When considered as a class, the imidazolines have higher affinities for the α-adrenergic receptor than the phenethylamines (Ruffolo, Dillard, Yaden & Waddell, 1979). One of the reasons for this observation may be that the dispersed-charge arrangement within the imidazole moiety helps it to achieve a better interaction with the α-receptors, which may not be possible in the phenethylamines since the positive charge is concentrated at the N atom in the protonated forms of these molecules.

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Structure of 2,2′-Anhydro-1-β-D-arabinofuranosyl-6-methyluracil Hemihydrate*

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Abstract. $C_{10}H_{12}N_2O_5.\frac{1}{2}H_2O$, $M_r = 249{\cdot}2$ ($240{\cdot}2$ for $C_{10}H_{12}N_2O_5$), monoclinic, space group $P2_1$, $a = 9{\cdot}595\,(1)$, $b = 16{\cdot}196\,(2)$, $c = 7{\cdot}341\,(2)$ Å, $\beta = 109{\cdot}39\,(2)°$, $V = 1076{\cdot}1\,(4)$ Å^3, $Z = 4$, D_m not measured, $D_x = 1{\cdot}538$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}14$ mm^{-1}, $T = 294$ K, $F(000) = 524$. Final $R = 0{\cdot}037$ for 2428 unique X-ray diffractometer data. The two independent molecules of the asymmetric unit have different conformations for sugar rings and side chains; C(4′)-*endo*-C(3′)-*exo*, g^+ (molecule *A*) and C(4′)-*exo*-O(4′)-*endo*, *t* (*B*). There are short intramolecular O(5′)···base contacts in molecule *A*. All hydroxyl and water hydrogen atoms participate in six hydrogen bonds with O(4*A*) (bifurcated), O(4*B*), O(water), O(3′*B*) and N(3*B*) atoms as their acceptors.

Introduction. *O*-Cyclonucleosides can be used as intermediates in nucleoside syntheses owing to easy breaking of their anhydro linkage. The best known example is cycloara C, which can be converted into therapeutically active ara C in physiological conditions (Hoshi, Kanzawa & Kuretani, 1972; Loo, Ho, Bodey & Freireich, 1975). Cycloara C, unlike its acyclic form,

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* IUPAC name: 2,3,3a,9a-tetrahydro-3-hydroxy-2-hydroxymethyl-8-methyl-6*H*-furo[2′,3′:4,5]oxazolo[3,2-*a*]pyrimidin-6-one.

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is resistant to cytidine deaminase which converts ara C to the inactive metabolite ara U. These findings have led to increasing interest in the conformation and electronic structure of cyclonucleosides. Until now the crystal structures of several *O*-cyclouridine derivatives have been examined by X-ray methods: 2,2′-anhydro-1-β-D-arabinofuranosylthymine (Harrison, Schinazi & Rubin, 1982); 2,2′-anhydro-1-β-D-arabinofuranosyluracil (Suck & Saenger, 1973; Delbaere & James, 1973); 2,2′-anhydro-1-α-D-xylofuranosyluracil (Birnbaum, Giziewicz, Huber & Shugar, 1976) and 2,2′-anhydro-1-β-D-arabinofuranosyl-2-thiouracil (Yamagata, Yoshimura, Fujii, Fujiwara, Tomita & Ueda, 1980). The title compound, containing two molecules and one water in the asymmetric unit, is a good model for studying the conformational flexibility of these cyclonucleosides.

Experimental. Crystallized from methanol solution by slow evaporation of the solvent. A single crystal of dimensions 0·14 × 0·21 × 0·41 mm was used for data collection and accurate cell determination on a CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation, θ up to 27°. Unit-cell parameters determined by least squares from 2θ values of 25 reflections in the range $9 \le \theta \le 13$°. 2428 independent intensities [2045 considered observed with $I \ge 2\sigma(I)$] were measured with $\omega/2\theta$ scan technique ($0 \le h \le 12$, $0 \le k \le 20$, $-9 \le l \le 9$). Three standards monitored during data collection did not show significant change in intensity. No corrections for absorption or secondary extinction.

