Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1141). Services for accessing these data are described at the back of the journal.

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FF-β-D-Arabinofuranosyluracil

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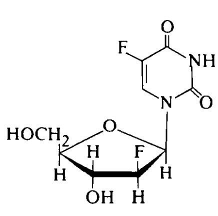
Abstract

In the title compound, 1-(2-deoxy-2-fluoro-β-D-arabinofuranosyl)-5-fluoropyrimidine-2,4(1*H*,3*H*)-dione, $C_9H_{10}F_2N_2O_5$, the furanosyl ring adopts the twisted conformation (*T*) with O1′ *endo* and C1′ *exo*. The crystal structure is characterized by a three-dimensional hydrogen-bond network involving the three H atoms bonded to heteroatoms.

Comment

FF-β-D-Arabinofuranosyluracil (FFaraU) was synthesized for possible use in positron-emission-tomography (PET) imaging of *Herpes-simplex*-virus-transformed cells and tumors. Expression of the *Herpes simplex* thymidine kinase gene in tumors, after transfection with a gene therapy retroviral vector, could be monitored through the use of positron-emitting ^{18}F-labeled nucleosides which are incorporated preferentially into the target cells. The antiviral properties of FFaraU suggest it is 5′-monophosphorylated by the viral kinase; the phosphorylated compound is known to bind to thymidylate synthetase (Coderre *et al.*, 1983), and it is not phosphorylated by host-cell thymidine kinase, indicating its suitability for these studies. We undertook the structure elucidation of FFaraU in conjunction with biological testing of a series of fluorinated nucleosides (Shields *et al.*, 1996) in order to correlate structural characteristics with cellular activity.



FFaraU

The molecular structure is presented in Fig. 1. The molecule can be described as having an *anti* orientation of the base with respect to the sugar ring; the χ torsion angle O1′—C1′—N1—C6 is 21.4 (3)° (the ring oxygen O1′ is often designated O4′ by organic chemists). The sugar moiety adopts a twisted conformation (*T*) with O1′ *endo* and C1′ *exo*. The deviations of the two atoms from the best plane containing the remaining sugar ring atoms are: 0.56 (1) and 0.54 (1) Å for O1′ and C1′, respectively. Puckering parameters for the sugar moiety are: $Q(2) = 0.388\,(3)$ Å and $\varphi(2) = 12.1\,(4)°$ (Cremer & Pople, 1975). Pseudorotation parameters are: $P = 101.6\,(2)$ and $\tau(M) = 43.2\,(1)°$ for reference bond C2′—C3′ (Rao *et al.*, 1981). The sugar pucker differs from that observed in 5-nitro-1-β-D-arabinofuranosyluracil (Biswas *et al.*, 1988) and uracil-β-D-arabinofuranoside (Tollin *et al.*, 1973), where the puckering is C2′-*endo*, and 1-β-D-arabinofuranosyl-4-thiouracil (Saenger, 1972), where the puckering is C3′-*endo*. The 2′-F atom is axial to the ring. The uracil ring is planar and the bond lengths and angles for the whole molecule conform to accepted values with no significant deviations. In the crystal structure, the molecules are held together through hydrogen bonds from the donating heteroatoms (N3, O3′ and O5′ to O3′, O4 and O5′), forming a three-dimensional infinite network.

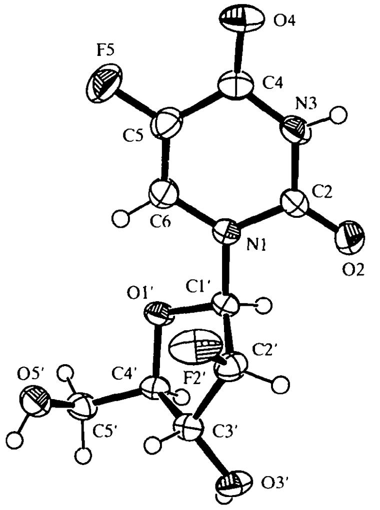


Fig. 1. *ORTEP*-3 (Farrugia, 1998) view of the title compound showing 50% probability displacement ellipsoids. H atoms are drawn as small circles of arbitrary radii.

Experimental

FFaraU (Watanabe *et al.*, 1979) was synthesized at the University of Washington (Seattle, USA). Crystals were obtained by crystallization from a methanol–water mixture.

Crystal data

$C_9H_{10}F_2N_2O_5$
$M_r = 264.19$
Tetragonal
$P4_1$
$a = 9.975 (2)$ Å
$c = 10.418 (2)$ Å
$V = 1036.6 (4)$ Å^3
$Z = 4$
$D_x = 1.693$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 16 reflections
$\theta = 25$–$50°$
$\mu = 1.419$ mm^{-1}
$T = 293 (2)$ K
Transparent block
0.6 × 0.4 × 0.4 mm
Colorless

Data collection

Picker FACS-1 four-circle diffractometer
$\theta/2\theta$ scan
Absorption correction: none
1049 measured reflections
937 independent reflections
918 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.027$
$\theta_{\rm max} = 64.77°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 12$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R = 0.028$
$wR = 0.092$
$S = 0.865$
937 reflections
166 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.193$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.143$ e Å^{-3}

Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.026 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Absolute structure: Flack (1983)
Flack parameter = −0.10 (18)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O3′$^{\rm i}$	0.86	1.96	2.811 (3)	169
O3′—H3′1···O4$^{\rm ii}$	0.82	1.90	2.715 (4)	175
O5′—H5′···O5′$^{\rm iii}$	0.82	2.15	2.920 (4)	157

Symmetry codes: (i) $y, 1-x, \frac{3}{4}+z$; (ii) $x, y, z-1$; (iii) $1+y, 1-x, z-\frac{1}{4}$.

The space group was determined from precession camera photographs. Cell parameters were determined by least-squares refinement of 16 high-angle reflections. The intensities were collected up to the geometrical limits of a PICKER FACS-1 four-circle diffractometer ($3 < 2\theta < 130°$). A total of 937 independent reflections, subjected to Lorentz and polarization corrections, were measured and used in the structure analysis and refinement. All non-H atoms were found from the best solution E map. Refinement was by full-matrix least squares. Due to paucity of data, all H atoms were refined as riding on their respective host atoms. Dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C).

Data collection: Picker software. Cell refinement: Picker software. Data reduction: Picker software. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1998). Software used to prepare material for publication: *SHELXL*97.

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