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Structure of 3′-*O*-Acetylthymidine

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Abstract. $C_{12}H_{16}N_2O_6$, $M_r = 284{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}485\,(4)$, $b = 14{\cdot}043\,(7)$, $c = 17{\cdot}145\,(12)$ Å, $U = 1320{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}43$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}08$ cm^{-1}, $F(000) = 600$, $T = 293$ K, $R = 0{\cdot}049$ for 1769 observed reflections. The molecule adopts an *anti* conformation, with a sugar pucker of $^2T_3$ [C(2′)-*endo*/C(3′)-*exo*].

Introduction. The structure determination was performed as part of a series of studies of acetylated nucleosides intended to investigate base-pairing patterns, propeller twisting and acetyl conformations in such compounds. Earlier work has revealed possible biologically interesting acetyl conformations (Wilson & Tollin, 1985) and induced base-pairing in such substituted nucleosides (Wilson, 1985). Since both features of acetyl–base stacking and base-pairing are present in the structure of the closely related compound 3′,5′-di-*O*-acetylthymidine (Wilson, Low, Tollin & Wilson, 1984), it was hoped that the title compound would also show these features.

Experimental. The crystals used were grown from aqueous solution and that used in the structural determination had dimensions 1·2 × 0·34 × 0·19 mm. Cell dimensions and orientation were obtained from the 254, 082, 0,0,10 and symmetry-related reflections. Data were collected on a Nicolet *P*3 four-circle diffractometer in Aberdeen. 2168 reflections were measured using Mo $K\alpha$ radiation. Data collection parameters: $0 < 2\theta < 60°$, $\theta/2\theta$ scans with a 2θ scan range of 2·41–2·8° depending on 2θ value, and a variable scan rate in the range 5·33° min^{-1} ($I < 150$) to 58·6° min^{-1} ($I > 2500$). Range of indices: $h \le 7$, $k \le 19$, $l \le 24$. The reflections 284 and 082 were measured every 50 reflections and showed no significant change in their intensities. No absorption or extinction corrections were applied.

Structure solution and refinement. The Patterson-methods program *PATMET* (Wilson & Tollin, 1986) was used to obtain an oriented model of the planar base group in the structure which was used as *ab initio* structural knowledge (Main, 1976) in the direct-methods package *MITHRIL* (Gilmore, 1984). A direct-methods run led to the appearance of most non-hydrogen atoms in the first calculated *E* map. Fourier recycling completed the definition of the structure, and a series of difference Fourier maps revealed all the H-atom positions. All non-hydrogen atoms were refined anisotropically and the H atoms isotropically. In the final stages of refinement all H parameters were fixed. Refinement was by least squares on *F* using the *BLOCK* program in the *GX* system (Mallinson & Muir, 1985). Final refinement parameters: $R = 0{\cdot}049$ for 1769 observed reflections [$I > 3\sigma(I)$]; $wR = 0{\cdot}053$, $w = 1/[\sigma(F)]^2$; 181 parameters refined; max. shift/e.s.d. = 0·022; max., min. difference peaks = 0·22 and −0·27 e Å^{-3}. Scattering factors taken from *International Tables for X-ray Crystallography* (1974). The *PLUTO* program (Motherwell & Clegg, 1978) was used for structure drawing. All other calculations were carried out using the *GX* package. All computations were performed on

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the Rutherford Appleton Laboratory Neutron Division HUB computer, a VAX 8600.

Discussion. The atomic numbering used is shown in the perspective drawing (Fig. 1) with atomic parameters, bond lengths and angles given in Tables 1 and 2.*

The only noteworthy feature in the bond parameters is that the ring exocyclic carbonyl bonds are somewhat longer [C(2)—O(2), 1·205 (4); C(4)—O(4), 1·234 (4) Å] than the acetyl carbonyl bond [C(3′1)—O(3′1), 1·188 (6) Å]. The short value for this latter bond length is in agreement with other acetyl carbonyl bond lengths in this series of compounds (Wilson, 1985). The length of these short carbonyl bonds seems not to be related to the thermal vibration parameters on either the C or O of the carbonyl group (Wilson & Tollin, 1988).

The pyrimidine base and the acetyl group are approximately planar, as expected, with the largest deviations from the mean planes being atom N(1) [0·03 (1) Å] in the base and atom O(3′) [0·02 (1) Å] in the acetyl group.

Molecular conformation. The molecule adopts an *anti* conformation about the N-glycosidic bond, with χ[C(2)—N(1)—C(1′)—O(4′)] = −123·5 (4)°, and exhibits a sugar pucker of $^2T_3$ [C(2′)-*endo*/C(3′)-*exo*] described by the pseudorotation parameters P = 173·9 (5)°, ψ_m = 26·0 (5)°. The C(4′)—C(5′) conformation is *gg* (*gauche–gauche*), with γ[O(5′)—C(5′)—C(4′)—C(3′)] = 50·1 (4)°. All of these conformational parameters are quite normal for a pyrimidine nucleoside.

