The Crystal Structure of Ba-Ribose-5-phosphate

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The structure of the barium salt of ribose-5-phosphate has been determined by X-ray crystallographic methods with moderate accuracy. The five-membered ring is non-planar with atom C_2 out of the plane of the other atoms. The configuration at the anomeric carbon atom appears to be of the α-type. Each barium ion is surrounded by twelve oxygen atoms, three of which are the hydroxyl oxygens of the same molecule.

Continuing our investigations of simple compounds related to the nucleic acids, we have determined the crystal structure of the barium salt of ribose-5-phosphate. This substance was first prepared by Levene and Jacobs[1] by hydrolyzing inosinic acid. They also determined its structure except for the configuration at the anomeric carbon atom which still appears to be unknown. The mutarotation curves are complex[2] and permit no safe conclusions about the structure to be drawn. However, it seems to be generally assumed that the compound is of the β-type, as in the nucleotides, from which it is isolated. In view of its relation to nucleic acid structure it would also appear to be of interest to determine the conformation of the ribose in this compound. In earlier studies of cytidine[3] and cytidylic acid[4] we have observed two different conformations of ribose.

EXPERIMENTAL. CRYSTAL DATA

A sample of the compound was supplied by Fluka AG. Crystals were grown by slowly adding alcohol to an aqueous solution, the solution being placed on a watch-glass in a desiccator containing alcohol. From a cluster of crystals a cube with edges approximately 0.1 mm was cut out and used for the investigation. Weissenberg diagrams were taken about the b and c axes using copper radiation ($\lambda = 1.542$ Å). The crystals are monoclinic, with unit cell dimensions $a = 15.38$ Å, $b = 8.56$ Å, $c = 12.86$ Å and $\beta = 118.6°$ (all ± 0.5 %). All reflections with $h + k$ odd are absent and the lattice is therefore C-centered. As the molecules are optically active, the space group must be $C2$. By flotation in mixtures of bromoform and carbon tetrachloride the density was found to be 2.05 g/cm³, corresponding to four (calculated 4.03) molecules $C_5H_9O_5 \cdot PO_3 \cdot Ba \cdot 5H_2O$ per unit cell.

205 $h0l$ reflections and 52 $hk0$ reflections were recorded with measurable intensity on the films, the numbers obtainable by copper radiation being 220 and 80, respectively. The intensities were estimated visually and corrected for Lorentz-polarization effects. No correction for absorption was made.
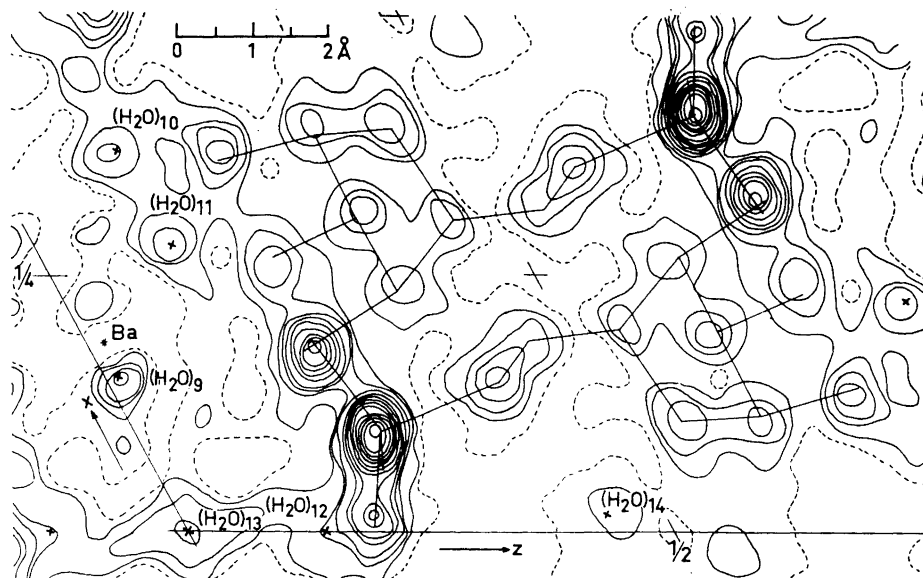
Fig. 1. Electron density projection in direction of *b* axis. Contours at 3, 5, 7 . . .e.Å^{-2}, the first one being dotted. The barium ion is subtracted. Crosses indicate water molecules.

