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4-Nitrophenyl tetra-*O*-acetyl-β-D-glucopyranoside

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.006 Å
R factor = 0.040
wR factor = 0.108
Data-to-parameter ratio = 7.1

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

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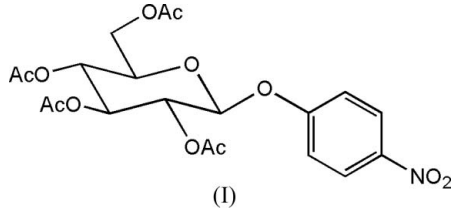
In the title compound, $C_{20}H_{23}NO_{12}$, the pyranoside ring adopts a $^4C_1$ chair conformation, with all substituents oriented in equatorial positions. The endocyclic C—O—C angle is in agreement with the geometry of the equatorial glycosidic bond, typical for the β-D-$^4C_1$ pyranoside conformation.

Comment

4-Nitrophenyl-β-D-glycosides constitute an important class of aromatic *O*-glycosides, especially because they have been frequently used as substrates for enzyme detection of most of the β-glycosidases (Esen, 1993). These synthetic glycosides, when incubated with the appropriate glycosidase, produced 4-nitrophenol release which is observed as a yellow color and results in an increase in absorbance at 405 cm^{-1} (Manafi *et al.*, 1991).

In the title compound, (I) (Fig. 1), the glucopyranoside ring adopts a $^4C_1$ chair conformation as shown by its puckering parameters (Cremer & Pople, 1975) Q = 0.583 (4) Å, θ = 5.4 (4)° and ψ = 352 (4)°. The anomeric C1′—O1 bond is slightly elongated [1.398 (4) Å] with respect to the reference value (1.385 Å; Jeffrey, 1990). From the six possible C—H···O hydrogen bonds (Table 1) suggested by *PLATON* (Spek, 2003), three contacts are intermolecular, but with C···O distances larger than 3.25 Å, and the other three are intramolecular (C2—H2···O5′, C2′—H2′···O13 and C3′—H3′···O11). Any of them is considered as a formal hydrogen bond, although these attractive close contacts certainly contribute to the stabilization of the molecular conformation and the crystal packing.



(I)

Experimental

The title compound was prepared according to a standard protocol consisting of the reaction of 2,3,4,6-tetra-*O*-acetyl-1-bromo-bβ-D-glycopyranose (1.18 g, 2.8 mmol) with 4-nitrophenol sodium salt (0.4 g, 2.8 mmol) in acetone–water (10 ml, 1:1 *v*/*v*) as solvent system to yield 0.67 g (50%) of the desired product as a colorless solid, which was recrystallized from 95% ethanol (scheme). ^{1}H NMR ($CDCl_3$): δ 2.01–2.10 (4 *s*, 12H, CH_3CO), 3.95 (*m*, 1H, H-5′), 4.20 (*dd*, 1H, H6*a*), 4.26 (*dd*, 1H, H-6*b*), 5.07 (*dd*, 1H, H-2′), 5.22 (*d*, 1H, H-1′), 5.25 (*t*, 1H,

H4′), 5.34 (*t*, 1H, H-3′), 7.07 (*d*, 2H, *J* = 9.3 Hz), 8.20 (*d*, 2H, *J* = 9.3 Hz). ^{13}C NMR ($CDCl_3$): δ 20.78 (CH_3CO), 61.99 (C-5′), 68.15 (C-6′), 71.08 (C-2′), 72.57 (C-4′), 77.68 (C-3′), 98.19 (C-1′), 116.80 (C-2), 126.02 (C-3), 143.42 (C-4), 161.36 (C-1), 170.71 (C═O).

Crystal data

$C_{20}H_{23}NO_{12}$
M_r = 469.39
Monoclinic, $P2_1$
a = 5.7832 (10) Å
b = 16.204 (3) Å
c = 12.7389 (16) Å
β = 100.044 (12)°
V = 1175.5 (3) Å^3
Z = 2
D_x = 1.326 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
Prism, colorless
0.51 × 0.21 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: part of the refinement model (ΔF) (Walker & Stuart, 1983)
T_{min} = 0.948, T_{max} = 0.979
2242 measured reflections
2147 independent reflections
1710 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 25.0°
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
S = 1.06
2147 reflections
302 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.062P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O5′	0.93	2.55	3.113 (5)	120
C2′—H2′···O13	0.98	2.25	2.674 (6)	105
C3′—H3′···O9^{i}	0.98	2.58	3.387 (5)	140
C3′—H3′···O11	0.98	2.25	2.699 (6)	106
C5′—H5′···O9^{i}	0.98	2.38	3.279 (5)	152
C6—H6···O7^{ii}	0.93	2.54	3.320 (6)	141
O11—H3′···O9^{i}				113

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

The assumed chirality derives from the known absolute configuration of the synthetic precursor. In the absence of significant anomalous scattering effects, Friedel pairs were merged. H atoms were introduced in calculated positions and refined as riding on their parent atoms with constraints as follows: C—H = 0.96, 0.97, 0.98 and 0.93 Å for CH_3, CH_2, CH and aromatic CH. U_{iso}(H) = 1.5U_{eq}(C) for the methyl H atoms and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms. The methyl groups were allowed to rotate around the C—C bonds.

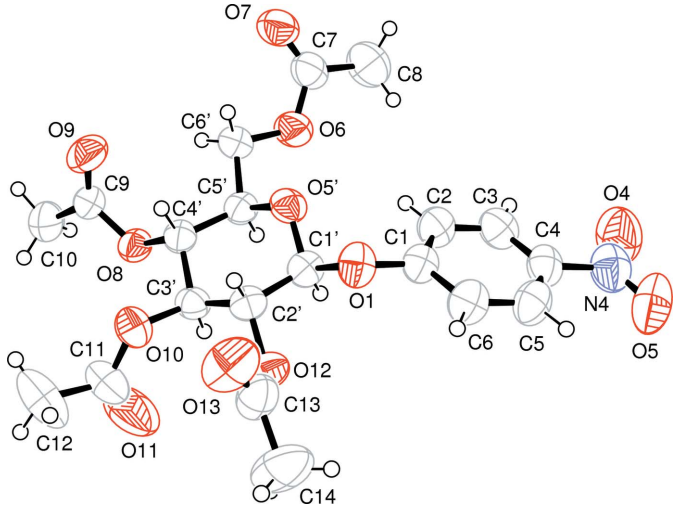


Figure 1
The molecular structure of (I), with the atom-labeling scheme. Displacement ellipsoids are shown at the 50% probability level.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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