

# 5-Fluoro-1-[(4*S*,5*R*)-5-(2-hydroxyethyl)-2,2-dimethyl-1,3-dioxolan-4-yl]-pyrimidine-2,4(1*H*,3*H*)-dione

Angel Mendoza,<sup>a\*</sup> Martha Sosa-Rivadeneira,<sup>b</sup> Fernando Sartillo-Piscil,<sup>b</sup> Leticia Quintero<sup>b</sup> and Marcos Flores-Alamo<sup>c</sup>

<sup>a</sup>Centro de Química, ICUAP, Benemérita Universidad Autónoma de Puebla, Puebla, Pue., Mexico, <sup>b</sup>Facultad de Ciencias Químicas, Benemérita Universidad Autónoma de Puebla, Puebla, Pue., Mexico, and <sup>c</sup>Facultad de Química, Universidad Nacional Autónoma de México, 04510, México, DF, Mexico  
Correspondence e-mail: angel.mendoza.m@gmail.com

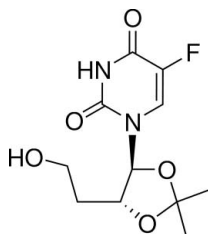
Received 26 April 2010; accepted 30 April 2010

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.027;  $wR$  factor = 0.071; data-to-parameter ratio = 9.9.

In the title compound,  $\text{C}_{11}\text{H}_{15}\text{FN}_2\text{O}_5$ , the five-membered ring has an envelope conformation, while the six-membered ring is essentially planar, with a maximum deviation of 0.032 (2) Å from the mean plane. The crystal packing is stabilized by intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, generating a layer structure parallel to (001).

## Related literature

For applications of modified nucleosides in medical chemistry, see: Huryn & Okabe (1992); Minuk *et al.* (1992); Luscombe *et al.* (1996); Korba & Boyd (1996). For the synthesis, see: Valdivia *et al.* (2005); Xie *et al.* (1996). For ring conformation analysis, see: Cremer & Pople (1975).



## Experimental

### Crystal data

$\text{C}_{11}\text{H}_{15}\text{FN}_2\text{O}_5$   
 $M_r = 274.25$   
Monoclinic,  $C2$

$a = 20.8905$  (8) Å  
 $b = 5.5751$  (1) Å  
 $c = 13.5639$  (5) Å

$\beta = 126.297$  (6)°  
 $V = 1273.21$  (12) Å<sup>3</sup>  
 $Z = 4$   
Cu  $K\alpha$  radiation

$\mu = 1.06$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.40 \times 0.12 \times 0.08$  mm

### Data collection

Oxford Diffraction Gemini Atlas CCD diffractometer  
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2009)  
 $T_{\min} = 0.885$ ,  $T_{\max} = 0.964$

4606 measured reflections  
1786 independent reflections  
1732 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.013$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.071$   
 $S = 1.04$   
1786 reflections  
181 parameters  
1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.23$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.20$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983), 498 Friedel pairs  
Flack parameter: 0.0 (2)

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H1N}\cdots\text{O5}^i$	0.83 (2)	2.01 (2)	2.828 (2)	167 (3)
$\text{O5}-\text{H1O}\cdots\text{O2}^ii$	0.75 (3)	2.16 (3)	2.876 (2)	160 (3)

Symmetry codes: (i)  $-x + \frac{3}{2}, y - \frac{1}{2}, -z + 2$ ; (ii)  $x + \frac{1}{2}, y + \frac{1}{2}, z$ .

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Special thanks to BUAP for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2543).

## References

- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.  
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.  
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
Huryn, D. M. & Okabe, M. (1992). *Chem. Rev.* **92**, 1745–1768.  
Korba, B. E. & Boyd, M. R. (1996). *Antimicrob. Agents Chemother.* **40**, 1282–1284.  
Luscombe, C., Pedersen, J., Uren, E. & Locarnini, S. (1996). *Hepatology* **24**, 766–773.  
Minuk, G. Y., German, G. B., Bernstein, C., Benarroch, A., Gauthiar, T. & Sekla, L. (1992). *Clin. Invest. Med.* **15**, 506–512.  
Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Valdivia, V., Hernández, A., Rivera, A., Sartillo-Piscil, F., Loukaci, A., Fourrey, J.-L. & Quintero, L. (2005). *Tetrahedron Lett.* **46**, 6511–6514.  
Xie, M., Berges, D. A. & Robins, M. (1996). *J. Org. Chem.* **61**, 5178–5179.

