order to prove the predicted reaction pathway and to determine the preferred direction of the bridge-opening reaction, a crystal structure determination was performed. For further details see Lee, Hahn & Noland (1978), Sewarte-Roß (1989) and Sha & Tsou (1990). For preparation and spectroscopic data of tricyclic 14π-hetarenes see Dyker (1988) and Götte (1991).

References

Acheson, R. M. & Vernon, J. M. (1962). *J. Chem. Soc.* pp. 1148–1157.

Bender, C. O., Bonnett, R. & Smith, R. G. (1970). *J. Chem. Soc. C*, pp. 1251–1257.

Diels, O., Alder, K. & Winckler, H. (1931). *Justus Liebigs Ann. Chem.* **490**, 267–276.

Dyker, G. (1988). Dissertation, Univ. of Dortmund, Germany.

Götte, H. (1991). Dissertation (in progress), Univ. of Dortmund, Germany.

Kreher, R. P., Hennige, H., Jelitto, F. & Preut, J. (1989). *Z. Naturforsch. Teil B*, **44**, 1132–1148.

Kricka, L. J. & Vernon, J. M. (1971). *J. Chem. Soc. Chem. Commun.* pp. 942–943.

Kricka, L. J. & Vernon, J. M. (1972). *J. Chem. Soc. Perkin Trans.* 1, pp. 904–908.

Lee, C. K., Hahn, C. S. & Noland, W. E. (1978). *J. Org. Chem.* **43**, 3727–3729.

Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Palmer, M. H. & Kennedy, S. M. F. (1974). *J. Chem. Soc. Perkin Trans.* 2, pp. 1893–1903.

Rogers, D. (1981). *Acta Cryst.* A**37**, 734–741.

Schmitt, D. (1967). Dissertation, TH Darmstadt, Germany.

Sewarte-Roß, G. (1989). Dissertation, Univ. of Dortmund, Germany.

Sha, C. K. & Tsou, C. P. (1990). *J. Org. Chem.* **55**, 2446–2450.

Sheldrick, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet *R3m/V* crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Spek, A. L. (1982). *The EUCLID Package*. In *Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.

Williams, D. E. (1984). *PCK83. A Crystal and Molecular Packing Analysis Program*. Univ. of Louisville, QCPE Program No. 481. Department of Chemistry, Indiana Univ., Indiana, USA.

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Structure of 1-(2-Deoxy-2-fluoro-α-D-arabinopyranosyl)-5-iodouracil*

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Abstract. 1-(2-Deoxy-2-fluoro-α-D-arabinopyranosyl)-5-iodouracil, $C_9H_{10}FIN_2O_5$, $M_r = 372{\cdot}09$, triclinic, $P1$, $a = 5{\cdot}098\,(2)$, $b = 6{\cdot}470\,(2)$, $c = 9{\cdot}293\,(6)$ Å, $\alpha = 81{\cdot}10\,(2)$, $\beta = 84{\cdot}91\,(3)$, $\gamma = 79{\cdot}38\,(2)^\circ$, $V = 297{\cdot}1\,(2)$ Å^3, $Z = 1$, $D_m = 2{\cdot}08$, $D_x = 2{\cdot}080$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 2{\cdot}692$ mm^{-1}, $F(000) = 180$, $T = 293$ K, final $R = 0{\cdot}024$ for 1702 unique observed $[F \geq 4\sigma(F)]$ reflections. The pyranose ring adopts a chair conformation with $\varphi_2 = -19\,(4)^\circ$, $\theta_2 = 8{\cdot}9\,(5)^\circ$ and $Q_t = 0{\cdot}596\,(5)$ Å. The N-glycosidic torsion angle χ [O(5′)—C(1′)—N(1)—C(2)] between this ring and the pyrimidine base is oriented $+ac$ [95·1 (4)°]. 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 crystals were crystallized from a methanol solution, $0{\cdot}38 \times 0{\cdot}15 \times 0{\cdot}23$ mm. Density measured by flotation in 1,1,2,2-tetrabromoethane/CCl_4. Weissenberg photographs showed no systematically absent reflections. Stoe STADI-4 computer-controlled diffractometer, cell constants by least-squares refinement of the θ angles of 28 reflections with $20 \leq 2\theta \leq 30^\circ$, $\omega/2\theta$ scan, $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}7034$ Å^{-1}, $-7 \leq h \leq 7$, $-9 \leq k \leq 9$, $-12 \leq l \leq 12$. Intensities of two standard reflections $(1\bar{1}\bar{1}, 1\bar{1}1)$ monitored every two hours showed no

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[*] Structural Studies on Modified Nucleosides. XII. Part XI: Van Aerschot, Everaert, Gosselin, Peeters, Blaton, De Ranter, Imbach, Balzarini, De Clercq & Herdewijn (1990).