The structure was solved by direct methods (*MULTAN*81: Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1981) and refined by full-matrix least squares with anisotropic temperature factors for non-H and isotropic ones for H atoms. H atoms located from difference Fourier synthesis. The largest shift-over-error ratio in final cycle was 0·19. Quantity minimized was $\sum w(F_o - F_c)^2$, $w^{-1} = \sigma^2(I) + 0{\cdot}04\,|F_o|^2$. The final R (wR) factor was 0·037 (0·046) and final difference Fourier map showed no peaks above 0·15 e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed by the CAD-4 *SDP* system (Frenz, 1978) and *PARST* (Nardelli, 1983).

Discussion. Atomic parameters and selected geometrical data are given in Tables 1 to 3.* Fig. 1 shows general conformations of the two independent

Table 1. *Final positional and equivalent isotropic temperature parameters for non-*H *atoms with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i \sum_j \beta_{ij}(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	$B_{\rm eq}$(Å^2)
Molecule *A*				
O(4)	−0·4901 (2)	0·8044	0·9440 (3)	4·10 (4)
O(2)	−0·0888 (2)	0·9700 (1)	1·0662 (2)	3·07 (3)
O(1′)	0·1749 (2)	0·8346 (1)	1·2269 (2)	2·96 (3)
O(3′)	0·2940 (2)	1·0226 (1)	1·2784 (2)	3·77 (4)
O(5′)	−0·0332 (2)	0·8743 (1)	1·4263 (3)	4·21 (4)
N(1)	−0·0694 (2)	0·8381 (1)	0·9987 (3)	2·49 (4)
N(3)	−0·2969 (2)	0·8918 (1)	1·0037 (3)	2·96 (4)
C(1)	−0·0248 (3)	0·6958 (2)	0·9147 (4)	3·97 (6)
C(2)	−0·1599 (3)	0·8977 (2)	1·0221 (3)	2·59 (4)
C(4)	−0·3589 (3)	0·8144 (2)	0·9551 (3)	2·97 (5)
C(5)	−0·2702 (3)	0·7485 (2)	0·9195 (3)	3·00 (5)
C(6)	−0·1265 (3)	0·7599 (2)	0·9436 (3)	2·65 (5)
C(1′)	0·0838 (2)	0·8665 (2)	1·0508 (3)	2·57 (4)
C(2′)	0·0643 (2)	0·9592 (2)	1·0787 (3)	2·65 (4)
C(3′)	0·1677 (3)	0·9791 (1)	1·2810 (3)	2·65 (4)
C(4′)	0·2037 (2)	0·8948 (2)	1·3814 (3)	2·79 (5)
C(5′)	0·1207 (3)	0·8732 (2)	1·5172 (3)	3·54 (5)
Molecule *B*				
O(4)	−0·0803 (2)	0·6058 (2)	0·3058 (3)	4·81 (5)
O(2)	−0·5453 (2)	0·5387 (1)	0·2830 (2)	3·14 (3)
O(1′)	−0·6070 (2)	0·6797 (1)	0·5650 (2)	3·15 (3)
O(3′)	−0·8479 (2)	0·5039 (1)	0·4181 (2)	3·43 (4)
O(5′)	−0·6750 (2)	0·6750 (1)	0·9236 (2)	4·61 (4)
N(1)	−0·4644 (2)	0·6664 (1)	0·3625 (2)	2·33 (3)
N(3)	−0·3109 (2)	0·5669 (1)	0·2895 (3)	2·95 (4)
C(1)	−0·4044 (3)	0·8128 (2)	0·4346 (4)	3·72 (6)
C(2)	−0·4331 (2)	0·5907 (2)	0·3125 (3)	2·54 (4)
C(4)	−0·2000 (3)	0·6252 (2)	0·3247 (3)	3·05 (5)
C(5)	−0·2286 (3)	0·7072 (2)	0·3775 (4)	3·25 (5)
C(6)	−0·3603 (3)	0·7284 (2)	0·3928 (3)	2·68 (5)
C(1′)	−0·6130 (2)	0·6703 (1)	0·3718 (3)	2·40 (4)
C(2′)	−0·6698 (2)	0·5829 (2)	0·3080 (3)	2·57 (4)
C(3′)	−0·7125 (2)	0·5477 (2)	0·4762 (3)	2·62 (4)
C(4′)	−0·7119 (2)	0·6236 (2)	0·6004 (3)	2·61 (4)
C(5′)	−0·6627 (3)	0·6059 (2)	0·8126 (3)	3·84 (6)
Water				
O(*W*)	0·3576 (2)	0·9415 (1)	0·9986 (3)	4·23 (4)