## supporting information

*Acta Cryst.* (2010). E66, o1317 [https://doi.org/10.1107/S1600536810016065]

## 5-Fluoro-1-[(4*S*,5*R*)-5-(2-hydroxyethyl)-2,2-dimethyl-1,3-dioxolan-4-yl]pyrimidine-2,4(1*H*,3*H*)-dione

Angel Mendoza, Martha Sosa-Rivadeneira, Fernando Sartillo-Piscil, Leticia Quintero and Marcos Flores-Alamo

### S1. Comment

For many years, design of modified nucleosides has been a focal point of research in medicinal chemistry (Huryn & Okabe, 1992). Modified nucleosides have acquired an important role as therapeutic agents for the treatment of patients with devastating infections with viruses such as human immunodeficiency virus (HIV), hepatitis B virus (HBV), and herpes viruses. A class of nucleoside analogues for antiviral chemotherapy is that where cyclic carbohydrate moiety is replaced with open-chain "acyclic" sugar moieties. Among purine acyclic nucleosides, are Acyclovir, Ganciclovir and Penciclovir (Minuk *et al.*, 1992; Luscombe *et al.*, 1996; Korba & Boyd, 1996).

In this context and as result of our continuing investigations on the synthesis of nucleoside analogues, we report a new compound **1** (Scheme 1). This new analogue might present a similarity with a number of acyclic nucleosides, which showed remarkable antiviral properties.

In the present paper, we report the structure of title compound **1**. In the [(1'*S*, 2'*R*)-(1', 2'-*O*-isopropylidene-4'-hydroxy-1-butyl)], the five member ring (C5/C6/O3/O4/C9) shows an envelope conformation on atom C6 with puckering parameters (Cremer & Pople, 1975)  $q_2 = 0.238$  (2) Å and  $\varphi_2 = 69.8$  (6)°. For the six member ring uracil, shows a planar configuration with torsion angle (N1—C4—N2—C3) of 4.8 (3)°, and C1—C2 = 1.325 (3) Å and N2—C4 = 1.384 (2) Å (double bond). The crystal packing is stabilized by two intermolecular hydrogen bonds [O5...O2 = 2.828 (2) Å and N2...O5 = 2.876 (2) Å], generating a layer parallel to the (001) plane.

### S2. Experimental

Deshomologation of previous nucleoside analogue, 1-[(1'*S*,2'*R*,4'*S*)-(1',2'-*O*-isopropylidene-4',5'-dihydroxy-1'-pentyl)]-5-fluorouracil, was achieved following non-aqueous protocol (Valdivia *et al.*, 2005; Xie *et al.*, 1996). Reaction was carried out by two steps: a) periodic acid/ethyl acetate, 30 min, b) EtOH/H<sub>2</sub>O/ NaBH<sub>4</sub>, 20 min, rt. Final purification of compound **1** was achieved by crystallization from hexane. Yield 80%, white solid, m.p. 184 °C;  $[\alpha]_D -18.51$  (c 1.0, CH<sub>3</sub>OH). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>/TMS) 1.53 (s, 3H), 1.57 (s, 3H), 1.95 (m, 1H), 2.11 (m, 1H), 3.80 (m, 2H), 4.21 (m, 1H), 5.90 (d, 1H, J = 5.1 Hz), 7.3 (s, 1H). <sup>13</sup>C MNR (75 MHz, CDCl<sub>3</sub>/TMS) 26.9, 27.9, 34.9, 55.8, 58.9, 79.4, 86.8, 111.6, 142.2, 149.5, 157.5.

### S3. Refinement

H atoms bonded to N2 and O5 atoms were located in a difference Fourier map and refined with free coordinates and isotropic *U* parameters. H atoms linked to C atoms were placed in geometrical idealized positions and refined as riding on their parent atoms, with C—H = 0.93–0.98 Å, and with  $U_{iso}(H) = 1.2U_{eq}(C)$  or  $1.5U_{eq}(C)$  for methyl groups.

