of the cell constants and Drs R. Olthof-Hazekamp for writing and modifying many of the programs used. We are indebted to Dr K. Toman, Dr B. Sedlaček and Prof. E. H. Wiebenga for their interest in this work. We thank the staff of the Computing Centre of the University, Groningen, for their cordial cooperation.

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Hydrogen Bond Studies. XVII.* The Crystal Structure of Dimethylammonium Chloride

By Jan Lindgren and Ivar Olovsson

Institute of Chemistry, University of Uppsala, Uppsala, Sweden

(Received 11 May 1967)

The crystal structure of dimethylammonium chloride, $(CH_3)_2NH_2Cl$, has been determined from threedimensional single-crystal X-ray data obtained at room temperature. The crystals are orthorhombic, space group *Ibam*, with eight formula units in the cell of dimensions $a=7\cdot301$, $b=14\cdot515$, $c=9\cdot926$ Å. The structure contains infinite planar chains of chlorine and nitrogen atoms linked together by hydrogen bonds of lengths $3\cdot12$ and $3\cdot09$ Å. The dimethylammonium and chloride ions form a distorted NaCltype structure.

Introduction

In conjunction with the study of simple hydrogenbonded structures in progress at this Institute an investigation of the changes in the N-H \cdots Cl bond lengths accompanying replacement of hydrogen in ammonium by methyl groups is of considerable interest. The crystal structure of monomethylammonium chloride was studied by X-ray diffraction technique by Hughes & Lipscomb (1946).

In the present investigation the crystal structure of dimethylammonium chloride has been determined from single-crystal X-ray diffraction data obtained at 20 °C.

Experimental

The sample used was commercial dimethylammonium chloride. The salt was recrystallized three times from chloroform, and dried in a vacuum desiccator as described by Whitlow & Felsing (1944). The crystals had to be handled in a dry-box as they were highly hygroscopic. In the first attempts powder samples were sealed in glass capillaries and single crystals were grown by a

zone-melting technique, the growth of the crystals being followed with a polarizing microscope. The melting point was found to agree with that earlier published, 167°C (Hunter & Byrkit, 1932). Most of the crystals thus obtained had their crystallographic axes orientated at an angle of 45° to the capillary axis. It proved very difficult to obtain a crystal with suitable orientation. Weissenberg photographs demonstrated that the crystals were probably twinned; furthermore the X-ray reflexions became unusually diffuse on passing from lower to higher layers. Owing to these difficulties crystals grown from solution were tried instead. Plate-like crystals were obtained with chloroform alone as a solvent, but from a solution with equal amounts of chloroform and ethyl alcohol needle-like crystals with a nearly circular cross-section were obtained. One of these with a diameter of 0.15 mm and a length of approximately 2 mm was chosen and sealed in a thin-walled glass capillary. An appropriate crystallographic axis was thus obtained parallel to the capillary axis. With this, the a axis, as rotation axis, equi-inclination Weissenberg photographs, layers $0 \le h \le 6$, were recorded with Ni-filtered Cu Ka radiation. It was now clear that the previous crystal, grown by the zone-melting procedure, was indeed a twin

^{*} Part XVI: Acta Cryst. (1967), 23, 971.

composed of two or more components rotated 90° about c in such a way that the *a* axis of some components coincided with the *b* axis of the rest. Owing to the particular type of twinning it was not possible to detect the twinning phenomenon with a polarizing microscope. This strong tendency to twinning is readily explained in terms of the atomic configuration as shown in the section dealing with the description of the structure.

The number of independent reflexions recorded was 507, but the intensities of 121 of these were too weak to be measured. The relative intensities were estimated visually employing the multiple film technique (5 films) and comparison with an intensity scale. The data were corrected for the Lorentz and polarization effects. No absorption corrections were applied; the linear absorption coefficient is 50.0 cm⁻¹ for Cu $K\alpha$; and for a crystal radius of 0.008 cm, $\mu R = 0.4$.

The crystal was also mounted in a Weissenberg camera modified for operation at low temperatures (Olovsson, 1960) and gradually cooled. Oscillation photographs were taken at 25° intervals from -25° to -150°C and finally at -186°C to investigate possible phase transformations. No such transformation was observed but the crystal was evidently subjected to strong thermal strains as the X-ray reflexions became progressively more diffuse as the temperature was lowered. However, the intensity distribution remained the same as on the photographs taken at room temperature.

