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The Crystal and Molecular Structure of O^2,2′-Cyclouridine. Influence of O(2)–C(2′) Cyclization on the Sugar Conformation of Pyrimidine Nucleosides

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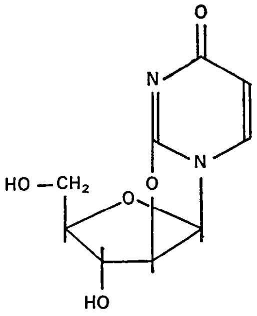
The molecular and crystal structure of O^2,2′-cyclouridine, a useful intermediate in the inversion or replacement of the O(2′) hydroxyl group of uridine, has been investigated by X-ray analysis. The structure of the crystals, space group $P2_12_12_1$, with cell dimensions $a=13{\cdot}681$ (3), $b=7{\cdot}441$ (2), $c=18{\cdot}242$ (3) Å and two molecules in the asymmetric unit, was solved by direct methods and refined to a discrepancy index $R=4{\cdot}5$% for 1762 independent reflexions. The cyclization between atoms O(2) and C(2′) results in a considerable alteration of the geometry of the base compared with normal uridine derivatives. Strongly affected are the bond angles at C(2), N(1) and C(1′), which are directly involved in the formation of the additional five-membered ring. The unusual C(4′)-*endo* and C(4′)-*endo*-C(3′)-*exo* sugar conformations of the two crystallographically independent molecules reflect the rigidity imposed on the ring system by the O(2)–C(2′) cyclization. The orientation of the C(5′)–O(5′) bond is *gauche-trans* in both molecules. Within the crystal, hydrogen-bonded dimers of O^2,2′-cyclouridine form stacks along the *b* axis with alternating interplanar spacings of 3·3 and 3·8 Å respectively.

Introduction

Pyrimidine O^2,2′-cyclonucleosides have successfully been used as intermediates for the specific inversion or replacement of the ribose *O*(2′)-hydroxyl group. Thus spongouridine (3-β-D-arabinofuranosyluracil) could be obtained by hydrolysis of O^2,2′-cyclouridine with dilute acid (Brown, Todd & Varadarajan, 1956) and a similar ring opening by treatment with anhydrous hydrogen halide gave the various 2′-halogeno-2′-deoxyuridines (Codington, Doerr, Praag, Bendich & Fox, 1961). From the reactivity of these cyclonucleosides one would expect the molecules to be strained to some extent. In order to establish the influence of the O(2)–C(2′)-cyclization on bond angles and distances and on the sugar conformation in O^2,2′-cyclouridine, we have investigated its molecular structure by X-ray structural analysis.



Materials and methods

We are grateful to Dr W. Guschlbauer for a sample of O^2,2′-cyclouridine. The material crystallized in the form of stout colourless prisms in the orthorhombic space group $P2_12_12_1$ with two molecules per asymmetric unit. The crystallographic data presented in Table 1 were obtained from Weissenberg and precession photographs and measurements by means of an automatic Stoe four-circle diffractometer using graphite-monochromatized Cu $K\alpha$ radiation. The intensities of 1831 reflexions up to $\theta=65°$ were measured using the θ–2θ scan technique and corrected for Lorentz and polarization factors, but not for absorption because of the small linear absorption coefficient (Table 1). From these data normalized structure factors E (Karle & Hauptman, 1956) were calculated.

Table 1. *Crystallographic data*

Space group $P2_12_12_1$
$a=13{\cdot}681$ (3) Å
$b=\ 7{\cdot}441$ (2)
$c=18{\cdot}242$ (3)
Molecules per cell $Z=8$ (two molecules per asymmetric unit)
Cu $K\alpha$ radiation $\lambda=1{\cdot}54182$ Å
Density observed $\varrho_{\text{obs}}=1{\cdot}623$ g cm^{-3} (flotation in a toluene/CH_3I mixture).
Density calculated $\varrho_{\text{calc}}=1{\cdot}618$ g cm^{-3}
Chemical formula $C_9H_{10}N_2O_5$
M. W. 226·19
Linear absorption coefficient $\mu=11{\cdot}7$ cm^{-1}

The structure was solved by direct methods using the program *MULTAN* (Main, Germain & Woolfson, 1970) which combines the cyclic application of the tangent formula (Karle & Hauptman, 1956) with multi-solution techniques (Germain & Woolfson, 1968). The starting set was automatically chosen by the program and consisted of 9 reflexions with large E values, including three origin reflexions and one enantiomorph defining reflexion. For all the 16 possible phase angle combinations within this starting set we obtained phase angles for the 231 E's $>1{\cdot}45$. From a Fourier synthesis (E map), calculated with the phase information of the most consistent solution, we found the positions of all the nonhydrogen atoms of the two cyclouridine molecules.

The initial R index ($R=\sum||F_o|-|F_c||/\sum|F_o|$) of 23 % for the 1762 significant reflexions dropped to 10 % after three cycles of isotropic full-matrix least-squares refinement (Busing, Martin & Levy, 1962). We applied a weighting scheme based on counting statistics with 2 % allowance for machine error (Stout & Jensen, 1968) and data with $F_o<3/\sqrt{w_{F_o}}$ were treated as unobserved and not included in the refinement process. All hydrogen atoms could be located in a subsequent difference Fourier synthesis and were assigned the isotropic temperature factors of the 'heavy' atoms to which they were covalently bound. After four cycles of anisotropic full-matrix least-squares refinement of the positional and thermal parameters of the heavy atoms and three cycles of refinement of the hydrogen positions the average parameter shifts were less than

Table 2. *Atomic coordinates in fractions of the crystallographic axes and thermal parameters*

Thermal parameters are in the form $T=\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\times10^4]$. Isotropic temperature factors are given for the hydrogen atoms. Estimated standard deviations for the last decimal place are given in parentheses. Coordinates for nonhydrogen atoms are $\times10^4$ and for hydrogen atoms, $\times10^3$.

(1) Molecule *A*

	x	y	z	β_{11} or B	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
N(1)	863 (2)	2669 (5)	3494 (2)	28 (2)	138 (7)	17 (1)	3 (3)	1 (1)	−2 (2)
C(2)	1032 (2)	2663 (5)	4215 (2)	24 (2)	112 (7)	19 (1)	9 (3)	0 (1)	5 (2)
O(2)	221 (2)	2285 (4)	4599 (1)	25 (1)	165 (6)	18 (1)	−0 (2)	0 (1)	6 (2)
N(3)	1851 (2)	3003 (5)	4545 (2)	27 (2)	148 (7)	22 (1)	3 (3)	−5 (1)	8 (2)
C(4)	2640 (3)	3406 (6)	4088 (2)	24 (2)	142 (8)	31 (8)	8 (3)	−2 (1)	13 (3)
O(4)	3441 (2)	3740 (5)	4376 (2)	30 (1)	258 (8)	35 (1)	−15 (3)	−6 (1)	17 (3)
C(5)	2497 (3)	3413 (7)	3304 (2)	32 (2)	195 (9)	25 (1)	1 (4)	5 (1)	5 (3)
C(6)	1612 (3)	3052 (6)	3021 (2)	32 (2)	162 (9)	20 (1)	10 (4)	4 (1)	0 (3)
C(1′)	−176 (3)	2373 (6)	3320 (2)	27 (2)	139 (8)	20 (1)	4 (3)	−2 (1)	−10 (3)
C(2′)	−587 (3)	1993 (5)	4089 (2)	24 (2)	122 (7)	22 (1)	−2 (3)	−2 (1)	0 (2)
C(3′)	−1375 (2)	3437 (5)	4203 (2)	23 (2)	111 (7)	19 (1)	−10 (3)	−2 (1)	−5 (2)
O(3′)	−2321 (2)	2696 (4)	4044 (1)	20 (1)	146 (5)	23 (1)	−13 (2)	1 (1)	−4 (2)
C(4′)	−1110 (2)	4884 (5)	3644 (2)	24 (2)	126 (7)	17 (1)	3 (3)	1 (1)	−2 (2)
C(5′)	−491 (3)	6355 (5)	3975 (2)	39 (2)	125 (7)	21 (1)	−14 (4)	−2 (1)	5 (2)
O(5′)	−187 (2)	7589 (4)	3431 (2)	47 (2)	179 (7)	26 (1)	−25 (3)	−4 (1)	24 (2)
O(1′)	−620 (2)	3933 (4)	3052 (1)	31 (1)	156 (5)	15 (1)	15 (2)	−1 (1)	−1 (2)
H(5)	305 (3)	356 (7)	302 (2)	3·2689					
H(6)	146 (3)	288 (6)	250 (2)	2·7867					
H(1′)	−25 (3)	142 (7)	296 (2)	2·5616					
H(2′)	−89 (3)	67 (6)	415 (2)	2·5741					
H(3′)	−134 (3)	385 (6)	472 (2)	2·2407					

