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5-*O*-Acetyl-D-ribono-1,4-lactone

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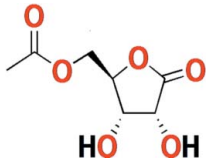
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.135; data-to-parameter ratio = 10.6.

The title compound, $C_7H_{10}O_6$, was obtained from a regioselective enzyme-catalysed acylation of D-ribono-1,4-lactone. The five-membered ring of the acylated sugar shows an envelope conformation. In the crystal, the molecules are linked by intermolecular O—H···O hydrogen-bonds, forming a one-dimensional polymeric structure parallel to [010]. In addition, packing analysis shows stacking along the b axis.

Related literature

For general background to carbohydrates, see: Corma *et al.* (2007); Han *et al.* (1993); Simone *et al.* (2005). For biocatalysed acylation reactions, see: Díaz-Rodríguez *et al.* (2005); Wu *et al.* (2008). For related structures, see: Shalaby *et al.* (1994); Bye (1979); Amador *et al.* (2004); Sá *et al.* (2008); Gress & Jeffrey (1976).



Experimental

Crystal data

$C_7H_{10}O_6$
M_r = 190.15
Monoclinic, $P2_1$
a = 6.1409 (4) Å
b = 5.1952 (15) Å
c = 13.1844 (18) Å
β = 95.118 (12)°
V = 418.95 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.50 × 0.30 × 0.13 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
2164 measured reflections
1346 independent reflections
1015 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.135
S = 1.07
1346 reflections
127 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O4^{i}	0.85 (5)	1.95 (5)	2.781 (3)	164 (3)
O4—H4···O2^{i}	0.85 (5)	2.15 (5)	2.910 (3)	148 (5)
O4—H4···O3^{i}	0.85 (5)	2.41 (6)	3.086 (4)	136 (4)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *SET4* in *CAD-4 Software*; data reduction: *HELENA* (Spek, 1996); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2026).

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supporting information

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5-*O*-Acetyl-D-ribono-1,4-lactone

Adailton J. Bortoluzzi, Damianni Sebrão, Marcus M. Sá and M. G. Nascimento

S1. Comment

Carbohydrates are valuable sources for the production of synthetic compounds of general relevance (Corma *et al.*, 2007). D-Ribono-1,4-lactone (1) is an inexpensive and abundant sugar derivative that is commercially available from renewable resources (Han *et al.*, 1993, Simone *et al.*, 2005). Many synthetic transformations involving 1 lead to unexpected processes ranging from rearrangements to functional group migrations. In such cases, single-crystal X-ray analysis is the only reliable method for the correct structural and conformational assignments (Sá *et al.*, 2008). Enzyme-catalyzed acylation of sugars is, in general, regioselective. Lipases (EC 3.1.1.3) are the most used biocatalyst for this purpose, especially *Candida antarctica* lipase B - CAL-B (Díaz-Rodríguez *et al.*, 2005; Wu *et al.*, 2008). We describe herein the crystal structure of 5-*O*-acetyl-D-ribono-1,4-lactone (2), synthesized from the regioselective acetylation of 1 using CAL-B (Fig. 1).

The molecular structure of the title compound exhibits its 1,4-lactone ring with envelope conformation, which is enveloped on C3 (Fig. 2). Hydroxyl groups are involved in different types of intermolecular O—H···O hydrogen-bonds (Table 1). Hydroxyl group (O3) is the donor for linear hydrogen-bond (O3—H3···O4), whereas hydroxyl group (O4) is the donor for bifurcated interactions (O4—H4···O2 and O4—H4···O3). These interactions link molecules forming one-dimensional zigzag infinite chain parallel to [010] direction. Also, packing analysis shows stack along the *b* crystallographic axis (Fig. 3).

S2. Experimental

The reaction was initiated by dissolving D-ribono-1,4-lactone (74.0 mg, 0.5 mmol) and vinyl acetate (0.14 ml, 1.5 mmol) in anhydrous acetonitrile (10.0 ml) followed by the addition of CAL-B (10.0 mg, Novozym 435, 10,000 PLU/g). The mixture was shaken at 308 K and 150 rpm for 24 h. The reaction was stopped by filtering off the lipase. Finally, solvent was evaporated and the product 5-*O*-acetyl-D-ribono-1,4-lactone was obtained as a white solid (94% yield). Careful recrystallization from acetone provided the crystals (413–414 K) suitable for X-ray diffraction analysis.