Unit cell and space group

The diffraction symmetry mmm with systematic absences for hkl with h+k+l odd, for h0l with h odd and 0kl with k odd indicate either the non-centrosymmetric space group *Iba2* or the centrosymmetric *Ibam*. As will be shown below, the structure is appropriately described in terms of the space group *Ibam* (No. 72, *International Tables for X-ray Crystallography*, 1952).

The b and c axes were determined from quartz-calibrated zero-layer Weissenberg photographs employing the following constants: a=4.9128 Å for α -quartz at 18 °C; λ Cu $K\alpha_1=1.54051$ and λ Cu $K\alpha_2=1.54433$ Å. The most probable b and c dimensions were then derived from the observed θ values by the least-squares method. The a axis was determined from a quartz-calibrated rotation photograph (c=5.4045 Å for α -quartz at 18 °C). The cell dimensions together with their estimated standard deviations are

 $a = 7.301 \pm 0.002, b = 14.515 \pm 0.001, c = 9.926 \pm 0.003$ Å.

A rough estimate of the density was obtained by measuring the volume and weight of a large homogeneous sample in a glass tube. The measured density was 1.01 ± 0.05 g.cm⁻³ as compared with a value of 1.030 g.cm⁻³ calculated with a cell content of eight formula units.

Determination of the crystal structure

The positions of the chlorine atoms were determined from a three-dimensional Patterson synthesis. The interpretation was then based on the centrosymmetric space group *Ibam*. At first the chlorine atoms were placed on the glide plane b. A Fourier synthesis based on these positions gave a very poorly resolved electron density map, evidently due to the introduction of too high a symmetry. However, when the chlorine atoms were moved away from the glide plane the Fourier synthesis gave well-resolved nitrogen and carbon peaks. Carbon was thus found to occupy the 16(k) positions while nitrogen and chlorine each occupies an 8(j) set in the space group *Ibam*.

The preliminary atomic coordinates were first improved in a three-dimensional electron density calculation. The Patterson and Fourier syntheses were all carried out with the crystallographic programs STRIX and PROFFS (Liminga & Olovsson, 1964) written for the computer FACIT EDB.

A series of least-squares refinements was then calculated, using the program SFLS written by Åsbrink & Brändén (1962). A block-diagonal approximation is used in this program which minimizes the function $\Sigma w(|F_o| - |F_c|)^2$. The weighting function was that suggested by Cruickshank, Pilling, Bujosa, Lovell & Truter (1961), namely $w = 1/(a + |F_0| + c|F_0|^2)$, in which the final values for the constants were a = 5.0 and c = 0.01. Only observed reflexions were included in the refinements. During these refinements, individual isotropic temperature factors and individual scale factors for the separate layers were refined. The structure was successfully refined in accordance with the space group *Ibam.* When all shifts in the parameters were less than one tenth of the estimated standard deviations the Rvalue was: $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 11.8 \%$. A difference synthesis was now calculated to locate the hydrogen atoms. The hydrogen atoms thus determined were introduced with fixed positional and temperature parameters in the subsequent refinements (see below). The isotropic temperature factors for the methyl hydrogen atoms were set to 10 Å², and for the others to 7 Å². Three least-squares cycles were then carried out with the full matrix program OR FLS (Busing, Martin & Levy, 1962), otherwise under the same conditions as above, giving a R value of 10.7 %. The above difference synthesis showed characteristic dumb-bell formed peaks, indicating considerable thermal anisotropy. An anisotropic refinement with one over-all scale factor was therefore performed giving an *R*-value of 8.2%. The inter-layer scaling was then based on the previously obtained individual scale factors. The hydrogen atoms were included with the same fixed parameters as above. In total only five more parameters were refined by allowing for thermal anisotropy.

The structure was also refined in *Iba2*, starting with the preliminary coordinates derived from the Patterson synthesis but with the atoms shifted somewhat from the centrosymmetric positions. A significance test was made on the *R*-factor ratio as described by Hamilton (1965) with the hypothesis that *Ibam* rather than *Iba2* is the correct space group. The ratio obtained after final anisotropic refinements is 1.004. According to Hamilton's table the hypothesis cannot be rejected even at the 50 % level. The space group *Ibam* is therefore considered to be the best choice. The differences in the final atomic coordinates and interatomic distances in the two space groups are within one standard deviation obtained using *Iba2*.

