N^1,N^1-Diethyl-N^2-(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)acet-amidine

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The solid-state conformation of the title compound, $C_{20}H_{32}N_2O_9$, has been determined at 150 K. The pyranose ring has a distorted chair conformation. Among the possible conformations of the C—N glycosidic bond, that of the E rotamer is observed and a short intramolecular $C_{methyl}\cdots O$ contact may partly stabilize this conformation. Crystal cohesion is stabilized by an extensive network of weak C—H$\cdots$O hydrogen bonds and close contacts.

Comment

The chemical and biological properties of organic compounds depend on their structure and conformation and, as many natural products and their derivatives have an N-acyl group joined to a sugar moiety, correct determination of their structure is sometimes challenging. Crystals of the title compound, (I), were obtained by Avalos *et al.* (1995) from the reaction of 2,3,4,6,-tetra-O-acetyl-N-thioacetyl-β-D-gluco-pyranosylamine, mercury oxide and diethylamine in dichloromethane. The same authors studied several glyco-amidines by NMR spectroscopy, including the title compound, and confirmed the β-configuration of the prepared glyco-amidine. The absolute configuration was assigned from the absolute stereochemistry of the starting material used in the synthesis. We therefore undertook the crystal structure determination of (I). The structural analysis shows that only one rotamer along the C—N bond (E) is present in the crystal.
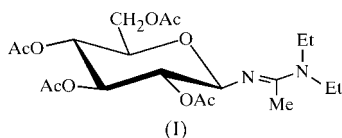
The structure of compound (I) was determined first at room temperature and then at 150 K, to reduce the dynamic disorder affecting the terminal groups. The data at 150 K are presented here. The isotropic displacement parameters at 150 K are almost three times smaller than those determined at room temperature.

Fig. 1 shows an *ORTEP*II view (Johnson, 1976) of the molecule of (I) viewed along the c axis, together with the atomic numbering scheme. Bond lengths and angles (Table 1) agree with those of analogous compounds (Vega *et al.*, 1986; Diánez *et al.*, 1997), although the pyranose endocyclic bond lengths [O—C1 1.445 (3) Å and O—C5 1.436 (3) Å] do not show the anomeric effect characteristic of this system. The acetoxy and methylacetoxy groups are essentially planar.
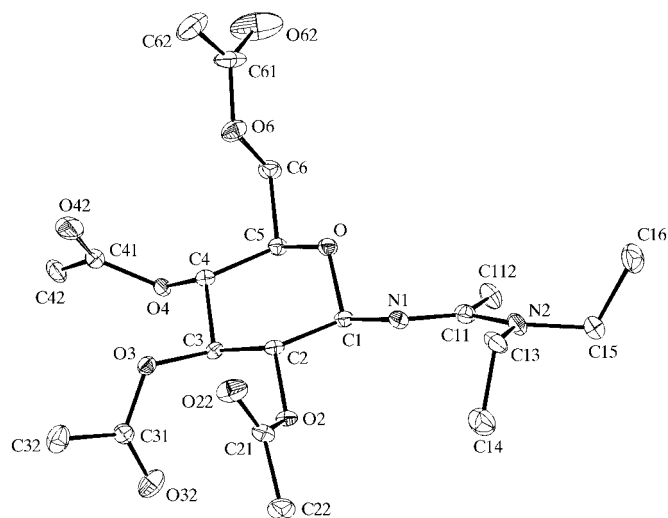
Figure 1
A view of the molecule of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

The geometry observed for the pyranose ring is a distorted chair, with ring substituents O2, O4, C6, O3 and N1 all in equatorial positions. The ring puckering parameters (Cremer & Pople, 1975) are $Q = 0.591$ (3) Å, $\varphi = 42$ (2)° and $\theta = 10$ (1)°; the asymmetry parameters (Nardelli, 1983a) are $\Delta C_s(C1) = 0.028$ and $\Delta C_2(C1-O) = 0.011$. The glycosidic O—C1—N1—C11 torsion angle is -80.0 (3)°, which is within the range of the E rotamer of the glycosidic linkage; the O—C5—C6—O6 torsion angle is -67.4 (3)°. This conformation may be partly stabilized by a close intramolecular C—H$\cdots$O interaction between C12 and the O atom in the pyranose ring, with C12$\cdots$O = 3.174 (1) Å, H$\cdots$O = 2.525 Å and C12—H$\cdots$O = 123.6 (1)°.

