

Table 2. Bond distances (\AA) and angles ($^\circ$) for xanthone with e.s.d.'s in parentheses

C(11)—C(12)	1.39 (1)	C(21)—C(22)	1.40 (1)
C(12)—C(13)	1.35 (2)	C(22)—C(23)	1.35 (2)
C(13)—C(14)	1.37 (2)	C(23)—C(24)	1.37 (2)
C(14)—C(15)	1.38 (2)	C(24)—C(25)	1.37 (2)
C(15)—C(16)	1.42 (1)	C(25)—C(26)	1.41 (1)
C(16)—C(11)	1.39 (1)	C(26)—C(21)	1.38 (1)
O(1)—C(11)	1.36 (1)	O(1)—C(21)	1.37 (1)
C(7)—C(16)	1.45 (1)	C(7)—C(26)	1.48 (1)
C(7)—O(2)	1.22 (1)		
C(11)—C(12)—C(13)	118.1 (9)	C(21)—C(22)—C(23)	118.9 (9)
C(12)—C(13)—C(14)	124.0 (10)	C(22)—C(23)—C(24)	121.4 (10)
C(13)—C(14)—C(15)	118.4 (10)	C(23)—C(24)—C(25)	120.8 (10)
C(14)—C(15)—C(16)	120.3 (9)	C(24)—C(25)—C(26)	119.2 (9)
C(15)—C(16)—C(11)	118.4 (8)	C(25)—C(26)—C(21)	118.7 (8)
C(16)—C(11)—C(12)	120.7 (9)	C(26)—C(21)—C(22)	121.0 (9)
O(1)—C(11)—C(12)	116.3 (8)	O(1)—C(21)—C(22)	115.6 (8)
O(1)—C(11)—C(16)	123.0 (8)	O(1)—C(21)—C(26)	123.3 (8)
C(11)—C(16)—C(7)	119.3 (8)	C(21)—C(26)—C(7)	118.6 (8)
C(15)—C(16)—C(7)	122.3 (8)	C(25)—C(26)—C(7)	122.7 (8)
C(16)—C(7)—O(2)	123.4 (8)	C(26)—C(7)—O(2)	120.5 (8)
C(11)—O(1)—C(21)	119.4 (6)	C(16)—C(7)—C(26)	116.2 (7)

to that in the gas phase (Iijima, Misu, Ohnishi & Onuma, 1989), and we conclude that the p_z electrons of atoms O(1) and C(7) are used for conjugation in

the central ring and this conjugation makes the skeleton of the molecule planar.

The benzene rings are apparently not regular hexagons in shape. This distortion, however, appears to be symmetric, and the whole molecule has approximate C_2 symmetry.

References

- BISWAS, S. C. & SEN, R. K. (1982). *Indian J. Pure Appl. Phys.* **20**, 414–415.
- IIJIMA, K., MISU, T., OHNISHI, S. & ONUMA, S. (1989). *J. Mol. Struct.* **213**, 263–269.
- IIJIMA, T. (1972). *Bull. Chem. Soc. Jpn.* **45**, 3526–3530.
- International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagatu Kenkyusho Hokoku*, **55**, 69–77.
- TAMAGAWA, K., TAKEMURA, M., KONAKA, S. & KIMURA, M. (1984). *J. Mol. Struct.* **125**, 131–142.

Acta Cryst. (1990). **C46**, 1727–1730

Structure of 2,4,4,6-Tetraphenyl-4*H*-pyran

BY JAROSLAV VOJTEČHOVSKÝ AND JINDŘICH HAŠEK

Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, Heyrovský sq. 2, 162 06 Prague 6, Czechoslovakia

(Received 25 September 1989; accepted 5 January 1990)

Abstract. $C_{29}H_{22}O$, $M_r = 386.49$, monoclinic, $P2_1/c$, $a = 12.128 (5)$, $b = 12.372 (5)$, $c = 14.599 (5) \text{ \AA}$, $\beta = 105.57 (3)^\circ$, $V = 2110 (1) \text{ \AA}^3$, $Z = 4$, $D_m = 1.21 (1)$, $D_x = 1.217 (1) \text{ Mg m}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.5418 \text{ \AA}$, $\mu = 0.561 \text{ mm}^{-1}$, $F(000) = 816$, $T = 298 \text{ K}$, $R = 0.039$ for 2729 unique observed reflections. The 4*H*-pyran ring is roughly planar with the phenyl rings substituted on the $C(sp^2)$ atoms C2 and C6. The ring shows a slightly distorted boat conformation with the boat angles 4.3 (1) and 7.7 (1) $^\circ$ on $C(sp^2)$ and O, respectively. The observed conformation is compared with three other structures containing similar pyran rings.

