	4	5.1	-5.4	5	8.1	-8.5
9	2.8	4.2	3	11.3	-10.5	6	19.9	-17.8	5	2.8	3.8	6	16.7	-16.7
10	1.8-	-1.2	4	6.0	-6.0	7	12.2	12.0	6	2.4-	-0.1	7	3.5-	3.8
11	7.4	-7.0	5	6.5	6.6	8	7.7	8.0	7	1.8-	0.8	8	7.9	7.6
2 0 0	58.4	65.5	6	2.1-	0.8	9	5.5	5.6	11 1 1	3.0-	-0.4	9	3.3	3.0
1	15.4	12.6	7	1.9-	-0.3	10	6.0	5.0	2	2.9-	-1.4	10	1.8-	3.3
2	12.2	-10.3	8	1.6-	-0.6	11	3.4	-3.9	3	2.7-	-1.5	6 2 0	2.9-	-0.6
3	16.9	-13.9	9	5.1	-6.0	4 1 0	34.0	35.0	4	2.5-	1.0	1	3.0-	0.0
4	27.9	-28.5	9 0 1	13.1	-12.5	1	10.3	-7.9	5	2.2-	1.2	2	6.0	5.8
5	25.5	26.7	2	4.5	-5.5	2	2.1-	-0.9	6	2.6	3.5	3	14.5	14.1
6	15.3	15.0	3	10.8	10.5	3	2.3-	2.6	12 1 0	2.5-	0.2	4	3.4-	4.4
7	2.1-	2.4	4	4.8	3.4	4	14.1	-13.2	1	2.5-	-1.3	5	14.5	-14.3
8	9.9	9.1	5	2.2	2.8	5	2.9-	-0.3	2	2.4-	0.7	6	3.6-	-1.7
9	4.8	-4.8	6	4.3	4.6	6	9.3	8.5	3	3.5	-4.4	7	3.5-	4.1
10	8.6	-8.6	7	3.4	-2.6	7	7.8	7.8	4	1.8-	1.6	8	3.1-	-2.5
11	2.2	2.4	8	1.3-	0.6	8	5.6	4.4	13 1 1	1.6-	-1.2	9	6.1	7.0
3 0 1	17.3	16.8	10 0 0	2.2-	2.0	9	3.3	3.9	2	1.4-	1.6	7 2 1	3.3-	2.3
2	18.2	-20.3	1	2.1-	-2.1	10	5.9	-5.7	0 2 2	21.1	21.6	2	7.6	6.0
3	15.5	-14.3	2	3.9	3.3	5 1 1	12.8	12.5	4	31.2	28.4	3	8.7	-8.5
4	1.5-	-3.2	3	2.1-	1.8	2	15.5	16.2	6	6.4	-4.4	4	3.5-	-1.9
5	1.8-	-2.3	4	2.0-	1.7	3	23.7	-22.7	8	15.5	-15.5	5	3.6-	1.5
6	8.3	7.1	5	1.8-	1.1	4	3.0	-4.1	10	8.6	8.2	6	12.0	-13.1
7	8.8	8.3	6	1.6-	-1.8	5	3.0-	2.7	1 2 1	7.2	-6.4	7	3.2-	0.9
8	13.1	-12.8	7	1.3-	-2.3	6	17.7	-17.7	2	19.8	20.0	8	5.3	5.6
9	2.0-	-1.6	11 0 1	4.1	-5.1	7	8.3	8.2	3	36.6	33.1	9	1.9-	2.4
10	1.7-	-2.8	2	1.9-	-1.9	8	3.1-	1.9	4	9.4	10.0	8 2 0	7.3	6.4
11	3.7	-4.0	3	1.9	3.8	9	2.7-	0.2	5	5.5	-5.1	1	7.2	8.6
4 0 0	51.5	46.8	4	1.7-	-1.1	10	2.0-	1.2	6	10.9	-11.6	2	7.4	-7.4
1	27.3	-24.8	5	1.5-	-0.1	6 1 0	38.1	38.7	7	9.6	-8.5	3	9.1	9.1