S3. Refinement

H atoms attached to carbon atoms were placed at their idealized positions with distances of 0.98, 0.97 and 0.96 Å and U_{eq} fixed at 1.2 and 1.5 times U_{iso} of the preceding atom for CH, CH_2 and CH_3, respectively. H atoms of the hydroxyl groups were found from difference map and treated as free atoms. The final refinement of the structure was done averaging all equivalents.

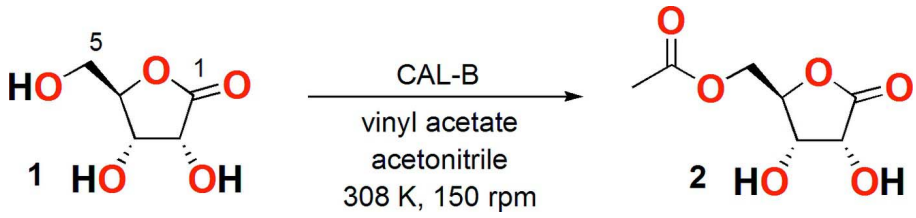

Figure 1

Biocatalyzed acylation reaction.

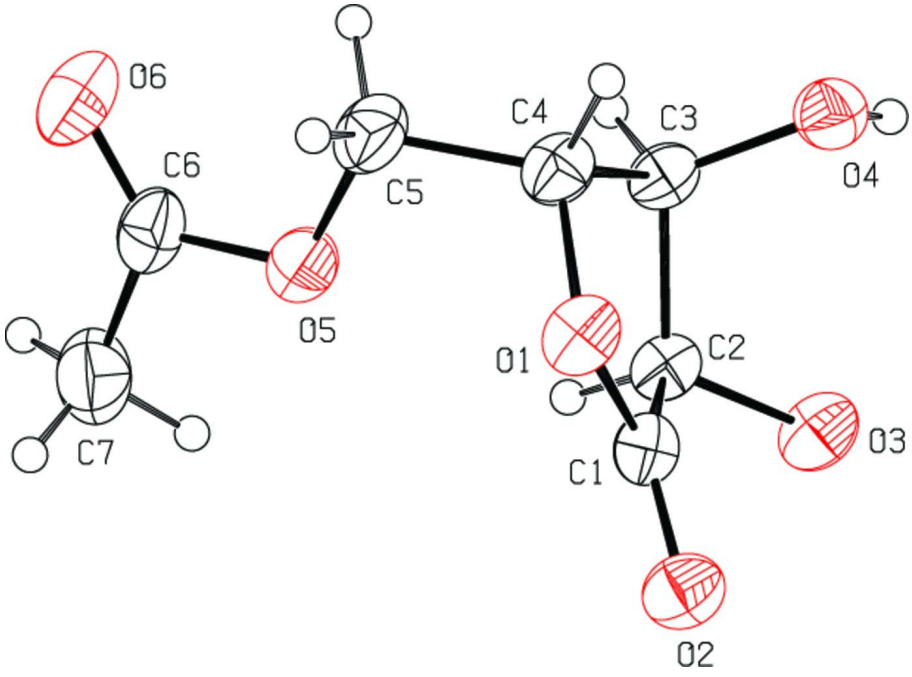

Figure 2

The molecular structure of enantiomeric pair of the title compound showing the atom-labelling scheme. Ellipsoids are drawn at the 40% probability level.