Introduction. The structure of 2,4,4,6-tetraphenyl-4*H*-pyran has been determined by the X-ray method because by analogy with three similar structures which were available from the Cambridge Structural Database (Allen *et al.*, 1979; release 1988), neither

the conformation of the 4*H*-pyran moiety nor the orientation of its substituents could be predicted unambiguously. The photochromic behaviour is similar to that of differently substituted 1,4-dihydropyridines observed by Iwasaki, Watanabe & Maeda (1987). The molecular geometry is considered to be significant for the explanation of photochromic properties.

Experimental. 2,4,4,6-Tetraphenyl-4*H*-pyran crystallized as colourless, well developed crystals from an oxygen-free benzene solution. The crystals turn blue in light and X-rays, but no changes in diffracted intensities were observed. The density was determined by flotation in an aqueous KBr solution.

The crystal used was a prism with dimensions $0.6 \times 0.3 \times 0.3 \text{ mm}$. Weissenberg photographs confirmed the good quality of the crystal and indicated

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

	x	y	z	U_{eq} (\AA^2)
C1	0.2724 (1)	0.0456 (1)	0.9590 (1)	0.0415 (5)
C2	0.1951 (1)	-0.0319 (1)	0.9327 (1)	0.0459 (6)
C3	0.2040 (1)	-0.1244 (1)	0.8673 (1)	0.0438 (5)
C4	0.3027 (1)	-0.0997 (1)	0.8258 (1)	0.0476 (5)
C5	0.3758 (1)	-0.0192 (1)	0.8540 (1)	0.0425 (5)
C6	0.4754 (1)	0.0073 (1)	0.8178 (1)	0.0448 (5)
C7	0.5421 (1)	0.0986 (2)	0.8505 (1)	0.0552 (6)
C8	0.6370 (2)	0.1220 (2)	0.8183 (1)	0.0658 (8)
C9	0.6664 (2)	0.0563 (2)	0.7530 (1)	0.0676 (8)
C10	0.6013 (2)	-0.0343 (2)	0.7195 (1)	0.0664 (8)
C11	0.5074 (2)	-0.0591 (2)	0.7519 (1)	0.0549 (6)
C12	0.2720 (1)	0.1337 (1)	1.0276 (1)	0.0441 (6)
C13	0.3553 (1)	0.2141 (1)	1.0442 (1)	0.0552 (6)
C14	0.3568 (2)	0.2956 (2)	1.1097 (1)	0.0640 (7)
C15	0.2751 (2)	0.2989 (2)	1.1591 (1)	0.0663 (7)
C16	0.1916 (2)	0.2204 (2)	1.1431 (1)	0.0659 (8)
C17	0.1902 (2)	0.1383 (2)	1.0785 (1)	0.0567 (7)
C18	0.2244 (1)	-0.2285 (1)	0.9278 (1)	0.0440 (5)
C19	0.1412 (2)	-0.2653 (1)	0.9702 (1)	0.0532 (6)
C20	0.1592 (2)	-0.3556 (2)	1.0276 (1)	0.0652 (6)
C21	0.2613 (2)	-0.4115 (2)	1.0444 (1)	0.0686 (7)
C22	0.3437 (2)	-0.3767 (2)	1.0028 (1)	0.0701 (8)
C23	0.3259 (1)	-0.2859 (2)	0.9452 (1)	0.0561 (6)
C24	0.0951 (1)	-0.1337 (1)	0.7839 (1)	0.0436 (5)
C25	0.0604 (1)	-0.2316 (1)	0.7392 (1)	0.0525 (6)
C26	-0.0348 (2)	-0.2378 (2)	0.6610 (1)	0.0626 (7)
C27	-0.0965 (2)	-0.1468 (2)	0.6262 (1)	0.0649 (8)
C28	-0.0631 (2)	-0.0491 (2)	0.6694 (1)	0.0702 (8)
C29	0.0329 (2)	-0.0426 (2)	0.7472 (1)	0.0609 (7)
O30	0.3666 (1)	0.0524 (1)	0.9237 (1)	0.0574 (4)

