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1-Deoxy-4-*C*-hydroxymethyl-3,4-*O*-isopropylidene-*β*-L-ribulofuranose [(2*R*,3*S*,4*S*)-4-(hydroxymethyl)-3,4-*O*-isopropylidene-2-methyltetrahydrofuran-2,3,4-triol]

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The stereochemistry of crystalline 1-deoxy-4-*C*-hydroxymethyl-3,4-*O*-isopropylidene-*β*-L-ribulofuranose, $C_9H_{16}O_5$, was unequivocally established by X-ray crystallographic analysis to be the *β*-anomer; the absolute configuration was determined by the use of D-ribose as a starting material.

Key indicators

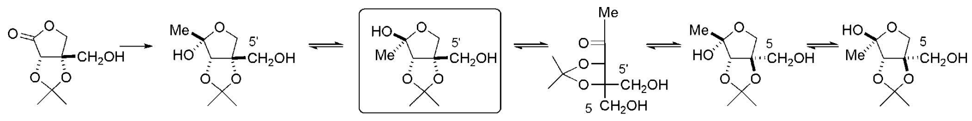
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.106
Data-to-parameter ratio = 10.4

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

Comment

Novel monosaccharides have considerable potential as nutraceuticals (DeFelice, 1995; Liang *et al.*, 1999; Dillard & German, 2000); while rare sugars have considerable potential as alternative foodstuffs (Sun *et al.*, 2007; Levin, 2002; Skytte, 2002), monosaccharides also have a range of other desirable medicinal activities (Nakajima *et al.*, 2004; Menavuvu *et al.*, 2006; Hossain *et al.*, 2006)

In a synthesis of a hitherto unknown carbohydrate, 1-deoxy-4-*C*-hydroxymethyl-L-ribulofuranose, the unprotected form of (4), methyl magnesium bromide was added to the lactone (1) (Best *et al.*, 2007) to give a single-crystalline compound. Addition of the methyl anion to (1) from the top face of the carbonyl would give (2) or from the bottom face would produce (3). Either (2) or (3) could equilibrate through the open chain derivative (4) to the lactols (5) and (6) which would be formed by cyclization of C5′ CH_2OH on to the ketone function in (4); (2) and (3) are formed from (4) by cyclization of C5 CH_2OH. Although structure (4) may easily be eliminated as the structure formed, using other spectroscopic techniques it is difficult to determine which of the other structures is formed. X-ray crystallographic analysis of the crystals formed in the reaction show unequivocally that the compound isolated is (3). The relative configuration shows that the *β*- (rather than the *α*-) anomer is formed at C2 and that the lactol is formed by cyclization of C5 (rather than the C5′) CH_2OH group. The absolute configuration was determined by the use of D-ribose as the synthetic starting material for the lactone (1).



(1) (2) (3) (4) (5) (6)

The crystal structure is connected by a three-dimensional network of hydrogen bonds with each molecule acting as a donor and acceptor for 2 hydrogen bonds (Fig. 2, Fig. 3). There are no unusual bond lengths or angles.

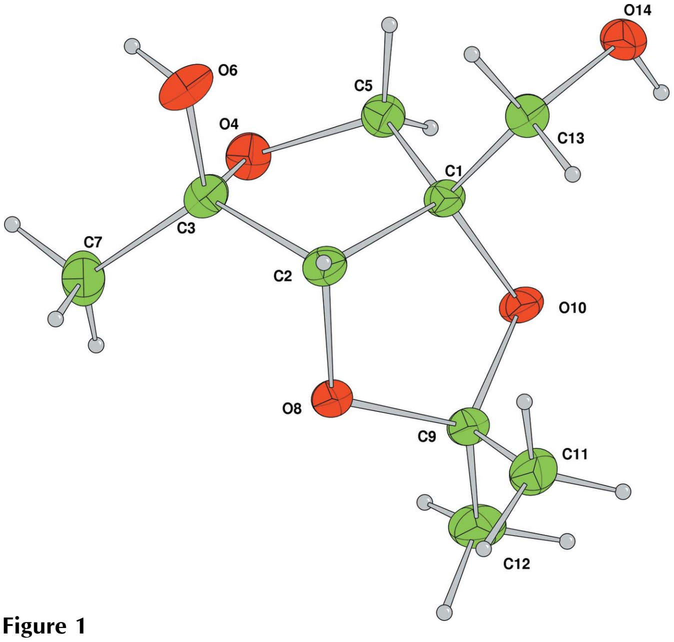


Figure 1
The molecular structure of (3) with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

