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The Crystal Structure of 5-Hydroxyuridine

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Crystals of 5-hydroxyuridine are monoclinic, space group $P2_1$, with $a = 11.362$ (3), $b = 6.593$ (2), $c = 6.897$ (2) Å and $\beta = 97.65$ (3)°. X-ray diffraction data were collected with an automated diffractometer. The structure was solved by Patterson and trial-and-error methods and was refined by the least-squares method to $R = 0.026$. Hydroxyuracil bases are hydrogen bonded to form ribbons that run in the b direction and are stacked in the c direction. The stacking pattern, which is similar to that found in other crystal structures of 5-substituted pyrimidines, involves slight base overlap and interactions of atoms O(2) and O(5) with the pyrimidine rings of adjacent bases. The conformation about the glycosidic C–N bond is *anti*, with $\chi_{CN} = 42.1°$. The ribose moiety is in the C(2')-*endo* conformation.

Introduction

The base-stacking patterns in crystals of purines and pyrimidines, and in fibers of helical polynucleotides, generally involve slight overlap of bases and are such that hetero-atom substituents interact with the ring systems of adjacent bases within the stacks (Bugg, Thomas, Sundaralingam & Rao, 1971; Bugg, 1972). Thus base-stacking patterns are largely governed by the distribution of hetero-atom substituents. An interesting effect on base-stacking patterns apparently results from the addition of halogen substituents to the 5-position of uracil bases, since in most crystal structures of 5-halogenated uracil derivatives the stacking patterns involve interactions between the halogen substituents and the pyrimidine rings of neighboring bases (Bugg, Thomas, Sundaralingam & Rao, 1971; Bugg & Thewalt, 1972). We determined the crystal structure of 5-hydroxyuridine (Fig. 1) to see if a hydroxyl group at the 5 position of uracil would exert similar effects on the base-stacking pattern.

Experimental

Hydroxyuridine was crystallized as large clear plates by evaporating an acetonitrilic solution that contained hydroxyuridine and a few drops of water. Weissenberg and oscillation photographs showed that these crystals are monoclinic; the space group is $P2_1$, as indicated by the systematic absence of reflections 0k0 with k odd. A fragment (with approximate dimensions 0·05, 0·30 and 0·15 mm in the **a**, **b** and **c** directions, respectively)

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Table 1. *Crystal data*

Stoichiometry	$C_9H_{12}N_2O_7$
Z	2
Space group	$P2_1$
a	11·362 (3) Å
b	6·593 (2)
c	6·897 (2)
β	97·65 (3)°
ϱ(calculated)	1·687 g cm^{-3}
ϱ(observed)	1·69 g cm^{-3}
μ(Cu $K\alpha$)	13·0 cm^{-1}

(The reported standard deviations are five times greater than those obtained from the least-squares analysis. The density was measured by flotation in a mixture of benzene and ethylene dibromide.)

was sliced from a larger crystal and then mounted on a Picker FACS-1 diffractometer with its b axis slightly inclined to the φ axis of the diffractometer. Unit-cell parameters were obtained by a least-squares analysis of 2θ values for 11 high-angle reflections (Cu $K\alpha_1$, $\lambda =$ 1·54051 Å) measured with the diffractometer. Table 1 lists the crystal data.

Intensity data were collected with the diffractometer, by use of a scintillation counter, nickel-filtered copper radiation, and a $\theta-2\theta$ scanning technique. The scanning speed was 1°/min, and a 20 sec background measurement was performed at each terminus of the scans. Measurements were made for the 915 independent reflections with $2\theta \le 127°$.

The intensities were assigned variances, $\sigma^2(I)$, according to the statistics of the scan and background counts plus a correctional term, $(0.03S)^2$, S being the scan counts. The intensities and their variances were corrected for Lorentz and polarization factors; absorption corrections were applied by using the program *ORABS* (Wehe, Busing & Levy, 1962), and then the data were scaled by means of a Wilson (1942) plot.