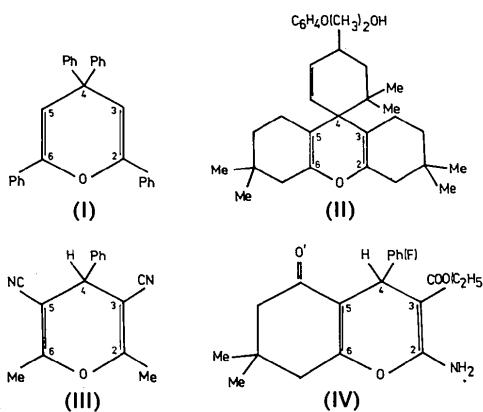
space group $P2_1/c$. The lattice parameters were refined from 15 centered reflections ($12 < 2\theta < 27^\circ$). A total of 3144 unique and 2729 observed reflections [$I > 1.96\sigma(I)$] were collected by $\theta-2\theta$ scan, using a Syntex $P2_1$ diffractometer. Intensities were measured up to $2\theta = 120^\circ$ with $h = -13/13$, $k = 0/13$, $l = 0/16$; only a unique data set was collected. Three standard reflections (200, 132, 202) showed no significant fluctuations during the measurement.

The phase problem was solved by *MULTAN87* (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987) and refined by *SHELX76* (Sheldrick, 1976). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The atomic coordinates and the anisotropic temperature parameters were freely refined for all the non-H atoms; the H atoms were refined isotropically. The function minimized was $\sum(w|\Delta F|^2)$, where $w = [\sigma^2(F_o) + 0.0009|F_o|^2]^{-1}$. The refinement of 358 parameters in full-matrix mode was finished when all shifts fell below 0.15 of their e.s.d.'s. An empirical isotropic extinction factor was refined to 1.14×10^{-6} . Final $R = 0.039$, $wR = 0.057$, $S = 1.509$. Maximum and minimum peaks in the final difference map were 0.13 and -0.18 e \AA^{-3} . No significant correlations among refined parameters were observed. The geometry was calculated with *PARST* (Nardelli, 1983). The final positional parameters and equivalent

isotropic thermal parameters for non-H atoms are listed in Table 1.*

Discussion. The molecular structure of 2,4,4,6-tetraphenyl-4H-pyran with the atom numbering is shown in Fig. 1. Bond distances and angles are listed in Table 2 and selected torsion angles are listed in Table 3.

The geometries of the 4H-pyran rings in four compounds are compared in Table 3 [(I) title compound, (II) 2-(3,3,5',5',6,6-hexamethyl-1,8-dioxo-1,2,3,4,5,6,7,8-octahydroxanthen-9-spirocyclohex-2'-en-3'-yl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (Declercq, Germain, van Meerssche, Greenhill, Chaaban & de Kimpe, 1981), (III) 2,6-dimethyl-4-phenyl-4H-pyran-3,5-dicarbonitrile (Florencio & Garcia-Blanco, 1987) and (IV) 2-amino-7,7-dimethyl-4-(3-fluorophenyl)-5-oxo-3-ethoxycarbonyl-5,6,7,8-tetrahydro-4H-benzo[b]pyran (Sharanina *et al.*, 1986)].



* Lists of refined parameters for H atoms, anisotropic temperature parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52930 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

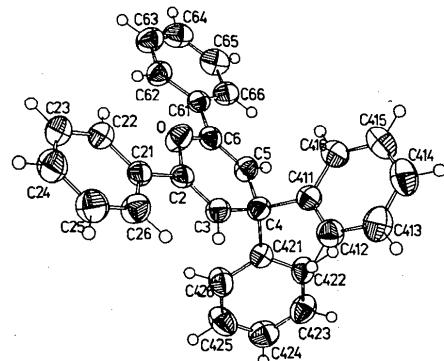


Fig. 1. Molecular structure with the atom numbering.

