

C5—C4—C411, 112.1 (2)° with C3—C4—C411, 107.3 (2), C5—C4—C421, 107.5 (2)°.

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.

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

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**Abstract.** 1-(2,3-Dideoxy-3-fluoro- $\beta$ -D-erythro-pentofuranosyl)thymine, C<sub>10</sub>H<sub>13</sub>FN<sub>2</sub>O<sub>4</sub>,  $M_r = 244.22$ , monoclinic,  $P2_1$ ,  $a = 6.408$  (14),  $b = 18.716$  (26),  $c = 9.329$  (7) Å,  $\beta = 98.4$  (1)°,  $V = 1107$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.46$ ,  $D_x = 1.465$  Mg m<sup>-3</sup>, graphite-monochromated Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å,  $\mu = 0.1169$  mm<sup>-1</sup>,  $F(000) = 512$ ,  $T = 298$  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  $\chi$  has a value of  $-138.4$  (5)° in the *anti* range; the sugar pucker is <sup>2</sup>*E* with  $P = 164$  (1)° and  $\psi_m = 36$  (1)° and the C4'—C5' conformation is *+sc* with  $\gamma = 50.2$  (7)°. For molecule *B*: the *N*-glycosidic torsion angle  $\chi$  has a value of  $-159.6$  (5)° in the *anti* range; the sugar pucker is <sup>2</sup>*T*<sub>3</sub> with  $P = 169$  (1)° and  $\psi_m = 32$  (1)° and the C4'—C5' conformation is *+sc* with  $\gamma = 52.8$  (7)°. 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/CCl<sub>4</sub>. 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 ( $\bar{1}0\bar{3}$ ,  $10\bar{2}$ ,  $\bar{2}51$ ,  $03\bar{2}$ ) monitored every 120 min showed no significant decrease in intensity per hour, 4288

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reflections measured, 2021 unique reflections ( $R_{\text{int}} = 0.020$ ) of which 1428 considered as observed [ $F > 4\sigma(F)$ ]. Three reflections (040, 100, 10 $\bar{2}$ ) badly affected by extinction were eliminated. Lorentz-polarization corrections, no absorption corrections, scattering factors from *International Tables for X-ray Crystallography* (1974) and Stewart, Davidson & Simpson (1965) (for H). The structure was solved by *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The *E* map calculated from the solution with the best figure of merit revealed 17 of the 34 non-H atoms in the asymmetric unit. The remaining atoms were obtained from a subsequent Fourier synthesis. A difference synthesis revealed the position of all H atoms, which were refined with fixed isotropic temperature factors ( $B_{\text{eq}} = 4 \text{ \AA}^2$ ). All other atoms were refined anisotropically on *F* by full-matrix least squares. The refinement converged at  $R = 0.035$ ,  $wR = 0.045$ ,  $S = 0.284$ .  $w = (C_0 + C_1|F_o| + C_2|F_o|^2 + C_3|F_o|^3)^{-1}$ , where  $C_0 = 1000$ ,  $C_1 = 1$ ,  $C_2 = 0.00004$ ,  $C_3 = 0.000009$ . 385 refined parameters, maximum shift/e.s.d. = 0.03, minimum and maximum electron density  $-0.194$  and  $0.152 \text{ e \AA}^{-3}$ . The number of reflections per refined variable was  $1425/385 = 3.7$ . All calculations were performed on a PDP 11/73 micro-computer using *SDP/PDP* (Enraf-Nonius, 1982) and *PARST* (Nardelli, 1983).

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters with e.s.d.'s of the refined parameters in parentheses

