Monoclinic
$P2_1/c$
a = 13.113 (4) Å
b = 15.260 (6) Å
c = 6.762 (5) Å
β = 92.36 (4)°
V = 1352.0 (12) Å^3
Z = 4
D_x = 1.407 Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
θ = 8–12°
μ = 0.243 mm^{-1}
T = 293 (2) K
Prismatic
0.38 × 0.35 × 0.18 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2274 measured reflections
2088 independent reflections
1749 reflections with $I > 2\sigma(I)$

R_{int} = 0.018
θ_{max} = 25.00°
$h = -15 \rightarrow 15$
$k = -18 \rightarrow 0$
$l = 0 \rightarrow 8$
2 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.131
S = 1.058
2088 reflections
237 parameters
H-atom coordinate parameters refined only

$w = 1/[\sigma^2(F_o^2) + (0.0823P)^2 + 0.3635P]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.329 e Å^{-3}
$\Delta\rho_{min}$ = −0.280 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 2. *Selected geometric parameters (Å, °) for (II)*

S—C10	1.783 (3)	C3—C2	1.513 (4)
S—C3	1.811 (3)	C3—C4	1.516 (4)
O1—C9′	1.367 (3)	C9—C4	1.478 (3)
O1—C2	1.429 (3)		
C10—S—C3	99.3 (1)	O1—C9′—C8	116.8 (2)
C9′—O1—C2	114.3 (2)	O1—C9′—C9	123.1 (2)
C2—C3—C4	108.0 (2)	C9′—C9—C4	120.4 (2)
C2—C3—S	111.8 (2)	O2—C4—C9	124.2 (2)
C4—C3—S	112.7 (2)	O2—C4—C3	123.5 (2)
O1—C2—C3	110.6 (2)	C9—C4—C3	112.2 (2)
C9′—O1—C2—C3	50.7 (3)	C2—C3—C4—O2	−141.2 (3)
C4—C3—C2—O1	−64.8 (3)	S—C3—C4—O2	−17.2 (4)
S—C3—C2—O1	170.8 (2)	C2—C3—C4—C9	42.2 (3)
C2—O1—C9′—C9	−14.4 (4)	S—C3—C4—C9	166.1 (2)
O1—C9′—C9—C4	−7.3 (4)	C3—S—C10—C15	−54.1 (3)
C9′—C9—C4—C3	−8.7 (3)	C3—S—C10—C11	128.1 (3)

For both compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976).

The authors wish to thank the Regional Sophisticated Instrumentation Centre, Indian Institute of Technology, Madras, India, for data collection.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1051). Services for accessing these data are described at the back of the journal.

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β-1-*N*-Acetamido-D-glucopyranose

D. SRIRAM,[a] H. SREENIVASAN,[a] S. SRINIVASAN,[a] K. PRIYA,[b] M. VISHNU THIRTHA[b] AND D. LOGANATHAN[b]

[a]*Department of Physics, Indian Institute of Technology, Madras 600 036, India, and* [b]*Department of Chemistry, Indian Institute of Technology, Madras 600 036, India.* *E-mail: phy13@iitm.ernet.in*

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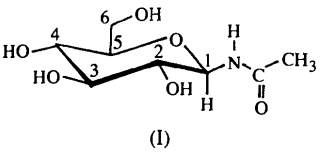
Abstract

In the title molecule, $C_8H_{15}NO_6$, the pyranose ring adopts the usual $^4C_1(D)$ conformation and the *N*-acetyl group exists in the *Z–anti* conformation. The orientation of the primary alcohol group is *gauche*.

Comment

The glycan (oligosaccharide) components of glycoproteins are known to play vital roles in protein folding, protein targeting and cellular recognition (Cumming, 1992; Imperiali & Rickert, 1994). Elucidation of the structural basis of their biological roles represents a fundamental and challenging problem in glycobiological research (Dwek, 1996). A systematic study initiated recently by the present authors is focused on the structural aspects of *N*-glycoproteins. The title compound, (I), was chosen for the current X-ray crystallographic investigation as it represents the simplest model of the linkage region (2-deoxy-2-acetamido-β-D-glucopyranosylasparagine, GlcNAcβAsn).



(I)

An *ORTEP*II (Johnson, 1976) diagram of the molecule with the atom numbering is shown in Fig. 1. The title compound exhibits some differences from and shares a few similarities with other β-pyranoses. Unlike in other β-pyranoses (Samudzi, Ruble & Jeffrey, 1985), (i) there is no shortening of the C—N bond and (ii) the two bonds O5—C1 and O5—C5 are equal within their s.u.'s. As a consequence, there is very little deviation of the valence angle O5—C1—N from the ideal value for tetrahedral geometry.