Determination and refinement of the structure

A sharpened three-dimensional Patterson map had major features in the two sections $w=0$ and $w=0.5$, suggesting that the planar pyrimidine moieties are approximately parallel to the (001) plane and are separated by $c/2$. This indicated that the pyrimidine moieties are situated at approximately $z=\frac{1}{4}$ and $z=\frac{3}{4}$. Approximate x and y coordinates for these atoms were obtained by evaluating the R index, $(\sum||F_o| - |F_c||/\sum|F_o|)$, while varying the position and orientation of the hydroxyuracil moiety within the $z=\frac{1}{4}$ plane. This was accomplished by use of a computer program which systematically shifted the molecular fragment to various points in the x direction (at $z=\frac{1}{4}$) and rotated the fragment around a vector that lay perpendicular to the pyrimidine ring and passed through its center. The R index, which was calculated for each orientation of the hydroxyuracil moiety, showed a pronounced

Fig. 1. Structural formula of 5-hydroxyuridine.

Table 2. *The final heavy-atom parameters and their standard deviations*

The values have been multiplied by 10^4. The y coordinate of atom N(1) was not refined. Temperature factors are in the form $T = \exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$. The final value of the isotropic extinction parameter is $g = 0.0514$ (6).

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
N(1)	3946 (2)	4964	2814 (3)	31 (1)	99 (5)	153 (4)	−4 (2)	5 (2)	−4 (4)
C(2)	4469 (2)	6815 (5)	2789 (4)	36 (2)	117 (6)	145 (6)	−3 (3)	4 (2)	−4 (5)
O(2)	3958 (2)	8435 (4)	2965 (3)	46 (1)	109 (5)	292 (6)	−6 (2)	23 (2)	−12 (4)
N(3)	5649 (2)	6786 (4)	2531 (3)	36 (2)	115 (5)	190 (5)	−26 (2)	18 (2)	−15 (4)
C(4)	6331 (2)	5104 (5)	2243 (3)	41 (2)	166 (7)	130 (5)	0 (3)	13 (2)	−14 (5)
O(4)	7375 (2)	5270 (4)	1997 (3)	41 (1)	215 (6)	245 (5)	−13 (2)	43 (2)	−50 (5)
C(5)	5696 (2)	3198 (5)	2237 (3)	45 (2)	118 (6)	136 (5)	6 (3)	18 (2)	−1 (5)
O(5)	6290 (2)	1442 (4)	2017 (3)	53 (2)	136 (5)	317 (6)	14 (2)	67 (2)	9 (5)
C(6)	4551 (2)	3189 (5)	2497 (4)	40 (2)	117 (6)	147 (5)	0 (3)	12 (2)	2 (5)
C(1′)	2673(2)	4891(5)	2987 (3)	33 (2)	98 (6)	137 (5)	−1 (3)	10 (2)	4 (5)
O(1′)	2458 (1)	3192 (4)	4145 (2)	37 (1)	135 (4)	156 (4)	−11 (2)	5 (2)	29 (4)
C(2′)	1871 (2)	4527 (5)	1054 (3)	32 (2)	90 (5)	132 (5)	6 (3)	11 (2)	−0 (5)
O(2′)	1763(1)	6190 (4)	−228 (2)	39 (1)	129 (4)	150 (4)	5 (2)	13 (2)	18 (3)
C(3′)	729 (2)	3850 (5)	1814 (3)	31 (2)	106 (6)	155 (5)	−6 (3)	8 (2)	−20 (5)
O(3′)	88 (1)	5598 (4)	2305 (3)	35 (1)	144 (5)	236 (5)	10 (2)	22 (2)	−12 (4)
C(4′)	1211 (2)	2631 (5)	3658 (4)	35 (2)	130 (6)	142 (5)	−8 (3)	27 (2)	−4 (5)
C(5′)	1121 (2)	373 (5)	3366 (4)	48 (2)	130 (6)	177 (6)	−10 (3)	26 (3)	9 (5)
O(5′)	1649 (2)	−251 (4)	1702 (3)	61 (2)	130 (5)	218 (4)	−1 (2)	39 (2)	−28 (4)

minimum value of 0·45 as soon as the correct orientation of this molecular fragment was attained. Coordinates for the remaining non-hydrogen atoms were then obtained from a three-dimensional Fourier map that was calculated with phase angles derived from the atoms of the hydroxyuracil moiety.

