Table 1. *Positional (fractional* × 10^4*) and equivalent isotropic* ($U \times 10^3$ Å^2) *thermal parameters, with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}$(trace of diagonalized U tensor).

	x	*y*	*z*	U_{eq}
C1	6829 (4)	305 (3)	3738 (4)	68
C2	5566 (5)	369 (5)	3228 (4)	80
C3	4655 (5)	1440 (5)	3302 (4)	80
C4	4974 (4)	2495 (4)	3866 (4)	69
C4a	6213 (3)	2455 (3)	4377 (3)	56
C5	8601 (4)	5039 (3)	3726 (4)	69
C6	9977 (6)	5141 (5)	3125 (4)	86
C7	10893 (4)	4062 (5)	3114 (4)	83
C8	10455 (4)	2852 (4)	3682 (4)	69
C8a	9100 (3)	2738 (3)	4271 (3)	55
C9	8424 (3)	1487 (3)	4960 (3)	53
C9a	7137 (3)	1357 (3)	4317 (3)	55
C10	6739 (3)	3508 (3)	5053 (3)	56
C10a	8172 (3)	3835 (3)	4310 (3)	55
C11	7892 (3)	1790 (3)	6355 (3)	50
C12	7014 (3)	2841 (3)	6404 (3)	50
C13	8507 (4)	982 (3)	7448 (4)	60
C14	8166 (5)	−631 (4)	9285 (5)	96
C15	7077 (6)	−1453 (6)	9985 (5)	118
C16	7511 (6)	−2327 (5)	11126 (5)	110
C17	6370 (4)	3434 (4)	7581 (4)	61
C18	6269 (6)	3373 (7)	9880 (5)	120
C19	7256 (8)	3813 (9)	10515 (7)	162
C20	8588 (7)	3225 (8)	10618 (6)	142
O1	7633 (3)	190 (3)	8185 (3)	79
O2	9704 (3)	1017 (3)	7589 (4)	109
O3	6847 (3)	2908 (3)	8657 (3)	95
O4	5516 (4)	4336 (3)	7548 (3)	108

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

C1—C2	1·397 (6)	C10—C10a	1·526 (5)
C1—C9a	1·376 (5)	C10—C12	1·530 (5)
C2—C3	1·365 (6)	C11—C12	1·329 (4)
C3—C4	1·374 (6)	C11—C13	1·487 (5)
C4—C4a	1·378 (5)	C12—C17	1·475 (5)
C4a—C9a	1·392 (4)	C13—O1	1·310 (4)
C4a—C10	1·521 (5)	C13—O2	1·194 (4)
C5—C6	1·394 (6)	C14—C15	1·450 (7)
C5—C10a	1·372 (4)	C14—O1	1·462 (5)
C6—C7	1·372 (6)	C15—C16	1·483 (6)
C7—C8	1·375 (6)	C17—O3	1·307 (4)
C8—C8a	1·373 (5)	C17—O4	1·200 (4)
C8a—C9	1·531 (4)	C18—C19	1·371 (8)
C8a—C10a	1·393 (5)	C18—O3	1·439 (5)
C9—C9a	1·520 (4)	C19—C20	1·399 (9)
C9—C11	1·530 (5)		