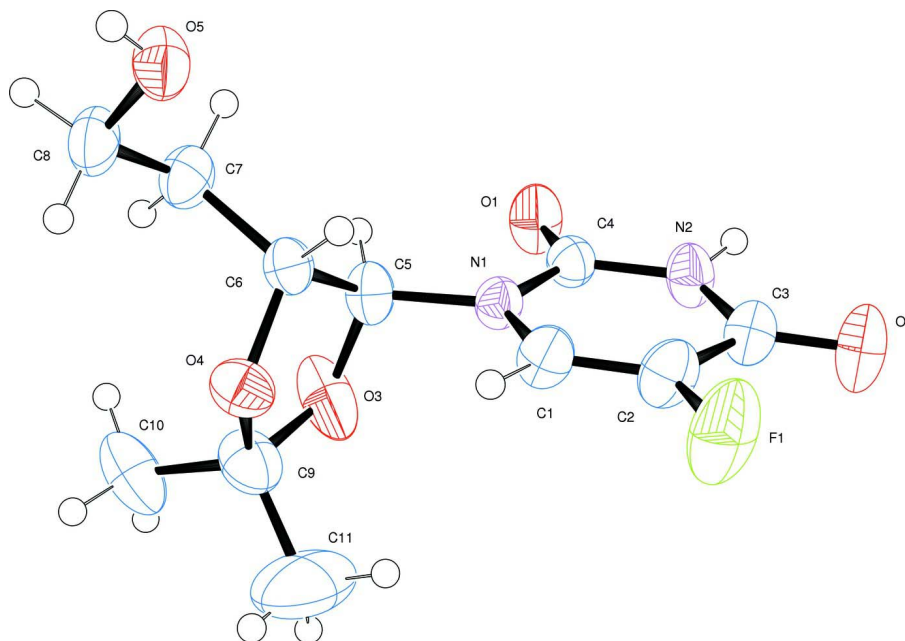


Figure 1

The molecular structure of compound **1**, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

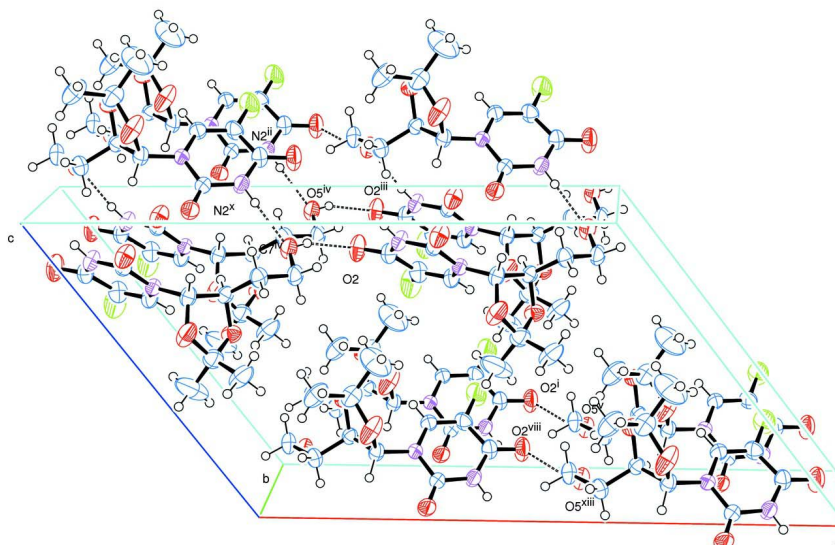
H1n<sup>x</sup>

Figure 2

The packing of compound **1**, viewed down the *b* axis, showing one layer of molecules connected by O5—H $\cdots$ O2 and N2—H $\cdots$ O5 hydrogen bonds (dashed lines).

5-Fluoro-1-[(4*S*,5*R*)-5-(2-hydroxyethyl)-2,2-dimethyl-1,3-dioxolan-4-yl]pyrimidine-2,4(1*H*,3*H*)-dione

*Crystal data*

C<sub>11</sub>H<sub>15</sub>FN<sub>2</sub>O<sub>5</sub>  
*M<sub>r</sub>* = 274.25

Monoclinic, *C*2  
 Hall symbol: C 2y

$a = 20.8905$  (8) Å  
 $b = 5.5751$  (1) Å  
 $c = 13.5639$  (5) Å  
 $\beta = 126.297$  (6)°  
 $V = 1273.21$  (12) Å<sup>3</sup>  
 $Z = 4$   
 $F(000) = 576$   
 $D_x = 1.431$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54184$  Å  
 Cell parameters from 4215 reflections  
 $\theta = 4.0$ – $68.0$ °  
 $\mu = 1.06$  mm<sup>-1</sup>  
 $T = 298$  K  
 Prism, colorless  
 $0.40 \times 0.12 \times 0.08$  mm