The atomic parameters with standard deviations after final refinement in *Ibam* are found in Table 1. The observed and calculated structure factors are listed in Table 2. The atomic scattering factors used were those for Cl⁻, N⁰, C⁰, and H⁰ listed in *International Tables for X-ray Crystallography* (1962, p. 202).

Table 1. Atomic parameters with standard deviations ($\times 10^4$)

The form of the anisotropic temperature factor is: $\exp \left[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+2b_{12}hk+2b_{13}hl+2b_{23}kl)\right]$. The r.m.s. components (R_i) of thermal displacement of the atoms along the ellipsoid axes are also listed (Å × 10³).

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	Chlorine	Nitrogen	Carbon
x	2116 (3)	2887 (10)	2131 (12)
у	1392 (1)	3510 (5)	3912 (5)
Z	0	0	1222 (7)
<i>b</i> ₁₁	228 (6)	270 (21)	416 (22)
b_{22}	51 (1)	60 (4)	85 (4)
b_{33}	150 (3)	120 (8)	118 (8)
b_{12}	1 (2)	9 (6)	30 (8)
b_{13}	0	0	24 (10)
b23	0	0	8 (4)
R_1	233 (2)	245 (8)	239 (8)
R_2	248 (4)	247 (8)	287 (8)
R_3	274 (3)	274 (10)	350 (9)

Table 2. Observed and calculated structure factors

Reflexions too weak to be measured are marked with an asterisk and the values of $|F_o|$ given correspond to minimum observable intensities.

