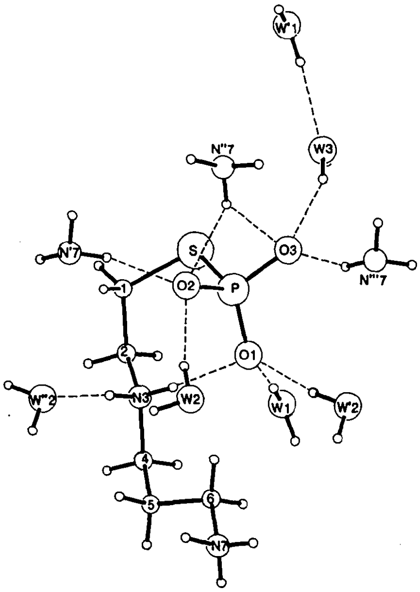

Fig. 2. Diagram illustrating the numbering scheme for the WR 2721 and water molecules. Eleven of the hydrogen bonds are depicted by the dashed lines. The $W(3)H2$ hydrogen bond to $W''(1)$ is not shown. Several fragments of surrounding WR 2721 molecules are shown in order to illustrate the hydrogen bonding which occurs between one WR 2721 molecule and its neighbors. The size of the circles was arbitrarily chosen to correspond to the atomic weight of the atom.

between the nitrogen atoms $[H_2N(CH_2)_nNH-]$ all demonstrate some radioprotection except for the WR 1065 analog with $n = 4$. Analogs of WR 1065 with $n = 7$ or greater are inactive. The most potent analog of WR 2721 is WR 2823 with $n = 5$, which showed essentially the same radioprotective strength as WR 2721; however, WR 2823 displayed more toxicity upon oral dosing. Thus, the interatomic distances found in the crystalline structure of WR 2721 between N(3) and N(7) of 4·45 Å and between N(3) and S of 3·49 Å are desirable for optimum radioprotective properties.

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Structure of 6,5'-Anhydro-6-hydroxy-2',3'-*O*-isopropylideneuridine

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Abstract. $C_{12}H_{14}N_2O_6$, $M_r = 282·3$, orthorhombic, $P2_12_12_1$, $a = 10·412\,(2)$, $b = 14·936\,(2)$, $c = 16·651\,(3)$ Å, $V = 2589·46$ Å^3, $Z = 8$, $D_m = 1·450$, $D_x = 1·447$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1·5418$ Å, $\mu = 0·902$ mm^{-1}, $F(000) = 1184·00$, $T = 293$ K, $R = 0·039$, $wR = 0·038$ for 2548 unique reflections with $F > 3\sigma(F)$. The two crystallographically independent molecules in the asymmetric unit have similar geometries with the ribose ring having an O(4')-*exo*, C(4')-*endo* pucker and the uracil base in the *anti* conformation. The geometry about the exocyclic C(4')—C(5') bond in both molecules is *gauche–gauche*. The dioxolane ring assumes twist conformations in both molecules.

Introduction. We report here the crystal structure of the title compound which was undertaken as part of our studies on the isopropylidene derivatives of nucleosides (Katti, Seshadri & Viswamitra, 1981; Gautham, Seshadri & Viswamitra, 1983; Gautham, Seshadri, Viswamitra & Salisbury, 1983). Such structures serve as models for studying the flexibility of nucleoside conformation under external cyclic constraints. The present structure, in addition to the isopropylidene group, has a further cyclic constraint imposed by the cyclization of the base–sugar moiety through O(6) of the hydroxyuracil base.

