

Fig. 2. *PLUTO* plot (Motherwell & Clegg, 1978) of the crystal packing along *b*. Thin lines indicate hydrogen bonds.

hydrogen-bond network along the crystallographic *a* axis is formed by O2*B* and H11*B*—N4*B*, while the packing along *b* is determined by hydrogen bonds between O5'*A*—H1*A* and O2*A*.

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Structure of 1-(2-Deoxy- β -D-ribofuranosyl)-5-iodouracil*

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Abstract. 1-(2-Deoxy- β -D-ribofuranosyl)-5-iodouracil, C₉H₁₁IN₂O₅, *M_r* = 354.10, monoclinic, *P*2₁, *a*

= 5.458 (3), *b* = 8.237 (4), *c* = 12.812 (6) Å, β = 98.42 (4)°, *V* = 569.8 (5) Å³, *Z* = 2, *D_m* = 2.05, *D_x* = 2.063 Mg m⁻³, λ (Mo *K* α) = 0.71069 Å, μ = 2.789 mm⁻¹, *F*(000) = 344, *T* = 293 K, final *R* = 0.039 for 1701 unique observed [*F* ≥ 4 σ (*F*)] reflections. The sugar ring adopts a slightly flattened chair conformation. The heterocyclic base is placed in an

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–*ap* (antiperiplanar) orientation [$\chi = 196.4(3)^\circ$] with respect to the sugar moiety. Of the three sugar ring substituents, only O(33') is in an axial position. The packing of the crystal is determined by three intermolecular hydrogen bonds, involving O(33'), O(2) and O(44'). The conformational parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines.

Experimental. Colourless plate-shaped crystals were crystallized from a methanol–acetone solution. Crystal $0.65 \times 0.65 \times 0.11$ mm. Density measured by flotation in 1,1,2,2-tetrabromoethane/ CCl_4 . Space group symmetry $P2_1$ from systematic absences. Stoe STADI-4 diffractometer, cell constants by least-squares refinement of the setting angles of 26 reflections with $20 \leq 2\theta \leq 30^\circ$, $\omega/2\theta$ scan, $[(\sin\theta)/\lambda]_{\text{max}} = 0.7035 \text{ \AA}^{-1}$, $0 \leq h \leq 7$, $0 \leq k \leq 11$, $-18 \leq l \leq 18$ and their Friedel pairs at $-\omega$ and -2θ . Intensities of three standard reflections (200, 020, 002) monitored every 2 h showed no significant decrease in intensity, 3886 reflections measured, 1771 unique reflections of which 1701 were considered observed with $F \geq 4\sigma(F)$, $R_{\text{int}} = 0.0153$. Data reduction with a locally modified version of *REDU4* (Stoe & Co., 1985), Lorentz and polarization corrections, absorption corrections by the method of North, Phillips & Mathews (1968) based on six reflections (151, 130, $12\bar{2}$, 110, $22\bar{2}$, $23\bar{1}$). Scattering factors were taken from Cromer & Waber (1974) and for H atoms from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were included for all non-H atoms (Ibers & Hamilton, 1964). The position of I(5) was obtained from a sharpened Patterson synthesis.

Subsequent Fourier syntheses revealed the remaining non-H atoms. Refined on F by full-matrix least squares, first with isotropic temperature factors and then anisotropically. All H atoms were found in a difference synthesis and they were included in the refinement with a fixed temperature factor $B = 4.0 \text{ \AA}^2$. Final $R = 0.039$, $wR = 0.049$, with $w = 1/[\sigma(F)]^2$, $S = 1.32$. Largest parameter shift/e.s.d. = 0.07. Minimum and maximum residual electron density -1.59 and 1.54 e \AA^{-3} (both near I). The number of reflections per refined parameter $1701/186 = 9.1$. All calculations were performed on a Digital PDP-11/73 and a MicroVAX 2000 microcomputer using *SDP* (Enraf–Nonius, 1985) and *PARST* (Nardelli, 1983).

Discussion. A *PLUTO* (Motherwell & Clegg, 1978) view of the title compound with the atomic numbering scheme is shown in Fig. 1. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths, bond angles and selected torsion angles are given in Table 2.

All bond lengths and bond angles are within the normal range (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The C(2)—O(2) and C(4)—O(4) bonding distances are for a standard C=O double bond, reflecting the presence of the lactam tautomeric form. The pyrimidine heterocycle is essentially planar; only C(1') and O(4) are 0.172 (4) and 0.143 (4) \AA , respectively, above the plane.

