observed in the infrared spectrum (Linder, Lehner & Scheer, 1967). The degree of E—O lengthening is significantly greater in P than in As with respect to the corresponding bond in the free base, demonstrating a more dramatic disruption of the π character of the E—O bond. The B—O bond lengths in the two structures are indistinguishable and are typical of a single B—O bond (Greenwood & Earnshaw, 1984).

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Structure of Bis(cyclohexylammonium) D-Glucose 6-Phosphate Trihydrate

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Abstract. $C_6H_{11}O_9P^{2-}.2C_6H_{14}N^+.3H_2O$, $M_r = 512{\cdot}54$, triclinic, $P1$, $a = 9{\cdot}486\,(7)$, $b = 12{\cdot}696\,(8)$, $c = 11{\cdot}736\,(7)$ Å, $\alpha = 99{\cdot}93\,(5)$, $\beta = 102{\cdot}66\,(5)$, $\gamma = 104{\cdot}90\,(5)^\circ$, $V = 1293\,(2)$ Å^3, $Z = 2$, $D_m = 1{\cdot}30\,(2)$, $D_x = 1{\cdot}317\,(2)$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}173$ mm^{-1}, $F(000) = 556$, $T = 301\,(2)$ K, final $R = 0{\cdot}0442$ for 4760 observed reflections. Two crystallographically independent sugar moieties have different configurations at C(1). The first dianion exists as β-D-glucopyranose. The second is about 65% α-glucopyranose and about 35% β-glucopyranose forms. The orientation of the phosphate group with respect to the sugar system is similar in the two anions. The lengths of the phosphate ester bonds are 1·610 (4) and 1·625 (4) Å.

Introduction. Depending on pH, glucose 6-phosphate may exist in water solutions as free acid, monoanion and dianion. All these forms are difficult to obtain in the crystalline state. The only known crystal structures are of the Ba salt of the dianion (Katti, Seshadri & Viswamitra, 1982; Lis, 1985) and the Na salt of the monoanion (Lis, 1985; Narendra & Viswamitra, 1985). Since our attempts to isolate the crystalline Na and K salts of the dianion failed, it was decided to obtain some other crystalline salts of the D-glucose 6-phosphate dianion with different organic cations. Here the structure of D-glucose 6-phosphate dianion as the cyclohexylammonium salt is reported.

Experimental. Bis(cyclohexylammonium) D-glucose 6-phosphate trihydrate was obtained from the reaction between barium D-glucose 6-phosphate heptahydrate and bis(cyclohexylammonium) sulfate in water. $BaSO_4$ was filtered off and the title compound was grown from water solution as large many-faced colorless crystals. D_m by flotation in a C_6H_5Cl/CH_2Cl_2 mixture. Preliminary examination by rotation and Weissenberg photographs. A specimen $0{\cdot}6 \times 0{\cdot}5 \times 0{\cdot}6$ mm was cut from a large crystal. Syntex $P2_1$ diffractometer, Mo $K\alpha$ radiation for lattice parameters (15 reflections in the range $21 < 2\theta < 26^\circ$) and intensity measurements; θ–2θ scan technique; 5110 reflections measured up to $2\theta = 50^\circ$, $h\ 0\rightarrow 11$, $k\ -15\rightarrow 14$, $l\ -13\rightarrow 13$ (some Friedel opposites measured and not averaged); two standards measured after every 50 reflections, variation $\mp 7\%$. Absorption and extinction ignored. Structure solved

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by direct methods using the *SHELXS*86 program (Sheldrick, 1986). All non-H atoms were located on an *E* map. The C-bonded H atoms were included in geometrically calculated positions [*d*(C—H) = 1·08 Å]. Subsequent electron-density difference maps showed the O- and N-bonded H atoms and the presence of two anomers in the case of one of the two crystallographically independent sugar residues. The percentage of the α and β anomers in this dianion was assumed on the basis of the height of the peaks on the difference map after removing the O atoms bonded to anomeric C atoms. Blocked full-matrix least-squares refinement using *SHELX*76 (Sheldrick, 1976) based on *F* values (P, O, N and C anisotropic; the positional parameters of O- and N-bonded H atoms were refined with constraints $d_{\mathrm{O—H}} = 0{\cdot}96$ and $d_{\mathrm{N—H}} = 1{\cdot}00$ Å; isotropic temperature factors were fixed for all H atoms). Neutral-atom scattering factors including corrections for anomalous dispersion for all non-H atoms taken from *International Tables for X-ray Crystallography*, (1974). $\sum w||F_o| - |F_c||^2$ minimized, $w = 1/\sigma^2(F_o)$. Final $R = 0{\cdot}0442$, $wR = 0{\cdot}0438$ for 4760 reflections with $I > 3\sigma(I)$ and 700 refined parameters, $(\Delta/\sigma)_{\max} = 0{\cdot}19$, minimum and maximum heights on difference Fourier map $-0{\cdot}26$ and $0{\cdot}24$ e Å^{-3}. Final atom parameters are summarized in Table 1.*