	x	y	z	B
H(O3′)	−261 (3)	249 (6)	440 (2)	2·4553
H(4′)	−172 (3)	535 (6)	344 (2)	2·2829
H(5′)	6 (3)	587 (6)	424 (2)	2·5754
H(5′)	−83 (3)	685 (6)	433 (2)	2·5754
H(O5′)	27 (3)	651 (7)	346 (2)	3·5319

(2) Molecule *B*

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
N(1)	840 (2)	1088 (4)	−1801 (1)	29 (1)	131 (6)	18 (1)	1 (2)	1 (1)	−1 (2)
C(2)	1026 (2)	813 (5)	−1088 (2)	32 (2)	95 (6)	20 (1)	6 (3)	2 (1)	4 (2)
O(2)	211 (2)	331 (4)	−724 (1)	26 (1)	150 (5)	21 (1)	−2 (2)	−1 (1)	17 (2)
N(3)	1857 (2)	927 (4)	−757 (2)	31 (1)	159 (6)	21 (1)	−3 (3)	−2 (1)	14 (2)
C(4)	2651 (3)	1426 (6)	−1188 (2)	32 (2)	155 (7)	26 (1)	−5 (3)	1 (1)	2 (3)
O(4)	3469 (2)	1502 (5)	−909 (2)	30 (1)	309 (8)	34 (1)	−19 (3)	−4 (1)	17 (3)
C(5)	2486 (3)	1786 (6)	−1961 (2)	35 (2)	162 (8)	24 (1)	−4 (3)	8 (1)	6 (3)
C(6)	1597 (3)	1622 (5)	−2248 (2)	40 (2)	137 (7)	18 (1)	3 (3)	4 (1)	4 (2)
C(1′)	−204 (2)	969 (5)	−1966 (2)	28 (2)	104 (6)	21 (1)	7 (3)	−3 (1)	−3 (2)
C(2′)	−593 (2)	184 (5)	−1248 (2)	32 (2)	92 (6)	22 (1)	−2 (3)	−4 (1)	2 (2)
C(3′)	−1441 (2)	1409 (5)	−1050 (2)	23 (2)	120 (7)	24 (1)	−6 (3)	−1 (1)	7 (2)
O(3′)	−2287 (2)	826 (4)	−1431 (2)	30 (1)	156 (6)	40 (1)	−21 (2)	−9 (1)	14 (2)
C(4′)	−1142 (2)	3191 (5)	−1391 (2)	26 (2)	108 (6)	21 (1)	−2 (3)	1 (1)	4 (2)
C(5′)	−544 (3)	4369 (5)	−892 (2)	44 (2)	100 (6)	22 (1)	−7 (3)	2 (1)	0 (2)
O(5′)	−210 (2)	5904 (3)	−1279 (1)	46 (1)	101 (5)	28 (1)	−21 (2)	−5 (1)	6 (2)
O(1′)	−626 (2)	2696 (3)	−2055 (1)	36 (1)	100 (4)	17 (1)	3(2)	0 (1)	1 (1)
H(5)	303 (3)	214 (7)	−223 (2)	3·1029					
H(6)	144 (3)	160 (6)	−279 (2)	2·7625					
H(1′)	−33 (3)	31 (6)	−248 (2)	2·2732					
H(2′)	−81 (3)	−96 (6)	−130 (2)	2·5557					
H(3′)	−154 (3)	145 (6)	−53 (2)	2·4911					

	x	y	z	B
H(O3′)	−246 (3)	−35 (7)	−128 (3)	3·5007
H(4′)	−173 (3)	386 (6)	−155 (2)	2·4168
H(5′)	−106 (3)	456 (6)	−50 (2)	2·6741
H(5′)	4 (3)	366 (6)	−69 (2)	2·6741
H(O5′)	32 (3)	606 (6)	−109 (2)	3·1834

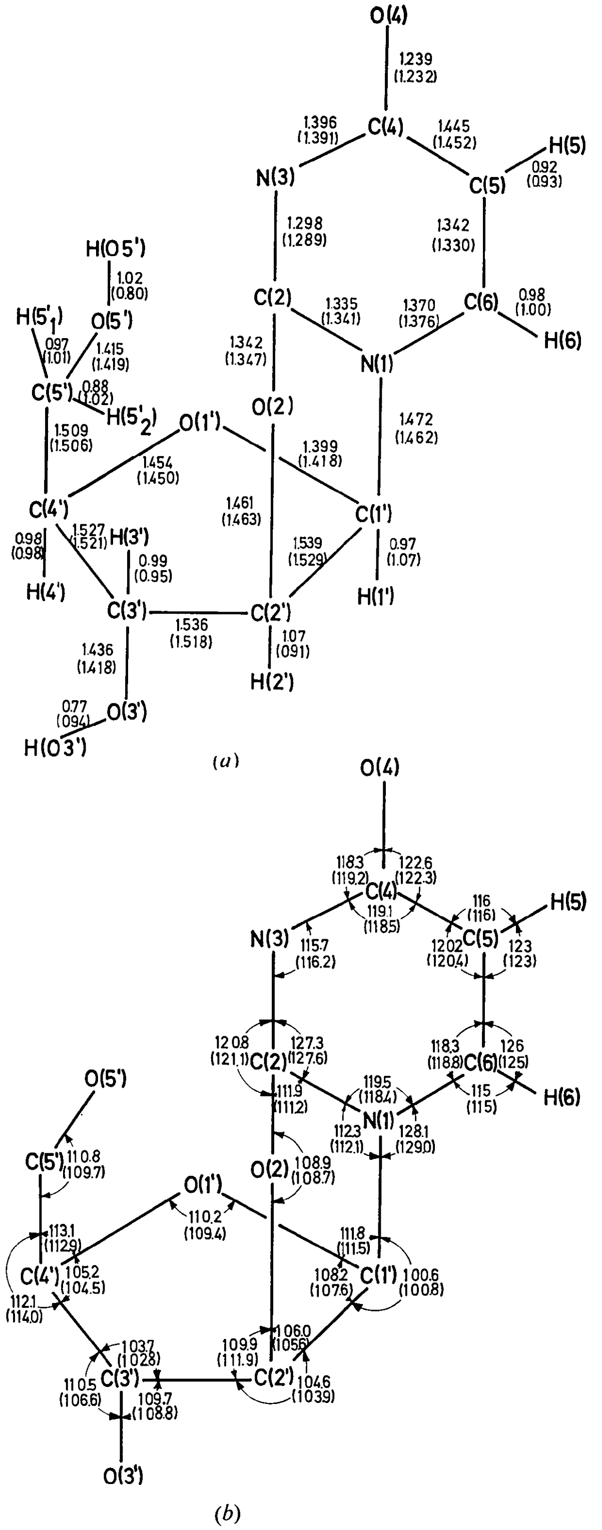


Fig. 1. Bond angles (*a*) and distances (*b*) in O^2,2′-cyclouridine. Data for molecule *B* are given in parentheses. The average estimated standard deviations (e.s.d.) are 0·005 Å and 0·3° for bonds and angles not involving hydrogen atoms. The hydrogen-containing bond angles within the sugar residues, not shown in the figure, are in the normal range, around 110°.

