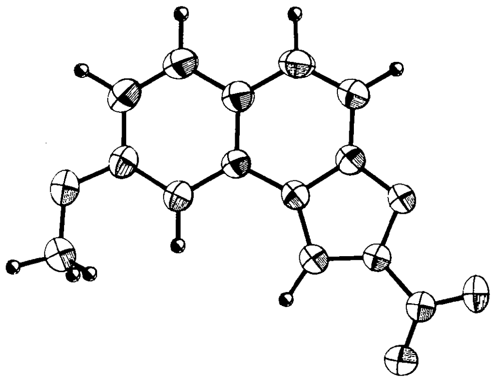

Fig. 2. Tracé *ORTEP* (Johnson, 1965) de la molécule projetée sur son plan moyen.

Tableau 2. *Longueurs* (Å) *et angles* (°) *de liaisons intramoléculaires avec leurs écarts-types*

O(1)–C(2)	1,360 (4)	C(2)–O(1)–C(13)	104,2 (2)
O(1)–C(13)	1,372 (4)	O(1)–C(2)–C(3)	114,1 (3)
C(2)–C(3)	1,350 (5)	O(1)–C(2)–N(21)	115,4 (3)
C(2)–N(21)	1,408 (5)	C(3)–C(2)–N(21)	130,4 (3)
C(3)–C(12)	1,423 (5)	C(2)–C(3)–C(12)	104,7 (3)
C(4)–C(5)	1,359 (5)	C(5)–C(4)–C(13)	115,3 (3)
C(4)–C(13)	1,400 (5)	C(4)–C(5)–C(11)	123,6 (3)
C(5)–C(11)	1,426 (5)	C(7)–C(6)–C(11)	121,2 (3)
C(6)–C(7)	1,365 (5)	C(6)–C(7)–C(8)	120,0 (3)
C(6)–C(11)	1,410 (5)	C(7)–C(8)–C(9)	121,1 (3)
C(7)–C(8)	1,414 (5)	C(7)–C(8)–O(81)	114,4 (3)
C(8)–C(9)	1,373 (5)	C(9)–C(8)–O(81)	124,5 (3)
C(8)–O(81)	1,367 (4)	C(8)–C(9)–C(10)	119,0 (3)
C(9)–C(10)	1,411 (5)	C(9)–C(10)–C(11)	120,6 (3)
C(10)–C(11)	1,425 (5)	C(9)–C(10)–C(12)	123,1 (3)
C(10)–C(12)	1,427 (4)	C(11)–C(10)–C(12)	116,3 (3)
C(12)–C(13)	1,389 (5)	C(5)–C(11)–C(6)	121,7 (3)
N(21)–O(22)	1,222 (4)	C(5)–C(11)–C(10)	120,2 (3)
N(21)–O(23)	1,221 (4)	C(6)–C(11)–C(10)	118,0 (3)
O(81)–C(82)	1,426 (5)	C(3)–C(12)–C(10)	134,1 (3)
		C(3)–C(12)–C(13)	106,3 (3)
		C(10)–C(12)–C(13)	119,6 (3)
		O(1)–C(13)–C(4)	124,4 (3)
		O(1)–C(13)–C(12)	110,7 (3)
		C(4)–C(13)–C(12)	124,9 (3)
		C(2)–N(21)–O(22)	117,5 (3)
		C(2)–N(21)–O(23)	119,1 (3)
		O(22)–N(21)–O(23)	123,4 (3)
		C(8)–O(81)–C(82)	117,5 (3)

La cohésion cristalline est assurée essentiellement par des interactions du type C—H···O mettant en jeu les oxygènes des groupes $-NO_2$ et $-OCH_3$, l'oxygène du cycle furannique n'étant impliqué dans aucune liaison (Fig. 1): (*a*) $O(22^{i})\cdots C(4^{iii+c})$: 3,310 (5); $O(22^{i})\cdots H(104^{iii+c})$: 2,41 (4) Å; (*b*) $C(3^{i})\cdots O(23^{iii+c})$: 3,312 (5); $H(103^{i})\cdots O(23^{iii+c})$: 2,34 (4) Å; (*c*) $O(81^{i})\cdots C(6^{iii+a})$: 3,473 (5); $O(81^{i})\cdots H(106^{iii+a})$: 2,43 (4) Å. Code de symétrie: (iii) $\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z$.

Contrairement au R7000 et à des molécules analogues (Cotrait, Bideau & Bravic, 1983) on n'observe pas d'empilements moléculaires résultant du recouvrement des orbitales π. Ceci est probablement dû au nombre plus élevé des liaisons CH···O, qui, de plus, sont présentement plus courtes et l'emportent donc sur les forces de stacking.