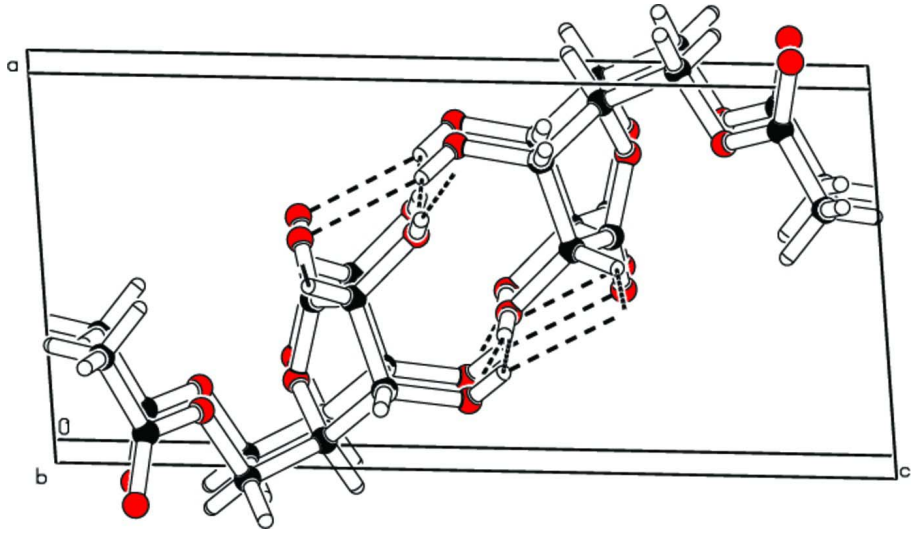

Figure 3

Partial packing of the title compound showing hydrogen bonds.

5-*O*-Acetyl-D-ribono-1,4-lactone

Crystal data

$C_7H_{10}O_6$
$M_r = 190.15$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.1409\ (4)$ Å
$b = 5.1952\ (15)$ Å
$c = 13.1844\ (18)$ Å
$\beta = 95.118\ (12)°$
$V = 418.95\ (14)$ Å^3
$Z = 2$
$F(000) = 200$
$D_x = 1.507$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 3.5–20.5°$
$\mu = 0.13$ mm^{-1}
$T = 293$ K
Prismatic, colorless
0.50 × 0.30 × 0.13 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω–2θ scans
2164 measured reflections
1346 independent reflections
1015 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 30.0°$, $\theta_{min} = 1.6°$
$h = -8\rightarrow8$
$k = -7\rightarrow2$
$l = -18\rightarrow2$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.135$
$S = 1.07$
1346 reflections
127 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0807P)^2 + 0.0065P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4264 (4)	−0.0865 (5)	0.32056 (19)	0.0355 (5)
C2	0.4464 (4)	0.1808 (5)	0.3674 (2)	0.0344 (5)
H2	0.4839	0.3062	0.3162	0.041*
C3	0.2154 (4)	0.2326 (5)	0.3976 (2)	0.0364 (6)
H3A	0.1774	0.4155	0.3913	0.044*
C4	0.0742 (4)	0.0687 (6)	0.3213 (2)	0.0405 (6)
H4A	−0.0460	−0.0050	0.3559	0.049*
C5	−0.0206 (5)	0.2048 (8)	0.2270 (2)	0.0507 (8)
H5A	−0.1029	0.0850	0.1820	0.061*
H5B	−0.1181	0.3413	0.2448	0.061*
C6	0.1100 (5)	0.5012 (7)	0.1095 (2)	0.0482 (7)
C7	0.3079 (6)	0.5894 (10)	0.0629 (3)	0.0660 (11)
H7A	0.3294	0.4848	0.0046	0.099*
H7B	0.4329	0.5748	0.1118	0.099*
H7C	0.2895	0.7658	0.0422	0.099*
O1	0.2157 (3)	−0.1406 (4)	0.29314 (16)	0.0433 (5)
O2	0.5706 (3)	−0.2355 (4)	0.30942 (17)	0.0480 (5)
O3	0.6139 (3)	0.1709 (5)	0.44808 (17)	0.0449 (5)
O4	0.1877 (3)	0.1377 (5)	0.49711 (16)	0.0445 (5)
O5	0.1590 (3)	0.3103 (5)	0.17745 (16)	0.0482 (6)
O6	−0.0705 (4)	0.5837 (6)	0.0916 (2)	0.0666 (8)
H3	0.661 (6)	0.325 (9)	0.455 (3)	0.047 (10)*
H4	0.265 (7)	0.229 (12)	0.540 (4)	0.070 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0392 (12)	0.0292 (12)	0.0375 (12)	−0.0038 (11)	0.0002 (10)	0.0020 (11)
C2	0.0301 (10)	0.0282 (12)	0.0445 (13)	−0.0040 (10)	0.0004 (9)	0.0007 (11)
C3	0.0332 (11)	0.0304 (14)	0.0452 (13)	0.0003 (10)	0.0004 (9)	−0.0002 (11)
C4	0.0320 (11)	0.0398 (16)	0.0489 (14)	−0.0059 (12)	−0.0006 (10)	0.0034 (13)
C5	0.0368 (12)	0.060 (2)	0.0535 (16)	−0.0001 (15)	−0.0068 (11)	0.0076 (17)
C6	0.0556 (16)	0.0446 (16)	0.0417 (14)	0.0035 (16)	−0.0107 (12)	−0.0012 (14)
C7	0.068 (2)	0.078 (3)	0.0513 (18)	−0.002 (2)	0.0015 (16)	0.015 (2)
O1	0.0416 (9)	0.0331 (10)	0.0535 (11)	−0.0079 (9)	−0.0054 (8)	−0.0032 (9)
O2	0.0480 (11)	0.0389 (12)	0.0566 (12)	0.0027 (10)	0.0018 (9)	−0.0052 (10)
O3	0.0371 (9)	0.0401 (13)	0.0555 (12)	−0.0052 (10)	−0.0078 (8)	−0.0051 (10)
O4	0.0411 (9)	0.0487 (13)	0.0438 (10)	0.0013 (10)	0.0033 (8)	−0.0021 (10)
O5	0.0429 (10)	0.0534 (14)	0.0478 (11)	0.0049 (10)	0.0014 (8)	0.0081 (11)
O6	0.0569 (13)	0.0666 (18)	0.0728 (15)	0.0090 (13)	−0.0140 (11)	0.0155 (15)