Experimental. Plate-shaped crystals of the title compound (Sigma Chemicals) were grown by slow evaporation of water/acetone solution. Density measured by flotation in carbon tetrachloride/benzene showed that there are two independent molecules in the asymmetric unit. Accurate unit-cell parameters were obtained from least-squares refinement of 25 high-angle reflections on a CAD-4 diffractometer. Cu $K\alpha$ intensity data were collected using a crystal of size 0·6 × 0·25 × 0·2 mm up to $(\sin\theta)/\lambda = 0{\cdot}617$ Å^{-1} using ω–2θ scans. Lorentz and polarization corrections were applied. An absorption correction was also applied with minimum and maximum values of the transmission factor equal to 82·106 and 99·614 respectively. 2565 reflections were measured for *h*, *k* and *l* values between 0 and 12, 0 and 18 and 0 and 20 respectively, of which 2548 were unique with $F > 3\sigma(F)$. Two reflections, $\bar{2}2\bar{3}$ and $\bar{3}11$, monitored periodically during the data collection showed negligible variation indicating crystal and instrument stability. The structure was solved by direct methods by *MULTAN*80 (Germain, Main & Woolfson, 1971). The *E* map computed with the best set of phases revealed the positions of most of the non-H atoms. The rest of the molecule was located from difference Fourier maps. The full-matrix refinement on *F* [all calculations with *SHELX*76 (Sheldrick, 1976)] with anisotropic temperature factors reduced *R* to 0·082. Of the 28 atoms present in the asymmetric unit, 24 were located from difference Fourier maps. The remaining four were fixed using geometric considerations. H atoms were refined isotropically. After the final cycles of refinement, $R = 0{\cdot}039$, $wR = 0{\cdot}038$ and *S* (goodness of fit) = 0·19. The function minimized in the final stages of refinement was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/(\sigma^2 F + 0{\cdot}03951F^2)$. Maximum and minimum electron densities in the final difference Fourier synthesis are 0·26 and −0·34 e Å^{-3} respectively. Maximum $\Delta/\sigma = 0{\cdot}927$. Scattering factors for non-H atoms were computed from the function of Cromer & Waber (1965) and for H atoms from Stewart, Davidson & Simpson (1965).*

Table 1. *Final positional parameters* (×10^4) *and equivalent isotropic temperature factors for non*-H *atoms, with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}(Å^2)
Molecule *A*				
N(1)	3081 (2)	255 (2)	1368 (1)	3·52 (5)
C(2)	3121 (2)	75 (2)	2193 (1)	4·00 (6)
O(2)	2209 (2)	−211 (2)	2563 (1)	6·66 (8)
N(3)	4280 (2)	240 (2)	2546 (1)	3·62 (5)
C(4)	5365 (2)	578 (1)	2192 (1)	3·07 (5)
O(4)	6346 (2)	656 (1)	2593 (1)	3·76 (4)
C(5)	5243 (2)	786 (2)	1357 (1)	3·31 (5)
C(6)	4151 (2)	593 (1)	979 (1)	3·02 (5)
O(6)	4024 (2)	761 (1)	182 (1)	3·43 (4)
C(1')	1903 (2)	8 (2)	924 (1)	3·59 (5)
C(2')	1335 (2)	765 (2)	418 (1)	3·52 (6)
O(2')	−25 (2)	711 (2)	468 (1)	4·47 (5)
C(3')	1658 (2)	499 (2)	−455 (1)	3·64 (6)
O(3')	451 (2)	225 (1)	−775 (1)	4·43 (5)
C(4')	2533 (3)	−305 (2)	−367 (1)	3·73 (5)
O(4')	2165 (2)	−702 (1)	386 (1)	4·08 (4)
C(5')	3914 (3)	−20 (2)	−340 (1)	3·93 (6)
C(7)	−522 (3)	689 (2)	−333 (1)	4·65 (7)
C(8)	−1715 (3)	117 (4)	−334 (2)	6·14 (12)
C(9)	−703 (4)	1633 (3)	−654 (2)	6·14 (10)
Molecule *B*				
N(1)	9637 (2)	−2569 (1)	2389 (1)	3·70 (5)
C(2)	10603 (3)	−3200 (2)	2240 (1)	3·66 (5)
O(2)	10615 (2)	−3664 (1)	1641 (1)	5·16 (5)
N(3)	11522 (2)	−3261 (1)	2825 (1)	3·84 (5)
C(4)	11580 (2)	−2775 (2)	3535 (1)	3·78 (6)
O(4)	12492 (2)	−2870 (2)	3993 (1)	5·27 (5)
C(5)	10535 (2)	−2165 (2)	3657 (1)	3·69 (5)
C(6)	9665 (2)	−2057 (1)	3083 (1)	3·20 (5)
O(6)	8704 (2)	−1452 (1)	3162 (1)	3·63 (4)
C(1')	8703 (3)	−2399 (2)	1742 (1)	3·66 (6)
C(2')	7294 (3)	−2413 (2)	2014 (2)	3·71 (6)
O(2')	6539 (2)	−2865 (1)	1440 (1)	5·14 (5)
C(3')	6887 (3)	−1422 (2)	1961 (1)	3·46 (5)
O(3')	6113 (2)	−1390 (1)	1259 (1)	4·47 (5)
C(4')	8137 (3)	−928 (2)	1829 (1)	3·62 (5)
O(4')	8917 (2)	−1555 (1)	1390 (1)	4·24 (4)
C(5')	8766 (3)	−693 (2)	2616 (2)	3·85 (6)
C(7)	5596 (3)	−2264 (2)	1126 (2)	5·08 (7)
C(8)	4354 (4)	7645 (3)	1614 (4)	8·25 (16)
C(9)	4550 (8)	2582 (3)	4756 (3)	9·12 (18)

Discussion. The final positional parameters of the non-H atoms are given in Table 1 and Table 2 lists the bond lengths and bond angles involving non-H atoms. Fig. 1 shows the molecular geometry viewed perpendicular to the base of molecule *B* and Fig. 2 indicates the hydrogen-bonding scheme in the unit cell viewed down the *a* axis.