Table 1. *Final atomic parameters for bis(cyclohexylammonium)* D-*glucose* 6-*phosphate trihydrate*

$$U_{\mathrm{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}(Å^2)
P(1)	0·725 (fixed)	0·1773 (2)	0·565 (fixed)	0·0346 (5)
P(2)	0·6815 (2)	0·18 (fixed)	0·0622 (2)	0·0298 (5)
C(1)	0·9679 (5)	0·0297 (4)	0·9463 (4)	0·033 (2)
C(2)	1·0969 (5)	−0·0219 (4)	0·9528 (4)	0·034 (2)
C(3)	1·2001 (5)	0·0264 (4)	0·8790 (4)	0·034 (2)
C(4)	1·1052 (5)	0·0079 (4)	0·7505 (4)	0·033 (2)
C(5)	0·9721 (5)	0·0576 (4)	0·7507 (5)	0·034 (2)
C(6)	0·8729 (6)	0·0360 (5)	0·6262 (5)	0·043 (3)
O(1)	0·8693 (4)	−0·0199 (3)	1·0062 (3)	0·043 (2)
O(2)	1·1875 (4)	0·0049 (4)	1·0750 (3)	0·050 (2)
O(3)	1·3149 (4)	−0·0266 (3)	0·8772 (3)	0·045 (2)
O(4)	1·1913 (4)	0·0547 (4)	0·6769 (3)	0·051 (2)
O(5)	0·8821 (4)	0·0042 (3)	0·8210 (3)	0·035 (2)
O(6)	0·7451 (4)	0·0770 (3)	0·6281 (4)	0·049 (2)
O(7)	0·5676 (4)	0·1803 (3)	0·5702 (3)	0·046 (2)
O(8)	0·8499 (5)	0·2821 (4)	0·6381 (4)	0·068 (2)
O(9)	0·7365 (5)	0·1449 (4)	0·4388 (4)	0·072 (3)
C(11)	0·4230 (6)	0·3245 (5)	0·3820 (5)	0·042 (3)
C(21)	0·2805 (6)	0·3624 (4)	0·3508 (4)	0·034 (2)
C(31)	0·1945 (5)	0·3162 (4)	0·2170 (4)	0·031 (2)
C(41)	0·3015 (5)	0·3513 (4)	0·1413 (4)	0·029 (2)
C(51)	0·4372 (5)	0·3061 (4)	0·1778 (4)	0·030 (2)
C(61)	0·5509 (6)	0·3376 (5)	0·1082 (5)	0·038 (2)
O(11*A*)*	0·3877 (6)	0·2167 (5)	0·3763 (5)	0·046 (3)
O(11*B*)*	0·4996 (11)	0·3591 (9)	0·4949 (8)	0·043 (4)
O(21)	0·1846 (4)	0·3301 (3)	0·4250 (4)	0·049 (2)
O(31)	0·0691 (4)	0·3580 (3)	0·1918 (4)	0·043 (2)
O(41)	0·2193 (4)	0·3039 (3)	0·0185 (3)	0·037 (2)
O(51)	0·5147 (4)	0·3571 (3)	0·3036 (3)	0·038 (2)
O(61)	0·6787 (4)	0·2960 (3)	0·1445 (3)	0·037 (2)
O(71)	0.6783 (4)	0·1962 (3)	−0·0620 (3)	0·041 (2)
O(81)	0·8326 (4)	0·1700 (3)	0·1293 (3)	0·037 (2)
O(91)	0·5458 (4)	0·0870 (3)	0·0626 (3)	0·046 (2)