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significant decrease in intensity, 3430 reflections measured, 1702 unique reflections of which all were considered observed [$F \geq 4\sigma(F)$], $R_{\rm int}$ on $F = 0{\cdot}013$. Data reduction with a locally modified version of the *REDU*4 program (Stoe & Co., 1985). Lorentz and polarization corrections. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) and for H atoms from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were included for all non-H atoms (Ibers & Hamilton, 1964). No absorption corrections were applied. The structure was solved by a Fourier synthesis with phases from the I atom which was placed at an arbitrary position. Refined on F by full-matrix least squares, first with isotropic temperature factors and finally anisotropically. All H atoms were found in a difference synthesis and, with the exception of H(4) and H(6), placed at a riding distance of 0·95 Å with a fixed temperature factor B 1·3 times the $B_{\rm eq}$ of their parent atoms. H(4) and H(6) were placed at their located positions and included in the refinement with a fixed temperature factor B 1·3 times the $B_{\rm eq}$ of O(44′) and O(33′) respectively. Final $R = 0{\cdot}024$, $wR = 0{\cdot}030$, with $w = 1/[\sigma^2(F_o) + 0{\cdot}0004F_o^2]$, $S = 1{\cdot}40$. Largest parameter shift/e.s.d = 0·18. Min. and max. residual electron density $-2{\cdot}06$ and $0{\cdot}80$ e Å^{-3} (both peaks near the I atom). The number of reflections per

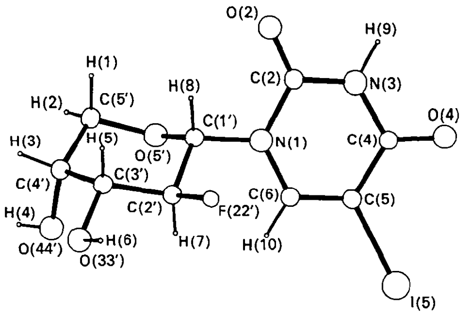

Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) plot of the title compound with atomic numbering scheme.

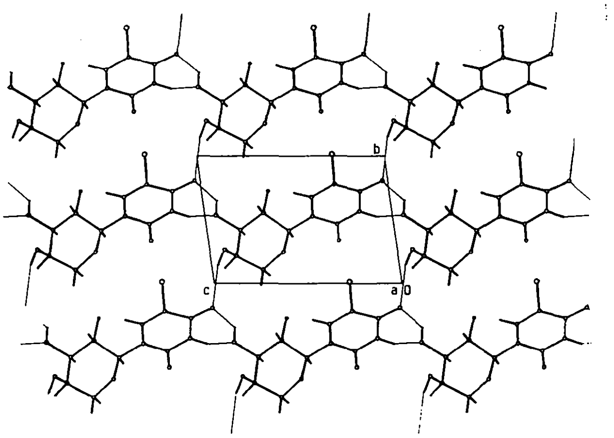

Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) plot of the crystal along **a** showing the packing. Thin lines indicate hydrogen bonds.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2 × 10^4) *with e.s.d.'s in parentheses*

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

	x	y	z	$U_{\rm eq}$
N(1)	0·4975 (7)	0·5290 (5)	0·4279 (3)	272 (7)
C(2)	0·4209 (9)	0·4693 (6)	0·3022 (4)	296 (8)
O(2)	0·2729 (9)	0·3409 (6)	0·3065 (4)	497 (9)
N(3)	0·5188 (9)	0·5683 (6)	0·1737 (3)	316 (8)
C(4)	0·6745 (8)	0·7238 (6)	0·1554 (4)	265 (8)
O(4)	0·7435 (8)	0·8053 (6)	0·0318 (3)	369 (9)
C(5)	0·7474 (8)	0·7786 (6)	0·2904 (4)	265 (8)
I(5)	0·976*	1·013*	0·281*	311 (1)
C(6)	0·6604 (8)	0·6783 (6)	0·4194 (4)	277 (9)
C(1′)	0·4074 (9)	0·4177 (6)	0·5660 (4)	283 (9)
C(2′)	0·3403 (8)	0·5535 (6)	0·6884 (4)	279 (8)
F(22′)	0·1383 (8)	0·7255 (6)	0·6453 (4)	485 (10)
C(3′)	0·2338 (9)	0·4221 (8)	0·8238 (4)	335 (11)
O(33′)	0·1971 (9)	0·5384 (8)	0·9430 (4)	509 (11)
C(4′)	0·430 (1)	0·2160 (8)	0·8636 (4)	362 (11)
O(44′)	0·6534 (8)	0·2681 (7)	0·9230 (4)	410 (9)
C(5′)	0·514 (2)	0·1061 (8)	0·7300 (6)	524 (18)
O(5′)	0·614 (1)	0·2476 (6)	0·6114 (4)	429 (10)

* Parameters kept fixed during refinement.