#### Data collection

Oxford Diffraction Gemini Atlas CCD  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 Detector resolution: 10.4685 pixels mm<sup>-1</sup>  
 $\omega$  scans  
 Absorption correction: analytical  
 (*CrysAlis RED*; Oxford Diffraction, 2009)  
 $T_{\min} = 0.885$ ,  $T_{\max} = 0.964$

4606 measured reflections  
 1786 independent reflections  
 1732 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.013$   
 $\theta_{\max} = 68.1$ °,  $\theta_{\min} = 4.0$ °  
 $h = -22 \rightarrow 24$   
 $k = -4 \rightarrow 6$   
 $l = -16 \rightarrow 16$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.071$   
 $S = 1.04$   
 1786 reflections  
 181 parameters  
 1 restraint  
 Primary atom site location: structure-invariant  
 direct methods  
 Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites

H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 0.4768P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.23$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.20$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97* (Sheldrick,  
 2008),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.0075 (4)  
 Absolute structure: Flack (1983), 498 Friedel  
 pairs  
 Absolute structure parameter: 0.0 (2)

#### Special details

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.69200 (8)	0.0051 (3)	0.95722 (12)	0.0519 (4)
N1	0.66380 (8)	0.2920 (3)	0.81679 (12)	0.0339 (3)
O4	0.72382 (7)	0.3751 (3)	0.65042 (11)	0.0438 (3)
F1	0.52029 (9)	0.7673 (3)	0.69645 (15)	0.0816 (5)
O5	0.90402 (9)	0.7069 (3)	0.88915 (13)	0.0478 (4)

O2	0.49424 (9)	0.5250 (3)	0.84757 (15)	0.0627 (5)
C5	0.72230 (11)	0.1894 (4)	0.80156 (17)	0.0403 (4)
H5	0.7609	0.0950	0.8745	0.048*
N2	0.59707 (9)	0.2801 (4)	0.90550 (15)	0.0434 (4)
C1	0.61955 (10)	0.4901 (4)	0.75175 (16)	0.0418 (4)
H1	0.6291	0.5673	0.7009	0.050*
O3	0.68552 (11)	0.0402 (3)	0.69943 (16)	0.0681 (5)
C4	0.65428 (10)	0.1784 (4)	0.89752 (15)	0.0362 (4)
C6	0.76688 (9)	0.3754 (4)	0.78102 (14)	0.0350 (4)
H6	0.7625	0.5332	0.8084	0.042*
C2	0.56349 (11)	0.5732 (4)	0.76007 (18)	0.0466 (5)
C3	0.54650 (10)	0.4653 (4)	0.83818 (17)	0.0435 (5)
C8	0.89973 (11)	0.4915 (4)	0.83002 (18)	0.0469 (5)
H8A	0.9528	0.4318	0.8649	0.056*
H8B	0.8733	0.5206	0.7435	0.056*
C7	0.85368 (10)	0.3099 (4)	0.84748 (17)	0.0437 (5)
H7A	0.8779	0.2948	0.9342	0.052*
H7B	0.8572	0.1551	0.8184	0.052*
C9	0.68429 (13)	0.1521 (4)	0.60232 (19)	0.0533 (6)
C10	0.72705 (17)	-0.0147 (5)	0.5721 (2)	0.0689 (7)
H10A	0.6979	-0.1621	0.5400	0.103*
H10B	0.7793	-0.0473	0.6448	0.103*
H10C	0.7313	0.0588	0.5121	0.103*
C11	0.60061 (17)	0.2034 (8)	0.4934 (3)	0.1094 (13)
H11A	0.5725	0.0550	0.4592	0.164*
H11B	0.6016	0.2893	0.4330	0.164*
H11C	0.5743	0.2986	0.5185	0.164*
H1N	0.5950 (12)	0.236 (5)	0.9622 (19)	0.048 (6)*
H1O	0.9348 (15)	0.789 (6)	0.896 (2)	0.072 (9)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0606 (7)	0.0506 (9)	0.0586 (7)	0.0216 (7)	0.0430 (7)	0.0239 (8)
N1	0.0336 (6)	0.0365 (9)	0.0345 (7)	0.0036 (7)	0.0218 (6)	0.0044 (6)
O4	0.0520 (7)	0.0397 (8)	0.0390 (6)	-0.0028 (6)	0.0265 (5)	0.0009 (6)
F1	0.0799 (8)	0.0748 (11)	0.1109 (11)	0.0463 (8)	0.0678 (8)	0.0532 (10)
O5	0.0529 (8)	0.0445 (10)	0.0622 (8)	-0.0061 (7)	0.0430 (7)	-0.0071 (7)
O2	0.0621 (8)	0.0584 (11)	0.0954 (11)	0.0167 (8)	0.0619 (8)	0.0137 (10)
C5	0.0465 (9)	0.0352 (11)	0.0509 (9)	0.0048 (9)	0.0353 (8)	0.0038 (9)
N2	0.0502 (8)	0.0455 (10)	0.0498 (8)	0.0083 (8)	0.0381 (7)	0.0092 (8)
C1	0.0410 (8)	0.0437 (12)	0.0447 (9)	0.0064 (9)	0.0275 (7)	0.0138 (9)
O3	0.1087 (12)	0.0487 (10)	0.0938 (11)	-0.0334 (9)	0.0857 (10)	-0.0292 (9)
C4	0.0368 (8)	0.0390 (11)	0.0359 (8)	0.0011 (8)	0.0232 (7)	0.0018 (8)
C6	0.0397 (8)	0.0320 (10)	0.0388 (8)	0.0022 (8)	0.0263 (7)	0.0006 (8)
C2	0.0444 (9)	0.0382 (12)	0.0563 (11)	0.0122 (9)	0.0294 (9)	0.0139 (9)
C3	0.0417 (8)	0.0401 (12)	0.0557 (10)	0.0025 (9)	0.0326 (8)	0.0013 (9)
C8	0.0430 (9)	0.0492 (13)	0.0608 (11)	0.0008 (10)	0.0374 (9)	-0.0079 (11)

