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1-(1,1-Dioxo-3′-deoxy-2,3,5,6-tetrahydro-1,4-oxathiine-β-D-ribofuranosyl)uracil: a bicyclic nucleoside analogue

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In the title compound, $C_{11}H_{14}N_2O_7S$, the furanose ring adopts a 3′-*endo*,4′-*exo* conformation $_4T^3$. The orientation of the pyrimidine ring with respect to the sugar group is *anti*. The crystal packing is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds.

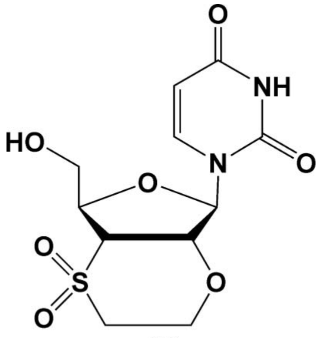
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.078
Data-to-parameter ratio = 15.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

2′,3′-Dideoxynucleosides, such as 3′-azido-2′,3′-dideoxythymidine (AZT, zidovudine) (Mitsuya *et al.*, 1985; Herdewijn *et al.*, 1987), 2′,3′-dideoxycytidine (ddC, zalcitabine) (Mitsuya & Broder, 1986; Chu *et al.*, 1988), 2′,3′-dideoxyinosine (ddI, didanosine) (De Clercq *et al.*, 1989; Schinazi *et al.*, 1990) and 2′,3′-didehydro-2′,3′-dideoxythymidine (d4T, stavudine) (Lin *et al.*, 1987; Balzarini *et al.*, 1989), constitute an important class of drugs in antivirus therapy. The title compound, (I), is a 2′,3′-α-fused tetrahydrooxathiine nucleoside synthesized *via* intramolecular Michael addition of the oxygen nucleophile to the vinyl sulfone. Here we present its crystal structure.



(I)

In (I) (Fig. 1), the five-membered ribose ring C7/C3/C4/C5/O4 adopts an approximate $_4T^3$ conformation, with a pseudorotational phase angle (P) of 36.8 (2)° and puckering amplitude (τ^m) of 47.5 (3)° (Sanger, 1984; Altona & Sundaralingam, 1972, 1973). The glycosidic torsion angle χ of −163.20 (19)° shows that the orientation of the pyrimidine ring with respect to the sugar group is *anti*. The torsion angle γ (C4—C5—C6—O5) is −169.76 (18)°. The C4—S1 and C3—O3 bond lengths are 1.781 (2) and 1.414 (3) Å, respectively. The fused six-membered tetrahydrooxathiine ring adopts a chair conformation.

The crystal packing (Fig. 2) is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds (Table 1).

Experimental

Compound (I) was synthesized from 1-{5′-*O*-trityl-2′,3′-dideoxy-3′-[2-(*tert*-butyldimethylsilyloxy)ethyl]sulfonyl-β-D-glyceropent-2′-enofuranosyl}uracil *via* intramolecular Michael addition (the manuscript is in preparation). It was crystallized slowly from a mixture of ethanol and methanol (2:1) at 298 K.

Crystal data

$C_{11}H_{14}N_2O_7S$
$M_r = 318.30$
Monoclinic, $P2_1$
$a = 5.2752\ (11)$ Å
$b = 7.1294\ (14)$ Å
$c = 17.425\ (4)$ Å
$\beta = 90.45\ (3)^\circ$
$V = 655.3\ (2)$ Å^3
$Z = 2$
$D_x = 1.613$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.28$ mm^{-1}
$T = 295\ (2)$ K
Block, colourless
$0.15 \times 0.11 \times 0.09$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.960$, $T_{\max} = 0.974$
6545 measured reflections
2969 independent reflections
2408 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.078$
$S = 1.04$
2969 reflections
195 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 0.0765P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 1343 Friedel pairs
Flack parameter: −0.04 (8)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H14···O7$^{\rm i}$	0.79 (3)	1.98 (3)	2.772 (3)	176 (2)
O5—H13···O6$^{\rm ii}$	0.82	2.02	2.777 (3)	153

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

The C- and O-bound H atoms were positioned geometrically (C—H = 0.93–0.98 Å, O—H = 0.82 Å) and refined as riding with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) of the parent atom. The atom H14 was located in a difference Fourier map and refined isotropically.