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	000222222	45.0 177.4 18.2 9.0 2.1* 26.0 13.8 40.7	43.3 204.1 20.0 7.8 0.3 20.3 10.5 38.5			1 164 1 164 1 108 1 108 1 64 1 108	1 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	1 1 4 27.2 1 16 5 5.1 1 14 5 7.0 1 12 5 6.0 1 10 5 25.8 1 8 5 25.8 1 6 5 32.8 1 4 5 32.8	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1 1 4 27,2 28,0 2 1 16 5 5,1 5,1 2 1 14 5 7,0 5,8 2 1 12 5 6,0 6,7 2 1 10 5 20,3 17,8 2 1 10 5 20,3 17,8 2 1 8 5 5,6 5,4 2 1 6 5 32,8 35,8 2 1 4 5 5 4,7 1,8 2	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1 16 5 77.2 28.0 2 2 4 24.2 1 16 5 5.1 2 0 4 4 24.2 1 16 5 7.0 5.8 2 15 5 1.2 1 14 5 5 6.0 6.7 2 15 5 1.6 1 12 5 20.3 17.8 2 11 5 2.1 1 10 5 20.3 17.8 2 11 5 2.1 1 8 5 5.4 2 9 5 9.1 1 8 5 32.8 35.8 2 7 5 1.8 1 4 5 32.8 35.8 2 7 5 1.8 1 4 5 32.8 35.8 2 7 5 1.8 1 4 5 32.8 35.8 35.8 2 7 5 1.8 1 4 5 32.8 35.8 35.8 35.8 3 1.8 1 4 5 3 32.8 35.8 35.8 3 2 7 5 1.8 1 4 5 3 32.8 35.8 35.8 35.8 3 2 7 5 1.8 1 4 5 3 32.8 35.8 35.8 35.8 3 2 7 5 1.8 1 4 5 3 32.8 35.8 35.8 35.8 3 1.8 1 5 3 32.8 35.8 35.8 3 1.8 1 6 5 3 32.8 35.8 35.8 3 2 7 5 1.8 1 7 8 5 32.8 35.8 35.8 35.8 3 2 7 5 1.8 1 8 8 5 8 8 8 5 8 8 5 8 8 5 8 8 5 8 8 5 8	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
	~~~~~~~~~~	26.0 13.8 40.7 53.6 106.1 33.2 135.2	20.3 10.5 38.5 42.9 54.6 110.1 37.0 173.3		1 1 1 1 1	1 8 1 6 1 2 1 13 1 11 1 9	1 8 5 1 6 5 5 1 2 5 1 13 6 1 11 6 1 9 6 7	1 8 5 5.6 1 6 5 32.8 1 4 5 4.7 1 2 5 47.7 1 13 6 2.0* 1 11 6 2.2* 1 9 6 2.2* 1 7 6 2.7	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{smallmatrix} 1 & 8 & 5 & 5 & 6 & 5 & 4 & 2 \\ 1 & 6 & 5 & 32 & 8 & 35 & 8 & 2 \\ 1 & 4 & 5 & 4 & 7 & 1 & 8 & 2 \\ 1 & 4 & 5 & 4 & 7 & 7 & 51 & 4 & 2 \\ 1 & 13 & 6 & 2 & 0^* & 1 & 8 & 2 \\ 1 & 11 & 6 & 2 & 2^* & 0 & 6 & 2 \\ 1 & 11 & 6 & 2 & 2^* & 0 & 6 & 2 \\ 1 & 19 & 6 & 2 & 2^* & 0 & 6 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 6 & 8 & 7 & 9 & 4 & 2 \\ 1 & 7 & 8 & 8 & 1 & 1 \\ 1 & 1 & 1 & 1 & 1 & 1 \\ 1 & 1 &$	1     8     5     5.6     5.4     2     9       1     6     5     32.8     35.8     2     7       1     4     5     32.4     7     1.8     2     3       1     2     5     47.7     1.8     2     3       1     2     5     47.7     1.8     2     1       1     13     6     2.2*     4.9     2     1       1     11     6     2.2*     0.4     2     2     12       1     9     6     2.2*     0.4     2     2     12       1     9     6     8.7     9.4     2     2     12	1     8     5     5.6     5.4     2     9     5       1     6     5     32.8     35.8     2     7     5       1     4     5     32.4     7     1.8     2     7     5       1     2     5     47.7     1.8     2     3     5       1     1.3     6     2.0*     1.8     2     1     5       1     1.3     6     2.2*     4.9     2     1     5       1     1.9     6     2.2*     0.4     2     2     12     6       1     9     6     8.7     9.4     2     2     12     6       1     7     6     8.7     9.4     2     12     12     6	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
	24444444	135.2 27.3 6.7 42.7 25.5 56.9 50.2	173.3 21.3 6.3 40.2 22.5 53.5 49.9			1 5 1 3 1 12 1 10 1 8	7 6 1 5 6 1 12 7 1 10 7 1 8	1 7 6 8.7 1 5 6 5.3 1 3 6 12.3 1 1 6 11.2 1 12 7 9.2 1 10 7 9.4 1 8 7 11.0	1 7 6 8.7 9.4 1 5 6 5.3 3.1 1 3 6 12.3 12.5 1 1 6 11.2 9.9 1 12 7 9.2 9.6 1 10 7 9.4 8.4 1 8 7 11.0 11.9			7     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6     6	7     5     6     7     7     10     6     17     8     6     10     10     6     17     10     6     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10	1     7     8     6     17     10     6     17     10     18     6     17     10     18     10     18     10     18     10     18     10     18     10     18     10     18     10     18     10     18     10     18     10     18     10     18     10     18     10     10     18     10     10     18     10     10     12     10     18     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10     10	1     7     6     6     17.8     17.1     17.8     17.1     17.8     17.1     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     17.8     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1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1</td> <td>7     6     6     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     