Table 2. Bond distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

C2—C3	1.324 (2)	C2—C21	1.481 (2)
C2—O	1.376 (2)	C3—C4	1.513 (2)
C4—C5	1.511 (2)	C4—C411	1.544 (2)
C4—C421	1.542 (2)	C5—C6	1.323 (2)
C6—C61	1.479 (2)	C6—O	1.376 (2)
C21—C22	1.392 (2)	C21—C26	1.391 (3)
C22—C23	1.386 (3)	C23—C24	1.374 (3)
C24—C25	1.377 (4)	C25—C26	1.383 (3)
C61—C62	1.397 (3)	C61—C66	1.397 (3)
C62—C63	1.386 (3)	C63—C64	1.371 (3)
C64—C65	1.383 (3)	C65—C66	1.380 (4)
C411—C412	1.394 (3)	C411—C416	1.384 (2)
C412—C413	1.378 (3)	C413—C414	1.382 (3)
C414—C415	1.371 (3)	C415—C416	1.385 (3)
C421—C422	1.387 (2)	C421—C426	1.382 (3)
C422—C423	1.391 (3)	C423—C424	1.372 (3)
C424—C425	1.373 (3)	C425—C426	1.394 (3)
C21—C2—O	111.2 (2)	C61—C66—C65	120.9 (2)
C3—C2—C21	126.8 (2)	C2—C21—C22	120.6 (2)
C3—C4—C421	111.3 (2)	C22—C21—C26	117.8 (2)
C3—C4—C411	107.3 (1)	C21—C22—C23	121.0 (2)
C411—C4—C421	111.2 (2)	C21—C26—C25	121.0 (2)
C5—C4—C421	107.5 (1)	C4—C411—C412	120.2 (2)
C5—C4—C411	112.1 (2)	C412—C411—C416	117.7 (2)
C5—C6—C61	126.8 (2)	C411—C412—C413	121.3 (3)
C61—C6—O	111.0 (2)	C411—C416—C415	120.9 (2)
C6—C61—C62	120.7 (2)	C4—C421—C422	121.6 (2)
C62—C61—C66	117.9 (2)	C422—C421—C426	117.7 (2)
C61—C62—C63	120.7 (2)	C421—C422—C423	121.0 (2)

Table 3. Comparison of the geometry (\AA , $^\circ$) of 4H-pyran with e.s.d.'s in parentheses

	(I)	(II)	(III)	(IV)
C2—C3	1.324 (2)	1.335 (7)	1.329 (6)	1.359 (4)
C5—C6	1.323 (2)	1.322 (7)	1.329 (6)	1.337 (3)
O—C2	1.376 (2)	1.385 (6)	1.382 (5)	1.371 (3)
C6—O	1.376 (2)	1.378 (6)	1.391 (5)	1.376 (3)
C3—C4	1.513 (2)	1.547 (7)	1.518 (6)	1.518 (4)
C4—C5	1.510 (2)	1.539 (7)	1.512 (5)	1.505 (3)
C3—C4—C5	107.4 (2)	105.4 (4)	107.3 (3)	110.1 (2)
C2—C3—C4	124.5 (2)	120.5 (5)	124.5 (4)	120.7 (2)
C4—C5—C6	124.3 (2)	120.6 (5)	125.8 (4)	121.7 (2)
C5—C6—O	122.3 (2)	121.9 (5)	120.7 (3)	122.8 (2)
O—C2—C3	122.0 (2)	120.9 (5)	122.0 (4)	122.6 (2)
C6—O—C2	118.5 (2)	117.4 (4)	119.3 (3)	118.8 (2)
C4—C5—C6—O	-1.6 (3)	7.0 (8)	-3.1 (6)	4.5 (5)
O—C2—C3—C4	4.1 (3)	-8.0 (8)	0.8 (7)	-5.4 (6)
C2—O—C6—C5	-5.9 (3)	22.9 (8)	-2.7 (5)	9.8 (5)
C6—O—C2—C3	4.7 (3)	-22.2 (9)	3.8 (6)	-9.3 (5)
C2—C3—C4—C5	-10.0 (3)	32.7 (8)	-5.4 (6)	17.5 (6)
C3—C4—C5—C6	8.8 (3)	-32.3 (9)	6.6 (5)	-17.2 (6)
Boat angle A, B	7.7 (1)	27.6 (5)	4.9 (3)	14.8 (3)
Boat angle A, C	4.3 (1)	19.1 (4)	2.7 (3)	8.0 (3)
Fold angle	8.2 (1)	30.7 (5)	5.1 (4)	14.8 (4)

A is the mean plane of C2, C3, C5 and C6, B is the plane of C3, C4 and C5, and C is the plane of C2, O and C6.