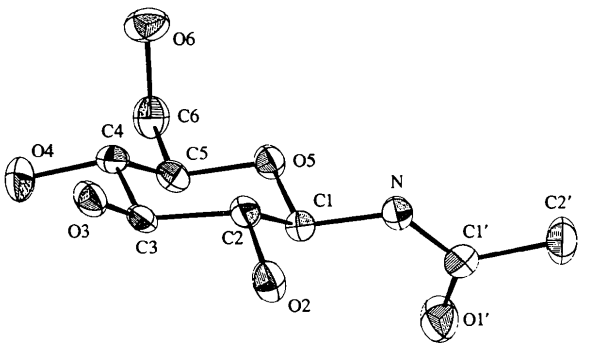


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the molecular structure and atom numbering of (I). The displacement ellipsoids are drawn at the 50% probability level.

As in other β-pyranoses, the orientation of the primary alcohol group is *gauche*, with the torsion angle O5—C5—C6—O6 = −71.8 (3)°. The observed ^{1}H NMR vicinal coupling constants, namely $J_{6,5}$ = 2.4 and $J_{6',5}$ = 4.9 Hz, are consistent with a similar torsion angle indicating that the orientation of the primary hydroxyl group in the solid state resembles that present in aqueous solution. The pyranose ring adopts a $^4C_1(D)$ conformation with atoms C4 and C1 deviating on opposite sides from the mean plane formed by the other atoms (C2, C3, C5 and O5) by 0.644 and 0.676 Å, respectively.

Motion of the GlcNAcβAsn linkage can influence the presentation of the sugar relative to the protein surface in *N*-glycoproteins (Dwek, 1996). Viewed in this context the conformation of the title compound around the amide bond assumes significance. The *ORTEP* diagram clearly shows that the *N*-acetyl group exists in the *Z*-*anti* conformation.

Experimental

The title compound was prepared by selective *N*-acetylation of β-D-glucopyranosylamine (Kitaoka, 1960). β-D-Glucopyranosylamine (Isbell & Frush, 1951) was reacted with acetic anhydride in dry *N*,*N*-dimethylformamide at 273 K for 4 h (Kitaoka, 1960). The product obtained in 77% isolated yield was recrystallized twice from aqueous methanol to give colourless prismatic crystals of the title compound. ^{1}H NMR (400 MHz, D_2O): δ 4.96 (*d*, 1H, 9.3 Hz, H-1), 3.89 (*dd*, 1H, $J_{6',6}$ = 12.2, $J_{6,5}$ = 2.4 Hz, H-6), 3.74 (*dd*, 1H, $J_{6,6'}$ = 12.2, $J_{6',5}$ = 4.9 Hz, H-6′), 3.56 (*t*, 1H), 3.54 (*m*, 1H, H-5), 3.46–3.36 (*m*, 2H) and 2.09 p.p.m. (*s*, 3H, $COCH_3$). ^{13}C NMR: δ 178.3 (C=O), 82.0 (C-1), 80.3, 79.2, 74.5, 72.0, 63.3 (C-6) and 24.9 p.p.m. (CH_3).

Crystal data

$C_8H_{15}NO_6$
M_r = 221.21
Orthorhombic
$P2_12_12_1$
a = 7.8642 (13) Å
b = 9.423 (4) Å
c = 14.008 (4) Å
V = 1038.0 (6) Å^3
Z = 4
D_x = 1.415 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8–12°
μ = 0.122 mm^{-1}
T = 293 (2) K
Prismatic
0.7 × 0.4 × 0.3 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
968 measured reflections
968 independent reflections
824 reflections with $I > 2\sigma(I)$

θ_{max} = 25°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 16$
2 standard reflections
frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0328
$wR(F^2)$ = 0.0760
S = 1.111
968 reflections
185 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2 + 0.1578P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.023

$\Delta\rho_{max}$ = 0.182 e Å^{-3}
$\Delta\rho_{min}$ = −0.152 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.006 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N—C1′	1.340 (4)	C5—C6	1.511 (5)
N—C1	1.431 (4)	C6—O6	1.427 (4)
C1—O5	1.441 (4)	C1′—O1′	1.239 (4)
C5—O5	1.441 (4)	C1′—C2′	1.496 (5)
C1′—N—C1	122.6 (3)	C4—C5—C6	113.7 (3)
N—C1—O5	108.0 (3)	C5—O5—C1	111.3 (2)
N—C1—C2	111.4 (3)	O1′—C1′—C2′	122.9 (3)
O3—C3—C4	111.8 (3)	N—C1′—C2′	115.7 (3)
C1′—N—C1—O5	−93.7 (4)	O5—C5—C6—O6	−71.8 (3)
O5—C1—C2—C3	55.7 (3)	C4—C5—C6—O6	50.1 (4)
C1—C2—C3—C4	−49.7 (3)	C4—C5—O5—C1	63.7 (3)
C2—C3—C4—C5	50.4 (4)	C2—C1—O5—C5	−63.3 (3)
C3—C4—C5—O5	−56.4 (3)		

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

The authors wish to thank the Regional Sophisticated Instrumentation Centre, Indian Institute of Technology, Madras, India, for data collection. Financial support from the Department of Science and Technology, New Delhi, India, is gratefully acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1056). Services for accessing these data are described at the back of the journal.