Table 2. *Bond lengths* (Å) *and angles* (°) *involving non-*H *atoms*

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(1)–C(6)	1·487 (5)	1·493 (5)	C(1′)–N(1)	1·465 (3)	1·451 (3)
C(2)–N(1)	1·347 (4)	1·342 (4)	C(1′)–C(2′)	1·535 (5)	1·533 (4)
C(2)–N(3)	1·280 (4)	1·297 (3)	C(2′)–O(2)	1·452 (3)	1·456 (3)
C(2)–O(2)	1·340 (4)	1·327 (3)	C(2′)–C(3′)	1·522 (3)	1·534 (4)
C(4)–O(4)	1·245 (4)	1·242 (4)	C(3′)–O(3′)	1·407 (3)	1·416 (3)
C(4)–N(3)	1·383 (5)	1·381 (4)	C(3′)–C(4′)	1·535 (4)	1·529 (4)
C(4)–C(5)	1·442 (5)	1·435 (5)	C(4′)–O(1′)	1·451 (3)	1·442 (3)
C(5)–C(6)	1·343 (4)	1·350 (4)	C(4′)–C(5′)	1·509 (4)	1·498 (3)
C(6)–N(1)	1·386 (4)	1·381 (4)	C(5′)–O(5′)	1·404 (3)	1·412 (3)
C(1′)–O(1′)	1·397 (3)	1·409 (3)			
O(2)–C(2)–N(1)	111·0 (3)	112·4 (2)	C(1′)–O(1′)–C(4′)	111·9 (2)	108·4 (2)
O(2)–C(2)–N(3)	120·9 (3)	120·6 (2)	O(1′)–C(1′)–N(1)	113·0 (2)	109·9 (2)
N(1)–C(2)–N(3)	128·0 (3)	126·9 (2)	O(1′)–C(1′)–C(2′)	107·7 (2)	107·4 (2)
O(4)–C(4)–N(3)	118·3 (3)	119·3 (3)	N(1)–C(1′)–C(2′)	100·7 (2)	101·6 (2)
O(4)–C(4)–C(5)	122·5 (3)	122·1 (3)	O(2)–C(2′)–C(1′)	106·0 (2)	105·6 (2)
N(3)–C(4)–C(5)	119·3 (3)	118·6 (3)	O(2)–C(2′)–C(3′)	110·8 (2)	111·6 (2)
C(4)–C(5)–C(6)	121·1 (3)	121·7 (3)	C(1′)–C(2′)–C(3′)	105·6 (2)	105·1 (2)
N(1)–C(6)–C(1)	117·7 (3)	117·4 (3)	O(3′)–C(3′)–C(2′)	112·2 (2)	114·0 (2)
N(1)–C(6)–C(5)	116·9 (3)	116·7 (3)	O(3′)–C(3′)–C(4′)	113·2 (2)	113·8 (2)
C(1)–C(6)–C(5)	125·4 (3)	126·0 (3)	C(2′)–C(3′)–C(4′)	104·5 (2)	103·4 (2)
C(2)–O(2)–C(2′)	109·5 (2)	108·8 (2)	O(1′)–C(4′)–C(3′)	105·6 (2)	105·3 (2)
C(2)–N(1)–C(6)	118·9 (2)	119·5 (2)	O(1′)–C(4′)–C(5′)	110·8 (2)	108·1 (2)
C(2)–N(1)–C(1′)	112·0 (2)	111·4 (2)	C(3′)–C(4′)–C(5′)	116·1 (2)	113·8 (3)
C(6)–N(1)–C(1′)	128·9 (2)	129·1 (2)	O(5′)–C(5′)–C(4′)	112·7 (2)	113·0 (3)
C(2)–N(3)–C(4)	115·7 (4)	116·5 (2)			

