Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2′-Deoxy-3′,5′-di-*O*-acetyluridine

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Received 15 November 2006
Accepted 22 November 2006

Key indicators

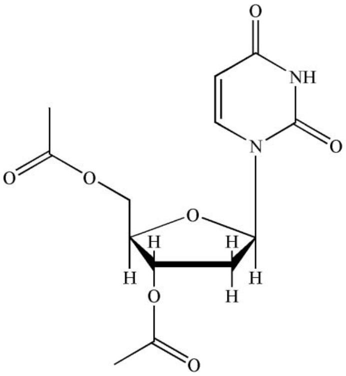
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.038
wR factor = 0.088
Data-to-parameter ratio = 9.0

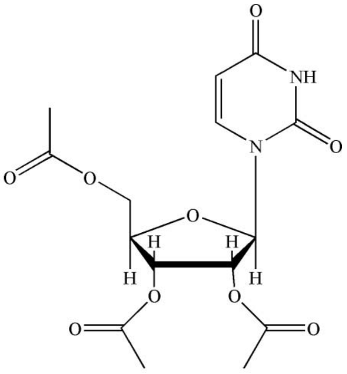
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the molecule of the title compound, $C_{13}H_{16}N_2O_7$, the furanose ring adopts an envelope conformation. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules, and these may be effective in the stabilization of the structure.

Comment

Uridine is a unique component of RNA structures. Hydrogen-bond behaviours of uridines have attracted considerable attention not only for the secondary and tertiary structures of RNAs but also for their therapeutic applications (Hulme *et al.*, 2005; Luchansky *et al.*, 2000; Yang & Rodgers, 2004). Although, as an important model compound, the photoreactive activity (Haga *et al.*, 1994) and hydrogen-bond behaviours in solution (Weisz *et al.*, 1997) of the title compound, (I), have been investigated, its crystal structure has not been reported yet. The structure of the analogous 2′,3′,5′-tri-*O*-acetyluridine, (II), has been reported (Low & Wilson, 1984).


(I)


(II)

In the molecule of (I) (Fig. 1), the bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987). In (I), the O3—C5 bond is a little shorter than the O3—C8 bond; the corresponding bonds in (II) are nearly the same. On the other hand, the acetyl carbonyl bonds, O7—C12 and O5—C10, in (I) are longer than the corresponding ones [1.184 (9), 1.185 (9) and 1.180 (11) Å] in (II). Ring A (O3/C5–C8) has an envelope conformation with atom C6 displaced by 0.598 (3) Å from the plane of the other four ring atoms.

In the crystal structure, N—H···O intermolecular hydrogen bonds (Table 2) link the independent molecules (Fig. 2), which may be effective in the stabilization of the structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