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

C(2)	O(2)	1·215 (7)	C(1′)	C(2′)	1·521 (5)
C(2)	N(3)	1·364 (5)	C(1′)	O(5′)	1·416 (5)
C(4)	O(4)	1·237 (5)	C(2′)	F(22′)	1·404 (5)
C(4)	C(5)	1·451 (6)	C(2′)	C(3′)	1·523 (5)
C(5)	I(5)	2·065 (4)	C(3′)	C(4′)	1·529 (6)
C(5)	C(6)	1·352 (5)	C(3′)	O(33′)	1·413 (6)
N(1)	C(2)	1·394 (5)	C(4′)	O(44′)	1·423 (7)
N(1)	C(6)	1·374 (6)	C(4′)	C(5′)	1·515 (7)
N(1)	C(1′)	1·452 (4)	C(5′)	O(5′)	1·440 (7)
N(3)	C(4)	1·374 (6)			

N(1)	C(2)	O(2)	122·4 (3)	I(5)	C(5)	C(6)	121·5 (3)
N(1)	C(2)	N(3)	115·4 (4)	C(6)	N(1)	C(1′)	122·5 (3)
N(1)	C(6)	C(5)	122·2 (4)	C(1′)	C(2′)	F(22′)	108·8 (3)
N(1)	C(1′)	C(2′)	114·5 (3)	C(1′)	C(2′)	C(3′)	109·3 (4)
N(1)	C(1′)	O(5′)	108·2 (3)	C(1′)	O(5′)	C(5′)	110·0 (4)
C(2)	N(1)	C(6)	121·0 (3)	C(2′)	C(1′)	O(5′)	107·5 (3)
C(2)	N(1)	C(1′)	116·4 (4)	C(2′)	C(3′)	O(33′)	110·0 (4)
C(2)	N(3)	C(4)	127·3 (4)	C(2′)	C(3′)	C(4′)	110·6 (3)
O(2)	C(2)	N(3)	122·2 (4)	F(22′)	C(2′)	C(3′)	108·2 (3)
N(3)	C(4)	O(4)	120·7 (4)	C(3′)	C(4′)	O(44′)	107·9 (4)
N(3)	C(4)	C(5)	114·5 (3)	C(3′)	C(4′)	C(5′)	110·2 (4)
C(4)	C(5)	I(5)	119·0 (3)	O(33′)	C(3′)	C(4′)	108·6 (3)
C(4)	C(5)	C(6)	119·5 (4)	C(4′)	C(5′)	O(5′)	111·1 (4)
O(4)	C(4)	C(5)	124·8 (4)	O(44′)	C(4′)	C(5′)	111·6 (5)

C(6)	N(1)	C(1′)	C(2′)	38·2 (5)	C(1′)	C(2′)	C(3′)	O(33′)	173·8 (4)
C(2)	N(1)	C(1′)	C(2′)	−144·9 (4)	F(22′)	C(2′)	C(3′)	O(33′)	−67·9 (5)
C(6)	N(1)	C(1′)	O(5′)	−81·8 (5)	F(22′)	C(2′)	C(3′)	C(4′)	172·2 (4)
C(2)	N(1)	C(1′)	O(5′)	95·1 (4)	C(2′)	C(3′)	C(4′)	O(44′)	73·1 (5)
N(1)	C(1′)	C(2′)	F(22′)	58·8 (5)	C(2′)	C(3′)	C(4′)	C(5′)	−49·0 (5)
N(1)	C(1′)	C(2′)	C(3′)	176·8 (3)	O(33′)	C(3′)	C(4′)	O(44′)	−47·7 (5)
N(1)	C(1′)	O(5′)	C(5′)	−50·0 (6)	O(33′)	C(3′)	C(4′)	C(5′)	−169·8 (4)
C(2′)	C(1′)	O(5′)	C(5′)	68·2 (5)	C(3′)	C(4′)	C(5′)	O(5′)	53·4 (6)
O(5′)	C(1′)	C(2′)	C(3′)	−62·9 (4)	O(44′)	C(4′)	C(5′)	O(5′)	−66·4 (6)
C(1′)	C(2′)	C(3′)	C(4′)	53·9 (5)	C(4′)	C(5′)	O(5′)	C(1′)	−64·4 (6)