O(*W*1)	0·4620 (4)	0·2179 (4)	0·7662 (4)	0·048 (2)
O(*W*2)	0·3358 (6)	−0·0227 (4)	0·5143 (5)	0·078 (3)
O(*W*3)	−0·0384 (4)	0·1241 (4)	0·3390 (4)	0·050 (2)
O(*W*4)	0·3506 (5)	0·3878 (4)	0·8593 (4)	0·060 (2)
O(*W*5)	0·1416 (5)	0·3905 (4)	0·6571 (4)	0·064 (2)
O(*W*6)	0·1120 (8)	−0·0413 (5)	0·2860 (5)	0·104 (4)
N(6)	0·8658 (6)	0·3748 (4)	0·3937 (5)	0·061 (3)
C(16)	0·9380 (7)	0·4988 (5)	0·4436 (5)	0·054 (3)
C(26)	0·8711 (8)	0·5472 (6)	0·5347 (6)	0·073 (4)
C(36)	0·9504 (10)	0·6721 (6)	0·5830 (7)	0·090 (4)
C(46)	0·9672 (10)	0·7339 (6)	0·4869 (7)	0·093 (4)
C(56)	1·0277 (9)	0·6833 (6)	0·3971 (7)	0·087 (4)
C(66)	0·9485 (9)	0·5585 (5)	0·3447 (6)	0·072 (4)
N(7)	0·5490 (5)	−0·0431 (4)	0·7575 (4)	0·046 (2)
C(17)	0·4815 (7)	−0·1678 (5)	0·7182 (5)	0·053 (3)
C(27)	0·5535 (7)	−0·2140 (5)	0·6274 (6)	0·059 (3)
C(37)	0·4842 (9)	−0·3435 (6)	0·5836 (7)	0·080 (4)
C(47)	0·4889 (9)	−0·3982 (6)	0·6856 (8)	0·087 (4)
C(57)	0.4199 (8)	−0·3487 (5)	0·7792 (7)	0·073 (4)
C(67)	0·4918 (8)	−0·2202 (5)	0·8231 (6)	0·063 (3)
N(8)	0·4810 (6)	0·0123 (4)	0·2522 (5)	0·052 (2)
C(18)	0·4733 (8)	−0·1106 (6)	0·2135 (8)	0·080 (4)
C(28)	0·4016 (10)	−0·1797 (6)	0·2789 (9)	0·103 (5)
C(38)	0·4038 (9)	−0·3000 (6)	0·2453 (8)	0·092 (5)
C(48)	0·5580 (12)	−0·3063 (8)	0·2385 (14)	0·176 (9)
C(58)	0·6330 (11)	−0·2355 (8)	0·1804 (11)	0·135 (7)
C(68)	0·6332 (8)	−0·1118 (6)	0·2097 (8)	0·085 (4)
N(9)	0·9234 (5)	0·3351 (4)	0·8934 (4)	0·060 (2)
C(19)	0·9233 (7)	0·4534 (5)	0·9432 (7)	0·063 (3)
C(29)	0·7785 (8)	0·4698 (6)	0·9111 (7)	0·077 (4)
C(39)	0·7832 (9)	0·5874 (6)	0·9738 (8)	0·087 (4)
C(49)	0·9084 (11)	0·6768 (7)	0·9536 (10)	0·116 (6)
C(59)	1·0517 (9)	0·6588 (6)	0·9780 (9)	0·119 (5)
C(69)	1·0511 (7)	0·5367 (5)	0·9185 (7)	0·066 (3)

*The occupancy factors for O(11*A*) and O(11*B*) are 0·65 and 0·35, respectively.