A ces liaisons spécifiques s'ajoutent de nombreux contacts carbone–carbone, carbone–hydrogène, *etc.*

La molécule présentement étudiée (R6998) et son homologue le R7000 ne diffèrent que par la forme globale de la molécule (position du groupement méthoxy). Bien qu'on ne sache pas encore comment agissent ces molécules, une différence d'activité entre les deux substances nous semble devoir être uniquement due à des facteurs stéréochimiques. Seule la comparaison des conformations moléculaires d'un plus grand nombre de composés analogues permettra éventuellement d'établir des relations structure–activité.

Dans le cristal, les liaisons de type C—H···O sont présentes dans les deux molécules mais seul le R7000 présente des empilements (interactions par électrons π).

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3′,5′-Di-*O*-acetyl-2′-deoxy-5-iodouridine, $C_{13}H_{15}IN_2O_7$

By C. C. Wilson, J. N. Low and D. W. Young

Carnegie Laboratory of Physics, University of Dundee, Dundee DD1 4HN, Scotland

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Abstract. $M_r = 438\cdot2$, monoclinic, $P2_1$, $a = 7\cdot674$ (8), $b = 7\cdot743$ (8), $c = 13\cdot414$ (5) Å, $\beta = 91\cdot2$ (1)°, $U = 796\cdot88$ Å^3, $Z = 2$, $D_x = 1\cdot826$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 18\cdot93$ cm^{-1}, $F(000) = 432$, $T = 293$ K, $R = 0\cdot064$ for 1828 observed reflexions (Friedel pairs not merged). The sugar-ring pucker is found to be

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C(1′)-*exo* ($_1E$), with $P = 132{\cdot}0\,(15)°$ and $\chi = -138{\cdot}3\,(13)°$. There is a short I···O contact of 3·081 (19) Å and another of 2·83 (3) Å between an acetyl O and a neighbouring base ring.

Introduction. The structure determination was undertaken to facilitate the use of this compound for electron spin resonance studies of free radicals produced by ionizing radiations (C. C. Wilson & R. A. Vaughan, work in progress). The atomic numbering used is shown in Fig. 1.

Experimental. Crystals from aqueous solution, intensities measured on a Stoe Stadi II diffractometer (two-circle), all calculations performed on the Dundee University DEC-10 computer using *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Cell dimensions from zero-level Weissenberg photographs of crystals mounted along **a** and **b**, and refined on the diffractometer. Intensities from *b*-axis crystal in the range $k = 0$ to 6 and $0° < 2\theta < 60°$, crystal dimensions $0{\cdot}31 \times 0{\cdot}155 \times 0{\cdot}116$ mm, and from *a*-axis crystal in the range $h = 0$ to 2, $0° < 2\theta < 60°$, crystal dimensions $0{\cdot}62 \times 0{\cdot}12 \times 0{\cdot}23$ mm. Absorption corrections applied (maximum and minimum transmission factors for *a*-axis crystal: 0·82, 0·60; for *b*-axis crystal: 0·80, 0·55). Range of indices: $-9 < h < 9$, $-10 < k < 10$, $0 < l < 18$. A standard reflexion was measured every 100 reflexions on each layer line: random variation (<5%) from mean intensities. 2875 reflexions measured, $R_{\rm int} = 0{\cdot}03$. Refinement (on F) by least-squares methods using 1828 reflexions $[F_o > 5\sigma(F_o)]$. Structure solved using heavy-atom method. All atoms except H refined using anisotropic thermal parameters. H atoms included as 'riding atoms' at calculated positions. The final refinement involved 206 refined parameters, $wR = 0{\cdot}065$, $w = 2{\cdot}8903/[\sigma^2(F) + 0{\cdot}000570F^2]$, maximum shift/e.s.d. = 0·005; in final difference Fourier synthesis maximum peak = 1·27 e Å^{-3}, minimum peak = −2 e Å^{-3}. The largest difference peaks were associated with the I atom. Scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The atomic parameters are given in Table 1.* Table 2 lists interatomic distances and bond angles. A comparison of these with the values in related compounds [the bromo-substituted compound (Low, 1983) and 3′,5′-di-*O*-acetyluridine (de Graaff, Admiraal, Koen & Romers, 1977)] shows good agreement of these parameters. The chemically similar O(3′)—C(3′1) and O(5′)—C(5′1) bonds, however, have lengths which are significantly different.

Selected torsion angles are shown in Table 2. Upon comparison of these with the values obtained for the bromo compound, several major differences were noted. Amongst these, most noticeable were the different values of the pseudorotation phase angle P, and the glycosyl torsion angle, χ[C(2)—N(1)—C(1′)—O(4′)]. Here $P = 132{\cdot}0\,(15)°$ and $\chi = -138{\cdot}3\,(11)°$. The C(4′)—C(5′) conformation of the sugar is g^+. The pyrimidine-base ring is essentially planar [maximum deviation of atom from plane: C(6) 0·029 Å] and the normals between screw-related bases are inclined at an angle of 62·7 (5)° to each other.