refined parameter 1702/166 = 10·3. 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. A *PLUTO* (Motherwell & Clegg, 1978) plot of the crystal packing is shown in Fig. 2. All calculations were performed on a Digital PDP-11/73 microcomputer using *SDP* (B. A. Frenz & Associates, Inc., 1982) and *PARST* (Nardelli, 1983).

Related literature. All bond lengths and bond angles are within the normal range (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The Cremer & Pople (1975) puckering parameters for the sequence C(1′)—C(2′)—C(3′)—C(4′)—C(5′)—O(5′) are $\varphi_2 = -19\,(4)°$ and $\theta_2 = 8{\cdot}9\,(5)°$ with $Q_t = 0{\cdot}596\,(5)$ Å, indicating a chair conformation. The crystal packing is stabilized by hydrogen bonds involving both rings (Table 3) and by base stacking forces (mean interplanar distance between adjacent bases = 3·5 Å; dihedral angle = 0·0°).

Table 3. *Geometry* (Å, °) *of intra- and intermolecular hydrogen bonds with e.s.d.'s in parentheses*

X—H···*Y*	*d*(H···*Y*)	*d*(*X*···*Y*)	*X*—H···*Y*
O(44′)—H(4)···O(4) (1)	2·2 (1)	2·973 (6)	138 (7)
O(33′)—H(6)···O(4) (2)	1·9 (1)	2·764 (6)	140 (10)
N(3)—H(9)···O(33′) (3)	2·013 (5)	2·862 (6)	147·7 (3)

Equivalent positions: (1) *x*, *y* − 1, *z* + 1; (2) *x* − 1, *y*, *z* + 1; (3) *x*, *y*, *z* − 1.

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54213 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ALLEN, F. H., KENNARD, O., WATSON, D. G., BRAMMER, L., ORPEN, A. G. & TAYLOR, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

B. A. FRENZ & ASSOCIATES, INC. (1982). *SDP Structure Determination Package.* College Station, Texas, USA.

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

IBERS, J. A. & HAMILTON, W. C. (1964). *Acta Cryst.* **17**, 781–782.

IUPAC–IUB JOINT COMMISSION ON BIOCHEMICAL NOMENCLATURE (1983). *Pure Appl. Chem.* **55**, 1273–1280.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Stoe & Co. (1985). *REDU4. Data Reduction Program.* Stoe & Co., Darmstadt, Germany.

VAN AERSCHOT, A., EVERAERT, D., GOSSELIN, G., PEETERS, O., BLATON, N., DE RANTER, C., IMBACH, J.-L., BALZARINI, J., DE CLERCQ, E. & HERDEWIJN, P. (1990). *Antiviral Res.* **14**, 357–369.

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Structures of Two Modifications of 2,2′:4′,4″:2″,2‴-Quaterpyridine

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Abstract. $C_{20}H_{14}N_4$, $M_r = 310{\cdot}4$. α-Form: monoclinic, $P2_1/c$, $a = 9{\cdot}434\,(8)$, $b = 6{\cdot}110\,(4)$, $c = 13{\cdot}322\,(10)$ Å, $\beta = 105{\cdot}02\,(6)°$, $V = 741{\cdot}7\,(9)$ Å^3, $Z = 2$, $D_x = 1{\cdot}39$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 324$, $T = 160$ K, $R = 0{\cdot}052$ for 976 independent observed reflections. β-Form: monoclinic, $P2_1/n$, $a = 7{\cdot}224\,(5)$, $b = 11{\cdot}530\,(6)$, $c = 8{\cdot}978\,(6)$ Å, $\beta = 91{\cdot}21\,(5)°$, $V = 747{\cdot}7\,(8)$ Å^3, $Z = 2$, $D_x = 1{\cdot}38$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 324$, $T = 160$ K, $R = 0{\cdot}047$ for 617 independent observed reflections. In both forms the molecules exist in a centrosymmetric planar conformation with *transoid* 2,2′- (and 2″,2‴-) linkages and pack with a common herringbone pattern but with different molecular stackings.

Experimental. Recrystallization of the title compound (1) from ethanol gave a mixture of two macroscopically distinguishable modifications, crystals of which were separated manually. The α-form

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