Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

1′-Deoxy-1′-hydroxymethyl-1,2′-*O*-isopropylidene-*β*-D-arabinofuranose

Bogdan Doboszewski[a] and Alexander Y. Nazarenko[b]*

[a]Department of Chemistry, Federal Rural University of Pernambuco, 52171-900 Recife, PE, Brazil, and [b]Chemistry Department, State University of New York, College at Buffalo, 1300 Elmwood Ave, Buffalo, NY 14222-1095, USA

Correspondence e-mail:
nazareay@buffalostate.edu

Received 16 December 2002
Accepted 6 January 2003
Online 17 January 2003

The six-membered ring of the title compound, $C_9H_{16}O_5$, adopts a chair conformation, while the furanose five-membered ring has an envelope conformation. The packing is stabilized by a two-dimensional network of intermolecular hydrogen bonds.

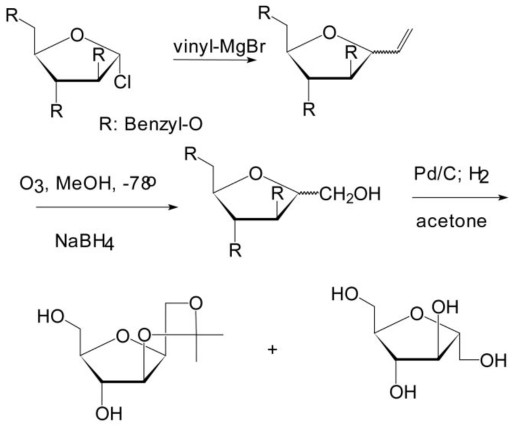
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.081
Data-to-parameter ratio = 9.2

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

Comment

C-Glycosides have received much attention recently as synthetic building blocks or as mimics of the naturally occurring compounds (Jimenez-Barbero *et al.*, 2001; Du & Linhardt, 1998; Postema, 1995; Levy & Tang, 1995). Nucleosides with a methylene or an ethylene linker between the carbohydrate and the nucleobase have been prepared for antiviral screening or for incorporation into antisense sequences (Boal *et al.*, 1996; Doboszewski, 1997; Efimtseva *et al.*, 1995; Hossain *et al.*, 1996). A novel approach (Doboszewski, 1997) to obtaining a carbohydrate substrate suitable for conversion into nucleosides is shown in the Scheme below. Easy separation of anomers was possible after the final isopropylidenation step, because the product (II) does not react with acetone. The target product, (I), was also obtained *via* acid-catalyzed dehydration of D-mannitol or 1,6-di-*O*-benzoyl-D-mannitol, followed by isopropylidenation (Koerner *et al.*, 1977). Acetonide (I) crystallized spontaneously upon standing in a refrigerator, yielding well-formed crystals which were subjected to X-ray study. Some further results dealing with the synthesis of C-glycosyl substrates with the *lyxo* configuration, suitable for conversion into C-analogues of the nucleoside, have recently been published (Doboszewski, 2002).



The bond distances in (I) are in good agreement with values observed in related compounds (Allen *et al.*, 1987). The six-membered ring adopts a chair conformation, while the five-membered ring has an envelope conformation with the C3

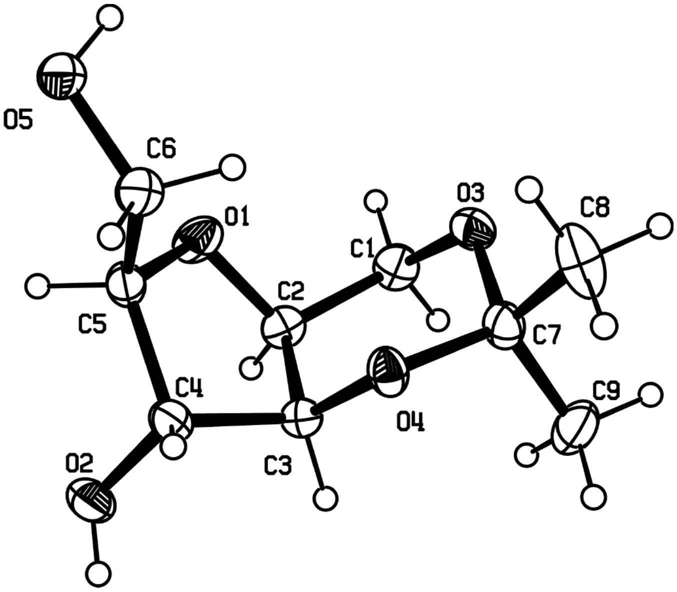


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

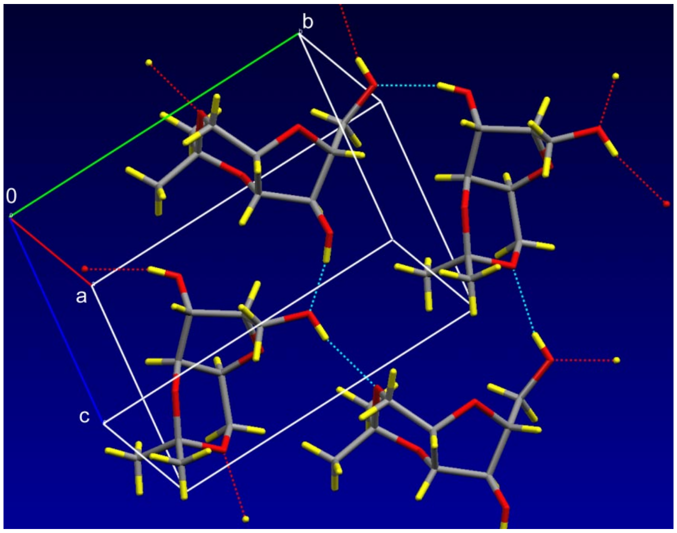


Figure 2
The molecular packing of (I).