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43084 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Structural characteristics of the hydrogen bonds and selected torsion angles (distances in Å, angles in °)*

Donor	Acceptor	D—H	$D\cdots A$	H$\cdots A$	D—H$\cdots A$
O(3′A)	O($W^{\rm i}$)	0·90 (4)	2·674 (3)	1·80 (3)	163 (3)
O(3′B)	O(4$B^{\rm ii}$)	0·85 (3)	2·675 (3)	1·83 (3)	174 (3)
O(5′A)	O(3′$B^{\rm iii}$)	1·03 (4)	2·807 (3)	1·79 (4)	173 (3)
O(5′B)	O(4$A^{\rm i}$)	0·85 (3)	2·718 (2)	1·90 (3)	162 (3)
O(W)	O(4$A^{\rm iv}$)	0·88 (3)	2·760 (2)	1·89 (3)	169 (3)
O(W)	N(3$B^{\rm v}$)	0·83 (3)	2·858 (3)	2·03 (3)	178 (4)

Torsion angle	Notation	Molecule A	Molecule B
O(1′)—C(1′)—N(1)—C(6)	χ	−69·7 (3)	−70·8 (3)
C(1′)—N(1)—C(2)—O(2)		7·0 (3)	0·4 (3)
N(1)—C(2)—O(2)—C(2′)		−1·4 (3)	2·2 (3)
C(2)—O(2)—C(2′)—C(1′)		−4·3 (3)	−3·7 (3)
O(2)—C(2′)—C(1′)—N(1)		7·6 (2)	3·7 (2)
C(2′)—C(1′)—N(1)—C(2)		−8·9 (3)	−2·6 (2)
O(1′)—C(1′)—C(2′)—C(3′)	τ_1	6·5 (3)	6·5 (2)
C(1′)—C(2′)—C(3′)—C(4′)	τ_2	−17·3 (3)	13·0 (3)
C(2′)—C(3′)—C(4′)—O(1′)	τ_3	21·9 (2)	−27·9 (3)
C(3′)—C(4′)—O(1′)—C(1′)	τ_4	−19·0 (3)	33·7 (2)
C(4′)—O(1′)—C(1′)—C(2′)	τ_0	7·9 (3)	−25·2 (2)
O(1′)—C(4′)—C(3′)—O(3′)		−100·5 (2)	−152·2 (2)
O(1′)—C(4′)—C(5′)—O(5′)	$\psi_{\rm OO}$	−62·6 (3)	68·6 (3)
C(3′)—C(4′)—C(5′)—O(5′)	$\psi_{\rm CO}$	57·8 (3)	185·1 (2)

Symmetry code for acceptor atoms: (i) x, y, z; (ii) $1+x, y, z$; (iii) $1-x, y-\frac{1}{2}, -z$; (iv) $x-1, y, z$; (v) $-x, y-\frac{1}{2}, 1-z$.

