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1-[4,5-Bis(hydroxymethyl)-1,2,3-triazol-1-ylmethyl]thymine

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The title compound, $C_{10}H_{13}N_5O_4$, can be classed as both a homo-*C*-nucleoside and an analogue of nucleosides possessing a 1,2,3-triazole ring instead of a furanose residue, some derivatives of which are potent antiviral agents. The mutual molecular arrangement of the five- and six-membered cyclic residues, as well as their disposition relative to the bridging $-CH_2-$ group, has been analyzed. In the crystal structure, intermolecular N—H···O and O—H···O hydrogen bonds form an infinite three-dimensional molecular network.

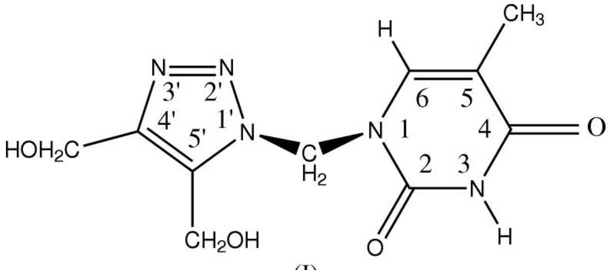
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.086
Data-to-parameter ratio = 13.9

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

Comment

In recent years, the synthesis of nucleoside analogues bearing different heterocyclic residues instead of the furanose ring has attracted considerable attention, since some of their derivatives are potent antiviral agents. A versatile method of constructing five-membered heterocycles possessing variable substituents is the 1,3-dipolar cycloaddition reaction (Xiang *et al.*, 1996; Adams *et al.*, 1998, and references therein), yielding compounds such as the title compound, (I). Furthermore, compound (I) can also be classed as a homo-*C*-nucleoside analogue (Doboszewski, 1997; Sallam & Townsend, 1998). The spatial arrangement of the heterocycles in (I) (Fig. 1) has now been analysed and the results are presented here.



(I)

The dihedral angle between the planes of the six-membered pyrimidine ring (N1/C2/N3/C4–C6) and the five-membered 1,2,3-triazole ring (N1′–N3′/C4′/C5′) is 77.0 (1)°. The dihedral angles between the N1/C1′/N1′ plane and the pyrimidine and triazole rings are 82.2 (1)° and 41.8 (1)°, respectively.

In the crystal structure, the molecules of (I) form a spiral along the 2_1 axis, with neighbouring molecules in the spiral connected by N3—H3···N3′^{i} and O4″—H4″···O4^{ii} hydrogen bonds, while O5″—H5″···O4″^{iii} hydrogen bonds link neighbouring spirals, forming an infinite three-dimensional molecular network (Table 1, Fig. 2; symmetry codes as in Table 1).

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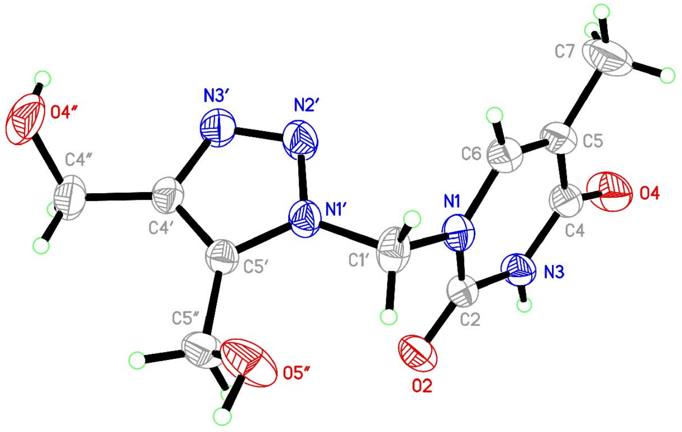

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

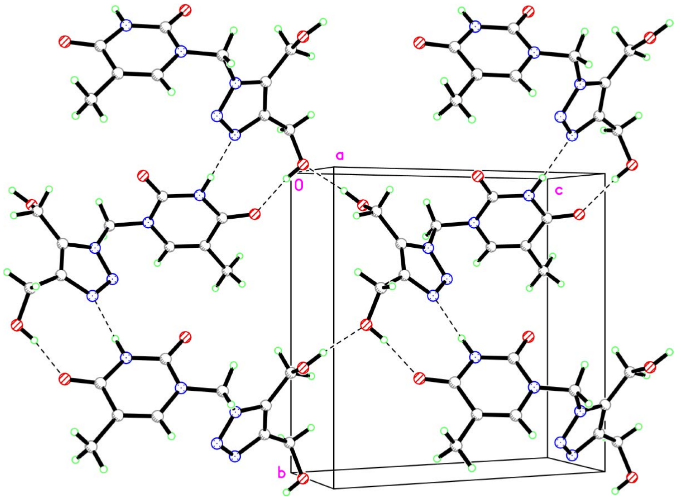

Figure 2
A view of the crystal packing in (I), showing the hydrogen bonding as dashed lines (see Table 1 for details).

Experimental

Compound (I) was obtained by the general procedure of cycloaddition of the azide dipole to alkyne dipolarophiles. 1-Methylthiomethylthymine (Zavgorodny *et al.*, 1997) was converted to 1-azidomethylthymine by the procedure developed earlier for *O*,*S*-acetals (Zavgorodny *et al.*, 2000), in 87% yield. This azido synthon gives the title compound in 88% yield by heating with 2-butyne-1,4-diol in methanol (m.p. 455–456 K). Crystals of (I) for X-ray analysis were prepared from a saturated solution of (I) in methanol, to which several drops of hexane had been added, kept at 277 K. Full details of the synthesis and characterization of (I) will be published elsewhere.

Crystal data

$C_{10}H_{13}N_5O_4$
$M_r = 267.25$
Monoclinic, $P2_1$
$a = 4.516$ (1) Å
$b = 11.618$ (2) Å
$c = 11.481$ (2) Å
$\beta = 91.43$ (2)°
$V = 602.2$ (2) Å^3
$Z = 2$
$D_x = 1.474$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 12.3$–14.2°
$\mu = 0.12$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.32 × 0.30 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
8497 measured reflections
2746 independent reflections
2350 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$
$\theta_{\max} = 35.0°$
$h = 0 \rightarrow 7$
$k = -18 \rightarrow 18$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.086$
$S = 1.04$
2746 reflections
197 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0712P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3⋯N3′$^{\rm i}$	0.90 (2)	2.00 (2)	2.8989 (12)	177 (2)
O4″—H4″⋯O4$^{\rm ii}$	0.81 (2)	1.94 (2)	2.7137 (15)	159 (2)
O5″—H5″⋯O4″$^{\rm iii}$	0.89 (1)	1.80 (1)	2.6863 (12)	175 (1)

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

All H atoms were located in difference syntheses and refined isotropically. In the refinement, atoms H4″, H5″, H3 and H6 were fully refined, while for the others, $U_{\rm iso}$(H) values were refined with the *xyz* coordinates constrained. The C—H bond lengths are in the range 0.92 (2)–0.98 (2) Å, N—H bond lengths are 0.904 (2) Å and O—H bond lengths are in the range 0.83 (2)–0.89 (1) Å.

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4/PC Software*; data reduction: *CAD-4/PC Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *CIFTAB97* (Sheldrick, 1997*a*) and *SHELXL97*.

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