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4-*N*-Acetylamino-5-[*N*-acetyl-*N*-(tetra-*O*-acetyl-β-D-glucopyranosyl)amino]-1,3-dimethyluracil

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.087
Data-to-parameter ratio = 8.8

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

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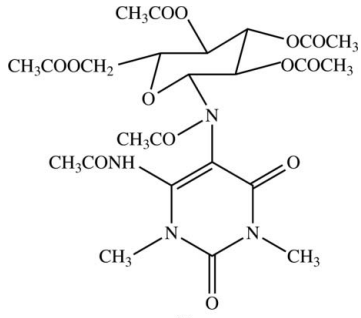
The title compound, $C_{24}H_{32}N_4O_{13}$, crystallizes with two molecules in the asymmetric unit. These have very similar conformations, and each contains an intramolecular N—H···O hydrogen bond. There are no significant intermolecular interactions.

Comment

The title compound, (I), has been prepared for use as an intermediate in the synthesis of nucleoside analogues with potential antitumour or antiviral applications.



(I)

Compound (I) crystallizes with two independent molecules (Fig. 1) in the space group $P2_1$, and the two molecules have very similar conformations. In the glucopyranose rings, the ring-puckering parameters (Cremer & Pople, 1975) in molecules 1 and 2 (containing N11 and N31 respectively, Fig. 1) are, for the atom sequences (O25, C21–C25) and (O45, C41–C45), $\theta = 3.5\ (2)$ and $4.5\ (2)^\circ$, respectively, and $\varphi = 104\ (4)$ and $342\ (3)^\circ$, respectively: thus each ring has an almost perfect chair conformation, for which the ideal value of θ is zero.

The conformation adopted by all the substituents exocyclic to these chair rings are again similar for the two molecules, as shown by the leading torsion angles (Table 1), but the differences between them are sufficient to preclude any additional symmetry. The bond distances within the uracil rings are also very similar for the two molecules.

In each molecule there is a single intramolecular N—H···O hydrogen bond (Table 2), forming an *S*(7) motif (Bernstein *et al.*, 1995), and these interactions may have an influence on the overall molecular conformations.

There are no significant direction-specific intermolecular interactions.

Experimental

Crystals of compound (I) were prepared according to a published procedure (Melgarejo Sampedro *et al.*, 1982).

Crystal data

$C_{24}H_{32}N_4O_{13}$
M_r = 584.54
Monoclinic, $P2_1$
a = 8.8490 (1) Å
b = 17.8429 (2) Å
c = 18.3058 (2) Å
β = 103.7830 (8)°
V = 2807.11 (6) Å^3
Z = 4
D_x = 1.383 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6642 reflections
θ = 2.9–27.5°
μ = 0.11 mm^{-1}
T = 120 (2) K
Block, colourless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.954, T_{max} = 0.980
46614 measured reflections
6642 independent reflections
5791 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.5°
$h = -11 \rightarrow 10$
$k = -22 \rightarrow 23$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.087
S = 1.08
6642 reflections
755 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.4658P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N11—C12	1.382 (3)	N31—C32	1.368 (4)
C12—N13	1.386 (3)	C32—N33	1.396 (3)
N13—C14	1.376 (3)	N33—C34	1.374 (3)
C14—C15	1.361 (3)	C34—C35	1.362 (3)
C15—C16	1.449 (3)	C35—C36	1.448 (3)
C16—N11	1.400 (3)	C36—N31	1.389 (3)
C15—C14—N14—C141	−116.1 (2)	C35—C34—N34—C341	−120.4 (2)
C15—N15—C21—C22	−35.7 (3)	C35—N35—C41—C42	−27.7 (3)
C22—C21—N15—C151	151.98 (19)	C42—C41—N35—C351	145.74 (19)
C21—C22—O22—C221	129.62 (18)	C41—C42—O42—C421	132.84 (19)
C22—C23—O23—C231	−150.68 (18)	C42—C43—O43—C431	−146.41 (19)
C23—C24—O24—C241	104.9 (2)	C43—C44—O44—C441	100.1 (2)
C24—C25—C26—O26	61.3 (3)	C44—C45—C46—O46	49.3 (3)
C25—C26—O26—C261	−136.4 (2)	C45—C46—O46—C461	−124.2 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N14—H14···O25	0.88	2.00	2.787 (3)	148
N34—H34···O45	0.88	2.06	2.867 (2)	152