C2—C1—C9a	118·1 (4)	C10a—C10—C12	105·4 (3)
C1—C2—C3	121·3 (4)	C5—C10a—C8a	119·9 (3)
C2—C3—C4	120·4 (4)	C5—C10a—C10	127·6 (3)
C3—C4—C4a	119·4 (4)	C8a—C10a—C10	112·5 (3)
C4—C4a—C9a	120·3 (3)	C9—C11—C12	113·7 (3)
C4—C4a—C10	127·2 (3)	C9—C11—C13	117·5 (3)
C9a—C4a—C10	112·6 (3)	C12—C11—C13	128·5 (3)
C6—C5—C10a	118·8 (4)	C10—C12—C11	113·5 (3)
C5—C6—C7	121·0 (4)	C10—C12—C17	119·0 (3)
C6—C7—C8	120·1 (4)	C11—C12—C17	127·4 (3)
C7—C8—C8a	119·5 (4)	C11—C13—O1	113·5 (3)
C8—C8a—C9	127·2 (3)	C11—C13—O2	122·4 (3)
C8—C8a—C10a	120·7 (3)	O1—C13—O2	124·0 (3)
C9—C8a—C10a	112·1 (3)	C15—C14—O1	109·4 (4)
C8a—C9—C9a	106·8 (2)	C14—C15—C16	113·8 (4)
C8a—C9—C11	104·8 (2)	C12—C17—O3	113·7 (3)
C9a—C9—C11	106·3 (3)	C12—C17—O4	123·1 (4)
C1—C9a—C4a	120·5 (3)	O3—C17—O4	123·2 (4)
C1—C9a—C9	127·2 (3)	C19—C18—O3	113·0 (5)
C4a—C9a—C9	112·3 (3)	C18—C19—C20	126·0 (7)
C4a—C10—C10a	106·5 (3)	C13—O1—C14	116·5 (3)
C4a—C10—C12	105·9 (2)	C17—O3—C18	119·0 (3)

Related literature. This work is part of a photochemical study of dibenzobarrelene diesters (Garcia-Garibay, Scheffer, Trotter & Wireko, 1990).

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Structure of the Hexagonal Modification of 3′-*O*-Acetylthymidine

By A. Schouten* and J. A. Kanters

Laboratorium voor Kristal- en Structuurchemie, Rijksuniversiteit Utrecht, Transitorium 3, *Padualaan* 8,
3584 *CH Utrecht, The Netherlands*

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Abstract. $C_{12}H_{16}N_2O_6$, $M_r = 284{\cdot}27$, hexagonal, $P6_3$, $a = 22{\cdot}120$ (2), $c = 4{\cdot}960$ (5) Å, $V = 2102$ (2) Å^3, $Z = 6$, $D_x = 1{\cdot}347$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 9{\cdot}4$ cm^{-1}, $F(000) = 900$, $T = 293$ K, $R = 0{\cdot}050$ for 1163 unique observed reflections with $I > 2{\cdot}5\sigma(I)$. The molecule has an *anti* conformation about the N-glycosidic bond with χ[C(2)—N(1)—C(1′)—O(4′)] = −116·8 (5)° and the sugar ring is in a half-chair conformation with pseudorotation parameters $P = 169{\cdot}3$ (6)° and $\psi = 34{\cdot}9$ (4)°. The C(4′)—C(5′) conformation is *gauche–gauche*. The structure does not exhibit base pairing; there is only one intermolecular head-to-head hydrogen bond between the bases [N(3)—H···O(4) ($-x$, $1-y$, $\frac{1}{2}+z$), N···O 2·876 (7) Å

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* Author to whom correspondence should be addressed.

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and N—H···O 170 (4)°]. A second hydrogen bond [O(5′)—H···O(4) ($1-y$, $1+x-y$, z), O···O 2·842 (7) Å and O—H···O 162 (5)°] links the sugar to the base.