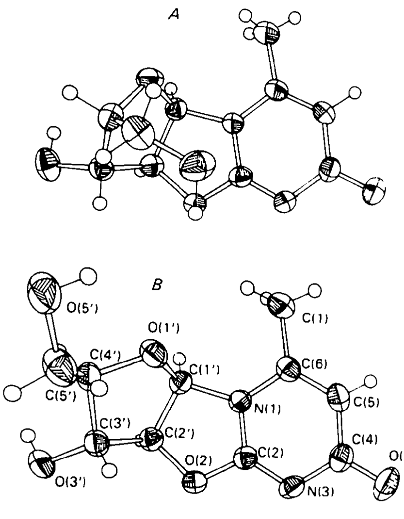

Fig. 1. Conformations and numbering system of the two independent molecules of the title compound projected on their base planes.

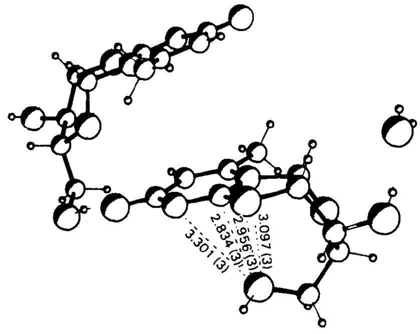

Fig. 2. The asymmetric unit viewed along [010].

molecules of the title compound as projected on the mean uracil ring and the numbering of atoms while the projection of the asymmetric unit on to (010) is shown in Fig. 2. Most equivalent bond lengths and angles in both independent molecules agree to within 0·01 Å and 0·5°. Greater variations occur for the N(3)—C(2) bond length (5σ) and C(1′)—O(1′)—C(4′), O(1′)—C(1′)—N(1), O(1′)—C(4′)—C(5′) and C(3′)—C(4′)—C(5′) angles (16, 16, 14 and 10σ, respectively). The lengthening of the C(2′)—O(2) bond and simultaneous shortening of C(2)—O(2), making a difference of about 0·12 Å, is caused by different overlapping of the lone electron pair at O(2) with the π-electron system of the uracil ring, a phenomenon which is certainly related to the easy breaking of the C(2′)—O(2) bond in this class of compound. As in other nucleoside structures, the C(4′)—O(1′) bond in the title compound is significantly longer than the C(1′)—O(1′) bond, the difference in this case being 0·054 and 0·033 Å for the A and B molecules, respectively. The shortening of C(1′)—O(1′) is due to an anomeric effect (Sundaralingam, 1965) on the C(1′) atom connected to two electronegative atoms :O—C—N $\leftrightarrow$ $^+$O=C—N$^-$. The effect would be perfect if the N(1)—C(1′)—O(1′)—C(4′) torsion angle were ±60° (impossible in the furanose ring). As the torsion angles are −102·1 (3) and −134·7 (3)° in A and B, respectively, the effect is small but still relevant, and stronger in molecule A where the torsion angle is smaller.

The general conformations of the two independent molecules are different except for the base orientation which is *anti* in both cases [$\chi = -69{\cdot}7\,(3)°$ in A and $-70{\cdot}8\,(3)°$ in B], which is forced by 2,2′-cyclization. The uracil rings are essentially planar. The mean distances of the ring atoms from the least-squares planes are 5·0 (A) and 4·5 (B) times greater than their standard deviations for uracil, and 15 (A) and 7 (B) times greater for 1,3-oxazolidine ring atoms. The interplanar angles between the base and the oxazolidine rings are 3·70 (8) and 1·35 (9)° for A and B, respectively. The base planes of the A and B molecules form an angle of 6·18 (7)°; they are roughly parallel to the xy plane and show some tendency to stacking. The conformations of the 1,3-oxazolidine rings are significantly different (Table 3). The sugar rings are more puckered and differentiated than the oxazolidine ones and they can be termed C(4′)-*endo*-C(3′)-*exo* [pucker] ($^4T_3$) in A and C(4′)-*exo*-O(4′)-*endo* [pucker] ($_4E^{\rm O}$) in B ($P_A = 218$, $P_B = 67$, $\tau_A = 22{\cdot}3$, $\tau_B = 33{\cdot}2°$; Altona & Sundaralingam, 1972).

