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The Crystal Structure of n-Dodecylammonium Bromide

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Crystals of n-dodecylammonium bromide ($C_{12}H_{28}NBr$) are monoclinic ($P2_1/c$) with $a=6\cdot030$, $b=6\cdot958$, $c=35\cdot639$ Å and $\beta=92\cdot09°$. The molecules are arranged 'head-to-tail' in single layers. The hydrocarbon chains pack according to the orthorhombic subcell $O'\perp$ with $a_s=6\cdot95$, $b_s=5\cdot40$ and $c_s=2\cdot54$ Å; a_s and b_s differ considerably from earlier reported values. The chain axes are tilted 63·0° to the end-group planes. The bromide ions are roughly octahedrally surrounded by four ammonium and two methyl groups. Three nitrogen–bromine contacts of 3·28, 3·34 and 3·38 Å involve hydrogen bonds. The other distance is 3·62 Å.

Introduction

Long-chain alkylammonium halides exhibit a pronounced surface activity and exist in several polytypic forms. Gordon, Stenhagen & Vand (1953) have given crystal data and phase-transition temperatures for dodecylammonium chloride and bromide. Positional parameters for the atoms were, however, limited to one projection of the bromide. In connexion with our general studies of structure and phase behaviour of lipids, a three-dimensional single-crystal study of dodecylammonium bromide was undertaken.

Experimental

A sample of dodecylammonium bromide (DAB) was kindly provided by Professor E. Stenhagen. Crystals for the X-ray work were obtained from acetone in a temperature-controlled apparatus while the temperature was lowered from 20 to +4°C. The crystals grew as thin plates and most of them were twins. In the hot-stage microscope DAB showed three phase transitions at 56, 62 and 73°C before it melted under decomposition at about 200°C. Gordon, Stenhagen & Vand (1953) give the transition temperatures 57, 61 and 72°C and a melting point of about 200°C.

Crystal data

Structural formula $CH_3(CH_2)_{11}NH_3^+Br^-$, n-dodecylammonium bromide, M.W. 266·26.

Unit cell

Monoclinic $a=6\cdot030$ (3), $b=6\cdot958$ (4),
$c=35\cdot639$ (23) Å,
$\beta=92\cdot09$ (5)°.

$V=1496\cdot29$ Å^3
$Z=4$
$D_c=1\cdot18$ g cm^{-3}
$D_m=1\cdot18$
$\lambda=1\cdot54051$ Å (Cu $K\alpha_1$ radiation)
$\mu=38\cdot75$ cm^{-1}
Systematic absences: $h0l$, $l=2n+1$; $0k0$, $k=2n+1$.
Space group $P2_1/c$.

The intensity data were collected on a Picker FACS-1 automatic diffractometer using graphite monochromated Cu $K\alpha$ radiation. The crystal had the dimensions $0\cdot34\times0\cdot14\times0\cdot02$ mm and was mounted with **a** coincident with the φ axis of the diffractometer. The reflexions were scanned in 6 steps (θ–2θ) of 2 s and a width of 0·40°. The background was determined from measurements on each side of the peak with the crystal and detector stationary. In all, 2634 reflexion intensities were measured with $2\theta<120°$ of which 1640 were considered to be above background ($I\geq2\sigma$). Most of the reflexions with l odd were weak and only 20% of their total number were considered observable.

The intensities were corrected for the Lorentz, polarization and absorption factors but not for extinction.

Structure solution and refinement

The bromine atom coordinates were determined from a sharpened Patterson series. The remaining non-hydrogen atoms were then located from a bromine phased Fourier synthesis. After three cycles of block-diagonal refinement with isotropic temperature factors the R value was 0·15. Another three cycles with anisotropic refinement reduced R to 0·093. The hydrogen atoms of the chain were included in the calculations with their expected positions 1·09 Å from the parent atom, but those of the nitrogen and of the methyl group with coordinates obtained from a difference synthesis. (The hydrogen atoms of the two isoelectronic groups showed, as expected, the normal staggered conformation.) All hydrogen atoms were given isotropic temperature factors corresponding to those of the parent atom. Owing to the relatively large number of parameters the hydrogen atoms and other atoms were refined separately in alternating cycles. The hydrogen temperature factors were not refined. At an R value of 0·074 no further improvement occurred and the refinement was terminated. The shifts were then all less than 0·001 Å for the non-hydrogen atoms and 0·02 Å for the hydrogen atoms.