Experimental. After several unsuccessful attempts using conventional methods to obtain crystals suitable for X-ray analysis, crystals of good quality were prepared by dissolving 10 mg of the title compound in 200 μl *n*-propanol at 333 K. After cooling very fine colourless needles appeared. The resulting slurry was heated until almost all solid material was dissolved. This solution was cooled to room temperature in a Dewar flask over a period of five days which resulted in single crystals with sufficient thickness. For the X-ray study a crystal with dimensions 0·1 × 0·1 × 1·1 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. Unit-cell parameters were determined by least-squares refinement of the angular settings of 25 reflections in the θ range 10·84–15·85°. The diffracted intensities of 2798 reflections were collected within the reflection quadrant limited by $2\theta = 140°$ and $0 \leq h \leq 26$, $0 \leq k \leq 26$, $-6 \leq l \leq 6$ using Ni-filtered Cu $K\alpha$ radiation. After merging equivalent reflections ($R_{\rm int} = 0{\cdot}058$) 1489 unique reflections remained, of which 1163 were above the $2{\cdot}5\sigma(I)$ level. The ω–2θ scan technique was applied with $\Delta\omega = (0{\cdot}55 + 0{\cdot}15\tan\theta)°$. The intensities of three reference reflections ($1\bar{3}1$, 211 and $\bar{3}21$), measured every hour, did not vary by more than 2% over the duration of the data collection. Intensities were corrected for Lp effects but not for absorption. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986), which resulted in the coordinates of all non-H atoms. The positions of the H atoms were determined from subsequent difference Fourier maps. The H atoms of the methyl groups were placed at idealized positions (C—H 1·00 Å, H—C—H 109·5°) and refined riding on their bonded atoms. All H atoms were given constant isotropic thermal parameters equal to those of their carrier atoms. The distance C(3′1)—O(3′) was found to be short (1·27 Å) and therefore was fixed at an acceptable value of 1·35 Å. As the refinement proceeded a peak at a distance of about 1·4 Å from C(3′1) and O(3′1) was found. This was interpreted as a disorder of O(3′1) over two positions as supported by the high thermal parameter U_{11} (0·251 Å^2) of O(3′1). By anisotropic refinement of the stronger peak and isotropic refinement of the weaker peak, the occupancy factor converged to a value of 0·81 (1). Anisotropic full-matrix least-squares refinement on F of 215 parameters converged at $R = 0{\cdot}050$ and $wR = 0{\cdot}039$ with $w = 2{\cdot}9764/\sigma^2(F_o)$, $S = 0{\cdot}79$, $(\Delta/\sigma)_{\rm av} = 0{\cdot}004$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}03$. The isotropic extinction parameter converged to 0·017. Maximum and minimum residual densities in the final difference map were 0·31 and $-0{\cdot}27$ e Å^{-3}, respectively. The scattering factors were those of Cromer & Mann (1968) and anomalous-dispersion terms from Cromer & Liberman (1970). Calculations were performed with *SHELX*76 (Sheldrick, 1976) (refinement) and the *EUCLID* package (Spek, 1982) (geometry and illustrations) on a MicroVAX II computer.

A perspective view of the molecule with adopted numbering and the hydrogen-bond network are shown in Fig. 1 and Fig. 2, respectively. Fractional atomic coordinates and equivalent isotropic temperature factors for non-H atoms are listed in Table 1,* bond distances and angles in Table 2.

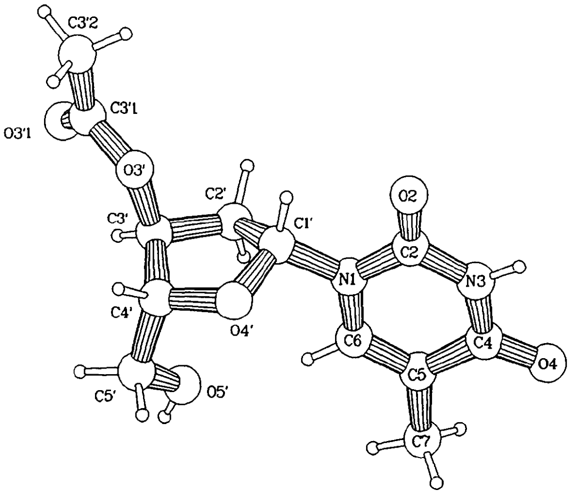

Fig. 1. Perspective view and atomic numbering of the title compound. The lower occupancy O(3′2) has been omitted.

