Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Benzotriazol-1-yl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-*erythro*-hex-2-enpyranoside

John Nicolson Low,[a]*† Justo Cobo,[b] M. Dolores López,[b] Manuel Nogueras[b] and Adolfo Sánchez[b]

[a]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and [b]Departamento de Química Inorgánica y Orgánica, Universidad de Jaén, 23071 Jaén, Spain

† Postal address: Department of Electrical Engineering and Physics, University of Dundee, Dundee DD1 4HN, Scotland

Correspondence e-mail: jnlow111@hotmail.com

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.046
wR factor = 0.094
Data-to-parameter ratio = 8.6

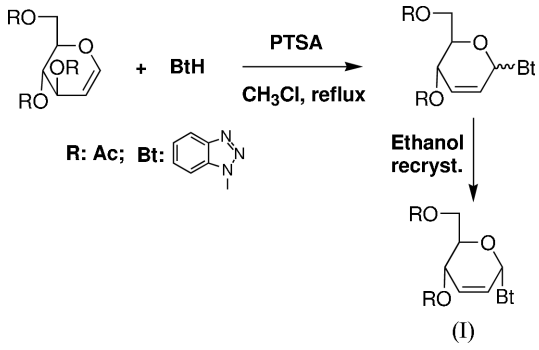
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 5 February 2003
Accepted 11 February 2003
Online 21 February 2003

In the title compound {systematic name: [3(*S*)-acetyloxy-6(*S*)-(1*H*-1,2,3-benzotriazol-1-yl)-3,6-dihydro-2*H*-pyran-2(*S*)-yl]-methyl acetate}, $C_{16}H_{17}N_3O_5$, weak C—H···O hydrogen bonds link the molecules to form a three-dimensional network consisting of linked helical chains.

Comment

The title compound, (I), was prepared as an intermediate in an attempt to optimize the stereoselective synthesis of 2-deoxyglycosyl derivatives, which are of interest as potentially biologically active compounds (see *Scheme*). There are no unusual bond distances or angles in (I). The benzotriazole moiety is planar.



(I)

There are two short intramolecular contacts in the molecule which play a part in the orientation of the acetyl substituents; these are C14—H14···O141 [C···O = 2.690 (4) Å] and C151—H15*A*···O151 [C···O = 2.670 (4) Å, see Fig. 1]. For details of these contacts, see Table 1.

In the following description, the symmetry codes are as in Table 1. Weak C—H···O hydrogen bonds, listed in Table 1, determine the supramolecular structure of the compound. Atom C7 in the molecule at (*x, y, z*) acts as a hydrogen-bond donor, *via* H7, to O141^{i}, while C6 in the molecule at (*x, y, z*) acts as a hydrogen-bond donor, *via* H6, to O151^{ii}. This gives rise to a $C_2^2(13)$ (Bernstein *et al.*, 1995) helical chain which runs parallel to the *a* axis. Fig. 2 shows a stereoview of this chain. Atom C12 in the molecule at (*x, y, z*) acts as a hydrogen-bond donor, *via* H12, to O151^{iii} to form a *C*(9) chain which runs parallel to [101] (Fig. 3). Bonds of this type link the helical chains together, forming a three-dimensional network.

Experimental

An equimolar amount of *p*-toluenesulfonic acid (0.513 ml, 2.7 mmol) was added to a solution of tri-*O*-acetylglucal (0.743 g, 2.7 mmol) and benzotriazole (0.59 g, 2.7 mmol) in 12 ml of chloroform and heated to

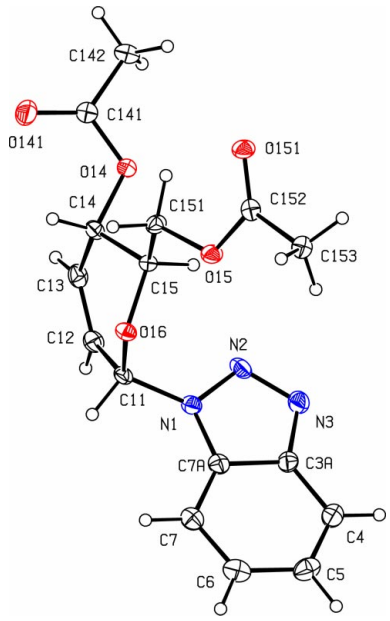


Figure 1
A view of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

reflux overnight. 20 ml of dichloromethane was added to the resulting mixture and then washed subsequently with aqueous NaOH (2 *N*, 2 × 10 ml) and brine (10 ml). The organic layer was dried over anhydrous sodium sulfate and then the solvent was removed to give 0.64 g of a complex mixture as solid foam. The title compound (0.16 g, yield 18%, m.p. 382 K) was isolated in several fractions by crystallization from ethanol at 273 K, to give crystals suitable for X-ray diffraction. Analysis calculated for $C_{16}H_{17}N_3O_5$: C 58.00, H 5.17, N 12.68%; found: C 58.01, H 4.90, N 12.54%.