The form factors used were those given in *Interna-*

Table 1. *Fractional atomic coordinates and hydrogen atom isotropic thermal parameters*

The estimated standard deviations are multiplied by 10^4 for Br, N and C and for hydrogen by 10^3. For the hydrogen atoms the first appended number refers to that of the parent atom.

	x	*y*	*z*	*B*
Br(1)	1·7595 (2)	0·7656 (2)	−0·0140 (0)	
N(1)	1·2732 (16)	0·7168 (14)	0·0231 (3)	
C(1)	1·3065 (23)	0·7656 (17)	0·0634 (4)	
C(2)	1·1165 (24)	0·6949 (18)	0·0872 (4)	
C(3)	1·1468 (23)	0·7583 (18)	0·1279 (4)	
C(4)	0·9572 (24)	0·6883 (18)	0·1509 (4)	
C(5)	0·9698 (25)	0·7582 (19)	0·1920 (4)	
C(6)	0·7882 (24)	0·6877 (22)	0·2157 (4)	
C(7)	0·7980 (24)	0·7622 (18)	0·2558 (4)	
C(8)	0·6094 (23)	0·6887 (20)	0·2789 (4)	
C(9)	0·6220 (22)	0·7603 (19)	0·3196 (4)	
C(10)	0·4349 (22)	0·6926 (21)	0·3431 (4)	
C(11)	0·4419 (27)	0·7667 (20)	0·3831 (5)	
C(12)	0·2540 (27)	0·7015 (25)	0·4068 (5)	
*H(131)	1·128 (13)	0·788 (11)	0·012 (2)	2·4
*H(132)	1·254 (13)	0·560 (12)	0·020 (2)	2·4
*H(133)	1·420 (13)	0·762 (11)	0·009 (2)	2·4
H(11)	1·432 (17)	0·704 (14)	0·074 (3)	4·5
H(12)	1·289 (17)	0·916 (15)	0·068 (3)	4·5
H(21)	0·981 (17)	0·776 (14)	0·075 (3)	4·7
H(22)	1·128 (17)	0·543 (15)	0·081 (3)	4·7
H(31)	1·290 (17)	0·706 (15)	0·142 (3)	4·9
H(32)	1·129 (17)	0·920 (15)	0·127 (3)	4·9
H(41)	0·816 (17)	0·745 (14)	0·141 (3)	4·6
H(42)	0·946 (17)	0·540 (14)	0·151 (3)	4·6
H(51)	1·138 (18)	0·708 (16)	0·203 (3)	5·5
H(52)	0·972 (18)	0·909 (16)	0·191 (3)	5·5
H(61)	0·648 (16)	0·718 (14)	0·201 (3)	4·4
H(62)	0·787 (17)	0·532 (13)	0·212 (3)	4·4
H(71)	0·944 (18)	0·717 (15)	0·270 (3)	5·3
H(72)	0·792 (17)	0·885 (16)	0·255 (3)	5·3
H(81)	0·460 (18)	0·715 (15)	0·266 (3)	5·2
H(82)	0·611 (18)	0·527 (15)	0·283 (3)	5·2
H(91)	0·763 (17)	0·738 (15)	0·334 (3)	5·0
H(92)	0·596 (17)	0·890 (15)	0·320 (3)	5·0
H(101)	0·294 (16)	0·721 (14)	0·330 (3)	4·3
H(102)	0·460 (16)	0·529 (14)	0·347 (3)	4·3
H(111)	0·592 (17)	0·743 (14)	0·395 (3)	4·6
H(112)	0·436 (17)	0·913 (15)	0·381 (3)	4·6
H(121)	0·250 (16)	0·553 (13)	0·411 (3)	4·0
H(122)	0·104 (16)	0·754 (13)	0·394 (3)	4·0
H(123)	0·236 (15)	0·745 (13)	0·432 (3)	4·0