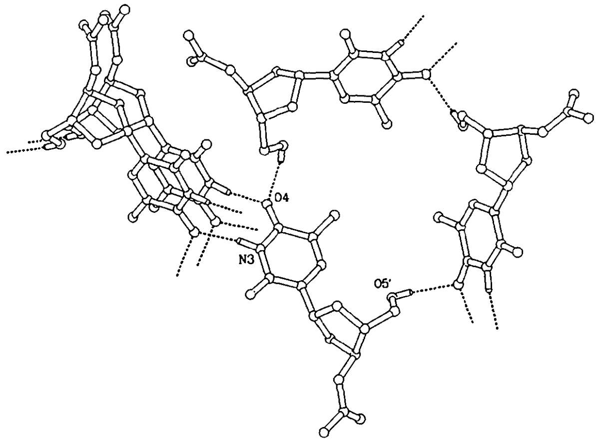

Fig. 2. Projection of a part of the structure down the c axis, showing the hydrogen-bond network.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53692 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å²) *with their e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}/U_{iso}
C(2)	0·0680 (3)	0·4147 (3)	0·289 (1)	0·050 (2)
C(4)	0·0934 (2)	0·5095 (2)	−0·033 (1)	0·041 (2)
C(5)	0·1589 (2)	0·5138 (2)	−0·095 (1)	0·040 (2)
C(6)	0·1740 (2)	0·4695 (2)	0·032 (1)	0·040 (2)
C(7)	0·2071 (3)	0·5671 (3)	−0·290 (2)	0·060 (2)
C(1′)	0·1513 (3)	0·3732 (3)	0·339 (1)	0·044 (2)
C(2′)	0·1678 (3)	0·3316 (3)	0·138 (1)	0·051 (2)
C(3′)	0·2219 (3)	0·3225 (3)	0·287 (1)	0·053 (2)
C(4′)	0·2616 (3)	0·3882 (3)	0·448 (1)	0·052 (2)
C(5′)	0·3286 (3)	0·4420 (3)	0·325 (1)	0·057 (2)
C(3′1)	0·1817 (3)	0·2044 (2)	0·446 (2)	0·074 (3)
C(3′2)	0·1401 (3)	0·1541 (3)	0·656 (2)	0·095 (3)
N(1)	0·1302 (2)	0·4197 (2)	0·2132 (–)	0·041 (2)
N(3)	0·0534 (2)	0·4611 (2)	0·159 (1)	0·045 (2)
O(2)	0·0286 (2)	0·3735 (2)	0·456 (1)	0·083 (2)
O(4)	0·0718 (2)	0·5454 (2)	−0·141 (1)	0·053 (1)
O(3′)	0·1862 (2)	0·2669 (2)	0·482 (1)	0·093 (2)
O(4′)	0·2142 (2)	0·4153 (2)	0·484 (1)	0·050 (1)
O(5′)	0·3156 (2)	0·4581 (2)	0·059 (1)	0·058 (2)
O(3′1)	0·2047 (5)	0·1940 (3)	0·258 (2)	0·138 (4)*
O(3′2)	0·258 (1)	0·240 (1)	0·436 (5)	0·098 (8)†

* S.o.f. 0·81 (1).

† S.o.f. 0·19 (1) and U_{iso}.

Related literature. The molecular geometry of the title compound is very similar to that of the recently reported structure of the orthorhombic modification (Eccleston, Wilson & Howie, 1988). In the two modifications the base residues are head-to-head linked by N(3)—H···O(4) hydrogen bonds. However, the modifications differ in the second hydrogen bond: in the hexagonal form the bond is between sugar O(5′)—H and base O(4), whereas in the orthorhombic form the bond connects O(5′)—H and O(4′) of neighbouring sugar residues.