⅓ the estimated standard deviations. The final *R* index for the 1762 significant reflexions is 4·5%, and for all the 1831 data, 5·5%.

Results and discussion

In Tables 2, 3, 4 and 5 are listed the final atomic parameters, the deviations of some atoms from the least-

Table 3. *Deviations* (Å) *from least-squares planes through several parts of molecules A and B*

The plane-defining atoms are marked by *.

(*a*) Base

	I*a*	I*b*	II*a*	II*b*
N(1)	0·002*	0·013*	−0·028*	0·054*
C(2)	−0·004*	0·009*	0·009*	−0·008*
O(2)	0·014	0·011	0·015*	−0·043*
N(3)	0·001*	−0·001*	0·062	−0·006
C(4)	0·003*	0·007*	0·071	0·040
O(4)	0·006	0·059	0·120	0·087
C(5)	0·004*	−0·003*	0·020	0·073
C(6)	0·001*	−0·007*	−0·024	0·073
C(1′)	0·112	−0·109	0·033*	−0·073*
C(2′)	0·028	0·084	−0·029*	0·069*

(*b*) Sugar

	III*a*	III*b*	IV*a*	IV*b*	V*a*	V*b*
C(1′)	−0·024*	0·077*	0·075*	−0·046*	0·000*	0·000*
C(2′)	0·023*	−0·073*	−0·043*	0·027*	0·000*	0·000*
C(3′)	−0·014*	0·046*	−0·389	0·505	−0·109	0·339
O(3′)	−1·355	1·420	−1·805	1·919	−1·474	1·748
C(4′)	0·423	−0·476	0·046*	−0·029*	0·338	−0·208
C(5′)	1·903	−1·963	1·439	−1·415	1·804	−1·644
O(5′)	2·298	−2·380	1·857	−1·885	2·214	−2·110
O(1′)	0·016*	−0·051*	−0·078*	0·048*	0·000*	0·000*

Coefficients of plane equations in the form $lX+mY+nZ+p=0$ where X, Y and Z are along a, b, and c respectively

	l	m	n	p
I*a*	−0·2368	0·9707	0·0403	−1·902
I*b*	0·1878	−0·9605	−0·2052	−0·099
II*a*	−0·2095	0·9754	0·0683	−2·153
II*b*	0·1974	−0·9515	−0·2361	−0·178
III*a*	0·6938	0·5788	0·4286	−3·475
III*b*	−0·6561	−0·4692	−0·5911	−1·887
IV*a*	0·8293	0·4447	0·3384	−2·560
IV*b*	−0·8265	−0·3534	−0·4382	−1·593
V*a*	0·7292	0·5523	0·4041	−3·247
V*b*	−0·7674	−0·4078	−0·4948	−1·694

Table 4. *Dihedral angles defining the conformation of the sugar residues in* O^2,*2′-cyclouridine*

	Molecule *A*	Molecule *B*
C(6) −N(1) −C(1′)−C(2′)	179·3	176·6°
C(1′)−C(2′)−C(3′)−C(4′)	−19·7	−28·7
C(2′)−C(3′)−C(4′)−O(1′)	+28·3	+34·4
C(3′)−C(4′)−O(1′)−C(1′)	−27·2	−27·3
C(4′)−O(1′)−C(1′)−C(2′)	+14·4	+8·7
O(1′)−C(1′)−C(2′)−C(3′)	+4·2	+13·3
O(5′)−C(5′)−C(4′)−O(1′)	+56·1	+54·6
O(5′)−C(5′)−C(4′)−C(3′)	+174·9	+173·7
O(2)−C(2′)−C(3′)−O(3′)	−148·3	−162·5
C(1′)−C(2′)−C(3′)−O(3′)	+98·3	+84·1
O(3′)−C(3′)−C(4′)−C(5′)	+147·6	+156·3
O(3′)−C(3′)−C(4′)−O(1′)	−89·1	+80·0

squares planes through several parts of the two molecules within the asymmetric unit, the dihedral angles describing the conformation of the sugar moieties and the observed and calculated structure factors. Bond angles and distances within the two molecules are presented in Fig. 1. Figs. 2 and 3 represent projections

Table 5. *Observed and calculated structure factors* (× 10) *for* O^2,2′-*cyclouridine*