* Refers to the nitrogen atom N(1).

tional Tables for X-ray Crystallography (1962), except for hydrogen, for which the values of Stewart, Davidson & Simpson (1965) were used. The bromine atom scattering values were corrected for the real part of the anomalous dispersion. All calculations were performed on a Datasaab D21–PDP15 dual computer system with programs developed at this research unit. The weight assigned to each observation in the least-squares refinement was (Mills & Rollett, 1961)

$$w=1/\left(1+\left[\frac{|F_o|-2{\cdot}12F_{\min}}{4{\cdot}24F_{\min}}\right]^2\right).^{*}$$

Results

The atomic parameters are given in Tables 1 and 2. The atomic coordinates agree in general with the *x* and *z* projection values reported by Gordon, Stenhagen & Vand (1953). Bond distances and angles, with standard deviations, are shown in Table 3. The atomic numbering is indicated in Fig. 1. Fig. 2 shows the molecule with thermal ellipsoids, viewed down **b**.

The molecule exhibits normal geometry. The average C–C distance in the chain is 1·523 Å $\{\sigma=0{\cdot}006$ Å, where σ is defined by $[\sum_N(X_N-\bar{X})^2/(N-1)]^{1/2}\}$ and the average bond angle 113·8° ($\sigma=0{\cdot}3°$). They compare well with the values found in other long-chain structures, *e.g.* 1·510 Å and 114·6° for β-tricaprin (Jensen & Mabis, 1966), 1·511 Å and 114·2° for DL-2-methyl-7-oxododecanoic acid (O'Connell, 1968), 1·515 Å and 113·3° for isostearic acid (Abrahamsson & Lundén, 1972). The C–N distance is 1·482 Å ($\sigma=0{\cdot}017$ Å) in good agreement with the values of *e.g.* 1·47 Å for the low-temperature form of monoethylamine hydrobromide (Jellinek, 1958) and 1·466 resp. 1·487 Å for trimethylammonium chloride (Lindgren & Olovsson,

Table 2. *Anisotropic thermal parameters*

The temperature factor is $\exp[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2klb^*c^*U_{23}+2lha^*c^*U_{31}+2hka^*b^*U_{12})]$. Standard deviations are given in parentheses. All values have been multiplied by 10^4.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
Br(1)	463 (6)	571 (2)	814 (8)	41 (8)	14 (6)	−21 (7)
N(1)	542 (55)	539 (60)	711 (62)	16 (50)	−25 (46)	15 (47)
C(1)	657 (78)	548 (75)	630 (74)	−88 (68)	−135 (62)	132 (65)
C(2)	769 (87)	573 (70)	539 (71)	−61 (58)	−14 (62)	53 (62)
C(3)	620 (73)	598 (78)	660 (75)	94 (71)	−9 (57)	−27 (64)
C(4)	732 (85)	485 (66)	644 (81)	0 (59)	−4 (67)	−44 (60)
C(5)	714 (86)	706 (89)	776 (87)	−54 (80)	−15 (69)	−21 (70)
C(6)	645 (79)	853 (91)	554 (80)	−1 (73)	−5 (63)	15 (74)
C(7)	636 (78)	549 (80)	878 (87)	47 (77)	4 (64)	−27 (68)
C(8)	689 (86)	619 (75)	668 (84)	−12 (65)	−60 (68)	−32 (65)
C(9)	575 (73)	720 (88)	781 (81)	24 (75)	−43 (61)	247 (67)
C(10)	548 (77)	801 (82)	535 (74)	13 (67)	−80 (60)	18 (66)
C(11)	796 (93)	684 (97)	848 (93)	205 (86)	−21 (73)	40 (77)
C(12)	700 (92)	1110 (118)	825 (101)	275 (95)	254 (77)	−37 (89)

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* A list of the final observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30405 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England, or from the author.