Table 3. *The final hydrogen-atom parameters and their standard deviations*

The positional parameters have been multiplied by 10^3, thermal parameters by 10.

	x	y	z	$B(\text{Å}^2)$
H(N3)	596 (2)	790 (4)	251 (3)	27 (5)
H(O5)	697 (2)	170 (5)	167 (4)	35 (6)
H(C6)	406 (2)	196 (4)	247 (4)	31 (5)
H(C1′)	249 (2)	617 (3)	358 (3)	11 (4)
H(C2′)	223 (2)	337 (4)	35 (3)	27 (5)
H(O2′)	155 (3)	722 (6)	47 (5)	59 (9)
H(C3′)	28 (2)	302 (4)	90 (3)	21 (4)
H(O3′)	− 67 (4)	524 (8)	188 (6)	90 (12)
H(C4′)	82 (2)	302 (4)	480 (3)	16 (4)
H(C5′)	29 (2)	− 6 (5)	313 (4)	38 (6)
H(C5′)′	150 (2)	− 39 (5)	447 (3)	29 (5)
H(O5′)	241 (3)	− 29 (6)	207 (4)	42 (6)

The trial structure was refined by use of a modified version of the full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962; Busing, 1971). The quantity minimized was $\sum w(F_o^2 - F_c^2/k^2)^2$, where k is a scale factor and the weight w is equal to $1/\sigma^2(F_o^2)$. Scattering factors for the non-hydrogen atoms were from *International Tables for X-Ray Crystallography* (1962); anomalous dispersion correction factors for these atoms were from Cromer & Liberman (1970), and hydrogen-atom scattering factors were from Stewart, Davidson & Simpson (1965). All hydrogen atoms were located in difference Fourier maps that were calculated during the latter stages of refinement. Final cycles of refinement included all positional parameters, with anisotropic temperature factors for the heavy atoms, isotropic temperature factors for the hydrogen atoms, and Zachariasen's (1963) isotropic extinction parameter g (as formulated by Coppens & Hamilton, 1970). Because of the limited core storage capacity of the computer, it was impracticable to refine all parameters simultaneously; consequently, the nonhydrogen atoms were refined together, and the hydrogen atoms were refined in the alternate cycles.

Table 4. *Observed and calculated structure factors*

From left to right, the columns contain values of h, $10F_o$ and $10|F_c|$.

[Structure factor table — dense numeric data not legibly reproducible]

Table 5. *Hydrogen bond distances and angles*

Donor atom (D)	Hydrogen atom	Acceptor atom (A)	Distances D–A	A–H	D–H–A angle
N(3)	H(N3)	O(5) a	3·185 Å	2·40 Å	163°
O(5)	H(O5)	O(2′) b	2·678	1·89	153
C(6)	H(C6)	O(2) c	3·321	2·35	148
O(2′)	H(O2′)	O(5′) a	2·709	1·87	158
O(3′)	H(O3′)	O(4) d	3·069	2·23	154
O(5′)	H(O5′)	O(2) c	2·789	1·98	156

Symmetry codes: a: $x, y+1, z$; b: $1-x, y-\frac{1}{2}, -z$; c: $x, y-1, z$; d: $x-1, y, z$.

The final R index is 0·026, and the R index based on F^2 values $(\sum|F_o^2 - F_c^2|/\sum F_o^2)$ is 0·037. The goodness-of-fit $\{[\sum w(F_o^2 - F_c^2/k^2)/(m-s)]^{1/2}$, where m is the number of reflections used and s is the number of parameters refined$\}$ is 1·73. During the last cycle of refinement, no heavy-atom parameter shifted more than one third of its standard deviation and no hydrogen-atom parameter shifted more than one half of its standard deviation. A final three-dimensional difference Fourier map showed no peaks or troughs with magnitudes that exceeded 0·3 e Å^{-3}.

The heavy-atom parameters and their estimated standard deviations are listed in Table 2, the hydrogen-atom parameters and their estimated standard deviations in Table 3, and observed and calculated structure factors are given in Table 4.