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

C(4)—C(5)	1·437 (7)	N(3)—C(2)	1·381 (8)
C(5)—C(6)	1·341 (7)	N(3)—C(4)	1·375 (7)
C(5)—C(7)	1·486 (8)	O(2)—C(2)	1·215 (8)
C(1′)—C(2′)	1·52 (1)	O(4)—C(4)	1·235 (6)
C(2′)—C(3′)	1·50 (1)	O(3′)—C(3′)	1·449 (8)
C(3′)—C(4′)	1·497 (9)	O(3′)—C(3′1)	1·348 (6)
C(4′)—C(5′)	1·490 (9)	O(4′)—C(1′)	1·424 (8)
C(3′1)—C(3′2)	1·46 (1)	O(4′)—C(4′)	1·456 (8)
N(1)—C(2)	1·377 (9)	O(5′)—C(5′)	1·434 (9)
N(1)—C(6)	1·374 (6)	O(3′1)—C(3′1)	1·14 (1)
N(1)—C(1′)	1·465 (8)	O(3′2)—C(3′1)	1·47 (3)

C(3′)—O(3′)—C(3′1)	120·7 (6)	O(4′)—C(1′)—N(1)	107·9 (4)
C(1′)—O(4′)—C(4′)	109·7 (4)	O(4′)—C(1′)—C(2′)	106·1 (6)
C(2)—N(1)—C(6)	121·4 (4)	N(1)—C(1′)—C(2′)	113·8 (5)
C(2)—N(1)—C(1′)	118·9 (4)	C(1′)—C(2′)—C(3′)	101·8 (5)
C(6)—N(1)—C(1′)	119·7 (5)	O(3′)—C(3′)—C(2′)	108·1 (5)
C(2)—N(3)—C(4)	127·2 (5)	O(3′)—C(3′)—C(4′)	105·9 (5)
O(2)—C(2)—N(1)	124·1 (6)	C(2′)—C(3′)—C(4′)	104·6 (5)
O(2)—C(2)—N(3)	121·9 (6)	O(4′)—C(4′)—C(3′)	105·7 (5)
N(1)—C(2)—N(3)	114·0 (5)	O(4′)—C(4′)—C(5′)	110·8 (5)
O(4)—C(4)—N(3)	119·8 (5)	C(3′)—C(4′)—C(5′)	115·1 (6)
O(4)—C(4)—C(5)	124·5 (5)	O(5′)—C(5′)—C(4′)	109·4 (6)
N(3)—C(4)—C(5)	115·8 (4)	O(3′)—C(3′1)—O(3′1)	120·3 (6)
C(4)—C(5)—C(6)	117·5 (5)	O(3′)—C(3′1)—O(3′2)	89 (1)
C(4)—C(5)—C(7)	119·4 (5)	O(3′)—C(3′1)—C(3′2)	112·4 (6)
C(6)—C(5)—C(7)	123·0 (5)	O(3′1)—C(3′1)—C(3′2)	127·1 (5)
N(1)—C(6)—C(5)	124·0 (5)	O(3′2)—C(3′1)—C(3′2)	123 (1)

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Structure of 6-Allyl-3-hydroxymethyl-6,10b-dimethyl-2-phenyl-2,3,5,6-tetrahydroisoquinolino[1,2-*b*][1,3]oxazol-5-one

By Kevin A. Andersen and Oren P. Anderson*

Department of Chemistry, Colorado State University, Fort Collins, Colorado 80523, *USA*

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Abstract. $C_{23}H_{25}NO_3$, $M_r = 363·46$, orthorhombic, $P2_12_12_1$, $a = 10·047$ (4), $b = 10·599$ (4), $c = 19·003$ (6) Å, $V = 2024$ (1) Å^3, $Z = 4$, $D_x = 1·19$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0·9$ cm^{-1}, $F(000) = 776$, $T = 151$ K, $R = 0·050$ ($wR = 0·068$) for 1888 unique observed reflections. Three fused (six-, six- and five-membered) heteroatom rings contain an amide N bridgehead atom.

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* Author to whom correspondence should be addressed.

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