C7	0.0400 (9)	0.0402 (12)	0.0531 (10)	0.0056 (9)	0.0288 (8)	0.0001 (9)
C9	0.0641 (12)	0.0483 (14)	0.0551 (11)	-0.0145 (11)	0.0394 (10)	-0.0142 (10)
C10	0.1132 (18)	0.0523 (16)	0.0761 (14)	-0.0072 (15)	0.0752 (15)	-0.0113 (13)
C11	0.0695 (16)	0.110 (3)	0.086 (2)	-0.0123 (19)	0.0121 (15)	-0.043 (2)

*Geometric parameters (Å, °)*

O1—C4	1.206 (2)	O3—C9	1.444 (3)
N1—C1	1.373 (3)	C6—C7	1.516 (2)
N1—C4	1.378 (2)	C6—H6	0.9800
N1—C5	1.471 (2)	C2—C3	1.434 (3)
O4—C9	1.419 (3)	C8—C7	1.509 (3)
O4—C6	1.435 (2)	C8—H8A	0.9700
F1—C2	1.345 (2)	C8—H8B	0.9700
O5—C8	1.417 (3)	C7—H7A	0.9700
O5—H10	0.75 (3)	C7—H7B	0.9700
O2—C3	1.218 (2)	C9—C11	1.504 (4)
C5—O3	1.393 (3)	C9—C10	1.505 (3)
C5—C6	1.526 (3)	C10—H10A	0.9600
C5—H5	0.9800	C10—H10B	0.9600
N2—C3	1.369 (3)	C10—H10C	0.9600
N2—C4	1.384 (2)	C11—H11A	0.9600
N2—H1N	0.83 (2)	C11—H11B	0.9600
C1—C2	1.325 (3)	C11—H11C	0.9600
C1—H1	0.9300		
C1—N1—C4	121.43 (14)	N2—C3—C2	112.25 (16)
C1—N1—C5	121.47 (14)	O5—C8—C7	108.20 (15)
C4—N1—C5	117.10 (15)	O5—C8—H8A	110.1
C9—O4—C6	109.72 (14)	C7—C8—H8A	110.1
C8—O5—H10	110 (2)	O5—C8—H8B	110.1
O3—C5—N1	110.92 (16)	C7—C8—H8B	110.1
O3—C5—C6	105.14 (14)	H8A—C8—H8B	108.4
N1—C5—C6	114.14 (16)	C8—C7—C6	113.23 (17)
O3—C5—H5	108.8	C8—C7—H7A	108.9
N1—C5—H5	108.8	C6—C7—H7A	108.9
C6—C5—H5	108.8	C8—C7—H7B	108.9
C3—N2—C4	128.17 (16)	C6—C7—H7B	108.9
C3—N2—H1N	113.4 (16)	H7A—C7—H7B	107.7
C4—N2—H1N	118.1 (16)	O4—C9—O3	105.54 (16)
C2—C1—N1	121.28 (17)	O4—C9—C11	107.9 (2)
C2—C1—H1	119.4	O3—C9—C11	111.2 (2)
N1—C1—H1	119.4	O4—C9—C10	112.88 (19)
C5—O3—C9	110.51 (16)	O3—C9—C10	106.8 (2)
O1—C4—N1	123.91 (16)	C11—C9—C10	112.5 (2)
O1—C4—N2	121.80 (16)	C9—C10—H10A	109.5
N1—C4—N2	114.29 (16)	C9—C10—H10B	109.5
O4—C6—C7	112.88 (14)	H10A—C10—H10B	109.5