$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}(\text{\AA}^2)$
N1A	4245 (6)	3740*	2103 (4)	3.45 (8)
C2A	5720 (8)	4252 (2)	2591 (4)	3.9 (1)
O2A	7416 (6)	4109 (2)	3316 (4)	4.95 (8)
N3A	5114 (6)	4940 (2)	2217 (4)	3.74 (8)
C4A	3227 (8)	5157 (2)	1454 (4)	3.8 (1)
O4A	2907 (6)	5789 (2)	1169 (4)	5.64 (9)
C5A	1711 (7)	4595 (2)	1039 (4)	3.5 (1)
C6A	2286 (7)	3929 (2)	1382 (4)	3.3 (1)
C7A	-400 (9)	4793 (3)	249 (6)	4.7 (1)
C1'A	4730 (8)	2983 (2)	2449 (5)	3.8 (1)
C2'A	3522 (8)	2692 (2)	3603 (4)	3.9 (1)
C3'A	3329 (8)	1912 (2)	3202 (4)	3.8 (1)
F3'A	5224 (5)	1568 (1)	3749 (3)	6.10 (7)
C4'A	3086 (8)	1916 (2)	1559 (4)	4.0 (1)
O4'A	4008 (6)	2587 (2)	1183 (3)	4.26 (7)
C5'A	880 (9)	1857 (3)	812 (5)	4.8 (1)
O5'A	-415 (6)	2361 (2)	1383 (4)	5.75 (9)
N1B	3741 (6)	3166 (2)	7697 (3)	3.31 (8)
C2B	5178 (7)	2648 (2)	8209 (4)	3.49 (9)
O2B	6940 (5)	2793 (2)	8818 (3)	4.37 (7)
N3B	4483 (6)	1966 (2)	7928 (4)	3.66 (8)
C4B	2584 (8)	1756 (2)	7156 (5)	3.8 (1)
O4B	2199 (6)	1114 (2)	6914 (4)	5.37 (9)
C5B	1155 (8)	2325 (2)	6664 (4)	3.7 (1)
C6B	1811 (8)	2997 (2)	6963 (5)	3.9 (1)
C7B	-950 (9)	2152 (3)	5834 (6)	5.3 (1)
C1'B	4475 (8)	3916 (2)	7853 (4)	3.6 (1)
C2'B	5694 (8)	4146 (2)	6678 (5)	4.4 (1)
C3'B	5188 (8)	4928 (2)	6522 (5)	4.1 (1)
F3'B	6599 (5)	5299 (1)	7550 (3)	6.26 (7)
C4'B	3008 (8)	4989 (2)	6889 (5)	4.0 (1)
O4'B	2715 (5)	4363 (2)	7766 (3)	4.29 (7)
C5'B	1302 (9)	5019 (3)	5619 (6)	5.3 (1)
O5'B	1503 (6)	4435 (2)	4690 (4)	6.16 (9)

\*Parameter kept fixed for origin definition.

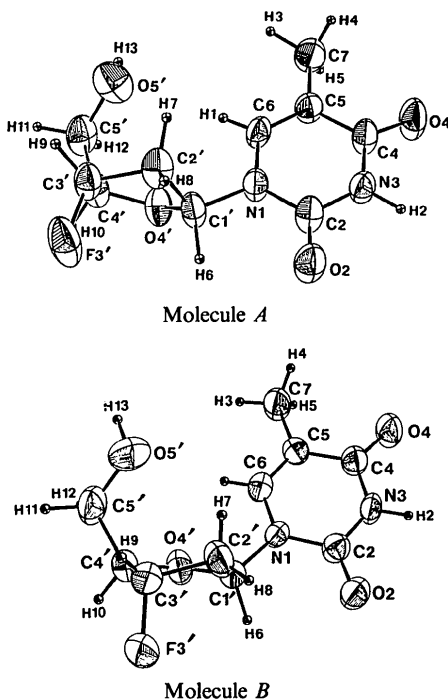


Fig. 1. A view of molecules *A* and *B* of the title compound with 50% probability anisotropic displacement ellipsoids for the non-H atoms and atomic numbering scheme.

**Discussion.** An *ORTEP* view (Johnson, 1976) of molecules *A* and *B* with the atomic numbering scheme is shown in Fig. 1.\* The final fractional atomic coordinates are given in Table 1. Bond lengths, bond angles and some selected torsion angles are given in Table 2. Fig. 2 shows the intramolecular hydrogen bonds and the classical intermolecular hydrogen bonds. Table 3 gives the geometry of all hydrogen bonds. Between the two molecules *A* and *B* there is base-pair formation including two so-called 'cyclic' N3—H...O4 hydrogen bonds ( $-x + 1, y + \frac{1}{2}, -z + 1$ ;  $-x + 1, y - \frac{1}{2}, -z + 1$ ) in order to produce a stable base pair; the geometrical characteristics of the hydrogen bonds involved in this base-base interaction are all within the normal limits (Saenger, 1984).

Both O5'*A* and O5'*B* donate their H13 to the O2 of a translational symmetry equivalent ( $x - 1, y, z - 1$ ;  $x - 1, y, z$ ). These hydrogen bonds create a net-

\* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, least-squares planes, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52900 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