Table 3. *Bond distances and angles*

(*a*) Non-hydrogen atoms. The estimated standard deviations for the distances are multiplied by 10^3.

N(1)—C(1)	1·482 (17) Å	N(1)—C(1)—C(2)	112·7 (1)°
C(1)—C(2)	1·536 (20)	C(1)—C(2)—C(3)	111·8 (1)
C(2)—C(3)	1·528 (19)	C(2)—C(3)—C(4)	111·0 (1)
C(3)—C(4)	1·516 (20)	C(3)—C(4)—C(5)	113·8 (1)
C(4)—C(5)	1·550 (20)	C(4)—C(5)—C(6)	115·1 (1)
C(5)—C(6)	1·494 (21)	C(5)—C(6)—C(7)	114·7 (1)
C(6)—C(7)	1·525 (21)	C(6)—C(7)—C(8)	113·2 (1)
C(7)—C(8)	1·522 (21)	C(7)—C(8)—C(9)	113·3 (1)
C(8)—C(9)	1·542 (20)	C(8)—C(9)—C(10)	114·5 (1)
C(9)—C(10)	1·508 (19)	C(9)—C(10)-C(11)	115·0 (1)
C(10)-C(11)	1·521 (21)	C(10)-C(11)-C(12)	115·6 (1)
C(11)-C(12)	1·514 (23)		

(*b*) Bond distances involving hydrogen atoms. The estimated standard deviations for the distances are multiplied by 10^2.

N(1)-H(131)	1·07 (8) Å	C(6)—H(62)	1·10 (10) Å
N(1)-H(132)	1·10 (8)	C(7)—H(71)	1·06 (11)
N(1)-H(133)	1·08 (8)	C(7)—H(72)	0·86 (12)
C(1)-H(11)	0·94 (10)	C(8)—H(81)	1·01 (11)
C(1)-H(12)	1·07 (11)	C(8)—H(82)	1·13 (11)
C(2)-H(21)	1·06 (10)	C(9)—H(91)	0·99 (11)
C(2)-H(22)	1·08 (11)	C(9)—H(92)	0·92 (11)
C(3)-H(31)	1·06 (11)	C(10)-H(101)	0·98 (10)
C(3)-H(32)	1·13 (11)	C(10)-H(102)	1·16 (10)
C(4)-H(41)	0·99 (10)	C(11)-H(111)	1·00 (11)
C(4)-H(42)	1·04 (11)	C(11)-H(112)	1·02 (11)
C(5)-H(51)	1·13 (11)	C(12)-H(121)	1·05 (10)
C(5)-H(52)	1·05 (12)	C(12)-H(122)	1·06 (10)
C(6)-H(61)	1·01 (10)	C(12)-H(123)	0·96 (10)

1968). The average C–H bond distance is 1·03 Å (σ = 0·02 Å), the average H–C–H angle 108·4° (σ = 2·3°) and the average H–C–C angle 107·0° (σ = 1·3°). The N–H distances are 1·07, 1·10 and 1·08 Å and the mean value of the H–N–H angles is 110·1° (σ = 5·7°).

The molecule is planar within 0·06 Å (Table 4) but the chain axis is somewhat bent in a direction perpendicular to the zigzag plane. Thus the carbon atoms C(2) and C(12) at the ends of the chain are 0·06 Å out of plane on one side and C(6) in the middle of the chain is 0·06 Å on the other side. The nitrogen atom is out of plane by 0·24 Å and the bromine atom 0·70 Å on the opposite side.

Table 4. *Deviations from the least-squares plane through the carbon chain*

The equation is expressed in terms of the crystal axes.

$$-0{\cdot}29302X+0{\cdot}52186Y-0{\cdot}80113Z+0{\cdot}03975=0$$

C(1)	0·062 Å	C(8)	−0·031 Å
C(2)	0·058	C(9)	−0·019
C(3)	−0·033	C(10)	−0·012
C(4)	−0·026	C(11)	0·037
C(5)	−0·028	C(12)	0·059
C(6)	−0·055	*N(1)	0·241
C(7)	−0·013	*Br(1)	−0·703

* Atoms not included in the plane calculation.