Determination of the structure

The b projection. The barium atom was located by means of the Patterson projection, and a Fourier synthesis based on the corresponding signs indicated the positions of most of the lighter atoms. These were more clearly shown on the difference map reproduced in Fig. 1, which was calculated by subtracting the contribution from the barium atom. The structure was further refined by means of a number of difference syntheses. It was, however, found difficult to locate some of the water molecules unambigously from the maps. Three of the sites are fairly clearly shown, namely those of O_9, O_{10} and O_{11}. In addition there appears to lie a water molecule (O_{13}) on the two-fold axis through origo. The position derived for O_{12} is less certain, as equally good agreement was obtained by substituting it by two half-oxygens with coordinates (0.027, 0.125) and (0.440, 0.140). Finally the water molecule O_{14} lies so near a two-fold axis that the distance to the equivalent atom is only 2.1 Å. This is clearly not possible, and we assume therefore that the water molecules are statistically distributed over these two sites. Consequently, the calculations were based on half an oxygen atom in this position, giving in all five water molecules per barium ion.

The positions finally selected for the atoms are given in Fig. 1 and Table 1. The corresponding values of $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$ is 0.09, including the extinction error occurring in the two strongest reflections (001) and (200). In Table 4 the observed (F_o) and calculated (F_c) values of the structure factors are compared. The analytical approximation to the atomic scattering factors given by Forsyth and Wells [5] was used. The vibrations of the Ba ion are strongly anisotropic. In the temperature factor, exp.$(B \sin^2\Theta/\lambda^2)$, the value of B varies between 1.1 and 2.5 for the lighter atoms (Table 1), whereas for the Ba ion $B = 0.8 + 1.0 \cos^2\varphi$, φ being the angle between the *a* axis and the normal to the reflecting plane. The water molecules have B-values in the range 3.5—6.0.

The c projection. The coordinates of the barium and phosphorus atoms were derived from the sharpened Patterson synthesis (Fig. 2 b), which showed that their *y* coordinates

Table 1. Atomic coordinates as fractions of the corresponding cell edge.

	x	y	z	B_{tot}
Ba	0.1862	0.000	0.0262	0.8—1.8
P	0.098	0.495	0.251	1.1
O_1	0.137	0.425	0.765	1.8
O_2	0.231	0.678	0.757	2.0
O_3	0.325	0.395	0.772	2.2
O_4	0.105	0.349	0.570	2.5
O_5	0.145	0.490	0.398	2.0
O_6	0.186	0.450	0.234	2.0
O_7	0.007	0.392	0.197	2.0
O_8	0.060	0.660	0.219	2.0
O_9	0.144	0.307	0.012	3.0
O_{10}	0.365	0.267	0.130	4.5
O_{11}	0.274	0.622	0.139	4.5
O_{12}	0.000	0.093	0.140	6.0
O_{13}	0.000	0.810	0.000	6.0
O_{14}	0.022	0.710	0.438	6.0
C_1	0.112	0.480	0.649	2.2
C_2	0.192	0.578	0.651	2.2
C_3	0.268	0.477	0.653	2.5
C_4	0.199	0.341	0.571	2.2
C_5	0.184	0.341	0.451	2.5

differ by about $b/2$. The y coordinates of the other atoms were derived from the b projection and approximately known bond lengths and bond angles, considering also the Patterson synthesis. They were refined by a number of difference syntheses. The final value obtained for R was 0.12. However, the coordinates derived from this projection are inaccurate because of lack of centre of symmetry and overlap of atoms. The value used for B was
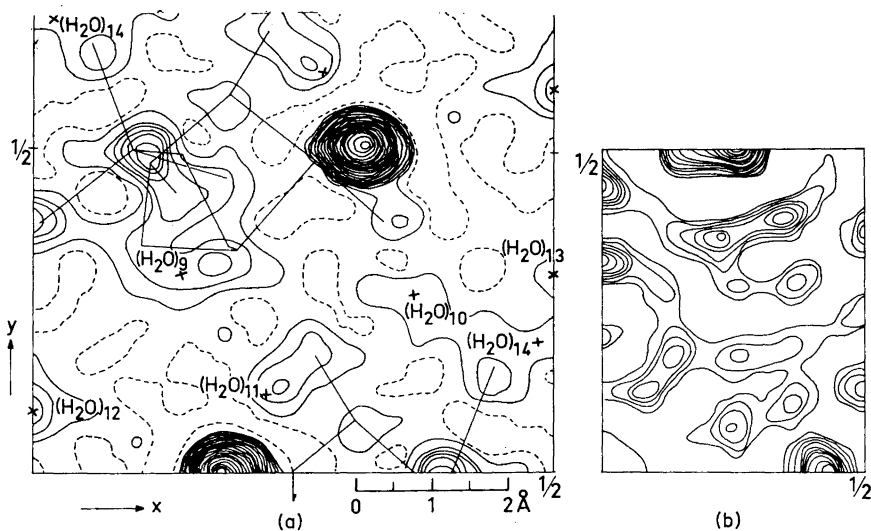

