Fig. 2. Stereoscopic drawing of the crystal packing. The dashed line indicates the hydrogen bond.

configuration, which is found to be preferred for the N—C—C—O system in a variety of molecules, has been attributed to favorable intramolecular Coulombic interactions (Sundaralingam, 1972). The hydroxyl O6 is brought into proximity with the lactam carbonyl C2, along a pathway suitable for nucleophilic attack (Bürgeil, Dunitz & Shefter, 1974). However, the C···O distance (3·27 Å) is not unusually short and the carbonyl C atom is found to be coplanar within experimental error with the three atoms to which it is covalently bonded. In the crystal structure (Fig. 2), the hydroxyl group forms an intermolecular hydrogen bond with the carbonyl oxygen, O6—H6···O14 having O6···O14 distance 2·78 Å and O—H···O angle 169 (4)°.

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structure of a closely related azetidone derivative (1·342 Å; Lee, Cho, Kim, Shin, Ruble & Craven, 1990) and in the crystal structure of a Δ^2cephalosperin (1·339 Å; Sweet & Dahl, 1970), all three compounds inactive as antibiotics. In contrast, C—N bond lengths of 1·392 (4) Å are reported for an active penicillin derivative by Domiano, Nardelli, Blasmo, Macchia & Macchia (1979). Thus the results from the present structure determination are consistent with the structure–activity relationship proposed by Sweet & Dahl (1970).

As can be seen in Fig. 1, the molecule adopts a conformation with O6 lying over the face of the β-lactam ring. This gives rise to unfavorable intramolecular repulsions which could be avoided in the isolated molecule by rotations of ± 120° about the C4—C5 bond. In the crystal structure, the N1—C4—C5—O6 group with torsion angle − 58° (Table 2) has the *gauche* configuration. The twisted

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Structure of Tetramethylammonium Bromide: a Redetermination

BY DAVID J. EVANS AND DAVID L. HUGHES

AFRC Institute of Plant Science Research, Nitrogen Fixation Laboratory, University of Sussex, Brighton BN1 9RQ, England

Abstract. $C_4H_{12}N^+.Br^-$, $M_r = 154·0$, tetragonal, $P4/nmm$, $a = b = 7·708$ (1), $c = 5·498$ (1) Å, $V = 326·6$ Å^3, $Z = 2$, $D_x = 1·566$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 61·1$ cm^{-1}, $F(000) = 156$, $T = 293$ K, $R = 0·055$ for all 298 unique reflections. In this redetermination in a higher symmetry, the structure

is essentially unchanged, with the Br anions and the cation N atoms on sites of $4mm$ and $\bar{4}2m$ symmetry respectively (rather than the 4 and $\bar{4}$ sites in $P4/n$). However, the methyl groups now have mirror-plane symmetry. In the distorted CsCl-type lattice, the anions are removed from a precise cubic arrangement by strong interactions with surrounding methyl groups.

Introduction. Crystals obtained as a byproduct in the preparation of the iron(II) tetrahalide $[NMe_4]_2[FeBr_4]$ were shown by X-ray analysis to be tetramethylammonium bromide. We note that in a similar investigation of this product (Cheban, Dvorkin, Rotaru & Malinovskii, 1987) the structure was determined in the space group $P4/n$. We report now our analysis with the higher symmetry group $P4/nmm$ (No. 129, origin at $2/m$ centre) and propose that this describes the structure more accurately.

Experimental. Recrystallization from acetonitrile gave almost colourless, translucent crystals which had some good faces but no regular shape.