doi:10.1107/S1600536806050264

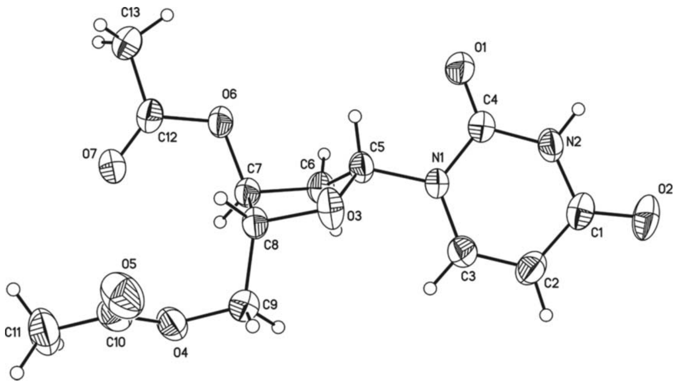


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

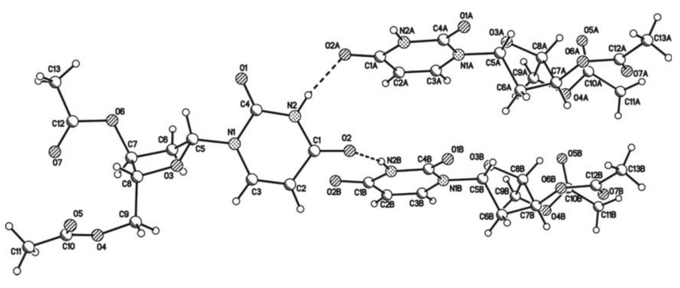


Figure 2
A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

Experimental

As a known compound (Haga *et al.*, 1994; Weisz *et al.*, 1997), the synthesis of (I) was achieved according to a similar literature procedures (Lohrmann & Khorana, 1964). Acetic anhydride (4 ml) was added to an anhydrous pyridine solution (10 ml) of 2′-deoxyuridine (2.282 g, 10 mmol), stirred at room temperature for 4 h and then poured into ice–water (100 ml). The mixture was evaporated to dryness. Chromatography [dichloromethane–methanol (99:1)] gave (I) as a white solid (yield 2.811 g, 90%). Slow evaporation of a 2-propanol solution of (I) at 278 K yielded single crystals suitable for X-ray analysis after a period of 7–8 weeks.

Crystal data

$C_{13}H_{16}N_2O_7$
$M_r = 312.28$
Monoclinic, $P2_1$
$a = 11.284\ (2)$ Å
$b = 5.3270\ (11)$ Å
$c = 13.184\ (3)$ Å
$\beta = 109.91\ (3)^\circ$
$V = 745.2\ (3)$ Å^3

$Z = 2$
$D_x = 1.392$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 293\ (2)$ K
Block, colourless
0.45 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.950$, $T_{max} = 0.977$

3709 measured reflections
1842 independent reflections
1274 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.088$
$S = 1.05$
1842 reflections
204 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.044P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.092 (7)

Table 1
Selected geometric parameters (Å, °).

O3—C5	1.427 (3)	O5—C10	1.213 (4)
O3—C8	1.459 (3)	O7—C12	1.213 (4)
O5—C10—O4	122.3 (3)	O7—C12—O6	121.5 (3)
O5—C10—C11	127.9 (3)	O7—C12—C13	126.8 (2)
O4—C10—C11	109.8 (3)	O6—C12—C13	111.7 (2)
C4—N1—C5—O3	138.1 (2)	C6—C7—C8—O3	−20.2 (3)
C5—C6—C7—C8	35.3 (3)	C7—C8—C9—O4	62.5 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H*N*2···O2^{i}	0.87 (4)	1.98 (3)	2.831 (3)	165 (3)

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

The H atom of the NH group was located in a difference synthesis and refined isotropically [N—H = 0.87 (4) Å and U_{iso}(H) = 0.063 (10) Å^2]. The remaining H atoms were positioned geometrically, with C—H = 0.93, 0.98, 0.97 and 0.96 Å for aromatic, methine, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *RAPID-AUTO* (Rigaku/MSC, 2000); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL97*.

The authors thank the National Natural Science Foundation of China (grant No. 20374058) for financial support.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Haga, N., Ishikawa, I., Takayanagi, H. & Ogura, H. (1994). *Bull. Chem. Soc. Jpn*, **67**, 728–737.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Hulme, A. T., Price, S. L. & Tocher, D. A. (2005). *J. Am. Chem. Soc.* **127**, 1116–1117.
Lohrmann, R. & Khorana, H. G. (1964). *J. Am. Chem. Soc.* **86**, 4188–4194.
Low, J. N. & Wilson, C. C. (1984). *Acta Cryst.* C**40**, 1030–1032.

Luchansky, S. J., Nolan, S. J. & Baranger, A. M. (2000). *J. Am. Chem. Soc.* **122**, 7130–7131.

Rigaku/MSC (2000). *CrystalStructure* and *RAPID-AUTO*, Rigaku/MSC, The Woodlands, Texas, USA.

Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*b*). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

Weisz, K., Johnchen, J. & Limbach, H.-H. (1997). *J. Am. Chem. Soc.* **119**, 6436–6437.

Yang, Z. & Rodgers, M. T. (2004). *J. Am. Chem. Soc.* **126**, 16217–16226.