Fig. 2. Electron density (a) and Patterson (b) projections in direction of c axis. Contours as in Fig. 1 in (a), in (b) at arbitrary intervals.

Table 2. Bond lengths (in Å units) and bond angles.

Bond lengths		Bond angles	
$P-O_5$	1.67	O_6-P-O_7	118°
$P-O_6$	1.52	O_6-P-O_8	117°
$P-O_7$	1.51	O_6-P-O_5	103°
$P-O_8$	1.51	O_7-P-O_8	106°
O_5-C_5	1.44	O_7-P-O_5	108°
C_5-C_4	1.45	O_8-P-O_5	104°
C_4-C_3	1.59	$P-O_5-C_5$	115°
C_3-C_2	1.44	$O_5-C_5-C_4$	109°
C_2-C_1	1.48	$C_5-C_4-C_3$	115°
C_1-O_4	1.48	$C_4-C_3-C_2$	99°
O_4-C_4	1.45	$C_3-C_2-C_1$	109°
C_1-O_1	1.43	$C_2-C_1-O_4$	103°
C_2-O_2	1.47	$C_1-O_4-C_4$	107°
C_3-O_3	1.49	$O_4-C_4-C_5$	111°
		$C_4-C_3-O_3$	104°
		$C_2-C_3-O_3$	110°
		$C_3-C_2-O_2$	111°
		$C_1-C_2-O_2$	108°
		$C_2-C_1-O_1$	111°
		$O_4-C_1-O_1$	111°
		$O_4-C_4-C_3$	109°

1.7 for the barium and phosphorus atoms and 2.0—4.0 for the other atoms. The electron density map is shown in Fig. 2 a.

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The molecular structure. The bond lengths and bond angles calculated from the coordinates in Table 1 are given in Table 2. Lack of resolution and the presence of a very heavy atom make it difficult to determine the structure accurately by two-dimensional methods and we believe that errors as great as about 0.10 Å and 5° may occur. Within these wide limits, the values derived agree with those found in similar compounds. The P—O ester linkage ($P-O_5$) is thus significantly longer than the other P—O distances, as was found also in cytidylic acid [4] and in calcium thymidylate [7].

If our interpretation of the Fourier maps is correct, the hydroxyl groups at C_1, C_2 and C_3 all are in *cis* relation, which means that the compound is
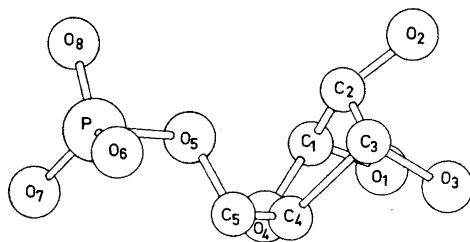

Fig. 3. The molecular structure.

the α-anomer. According to Fluka AG., our sample has the same optical configuration as the natural material. We have tried to accomodate a β-structure on the Fourier- and difference-maps. Although this possibility cannot definitely be excluded due to uncertainty in the positions of some of the water molecules, the α-structure fits the maps much better. We therefore consider it highly probable that this anomer occurs in the crystals. As described below, a complex involving three-point attachment of the Ba ion is present in the crystals. Only α-ribose can form the complex, which thus may favour the crystallization of this anomer.