Being moisture sensitive, crystals were mounted in capillaries in a glove box. One, ca $0.28 \times 0.30 \times 0.40$ mm, showed strong diffraction on photographs and was transferred to our Enraf–Nonius CAD-4 diffractometer (with monochromated radiation) for determination of accurate cell parameters (from the settings of 25 reflections with θ ca $15°$) and measurement of diffraction intensities (to $\theta_{max} = 30°$; h, k each 0–10, l 0–7). During processing, data were corrected for Lorentz–polarization effects, absorption (by semi-empirical ψ-scan methods; T_{min}, T_{max} $= 0.80$, 0.98) and for negative intensities (by Bayesian statistical methods); there was no deterioration of the crystal as evidenced by 2 standard reflections measured every 10 000 s X-ray exposure time.

Intensity data for 298 independent reflections (222 of which have $I > 2\sigma_I$) were used in the *SHELX* program system (Sheldrick, 1976) for determination of the molecular structure (by the heavy-atom method) and refinement (on F, by full-matrix least-squares methods). The Br, C and N atoms were allowed anisotropic thermal parameters; the H atoms, appearing clearly in difference maps, were refined independently with isotropic temperature factors. At convergence, $R = 0.055$, $wR = 0.038$ for all data, weighted $w = \sigma_F^{-2}$; in the final cycle, max. Δ/σ $= 0.04$. A final difference map showed peaks of ca 2.1 and 1.4 e Å^{-3} close to the bromide anion, but nothing else of significance in the structure.

Scattering factors for C, H, Br$^-$ and N atoms were from *International Tables for X-ray Crystallography* (1974). Computer programs used in this analysis (in addition to *SHELX*) are detailed in Anderson,

Richards & Hughes (1986), and were run on a DEC-MicroVAX II system.

Discussion. The crystal structure of this $[NMe_4]^+$ salt (Fig. 1) is very similar to those of the perchlorate (McCullough, 1964) and the hexafluorophosphate (Wang, Calvert & Brownstein, 1980) salts of the same cation. They share the same space group, in which the cation lies on a site of $\bar{4}2m$ symmetry and the anion is at a site of $4mm$ symmetry. In the bromide crystals, there is no sign of any disorder (whereas the perchlorate anion is disordered in its crystals) and the H atoms in the cation were identified and refined well. Atomic coordinates are in Table 1.* The dimensions in the cation are normal and are listed in Table 2.

In the previous report (Cheban *et al.*, 1987) on the structure of tetramethylammonium bromide, described in space group $P4/n$, the positions of the Br and N atoms, at sites of 4 and $\bar{4}$ symmetry, were identical to those in our structure. However, there are differences in the methyl-group arrangements. In the $P4/n$ study, the C atoms are in general positions and, according to the diagram, well removed from the planes $x = 0$ or $\frac{1}{2}$, *etc.*; the x coordinate -0.4988 (21) for this atom, however, is not significantly removed from -0.5. We have tried refining our structure in the lower-symmetry space group and confirm that the C atom comes very close to the plane $x = 0$ (or $\frac{1}{2}$). In $P4/nmm$, this is a plane of mirror symmetry, and the methyl C atom is required to lie on this plane (or to be disordered about the

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52779 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
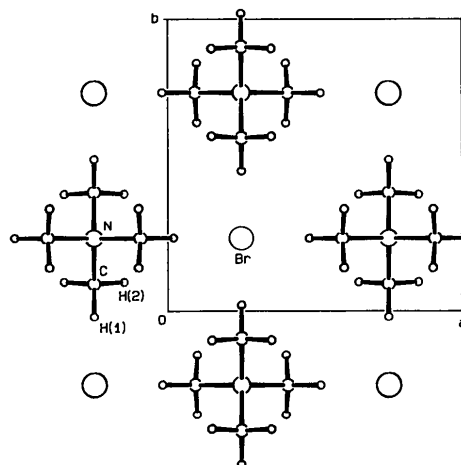

Fig. 1. View of the lattice of $[NMe_4]Br$, down the c axis.