The molecular packing is shown in Fig. 3. The molecules are arranged in single molecular layers in a 'head-to-tail' fashion. The chain axes are tilted 63·0° to the (001) plane.

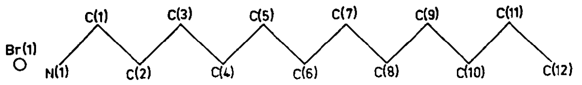

Fig. 1. Atomic numbering for DAB.

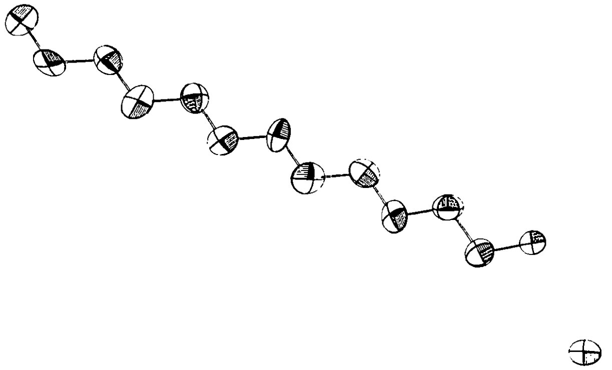

Fig. 2. Drawing of DAB showing the thermal ellipsoids viewed along the *b* axis.

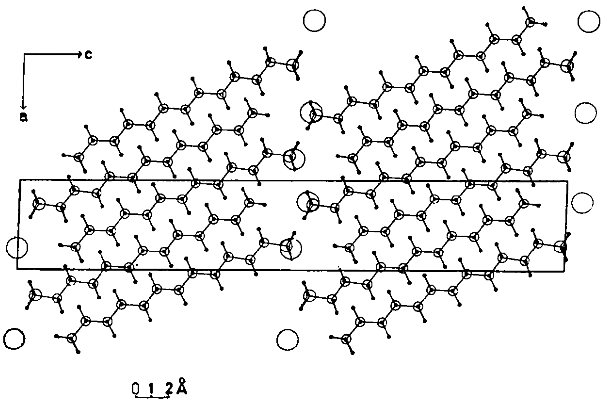

Fig. 3. Molecular packing of DAB viewed along the *b* axis.

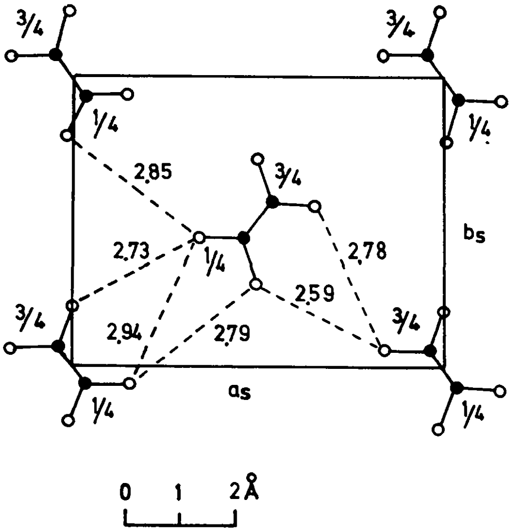

Fig. 4. The $O'\perp$ subcell in DAB viewed along the c_s axis. The hydrogen van der Waals contacts are those found in the middle of the chains.

The subcell of the hydrocarbon chains is orthorhombic with the dimensions $a_s = 6{\cdot}96$, $b_s = 5{\cdot}40$ and $c_s = 2{\cdot}54$ Å (Fig. 4). These values do not correspond with those of any previously known subcell. However, analysis of the subcell positions shows that the packing is similar to the $O'\perp$ arrangement found earlier in only two structures, 2-D-methyloctadecanoic acid (MOA) (Abrahamsson, 1959) and DL-2-methyl-7-oxododecanoic acid (ODA) (O'Connell, 1968). The dimensions (Å) of the subcell are in these cases

	a_s	b_s	c_s
MOA	7·43	5·01	2·50
ODA	7·57	5·06	2·53.