Crystal data

$C_{16}H_{17}N_3O_5$
M_r = 331.33
Monoclinic, $P2_1$
a = 5.5167 (3) Å
b = 17.7388 (11) Å
c = 8.2217 (5) Å
β = 94.139 (3)°
V = 802.47 (8) Å^3
Z = 2
D_x = 1.371 Mg m^{-3}
Mo Kα radiation
Cell parameters from 1883 reflections
θ = 3.4–27.5°
μ = 0.10 mm^{-1}
T = 120.0 (2) K
Plate, colourless
0.26 × 0.24 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (DENZO–SMN; Otwinowski & Minor, 1997)
T_{min} = 0.974, T_{max} = 0.996
6197 measured reflections
1883 independent reflections
1341 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 27.5°
$h = -7 \rightarrow 7$
$k = -19 \rightarrow 23$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.094
S = 1.00
1883 reflections
219 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

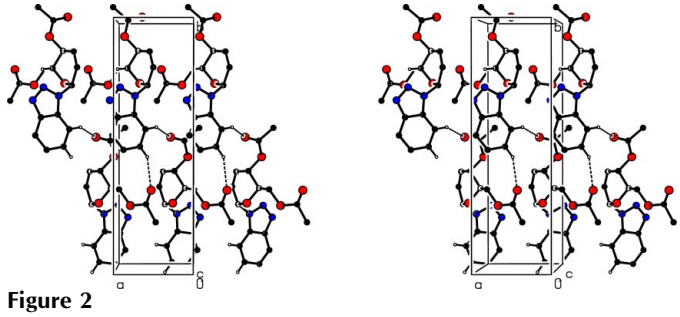


Figure 2
A stereoview of the crystal structure, showing the helical chain running parallel to the *a* axis. All H atoms, except for those involved in the hydrogen bonding, have been omitted for the sake of clarity.

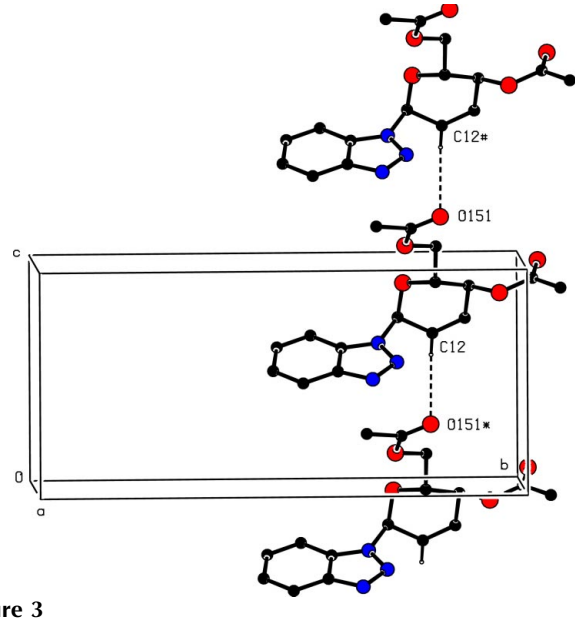


Figure 3
View of the *C*(9) chain which runs parallel to [101]. The molecule labelled with a hash (#) is at $(1 + x, y, 1 + z)$ and that with an asterisk (*) is at $(x - 1, y, z - 1)$. All H atoms, except for those involved in the hydrogen bonding, have been omitted for the sake of clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O141^{i}	0.95	2.40	3.198 (4)	141
C6—H6···O151^{ii}	0.95	2.50	3.369 (5)	152
C12—H12···O151^{iii}	0.95	2.53	3.378 (4)	148
C14—H14···O141	1.00	2.34	2.690 (4)	99
C151—H15*A*···O151	0.99	2.28	2.670 (4)	102

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

H atoms were treated as riding atoms, with C—H = 0.95–1.00 Å. Friedel pairs were merged.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2002); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton; the authors thank the staff for all their help and advice. JNL thanks NCR Self-Service, Dundee, for grants which have provided computing facilities for this work.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Nonius (1997). *KappaCCD Server Software*. Windows 3.11 Version. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (2002). *PLATON*. University of Utrecht, The Netherlands.