The phosphate group is pointing away from the ribose ring, the $P-O_8$ bond being nearly parallel to C_5-O_5 and $P-O_5$ to C_4-C_5, as shown in Fig. 3. This makes the molecule as extended as possible. The bond C_5-C_4 is in *gauche* position with respect to the ring bonds to C_4, and there is a staggered arrangement around all the single bonds. The same structural features were found in Ca thymidylate, the only other 5-phosphate hitherto investigated, and are also in exact agreement with space models proposed for nucleic acids [10].

As found in other furanose structures, the five-membered ring is non-planar. Four of the atoms, namely C_1, C_3, C_4 and O_4 lie nearly in one plane, but C_2 is out of this plane by about 0.5 Å in such a direction that O_2 falls nearly on to the plane. The ring angles at carbon atoms C_3 and C_1 are probably significantly smaller than the tetrahedral angle, and the conformation appears to differ from those observed in the ribose derivatives mentioned above [3,4,7].

The water molecules. After recrystallization from water the density corresponds to five molecules of water per molecule, with an accuracy of about half a water molecule. Levene and Jacobs [1] found the number to be five or five and a half. Some of the water of crystallization is, however, easily lost and the sample supplied by Fluka AG. is stated to contain only about three water molecules per barium atom. In the final structure there are, as described above, five water molecules. The electron density maxima corresponding to some of the water sites are considerably lower than found for the other oxygen atoms, and it seems possible that some of the water molecules are lost during the exposure time. We have not tried to take this into account as the data are not accurate enough to disclose such details in the presence of the heavy ion, but have assigned to these atoms anomalously large values of B. This may be interpreted either as thermal motions or as localized disorder, the coordinates given for the water molecules in Table 1 representing the average value of a certain distribution of parameters. This holds particularly for the water molecule at O_{14}. Similar problems as regards the water of crystallization have been encountered in a number of structures, *e.g.* in thymine monohydrate [11].

The ionic environment. In the structure there are corrugated sheets of hydrated barium ions at distances of about 11 Å. The radius of the barium ion is about the same as that of an oxygen atom, and it is recognized that in such cases the most stable anion polyhedron corresponds to coordination numbers in the range nine to twelve. With twelve neighbours the icosahedral configuration appears to be preferred as it is found in a number of barium salts, *e.g.* barium perchlorate trihydrate [8], in which there are twelve Ba to O contacts between 2.96 Å and 3.18 Å. The usual arrangement for coordination number nine arises by adding atoms beyond the centres of the vertical faces
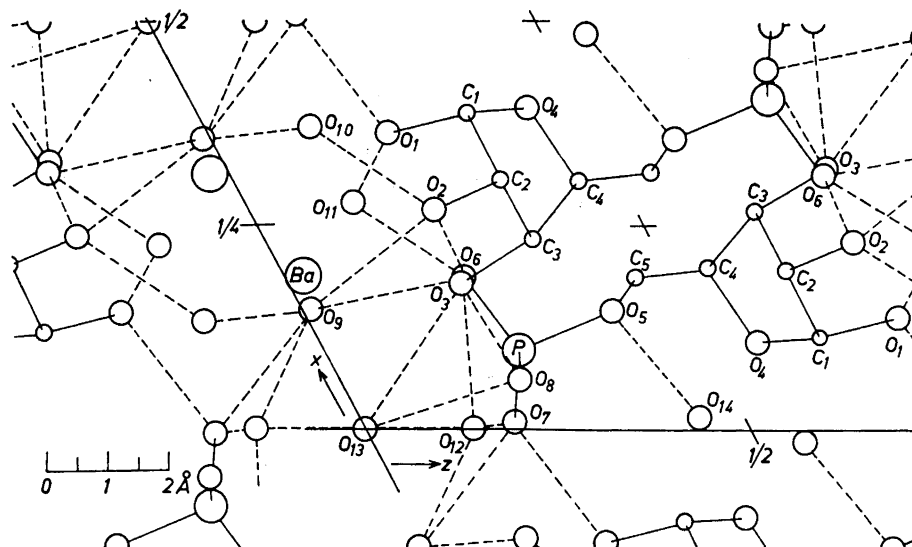
Fig. 4. The *b* projection of the structure. Short O—O contacts are indicated by dotted lines.

of a right triangular prism. An example of a barium salt with an anion poly-hedron of this type is provided by barium tetrathionate dihydrate [5], in which the barium ion has nine neighbours at distances 2.73 Å — 2.90 Å. In the pre-sent structure there are seven oxygen neighbours with Ba—O separations 2.67 Å—2.88 Å, and in addition five oxygens further apart (3.14 Å—3.80 Å). These twelve neighbours are situated in the corners of a distorted icosahedron. One out of the twenty triangles is common to the anion polyhedra of neigh-bouring barium ions.