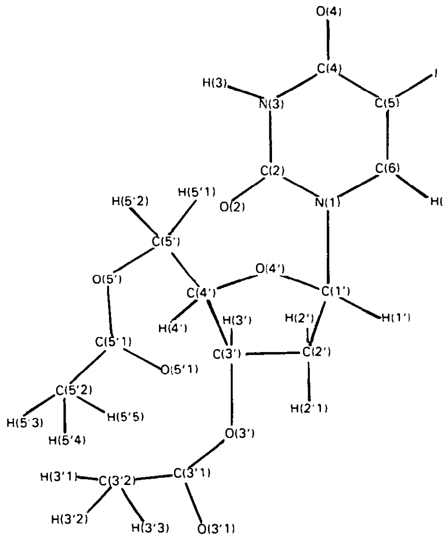

Fig. 1. Atomic numbering.

Table 1. *Coordinates* ($\times 10^4$) *for non-hydrogen atoms with e.s.d.'s in parentheses, and equivalent isotropic temperature factors* (Å$^2 \times 10^3$)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
I(1)	816 (1)	67	4582 (1)	38 (1)
N(1)	3808 (16)	1071 (11)	1955 (7)	26 (3)
C(2)	3235 (19)	2594 (15)	1529 (9)	27 (4)
O(2)	3752 (16)	3217 (11)	805 (6)	42 (3)
N(3)	1835 (17)	3301 (12)	2043 (8)	37 (4)
C(4)	1000 (24)	2806 (17)	2938 (9)	38 (5)
O(4)	−1 (15)	3641 (12)	3335 (7)	48 (3)
C(5)	1768 (20)	1134 (15)	3308 (9)	31 (4)
C(6)	3206 (20)	383 (16)	2828 (8)	33 (4)
C(1′)	5345 (19)	279 (22)	1572 (8)	30 (4)
C(2′)	6989 (22)	482 (14)	2236 (10)	34 (5)
C(3′)	8095 (21)	−1140 (16)	2021 (10)	33 (4)
O(3′)	9206 (13)	−678 (10)	1242 (7)	32 (3)
C(3′1)	10408 (22)	−1959 (17)	987 (11)	37 (5)
C(3′2)	11502 (21)	−1503 (20)	141 (10)	44 (5)
O(3′1)	10427 (18)	−3299 (12)	1451 (9)	59 (4)
C(4′)	6728 (19)	−2424 (15)	1600 (9)	24 (4)
C(5′)	6404 (25)	−4018 (16)	2254 (11)	45 (5)
O(5′)	5724 (18)	−3486 (13)	3185 (8)	38 (4)
C(5′1)	5116 (19)	−4757 (27)	3741 (9)	34 (4)
C(5′2)	4261 (28)	−4126 (19)	4656 (11)	58 (6)
O(5′1)	5154 (19)	−6254 (16)	3474 (9)	52 (5)
O(4′)	5053 (14)	−1540 (11)	1512 (6)	34 (3)

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* Lists of structure amplitudes, anisotropic thermal parameters H-atom coordinates, close intermolecular contact distances, and Fig. 2 showing the hydrogen bonding have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38585 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances* (Å) *and angles* (°) *and selected torsion angles* (°)

C(5)–I(1)	2·046 (13)	C(3′)–C(2′)	1·546 (19)
C(2)–N(1)	1·379 (15)	O(3′)–C(3′)	1·408 (17)
C(6)–N(1)	1·374 (15)	C(4′)–C(3′)	1·544 (19)
C(1′)–N(1)	1·434 (18)	C(3′1)–O(3′)	1·402 (18)
O(2)–C(2)	1·161 (15)	C(3′2)–C(3′1)	1·468 (21)
N(3)–C(2)	1·400 (18)	O(3′1)–C(3′1)	1·211 (17)
C(4)–N(3)	1·425 (18)	C(5′)–C(4′)	1·537 (18)
O(4)–C(4)	1·143 (19)	O(4′)–C(4′)	1·459 (17)
C(5)–C(4)	1·503 (18)	O(5′)–C(5′)	1·425 (19)
C(6)–C(5)	1·414 (20)	C(5′1)–O(5′)	1·326 (21)
C(2′)–C(1′)	1·537 (21)	C(5′2)–C(5′1)	1·485 (21)
O(4′)–C(1′)	1·428 (19)	O(5′1)–C(5′1)	1·214 (23)