Discussion. In the unit cell there are two crystallographically independent D-glucose 6-phosphate dianions, four cyclohexylammonium cations and six water molecules. The overall molecular configuration and conformation and the atom-numbering schemes of dianions are shown in Fig. 1. The principal bond lengths and bond angles are given in Table 2. The first dianion exists as β-D-glucopyranose. This form was found in the Ba and Na crystals. The second dianion is about 65% α-glucopyranose and about 35% β-glucopyranose. In this anion, the anomeric forms differ essentially in the positions of the OH groups and C-bonded H atoms at atom C(11) only (the positions of other atoms do not differ in the crystal). The sugar rings have the $^4C_1$ conformation, with little distortion from the ideal, having Cremer–Pople (1975) puckering parameters of $Q = 0{\cdot}607\,(5)$ Å [0·606 (6) Å], $\theta = 3{\cdot}4\,(5)^\circ$ [4·5 (5)°], $\varphi = 10\,(9)^\circ$ [281 (6)°] for rings containing the O(5) [O(51)] atoms. Such a conformation is normally found in D-glucose crystals. The conformation about the exocyclic bond C(6)—C(5) [C(61)—C(51)] is similar to that found in the Na salt; the ester O(6) [O(61)] atom is *trans* to C(4) [C(41)] and *gauche* to O(5) [O(51)]. The lengths of the two endocyclic C—O bonds C(1)—O(5) and C(5)—O(5) [C(11)—O(51) and C(51)—O(51)] are almost the same. The C(11)—O(11*A*) and C(11)—O(11*B*) bond lengths

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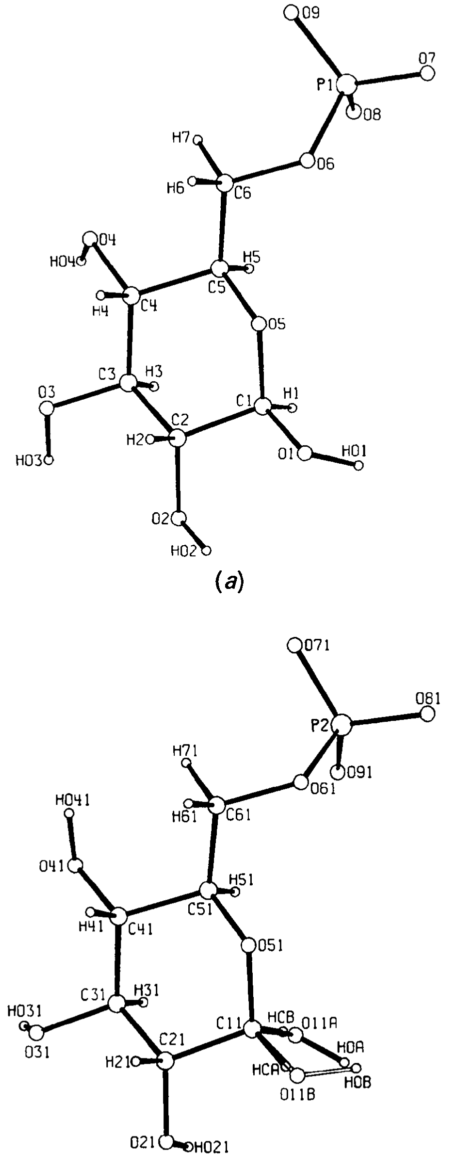
* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and principal bond lengths and angles of the cyclohexylammonium cations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52188 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

are unrealistically short. This is probably because of partial disorder of the C(11) atom as a result of the slightly different positions of this atom in the α and β anomers. All other bond lengths and bond angles in the sugar moieties are similar to those found in the Na and Ba salts.



(a)

(b)

Fig. 1. (a), (b). The molecular configurations and the atom-numbering schemes of D-glucose 6-phosphate dianions.

Table 2. *Principal bond lengths* (Å), *bond angles* (°) *and torsion angles* (°) *in* D-*glucose* 6-*phosphate dianions*