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

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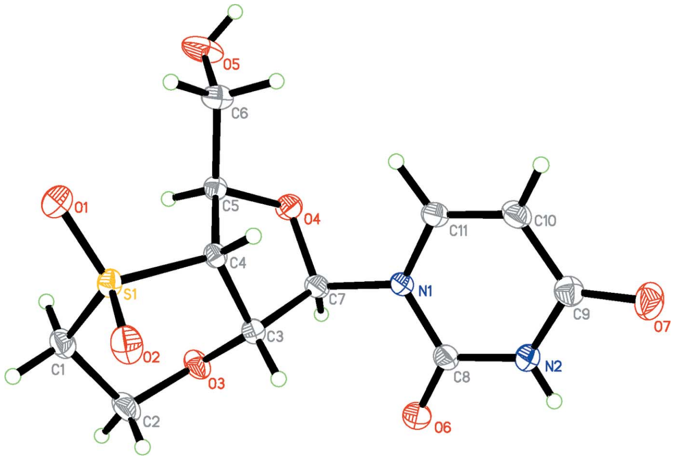

Figure 1
The molecular structure of (I), showing the atom-labelling scheme and displacement ellipsoids at the 30% probability level. H atoms are represented by circles of arbitrary size.

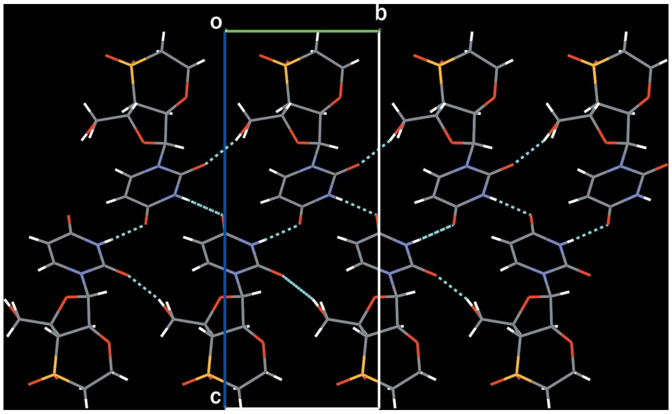

Figure 2
The molecular packing of (I), viewed along the *a* axis. The dashed lines indicate hydrogen bonds.

References

Altona, C. & Sundaralingam, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.
Altona, C. & Sundaralingam, M. (1973). *J. Am. Chem. Soc.* **95**, 2333–2344.
Balzarini, J., Van Aerschot, A., Herdewijn, P. & De Clercq, E. (1989). *Biochem. Pharmacol.* **38**, 869–874.
Chu, C. K., Schinazi, R. F., Arnold, B. H., Cannon, D. L., Doboszewski, B., Bhadti, V. B. & Gu, Z. (1988). *Biochem. Pharmacol.* **37**, 3543–3548.
De Clercq, E., Van Aerschot, A., Herdewijn, P., Baba, M., Pauwels, R. & Balzarini, J. (1989). *Nucleosides Nucleotides*, **8**, 659–671.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Herdewijn, P., De Clercq, E., Pauwels, R., Baba, M. & Vanderhaeghe, H. (1987). *J. Med. Chem.* **30**, 1270–1278.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Lin, T. S., Schinazi, R. F. & Prusoff, W. H. (1987). *Biochem. Pharmacol.* **36**, 2713–2718.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Mitsuya, H. & Broder, S. (1986). *Proc. Natl Acad. Sci. USA*, **83**, 1911–1915.
Mitsuya, H., Weinhold, K. J., Furman, P. A., St Clair, M. H., Nusinoff-Lehrman, S., Gallo, R. C., Bolognesi, D., Barry, D. W. & Broder, S. (1985). *Proc. Natl Acad. Sci. USA*, **82**, 7096–7100.
Rigaku Corporation (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sanger, W. (1984). *Principles of Nucleic Acid Structure*, edited by C. R. Cantor. New York: Springer Verlag.
Schinazi, R. F., Sommadossi, J. P., Saalmann, V., Cannon, D. L., Xie, M. Y., Hart, G. C., Smith, G. A. & Hahn, E. F. (1990). *Antimicrob. Agents Chemother.* **34**, 1061–1067.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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