The relative orientation of the base with respect to the sugar ring is given by the *N*-glycosidic torsion angle χ [O(5')—C(1')—N(1)—C(2)], which is $-163.6(3)^\circ$, –antiperiplanar (–*ap*). This is in quite good agreement with the antibiotic amicitin in which $\chi = 199.8(5)^\circ$ (Smith & Sundaralingam, 1981), but in total discrepancy with the crystal structure conformation of the nucleoside fragment of the antibiotic gougertin (*C*-substance), in which both bases of the asymmetric unit adopt a –antiperiplanar [–*ac*; $\chi = -93.2(6)^\circ$ for *A* and $-110.2(5)^\circ$ for *B*] orientation (Swaminathan, McAlister & Sundaralingam, 1980).

Using the method of Cremer & Pople (1975), we calculated as phase angles $\varphi_2 = 162(4)^\circ$ and $\theta_2 = 174.9(3)^\circ$, with a total puckering amplitude $Q = 0.566(4) \text{ \AA}$ for the sequence C(1')—C(2')—C(3')—C(4')—C(5')—O(5'). These puckering parameters describe a slightly distorted chair conformation, with Q only slightly lower than for an ideal cyclohexane

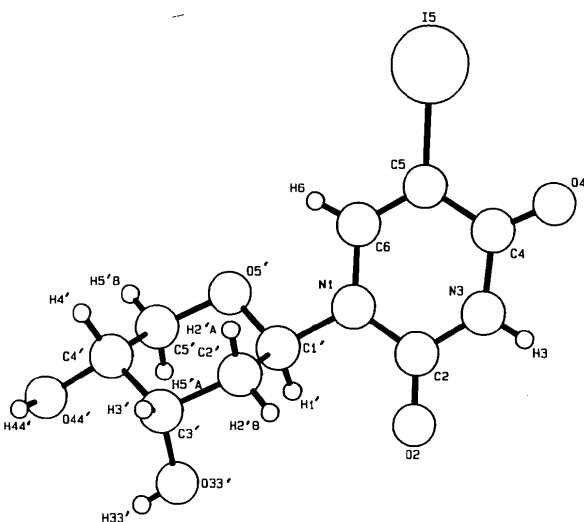


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) plot of the title compound with atomic numbering scheme.

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles including H atoms, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53587 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^4$) with *e.s.d.*'s in parentheses
$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
N(1)	0.1520 (6)	-0.1276 (3)	0.2557 (3)	256 (6)
C(2)	0.0849 (7)	-0.2696 (5)	0.2032 (3)	274 (8)
O(2)	0.1672 (6)	-0.3124 (5)	0.1242 (3)	372 (6)
N(3)	-0.0896 (6)	-0.3587 (5)	0.2444 (3)	330 (8)
C(4)	-0.2092 (7)	-0.3194 (5)	0.3303 (3)	320 (8)
O(4)	-0.3773 (7)	-0.4047 (5)	0.3523 (3)	488 (8)
C(5)	-0.1141 (7)	-0.1733 (5)	0.3845 (3)	276 (8)
I(5)	-0.26722 (4)	-0.1 (†)	0.51567 (2)	429 (6)
C(6)	0.0577 (6)	-0.0831 (5)	0.3459 (3)	268 (6)
C(1')	0.3203 (6)	-0.0192 (4)	0.2077 (3)	232 (6)
C(2')	0.1816 (6)	0.0707 (5)	0.1131 (3)	275 (8)
C(3')	0.3578 (6)	0.1867 (5)	0.0696 (3)	256 (6)
O(33')	0.5315 (6)	0.0891 (4)	0.0245 (3)	350 (6)
C(4')	0.4882 (6)	0.2945 (4)	0.1576 (3)	254 (6)
O(44')	0.6869 (5)	0.3790 (3)	0.1208 (3)	317 (6)
C(5')	0.5991 (7)	0.1935 (5)	0.2522 (3)	302 (8)
O(5')	0.4173 (5)	0.0900 (4)	0.2865 (3)	312 (6)

† Origin-defining.

Table 2. Bond lengths (\AA), bond angles ($^\circ$) and selected torsion angles ($^\circ$) with *e.s.d.*'s in parentheses