C(1)—C(2)	1·523 (7)	C(11)—C(21)	1·540 (7)
C(1)—O(1)	1·379 (6)	[C(11)—O(11*A*)	1·309 (8)]
		[C(11)—O(11*B*)	1·300 (10)]
C(2)—C(3)	1·527 (7)	C(21)—C(31)	1·533 (6)
C(2)—O(2)	1·433 (6)	C(21)—O(21)	1·426 (6)
C(3)—C(4)	1·521 (6)	C(31)—C(41)	1·522 (7)
C(3)—O(3)	1·422 (6)	C(31)—O(31)	1·417 (6)
C(4)—C(5)	1·550 (7)	C(41)—C(51)	1·547 (7)
C(4)—O(4)	1·415 (6)	C(41)—O(41)	1·419 (6)
C(5)—O(5)	1·440 (6)	C(51)—O(51)	1·445 (6)
O(5)—C(1)	1·451 (6)	O(51)—C(11)	1·439 (6)
C(5)—C(6)	1·491 (6)	C(51)—C(61)	1·506 (7)
C(6)—O(6)	1·441 (6)	C(61)—O(61)	1·453 (6)
O(6)—P(1)	1·610 (4)	O(61)—P(2)	1·625 (3)
O(7)—P(1)	1·518 (4)	O(71)—P(2)	1·502 (4)
O(8)—P(1)	1·503 (4)	O(81)—P(2)	1·525 (3)
O(9)—P(1)	1·503 (3)	O(91)—P(2)	1·508 (4)
O(5)—C(1)—O(1)	106·7 (4)	O(51)—C(11)—O(11*A*)	112·5 (5)
		O(51)—C(11)—O(11*B*)	112·4 (6)
O(5)—C(1)—C(2)	108·1 (4)	O(51)—C(11)—C(21)	108·5 (4)
O(1)—C(1)—C(2)	110·2 (4)	O(11*A*)—C(11)—C(21)	111·6 (5)
		O(11*B*)—C(11)—C(21)	114·3 (6)
C(1)—C(2)—C(3)	110·4 (4)	C(11)—C(21)—C(31)	111·2 (4)
C(1)—C(2)—O(2)	109·7 (4)	C(11)—C(21)—O(21)	110·5 (4)
O(2)—C(2)—C(3)	107·5 (4)	O(21)—C(21)—C(31)	111·1 (4)
C(2)—C(3)—C(4)	109·1 (4)	C(21)—C(31)—C(41)	109·4 (4)
C(2)—C(3)—O(3)	111·1 (4)	C(21)—C(31)—O(31)	109·0 (4)
O(3)—C(3)—C(4)	108·9 (4)	O(31)—C(31)—C(41)	111·0 (4)
C(3)—C(4)—C(5)	109·9 (4)	C(31)—C(41)—C(51)	107·5 (4)
C(3)—C(4)—O(4)	112·8 (4)	C(31)—C(41)—O(41)	107·6 (4)
O(4)—C(4)—C(5)	109·8 (4)	O(41)—C(41)—C(51)	111·6 (4)
C(4)—C(5)—C(6)	111·1 (4)	C(41)—C(51)—C(61)	111·5 (4)
C(4)—C(5)—O(5)	108·5 (4)	C(41)—C(51)—O(51)	108·6 (4)
C(5)—O(5)—C(1)	111·5 (4)	C(51)—O(51)—C(11)	112·9 (4)
C(5)—C(6)—O(6)	110·6 (4)	C(51)—C(61)—O(61)	111·0 (4)
O(5)—C(5)—C(6)	108·3 (4)	O(51)—C(51)—C(61)	107·0 (4)
C(6)—O(6)—P(1)	120·1 (4)	C(61)—O(61)—P(2)	118·7 (3)
O(6)—P(1)—O(7)	102·5 (2)	O(61)—P(2)—O(71)	108·0 (2)
O(6)—P(1)—O(8)	107·3 (3)	O(61)—P(2)—O(81)	101·3 (2)
O(6)—P(1)—O(9)	107·5 (3)	O(61)—P(2)—O(91)	107·9 (2)
O(7)—P(1)—O(8)	113·8 (3)	O(71)—P(2)—O(81)	112·9 (2)
O(7)—P(1)—O(9)	113·2 (3)	O(71)—P(2)—O(91)	112·3 (3)
O(8)—P(1)—O(9)	111·7 (3)	O(81)—P(2)—O(91)	113·5 (2)
O(7)—P(1)—O(6)—C(6)	171·2 (9)	O(71)—P(2)—O(61)—C(61)	59·7 (7)
O(8)—P(1)—O(6)—C(6)	−68·6 (10)	O(81)—P(2)—O(61)—C(61)	178·6 (8)
O(9)—P(1)—O(6)—C(5)	51·6 (9)	O(91)—P(2)—O(61)—C(61)	−61·9 (8)
P(1)—O(6)—C(6)—C(5)	116·1 (11)	P(2)—O(61)—C(61)—C(51)	98·7 (8)
O(6)—C(6)—C(5)—O(5)	57·7 (11)	O(61)—C(61)—C(51)—O(51)	60·5 (8)
O(6)—C(6)—C(5)—C(4)	176·8 (11)	O(61)—C(61)—C(51)—C(41)	179·2 (8)

