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# 1,5-Anhydro-2,3-*O*-isopropylidene-L-lyxofuranose [(1*S*,4*S*,5*S*,6*R*)-5,6-*O*-isopropylidene-2,7-dioxabicyclo[2.2.1]heptane-5,6-diol]

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**Key indicators**

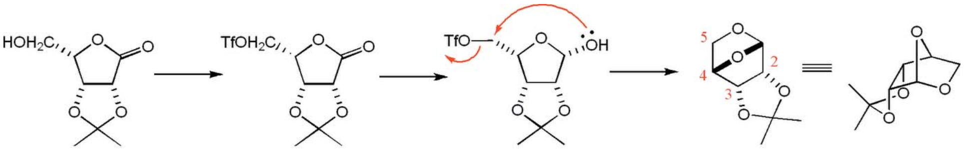
Single-crystal X-ray study
$T$ = 150 K
Mean $\sigma$(C–C) = 0.003 Å
$R$ factor = 0.031
$wR$ factor = 0.031
Data-to-parameter ratio = 7.7

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

The tricyclic title compound, $C_8H_{12}O_4$, was formed in the reduction by diisobutylaluminium hydride of a 5-*O*-trifluromethanesulfonyl lactone and is likely to be useful as a chiral intermediate for the synthesis of bioactive compounds. The absolute configuration was determined by the use of 2,3-*O*-isopropylidene-L-lyxono-1,4-lactone as the starting material.

## Comment

Branched sugar lactones may be useful as versatile intermediates in the synthesis of novel branched biologically important molecules and biopolymeric materials (Asano *et al.*, 2000; Ichikawa & Igarashi, 1995; Ichikawa *et al.*, 1998; Soengas *et al.*, 2005; Hotchkiss *et al.*, 2004, 2006). The Ho crossed-aldol reaction (Ho, 1979, 1985) of lactols with formaldehyde is one of the most powerful strategies for the synthesis of these compounds. In the course of the synthesis of a branched sugar lactone by a Ho procedure (Simone *et al.*, 2005), the trifluoromethanesulfonate, (2), derived from the 2,3-*O*-isopropylidene-L-lyxono-1,4-lactone, (1) (Simone *et al.*, 2005), was treated with diisobutylaluminium hydride (DIBAL-H). A rationalization for the formation of the title compound, (4), is that initial reduction afforded the $\beta$-lyxose, (3), which spontaneously cyclized to (4) by $S_N2$ displacement of the trifluoromethanesulfonate. The value of such highly oxygenated bicyclic intermediates as (4) is well established (Cossy *et al.*, 1995; Pechy *et al.*, 1993), and the unexpected formation of (4) may provide another valuable chiron.



The structure of (4) has been determined by X-ray diffraction (Fig. 1), which showed that it is formed by a six-membered ring fused to a five-membered ring (the acetonide protecting group). The five-membered ring adopts an envelope conformation. The six-membered ring is a $B_{2,5}$-type conformationally constrained structure, due to the existence of the oxygen bridge linking the 1- and 4-positions.

## Experimental

1,5-Anhydro-2,3-*O*-isopropylidene-L-lyxofuranose, (4), was obtained in 28% yield upon overnight reduction of the trifluoromethanesulfonate (2) with DIBAL-H in tetrahydrofuran at low concentration (16 mg ml$^{-1}$). The title material, (4), isolated as a crystalline but volatile product, was recrystallized *via* solvent evaporation (ethyl acetate–cyclohexane) ($R_f$ 0.57; m.p. 338–339 K). HRMS (FI$^+$), found: 172.0732 $[M]^+$; $C_8H_{12}O_4$ requires: 172.0736; $[\alpha]_D^{21}$ 106.8 (*c*, 0.72 in methanol); IR (thin film, $\nu_{max}$, cm$^{-1}$): 2992, 2955, 2906 (*s*, C—H), 1378 (–O—CO—CH), 1291 (C—O); $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 1.34, 1.60 [2 × 3H, 2 × *s*, C(CH$_3$)$_2$], 3.55 (1H, *ddd*, $J_{H5,H5'} = 6.6$ Hz, $J_{H5,H4} = 3.5$ Hz, $J_{H5,H3} = 1.3$ Hz, H5), 4.31 (1H, *a–d*, $J = 6.8$ Hz, H5′), 4.45 (1H, *a–dd*, $J = 8.2$ and 2.3 Hz, H2), 4.64 (1H, *dd*, $J_{H3,H2} = 8.1$ Hz, $J_{H3,H4} = 4.6$ Hz, H3), 4.72–4.77 (1H, *m*, H4), 5.45 (1H, *d*, $J_{H1,H2} = 2.2$ Hz, H1); $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta$ 25.6, 26.1 [C(*C*H$_3$)$_2$], 63.7 (C5), 76.5 (C3), 78.8 (C4), 81.6 (C2), 100.1 (C1), 119.7 [*C*(CH$_3$)$_2$].

### *Crystal data*

$C_8H_{12}O_4$
$M_r = 172.18$
Orthorhombic, $P2_12_12_1$
$a = 8.1279$ (3) Å
$b = 9.4993$ (4) Å
$c = 10.8126$ (4) Å
$V = 834.83$ (6) Å$^3$
$Z = 4$
$D_x = 1.370$ Mg m$^{-3$}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm$^{-1}$
$T = 150$ K
Fragment, colourless
0.38 × 0.32 × 0.14 mm

### *Data collection*

Nonius KappaCCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.96$, $T_{max} = 0.98$
5825 measured reflections
1111 independent reflections
834 reflections with $I > 3\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.4°$

### *Refinement*

Refinement on $F$
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.031$
$S = 1.15$
834 reflections
109 parameters
H-atom parameters constrained
$w = [1 - (F_o - F_c)^2/36\sigma^2(F)]^2/[0.275T_0(x) + 0.0396T_1(x) + 0.0219T_2(x)]$ where $T_i$ are Chebychev polynomials and $x = F_c/F_{max}$ (Prince, 1982; Watkin, 1994)
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.14$ e Å$^{-3}$
$\Delta\rho_{min} = -0.14$ e Å$^{-3}$

A large single-crystal was cut to give a small fragment. This was mounted on a glass fibre using perfluoropolyether oil and cooled rapidly to 150 K in a stream of cold N$_2$ using an Oxford Cryosystems CRYOSTREAM unit. H atoms were positioned geometrically after each cycle of refinement, with C—H = 1.00 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*and *SCALEPACK* (Otwinowski & Minor, 1997); 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*.

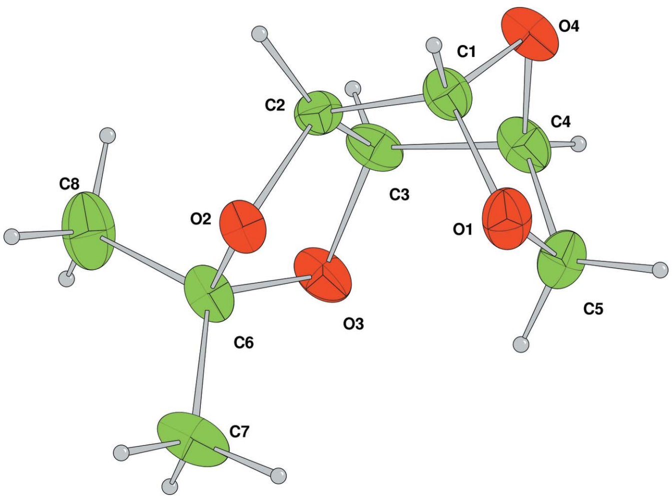


**Figure 1**
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

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