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1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1</td> <td>1     7     0     6     7     0     6     7     1     1     6     6     1     2     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     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    1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1	1     7     0     6     7     0     6     7     1     1     6     6     1     2     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1    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	4446666	50.2 38.2 57.4 15.1 6.7 26.7 28.0	49.9 36.9 64.1 12.8 22.8 23.2 23.2		1	1 8 1 6 ! 4 1 2 1 11 1 9 1 7	1 8 7 1 6 7 1 2 7 1 11 8 1 9 8 1 7 8	1 8 7 11.0 1 6 7 13.6 1 4 7 7.3 1 2 7 9.5 1 11 8 1.9* 1 9 8 2.2* 1 7 8 4.0	1 8 7 11.0 11.9 1 6 7 13.6 13.5 1 4 7 7.3 8.1 1 2 7 9.5 14.7 1 1 8 1.9* 3.7 1 9 8 2.2* 1.9 1 7 8 4.0 5.6	1     8     7     11.0     11.9     2       1     6     7     13.6     13.5     2       1     4     7     7.3     8.1     2       1     2     7     9.5     14.7     2       1     2     7     9.5     14.7     2       1     1     8     1.9*     3.7     2       1     9     8     2.2*     1.9     3.7     2       1     7     8     4.0     5.6     2     2       1     7     8     4.0     5.6     2     2	1 8 7 11.0 11.9 2 13 1 6 7 13.6 13.5 2 11 1 4 7 7.3 8.1 2 9 1 2 7 9.5 14.7 2 7 1 11 8 1.9* 3.7 2 5 1 9 8 2.2* 1.9 2 3 1 7 8 4.0 5.6 2 1 1 7 4 7 5	1   8   7   11.0   11.9   2   13   7     1   6   7   13.6   13.5   2   11   7     1   4   7   7.3   8.1   2   9   7     1   2   7   7.5   14.7   2   7   7     1   2   7   7.5   14.7   2   7   7     1   1   8   1.9*   3.7   2   5   7     1   9   8   2.2*   1.9   2   3   7     1   9   8   4.2*   5.6   2   1   7     1   9   8   4.0   5.6   2   1   7     1   9   8   4.0   5.6   2   1   7     1   9   8   4.0   5.6   2   1   7	1   8   7   11.0   11.9   2   13   7   1.6**     1   6   7   13.6   13.5   2   11   7   2.6**     1   4   7   7.3   8.1   2   9   7   6.2     1   2   7   7.5   8.1   2   9   7   7.3     1   1   8   1.9*   1.4,7   2   7   7.4     1   9   8   2.2*   1.9   2   3   7   7.4     1   9   8   2.5*   3   7   7.6   3   7   7.4     1   9   8   2.5*   1.9   2   3   7   7.4     1   9   8   5.6   2   1   7   9.6     1   7   8   5.6   2   1   9   9.6	1   8   7   11.0   11.9   2   13   7   7.6*   3.3     1   6   7   13.6   13.5   2   11   7   2.0*   1.1     1   4   7   7.3   8.1   2   9   7   6.2   6.3     1   2   7   7   6.3   4.8   1.1   1.9   2.5   7   7.4   9.0     1   1.9   8   2.2*   1.9   2   3   7   7.8   8.9   0.2     1   9   8   2.2*   1.9   2   3   7   7.8   8.9   0.2     1   9   8   2.2*   1.9   2   3   7   7.8   8.9   0.2     1   9   8   5.6   2   1   7   8.6   10.2     1   2   8   5.6   2   1   7   8.6   10.2     1   7   8   5.6   2   1   7   8.6   10.2	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
064 20411		27.5 52.8 15.9 65.0 5.8	26.8 48.4 14.7 63.6 5.3		1	1 5 1 3 1 12 1 10 1 8	1 / 0 1 5 8 1 3 8 1 12 9 1 10 9 1 8 9	1 / 8 4.0 1 5 8 5.6 1 1 8 5.1 1 1 8 2.1 1 12 9 5.1 1 10 9 4.8 1 8 9 8.7	1 / 0 4.0 3.0 1 5 8 4.0 0.0 1 3 8 5.6 6.1 1 1 8 2.1* 2.6 1 12 9 5.1 6.2 1 10 9 4.8 5.1 1 8 9 8.7 7.6	1 7 6 4.0 5.6 2 1 8 4.0 0.0 2 1 3 8 5.6 6.1 2 1 1 8 2.14 2.6 2 1 12 9 5.1 6.2 2 1 10 9 4.8 5.1 2 1 8 9 8.7 7.6 2	1     7     0     4.0     5.0     2     14       1     5     8     4.0     0.0     2     14       1     3     8     5.6     6.1     2     12       1     1     8     2.1     4.0     0.0     2     14       1     3     8     5.6     6.1     2     12       1     1     8     2.1     4.8     5.1     2     18       1     12     9     5.1     6.2     2     18       1     10     9     4.8     5.1     2     6       1     8     9     8.7     7.6     2     4	1     7     6     4.0     5.6     2     1     8       1     5     8     4.0     0.0     2     14     8       1     3     8     5.6     6.1     2     12     8       1     1     8     2.14     2.6     2     10     8       1     1     8     2.14     2.6     2     10     8       1     12     9     5.1     6.2     2     8     8       1     10     9     4.8     5.1     2     6     8       1     8     9     8.7     7.6     2     4	1     7     8     4.0     5.0     4.1     6     5.5       1     5     8     4.6     0.01     