Uracil: The uracil base is essentially planar and is *anti* with respect to the sugar ring in both the crystallographically independent molecules, as in the

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares planes and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44333 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
N(1)–C(2)	1·401 (2)	1·400 (4)	C(2)–N(1)–C(6)	120·3 (2)	120·3 (2)
C(2)–O(2)	1·210 (3)	1·215 (3)	C(1′)–N(1)–C(2)	117·8 (2)	117·5 (2)
C(2)–N(3)	1·366 (3)	1·368 (3)	C(1′)–N(1)–C(6)	121·7 (2)	121·8 (2)
N(3)–C(4)	1·370 (3)	1·390 (3)	N(1)–C(2)–N(3)	114·4 (2)	115·0 (2)
C(4)–O(4)	1·226 (3)	1·226 (3)	N(1)–C(2)–O(2)	123·0 (2)	122·5 (2)
C(4)–C(5)	1·430 (2)	1·434 (4)	N(3)–C(2)–O(2)	122·6 (2)	122·5 (2)
C(5)–C(6)	1·330 (3)	1·326 (3)	C(2)–N(3)–C(4)	127·6 (2)	126·9 (2)
C(6)–O(6)	1·358 (2)	1·354 (3)	N(3)–C(4)–C(5)	115·1 (2)	114·8 (2)
C(6)–N(1)	1·384 (3)	1·388 (2)	N(3)–C(4)–O(4)	119·2 (2)	120·1 (2)
N(1)–C(1′)	1·478 (3)	1·473 (3)	C(5)–C(4)–O(4)	125·7 (2)	125·0 (2)
C(1′)–C(2′)	1·530 (4)	1·535 (4)	C(4)–C(5)–C(6)	119·3 (2)	119·6 (2)
C(2′)–C(3′)	1·544 (3)	1·541 (4)	C(5)–C(6)–N(1)	123·0 (2)	123·2 (2)
C(3′)–C(4′)	1·515 (4)	1·512 (4)	C(5)–C(6)–O(6)	120·4 (2)	121·1 (2)
C(4′)–O(4′)	1·440 (3)	1·439 (3)	N(1)–C(6)–O(6)	116·5 (2)	115·7 (2)
O(4′)–C(1′)	1·416 (3)	1·408 (3)	N(1)–C(1′)–O(4′)	110·0 (2)	110·8 (2)
C(4′)–C(5′)	1·500 (4)	1·507 (4)	N(1)–C(1′)–C(2′)	114·3 (2)	114·4 (2)
C(5′)–O(6)	1·459 (3)	1·455 (4)	O(4′)–C(1′)–C(2′)	106·2 (2)	106·7 (2)
C(2′)–O(2′)	1·420 (3)	1·409 (4)	C(1′)–C(2′)–C(3′)	104·2 (2)	103·5 (2)
O(2′)–C(7)	1·430 (3)	1·430 (4)	O(2′)–C(2′)–C(1′)	108·1 (2)	109·8 (2)
C(7)–O(3′)	1·432 (3)	1·429 (3)	O(2′)–C(2′)–C(3′)	105·0 (2)	105·5 (2)
O(3′)–C(3′)	1·425 (3)	1·420 (3)	C(2′)–C(3′)–C(4′)	104·1 (2)	103·9 (2)
C(7)–C(8)	1·508 (5)	1·533 (6)	O(3′)–C(3′)–C(2′)	103·5 (2)	103·7 (2)
C(7)–C(9)	1·520 (5)	1·494 (6)	O(3′)–C(3′)–C(4′)	109·8 (2)	110·7 (2)
			C(3′)–C(4′)–O(4′)	104·5 (2)	104·0 (2)
			C(3′)–C(4′)–C(5′)	110·7 (2)	111·2 (2)
			O(4′)–C(4′)–C(5′)	110·2 (2)	110·4 (2)
			C(1′)–O(4′)–C(4′)	107·1 (2)	106·3 (2)
			C(4′)–C(5′)–O(6)	108·7 (2)	110·0 (2)
			C(5′)–O(6)–C(6)	116·3 (2)	115·3 (2)
			C(2′)–O(2′)–C(7)	107·9 (2)	109·3 (2)
			O(2′)–C(7)–C(8)	108·2 (2)	109·3 (3)
			O(2′)–C(7)–O(3′)	103·6 (2)	104·9 (2)
			C(8)–C(7)–C(9)	115·0 (3)	114·9 (4)
			C(9)–C(7)–O(3′)	110·9 (2)	109·4 (3)
			O(2′)–C(7)–C(9)	110·6 (2)	109·4 (3)
			O(3′)–C(7)–C(8)	108·0 (2)	108·5 (3)
			C(7)–O(3′)–C(3′)	107·0 (2)	108·1 (2)