Hydrogen bonding. Unlike the closely related 3′,5′-di-*O*-acetylthymidine (Wilson, Low, Tollin & Wilson, 1984) the structure does not exhibit base-pairing, there being only one hydrogen bond present between the bases. This is between O(4) and N(3) and is detailed as follows:

O(4)···N(3) (−0·5 + x, −0·5 − y, −z) 2·857 (3) Å
O(4)···H(3) (−0·5 + x, −0·5 − y, −z) 1·96 (2) Å.

The molecules pack in the crystal structure as shown in Fig. 2, with lines along **c** stacked in a head-to-head and tail-to-tail arrangement, with the head-to-head molecules linked by the hydrogen bond. The molecules

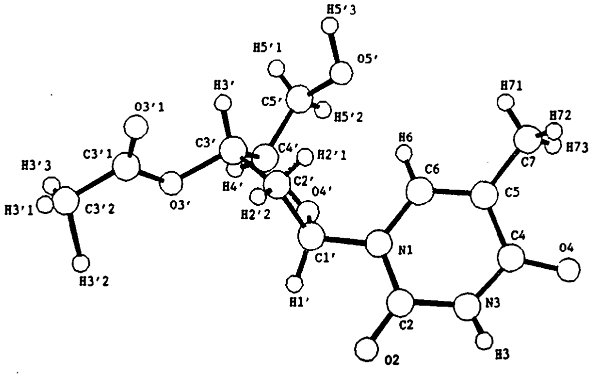

Fig. 1. Perspective view of the title compound, showing atomic numbering.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2), *with e.s.d.'s in the least significant digits in parentheses*

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
N(1)	−0·7310 (5)	−0·3528 (2)	0·2117 (1)	0·030 (2)
C(2)	−0·5469 (6)	−0·2947 (2)	0·1846 (2)	0·027 (2)
O(2)	−0·3998 (5)	−0·2547 (2)	0·2252 (1)	0·040 (1)
N(3)	−0·5457 (5)	−0·2850 (2)	0·1037 (1)	0·026 (1)
C(4)	−0·7040 (6)	−0·3266 (2)	0·0508 (2)	0·026 (2)
O(4)	−0·6716 (4)	−0·3122 (2)	−0·0195 (1)	0·032 (1)
C(5)	−0·8968 (5)	−0·3837 (2)	0·0842 (2)	0·029 (2)
C(7)	−1·0788 (8)	−0·4310 (3)	0·0319 (2)	0·040 (2)
C(6)	−0·9006 (6)	−0·3944 (2)	0·1624 (2)	0·030 (2)
C(1′)	−0·7337 (6)	−0·3784 (2)	0·2951 (2)	0·029 (2)
C(2′)	−0·9651 (7)	−0·3464 (2)	0·3380 (2)	0·035 (2)
C(3′)	−1·0259 (6)	−0·4279 (3)	0·3920 (2)	0·033 (2)
O(3′)	−0·9018 (5)	−0·4170 (2)	0·4669 (1)	0·041 (2)
C(3′1)	−1·0191 (8)	−0·3632 (2)	0·5212 (2)	0·042 (3)
O(3′1)	−1·2104 (6)	−0·3259 (2)	0·5099 (2)	0·059 (2)
C(3′2)	−0·8770 (11)	−0·3619 (3)	0·5956 (2)	0·068 (4)
C(4′)	−0·9147 (7)	−0·5155 (2)	0·3533 (2)	0·033 (2)
C(5′)	−1·0956 (8)	−0·5784 (3)	0·3113 (2)	0·045 (2)
O(5′)	−1·2472 (5)	−0·5265 (2)	0·2599 (2)	0·049 (2)
O(4′)	−0·7319 (5)	−0·4805 (2)	0·2995 (1)	0·034 (1)

Table 2. *Interatomic distances* (Å) *and angles* (°), *excluding* H *atoms*

N(1)—C(2)	1·380 (5)	N(1)—C(6)	1·386 (5)
N(1)—C(1′)	1·475 (4)	C(2)—O(2)	1·205 (4)
C(2)—N(3)	1·392 (4)	N(3)—C(4)	1·386 (5)
C(4)—O(4)	1·234 (4)	C(4)—C(5)	1·445 (5)
C(5)—C(7)	1·497 (5)	C(5)—C(6)	1·349 (5)
C(1′)—C(2′)	1·534 (5)	C(1′)—O(4′)	1·436 (4)
C(2′)—C(3′)	1·510 (5)	C(3′)—O(3′)	1·461 (4)
C(3′)—C(4′)	1·525 (5)	O(3′)—C(3′1)	1·361 (5)
C(3′1)—O(3′1)	1·188 (6)	C(3′1)—C(3′2)	1·495 (6)
C(4′)—C(5′)	1·512 (6)	C(4′)—O(4′)	1·449 (5)
C(5′)—O(5′)	1·414 (5)		