2.12     8     3.5       1     3     8.5     5.5     5.6     2.10     8     8.3       1     1.2     9     5.1     6.2     2     8     8     15.0       1     1.0     9     8.7     7.6     2     4     8     28.0       1     8     9     8.7     7.6     2     4     8     28.0	1     7     8     4.0     5.0     4     1.6     5.1     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2     5.2	1     7     8     4:0     5:0     2     1     6     5:5     4:2     3       1     5     8     4:0     0     2     1     6     5:5     4:2     3       1     5     8     1:5     4:0     0     2:1     10     8     3:5     4:1     5:1     1:1     10     8     8:5     1:4:9     3:1     1:1     10     9     4:7     7:6     2     6     8     6:8     9:1     3:1     10     9     8:7     7:6     2     6     8     6:8     9:1     3:1     8:5     1:1     2:0     8     8:5     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1     1:1	1     7     8     4.0     5.0     4     1     6.0     7.0     3     1       1     5     8     4.5     3     1     1     1     5.0     1     1.5     8     1.5     3     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1	1     7     8     4.0     5.6     2     1.6     6     5.6     3     16     6       1     5     8     4.6     0.0     2     1.4     6     5.6     3     11     6       1     3     8     4.6     5     1     2     6     6     5     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1     1	1     7     8     4.0     5.0     4     1     6     5.2     7     6     7     6     7     6     7     6     7     6     7     6     7     6     7     7     6     7     7     6     7     7     6     7     7     6     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     7     8     1     1     8     6     7     7     8     1     1     8     6     7     8     1     1     8     1     1     6     7     8     1     1     6     7     8     1     1     1     1     1     1     1     1     1     1     1     1 </td <td>$\begin{array}{cccccccccccccccccccccccccccccccccccc$</td> <td>$\begin{array}{cccccccccccccccccccccccccccccccccccc$</td> <td>$\begin{array}{cccccccccccccccccccccccccccccccccccc$</td> <td>1     7     8     4.0     5.0     2     1     6     0.2     3     13     8     2     1     0.6     4     2     8       1     5     6     4     1     15     5     4     3     13     8     2     7     3.6     4     2     8       1     3     6     6     2     10     8     8.3     3     13     8     2.7     3.6     4     2     8       1     3     6     6     2.1     10     8     8.5     3     13     8     2.7     3.6     4     2     8       1     12     8     4.15     14.19     3     19     8     4.9     9     9     9     1     12.2     3     13     8     1.1.4     12.2.3     4     7     9     9     1     1     12.3     4     7     9     9     1     1     10</td> <td>1     7     8     4.0     5.6     4     4     8     1     1.8       1     5     8     4.0     5.6     4     4     8     1.1     8       1     5     8     4.0     5.6     4     4     8     1.1     8       1     5     8     4.0     5.6     4     0     8     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6&lt;</td> <td>$\begin{array}{cccccccccccccccccccccccccccccccccccc$</td>	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1     7     8     4.0     5.0     2     1     6     0.2     3     13     8     2     1     0.6     4     2     8       1     5     6     4     1     15     5     4     3     13     8     2     7     3.6     4     2     8       1     3     6     6     2     10     8     8.3     3     13     8     2.7     3.6     4     2     8       1     3     6     6     2.1     10     8     8.5     3     13     8     2.7     3.6     4     2     8       1     12     8     4.15     14.19     3     19     8     4.9     9     9     9     1     12.2     3     13     8     1.1.4     12.2.3     4     7     9     9     1     1     12.3     4     7     9     9     1     1     10	1     7     8     4.0     5.6     4     4     8     1     1.8       1     5     8     4.0     5.6     4     4     8     1.1     8       1     5     8     4.0     5.6     4     4     8     1.1     8       1     5     8     4.0     5.6     4     0     8     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6     1.6<	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