In all four structures the pyran ring has a boat conformation with the apexes $\text{C}(\text{sp}^3)$ and O, and in which the torsion angles around $\text{C}(\text{sp}^3)$ ring bonds are wider than those around O ring bonds. Fig. 2 shows a plot of the average torsion angle at the O

against that at the $\text{C}(\text{sp}^3)$ atom; (II) has the largest differences. A correlation between the ring distortion of both apexes in the similar case of dihydropyridines is discussed by Fossheim, Svarteng, Mostad, Rømming, Shefter & Triggle (1982). The greatest difference between the bond angles at $\text{C}(\text{sp}^3)$ and their ideal value is also found in the case of (II). A comparison of dihedral angles between the planes A, B and A, C (boat angles, see Table 3) and dihedral angles between both $\text{O}=\text{C}(\text{sp}^2)=\text{C}(\text{sp}^2)=\text{C}(\text{sp}^2)$ planes (fold angles) shows that the ring distortions of (II) and (IV) are considerably higher than those of (I) and (III). However, the planarity of the double-bond system is almost retained; the deviations of four $\text{C}(\text{sp}^2)$ atoms from the respective mean plane A are less than 0.006 \AA for all structures. The double-bond length C2—C3 of (IV) is elongated by the effect described by Sharanina *et al.* (1986).

The mean phenyl planes of 2,4,4,6-tetraphenyl-4H-pyran form dihedral angles 3.2, 71.8, 75.2 and 3.0 (1) $^\circ$ for the pivot atoms C61, C411, C421 and C21, respectively, with plane A (the corresponding χ^2 values for ring planarity range from 8 to 21). The bonds C6—C61, C4—C411, C4—C421 and C2—C21 form angles 3.0, 62.9, 48.0 and 1.1 (1) $^\circ$, respectively, with plane A. Asymmetry in the positioning of phenyls at $\text{C}(\text{sp}^3)$ is similar to the case of 1-methyl-2,4,4,6-tetraphenyl-1,4-dihydropyridine (Hašek & Ondráček, 1990). This follows from a comparison of the angles C3—C4—C421, 111.3 (2),

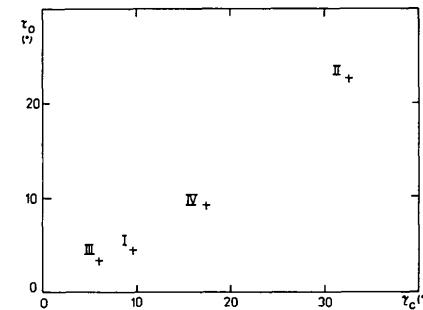


Fig. 2. A plot of the 4H-pyran ring distortion of O against that at $\text{C}(\text{sp}^3)$ [τ_O is the average torsion angle $|\text{C}(\text{sp}^2)=\text{C}(\text{sp}^2)=\text{O}=\text{C}(\text{sp}^2)|$, τ_C is the average torsion angle $|\text{C}(\text{sp}^2)=\text{C}(\text{sp}^2)=\text{C}(\text{sp}^3)=\text{C}(\text{sp}^3)|$].

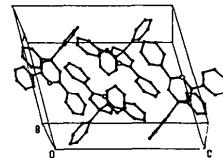
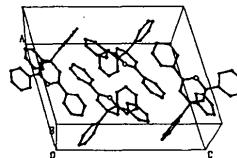


Fig. 3. Stereoscopic view of the unit cell (the H atoms have been omitted for clarity).

C5—C4—C411, 112.1 (2) $^{\circ}$ with C3—C4—C411, 107.3 (2), C5—C4—C421, 107.5 (2) $^{\circ}$.

Fig. 3 shows a stereoscopic view of the unit cell. There are no significant contacts shorter than 3.4 Å. No tendency for parallel stacking of the phenyl rings of symmetry-related molecules was observed.

The authors thank Dr S. Nešpůrek, Dr K. Huml and Ing J. Ječný for valuable discussions.