C(6)–N(1)–C(2)	125·1 (11)	C(3′)–C(2′)–C(1′)	104·9 (11)
C(1′)–N(1)–C(2)	118·3 (11)	O(3′)–C(3′)–C(2′)	106·0 (10)
C(1′)–N(1)–C(6)	115·6 (10)	C(4′)–C(3′)–C(2′)	102·7 (12)
O(2)–C(2)–N(1)	126·2 (13)	C(4′)–C(3′)–O(3′)	108·0 (10)
N(3)–C(2)–N(1)	111·8 (10)	C(3′1)–O(3′)–C(3′)	114·4 (10)
N(3)–C(2)–O(2)	121·9 (12)	C(3′2)–C(3′1)–O(3′)	114·1 (12)
C(4)–N(3)–C(2)	132·3 (11)	O(3′1)–C(3′1)–O(3′)	118·8 (14)
O(4)–C(4)–N(3)	123·9 (12)	O(3′1)–C(3′1)–C(3′2)	127·1 (14)
C(5)–C(4)–N(3)	109·2 (12)	C(5′)–C(4′)–C(3′)	115·2 (11)
C(5)–C(4)–O(4)	126·6 (12)	O(4′)–C(4′)–O(3′)	108·5 (10)
C(4)–C(5)–I(1)	118·6 (10)	O(4′)–C(4′)–C(5′)	105·7 (12)
C(6)–C(5)–I(1)	120·7 (9)	O(5′)–C(5′)–C(4′)	109·5 (10)
C(6)–C(5)–C(4)	120·5 (11)	C(5′1)–O(5′)–C(5′)	114·7 (11)
C(5)–C(6)–N(1)	120·5 (11)	C(5′2)–C(5′1)–O(5′)	112·8 (16)
C(2′)–C(1′)–N(1)	114·9 (11)	O(5′1)–C(5′1)–O(5′)	122·1 (13)
O(4′)–C(1′)–N(1)	108·2 (11)	O(5′1)–C(5′1)–C(5′2)	124·9 (15)
O(4′)–C(1′)–C(2′)	105·0 (11)	C(4′)–O(4′)–C(1′)	108·8 (10)

C(2)–N(1)–C(1′)–O(4′)	−138·3 (11)
O(4′)–C(1′)–C(2′)–C(3′)	32·0 (11)
C(2′)–C(1′)–O(4′)–C(4′)	−29·9 (11)
C(1′)–C(2′)–C(3′)–C(4′)	−21·6 (12)
C(2′)–C(3′)–O(3′)–C(3′1)	175·6 (10)
C(2′)–C(3′)–C(4′)–O(4′)	4·4 (12)
O(3′)–C(3′)–C(4′)–C(5′)	134·4 (11)
C(3′)–O(3′)–C(3′1)–C(3′2)	177·0 (12)
C(3′)–C(4′)–C(5′)–O(5′)	62·8 (11)
O(4′)–C(4′)–C(5′)–O(5′)	−56·9 (12)
C(3′)–C(4′)–O(4′)–C(1′)	16·2 (10)
C(4′)–C(5′)–O(5′)–C(5′1)	168·9 (11)
C(5′)–O(5′)–C(5′1)–C(5′2)	−174·3 (16)

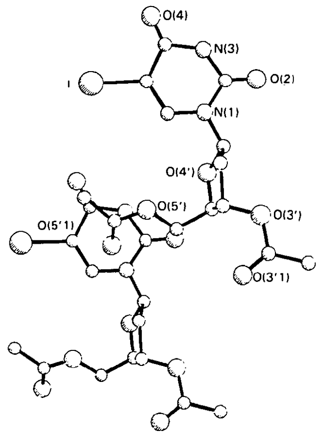

Fig. 3. View perpendicular to the pyrimidine ring, showing stacking of O(5′1).

Hydrogen bonding and molecular stacking. There is one hydrogen bond between N(3) and O(3′1) with an N···O distance of 2·949 (22) Å. The I···I close approach of 4·228 (16) Å has been noted previously (Rahman & Wilson, 1970). Of especial interest is the close approach between I and O(4), with interatomic distance 3·081 (19) Å. A similar configuration to this has been discussed (Camerman & Trotter, 1965) in relation to biological activity in another uridine derivative. The close contact of O(5′1) to the base in an adjacent molecule [2·83 (3) Å] indicates that some sort of interaction exists. The stacking force involved in such an approach could be due to the polarization of the pyrimidine ring by the polar carboxyl O atom, the carboxyl group acting as a dipole pointing down on the ring. This interaction in a different form, in terms of base–base stacking, has been noted (Delbaere & James, 1974) and discussed elsewhere (Bugg, Thomas, Sundaralingam & Rao, 1971). The appearance of it in a non-pyrimidine carboxyl, as here, has not been previously noted.

The above structural points are illustrated in Figs. 2* and 3, which show the molecular configuration and stacking arrangement.

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* See deposition footnote.

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