The nature of the linkage between divalent metal ions and ATP, nucleo-tides and sugar phosphates has been much discussed due to the biological importance of these complexes [9]. Several different modes of attachment have been proposed. The study of the crystal structures of these compounds may contribute to the solution of the problem. In the present structure the barium ion is bonded directly to the hydroxyl groups of α-ribose in a threedentate complex with Ba—O distances 2.84—2.88 Å. It is not linked directly to the phosphate oxygens, but is connected to these three atoms by bridges of the

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type Ba...O—H...O—P⟨. In crystals of Ca thymidylate the ion is bonded

directly to the phosphate oxygens. In this case, however, the sugar has only one free hydroxyl group.

The hydrogen bonds. The water molecules, the phosphate oxygens and the sugar hydroxyls are all involved in hydrogen bond formation. The three hyd-roxyl oxygens of α-ribose form a nearly equilateral triangle with edges of
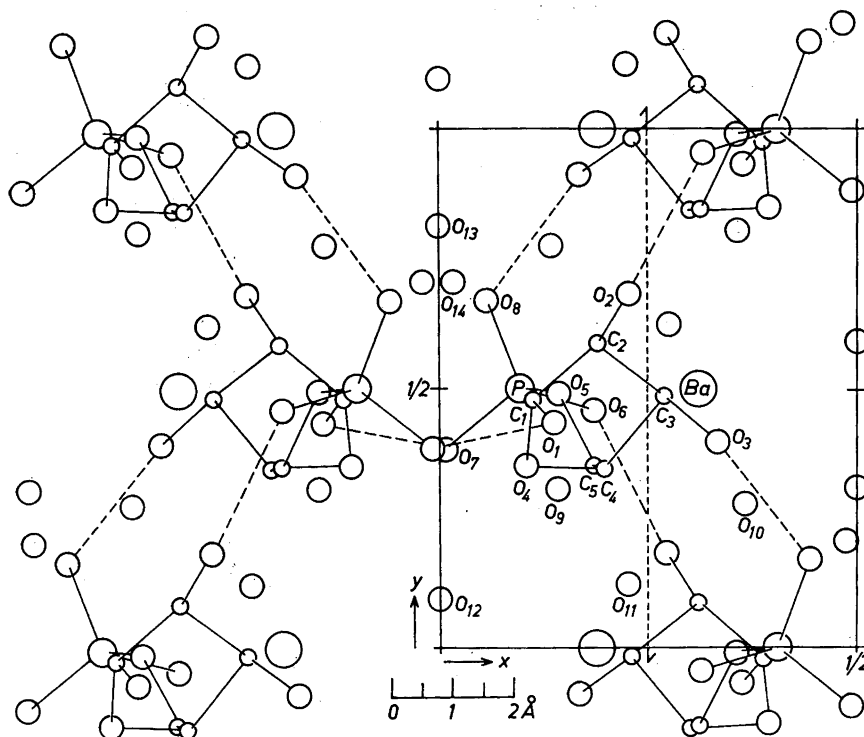
Fig. 5. The *c* projection of the structure. The broken lines show the hydrogen bonds between hydroxyl and phosphate oxygens.

Table 3. Short O—O and Ba—O distances (in Å units).

$O_7 - O_1$	2.50	$Ba - O_1$	2.84
$O_6 - O_2$	2.63	$Ba - O_2$	2.88
$O_8 - O_3$	2.64	$Ba - O_3$	2.86
$O_9 - O_6$	2.88	$Ba - O_9$	2.69
$O_9 - O_2$	2.80	$Ba - O_{10}$	2.67
$O_9 - O_{12}$	2.82	$Ba - O_{11}$	2.69
$O_9 - O_7$	2.68	$Ba - O_{12}$	2.74
$O_9 - O_{10}$	3.01	$Ba - O_{13}$	3.14
$O_{11} - O_1$	2.92	$Ba - O_9$	3.32
$O_{11} - O_6$	2.66	$Ba - O_{10}$	3.33
$O_{10} - O_2$	2.63	$Ba - O_{11}$	3.54
$O_{12} - O_3$	2.90	$Ba - O_{12}$	3.80
$O_{12} - O_7$	2.65		
$O_{12} - O_{13}$	3.02		
$O_{13} - O_3$	2.84		
$O_{13} - O_8$	2.79		
$O_{14} - O_5$	2.80		