The P(1)—O(6) [P(2)—O(61)] ester bond of 1·610 (4) Å [1·625 (4) Å] agrees with the mean value of the phosphate ester bond, 1·624 Å, calculated from data for 34 doubly ionized phosphate groups (Starynowicz, Lis & Weichsel, 1986). The deviation of the phosphate groups from tetrahedral symmetry is visible in the O—P—O bond angles, which range from 101·3 (2) to 113·8 (3)°. This differentiation is of the same type as in all monoesterified diionized phosphates (Lis, 1987). For example, the O atoms {O(7) [O(81)]} located *trans* to C(6) [C(61)] are involved in the smallest O—P—O bond angles. The conformations about the C(6)—O(6) and C(61)—O(61) bonds differ from those found previously in Na and Ba salts [the torsion angles P(1)—O(6)—C(6)—C(5) of 116·1 (11) and P(2)—O(61)—C(61)—C(51) of 98·7 (8)° are not identical].

The geometry of the four crystallographically independent cyclohexylammonium cations is normal and similar to that found in other salts of this cation (Weichsel & Lis, 1989). All four cyclohexyl rings have the chair conformation.

The O- and N-bonded H atoms are involved in hydrogen bonds. The data for these bonds are given in Table 3 and are also indicated on Fig. 2 showing the packing in the crystal. It should be noted that, in

Table 3. *Probable hydrogen bonds in bis(cyclohexylammonium)* D-*glucose 6-phosphate trihydrate crystals*

X—H···O	Translation *a b c*	*X*···O (Å)	H···O (Å)	*X*—*H*···O (°)
O(1)—H(O1)···O(81)	0 0 1	2·733 (5)	1·81 (6)	160 (6)
O(2)—H(O2)···O(*W*6)	1 0 1	2·837 (7)	2·04 (6)	139 (5)
O(3)—H(O3)···O(91)	1 0 1	2·611 (5)	1·74 (5)	150 (5)
O(4)—H(O4)···O(*W*1)	1 0 0	2·706 (5)	1·77 (5)	163 (5)
O(11*A*)—H(O*A*)···O(7)	0 0 0	2·715 (7)	1·77 (8)	169 (8)
O(11*B*)—H(O*B*)···O(7)	0 0 0	2·747 (11)	1·93 (17)	140 (15)
O(21)—H(O21)···O(*W*3)	0 0 0	2·764 (5)	1·92 (5)	146 (5)
O(31)—H(O31)···O(81)	$\bar{1}$ 0 0	2·681 (5)	1·83 (5)	146 (5)
O(41)—H(O41)···O(*W*4)	0 0 $\bar{1}$	2·683 (6)	1·75 (6)	165 (6)
O(*W*1)—H(*W*1)···O(7)	0 0 0	2·720 (5)	1·77 (5)	172 (5)
O(*W*1)—H(*W*11)···O(71)	0 0 1	2·650 (5)	1·69 (5)	174 (5)
O(*W*2)—H(*W*2)···O(7)	0 0 0	2·789 (6)	1·90 (5)	152 (5)
O(*W*2)—H(*W*21)···O(4)	$\bar{1}$ 0 0	2·771 (6)	2·01 (6)	135 (5)
O(*W*3)—H(*W*3)···O(81)	$\bar{1}$ 0 0	2·727 (5)	1·79 (5)	163 (5)
O(*W*3)—H(*W*31)···O(9)	$\bar{1}$ 0 0	2·698 (6)	1·81 (5)	153 (5)
O(*W*4)—H(*W*4)···O(*W*5)	0 0 0	2·750 (5)	1·83 (5)	160 (5)
O(*W*4)—H(*W*41)···O(*W*1)	0 0 0	2·798 (6)	1·84 (6)	173 (5)
O(*W*5)—H(*W*5)···O(8)	$\bar{1}$ 0 0	2·696 (6)	1·87 (5)	143 (4)
O(*W*5)—H(*W*51)···O(21)	0 0 0	2·846 (5)	1·98 (5)	150 (5)
O(*W*6)—H(*W*6)···*W*(2)	0 0 0	2·962 (8)	2·62 (5)	101 (4)
O(*W*6)—H(*W*61)···*W*(3)	0 0 0	2·883 (8)	2·68 (6)	92 (4)
N(6)—H(N61)···O(8)	0 0 0	3·301 (7)	2·31 (4)	174 (4)
N(6)—H(N61)···O(9)	0 0 0	3·046 (6)	2·40 (5)	122 (4)
N(6)—H(N62)···O(21)	1 0 0	3·173 (7)	2·44 (5)	130 (4)
N(6)—H(N62)···O(31)	1 0 0	3·379 (7)	2·54 (5)	141 (4)
N(6)—H(N63)···O(51)	0 0 0	3·202 (7)	2·31 (3)	149 (4)
N(6)—H(N63)···O(61)	0 0 0	2·917 (6)	2·16 (4)	131 (4)
N(7)—H(N71)···O(6)	0 0 0	2·944 (6)	2·18 (5)	132 (4)
N(7)—H(N71)···*W*(2)	0 0 0	3·209 (6)	2·60 (5)	119 (4)
N(7)—H(N72)···O(5)	0 0 0	2·956 (6)	2·05 (3)	150 (4)
N(7)—H(N73)···O(3)	$\bar{1}$ 0 0	2·908 (6)	2·00 (5)	149 (4)
N(7)—H(N73)···O(71)	0 0 1	3·154 (5)	2·69 (5)	108 (3)
N(8)—H(N81)···O(91)	0 0 0	2·694 (6)	1·72 (5)	162 (5)
N(8)—H(N82)···O(2)	$\bar{1}$ 0 $\bar{1}$	3·057 (6)	2·57 (5)	110 (4)
N(8)—H(N82)···O(11*A*)	0 0 0	3·175 (8)	2·42 (5)	132 (4)
N(8)—H(N83)···O(9)	0 0 0	2·794 (6)	1·85 (4)	155 (4)
N(9)—H(N91)···O(8)	0 0 0	2·840 (6)	1·85 (5)	173 (4)
N(9)—H(N92)···O(71)	0 0 1	2·745 (6)	1·77 (4)	164 (4)
N(9)—H(N93)···O(41)	1 0 1	3·026 (6)	2·04 (4)	169 (4)

