tallography (1974, Vol. IV). All geometrical calculations were performed using *PARST* (Nardelli, 1983). Fig. 1 shows the atom-labelling scheme and molecular conformation. Atomic coordinates are listed in Table 1,* bond distances and angles in Table 2. Hydrogen bonds are detailed in Table 3.

Related literature. The synthesis of the title compound forms part of a series of studies on stereoselective synthesis of α,β-dihydroxy ketones (Clerici & Porta, 1989). The ketyl radical generated by titanium(III) reduction of benzil contributes stereoselectively to the aldehydic C atom of 2-carboxy-

benzaldehyde, affording, after lactonization, the present phthalidyl derivative. Structural aspects of the phthalide system have been reported by Whalley, Ferguson & Roberts (1980) and by Roszak & Skrzat (1989).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53462 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Nucleoside Analogue: 3'-Azido-5-chloro-2',3'-dideoxyuridine*

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Abstract. 1-(3-Azido-2,3-dideoxy-β-D-*erythro*-pentofuranosyl)-5-chlorouracil, $C_9H_{10}ClN_5O_4$, $M_r = 287.66$, monoclinic, $P2_1$, $a = 5.780$ (3), $b = 11.730$ (6), $c = 17.670$ (9) Å, $\beta = 93.87$ (4)°, $V = 1195$ (1) Å^3, $Z = 4$, $D_m = 1.60$, $D_x = 1.598$ Mg m^{-3}, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.335$ mm^{-1}, $F(000) = 592$, $T = 290$ K. Final $R = 0.021$ for 1892 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 -128.2 (2)° in the *anti* range; the sugar pucker is $^2T_3$ with $P = 173$ (1) and $\psi_m = 33$ (1)° and the C4'—C5' conformation is $+sc$ with $\gamma = 50.4$ (3)°. For molecule B: the N-glycosidic torsion

angle χ has a value of -168.9 (2)° in the *anti* range; the sugar pucker is $_3T^4$ with $P = 208$ (1) and $\psi_m = 36$ (1)° and the C4'—C5' conformation is *ap* with $\gamma = 170.8$ (2)°. 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 in the crystal structure.

Experimental. Colourless prismatic crystals obtained at room temperature from an aqueous solution, dimensions $\sim 0.55 \times 0.3 \times 0.2$ mm. D_m by flotation. STADI4 computer-controlled four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique ($2\theta_{max} = 50°$, $-7 \le h \le 0$, $-14 \le k \le 14$, $-21 \le l \le 21$). Cell dimensions by least-squares refinement of the setting angles of 24 reflections with $20 < 2\theta < 29°$, space group $P2_1$ from

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

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
N1A	4489 (4)	8560*	8902 (1)	2·50 (4)
C2A	5980 (4)	8903 (2)	8375 (1)	2·35 (4)
O2A	7702 (3)	9476 (2)	8529 (1)	3·21 (4)
N3A	5376 (4)	8544 (2)	7649 (1)	2·46 (4)
C4A	3552 (4)	7863 (2)	7395 (1)	2·42 (4)
O4A	3214 (3)	7614 (2)	6727 (1)	3·53 (4)
C5A	2136 (4)	7514 (2)	8002 (1)	2·41 (5)
C15A	− 187 (1)	6630·1 (7)	7774·4 (4)	3·53 (1)
C6A	2624 (4)	7871 (2)	8711 (1)	2·62 (5)
C1'A	4873 (4)	8953 (3)	9694 (1)	2·60 (5)
C2'A	2994 (5)	9729 (2)	9946 (1)	3·09 (5)
C3'A	2945 (5)	9453 (3)	10781 (1)	2·90 (5)
N3'A	4752 (5)	10167 (3)	11191 (1)	4·49 (6)
N4'A	4716 (4)	10145 (2)	11884 (1)	3·36 (5)
N5'A	4842 (5)	10211 (3)	12520 (1)	4·20 (6)
C4'A	3552 (4)	8189 (3)	10827 (1)	2·67 (5)
O4'A	4880 (3)	7969 (2)	10165 (1)	2·82 (3)
C5'A	1532 (5)	7385 (3)	10829 (2)	3·64 (6)
O5'A	− 113 (3)	7645 (4)	10220 (1)	4·50 (5)
N1B	8593 (3)	5197 (2)	3849 (1)	2·42 (4)
C2B	9953 (4)	4891 (2)	3275 (1)	2·46 (4)
O2B	11616 (3)	4256 (2)	3394 (1)	3·35 (4)
N3B	9300 (4)	5328 (2)	2580 (1)	2·70 (4)
C4B	7466 (4)	6046 (3)	2382 (1)	2·68 (5)
O4B	7033 (4)	6342 (2)	1732 (1)	3·85 (4)
C5B	6192 (4)	6350 (2)	3032 (1)	2·66 (5)
C15B	3882 (1)	7261·2 (8)	2890·1 (4)	4·02 (1)
C6B	6760 (4)	5923 (2)	3724 (1)	2·60 (5)
C1'B	9090 (4)	4583 (2)	4589 (1)	2·51 (5)
C2'B	11368 (5)	4941 (3)	5002 (2)	3·42 (6)
C3'B	10744 (5)	5060 (3)	5821 (1)	3·13 (5)
N3'B	11001 (5)	3917 (3)	6172 (1)	4·20 (5)
N4'B	10729 (4)	3884 (2)	6856 (1)	3·53 (5)
N5'B	10530 (5)	3741 (3)	7477 (2)	4·79 (6)
C4'B	8217 (5)	5441 (3)	5727 (1)	2·73 (5)
O4'B	7296 (3)	4830 (2)	5060 (1)	2·66 (3)
C5'B	7942 (5)	6717 (3)	5618 (2)	3·58 (6)
O5'B	5613 (4)	7013 (2)	5417 (1)	4·65 (5)