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Table 4. Observed (F_o) and calculated (F_c) structure factors.

h k l	F_o	F_c	h k l	F_o	F_c	h k l	F_o	F_c
0 0 1	37	48	4 0 $\bar{2}$	10	−11	8 0 $\bar{6}$	12	− 9
0 0 2	15	15	4 0 $\bar{3}$	41	−44	8 0 $\bar{7}$	< 2	3
0 0 3	27	29	4 0 $\bar{4}$	36	−38	8 0 $\bar{8}$	6	4
0 0 4	51	51	4 0 $\bar{5}$	36	−40	8 0 $\bar{9}$	14	−11
0 0 5	59	57	4 0 $\bar{6}$	23	−21	8 0 $\overline{10}$	11	10
0 0 6	8	5	4 0 $\bar{7}$	40	−40	8 0 $\overline{11}$	17	14
0 0 7	14	14	4 0 $\bar{8}$	46	−43	8 0 $\overline{12}$	15	14
0 0 8	25	24	4 0 $\bar{9}$	31	−30	8 0 $\overline{13}$	6	7
0 0 9	18	19	4 0 $\overline{10}$	27	−27	8 0 $\overline{14}$	12	15
0 0 10	13	−14	4 0 $\overline{11}$	30	−29	8 0 $\overline{15}$	26	25
0 0 11	9	−11	4 0 $\overline{12}$	30	−27	8 0 $\overline{16}$	16	20
0 0 12	5	− 4	4 0 $\overline{13}$	21	−20	10 0 0	24	24
0 0 13	12	−11	4 0 $\overline{14}$	15	−15	10 0 1	23	23
0 0 14	14	−14	4 0 $\overline{15}$	11	−13	10 0 2	18	20
2 0 0	35	−48	6 0 0	37	39	10 0 3	14	13
2 0 1	40	−38	6 0 1	25	21	10 0 4	18	18
2 0 2	35	−41	6 0 2	22	19	10 0 5	23	23
2 0 3	30	−31	6 0 3	12	7	10 0 6	14	15
2 0 4	35	−36	6 0 4	10	7	10 0 7	12	14
2 0 5	52	−52	6 0 5	12	7	10 0 8	13	12
2 0 6	39	−41	6 0 6	6	1	10 0 $\bar{1}$	5	− 5
2 0 7	21	−22	6 0 7	14	−12	10 0 $\bar{2}$	5	− 1
2 0 8	27	−29	6 0 8	9	−10	10 0 $\bar{3}$	13	11
2 0 9	27	−28	6 0 9	9	−10	10 0 $\bar{4}$	12	9
2 0 10	11	−12	6 0 10	11	−11	10 0 $\bar{5}$	14	−16
2 0 11	13	−12	6 0 11	18	−17	10 0 $\bar{6}$	15	−13
2 0 12	6	− 7	6 0 $\bar{1}$	41	45	10 0 $\bar{7}$	< 2	2
2 0 13	13	−11	6 0 $\bar{2}$	46	47	10 0 $\bar{8}$	13	−13
2 0 $\bar{1}$	13	− 8	6 0 $\bar{3}$	33	30	10 0 $\bar{9}$	24	−25
2 0 $\bar{2}$	14	−18	6 0 $\bar{4}$	32	32	10 0 $\overline{10}$	33	−38
2 0 $\bar{3}$	41	−45	6 0 $\bar{5}$	50	55	10 0 $\overline{11}$	12	−12
2 0 $\bar{4}$	5	− 7	6 0 $\bar{6}$	43	46	10 0 $\overline{12}$	9	−10
2 0 $\bar{5}$	28	27	6 0 $\bar{7}$	39	38	10 0 $\overline{13}$	27	−26
2 0 $\bar{6}$	25	26	6 0 $\bar{8}$	13	13	10 0 $\overline{14}$	26	−26
2 0 $\bar{7}$	6	9	6 0 $\bar{9}$	29	25	10 0 $\overline{15}$	18	−18
2 0 $\bar{8}$	11	12	6 0 $\overline{10}$	39	32	10 0 $\overline{16}$	9	− 9
2 0 $\bar{9}$	24	21	6 0 $\overline{11}$	17	14	12 0 0	8	5
2 0 $\overline{10}$	27	27	6 0 $\overline{12}$	4	1	12 0 1	4	− 6
2 0 $\overline{11}$	29	27	6 0 $\overline{13}$	6	2	12 0 2	14	−15
2 0 $\overline{12}$	20	19	6 0 $\overline{14}$	7	5	12 0 3	5	2
2 0 $\overline{13}$	29	27	6 0 $\overline{15}$	< 2	0	12 0 4	2	− 4
2 0 $\overline{14}$	23	22	8 0 0	21	−22	12 0 5	17	−16
4 0 0	13	−13	8 0 1	30	−29	12 0 6	11	−11
4 0 1	9	4	8 0 2	26	−27	12 0 $\bar{1}$	18	15
4 0 2	12	5	8 0 3	32	−33	12 0 $\bar{2}$	16	14
4 0 3	18	14	8 0 4	19	−19	12 0 $\bar{3}$	6	− 4
4 0 4	12	13	8 0 5	17	−15	12 0 $\bar{4}$	20	19
4 0 5	20	19	8 0 6	16	−15	12 0 $\bar{5}$	31	32
4 0 6	35	36	8 0 7	10	−12	12 0 $\bar{6}$	16	14
4 0 7	26	27	8 0 8	< 2	− 4	12 0 $\bar{7}$	11	11
4 0 8	17	17	8 0 9	< 2	0	12 0 $\bar{8}$	19	22
4 0 9	20	20	8 0 $\bar{1}$	22	−23	12 0 $\bar{9}$	29	32
4 0 10	26	25	8 0 $\bar{2}$	33	−35	12 0 $\overline{10}$	19	21
4 0 11	25	25	8 0 $\bar{3}$	15	−18	12 0 $\overline{11}$	15	15
4 0 12	12	13	8 0 $\bar{4}$	39	−46	12 0 $\overline{12}$	13	14
4 0 $\bar{1}$	6	8	8 0 $\bar{5}$	24	−20	12 0 $\overline{13}$	20	21