The crystal packing

Fig. 2 shows the structure projected down the c axis, a direction which is approximately perpendicular to the pyrimidine rings. The hydrogen-bonding scheme is also
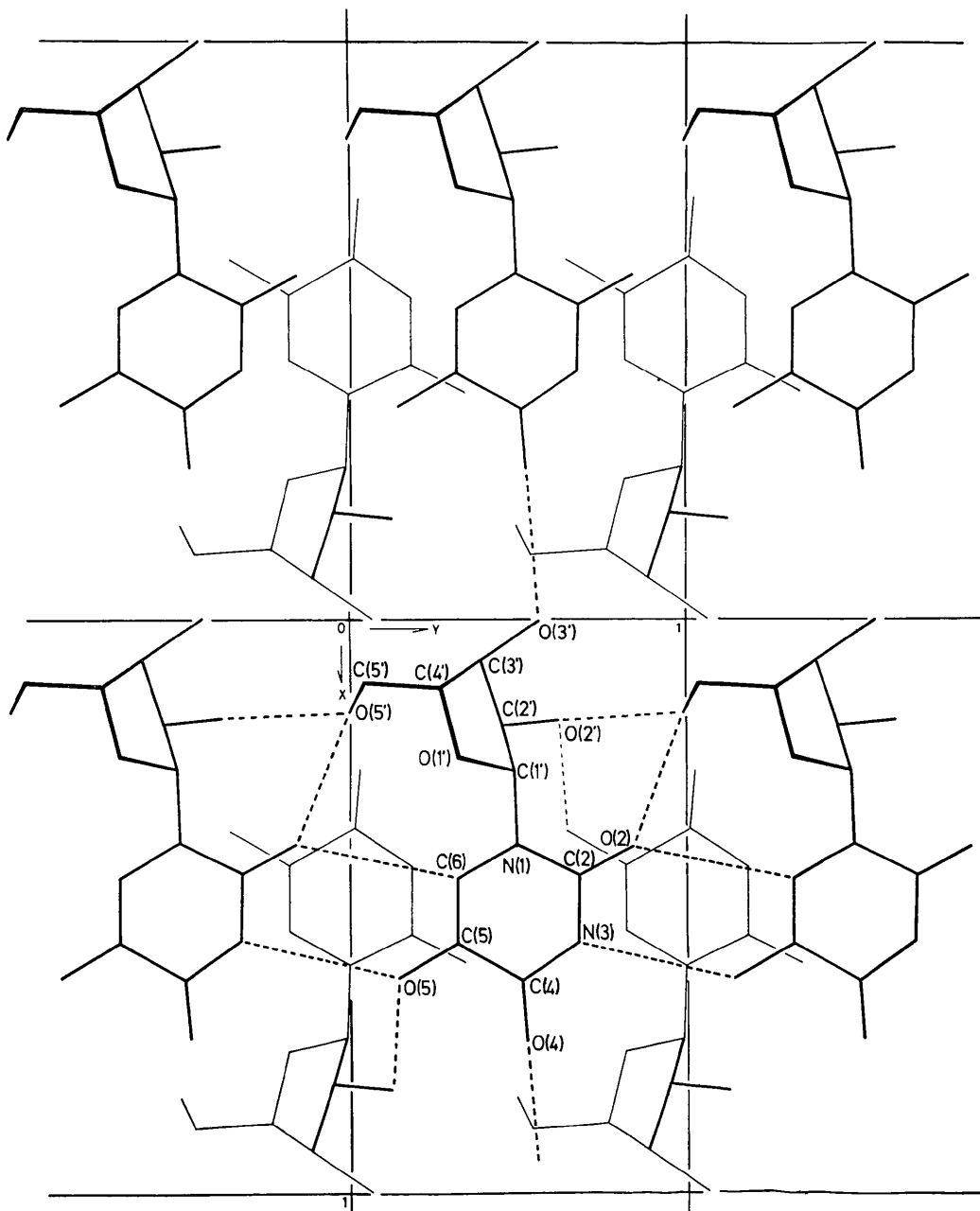
Fig. 2. The crystal structure viewed down the c axis. Only those hydrogen bonds that involve the labeled molecule are shown.

depicted in Fig. 2, and Table 5 gives the hydrogen-bond lengths and angles. All hydrogen atoms that are co-alently bonded to nitrogen and oxygen atoms appear to participate in hydrogen bonding. In addition there is a short $C(6)-H\cdots O(2)$ contact which may be considered a hydrogen bond.