structures of 2,5′-anhydro-1-(2′,3′-*O*-isopropylidene-*β*-D-ribofuranosyl)-2-thiouracil (Yamagata, Fujii, Fujiwara & Tomita, 1980). Cyclization of the base–sugar moiety constrains the molecules to this *anti* conformation. The glycosidic torsion angle χ[O(4′)–C(1′)–N(1)–C(2)] (IUPAC–IUB Joint Commission on Biochemical Nomenclature, 1983) has values of −112·1 (2) and −107·6 (2)° in molecules *A* and *B* respectively.

Ribose: The furanose ring assumes an O(4′)-*exo*, C(4′)-*endo* twist conformation in both molecules. The deviations of O(4′) and C(4′) from the best three-atom plane are 0·351 (2) and −0·196 (3) Å respectively in molecule *A* and 0·314 (2) and −0·273 (2) Å respectively in molecule *B*. The maximum amplitudes of pucker are 38·2 (2) and 40·5 (2)° in molecules *A* and *B* respectively. Although this conformation is not commonly found in the structures of normal nucleosides and nucleotides, it has been previously observed in 5′-deoxy-5′,6-epithio-5,6-dihydro-2′,3′-*O*-isopropylidene-3-methyluridine (Viswamitra & Gautham, 1984) and 2,5′-anhydro-1-(2′,3′-*O*-isopropylidene-*β*-D-ribofuranosyl)-2-thiouracil (Yamagata, Fujii, Fujiwara & Tomita, 1980). The conformation about the C(4′)–C(5′) bond is *gauche–gauche* in both molecules with torsion angles φ_{OO}[O(6)–C(5′)–C(4′)–O(4′)] and φ_{OC}[O(6)–C(5′)–C(4′)–C(3′)] equal to −71·6 (3) and 43·5 (3)° respectively in molecule *A* and −71·2 (3) and 43·7 (3)° respectively in molecule *B*.

The heptameric base–sugar heterocycle ring has a chair conformation in both molecules similar to that found in the structure of 5′-deoxy-5′,6-epithio-5,6-

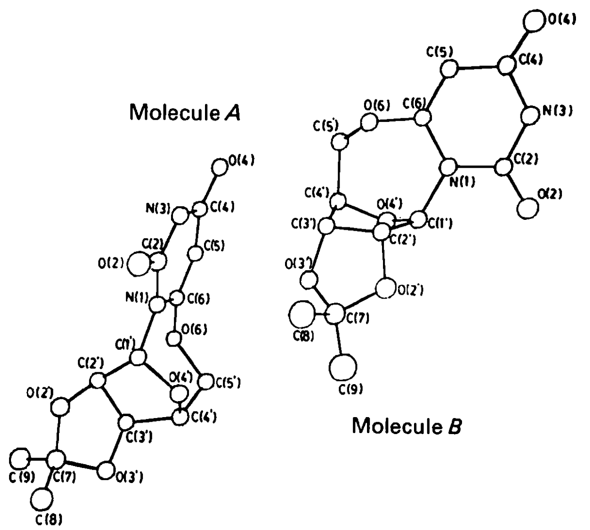

Fig. 1. The two molecules viewed perpendicular to the base of molecule *B*.