contrast to the Ba salt, there are no intramolecular H bonds between phosphate O atoms and sugar hydroxyl groups. Also interesting is the fact that the O(11*A*)H and O(11*B*)H hydroxyl groups are involved in hydrogen bonds to the same O(7) atom.

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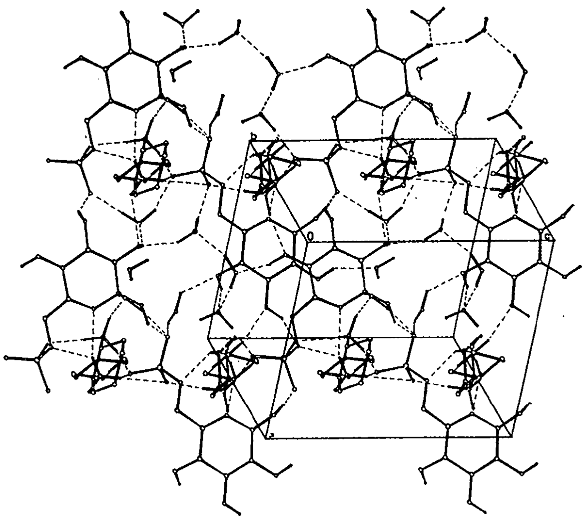

Fig. 2. The packing arrangement. Dashed lines show hydrogen bonds. (C-bonded H atoms are omitted for clarity.)

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Structure of a Novel Hexaaza Macrocyclic Ligand of Schiff-Base Type

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Abstract. 3,7,11,18,22,26-Hexaazapentacyclo[26.2.2.2^{13,16}.0^{2,7}.0^{17,22}]tetratriaconta-1(31),11,13(34),14,16(33),26,28(32),29-octaene, $C_{28}H_{38}N_6$, M_r = 458·6, orthorhombic, *Fdd*2, a = 16·328 (1), b = 56·632 (3), c = 5·426 (1) Å, V = 5017 (1) Å^3, Z = 8, D_x = 1·214 g cm^{-3}, λ = 1·54178 Å, μ(Cu $K\alpha$) =

0108-2701/90/010098-04$03.00