* Parameter kept fixed for origin definition.

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

	A	B
N1—C2	1·373 (4)	1·373 (3)
N1—C6	1·369 (3)	1·365 (3)
N1—C1'	1·477 (3)	1·502 (4)
C2—O2	1·217 (4)	1·223 (3)
C2—N3	1·372 (3)	1·361 (4)
N3—C4	1·373 (3)	1·380 (3)
C4—O4	1·219 (3)	1·211 (3)
C4—C5	1·452 (3)	1·450 (4)
C5—C6	1·331 (3)	1·340 (4)
C5—C15	1·723 (3)	1·715 (3)
C1'—C2'	1·508 (4)	1·522 (4)
C1'—O4'	1·423 (3)	1·403 (4)
C2'—C3'	1·513 (3)	1·520 (4)
C3'—N3'	1·489 (4)	1·482 (4)
C3'—C4'	1·525 (4)	1·525 (4)
N3'—N4'	1·226 (3)	1·231 (3)
N4'—N5'	1·123 (3)	1·123 (4)
C4'—O4'	1·464 (3)	1·451 (3)
C4'—C5'	1·501 (5)	1·516 (4)
C5'—O5'	1·420 (3)	1·413 (4)
C2—N1—C6	121·8 (2)	121·4 (3)
C2—N1—C1'	119·3 (2)	115·6 (3)
C6—N1—C1'	119·1 (2)	122·8 (3)
N1—C2—O2	123·6 (3)	121·0 (2)
N1—C2—N3	114·3 (3)	115·5 (2)
O2—C2—N3	122·1 (2)	123·4 (2)
C2—N3—C4	128·7 (3)	128·3 (2)
N3—C4—O4	121·7 (2)	121·4 (2)
N3—C4—C5	112·4 (2)	111·8 (2)
O4—C4—C5	125·9 (2)	126·8 (2)
C4—C5—C15	117·8 (2)	118·0 (2)
C4—C5—C6	120·8 (2)	· 121·6 (2)
C15—C5—C6	121·4 (2)	120·5 (3)
N1—C6—C5	122·0 (2)	121·4 (2)
N1—C1'—C2'	113·8 (2)	113·2 (2)
N1—C1'—O4'	107·1 (2)	108·2 (2)
C2'—C1'—O4'	106·9 (2)	108·0 (2)
C1'—C2'—C3'	102·7 (2)	103·3 (2)
C2'—C3'—N3'	107·2 (2)	107·0 (3)
C2'—C3'—C4'	103·9 (2)	102·2 (2)
N3'—C3'—C4'	111·7 (2)	112·3 (2)
C3'—N3'—N4'	114·6 (3)	115·1 (3)
N3'—N4'—N5'	173·0 (3)	173·1 (3)
C3'—C4'—O4'	105·1 (3)	103·9 (3)
C3'—C4'—C5'	115·8 (2)	113·2 (2)
O4'—C4'—C5'	109·9 (2)	110·9 (2)
C1'—O4'—C4'	110·1 (2)	109·8 (2)
C4'—C5'—O5'	110·1 (2)	111·4 (2)
C2—N1—C1'—O4'	− 128·2 (2)	− 168·9 (2)
C2'—C1'—O4'—C4'	− 14·8 (3)	6·1 (3)
O4'—C1'—C2'—C3'	30·0 (3)	16·8 (3)
C1'—C2'—C3'—C4'	− 33·2 (3)	− 31·6 (3)
C2'—C3'—C4'—O4'	25·0 (3)	35·6 (3)
C3'—C4'—O4'—C1'	− 6·6 (2)	− 26·5 (3)
C3'—C4'—C5'—O5'	50·4 (3)	170·8 (2)