$C_4H_{12}N^+.Br^-$

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
Br	0·2500	0·2500	0·1240 (1)	0·0410 (2)
N	–0·2500	0·2500	0·5000	0·031 (1)
C	–0·2500	0·0926 (6)	0·3435 (6)	0·050 (1)
H(1)	–0·2500	–0·0195 (57)	0·4417 (46)	0·049 (11)
H(2)	–0·1482 (38)	0·0988 (40)	0·2515 (26)	0·061 (7)

Table 2. *Dimensions in the cation; bond lengths (Å) and angles (°): e.s.d's are in parentheses*

N—C	1·487 (4)	C—H(2)	0·93 (2)
C—H(1)	1·02 (4)		

C—N—C′	109·3 (2)	N—C—H(2)	105·7 (19)
C—N—C″	109·5 (2)	H(1)—C—H(2)	109·3 (19)
N—C—H(1)	112·7 (19)	H(2)—C—H(2′)	114·1 (19)

Primed atom names indicate symmetry-related atoms.

plane, which we believe not to be the case). The H atoms, refined independently for both symmetry systems, give more acceptable bond dimensions in the higher-symmetry arrangement. We propose, therefore, that the correct space group for these crystals is *P4/nmm*, the same as previously determined for [NMe₄]⁺ perchlorate and hexafluorophosphate salts.

These structures of [NMe₄]⁺ salts have been described as distortions of the CsCl-type lattice (McCullough, 1964). A simple lattice defined only by the N atoms would be almost cubic; the bromide ions are removed from the sites of simple cubic packing along the unique, c, axis by interactions with the methyl groups of the cation. On one side along the c axis, the anion has close contacts with four H(1) atoms (related by the fourfold symmetry axis)

at a distance of 2·98 (3) Å with the C—H···Br angle of 159 (2)°; these contacts might be described as 'weak hydrogen bonding'. In the opposite direction along c, the anion lies between the three H atoms of a methyl group in each of the four neighbouring cations such that the Br···H distances are 3·48 (3) and 3·58 (3) Å and the N—C···Br angles are 171·1 (2)°. H(2) also contacts a second anion at 3·36 (3) Å. These are interactions and dimensions very similar to those described by Cheban and co-workers; we suspect that their C—H···Br angle of 127° should read 157°, a value close to ours.

The simple lattice of the N atoms described above has dimensions $a' = b' = 5·450$, $c' = 5·498$ Å. Within the *ab* layer, the cations are related to their immediate neighbours by inversion centres, and distances between these adjacent cations have normal van der Waals values, e.g. C···C 3·734 (4), C···H 3·23 (4) and 3·31 (3) and H···H 2·51 (4) and 2·69 (4) Å. Cations in the columns parallel to the c axis are related only by cell translation and contacts between these cations are much weaker, e.g. the closest are C···C 4·149 (7), C···H 3·50 (2), H···H 2·82 (3) Å; the principal interactions along the c axis are those which have been described involving the bromide anion.

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Structure du Diammonium-1,3 Propane *cyclo*-Tétraphosphate Dihydrate, $[NH_3—(CH_2)_3—NH_3]_2P_4O_{12}.2H_2O$

PAR MOHAMED BDIRI ET AMOR JOUINI

Faculté des Sciences et Techniques, Département de Chimie, 5000 Monastir, Tunisie

Abstract. $(C_3H_{12}N_2)_2P_4O_{12}.2H_2O$, $M_r = 504·20$, orthorhombic, *Pbca* (D_{2h}^{15}), $a = 14·627$ (1), $b = 13·391$ (1), $c = 10·068$ (1) Å, $V = 1972·0$ (5) Å³, $Z = 4$, $D_m = 1·633$, $D_x = 1·698$ Mg m⁻³, $\lambda(\text{Ag } K\alpha) = 0·5608$ Å, $\mu = 0·233$ mm⁻¹, $F(000) = 1056$, $T = 298$ K. The structure was solved by the Patterson method and refined to a final R of 0·040 for 2530 observed independent reflections. Centrosymmetric and distorted P_4O_{12} ring anions are located around the origin. They are linked in layers by hydrogen