In the absence of standard hydrogen-bonding donor or acceptor groups, the crystal packing is stabilized by a series of nine weak C—H$\cdots$O interactions, many of which have the near-linear molecular geometries expected for close C—H$\cdots$O contacts (Table 2).

Experimental

Compound (I) was synthesized in the Organic Chemistry Department of Extremadura University using the method of Avalos *et al.* (1995), from the reaction of 2,3,4,6-tetra-O-acetyl-N-thioacetyl-β-D-gluco-pyranosylamine, mercury oxide and diethylamine in dichloromethane. Crystals of (I) were grown from ethyl ether.

organic compounds

Crystal data

$C_{20}H_{32}N_2O_9$
$M_r = 444.48$
Orthorhombic, $P2_12_12_1$
$a = 10.440 (2)$ Å
$b = 27.647 (5)$ Å
$c = 8.012 (2)$ Å
$V = 2312.5 (8)$ Å^3
$Z = 4$
$D_x = 1.277$ Mg m^{-3}
$D_m = 1.28$ Mg m^{-3}

D_m measured by flotation in nitrobenzene and acetone
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 2$–$30°$
$\mu = 0.10$ mm^{-1}
$T = 150 (2)$ K
Prism, colourless
$0.60 \times 0.49 \times 0.40$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
5663 measured reflections
5663 independent reflections
4270 reflections with $I > 2\sigma(I)$
$\theta_{max} = 35°$

$h = 0 \rightarrow 16$
$k = 0 \rightarrow 44$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$S = 1.12$
5663 reflections
280 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0904P)^2 + 1.8423P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O—C5	1.436 (3)	N2—C11	1.360 (4)
O—C1	1.445 (3)	N2—C13	1.463 (4)
N1—C11	1.292 (4)	N2—C15	1.465 (4)
N1—C1	1.425 (4)	C11—C12	1.511 (5)
C5—O—C1	111.4 (2)	N1—C1—C2	108.7 (2)
C11—N1—C1	119.2 (3)	O—C1—C2	105.7 (2)
C11—N2—C13	119.5 (3)	N1—C11—N2	118.1 (3)
C11—N2—C15	124.3 (3)	N1—C11—C12	125.0 (3)
C13—N2—C15	115.7 (3)	N2—C11—C12	116.9 (3)
N1—C1—O	111.3 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3$\cdots$O32[i]	1.00	2.65	3.552 (4)	150
C5—H5$\cdots$O22[ii]	1.00	2.64	3.393 (4)	132
C12—H12$C\cdots$O22[ii]	0.98	2.61	3.434 (5)	142
C6—H6$B\cdots$O42[iii]	0.99	2.56	3.547 (5)	173
C22—H22$C\cdots$O32[iv]	0.98	2.73	3.338 (5)	121
C32—H32$B\cdots$O32[iv]	0.98	2.66	3.458 (5)	138
C22—H22$A\cdots$O62[v]	0.98	2.86	3.395 (6)	115
C32—H32$A\cdots$O42[vi]	0.98	2.44	3.359 (5)	156
C42—H42$A\cdots$O62[vi]	0.98	2.48	3.403 (6)	156

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

program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *PARST* (Nardelli, 1983b).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1111). Services for accessing these data are described at the back of the journal.

No Friedel pairs were collected. The absolute configuration was known from the absolute stereochemistry of the starting materials. All H atoms were placed at idealized positions using a riding model and were not refined.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SET*4 (de Boer & Duisenberg, 1984) and *CELDIM* in *CAD*-4 *Software*; data reduction: *XRAY*76 (Stewart *et al.*, 1976);

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