H	K	L	FOBS	FCAL
2	0	0	797	849
4	0	0	627	675
6	0	0	111	115
8	0	0	272	280
10	0	0	875	842
12	0	0	226	259
14	0	0	98	93
16	0	0	34	29
1	1	0	412	413
2	1	0	100	177
3	1	0	962	903
4	1	0	143	157
5	1	0	111	173
6	1	0	8	4
7	1	0	93	94
8	1	0	62	66
9	1	0	17	22
10	1	0	225	237
11	1	0	171	173
12	1	0	688	667
13	1	0	91	91
14	1	0	78	73
15	1	0	52	51
0	2	0	1132	1174
1	2	0	704	778
2	2	0	278	259
3	2	0	750	749
4	2	0	151	150
5	2	0	510	491
6	2	0	31	20
7	2	0	176	176
8	2	0	154	145
9	2	0	137	133
10	2	0	147	135
11	2	0	67	53
12	2	0	191	196
13	2	0	67	58
14	2	0	16	9
15	2	0	121	110
1	3	0	72	71
2	3	0	759	737
3	3	0	137	128
4	3	0	197	191
5	3	0	150	133
6	3	0	45	50
7	3	0	88	47
8	3	0	140	145
9	3	0	0	1
10	3	0	57	42
11	3	0	127	129
12	3	0	93	90
13	3	0	147	150
14	3	0	86	81
15	3	0	12	18
0	4	0	231	234
1	4	0	890	885
2	4	0	577	577
3	4	0	278	263
4	4	0	252	237
5	4	0	181	168
6	4	0	112	111
7	4	0	24	47
8	4	0	31	23
9	4	0	220	216
10	4	0	110	112
11	4	0	135	132
12	4	0	64	55
13	4	0	25	16
14	4	0	61	57
1	5	0	75	72
2	5	0	235	227
3	5	0	122	111
4	5	0	116	121
5	5	0	120	125
6	5	0	207	200
7	5	0	231	231
8	5	0	97	93
9	5	0	268	261
10	5	0	155	152
11	5	0	92	97
12	5	0	127	128
13	5	0	33	29
0	6	0	238	271
1	6	0	139	143
2	6	0	390	396
3	6	0	783	777
4	6	0	765	755
5	6	0	83	81
6	6	0	29	25
7	6	0	23	26
8	6	0	108	97
9	6	0	32	30
10	6	0	283	277
11	6	0	70	77
1	7	0	38	37
2	7	0	115	117
3	7	0	0	16
4	7	0	157	150
5	7	0	97	77
6	7	0	96	94
7	7	0	8	9
8	7	0	110	111
9	7	0	23	23
0	8	0	197	198
1	8	0	0	9
2	8	0	127	122
3	8	0	28	27
4	8	0	0	6
5	8	0	39	43
6	8	0	19	22
1	0	1	136	137
2	0	1	45	52
3	0	1	59	64
4	0	1	0	12
5	0	1	78	24
6	0	1	0	28
7	0	1	82	53
8	0	1	28	27
9	0	1	0	7
10	0	1	35	37
11	0	1	0	13
12	0	1	25	47
13	0	1	0	7
14	0	1	8	7
15	0	1	0	7
16	0	1	27	29
0	1	1	573	596
1	1	1	570	580
2	1	1	175	177
3	1	1	418	477
4	1	1	252	240
5	1	1	58	58
6	1	1	111	125
7	1	1	168	167
8	1	1	68	71
9	1	1	120	127
10	1	1	131	122
11	1	1	90	92
12	1	1	258	259
13	1	1	93	95
14	1	1	47	48
15	1	1	23	25
0	2	1	1275	1964
1	2	1	1085	1257
2	2	1	730	787
3	2	1	418	383
4	2	1	177	161
5	2	1	510	478
6	2	1	268	251
7	2	1	91	83
8	2	1	276	216
9	2	1	210	199
10	2	1	233	224
11	2	1	179	177
12	2	1	427	433
13	2	1	157	156
14	2	1	43	53
15	2	1	185	182
0	3	1	981	1069
1	3	1	724	708
2	3	1	272	284
3	3	1	840	845
4	3	1	245	229
5	3	1	173	114
6	3	1	150	183
7	3	1	73	84
8	3	1	47	66
9	3	1	297	280
10	3	1	149	164
11	3	1	44	68
12	3	1	125	126
13	3	1	75	81
14	3	1	57	91
15	3	1	71	88
0	4	1	95	101
1	4	1	430	406
2	4	1	644	637
3	4	1	157	156
4	4	1	134	177
5	4	1	190	167
6	4	1	54	57
7	4	1	215	210
8	4	1	94	99
9	4	1	190	197
10	4	1	178	179
11	4	1	192	187
12	4	1	180	183
13	4	1	22	36
14	4	1	59	84
0	5	1	48	55
1	5	1	97	92
2	5	1	145	153
3	5	1	217	209
4	5	1	44	58
5	5	1	90	94
6	5	1	109	107
7	5	1	99	97
8	5	1	140	139
9	5	1	97	83
10	5	1	47	62
11	5	1	113	109
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7	0	8	104	120
8	0	8	210	217
9	0	8	136	136
10	0	8	91	90
11	0	8	218	225
12	0	8	189	192
13	0	8	221	225
14	0	8	24	17
0	1	8	224	219
1	1	8	198	161
2	1	8	108	75
3	1	8	117	112
4	1	8	180	192
5	1	8	270	294
6	1	8	268	262
7	1	8	170	195
8	1	8	285	236
9	1	8	181	145
10	1	8	113	108
11	1	8	179	190
12	1	8	134	132
13	1	8	127	130
14	1	8	78	90
0	2	8	485	484
1	2	8	297	332
2	2	8	39	7
3	2	8	130	87
4	2	8	152	188
5	2	8	268	244
6	2	8	182	177
7	2	8	58	60
8	2	8	171	177
9	2	8	140	148
10	2	8	39	35
11	2	8	123	123
12	2	8	38	32
13	2	8	47	40
14	2	8	41	37
0	3	8	29	42
1	3	8	202	209
2	3	8	261	249
3	3	8	98	98
4	3	8	181	177
5	3	8	50	45
6	3	8	81	83
7	3	8	229	224
8	3	8	169	175
9	3	8	97	100
10	3	8	77	81
11	3	8	177	177
12	3	8	92	103
13	3	8	115	119
0	4	8	43	40
1	4	8	174	177
2	4	8	146	148
3	4	8	237	241
4	4	8	234	242
5	4	8	95	91
6	4	8	100	110
7	4	8	111	117
8	4	8	130	140
9	4	8	143	140
10	4	8	61	54
11	4	8	147	147
12	4	8	39	50
0	5	8	17	36
1	5	8	168	165
2	5	8	53	51
3	5	8	73	70
4	5	8	52	59
5	5	8	131	135
6	5	8	118	115
7	5	8	181	182
8	5	8	134	132
9	5	8	174	179
10	5	8	27	30
11	5	8	66	63
0	6	8	110	110
1	6	8	62	59
2	6	8	160	152
3	6	8	133	136
4	6	8	69	68
5	6	8	75	73
6	6	8	80	81
7	6	8	76	71
8	6	8	124	99
9	6	8	55	53
10	6	8	25	32
0	7	8	123	105
1	7	8	5	21
2	7	8	74	84
3	7	8	76	80
4	7	8	130	129
5	7	8	67	65
6	7	8	85	88
7	7	8	51	40
0	8	8	3	4
1	8	8	54	57
2	8	8	69	67
1	0	9	153	150
2	0	9	145	144
3	0	9	337	348
4	0	9	121	117
5	0	9	205	199
6	0	9	30	30
7	0	9	25	27
8	0	9	332	336
9	0	9	51	56

Table 5 (*cont.*)