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5,8-Dimethoxy-1-naphthoic Acid and Methyl 5,8-Dimethoxy-1-naphthoate

ANTHONY C. BLACKBURN AND ROGER E. GERKIN

Department of Chemistry, The Ohio State University, Columbus, Ohio 43210, USA. E-mail: gerkin@chemistry.ohio-state.edu

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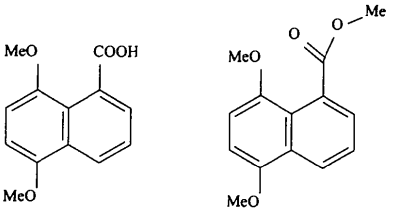
Abstract

In 5,8-dimethoxy-1-naphthoic acid, $C_{13}H_{12}O_4$, hydrogen bonding is of the cyclic dimer type. The acid H atom is modelled as being distributed equally over two sites. In addition to the conventional hydrogen bonds, there are three significantly attractive C—H···O interactions. The dihedral angle between the naphthalene core plane and the carboxyl plane is 80.0 (1)°. In methyl 5,8-dimethoxy-1-naphthoate, $C_{14}H_{14}O_4$, there are no conventional hydrogen bonds but there are three significantly attractive C—H···O interactions. With the exception of the C—O distances in the carboxyl groups, the molecular geometries of the acid and the ester are quite similar.

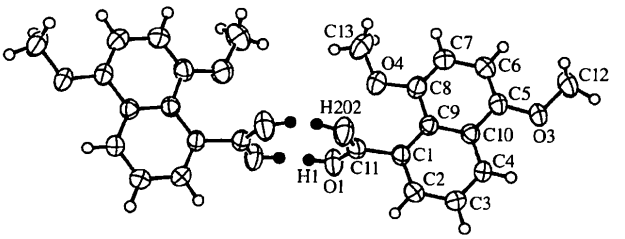
Comment

This study of 5,8-dimethoxy-1-naphthoic acid, (I), and its methyl ester, (II), was performed as part of a series of hydrogen-bonding studies of aromatic carboxylic acids. Of particular interest was the comparison of the hydrogen bonding in this acid with that in 1-naphthoic acid as previously determined by Fitzgerald & Gerkin (1993).

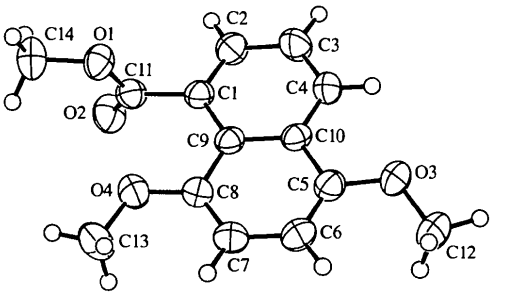


(I) (II)

In the title acid, (I), the hydrogen bonding is of the cyclic dimer type as shown in Fig. 1(*a*). Hydrogen-bonding details are given in Table 2. [Neutron-adjusted O—H distances result in H···*A* distances of 1.67 and 1.69 Å, and *D*—H···*A* angles of 176 and 145° for the two hydrogen bonds, respectively.] The donor–acceptor distances are below average for organic O···O hydrogen bonds (2.77 Å; Ceccarelli, Jeffrey & Taylor, 1981). Also as shown in Fig. 1(*a*), the carboxylic atoms H1 and H2 have been modelled as half-occupancy atoms.



(*a*)



(*b*)

Fig. 1. *ORTEPII* (Johnson, 1976) drawings of (*a*) the cyclic hydrogen-bonded dimer of 5,8-dimethoxy-1-naphthoic acid and (*b*) the methyl 5,8-dimethoxy-1-naphthoate molecule with our numbering scheme. Displacement ellipsoids are drawn for 50% probability for all atoms except H for which they have been set artificially small. In (*a*), the four half-occupancy acid H atoms are shown as solid circles. The symmetry code relating the second molecule to the base molecule is $x, \frac{1}{2}-y, z$.