systematic absences 0*k*0 for *k* odd. Four standard reflections (002, 040, $\bar{1}$30, $\bar{1}2\bar{1}$) monitored after every 120 min showed no significant decrease in intensity per hour, 4738 reflections measured, 2215 unique reflections ($R_{int} = 0·015$), of which 1894 were considered observed [$F > 4\sigma(F)$]. Two reflections (002, 012) badly affected by extinction were eliminated. Lorentz–polarization corrections, no absorption corrections, scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) and Stewart, Davidson & Simpson (1965) (for H atoms). The structure was solved by *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The *E* map calculated from the solution with the best figure of merit revealed 18 of the 38 non-H atoms in the asymmetric unit. The remaining atoms were obtained from a subsequent Fourier synthesis. A difference synthesis revealed the positions of all H atoms, which were refined with fixed isotropic temperature factors 1·3 times that of the parent atom. All other atoms were refined anisotropically on *F* by full-matrix least squares. The refinement converged at $R = 0·021$, $wR = 0·028$, $S = 0·266$. $w = (C_0 + C_1|F_o| + C_2|F_o|^2 + C_3|F_o|^3)^{-1}$, where $C_0 = 1000$, $C_1 = 1$, $C_2 = 0·002$, $C_3 = 0·000002$.

402 refined parameters, max. shift/e.s.d. = 0·03, min. and max. electron density $-0·166$ and $0·120$ e Å^{-3}. The number of reflections per refined variable was $1892/402 = 4·7$. All calculations were performed on a PDP-11/73 microcomputer using *SDP/PDP* (Enraf–Nonius, 1982) and *PARST* (Nardelli, 1983). A *PLUTO* plot (Motherwell & Clegg, 1978) of molecule *A* of the title compound with the atomic numbering scheme is shown in Fig. 1. The final fractional atomic coordinates are given in Table 1.*
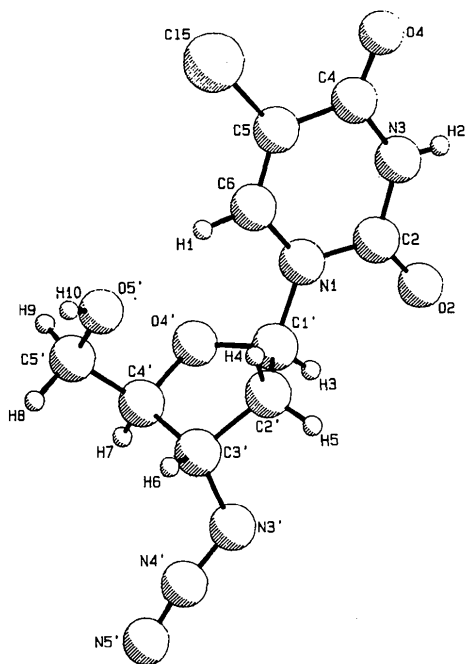
Fig. 1. Atomic numbering scheme of molecule *A* of the title compound.

Bond lengths, bond angles and some selected torsion angles are given in Table 2.

Related literature. The method of preparation of this possible anti-AIDS compound has been described by

Van Aerschot, Everaert, Balzarini, Augustyns, Jie, Janssen, Peeters, Blaton, De Ranter, De Clercq & Herdewijn (1990). There is a close conformational similarity between the title compound and 3′-azido-3′-deoxythymidine (Dyer, Low, Tollin, Wilson & Howie, 1988).

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Structural Studies of Mitomycins. VI. Structure of Mitiromycin Monohydrate

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Abstract. 1,4b,5,5a,6,11b-Hexahydro-10-methoxy-5,9-dimethylazirino[2′,3′:3,4]pyrrolo[1,2-*a*][1,3]oxazino[4,5-*b*]indole-3,8,11(4*H*)-trione monohydrate, $C_{16}H_{17}N_3O_5.H_2O$, $M_r = 349.35$, orthorhombic, $P2_12_12_1$, $a = 11.770$ (1), $b = 14.276$ (2), $c = 9.488$ (1) Å, $V = 1594.3$ (5) Å^3, $Z = 4$, $D_x = 1.46$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.5405$ Å, $\mu = 8.1$ cm^{-1}, $F(000) = 736$, $T = 293$ K, $wR = 0.044$ for 1650 observed reflections with $I > 3\sigma(I)$. The quinone ring adopts a flat envelope conformation in which one carbonyl group deviates from the plane defined by the other five atoms in the ring, C by 0.065 (2) and O by 0.203 (2) Å. The dihedral angle between the quinone and the aziridine rings is 56.7 (1)°. The oxazinone ring has a boat conformation. The stereochemistries of the two junctions where the oxazinone ring is fused are determined as α and α. The absolute configuration of the molecule was suggested by referring to that of 7-*p*-bromoanilino-7-demethoxymitomycin B [Hirayama & Shirahata (1987). *Acta Cryst.* B43, 555–559].

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