H	K	L	FOBS	FCAL
10	0	9	31	28
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13	0	9	105	101
14	0	9	64	59
0	1	9	553	552
1	1	9	368	370
2	1	9	95	90
3	1	9	144	146
4	1	9	209	205
5	1	9	224	218
6	1	9	157	149
7	1	9	88	91
8	1	9	172	179
9	1	9	80	86
10	1	9	111	115
11	1	9	40	40
12	1	9	81	87
13	1	9	22	19
14	1	9	21	22
0	2	9	473	492
1	2	9	74	68
2	2	9	200	190
3	2	9	307	289
4	2	9	258	253
5	2	9	168	169
6	2	9	180	168
7	2	9	137	130
8	2	9	111	111
9	2	9	96	101
10	2	9	74	77
11	2	9	24	15
12	2	9	92	94
13	2	9	102	103
14	2	9	20	18
0	3	9	107	105
1	3	9	88	86
2	3	9	416	421
3	3	9	63	56
4	3	9	103	96
5	3	9	149	153
6	3	9	69	63
7	3	9	230	229
8	3	9	128	129
9	3	9	211	215
10	3	9	203	212
11	3	9	141	134
12	3	9	28	26
13	3	9	96	91
0	4	9	207	204
1	4	9	264	273
2	4	9	259	267
3	4	9	201	198
4	4	9	167	179
5	4	9	57	57
6	4	9	127	128
7	4	9	177	171
8	4	9	282	281
9	4	9	100	98
10	4	9	96	94
11	4	9	38	39
12	4	9	40	42
0	5	9	138	150
1	5	9	34	39
2	5	9	193	188
3	5	9	201	222
4	5	9	251	252
5	5	9	180	180
6	5	9	148	156
7	5	9	66	61
8	5	9	223	219
9	5	9	61	44
10	5	9	148	139
11	5	9	30	34
0	6	9	117	124
1	6	9	137	132
2	6	9	132	95
3	6	9	48	51
4	6	9	95	90
5	6	9	57	67
6	6	9	106	110
7	6	9	30	37
8	6	9	192	191
9	6	9	145	143
0	7	9	0	7
1	7	9	74	41
2	7	9	119	115
3	7	9	66	67
4	7	9	4	18
5	7	9	0	10
6	7	9	0	11
0	0	10	404	403
1	0	10	356	381
2	0	10	48	50
3	0	10	164	155
4	0	10	31	39
5	0	10	48	40
6	0	10	367	355
7	0	10	116	116
8	0	10	165	171
9	0	10	71	72
10	0	10	413	422
11	0	10	0	3
12	0	10	0	10
13	0	10	108	111
14	0	10	107	103
0	1	10	25	26
1	1	10	292	291
2	1	10	487	478
3	1	10	183	160
4	1	10	256	243
5	1	10	196	195
6	1	10	410	416
7	1	10	154	148
8	1	10	63	61
9	1	10	91	97
10	1	10	232	240
11	1	10	161	160
12	1	10	97	95
13	1	10	65	65
14	1	10	88	87
0	2	10	41	21
1	2	10	114	114
2	2	10	432	431
3	2	10	130	95
4	2	10	18	2
5	2	10	184	181
6	2	10	282	281
7	2	10	247	247
8	2	10	40	34
9	2	10	186	188
10	2	10	214	221
11	2	10	140	140
12	2	10	60	66
13	2	10	155	146
0	3	10	222	219
1	3	10	302	313
2	3	10	366	335
3	3	10	220	223
4	3	10	72	68
5	3	10	193	202
6	3	10	158	155
7	3	10	29	25
8	3	10	297	298
9	3	10	87	78
10	3	10	45	47
11	3	10	70	82
12	3	10	46	45
13	3	10	48	48
0	4	10	353	350
1	4	10	77	77
2	4	10	318	325
3	4	10	123	122
4	4	10	129	128
5	4	10	44	39
6	4	10	179	180
7	4	10	158	167
8	4	10	111	113
9	4	10	68	70
10	4	10	36	42
11	4	10	58	51
12	4	10	60	42
0	5	10	47	41
1	5	10	92	92
2	5	10	56	69
3	5	10	156	150
4	5	10	197	200
5	5	10	72	75
6	5	10	150	168
7	5	10	88	92
8	5	10	67	68
9	5	10	0	18
10	5	10	31	36
0	6	10	15	26
1	6	10	45	48
2	6	10	133	142
3	6	10	95	96
4	6	10	110	108
5	6	10	135	122
6	6	10	86	84
7	6	10	43	41
8	6	10	41	78
0	7	10	90	92
1	7	10	117	121
2	7	10	70	68
3	7	10	47	48
4	7	10	43	80
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6	7	10	40	51
1	0	11	257	252
2	0	11	336	338
3	0	11	215	234
4	0	11	301	302
5	0	11	259	257
6	0	11	491	489
7	0	11	30	42
8	0	11	203	211
9	0	11	117	111
10	0	11	122	125
11	0	11	95	94
12	0	11	47	3
13	0	11	33	36
0	1	11	55	43
1	1	11	240	236
2	1	11	203	278
3	1	11	137	103
4	1	11	195	194
5	1	11	170	180
6	1	11	316	306
7	1	11	305	316
8	1	11	155	166
9	1	11	21	22
10	1	11	37	103
11	1	11	75	81
12	1	11	30	35
13	1	11	123	101
0	2	11	92	92
1	2	11	368	349
2	2	11	153	152
3	2	11	154	154
4	2	11	43	93
5	2	11	135	312
6	2	11	115	114
7	2	11	32	29
8	2	11	90	90
9	2	11	53	52
10	2	11	99	104
11	2	11	67	55
12	2	11	44	45
13	2	11	53	58
0	3	11	270	270
1	3	11	196	193
2	3	11	149	145
3	3	11	73	76
4	3	11	46	50
5	3	11	172	185
6	3	11	27	29
7	3	11	0	10
8	3	11	122	124
9	3	11	96	95
10	3	11	44	43
11	3	11	115	116
12	3	11	18	22
0	4	11	47	46
1	4	11	249	250
2	4	11	206	215
3	4	11	60	59
4	4	11	133	134
5	4	11	104	103
6	4	11	117	127
7	4	11	192	198
8	4	11	176	170
9	4	11	121	123
10	4	11	0	18
11	4	11	73	74
0	5	11	81	88
1	5	11	171	177
2	5	11	35	45
3	5	11	79	81
4	5	11	51	50
5	5	11	130	133
6	5	11	43	43
7	5	11	96	97
8	5	11	78	74
9	5	11	120	127
10	5	11	85	88
0	6	11	76	73
1	6	11	145	145
2	6	11	130	126
3	6	11	149	156
4	6	11	63	66
5	6	11	40	36
6	6	11	24	32
7	6	11	70	72
8	6	11	10	13
0	7	11	99	90
1	7	11	22	28
2	7	11	68	68
3	7	11	79	80
4	7	11	55	58
0	0	12	72	7
1	0	12	0	12
2	0	12	112	113
3	0	12	226	222
4	0	12	37	21
5	0	12	67	67
6	0	12	0	5
7	0	12	364	366
8	0	12	110	113
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11	0	12	115	132
12	0	12	32	34
13	0	12	94	93
0	1	12	88	80
1	1	12	126	132
2	1	12	27	36
3	1	12	148	154
4	1	12	368	369
5	1	12	113	114
6	1	12	68	48
7	1	12	211	213
8	1	12	268	272
9	1	12	65	76
10	1	12	134	138
11	1	12	57	54
12	1	12	47	47
13	1	12	44	23
0	2	12	95	95
1	2	12	126	126
2	2	12	143	153
3	2	12	95	92
4	2	12	295	309
5	2	12	18	20
6	2	12	160	168
7	2	12	263	272
8	2	12	369	376
9	2	12	147	151
10	2	12	51	56
11	2	12	64	57
12	2	12	7	31
0	3	12	21	92
1	3	12	132	132
2	3	12	24	25
3	3	12	22	27
4	3	12	154	156
5	3	12	662	670
6	3	12	135	138
7	3	12	62	39
8	3	12	171	102
9	3	12	92	97
10	3	12	23	32
11	3	12	135	131
12	3	12	77	27
0	4	12	0	23
1	4	12	95	101
2	4	12	196	186
3	4	12	125	115
4	4	12	168	166
5	4	12	118	118
6	4	12	66	58
7	4	12	214	212
8	4	12	115	112
9	4	12	123	106
10	4	12	92	85
11	4	12	11	17
0	5	12	0	5
1	5	12	59	57
2	5	12	115	122
3	5	12	71	74
4	5	12	32	24
5	5	12	56	74
6	5	12	143	132
7	5	12	67	67
8	5	12	57	62
9	5	12	0	4
0	6	12	11	19
1	6	12	64	71
2	6	12	55	63
3	6	12	29	23
4	6	12	90	95
5	6	12	78	70
6	6	12	31	33
7	6	12	105	95
0	7	12	27	23
1	7	12	127	123
2	7	12	122	113
3	7	12	163	162
1	0	13	309	315
2	0	13	132	129
3	0	13	105	120
4	0	13	229	232
5	0	13	226	225
6	0	13	220	214
7	0	13	74	73
8	0	13	458	457
9	0	13	44	51
10	0	13	173	175
11	0	13	120	118
12	0	13	64	64
0	1	13	0	20
1	1	13	221	219
2	1	13	108	113