Table 4. Continued.

h k l	F_o	F_c	h k l	F_o	F_c	h k l	F_o	F_c
12 0 $\overline{14}$	13	13	18 0 $\overline{4}$	3	— 1	12 4 0	<13	2
12 0 $\overline{15}$	< 2	2	18 0 $\overline{5}$	5	0	14 4 0	17	15
14 0 0	17	—14	18 0 $\overline{6}$	8	5	1 5 0	<10	2
14 0 1	15	—12	18 0 $\overline{7}$	13	10	3 5 0	25	21
14 0 2	3	— 3	18 0 $\overline{8}$	5	5	5 5 0	39	31
14 0 3	6	— 5	18 0 $\overline{9}$	5	5	7 5 0	<11	7
14 0 4	5	— 6	18 0 $\overline{10}$	9	11	9 5 0	13	13
14 0 $\overline{1}$	5	— 6	18 0 $\overline{11}$	11	14	11 5 0	16	16
14 0 $\overline{2}$	14	—16	18 0 $\overline{12}$	10	12	13 5 0	13	15
14 0 $\overline{3}$	20	—20	1 1 0	12	13	0 6 0	42	40
14 0 $\overline{4}$	19	—22	3 1 0	20	22	2 6 0	25	21
14 0 $\overline{5}$	14	—15	5 1 0	52	52	4 6 0	<11	6
14 0 $\overline{6}$	10	—11	7 1 0	20	13	6 6 0	<12	6
14 0 $\overline{7}$	16	—16	9 1 0	22	18	8 6 0	20	22
14 0 $\overline{8}$	18	—17	11 1 0	20	18	10 6 0	18	15
14 0 $\overline{9}$	11	—12	13 1 0	<13	12	12 6 0	< 9	2
14 0 $\overline{10}$	3	— 2	15 1 0	<11	3	14 6 0	11	13
14 0 $\overline{11}$	14	—14	17 1 0	< 8	9	1 7 0	<12	1
14 0 $\overline{12}$	4	— 5	0 2 0	42	69	3 7 0	27	25
14 0 $\overline{13}$	2	2	2 2 0	29	37	5 7 0	32	28
14 0 $\overline{14}$	< 2	0	4 2 0	29	26	7 7 0	<13	6
16 0 0	12	12	6 2 0	36	29	9 7 0	<11	11
16 0 1	12	14	8 2 0	31	29	11 7 0	< 8	11
16 0 $\overline{1}$	11	10	10 2 0	25	25	13 7 0	< 6	11
16 0 $\overline{2}$	13	13	12 2 0	<13	6	0 8 0	26	25
16 0 $\overline{3}$	14	14	14 2 0	17	17	2 8 0	<13	12
16 0 $\overline{4}$	5	7	1 3 0	26	26	4 8 0	<13	5
16 0 $\overline{5}$	5	8	3 3 0	38	32	6 8 0	<12	10
16 0 $\overline{6}$	13	11	5 3 0	45	43	8 8 0	12	13
16 0 $\overline{7}$	< 2	4	7 3 0	21	18	10 8 0	< 7	13
16 0 $\overline{8}$	< 2	1	9 3 0	25	17	12 8 0	< 3	3
16 0 $\overline{9}$	< 2	0	11 3 0	18	17	1 9 0	<12	7
16 0 $\overline{10}$	< 2	— 3	13 3 0	<12	15	3 9 0	21	21
16 0 $\overline{11}$	< 2	— 3	15 3 0	< 8	7	5 9 0	21	21
16 0 $\overline{12}$	11	—11	0 4 0	34	34	7 9 0	< 7	7
16 0 $\overline{13}$	8	— 9	2 4 0	27	26	9 9 0	9	9
16 0 $\overline{14}$	5	— 7	4 4 0	20	18	0 10 0	20	19
