

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–S19.
- Bocelli, G. & Cantoni, A. (1992). *Acta Cryst.* **C48**, 961–963.
- Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Polidori, G., Spagna, R. & Viterbo, D. (1989). *J. Appl. Cryst.* **22**, 389.
- Casella, L. (1984). *Inorg. Chem.* **23**, 2781–2787.
- Castro, J., Romero, J., Vázquez, J. A. G., Durán, M. L., Castineiras, A. & Sousa, A. (1990). *J. Chem. Soc. Dalton Trans.* pp. 3255–3258.
- Charles, R. G. & Freiser, H. (1953). *J. Org. Chem.* **18**, 422–425.
- Enraf–Nonius (1988). *CAD-4 VAX/PC Fortran System. Operator's Guide to the Enraf–Nonius CAD-4 Diffractometer Hardware, its Software and the Operating System.* Enraf–Nonius, Delft, The Netherlands.
- Enraf–Nonius (1993). *CAD-4 Express Software.* Version 1.1. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.
- Hordvik, A. (1966). *Acta Chem. Scand.* **20**, 1885–1891.
- İde, S., Öztaş, S. G., Ancin, N. & Tüzün, M. (1996). *Cryst. Res. Technol.* **31**, 427–431.
- Jadamus, H., Fernando, Q. & Freiser, H. (1964). *J. Am. Chem. Soc.* **86**, 3056–3059.
- Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Krasinski, R. A. & Fackler, J. P. Jr (1994). *Acta Cryst.* **C50**, 2039–2041.
- Lee, J. D. (1972). *Naturwissenschaften*, **15**, 36.
- Lee, J. D. & Bryant, M. W. R. (1970). *Acta Cryst.* **B26**, 1729–1735.
- Marsden, C. J. & Smith, B. J. (1988). *J. Phys. Chem.* **92**, 347–353.
- Meyer, B. (1976). *Adv. Inorg. Chem. Radiochem.* **18**, 287–317.
- Muller, A. & Diemann, E. (1987). *Adv. Inorg. Chem.* **31**, 89–122.
- Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1989). *J. Chem. Soc. Dalton Trans.* pp. S1–S83.
- Sugiura, Y. & Hirayama, Y. (1976). *Inorg. Chem.* **15**, 679–681.
- Sugiura, Y., Hirayama, Y., Tanaka, H. & Ishizu, K. (1975). *J. Am. Chem. Soc.* **97**, 5577–5581.
- Tinant, B. & Declercq, J.-P. (1986). *Acta Cryst.* **C42**, 383–384.

Acta Cryst. (1997). **C53**, 378–379

N-Acetyl-2,3,4,6-tetra-*O*-acetyl-*N*-benzyl- β -D-glucopyranosylamine

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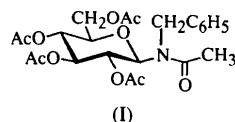
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Abstract

The solid-state conformation of the title compound, C₂₃H₂₉NO₁₀, has been unequivocally established. The configuration at C1 is β -D-glucopyranose and the pyranose ring is essentially a perfect chair. Only the *E* rotamer along the N—C bond is observed.

Comment

Many natural products and their derivatives have an *N*-acyl group joined to a sugar moiety and as the chemical and biological properties of these compounds depend on their structure and conformation, the correct determination is important. In some *N*-alkyl-*N*-glycosylamides, the presence of two conformational isomers is observed in solution due to hindered rotation about the amide C—N bond. In the present case, (I), no preferred conformation was established in solution so we undertook a crystal structure determination.



Crystals of (I) were obtained (Avalos, Babiano, Durán, Jiménez & Palacios, 1992) from the reaction of acetic anhydride with *N*-benzylglucopyranosylamine in pyridine. The same authors studied several sugar amides and thioamides, including the title compound, by NMR spectroscopy. Our structural analysis shows that only one anomer, *i.e.* β -D-glucopyranose, and one rotamer along the N—C bond (*E*) are present in the crystal.

A *PLATON* view (Spek, 1994) of the title molecule viewed along the *z* axis, together with the atomic numbering, is shown in Fig. 1. Bond lengths and angles agree with those of analogous compounds (Banic, Kojic-Prodic, Kroon-Beterburg & Keglevic, 1994; Vega, López-Castro & Márquez, 1986), although the pyranose endocyclic bond lengths [O1—C2 1.435 (3) and O—C5 1.427 (3) Å] do not show the anomeric effect characteristic of this ring. The phenyl, acetoxy and acetamide groups are essentially planar.