3	1	13	176	176
4	1	13	187	183
5	1	13	108	196
6	1	13	301	407
7	1	13	95	99
8	1	13	183	182
9	1	13	139	139
10	1	13	67	67
11	1	13	21	15
12	1	13	36	30
0	2	13	77	84
1	2	13	192	191
2	2	13	145	147
3	2	13	159	163
4	2	13	167	171
5	2	13	105	109
6	2	13	195	194
7	2	13	211	213
8	2	13	192	188
9	2	13	66	59
10	2	13	62	54
11	2	13	47	52
12	2	13	65	65
0	3	13	73	64
1	3	13	116	109
2	3	13	210	222
3	3	13	79	77
4	3	13	42	48
5	3	13	212	219
6	3	13	141	155
7	3	13	122	125
8	3	13	132	141
9	3	13	70	72
10	3	13	50	49
11	3	13	29	29
0	4	13	141	170
1	4	13	51	67
2	4	13	0	5
3	4	13	104	115
4	4	13	220	226
5	4	13	67	69
6	4	13	177	180
7	4	13	212	209
8	4	13	141	137
9	4	13	52	55
10	4	13	45	50
0	5	13	93	104
1	5	13	152	160
2	5	13	77	72
3	5	13	187	196
4	5	13	202	198
5	5	13	192	193
6	5	13	88	87
7	5	13	31	30
8	5	13	140	138
0	6	13	51	53
1	6	13	43	40
2	6	13	146	148
3	6	13	92	87
4	6	13	47	41
5	6	13	143	137
6	6	13	76	78
0	0	14	124	128
1	0	14	32	31
2	0	14	15	19
3	0	14	236	241
4	0	14	157	163
5	0	14	32	30
6	0	14	78	80
7	0	14	66	64
8	0	14	0	13
9	0	14	81	85
10	0	14	72	76
11	0	14	8	4
12	0	14	31	30
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1	1	14	81	81
2	1	14	62	67
3	1	14	58	65
4	1	14	175	182
5	1	14	129	131
6	1	14	160	165
7	1	14	102	104
8	1	14	30	74
9	1	14	108	110
10	1	14	44	50
11	1	14	48	79
12	1	14	0	9
0	2	14	74	69
1	2	14	170	178
2	2	14	50	54
3	2	14	82	80
4	2	14	316	318
5	2	14	275	294
6	2	14	179	175
7	2	14	128	131
8	2	14	95	96
9	2	14	131	138
10	2	14	114	118
11	2	14	61	66
0	3	14	58	56
1	3	14	51	52
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8	3	14	315	306
9	3	14	65	62
10	3	14	60	55
0	4	14	138	136
1	4	14	135	141
2	4	14	120	125
3	4	14	110	112
4	4	14	232	222
5	4	14	48	28
6	4	14	210	204
7	4	14	126	123
8	4	14	51	63
9	4	14	114	114
0	5	14	22	20
1	5	14	98	98
2	5	14	46	42
3	5	14	37	26
4	5	14	64	73
5	5	14	15	21
6	5	14	53	57
7	5	14	105	90
0	6	14	0	12
1	6	14	24	23
2	6	14	68	69
3	6	14	40	36
4	6	14	94	95
5	6	14	37	33
1	0	15	127	131
2	0	15	34	37
3	0	15	0	3
4	0	15	104	107
5	0	15	283	285
6	0	15	127	120
7	0	15	77	72
8	0	15	94	91
9	0	15	33	44
10	0	15	63	75
11	0	15	57	53
0	1	15	121	119
1	1	15	112	121
2	1	15	64	67
3	1	15	143	155
4	1	15	338	332
5	1	15	235	236
6	1	15	220	210
7	1	15	224	226
8	1	15	137	135
9	1	15	81	82
10	1	15	46	43
11	1	15	32	33
0	2	15	114	114
1	2	15	61	57
2	2	15	105	127
3	2	15	112	119
4	2	15	71	75
5	2	15	205	202
6	2	15	137	130
7	2	15	41	47
8	2	15	69	67
9	2	15	40	43
10	2	15	41	30
0	3	15	252	256
1	3	15	171	171
2	3	15	132	133
3	3	15	65	59
4	3	15	40	45
5	3	15	119	116
6	3	15	88	80
7	3	15	18	16
8	3	15	53	53
9	3	15	22	11
10	3	15	84	79
0	4	15	76	76
1	4	15	75	75
2	4	15	146	146
3	4	15	93	93
4	4	15	69	69
5	4	15	52	57
6	4	15	0	22
7	4	15	144	143
8	4	15	30	38
0	5	15	130	128
1	5	15	94	86
2	5	15	97	104
3	5	15	140	138
4	5	15	168	159
5	5	15	70	74
6	5	15	114	105
0	6	15	27	24
1	6	15	59	55
2	6	15	41	46
3	6	15	27	28
0	0	16	92	98
1	0	16	130	133
2	0	16	40	32
3	0	16	130	143
4	0	16	37	36
5	0	16	122	123
6	0	16	0	9
7	0	16	73	74
8	0	16	0	10
9	0	16	0	13
10	0	16	62	52
0	1	16	69	72
1	1	16	116	119
2	1	16	67	72
3	1	16	42	35
4	1	16	245	247
5	1	16	179	175
6	1	16	90	92
7	1	16	148	146
8	1	16	65	57
9	1	16	26	27
10	1	16	21	20
0	2	16	345	350
1	2	16	59	68
2	2	16	193	204
3	2	16	39	44
4	2	16	109	108
5	2	16	40	32
6	2	16	40	43
7	2	16	173	165
8	2	16	47	51
9	2	16	0	9
10	2	16	0	12
0	3	16	101	110
1	3	16	15	28
2	3	16	60	87
3	3	16	86	82
4	3	16	120	124
5	3	16	220	220
6	3	16	72	69
7	3	16	76	77
8	3	16	55	53
9	3	16	49	44
0	4	16	147	152
1	4	16	147	145
2	4	16	98	95
3	4	16	77	79
4	4	16	119	108
5	4	16	71	74
6	4	16	47	44
7	4	16	70	74
0	5	16	45	46
1	5	16	31	31
2	5	16	0	10
3	5	16	35	37
4	5	16	47	47
5	5	16	33	36
1	0	17	212	209
2	0	17	162	184
3	0	17	293	285
4	0	17	98	90
5	0	17	327	312
6	0	17	77	79
7	0	17	0	0
8	0	17	50	40
9	0	17	32	31
0	1	17	53	56
1	1	17	74	73
2	1	17	65	64
3	1	17	43	36
4	1	17	80	86
5	1	17	151	145
6	1	17	128	120
7	1	17	103	112
8	1	17	59	64
9	1	17	65	59
0	2	17	179	176
1	2	17	58	60
2	2	17	191	192
3	2	17	62	58
4	2	17	153	150
5	2	17	14	16
6	2	17	53	43
7	2	17	64	60
8	2	17	60	64
9	2	17	76	71
0	3	17	29	30
1	3	17	70	77
2	3	17	76	72
3	3	17	49	47
4	3	17	60	60
5	3	17	33	33
6	3	17	53	49
7	3	17	0	15
8	3	17	62	58
0	4	17	120	119
1	4	17	93	98
2	4	17	0	7
3	4	17	84	89
4	4	17	77	71
5	4	17	72	71
6	4	17	85	79
0	5	17	25	31
1	5	17	79	72
2	5	17	26	22
3	5	17	60	57
0	0	18	254	242
1	0	18	94	87
2	0	18	70	77
3	0	18	0	16
4	0	18	23	35
5	0	18	128	128
6	0	18	102	103
7	0	18	0	9
8	0	18	8	10
0	1	18	84	89
1	1	18	111	114
2	1	18	102	93
3	1	18	76	69
4	1	18	81	80
5	1	18	42	37
6	1	18	67	70
7	1	18	78	74
8	1	18	116	107
0	2	18	29	24
1	2	18	157	159
2	2	18	151	142
3	2	18	121	111
4	2	18	128	124
5	2	18	161	150
6	2	18	103	99
7	2	18	93	96
0	3	18	15	19
1	3	18	107	107
2	3	18	135	135
3	3	18	27	34
4	3	18	123	123
5	3	18	37	27
6	3	18	75	75
0	4	18	21	32
1	4	18	63	62
2	4	18	62	55
3	4	18	88	95
4	4	18	95	91
1	0	19	209	209
2	0	19	99	89
3	0	19	96	88
4	0	19	50	50
5	0	19	17	11
6	0	19	35	45
7	0	19	22	13
0	1	19	63	54
1	1	19	79	84
2	1	19	114	121
3	1	19	102	106
4	1	19	31	38
5	1	19	20	12
6	1	19	23	26
7	1	19	112	103
0	2	19	33	35
1	2	19	18	14
2	2	19	38	35
3	2	19	68	64
4	2	19	50	43
5	2	19	65	63
6	2	19	24	25
0	3	19	0	18
1	3	19	60	51
2	3	19	30	29
3	3	19	97	90
4	3	19	24	20
5	3	19	58	53
0	4	19	28	19
1	4	19	6	9
0	0	20	98	93
1	0	20	60	56
2	0	20	0	8
3	0	20	41	30
4	0	20	32	30
5	0	20	16	21
0	1	20	99	78
1	1	20	126	121
2	1	20	54	58
3	1	20	33	39
4	1	20	47	52
5	1	20	72	68
0	2	20	176	172
1	2	20	74	77
2	2	20	53	49
3	2	20	90	91
4	2	20	84	78
5	2	20	30	26
1	3	20	55	58
1	0	21	9	13
2	0	21	46	41
3	0	21	51	48
0	1	21	58	42
1	1	21	75	69