1086474	00000000	10.2 22.5 10.5 45.1 2.3*	10.9 19.2 11.8 37.8 48.5		1	1 6 1 4 1 2 1 7 5	1 6 9 1 6 9 1 2 9 1 7 10 1 5 10	1 6 9 8.7 1 6 9 3.9 1 4 9 6.3 1 2 9 2.2* 1 7 10 1.9* 1 5 10 2.1*	1 8 9 8.7 7.6 1 6 9 3.9 8.1 1 4 9 6.3 5.4 1 2 9 2.2* 3.1 1 7 10 2.1* 0.4 1 5 10 2.1* 0.4	1 6 9 3.9 8.1 2 1 4 9 6.3 5.4 2 1 2 9 2.2* 3.1 2 1 7 10 1.9* 3.1 2 1 5 10 2.1* 0.4 2	1 6 9 8.7 7.6 2 4 1 6 9 3.9 8.1 2 2 1 4 9 6.3 5.4 2 0 1 2 9 2.24 3.1 2 11 1 7 10 1.94 3.1 2 9 1 5 10 2.14 0.4 2 7 1 5 10 2.14 0.4 2 7	1 6 6 9 9 8 6 7 9 8 6 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8		$\begin{array}{cccccccccccccccccccccccccccccccccccc$	0     0     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2     2	0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0	0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0     0  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    4     6     4     6     4     6     4     6     4     6     4     6     6     3     3<	1     6     9     6.7     7.7     4     3     9       1     6     7.7     4     1     9     3     1     8     6.8     7.7     4     1     9       1     6     9     7.6     4     2     8     3.6     1     8     6.8     7.7     4     8     9       1     4     9     9     7.6     4     8     10     9     1.5     4.8     10     10     1     1     1.6     3     8     9     4.6     4.8     10     10     1.2     1     9     1.4     4     10     1.2     1.2     9     9     1.4     4     10     1.4     1.1     3     8     9     1.4     4     10     1.2     1.2     9     9     1.4     2.7     3     3     6     9     1.4     4     10     1.2     1.2     9     9     1.4     2.0	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	0   9   9   7   7   0   0   20   3   3   8   10   1   13   4   3   9   6.5   6.0   4.0   1   1   1   0   1   0   1   0   1   0   1   0   1   0   1   0   1   0   1   0   1   0   1   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Distances and angles with their standard deviations, given in Table 3, were calculated using the program $OR \ FFE$ written by Busing, Martin & Levy (1964). The standard errors of the distances and angles were computed from the errors of the atomic parameters in the form of a variance-covariance matrix, obtained in the final cycle using $OR \ FLS$. The errors of the cell parameters were also included in the error calculation, but their influence was negligible.

Table 3. Distances and angles with standard deviations

Distances	
N-C(1,2)	1.455 (8) Å
$N \cdots Cl(1)$	3.126(7)
$N \cdots Cl(2)$	3.091 (8)
N···Chlorine atoms in adjacent chains	> 4.2
$Cl(1)\cdots$ other chlorine atoms	> 4.8
Angles	
C(1)-N-C(2)	113·0 (0·8)°
$\hat{Cl(1)} \cdots \hat{N-C(1,2)}$	109.0 (0.4)
$Cl(2) \cdots N - C(1,2)$	111.1 (0.4)
$Cl(1) \cdots N \cdots Cl(2)$	103.0(0.2)

Description and discussion of the structure

General

The nitrogen and chlorine atoms are located in the planes z=0 and $z=\frac{1}{2}$, respectively. Fig. 1 gives the content in and close to the plane z=0. The relative positions of the atoms in the plane $z = \frac{1}{2}$ may be derived from the space group symmetry. The nitrogen and chlorine atoms are linked together by hydrogen bonds of 3.12 and 3.09 Å. Infinite planar chains are thus formed in the [100] direction and there are only weak bonds between adjacent chains (cf. Table 3). In the present compound, (CH₃)₂NH₂Cl, both hydrogen atoms bound to nitrogen are expected to be involved in hydrogen bonds. Since the only atom capable of accepting such hydrogen atoms is chlorine it may be concluded that each chlorine atom should, in turn, be hydrogen-bonded to two nitrogen atoms. This is the situation realized in the structure. Corresponding principles can of course be applied to all alkylammonium halides but in practice complications sometimes arise due to orientational disorder or restricted rotation of the groups involved in bonding. The situation will be surveyed in a later paper.

In analogy with many other methyl substituted ammonium halides, dimethylammonium chloride crystallizes with a structure similar to one of the simple inorganic ionic compounds. Dimethylammonium chloride thus has an arrangement of $(CH_3)_2NH_2^+$ and $Cl^$ ions which may be regarded as a distorted sodium chloride structure. The strong tendency of twinning of the present compound is now readily understood in terms of its crystal structure. From Fig. 1 it is evident that only small shifts of the ions are needed to convert the hydrogen bonds along [100] into similar bonds along [010]. In this connexion it is noteworthy that the *b* axis (14.515 Å) is close to twice the *a* axis (7.301 Å).