Hydroxyuracil moieties are arranged in planar ribbons that lie at $z = \frac{1}{4}$ and $z = \frac{3}{4}$ and run in the **b** direction. Adjacent bases within the ribbons are joined by relatively long $N(3)-H\cdots O(5)$ hydrogen bonds, and by the $C(6)-H\cdots O(2)$ contacts. The ribbons of bases are stacked in the **c** direction, and are separated by an interplanar spacing of about 3·4 Å. The ribose moieties are arranged in channels between stacks of bases. Thus the crystal structure consists of successive layers of sugars and bases, with the layers running parallel to the **bc** plane.

The base-stacking pattern can be seen in Fig. 2. The bases in adjacent ribbons are staggered, so that the pyrimidine rings do not overlap. The principal contact between neighboring bases in the ribbons involves the O(2) and O(5) substituents of the hydroxyuracil bases. This type of stacking pattern is common in crystals of purines and pyrimidines (Bugg, Thomas, Sundaralingam & Rao, 1971; Bugg, 1972). There are few examples of pyrimidine stacking patterns wherein the bases overlap to any great extent. Rather the stacking patterns are generally of the type found in this structure with the hetero-atom substituents forming the only close contacts with adjacent bases. The hydroxyuracil stacking pattern is analogous to that found in the crystal structures of many 5-halogenated uracil derivatives where the halogen substituent and atom O(2) interact with the pyrimidine rings of adjacent bases (Bugg, Thomas, Sundaralingam & Rao, 1971; Bugg & Thewalt, 1972).
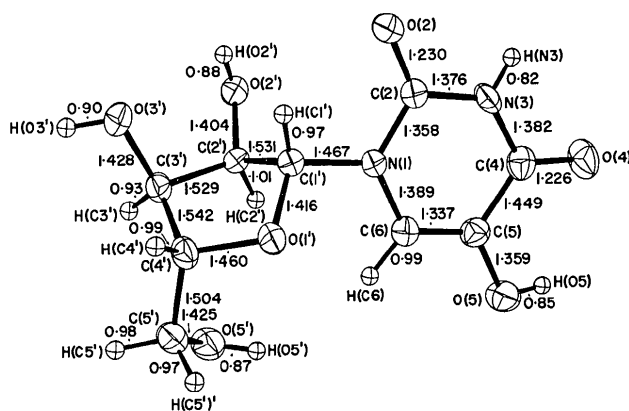

Fig. 3. Conformation of 5-hydroxyuridine, including the bond lengths. The estimated errors in bond lengths are about 0·004 Å for bonds that involve only nonhydrogen atoms and about 0·04 Å for bonds that involve hydrogen atoms. The non-hydrogen atoms are represented by ellipsoids defined by the principal axes of thermal vibration and scaled to include 50 % probability. The hydrogen atoms are represented by spheres of 0·1 Å radius. This drawing was prepared by using the computer program *ORTEP* (Johnson, 1965).

Molecular structure

The conformation, heavy-atom thermal ellipsoids, and bond lengths are shown in Fig. 3. Bond angles involving only nonhydrogen atoms are listed in Table 6. Torsion angles are given in Table 7. As in most other crystal structures of nucleosides and nucleotides (Donohue & Trueblood, 1960; Sundaralingam, 1969), the conformation around the glycosidic sugar-base bond is *anti*; the torsion angle, $\chi_{CN}[O(1')-C(1')-N(1)-C(6)]$, is 42·1°. The ribose ring assumes the $C(2')$-*endo* conformation (Sundaralingam, 1965, 1969). The best four-atom plane through the pentose ring is defined by atoms $C(1')$, $O(1')$, $C(4')$, and $C(3')$, none of which deviate more than 0·07 Å from the plane, with atom $C(2')$ displaced 0·64 Å from the plane in the same direction as atom $C(5')$. The conformation about the $C(4')-C(5')$ bond is *gauche-gauche* (Shefter & Trueblood, 1965; Sundaralingam, 1969) the torsion angle $\varphi_{OO}[O(5')-C(5')-C(4')-O(1')]$ is $-65\cdot7°$, and the torsion angle $\varphi_{OC}[O(5')-C(5')-C(4')-C(3')]$ is 52·8°. As shown in Table 8, which lists deviations from the least-

Table 6. *Bond angles involving only nonhydrogen atoms*

The standard deviations are about 0·2°.