The orientation of the side chain is g^+ [$\psi_{\rm CO} = 57{\cdot}8\,(3)°$] in molecule A and t [$\psi_{\rm CO} = -174{\cdot}9\,(2)°$] in molecule B. The *gauche–gauche* orientation of the C(5′)—O(5′) bond in molecule A gives rise to short intramolecular contacts between O(5′) and the base atoms (Fig. 2) though it has been suggested that a positive charge on the base is essential for O(5′)$\cdots$base electrostatic attraction (Brennan & Sundaralingam, 1973). There are indeed short intramolecular O(5′)···base contacts in all known structures of cyclic nucleosides with positive charge on the base, *i.e.* 2,2′-anhydro-1-β-D-arabinofuranosylcytosine-3′,5′-diphosphate (Yamagata, Suzuki, Fujii, Fujiwara & Tomita, 1979), 2,2′-anhydro-1-β-D-arabinofuranosyl-5-dimethylsulfonio-6-oxocytosine chloride (Yamagata, Koshibe, Tokuoka, Fujii, Fujiwara, Kanai & Tomita, 1979) and 2,2′-anhydro-1-β-D-arabinofuranosylcytosine hydrochloride (Brennan & Sundaralingam, 1973); and in just one structure with no formal charge: 2,2′-anhydro-1-(3′,5′-di-*O*-acetyl-β-D-arabinofuranosyl)-5-chloro-6-oxocytosine (Yamagata, Koshibe, Tokuoka, Fujii, Fujiwara, Kanai & Tomita, 1979).

The molecules in the crystal of the title compound are connected by a complicated system of hydrogen bonding employing all six H atoms bonded to oxygens, and O(4*A*) [bifurcated hydrogen bonding], O(4*B*), O(3′*B*), N(3*B*) and water as their acceptors. The donor···acceptor distances are between 2·67 and 2·86 Å while the *D*—H···*A* angles are between 162 and 178°. The absence of other than O(4) acceptors in molecule *A* may be related to the intramolecular O(5′*A*)···uracil(*A*) moiety interactions (Fig. 2).

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Structure of the 1/1 Complexes of [2.2.2]Paracyclophanetriene and [2.2]Paracyclophane with Tetracyanoethylene

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Abstract. $C_{24}H_{18}.C_6N_4$ (I), $M_r = 437\cdot51$, monoclinic, $P2_1/m$, $a = 8\cdot192\ (5)$, $b = 20\cdot579\ (8)$, $c = 6\cdot881\ (3)$ Å, $\beta = 94\cdot70\ (4)°$, $V = 1156$ Å^3, $Z = 2$, $D_x = 1\cdot26$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0\cdot604$ mm^{-1}, $F(000) = 452$, $T = 153$ K; $C_{16}H_{16}.C_6N_4$ (II), $M_r = 338\cdot60$, triclinic, $P\bar{1}$, $a = 8\cdot079\ (5)$, $b = 8\cdot632\ (4)$, $c = 7\cdot615\ (3)$ Å, $\alpha = 106\cdot80\ (5)$, $\beta = 111\cdot60\ (5)$, $\gamma = 101\cdot00\ (5)°$, $V = 446$ Å^3, $Z = 1$, $D_x = 1\cdot26$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0\cdot066$ mm^{-1}, $F(000) = 176$, $T = 293$ K. The structures were refined to wR values of 0·053 (I) and 0·068 (II) for 869 and 726 observed intensities respectively. In (I), the paracyclophane molecule has a crystallographic mirror plane and a threefold pseudo-axis of symmetry, as was observed in [2.2.2]paracyclophane. In (II), the paracyclophane molecule has a centre of symmetry. In both compounds, the tetracyanoethylene (TCNE) molecules are disordered. The disorder corresponds to two perpendicular, almost coplanar orientations of TCNE with 58–42% in (I) and 93–7% in (II). Each

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