2	18.2	-16.5	6	1.2-	0.8	1	4.0	-3.4	8	6.3	6.1	4	3.6-	4.9
3	15.3	-13.4	12 0 0	3.0	-2.7	2	18.8	-17.2	9	3.3-	-3.7	5	5.5	-5.7
4	29.8	-26.9	1	1.7-	-0.4	3	2.8-	1.6	10	2.7-	1.1	6	3.3-	-0.6
5	24.3	23.9	2	3.3	3.2	4	12.7	-12.2	11	4.7	6.2	7	2.9-	0.2
6	4.2	4.3	3	1.5-	0.2	5	6.4	6.1	2 2 0	47.0	-48.6	8	2.3-	0.6
7	6.8	7.3	4	1.3-	-0.3	6	3.3-	1.3	1	5.4	-7.1	9 2 1	10.0	10.8
8	12.1	11.7	5	0.9-	2.1	7	4.1	4.0	2	9.7	7.3	2	4.1	4.8
9	8.8	-9.4	13 0 1	4.0	-3.5	8	9.6	9.8	3	12.3	9.9	3	9.0	-9.0
10	1.6-	-1.2	2	1.0-	-0.1	9	2.5-	-0.5	4	25.5	22.8	4	3.4-	-3.0
5 0 1	4.2	2.1	0 1 1	37.7	-35.9	10	1.6-	-1.6	5	23.1	-21.5	5	3.2-	-2.5
2	28.3	-27.5	3	16.9	15.7	7 1 1	16.3	17.2	6	12.0	-12.4	6	2.9-	-4.0
3	5.0	6.1	5	37.8	-36.3	2	3.0-	-0.1	7	3.5-	-2.0	7	2.4-	2.2
4	1.8-	-1.4	7	3.8	2.6	3	15.0	-15.1	8	5.1	-7.9	10 2 0	3.5-	0.0
5	11.9	10.5	9	8.6	9.0	4	5.3	-5.8	9	4.3	4.2	1	3.5-	1.8
6	20.0	19.6	1 1 1	14.1	-10.8	5	3.3-	1.9	10	7.3	7.4	2	3.4-	-2.9
7	4.7	-4.7	2	59.2	68.6	6	3.3-	2.9	3 2 1	12.9	-12.5	3	3.3-	-1.6
8	8.8	-8.6	3	21.0	-16.3	7	3.1-	3.4	2	18.6	15.9	4	3.1-	-1.5
9	4.2	-3.5	4	6.3	5.7	8	2.7-	-2.9	3	12.8	11.3	5	2.8-	-1.0
10	3.2	-3.8	5	12.9	11.3	9	2.1-	1.0	4	4.3	2.7	6	2.3-	1.5
6 0 0	1.7-	0.7	6	34.3	-31.1	8 1 0	4.7	4.2	5	3.1-	2.3	11 2 1	3.1-	4.4
1	5.5	1.5	7	3.1-	-0.2	1	3.1-	-0.9	6	8.5	-6.2	2	3.7	1.7
2	8.3	-7.5	8	19.9	19.5	2	6.4	-6.6	7	7.1	-7.2	3	2.8-	-3.3
3	19.7	-17.4	9	3.1-	2.2	3	13.6	-13.4	8	11.6	11.0	4	2.4-	0.2
4	4.4	-5.5	10	6.6	6.0	4	4.8	-5.5	9	3.1-	1.4	5	2.1-	0.1
5	16.8	16.6	11	2.9	-2.8	5	11.1	10.3	10	2.5-	2.4	12 2 0	2.5-	2.6
6	2.2-	2.0	2 1 0	57.5	74.0	6	3.1-	3.6	4 2 0	28.7	-31.2	1	2.4-	0.5
7	4.4	-4.8	1	26.8	24.5	7	2.8-	1.3	1	17.6	18.6	2	2.3-	-2.8
8	2.1	3.0	2	12.3	-9.9	8	2.3-	2.9	2	12.2	11.4	3	2.0-	-0.1
9	8.0	-8.0	3	19.1	15.9	9	3.0	-3.6						