All H atoms were located in difference maps, and then treated as riding atoms with distances C—H = 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (CH), and N—H = 0.88 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(methyl C). In the absence of significant anomalous scattering, the Flack (1983) parameter was indeterminate (Flack & Bernardinelli, 2000); hence, the Friedel equivalents were merged prior to the final refinements. The absolute configuration was assigned by reference to the known configuration of the chiral starting material. There is evidence for considerable libration of the acetyl groups bonded to O26 and O46.

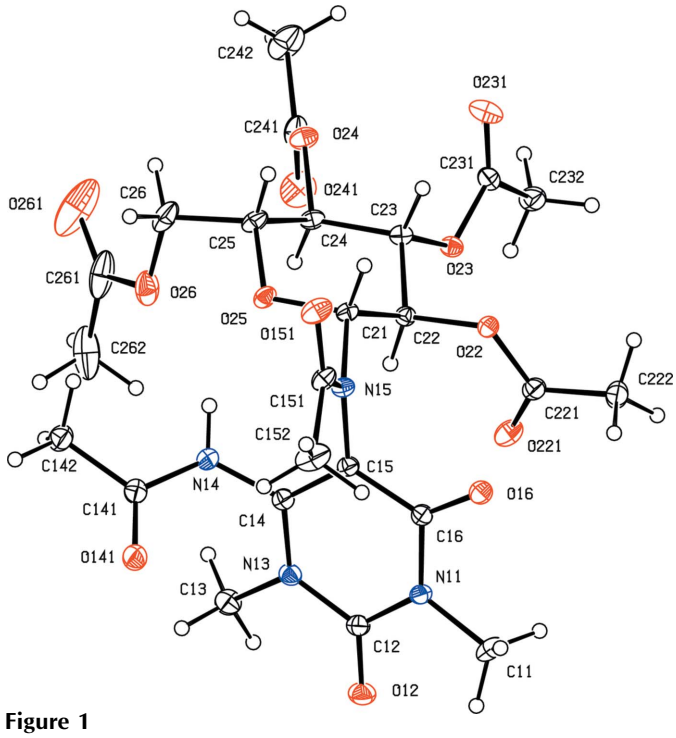


Figure 1
One of the two independent molecules of compound (I), *viz*. molecule 1, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

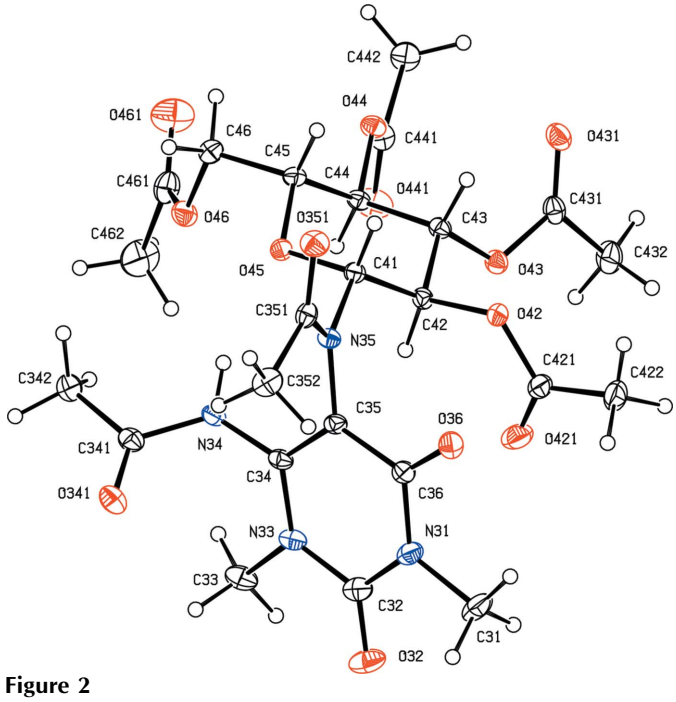


Figure 2
One of the two independent molecules of compound (I), *viz*. molecule 2, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); soft-

ware used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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