C(2)—N(1)—C(6)	122·4 (3)	C(2)—N(1)—C(1′)	118·6 (3)
C(6)—N(1)—C(1′)	118·8 (3)	N(1)—C(2)—O(2)	124·8 (3)
N(1)—C(2)—N(3)	113·3 (3)	O(2)—C(2)—N(3)	121·8 (3)
C(2)—N(3)—C(4)	127·5 (3)	N(3)—C(4)—O(4)	118·7 (3)
N(3)—C(4)—C(5)	115·6 (3)	O(4)—C(4)—C(5)	125·7 (3)
C(4)—C(5)—C(7)	119·8 (3)	C(4)—C(5)—C(6)	117·9 (3)
C(7)—C(5)—C(6)	122·3 (4)	N(1)—C(6)—C(5)	123·2 (3)
N(1)—C(1′)—C(2′)	113·7 (3)	N(1)—C(1′)—O(4′)	107·0 (3)
C(2′)—C(1′)—O(4′)	105·9 (3)	C(1′)—C(2′)—C(3′)	104·8 (3)
C(2′)—C(3′)—O(3′)	110·9 (3)	C(2′)—C(3′)—C(4′)	104·8 (3)
O(3′)—C(3′)—C(4′)	106·3 (3)	C(3′)—O(3′)—C(3′1)	116·1 (3)
O(3′)—C(3′1)—O(3′1)	123·3 (4)	O(3′)—C(3′1)—C(3′2)	110·2 (4)
O(3′)—C(3′1)—C(3′2)	126·5 (4)	C(3′)—C(4′)—C(5′)	114·6 (4)
C(3′)—C(4′)—O(4′)	106·3 (3)	C(5′)—C(4′)—O(4′)	110·4 (3)
C(4′)—C(5′)—O(5′)	112·5 (3)	C(1′)—O(4′)—C(4′)	111·5 (3)

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a full list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44744 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

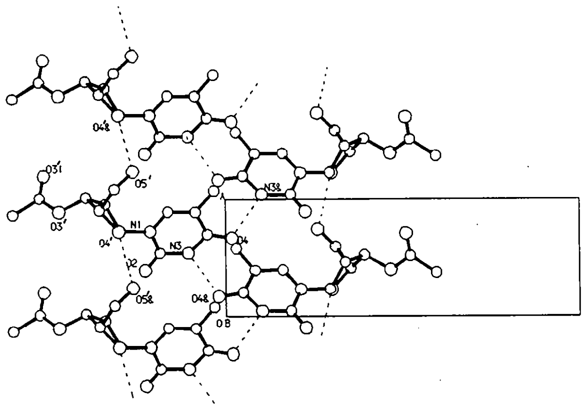

Fig. 2. The stacking in the title compound, showing the hydrogen-bonding scheme.

are linked along **a** by a hydrogen bond between O(4') and O(5'):

O(4')···O(5') (1 + *x*, *y*, *z*) 2·819 (4) Å
O(4')···H(5'3) (1 + *x*, *y*, *z*) 2·21 (3) Å.

Thus there are regions of bases and of sugar groups packing along **a**, as shown.

There are no other significant close contacts between the molecules in the structure. This goes against the strong trend in other acetylated pyrimidine and purine nucleosides for the C=O moiety of an acetyl group to stack above the base ring of a neighbouring molecule (Wilson, 1985). However, the interaction in 3',5'-di-*O*-acetylthymidine was the weakest of those found in the earlier work and while the 3'-acetyl group was involved it is not particularly surprising that in the less sterically congested mono-substituted title compound the interaction no longer plays a significant role. It has been found that in nucleoside structures the mono-acetylated compounds show less propensity to display this interaction.

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Ammonium Chloride Complexes with 18-Crown-6

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Abstract. (I): $2C_{12}H_{24}O_6.2NH_4^+.Cl^-.PF_6^-.0\cdot9CH_2Cl_2$, $M_r = 821\cdot6$, monoclinic, $P2_1/n$, $a = 8\cdot460$ (2), $b = 22\cdot852$ (7), $c = 21\cdot604$ (4) Å, $\beta = 92\cdot77$ (1)°, $V = 4172$ (2) Å^3, $Z = 4$, $D_x = 1\cdot31$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 3\cdot09$ mm^{-1}, $F(000) = 1752$, room temperature, $R = 0\cdot114$ for 2127 observed reflections with $|F_o| > 3\sigma(|F_o|)$. (II): $C_{12}H_{24}O_6.NH_4^+.Cl^-.2H_2O$, $M_r = 353\cdot8$, orthorhombic, *Pnam* (equivalent positions, $-x, -y, \frac{1}{2}+z$; $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}+z$; $\frac{1}{2}+x, \frac{1}{2}-y, z$), $a = 9\cdot751$ (1), $b = 12\cdot844$ (1), $c = 14\cdot972$ (2) Å, $V = 1875$ (1) Å^3, $Z = 4$, $D_x = 1\cdot26$ Mg m^{-3}, λ(Co $K\alpha$) = 1·54178 Å, $\mu = 2\cdot13$ mm^{-1}, $F(000) = 768$, room

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