atom located out of the least-squares plane of the other four atoms. The packing is stabilized by a two-dimensional network of hydrogen bonds, employing both hydroxy groups of compound (I). The shortest C—H···O contact is longer than 3.45 Å and can hardly play a significant role in intermolecular interactions.

Experimental

Compound (I) crystallized spontaneously after chromatographic purification with a chloroform–methanol mixture (gradient from 100:1 to 10:1), upon standing in the refrigerator, to yield well-formed prismatic crystals; m.p. 388–390 K, $[\alpha]_D$ 27° (c 1.5, EtOH). Other experimental details are shown in the *Scheme*.

Crystal data

$C_9H_{16}O_5$
$M_r = 204.22$
Monoclinic, $P2_1$
$a = 6.227$ (5) Å
$b = 9.817$ (8) Å
$c = 8.292$ (5) Å
$\beta = 102.56$ (5)°
$V = 494.8$ (6) Å^3
$Z = 2$

$D_x = 1.371$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 12$–13°
$\mu = 0.11$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.32 × 0.28 × 0.27 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
1315 measured reflections
1211 independent reflections
1007 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$

$\theta_{\rm max} = 27.6°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 12$
$l = -10 \rightarrow 10$
3 standard reflections every 100 reflections intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.081$
$S = 1.06$
1211 reflections
131 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.0294P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.437 (3)	O4—C7	1.424 (3)
O1—C2	1.439 (3)	C1—C2	1.519 (3)
O3—C1	1.426 (3)	C2—C3	1.522 (3)
O3—C7	1.438 (3)	C3—C4	1.524 (3)
O4—C3	1.422 (3)	C4—C5	1.531 (3)
C5—O1—C2	110.59 (18)	O4—C3—C2	111.74 (18)
C1—O3—C7	114.25 (17)	C2—C3—C4	102.5 (2)
C3—O4—C7	115.70 (17)	C3—C4—C5	103.07 (17)
O3—C1—C2	112.20 (19)	O1—C5—C4	106.25 (19)
O1—C2—C3	105.33 (19)	O4—C7—O3	108.64 (19)
C1—C2—C3	113.0 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2B···O5$^{\rm i}$	0.82	2.02	2.800 (4)	158
O5—H5B···O3$^{\rm ii}$	0.82	2.05	2.853 (4)	166

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

The absolute configuration was known from the synthetic route; there were no Friedel pairs in the data set. All H atoms were fixed geometrically and allowed to ride on their parent C and O atoms.

Data collection: *P*4 *Software* (Siemens, 1995); cell refinement: *P*4 *Software*; data reduction: *P*4 *Software*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 1990).

The authors thank Dr N. Kent Dalley for his kind permission to use the diffractometer.

Acta Cryst. (2003). E**59**, o158–o160 Bogdan Doboszewski *et al.* • $C_9H_{16}O_5$ **o159**

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Boal, J. H., Wilk, A., Scremin, C. L., Gray, G. N., Philips, L. R., Beaucage, S. L. (1996). *J. Org. Chem.* **61**, 8617–8626.

Doboszewski, B. (1997). *Nucleosides Nucleotides*, **16**, 1049–1052.

Doboszewski, B. (2002). *J. Carbohyd. Chem.* **21**, 79–88.

Du, Y. G. & Linhardt, R. J. (1998). *Tetrahedron.* **54**, 9913–9959.

Efimtseva, E. V., Mikhailov, S. N., Victorova, L. S., Rosovskaya, T. A. & Beabeashvilli, R. S. (1995). *Nucleosides Nucleotides*, **14**, 727–729.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Hossain, N., Blaton, N., Peters, O., Rozenski, J. & Herdewijn, P. A. (1996). *Tetrahedron*, **52**, 5563–5578.

Jimenez-Barbero, J., Espinosa, J. F., Asensio, J. L., Canada, F. J. & Poveda, A. (2001). *Adv. Carbohydr. Chem. Biochem.* **56**, 235–284.

Koerner, T. A. W., Voll, R. J. & Younathan, E. S. (1977). *Carbohydr. Res.* **59**, 403.

Levy, D. E. & Tang, C. (1995). *The Chemistry of C-Glycosides*. Oxford: Pergamon.

Postema, M. H. D. (1995). *C-Glycoside Synthesis*. Boca Raton: CRC Press.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

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

Siemens (1995). *P*4 *Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

o160 Bogdan Doboszewski *et al.* • $C_9H_{16}O_5$ *Acta Cryst.* (2003). E**59**, o158–o160