Bond distances and angles

Distances and angles are shown in Fig. 2 (cf. Table 3). The distances in Table 3 are not corrected for thermal motion. If the carbon atom is assumed to ride on the nitrogen atom the N-C distance changes from 1.455 to 1.476 Å. However, since no experimental scaling has been done and since part of the anisotropy has probably already been accounted for using individual scale factors for the separate layers, distances corrected this way will not be used in the following discussion.

The N-C bonds are 1.455 ± 0.008 Å and the C-N-C angle is $113.0 \pm 0.8^{\circ}$. The corresponding values for



Fig. 1. The plane z=0 as viewed along the c axis. One of the carbon atoms is lying below z=0 and is therefore hidden by the other one.



Fig. 2. Bond distances and angles around nitrogen.



Fig. 3. Part of a hydrogen-bonded chain showing the orientation of the methyl groups. The mirror plane perpendicular to the c axis is indicated.

gaseous dimethylamine are 1.46 ± 0.03 Å and $108 \pm 4^{\circ}$ (Bauer, 1938). Accordingly there is no significant difference in the N-C bond lengths. The bond angle C-N-C is, however, smaller in dimethylamine than in dimethylammonium chloride. This may, of course, be expected from the well-known empirical rule that a lone pair exerts a stronger repulsion than a bonding pair.

Hydrogen

The difference map calculated before the anisotropic refinement (see above) was used to locate the hydrogen atoms.

A very well resolved peak appeared on the 3.09 Å bond at about 1.03 Å from nitrogen. The other peak (on the 3.12 Å bond) was not equally well resolved but plainly distinguishable. These hydrogen atoms were therefore located on the bond axes at 1.03 Å from the nitrogen atom; the coordinates are (0.26, 0.28, 0.0) and (0.43, 0.35, 0.0). The following assumptions were made concerning the location of the methyl hydrogen atoms: (a) the C-H distances are about 1.1 Å, (b) the H-C-H angles are about 109.5°. One of the hydrogen atoms could be quite well located experimentally since the corresponding peak was very well resolved. From the position of this hydrogen and the assumptions made above, the other hydrogen atoms were located within the other two rather diffuse peaks found in the difference map. The coordinates determined are: (0.07, 0.37, 0.12, (0.21, 0.47, 0.12) and (0.29, 0.37, 0.21). These positions, illustrated in Fig.3, correspond to a staggered configuration of the methyl groups relative the bonds around nitrogen. It is also found that the three hydrogen atoms are nearly directed towards the three closest chlorine neighbours which are not hydrogen-bonded to the dimethylammonium ion now considered. The $H \cdots Cl$ distances are approximately 2.85 Å.

N-H···Cl distance

When the N-H \cdots Cl distances in the present compound are compared with those in related substances several effects must be taken into consideration. Thus, when one or more hydrogen atoms in an ammonium ion are replaced by other groups, the N-H \cdots Cl distance is expected to change depending on the difference in electronegativity of the substituents and hydrogen as discussed in a previous paper (Olovsson, 1960). The coordination numbers of both atoms involved in the hydrogen bond must also be considered. A detailed comparison between the $N-H\cdots$ Cl distances in the different alkylammonium halides will be postponed until data on more compounds become available. Structure determinations of such closely related compounds are at present in progress at this Institute.

The authors are indebted to Mr H. Karlsson for his skilful assistance in the preparation of crystals and in the preliminary X-ray work.

This work has been supported by grants from the Swedish Natural Science Research Council and the Malmfonden–Swedish Foundation for Scientific Research and Industrial Development – which are here gratefully acknowledged.

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