O4—C6—C5	102.72 (13)	C9—C10—H10C	109.5
C7—C6—C5	111.86 (15)	H10A—C10—H10C	109.5
O4—C6—H6	109.7	H10B—C10—H10C	109.5
C7—C6—H6	109.7	C9—C11—H11A	109.5
C5—C6—H6	109.7	C9—C11—H11B	109.5
C1—C2—F1	121.20 (18)	H11A—C11—H11B	109.5
C1—C2—C3	122.28 (19)	C9—C11—H11C	109.5
F1—C2—C3	116.51 (17)	H11A—C11—H11C	109.5
O2—C3—N2	121.46 (18)	H11B—C11—H11C	109.5
O2—C3—C2	126.3 (2)		
C1—N1—C5—O3	-82.0 (2)	N1—C5—C6—C7	141.06 (16)
C4—N1—C5—O3	96.83 (19)	N1—C1—C2—F1	179.91 (19)
C1—N1—C5—C6	36.5 (2)	N1—C1—C2—C3	1.0 (3)
C4—N1—C5—C6	-144.63 (16)	C4—N2—C3—O2	173.9 (2)
C4—N1—C1—C2	-3.0 (3)	C4—N2—C3—C2	-6.5 (3)
C5—N1—C1—C2	175.8 (2)	C1—C2—C3—O2	-177.1 (2)
N1—C5—O3—C9	107.78 (18)	F1—C2—C3—O2	4.0 (3)
C6—C5—O3—C9	-16.1 (2)	C1—C2—C3—N2	3.3 (3)
C1—N1—C4—O1	-179.53 (18)	F1—C2—C3—N2	-175.64 (18)
C5—N1—C4—O1	1.6 (3)	O5—C8—C7—C6	67.2 (2)
C1—N1—C4—N2	0.4 (3)	O4—C6—C7—C8	65.3 (2)
C5—N1—C4—N2	-178.50 (15)	C5—C6—C7—C8	-179.47 (16)
C3—N2—C4—O1	-175.3 (2)	C6—O4—C9—O3	15.2 (2)
C3—N2—C4—N1	4.8 (3)	C6—O4—C9—C11	134.1 (2)
C9—O4—C6—C7	96.42 (19)	C6—O4—C9—C10	-101.1 (2)
C9—O4—C6—C5	-24.21 (19)	C5—O3—C9—O4	1.4 (2)
O3—C5—C6—O4	24.16 (19)	C5—O3—C9—C11	-115.3 (2)
N1—C5—C6—O4	-97.61 (16)	C5—O3—C9—C10	121.8 (2)
O3—C5—C6—C7	-97.17 (18)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H1N $\cdots$ O5 <sup>i</sup>	0.83 (2)	2.01 (2)	2.828 (2)	167 (3)
O5—H1O $\cdots$ O2 <sup>ii</sup>	0.75 (3)	2.16 (3)	2.876 (2)	160 (3)

Symmetry codes: (i)  $-x+3/2, y-1/2, -z+2$ ; (ii)  $x+1/2, y+1/2, z$ .