of the crystal structure along the *b* and *c* axes respectively.

(*a*) *Conformation of the molecules*

The uracil ring systems as well as the five-membered rings consisting of atoms N(1), C(2), O(2), C(2′) and C(1′) are essentially planar (Table 3). The interplanar angle between the six- and five-membered rings is about 2° in both molecules. The puckering of the five-membered sugar rings consisting of atoms C(1′), C(2′), C(3′), C(4′) and O(1′) is somewhat different in molecules *A* and *B*, which is obvious from the deviations of the atoms from the four-atom least-squares planes (Table 3) and the endocyclic dihedral angles (Table 4). The best four-atom plane in molecule *A* is comprised by atoms C(1′), C(2′), C(3′) and O(1′) with atom C(4′) being displaced by 0·423 Å to the same side of this plane as C(5′) (plane III*a* of Table 3). The C(1′)–O(1′) and C(2′)–C(3′) bonds are nearly coplanar, the dihedral angle O(1′)–C(1′)–C(2′)–C(3′) being only 4·2°. Thus the conformation of the sugar moiety of molecule *A* is C(4′)-*endo*. The sugar ring of molecule *B* however shows a twist conformation rather than an envelope form and should be termed C(4′)-*endo*-C(3′)-*exo* (planes III*b*, IV*b*, V*b* of Table 3). Most of the nucleosides so far investigated exhibit a C(2′)-*endo* or C(3′)-*endo* sugar puckering. The unusual puckering modes found in this crystal structure have to be attributed to the cyclization between atoms C(2′) and O(2) holding C(2′) and C(1′) in a rather rigid arrangement. A similar geometric situation due to a cyclization through a five-membered ring occurs in the 2′,3′-

cyclophosphates (Saenger & Eckstein, 1970; Coulter & Greaves, 1970); the O(1′)-*endo* conformation found in one of the two molecules of sodium cytidine-2′,3′-cyclophosphate (Coulter & Greaves, 1970) corresponds to the C(4′)-*endo* conformation of molecule *A* of O^2,2′-cyclouridine.

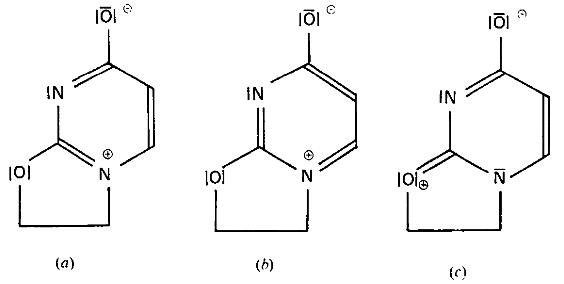
The orientation of the C(5′)–O(5′) bond with respect to bonds C(4′)–O(1′) and C(4′)–C(3′) is *gauche–trans* in molecules *A* and *B*; the corresponding dihedral angles are near 60° and 180° respectively (Table 4). The most common conformation around the C(4′)–C(5′) bond in nucleosides is *gauche–gauche* (Sundaralingam, 1965; Shefter & Trueblood, 1965). It cannot be decided from this analysis if the *gauche–trans* conformation found in O^2,2′-cyclouridine is due to hydrogen bonding requirements or if it is due to unfavourable short contacts between O(5′) and the heterocyclic ring system in case of a *gauche–gauche* conformation.

(*b*) *Bond angles and distances*

Comparable bond distances and angles within molecules *A* and *B*, with only a few exceptions, show no significant differences (Fig. 1). The maximum difference found for the O(3′)–C(3′)–C(4′) bond angle (3·9°) could be attributed to the different sugar puckering.

The cyclization between atoms O(2) and C(2′) results in a considerable alteration of the geometry of the base compared to normal uracil or uridine derivatives. The most prominent difference within the uracil moiety is the formation of the double bond in O^2,2′-cyclouridine between atoms C(2) and N(3) of length[*] 1·293 Å and the relatively short C(2)–N(1) distance, 1·338 Å, while in normal uracil derivatives these bonds were found to be 1·381 (22) Å and 1·374 (19) Å respectively (Voet & Rich, 1970).

The remaining bonds within the uracil moiety however are not changed significantly. This is somewhat unexpected because from the possible resonance forms of O^2,2′-cyclouridine one would expect the bonds N(1)–C(2) and N(3)–C(4) to have about equal amounts of double-bond character and the C(4)–O(4) bond to show partial single-bond character. Although the resonance forms (*a*) and (*b*) result in an aromatic system, their contributions are apparently small.



(*a*) (*b*) (*c*)

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[*] Throughout the following discussion averaged data for both O^2,2′-cyclouridine molecules are used.

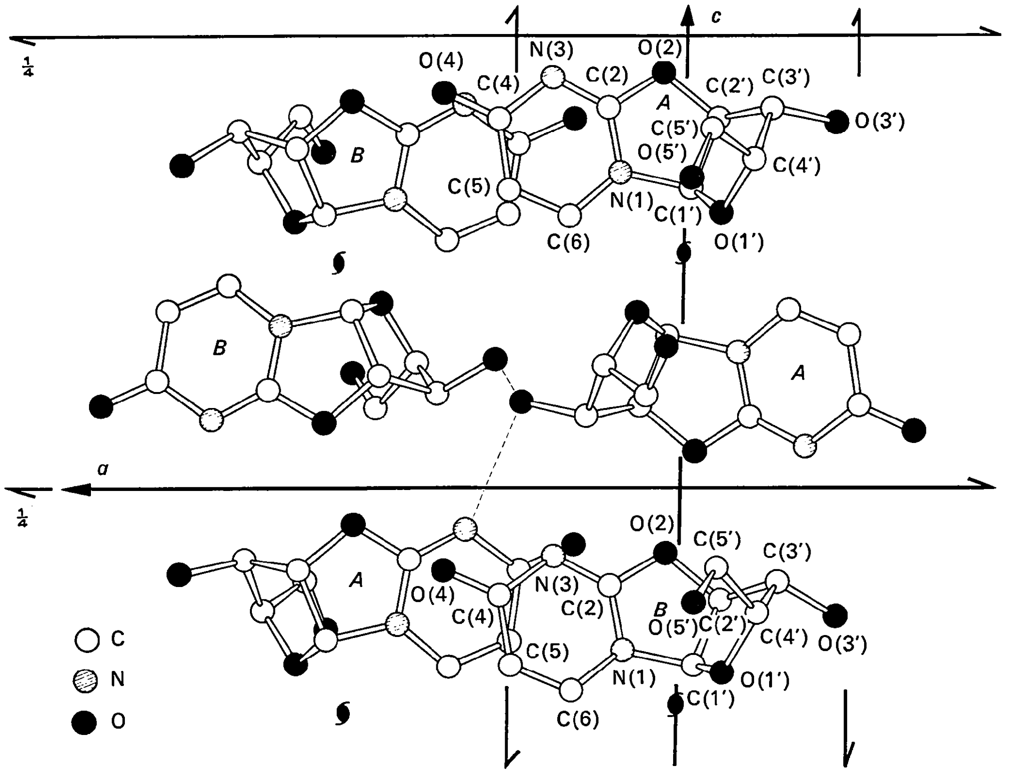

Fig. 2. Projection of the crystal structure along the *b* axis.