References

- ALLEN, F. H., BELLARD, S., BRICE, M. D., CARTWRIGHT, G. A., DOUBLEDAY, A., HIGGS, H., HUMMELINK, T., HUMMELINK-PETERS, B. J., KENNARD, O., MOTHERWELL, W. D. S., RODGERS, J. R. & WATSON, D. G. (1979). *Acta Cryst. B35*, 2331–2339.
- DEBAERDEMAEKER, T., GERMAIN, G., MAIN, P., TATE, C. & WOOLFSON, M. M. (1987). *MULTAN87. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- DECLERCQ, J.-P., GERMAIN, G., VAN MEERSCHE, M., GREENHILL, J. V., CHAABAN, I. & DE KIMPE, N. (1981). *Cryst. Struct. Commun.* **10**, 1335–1341.
- FLORENCIO, F. & GARCIA-BLANCO, S. (1987). *Acta Cryst. C43*, 1430–1432.
- FOSSHØM, R., SVARTENG, K., MOSTAD, A., RØMMING, C. H., SHEFTER, E. & TRIGGLE, D. J. (1982). *J. Med. Chem.* **25**, 126–131.
- HÁSEK, J. & ONDRÁČEK, J. (1990). *Acta Cryst. C46*, 1256–1259.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- IWASAKI, F., WATANABE, T. & MAEDA, K. (1987). *Bull. Chem. Soc. Jpn.* **60**, 1255–1259.
- NARDELLI, M. (1983). *PARST. A System of Computer Routines for Calculating Molecular Parameters from the Results of Crystal Structure Analysis*. Univ. of Parma, Italy.
- SHARANINA, G., NESTEROV, V. N., KLOKOL, G. V., RODINOVSKAYA, L. A., SHKLOVER, V. E., SHARANIN, YU. A., STRUCHKOV, YU. T. & PROMONENKOV, V. K. (1986). *Zh. Org. Khim.* **22**, 1315–1322.
- SHEDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1990). **C46**, 1730–1732

Structure of a Nucleoside Analogue, 3'-Deoxy-3'-fluorothymidine*

BY D. H. EVERAERT, O. M. PEETERS, N. M. BLATON AND C. J. DE RANTER†

Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Instituut voor Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium

(Received 25 September 1989; accepted 16 January 1990)

Abstract. 1-(2,3-Dideoxy-3-fluoro- β -D-erythro-pentofuranosyl)thymine, $C_{10}H_{13}FN_2O_4$, $M_r = 244.22$, monoclinic, $P2_1$, $a = 6.408$ (14), $b = 18.716$ (26), $c = 9.329$ (7) Å, $\beta = 98.4$ (1) $^{\circ}$, $V = 1107$ (3) Å 3 , $Z = 4$, $D_m = 1.46$, $D_x = 1.465$ Mg m $^{-3}$, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.1169$ mm $^{-1}$, $F(000) = 512$, $T = 298$ K, final $R = 0.035$ for 1425 unique observed reflections. The asymmetric unit contains two molecules (*A* and *B*). For molecule *A*: the *N*-glycosidic torsion angle χ has a value of -138.4 (5) $^{\circ}$ in the *anti* range; the sugar pucker is 2E with $P = 164$ (1) $^{\circ}$ and $\psi_m = 36$ (1) $^{\circ}$ and the C4'—C5' conformation is +sc with $\gamma = 50.2$ (7) $^{\circ}$. For molecule *B*: the *N*-glycosidic torsion angle χ has a value of -159.6 (5) $^{\circ}$ in the *anti* range; the sugar pucker is 2T_3 with $P = 169$ (1) $^{\circ}$ and $\psi_m = 32$ (1) $^{\circ}$ and the C4'—C5' conformation is +sc with $\gamma = 52.8$ (7) $^{\circ}$. The conformational parameters are in

accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines. Base-pair formation occurs between the two molecules *A* and *B*.

Introduction. This structure was determined as part of an investigation of potentially antiviral nucleoside analogues, with particular reference to possible anti-AIDS compounds.

Experimental. Colourless prismatic crystals obtained at room temperature from a methanol–ether solution, dimensions $\sim 0.6 \times 0.3 \times 0.2$ mm. Density measured by flotation in *n*-heptane/CCl₄. Stadi-4 computer-controlled four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique ($2\theta_{\max} = 50^{\circ}$, $-8 \leq h \leq 0$, $-22 \leq k \leq 22$, $-11 \leq l \leq 11$). Cell dimensions by least-squares refinement of the setting angles of 24 reflections with $17 < 2\theta < 25^{\circ}$, space group $P2_1$ from systematic absences $0k0$ for k odd. Four standard reflections ($\bar{1}0\bar{3}$, $\bar{1}02$, $\bar{2}51$, $0\bar{3}\bar{2}$) monitored every 120 min showed no significant decrease in intensity per hour, 4288

* Structural Studies of Modified Nucleosides. Part II. Part I: Van Aerschot, Everaert, Peeters, Blaton, De Ranter & Herdewijn (1990).

† To whom correspondence should be addressed.