$C(1')-N(1)-C(2)$	117·9°
$C(1')-N(1)-C(6)$	119·9
$C(2)-N(1)-C(6)$	121·9
$N(1)-C(2)-O(2)$	124·5
$N(1)-C(2)-N(3)$	115·1
$O(2)-C(2)-N(3)$	120·4
$C(2)-N(3)-C(4)$	127·2
$N(3)-C(4)-O(4)$	121·3
$N(3)-C(4)-C(5)$	114·1
$O(4)-C(4)-C(5)$	124·6
$C(4)-C(5)-O(5)$	119·0
$C(4)-C(5)-C(6)$	119·8
$O(5)-C(5)-C(6)$	121·2
$C(5)-C(6)-N(1)$	121·9
$N(1)-C(1')-O(1')$	108·6
$N(1)-C(1')-C(2')$	114·4
$O(1')-C(1')-C(2')$	103·9
$C(1')-O(1')-C(4')$	107·8
$C(1')-C(2')-O(2')$	114·7
$C(1')-C(2')-C(3')$	100·4
$O(2')-C(2')-C(3')$	116·0
$C(2')-C(3')-O(3')$	109·2
$C(2')-C(3')-C(4')$	102·2
$O(3')-C(3')-C(4')$	111·2
$O(1')-C(4')-C(3')$	106·5
$O(1')-C(4')-C(5')$	109·1
$C(3')-C(4')-C(5')$	113·3
$C(4')-C(5')-O(5')$	111·4

Table 7. *Conformational torsion angles*

The estimated standard deviations are about 0·4°.

$O(1')-C(1')-N(1)-C(6)$	42·1°
$O(5')-C(5')-C(4')-O(1')$	$-65\cdot7$
$O(5')-C(5')-C(4')-C(3')$	52·8
$C(2')-C(1')-O(1')-C(4')$	$-34\cdot6$
$C(3')-C(2')-C(1')-O(1')$	44·1
$C(4')-C(3')-C(2')-C(1')$	$-35\cdot8$
$O(1')-C(4')-C(3')-C(2')$	16·8
$C(1')-O(1')-C(4')-C(3')$	11·1

squares plane through the pyrimidine ring, the hydroxy-uracil moiety is significantly nonplanar.

Table 8. *Deviations from a least-squares plane through the 6 atoms of the pyrimidine ring*

N(1)	−0·016 Å
C(2)	0·013
N(3)	−0·002
C(4)	−0·005
C(5)	0·002
C(6)	0·009
C(1′)	0·065
O(2)	0·059
O(4)	0·003
O(5)	−0·035
H(N3)	0·017
H(O5)	0·104
H(C6)	0·042

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The Crystal Structure of Magnesium Diethyl Phosphate, Mg[PO2(OC2H5)2]2

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The crystal structure of magnesium diethyl phosphate, $Mg[PO_2(OC_2H_5)_2]_2$, has been determined from three-dimensional X-ray diffraction data. The crystal is monoclinic, space group $C2/c$, with $a = 22·14$ (2), $b = 7·998$ (4), $c = 9·300$ (3) Å, $\beta = 96·72$ (5)° and $Z = 4$. The intensities were measured using the θ–2θ scan method with a Picker automatic 4-circle diffractometer and filtered Mo K radiation. The structure was solved by Patterson and Fourier methods, and refined by full-matrix least squares. The final R value was 0·061 for 732 observed reflections. Magnesium ions are located on a twofold symmetry axis and coordinated in a nearly regular tetrahedral arrangement to four oxygen atoms with a mean O–Mg–O angle of 109·14° and a mean Mg–O distance of 1·091 Å. The ester conformation with respect to the phosphate group is *synclinal–synclinal*.

Introduction

Single-crystal X-ray diffraction analysis of phosphodiesters has provided information on the possible conformations of the sugar-phosphate backbone in polynucleotides and nucleic acids (Shefter, Barlow, Sparks & Trueblood, 1969; Sundaralingam, 1969).

The conformation of the diester phosphate ion shows a range of allowable values and, in particular, the diethyl phosphate anion conformation is different in the two crystal structures which have been determined, barium diethyl phosphate (Kyogoku & Iitaka, 1966) and silver diethyl phosphate (Hazel & Collin, 1972). The present study was carried out to determine the