The C(2)–O(2) bond (1·344 Å) is significantly shorter than a C–O single bond [1·426 (5) Å; Sutton, 1965) and resembles the average C–O bond length in aromatic phenols or ethers [1·36 (1) Å; Sutton, 1965], whereas the O(2)–C(2′) bond (1·462 Å) is even longer than the average C–O single bond. Kartha, Ahmed & Barnes (1962) reported similar aromatic and aliphatic C–O bond distances of 1·37 (2) Å and 1·47 (2) Å for the five-membered cyclic ether within codeine hydrobromide dihydrate.

As one would expect, the bond angles at N(1), C(2) and C(1′) are significantly different from those observed in uridine. The angles N(1)–C(2)–N(3), C(6)–N(1)–C(1′) and N(3)–C(4)–C(5) are increased by 12·0, 7·6 and 4·7° respectively, whereas the angles N(1)–C(2)–O(2), C(1′)–N(1)–C(2), N(1)–C(1′)–C(2′) are decreased by 11·4, 5·0 and 12·9° respectively, compared with the average values for uridine derivatives (Voet & Rich, 1970). The decrease of the latter bond angles enables bond formation between atoms O(2) and C(2′). The decrease of the C(2)–N(3)–C(4) bond angle of 10·5° is consistent with the observation, that the endocyclic angle at a nitrogen atom within a planar six-membered ring is about 10° smaller if there is no exocyclic bond attached to it (Singh, 1965; Sundaralingam & Jensen, 1965).

The bond distances within the sugar moieties are in general not affected by the formation of the additional 5-membered ring and agree well with data found for other ribose (Saenger & Eckstein, 1970) or arabinose (Saenger, 1972) residues. Much more affected are the bond angles, especially at C(1′) (discussed above) and C(2′) but also at C(3′) and C(4′). While the differences between the angles at C(1′) and C(2′) in O^2,2-cyclouridine and in normal ribose and arabinose residues should be considered to be a direct consequence of cyclization, differences between the corresponding exocyclic angles at C(3′) and C(4′) could be attributed to the unusual sugar puckering in O^2,2′-cyclouridine.

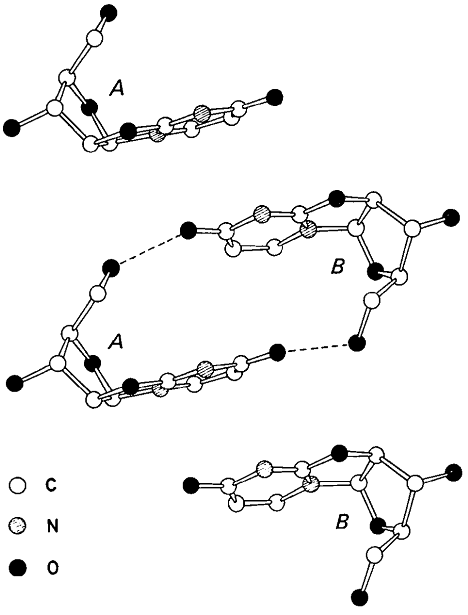

Fig. 3. Stacking of molecules along **b**; projection approximately along the *c* axis.

(*c*) *Packing arrangement*

The packing of the O^2,2′-cyclouridine molecules within the crystal is depicted in Figs. 2 and 3 which represent projections of the crystal structure along the *b* and *c* axes respectively. The base planes of molecules *A* and *B* form an angle of about 30° and are situated roughly parallel to the *a*, *c* plane. The bases are stacked along **b** at alternating distances of about 3·3 and 3·8 Å (Fig. 3). The C(4)–O(4) bond of one molecule overlaps with the ring system of the neighbouring molecules (Fig. 2). This type of base stacking involving overlap of polar groups with an aromatic ring system is quite common in nucleoside and nucleotide structures (Bugg, Thomas, Sundaralingam & Rao, 1971). The molecules form stacked dimers which are linked by two hydrogen bonds between atoms O(4) *A* and O(5′) *B* (2·712 Å) and atoms O(4) *B* and O(5′) *A* (2·727 Å), Fig. 3. Two further hydrogen bonds between atoms O(3′) *A* and O(3′) *B* and atoms N(3) *A* and O(3′) *A* of 2·812 Å and 2·860 Å respectively link molecules in different stacks (Fig. 2).

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The Crystal Structure of $CsMnCl_3$ and a Summary of the Structures of RMX_3 Compounds*

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Cesium trichloromanganate, $CsMnCl_3$, has been found by single-crystal X-ray diffraction studies to possess rhombohedral symmetry and to crystallize in the space group $R\overline{3}m$. At 23°C, the hexagonal unit-cell lattice constants are $a=7·290$ (5) and $c=27·317$ (4) Å with $V=1257·2$ Å^3, M.W.(calc) 293·63, $Z=9$, $D_M=3·35$ (4), $D_X=3·49$ g cm^{-3}. Full-matrix least-squares refinement of 481 observed three-dimensional diffractometer data (Mo $K\alpha$) led to a final weighted residual of 0·094 on F. The compound consists of facial-bridged $[MnCl_6]^{4-}$ octahedral trimers with each trimer linked to other trimers by sharing corners in such a way that the trimers spiral around the trigonal axis. The crystallographic site symmetries of the manganese atoms are D_{3d} for the central manganese atoms of a trimer and C_{3v} for the two terminal manganese atoms of a trimer. There are three crystallographically unique manganese–chlorine distances, 2·514 (3), 2·545 (9), and 2·557 (14) Å. The latter two facial-bridged distances compare favorably with the Mn–Cl distance 2·560 Å in $(CH_3)_4NMnCl_3$. The synthesis, lattice parameters, and space groups of the previously unreported compounds $CsVI_3$, $CsCrI_3$, $CsNiI_3$ and $CsMgI_3$ are described. Using the available structural data, the crystallographic properties of RMX_3 (R = univalent cation, M = divalent transition metal cation, X = halogen anion) compounds can be correlated with the properties of the individual ions, R, M, and X. The relation of these properties to the structural types of RMX_3 is briefly discussed.

Introduction

Compounds with the general formula $CsMX_3$ form a class of structurally related compounds whose crystal geometry can be described as a stacking of ordered, close-packed CsX_3 layers, with the M cations filling octahedral sites between these layers. There are two possible types of stacking of CsX_3 layers, cubic and hexagonal, both of which are observed (Longo & Kafales, 1969). It is also possible to have various combinations of cubic and hexagonal packing within the same crystal lattice. Complete or partial structural data published for $CsMgCl_3$ (McPherson, Kistenmacher & Stucky, 1970), $CsVCl_3$ (Seifert & Ehrlich, 1959), $CsCrCl_3$ (McPherson & Stucky, 1972), $CsFeCl_3$ (Seifert & Klatyk, 1966), $CsCoCl_3$ (Soling, 1968), $CsNiCl_3$ (Tishchenko, 1955), and $CsCuCl_3$ (Schlueter, Jacobson & Rundle, 1966) show that all the complexes have the same hexagonal close-packed structure except those of Cr(II) and Cu(II). The latter two compounds are subject to the Jahn–Teller effect and the Cu(II) complex in particular shows differences of 0·2 to 0·4 Å in metal–halogen bond distances.

In this paper, we report the results of a complete single-crystal structural investigation of $CsMnCl_3$. Initial preliminary results obtained from powder X-ray studies by Kestigian, Croft & Leipzig (1967) demonstrated that $CsMnCl_3$ has hexagonal symmetry with $a=b=7·288$, $c=27·44$ Å, and $Z=9$. This is an unusual number of molecules per unit cell for a $CsMX_3$ system and a detailed study of the structure of this compound seemed important in order to understand the structural and magnetic properties of RMX_3 complexes. It is also the purpose of this paper to show that through the correlation of the available data for known RMX_3 structures, it is possible to make some generalizations concerning the factors which determine the type of structure that is obtained. Interesting magnetic and spectroscopic properties of these one-dimensional

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