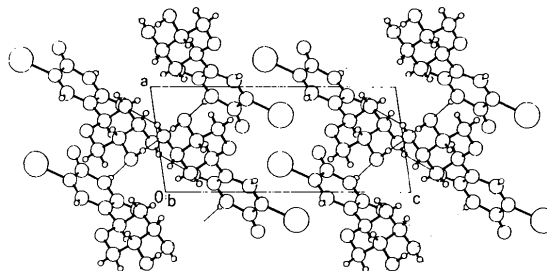
N(1)—C(2)	1.372 (5)	C(5)—C(6)	1.346 (5)
N(1)—C(6)	1.382 (5)	C(1')—C(2')	1.524 (6)
N(1)—C(1')	1.479 (5)	C(1')—O(5')	1.398 (5)
C(2)—O(2)	1.217 (5)	C(2')—C(3')	1.518 (5)
C(2)—N(3)	1.368 (5)	C(3')—O(33')	1.428 (6)
N(3)—C(4)	1.397 (6)	C(3')—C(4')	1.527 (6)
C(4)—O(4)	1.221 (6)	C(4')—O(44')	1.425 (5)
C(4)—C(5)	1.447 (6)	C(4')—C(5')	1.521 (5)
C(5)—I(5)	2.075 (4)	C(5')—O(5')	1.425 (6)
C(6)—N(1)—C(1')	121.2 (3)	N(1)—C(6)—C(5)	121.7 (3)
C(2)—N(1)—C(1')	116.6 (3)	N(1)—C(1')—O(5')	106.3 (3)
C(2)—N(1)—C(6)	122.1 (3)	N(1)—C(1')—C(2')	111.0 (3)
N(1)—C(2)—N(3)	114.7 (4)	C(2')—C(1')—O(5')	110.7 (3)
N(1)—C(2)—O(2)	123.4 (4)	C(1')—C(2')—C(3')	109.3 (3)
O(2)—C(2)—N(3)	121.8 (4)	C(2')—C(3')—C(4')	110.3 (3)
C(2)—N(3)—C(4)	127.7 (4)	C(2')—C(3')—O(33')	106.8 (3)
N(3)—C(4)—C(5)	113.2 (4)	O(33')—C(3')—C(4')	111.1 (3)
N(3)—C(4)—O(4)	120.1 (4)	C(3')—C(4')—C(5')	111.0 (3)
O(4)—C(4)—C(5)	126.6 (4)	C(3')—C(4')—O(44')	109.6 (3)
C(4)—C(5)—C(6)	120.2 (4)	O(44')—C(4')—C(5')	107.3 (3)
C(4)—C(5)—I(5)	118.2 (3)	C(4')—C(5')—O(5')	111.1 (3)
I(5)—C(5)—C(6)	121.4 (3)	C(1')—O(5')—C(5')	111.7 (3)
C(6)—N(1)—C(1')—C(2')	-100.7 (4)	C(1')—C(2')—C(3')—C(4')	-52.2 (4)
C(2)—N(1)—C(1')—C(2)	76.0 (4)	C(2')—C(3')—C(4')—O(44')	168.4 (3)
C(6)—N(1)—C(1')—O(5')	19.7 (4)	C(2')—C(3')—C(4')—C(5')	50.1 (4)
C(2)—N(1)—C(1')—O(5')	-163.6 (3)	O(33')—C(3')—C(4')—O(44')	50.2 (4)
N(1)—C(1')—O(5')—C(5')	175.3 (3)	O(33')—C(3')—C(4')—C(5')	-68.1 (4)
N(1)—C(1')—C(2')—C(3')	177.2 (3)	C(3')—C(4')—C(5')—O(5')	-53.4 (4)
C(2')—C(1')—O(5')—C(5')	-64.1 (4)	O(44')—C(4')—C(5')—O(5')	-173.1 (3)
O(5')—C(1')—C(2')—C(3')	59.4 (4)	C(4')—C(5')—O(5')—C(1')	60.9 (4)
C(1')—C(2')—C(3')—O(33')	68.6 (4)		

chair (0.63 \AA ; Cremer & Pople, 1975) and slightly flattened at the C(4') apex. Of the three sugar ring substituents, only O(33') is placed in an axial way on the ring, while both the base and O(44') are found to be in an equatorial position.

Table 3 summarizes all the intra- and intermolecular hydrogen bonds. A *PLUTO* (Motherwell & Clegg, 1978) plot of the crystal packing is shown in Fig. 2.

Table 3. Geometry of intra- and intermolecular hydrogen bonds (\AA , $^\circ$) with *e.s.d.*'s in parentheses

$X-H \cdots Y$	$d(H \cdots Y)$	$d(X \cdots Y)$	$X-H \cdots Y$
O(33')—H(33') \cdots O(44')	2.49 (8)	2.764 (4)	103 (8)
N(3)—H(3) \cdots O(44')	2.02 (8)	2.846 (5)	164 (8)
O(33')—H(33') \cdots O(2)''	2.14 (9)	2.813 (5)	147 (8)
O(44')—H(44') \cdots O(33)''	1.78 (8)	2.689 (4)	173 (8)

Symmetry code: (i) $x - 1, y - 1, z$; (ii) $1 - x, y + \frac{1}{2}, -z$.Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) plot of the crystal packing along *b*. Thin lines indicate hydrogen bonds.

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