These correspond to cross-sectional areas per chain of 18·6 and 19·2 Å^2 respectively. In DAB the area is very similar, 18·8 Å^2. The angle the zigzag plane makes with the a_sc_s-face is 59° in MOA, 67·5° in ODA and 52·1° in DAB. This means that the chain planes of adjacent chains intersect at 62, 45 and 75·8°. Obviously the $O'\perp$ packing can vary considerably both as to dimensions and chain orientation. Should the a_s axis decrease further and the b_s axis increase the packing would change to the common orthorhombic subcell $O\perp$ (Abrahamsson, Ställberg-Stenhagen & Stenhagen, 1963). As expected the infrared spectrum shows the double (720 and 730 cm^{-1}) CH_2-rocking absorption bands which are characteristic for chains with mutually perpendicular zigzag planes.

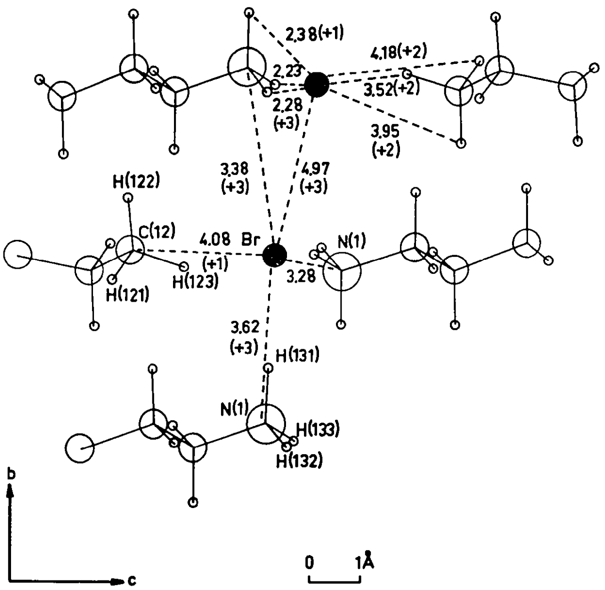

Fig. 5. Some packing contacts in the bromine region of DAB.

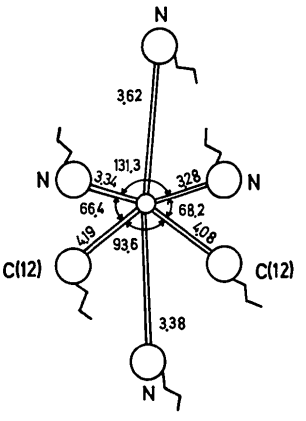

Fig. 6. Atomic arrangement around the bromine atom.

The arrangement in the polar region is given in Table 5 and Fig. 5. The bromine atom is roughly octahedrally surrounded by four NH_3^+-groups and two CH_3-groups (Fig. 6). Two equivalent nitrogen atoms are situated one above and one below the bromine atom in the **b** direction at distances of 3·38 and 3·62 Å, the latter not being involved in hydrogen bonding. The corresponding angle at the bromine atom is 167·8°. The other two nitrogen atoms are at distances of 3·28 and 3·34 Å and take part in the hydrogen-bond system. These four rather normal nitrogen–bromine contact distances could not have been obtained without the observed distortions in the chain subcell. With a normal a_s axis of 7·5 Å the nitrogen–bromine contacts would have been at least 3·75 Å. In the **a** direction an efficient packing could similarly not have been obtained with a b_s edge 0·4 Å shorter. At the remaining corners of the octahedron two methyl end carbon atoms are located but are, of course, at considerably greater distances (4·08 and 4·19 Å) from the bromine atom. Considering these last mentioned nitrogen and carbon atoms the angle N–Br–N is 131·3° and the angle C–Br–C 93·6°. The ammonium hydrogen atoms, H(131), H(132) and H(133), point in the direction of one bromine atom each, with the angles N–H–Br 148·6, 176·0 and 162·1°

Table 5. *Intermolecular contacts in the bromine environment*

i, ii, iii and iv refer to the equivalent positions x,y,z; $\bar{x},\bar{y},\bar{z}$; $\bar{x},\frac{1}{2}+y,\frac{1}{2}-z$; $x,\frac{1}{2}-y,\frac{1}{2}+z$.