It is of interest that an independent application of the 'coincidences' method (Grant, Howells & Rogers, 1957) gave for 62$h0l$ ($U \geq 0{\cdot}15$) reflections the same signs as the Cl atom contributions.

A first structure-factor calculation in which all the atoms of the organic cation were considered as carbon atoms, gave $R(h0l) = 0{\cdot}270$. At this stage the N and C atoms were separated in the $\varrho(X, Z)$ projection and a first set of isotropic thermal parameters was obtained: $R(h0l)$ dropped to 0·171. A further isotropic refinement was obtained with a $\Delta\varrho(X, Z)$ synthesis ($R(h0l) = 0{\cdot}158$).

Table 2. *Final atomic coordinates and their s.d.*

	x/a	y/b	z/c	x (Å)	y (Å)	z (Å)	$\sigma(x)$ $\times 10^4$ (Å)	$\sigma(z)$ $\times 10^4$ (Å)
Cl	0·5414	0·7500	0·3604	5·6468	4·2825	3·2400	22	14
N_I	0·4501	0·2500	0·2823	4·6945	1·4275	2·5379	43	41
N_{II}	0·4996	0·2500	0·1294	5·2108	1·4275	1·1633	50	54
C_I	0·4041	0·2500	0·0444	4·2148	1·4275	0·3992	72	68
C_{II}	0·2761	0·2500	0·1137	2·8797	1·4275	1·0222	64	62
C_{III}	0·3066	0·2500	0·2736	3·1978	1·4275	2·4597	73	80

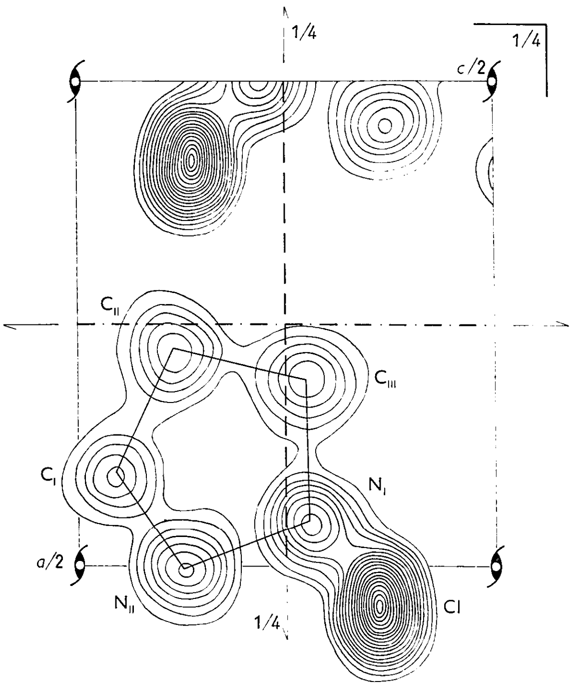


Fig. 2. $\varrho(X, Z)$ final projection. The contour interval is 1 e.Å^{-2}, and the lowest contour is at 2 e.Å^{-2}.

The final $\varrho(X, Z)$ projection is shown in Fig. 2. This indicates that the thermal motion of the chlorine atom is strongly anisotropic.

The complete refinement was then carried out by means of five cycles of Booth's 'differential synthesis', using all the recorded reflections; the anisotropic temperature parameters were determined following the method of Nardelli & Fava (1960) and refined by the procedure of Cruickshank (1956). The necessary computations were done with the IBM 650 programmes of Brown, Lingafelter, Stewart & Jensen (1959) for structure factors and those of Shiono (1957, 1959) for differential synthesis and refinement of thermal parameters. The refinement was considered complete when all the shifts of the atomic coordinates were less than their standard deviations. The R values were: $R(h0l) = 0·101$, $R(h1l) = 0·084$, $R(h2l) = 0·090$.