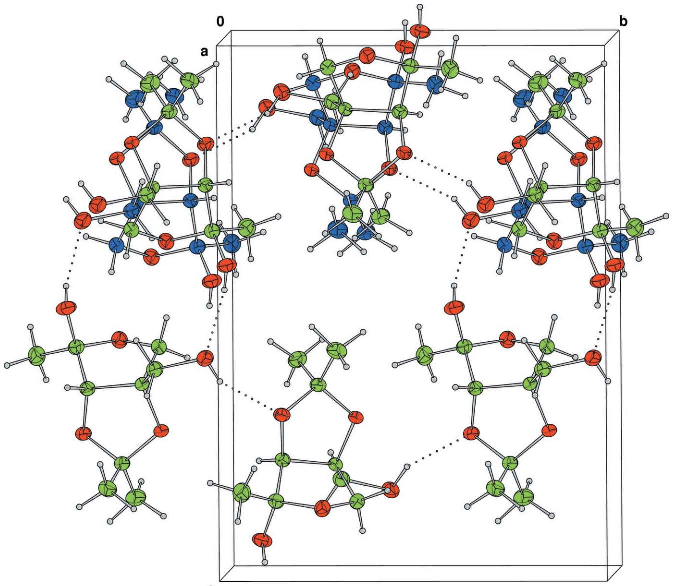


Figure 2
The packing of the title compound projected approximately along the *a* axis. Hydrogen bonds are shown as dotted lines.

Experimental

1-Deoxy-4-*C*-hydroxymethyl-L-ribulofuranose (3) was recrystallized from a mixture of ethyl acetate and cyclohexane by slow evaporation. M.p. 371–373 K; $[\alpha]_D^{18}$ +32 (*c*, 1.32 in chloroform).

Crystal data

$C_9H_{16}O_5$
M_r = 204.22
Orthorhombic, $P2_12_12_1$
a = 6.2405 (2) Å
b = 10.8088 (3) Å
c = 14.9369 (6) Å
V = 1007.53 (6) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.11 mm^{-1}
T = 150 K
0.50 × 0.20 × 0.10 mm

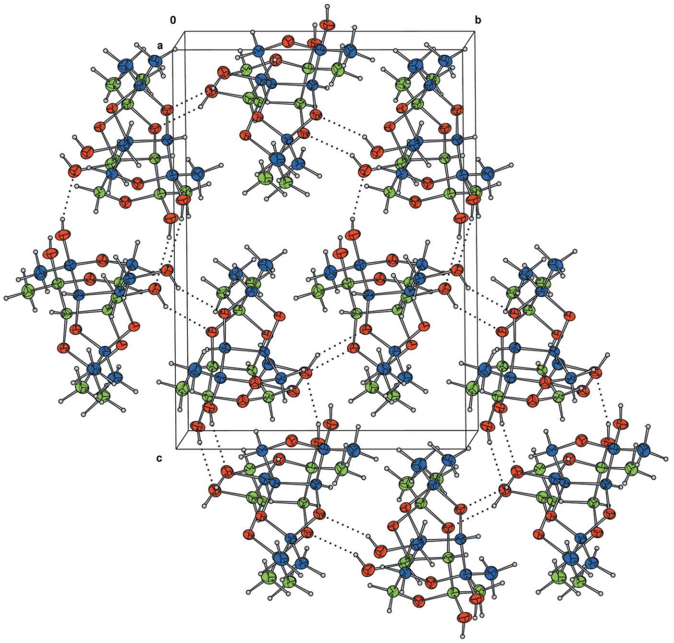


Figure 3
The packing of the title compound showing the three-dimensional hydrogen-bonding network. Hydrogen bonds are shown as dotted lines.

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min}$ = 0.69, $T_{\max}$ = 0.99
7730 measured reflections
1315 independent reflections
1210 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.066

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.106
S = 0.84
1315 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.27 e Å^{-3}
$\Delta\rho_{\min}$ = −0.27 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O14—H13···O8$^{\rm i}$	0.85	2.06	2.831 (2)	152
O6—H7···O14$^{\rm ii}$	0.83	2.01	2.783 (2)	155

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assigned from the starting material.

The relatively large ratio of minimum to maximum corrections applied in the multi-scan process (1:1.45) reflects changes in the illuminated volume of the crystal. These were kept to a minimum, and were taken into account (Görbitz, 1999) by the multi-scan inter-frame scaling (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997).

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98,

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O—H = 0.82 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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