Geometric parameters (Å, º)

C1—O2	1.195 (3)	C5—O5	1.439 (4)
C1—O1	1.342 (3)	C5—H5A	0.9700
C1—C2	1.521 (4)	C5—H5B	0.9700
C2—O3	1.413 (3)	C6—O6	1.192 (4)
C2—C3	1.531 (4)	C6—O5	1.352 (4)
C2—H2	0.9800	C6—C7	1.482 (5)
C3—O4	1.425 (4)	C7—H7A	0.9600
C3—C4	1.528 (4)	C7—H7B	0.9600
C3—H3A	0.9800	C7—H7C	0.9600
C4—O1	1.460 (4)	O3—H3	0.85 (5)
C4—C5	1.502 (4)	O4—H4	0.85 (5)
C4—H4A	0.9800		
O2—C1—O1	122.6 (3)	C3—C4—H4A	108.5
O2—C1—C2	127.4 (2)	O5—C5—C4	107.4 (2)
O1—C1—C2	110.0 (2)	O5—C5—H5A	110.2
O3—C2—C1	107.4 (2)	C4—C5—H5A	110.2
O3—C2—C3	116.1 (2)	O5—C5—H5B	110.2
C1—C2—C3	102.9 (2)	C4—C5—H5B	110.2
O3—C2—H2	110.0	H5A—C5—H5B	108.5
C1—C2—H2	110.0	O6—C6—O5	122.9 (3)
C3—C2—H2	110.0	O6—C6—C7	126.1 (3)
O4—C3—C4	107.8 (2)	O5—C6—C7	111.0 (3)
O4—C3—C2	111.6 (2)	C6—C7—H7A	109.5
C4—C3—C2	102.4 (2)	C6—C7—H7B	109.5
O4—C3—H3A	111.5	H7A—C7—H7B	109.5
C4—C3—H3A	111.5	C6—C7—H7C	109.5
C2—C3—H3A	111.5	H7A—C7—H7C	109.5
O1—C4—C5	109.6 (3)	H7B—C7—H7C	109.5
O1—C4—C3	105.5 (2)	C1—O1—C4	110.9 (2)
C5—C4—C3	116.0 (3)	C2—O3—H3	105 (2)
O1—C4—H4A	108.5	C3—O4—H4	108 (3)
C5—C4—H4A	108.5	C6—O5—C5	116.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4^{i}	0.85 (5)	1.95 (5)	2.781 (3)	164 (3)
O4—H4···O2^{i}	0.85 (5)	2.15 (5)	2.910 (3)	148 (5)
O4—H4···O3^{i}	0.85 (5)	2.41 (6)	3.086 (4)	136 (4)

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1.