The last step was the introduction of the hydrogen atoms, assuming that they are arranged tetrahedrally around C_{II}, C_{III} (C–H = 1·09 Å) and N_I (N–H = 1·03 Å) and trigonally around C_I (C–H = 1·08 Å). Their coordinates are:

	x/a	y/b	z/c
H_I	0·417	0·250	−0·052
H_{II}	0·222	0·406	0·083
H_{III}	0·265	0·406	0·325
H_{IV}	0·490	0·397	0·329

An isotropic B value 3·5 Å^2 was taken for all H atoms; this is the mean value of B for the C and N atoms.

The structure factors F_c reported in Table 1 are those calculated with H contributions and the coordinates obtained from the last differential synthesis. These coordinates with their standard deviations (Cruickshank, 1949) are reported in Table 2. The corresponding R (observed reflections only) and R' values (including $F_o = \frac{1}{2}F_{\text{min.}}$ when $F_c \geq F_{\text{min.}}$ for unobserved reflections) are:

$$R(h0l) = 0·093,\ R(h1l) = 0·081,\ R(h2l) = 0·089,$$
$$R'(h0l) = 0·104,\ R'(h1l) = 0·101,\ R'(h2l) = 0·122.$$
$$R\ (\text{over all}) = 0·088,\ R'\ (\text{over all}) = 0·108.$$

The standard deviation of the electron density is $\sigma(\varrho) = 0·114$ e.Å^{-3}. Thermal parameters and descrip-

Table 3. *Thermal parameters* (Å^2)

	B_{11}	B_{22}	B_{33}	B_{13}
Cl	6·85	3·44	2·98	0·26
N_I	3·65	3·31	2·73	0·10
N_{II}	3·36	3·77	3·93	1·40
C_I	4·44	3·67	2·66	−0·48
C_{II}	4·30	3·99	3·57	0·00
C_{III}	3·97	4·17	3·78	0·35

Table 4. *Atomic peak heights* (e.Å^{-3}) *and curvatures* (e.Å^{-5})

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{hl}
Cl	obs.	15·33	108·99	34·06	181·47	0·81
	calc.	15·22	110·84	33·27	181·31	0·80
N_I	obs.	6·78	55·49	13·22	61·27	0·17
	calc.	6·56	54·80	12·63	61·78	−0·09
N_{II}	obs.	4·77	47·79	8·95	47·11	0·55
	calc.	4·78	49·72	9·05	44·36	1·30
C_I	obs.	3·58	33·26	6·64	37·18	−2·02
	calc.	3·81	34·67	6·66	37·18	−1·85
C_{II}	obs.	4·37	37·30	7·90	40·75	−1·08
	calc.	4·32	37·73	8·29	40·82	−0·88
C_{III}	obs.	4·23	32·96	6·82	31·87	2·65
	calc.	4·21	32·46	7·56	32·22	2·75

tion of the peaks are reported in Table 3 and 4 respectively. These are strongly influenced by the omission of reflections with $k \geq 3$ ($k_{\text{max.}} = 7$ for Cu $K\alpha$ radiation) which reduces the values of ϱ, A_{hh} and A_{ll} to a half, and the values of A_{kk} to a tenth.

The atomic scattering factors used through-out the calculations are those of Berghuis *et al.* (1955) for C, N, Cl^- and that of McWeeny (1951) for H.

Discussion

The R values obtained at the end of the refinement operations, being practically the same for different values of k, indicate that the space group *Pnma* is the correct one, at least within the limits justified by the accuracy of the intensity measurements. The planarity of the organic cations is therefore required by the presence of the mirror planes in which they lie. The bond distances and angles (Fig. 3) are:

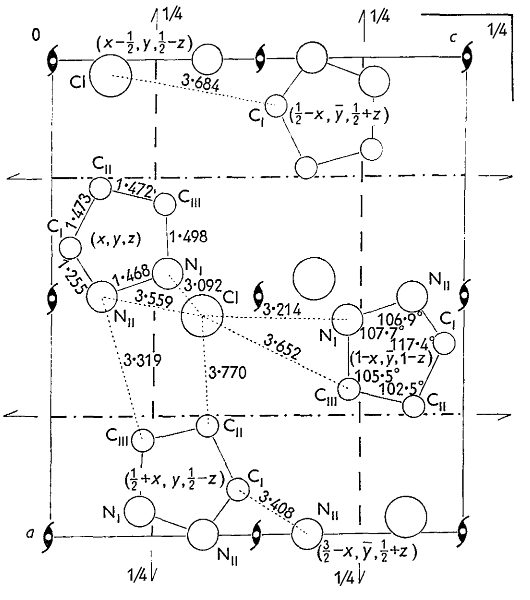


Fig. 3. Diagrammatic projection of a unit-cell on (010).

N_I–N_{II}	1·468 ± 0·007 Å
N_{II}–C_I	1·255 ± 0·009
C_I–C_{II}	1·473 ± 0·010
C_{II}–C_{III}	1·472 ± 0·010
C_{III}–N_I	1·498 ± 0·008
∠ C_I–C_{II}–C_{III}	102·5 ± 0·6°
∠ C_{II}–C_{III}–N_I	105·5 ± 0·6
∠ C_{III}–N_I–N_{II}	107·7 ± 0·4
∠ N_I–N_{II}–C_I	106·9 ± 0·5
∠ N_{II}–C_I–C_{II}	117·4 ± 0·6

The N_{II}–C_I distance corresponds well with the mean value of those generally assumed for N=C double bond (1·24 Å, Vaughan & Donohue, 1952; Penfold, 1953; 1·28 Å, Wheatley, 1955; 1·265 Å, Donohue, Lavine & Rollett, 1956). The N_I–N_{II} distance is consistent with the N–N single bond (1·47 Å, Bryden, 1958), while the C_{III}–N_I distance is significantly longer than that generally assigned to the C–N single bond (1·47 Å). It is probable that this standard value, which involves three-covalent nitrogen, must not be used for a true single bond, as conjugation effects can still occur. Perhaps the limiting value for a C–N single bond can be found when both atoms are in a sp^3 bond configuration, as in the present case. This view is supported by the value found for the C–NH_3^+ distance in amino-acids (mean value: 1·50 Å), amino-peptides (mean value: 1·49 Å) and aliphatic amine ions (mean value: 1·50 Å) (Donohue, Lavine & Rollett, 1956; a large list is quoted by Hahn, 1957). The lack of conjugation produces an increase of the distance greater than the decrease due to the presence of positive charge on the N atom. The two C–C distances are equal and noticeably shorter than the standard C–C single bond; perhaps the difference can be accounted for by some hyperconjugation effects.

If a tetrahedral sp^3 configuration is assumed for N_I, C_{II} and C_{III} and a trigonal sp^2 configuration for C_I and N_{II}, the most severely strained bond angle

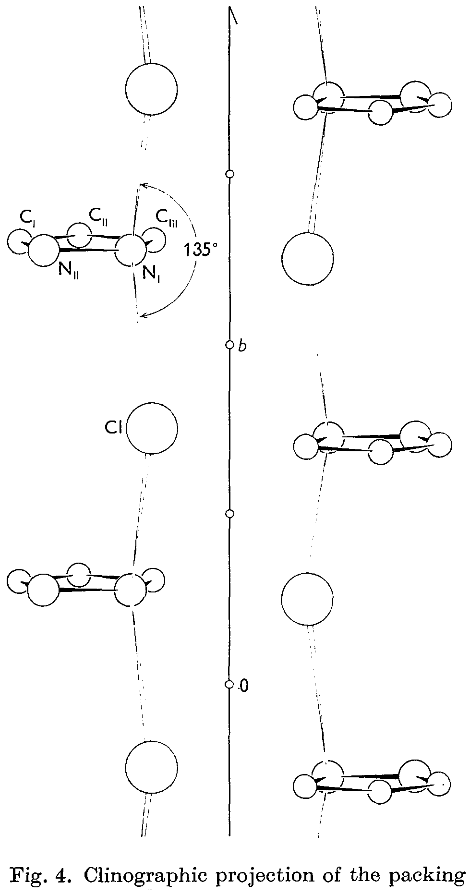


Fig. 4. Clinographic projection of the packing around the 2_1 axis at ($\frac{1}{2}$, y, $\frac{1}{2}$).