A *B*	Unit translation of atom *B*	
$Br(1)\cdots N(1^{i})$	(0, 0, 0)	3·28 Å
$Br(1)\cdots N(1^{i})$	(1, 0, 0)	3·34
$Br(1)\cdots N(1^{ii})$	(3, 1, 0)	3·38
$Br(1)\cdots N(1^{ii})$	(3, 2, 0)	3·62
$Br(1)\cdots C(12^{iv})$	(1, 1, −1)	4·08
$Br(1)\cdots C(12^{iv})$	(2, 1, −1)	4·19
$Br(1)\cdots H(131^{i})$	(0, 0, 0)	3·95
$Br(1)\cdots H(131^{i})$	(1, 0, 0)	2·38
$Br(1)\cdots H(132^{i})$	(0, 0, 0)	3·62
$Br(1)\cdots H(132^{ii})$	(3, 1, 0)	2·28
$Br(1)\cdots H(133^{i})$	(0, 0, 0)	2·23
$Br(1)\cdots H(121^{iii})$	(2, 0, 0)	4·18
$Br(1)\cdots H(121^{iv})$	(1, 1, −1)	4·19
$Br(1)\cdots H(121^{iv})$	(2, 1, −1)	4·25
$Br(1)\cdots H(122^{iv})$	(2, 1, −1)	3·95
$Br(1)\cdots H(123^{iv})$	(1, 1, −1)	3·64
$Br(1)\cdots H(123^{iv})$	(2, 1, −1)	3·52

respectively. The corresponding H···Br contacts are 2·38, 2·28 and 2·23 Å.

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The Crystal and Molecular Structure of Trisodium 6-Phospho-D-gluconate Dihydrate, $Na_3PO_4C_6H_{10}O_6.2H_2O$ and Comparison of Results from Filtered and Monochromatic Radiation

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The crystal and molecular structure of trisodium 6-phosphogluconate dihydrate has been determined by X-ray analysis. The crystals are monoclinic, space group $P2_1$, with $a=11{\cdot}588$ (2), $b=5{\cdot}876$ (1), $c=9{\cdot}859$ (2) Å, $\beta=97{\cdot}57$ (2)° and $Z=2$. The structure was solved from the Patterson synthesis and refined by full-matrix least-squares calculations to $R=3{\cdot}4\%$ with 1878 reflexions. Bond distances and angles in the 6-phosphogluconate ion are normal. The structure is held together by an extensive hydrogen-bond network as well as sodium-ion coordination by oxygen from several 6-phosphogluconate ions. Two complete sets of data were collected out to a $\sin\theta/\lambda$ of 0·703 Å^{-1} on a Picker FACS-1 diffractometer. The first set of data was collected with zirconium-filtered molybdenum radiation while the second set was collected with molybdenum radiation and a graphite monochromator. Normal and half-normal probability plots show that there are no significant differences in either the derived parameters or the structure factors.

Introduction

The pentose phosphate cycle is a source of energy to biological systems as well as a means of providing reduced nicotinamide adenine dinucleotide phosphate. One of the important intermediates in this cycle is 6-phospho-D-gluconate which has a free energy of hydrolysis that is probably comparable to the −3·3 kcal mole^{-1} reported for glucose-6-phosphate (*Handbook for Biochemistry*, 1970). This study was undertaken as a part of our continuing research on the structures of organic phosphates and to provide detailed structural information about this low-energy phosphate.

In addition, we have been interested in comparison of structural results obtained from filtered and monochromatized radiation. Different errors in the two data sets along with uncertainties in the polarization correction for the monochromatized radiation make such a comparison valuable.

Experimental

A clear, colorless crystal of trisodium phosphogluconate dihydrate, recrystallized from a mixture of water and ethanol, was mounted along the *b* axis. Preliminary Weissenberg photographs indicated a monoclinic space group; during the subsequent data collection the sys-