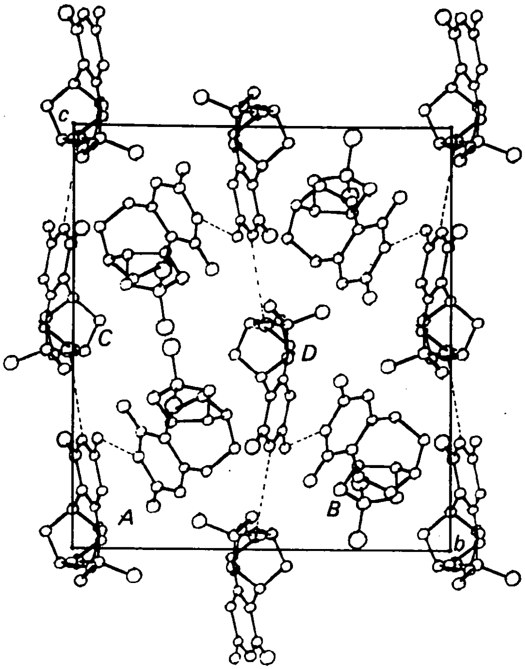

Fig. 2. Packing diagram viewed down the *a* axis showing hydrogen bonding between molecules in the unit cell. *A* and *B* – reference molecules; *C* and *D* – related to *A* by symmetry ($-x+\frac{1}{2}$, $-y$, $z+\frac{1}{2}$) and ($-x+2$, $y-\frac{1}{2}$, $-z+\frac{1}{2}$).

dihydro-2′,3′-*O*-isopropylidene-3-methyluridine (Gautham, Ramakrishnan, Seshadri & Viswamitra, 1982).

Dioxolane ring: In molecule *A*, C(7) and O(3′) are displaced by 0·377(3) and −0·154(2) Å respectively whereas in molecule *B* they are displaced by −0·187 (3) and 0·248 (2) Å respectively from the best three-atom planes of the dioxolane rings. Thus the dioxolane ring assumes a twist conformation in both molecules.

Molecular packing: The crystal structure is stabilized by two hydrogen bonds, N(3)—H···O(4) [2·832 (3) Å and 174·36°] and N(3)—H···O(3′) [2·893 (2) Å and 175·66°]. This seems to be the first isopropylidene nucleoside to show such an H bond involving the isopropylidene group. These two hydrogen bonds are shown in Fig. 2.

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Ionic Compounds from the 2:1 Addition of Tropolone* and 4-Hydroxy-3-methoxybenzaldehyde to 1,7,10,16-Tetraoxa-4,13-diazacyclooctadecane

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Abstract. $C_{12}H_{28}N_2O_4^{2+}.2C_7H_5O_2^-$ (1), $M_r = 506{\cdot}59$, triclinic, $P\bar{1}$, $a = 7{\cdot}513$ (3), $b = 9{\cdot}165$ (5), $c = 10{\cdot}458$ (5) Å, $\alpha = 81{\cdot}42$ (4), $\beta = 72{\cdot}75$ (3), $\gamma = 78{\cdot}47$ (4)°, $V = 670{\cdot}6$ (6) Å^3, $Z = 1$, $D_x = 1{\cdot}25$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}87$ cm^{-1}, $F(000) = 272$, $T = 300$ K, $R = 0{\cdot}047$ for 970 unique observed reflections. $C_{12}H_{28}N_2O_4^{2+}.2C_8H_7O_3^-$ (2), $M_r = 566{\cdot}65$, triclinic, $P\bar{1}$, $a = 7{\cdot}400$ (2), $b = 9{\cdot}111$ (3), $c = 10{\cdot}704$ (3) Å, $\alpha = 94{\cdot}21$ (2), $\beta = 93{\cdot}38$ (2), $\gamma = 94{\cdot}91$ (2)°, $V = 715{\cdot}6$ (3) Å^3, $Z = 1$, $D_x = 1{\cdot}31$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}93$ cm^{-1}, $F(000) = 304$, $T = 300$ K, $R = 0{\cdot}0437$ for 1542 unique observed reflections. The 'diaza-18-crown-6' dications adopt different conformations in the two structures. The conformer in (1) resembles that of free 18-crown-6; however, the conformer in (2) has not been observed previously. The tropolonate anion is planar and participates in a bifurcated hydrogen bond. The 4-hydroxy-3-methoxybenzaldehyde anion exhibits a quinoid-like arrangement of double bonds in the phenyl ring.

Introduction. In general, complexes between 1,7,10,16-tetraoxa-4,13-diazacyclooctadecane ('diaza-18-crown-6') and neutral organic molecules containing proton donors are stabilized by hydrogen bonds to the N atoms of the crown molecule. When Me_2SO solutions of tropolone and 'diaza-18-crown-6' (2:1 ratio) are mixed, a yellow solution is formed. This yellow solution arises because of proton transfer from the tropolone (3) to the 'diaza-18-crown-6' resulting in the formation of an ionic compound composed of a 'diaza-18-crown-6' dication and two tropolonate anions. The ^{13}C spectrum of pure tropolone contains only four lines which indicates the proton transfer between the two O atoms is fast on the NMR time scale (Weiler, 1972). In the crystal structure of tropolone (Shimanouchi & Sasada,

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* 2-Hydroxy-2,4,6-cycloheptatrien-1-one.

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