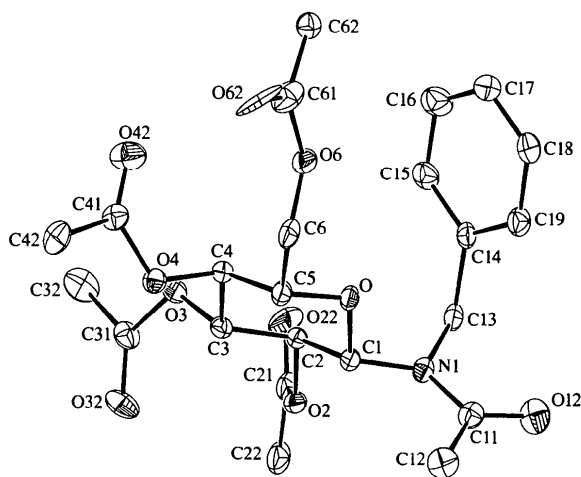


Fig. 1. A *PLATON* (Spek, 1994) plot of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

The geometry observed for the pyranose ring shows no significant distortion from a perfect chair. The O4 and O2 ring substituents are on one side, at distances of 0.541 (2) and 0.371 (2) Å, respectively, and C6 and O3 are on the other side, at distances of -0.171 (3) and -0.584 (2) Å, respectively, of the best plane defined by atoms O, C1, C2, C3, C4 and C5, while the N1 atom is practically in this plane at a distance of -0.012 (2) Å. In terms of ring-puckering coordinates (Cremer & Pople, 1975), amplitudes and phase magnitudes are $Q = 0.577$ (2) Å, $\varphi = -150$ (2) and $\theta = 172$ (3)°. The asymmetry parameters (Nardelli, 1983) are $\Delta C_s(O1) = 0.028$ (1) and $\Delta C_2(C2-O1) = 0.004$ (1). Crystal packing is governed only by van der Waals interactions.

Experimental

Crystals of (I) were obtained (Avalos, Babiano, Durán, Jiménez & Palacios, 1992) from a reaction in which acetic anhydride was gradually added to a solution of *N*-benzylglucopyranosylamine in pyridine at 273 K.

Crystal data

C₂₃H₂₉NO₁₀

$M_r = 479.47$

Monoclinic

$P2_1$

$a = 12.1510$ (10) Å

$b = 9.8460$ (10) Å

$c = 10.6420$ (10) Å

$\beta = 107.14$ (4)°

$V = 1216.7$ (2) Å³

$Z = 2$

$D_x = 1.309$ Mg m⁻³

$D_m = 1.32$ Mg m⁻³

D_m measured by flotation in ethanol/bromobenzene

Data collection

Enraf-Nonius CAD-4 diffractometer

ω - 2θ scans

Absorption correction: none

3677 measured reflections

3677 independent reflections

2812 reflections with

$I > 2\sigma(I)$

Refinement

Refinement on F^2

$R(F) = 0.0485$

$wR(F^2) = 0.1568$

$S = 1.094$

3677 reflections

307 parameters

H atoms not refined

$w = 1/[\sigma^2(F_o^2) + (0.0997P)^2 + 0.0201P]$

where $P = (F_o^2 + 2F_c^2)/3$

Mo $K\alpha$ radiation

$\lambda = 0.7107$ Å

Cell parameters from 25 reflections

$\theta = 5-10^\circ$

$\mu = 0.103$ mm⁻¹

$T = 293$ (2) K

Cube

$0.4 \times 0.4 \times 0.4$ mm

Colourless

Table 1. Selected geometric parameters (Å, °)

O2—C2	1.440 (3)	N1—C1	1.441 (3)
O3—C3	1.442 (3)	N1—C13	1.476 (4)
O4—C4	1.438 (3)	C5—C6	1.509 (4)
O12—C11	1.210 (4)	C11—C12	1.497 (5)
N1—C11	1.381 (4)		
C11—N1—C1	120.5 (2)	O12—C11—C12	120.5 (3)
C11—N1—C13	117.9 (2)	N1—C11—C12	119.2 (3)
C1—N1—C13	120.0 (2)	N1—C13—C14	113.1 (2)
O12—C11—N1	120.3 (3)		

H atoms were placed in geometrically calculated positions, with C—H = 1.00 Å, and included as fixed contributors in the structure-factor calculations, with isotropic displacement parameters related to those of the parent atom. The absolute configuration was not determinable from the diffraction data and was taken to be that of the starting material.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *SET4* (de Boer & Duisenberg, 1984) and *CELDIM* (Rettig, 1989). Data reduction: *XRAY76* (Stewart *et al.*, 1980). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1994). Software used to prepare material for publication: *SHELXL93* and *PARST95* (Nardelli, 1995).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BM1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435–436.
- Avalos, M., Babiano, R., Durán, C. J., Jiménez, J. L. & Palacios, J. C. (1992). *J. Chem. Soc. Perkin Trans. 2*, 2205–2215.
- Banic, Z., Kojic-Prodic, B., Kroon-Beterburg, L. & Keglevic, D. (1994). *Carbohydr. Res.* **259**, 159–174.
- Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst.* **A40**, C-410.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Nardelli, M. (1983). *Acta Cryst.* **C39**, 1141–1142.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Rettig, S. (1989). *CELDIM in CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Spek, A. L. (1994). *PLATON. Molecular Geometry Program*. Version of November 1994. University of Utrecht, The Netherlands.
- Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). *The XRAY76 System*. Technical Report TR-446. Computer Science Center, University of Maryland, College Park, Maryland, USA.
- Vega, R., López-Castro, A. & Márquez, R. (1986). *Acta Cryst.* **C42**, 1066–1068.