18 0 $\overline{1}$	5	— 6	6 4 0	15	15	2 10 0	< 7	8
18 0 $\overline{2}$	3	— 6	8 4 0	24	22	4 10 0	< 6	1
18 0 $\overline{3}$	3	1	10 4 0	20	14	6 10 0	10	10

about 2.8 Å and the three free phosphate oxygens another such triangle with edges about 2.5 Å. All these triangles lie roughly in planes parallel to the *ab* plane at approximately $z = 0.25$ and 0.75. Each of them is connected by hydrogen bonds to three triangles of the other type in the same plane. The lengths of these bonds are 2.50 Å, 2.63 Å and 2.64 Å, and they are evidently formed by the hydroxyl hydrogen atoms, as is apparent also from their directions. In addition, the water molecules which are associated with the barium ions take part in a number of hydrogen bonds. Thirteen such oxygen-oxygen contacts are shorter than 3.2 Å, lying in the range between 2.63 Å and 3.04 Å. However, only nine of those correspond presumably to hydrogen bonds as

this is the number of available hydrogen atoms (four and a half water mole-
cules). In addition there appears to be a hydrogen bond between $(H_2O)_{14}$ and
O_5. In Table 3 oxygen-oxygen contacts shorter than 3.2 Å are given. They are
all indicated in Fig. 4 by broken lines. In Fig. 5 only the hydrogen bonds be-
tween hydroxyl and phosphate oxygens are shown.

REFERENCES

1. Levene, P. A. and Jacobs, W. A. *Ber.* **44** (1911) 746.
2. Levene, P. A., Harris, S. A. and Stiller, E. T. *J. Biol. Chem.* **105** (1934) 153.
3. Furberg, S. *Acta Cryst.* **3** (1950) 325.
4. Alver, E. and Furberg, S. *Acta Chem. Scand.* **13** (1959) 910.
5. Forsyth, J. B. and Wells, M. *Acta Cryst.* **12** (1959) 412.
6. Foss, O., Furberg, S. and Zachariassen, H. *Acta Chem. Scand.* **8** (1954) 459.
7. Trueblood, K. N. and Horn, P. Luzzati. *Acta Cryst.* **14** (1961) 965.
8. Mani, N. V. and Ramaseshan, S. *Z. Krist.* **114** (1960) 200.
9. Brintzinger, H. *Helv. Chim. Acta* **44** (1961) 199.
10. Furberg, S. *Acta Chem. Scand.* **6** (1952) 634.
11. Gerdil, R. *Acta Cryst.* **14** (1961) 333.