is that on N_{II} (106·9°), but greater strains have been found in similar cases (103·0° and 106·7° in pyrazole, Ehrlich, 1960; 102° and 104° in xantazole, Nowacki & Bürki, 1955; 103° and 104° in 1,3-dimethyl-5-iminotetrazole hydrochloride, Bryden, 1955). The other angles are smaller by only a few degrees than the theoretical ones.

The interactions between the organic cations and the Cl^-'s occur through the H atoms bonded to N_I; the distance

$$N_I\text{–}Cl \quad 3{\cdot}092 \pm 0{\cdot}005 \text{ Å}$$

indicates a strong hydrogen bond which is formed owing to the high difference of charge between NH_2^+ and Cl^-. The N_I atom is bonded to two Cl^- ions, one above and the other below the plane of the cation (Fig. 4). The angle $Cl\text{–}N_I\text{–}Cl = 135{\cdot}0°$ is much larger than the tetrahedral angle, but the difference is justified by the repulsion exercised by a Cl^- lying between two Cl^- bonded to the same N_I. The Cl^-'s are packed around a 2_1 axis parallel to [010] and form a zigzag chain with $Cl\text{–}Cl = 3{\cdot}904$ Å contacts.

The following packing distances all involve van der Waals or longer contacts (when the coordinates are not indicated, the atom is at x, y, z):

$Cl\ \text{–}C_I(1-x, \frac{3}{4}, \bar{z})$	$3{\cdot}684 \pm 0{\cdot}007$ Å
$Cl\ \text{–}C_{II}(\frac{1}{2}+x, \frac{1}{4}, \frac{1}{2}-z)$	$3{\cdot}770 \pm 0{\cdot}006$
$Cl\ \text{–}C_{III}$	$3{\cdot}843 \pm 0{\cdot}006$
$Cl\ \text{–}C_{III}(1-x, \frac{3}{4}, 1-z)$	$3{\cdot}652 \pm 0{\cdot}008$
$Cl\ \text{–}N_I(1-x, \frac{3}{4}, 1-z)$	$3{\cdot}214 \pm 0{\cdot}004$
$Cl\ \text{–}N_{II}$	$3{\cdot}559 \pm 0{\cdot}005$
$C_I\ \text{–}C_I(1-x, \frac{3}{4}, \bar{z})$	$3{\cdot}578 \pm 0{\cdot}007$
$C_I\ \text{–}N_{II}(1-x, \frac{3}{4}, \bar{z})$	$3{\cdot}408 \pm 0{\cdot}006$
$C_{II}\ \text{–}N_I(x-\frac{1}{2}, \frac{1}{4}, \frac{1}{2}-z)$	$3{\cdot}526 \pm 0{\cdot}008$
$C_{II}\ \text{–}N_{II}(x-\frac{1}{2}, \frac{1}{4}, \frac{1}{2}-z)$	$3{\cdot}695 \pm 0{\cdot}008$
$C_{III}\text{–}N_I(x-\frac{1}{2}, \frac{1}{4}, \frac{1}{2}-z)$	$3{\cdot}751 \pm 0{\cdot}008$
$C_{III}\text{–}N_{II}(x-\frac{1}{2}, \frac{1}{4}, \frac{1}{2}-z)$	$3{\cdot}319 \pm 0{\cdot}009$
$N_{II}\text{–}N_{II}(1-x, \frac{3}{4}, \bar{z})$	$3{\cdot}685 \pm 0{\cdot}007$

It is interesting to observe that the whole structure is practically determined by the packing of Cl^- ions. There are no interactions among the organic cations which are separated by 5·71 Å in the [010] direction, so there is a relatively empty space between two rings.

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