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2,3-*O*-(*S*)-Benzylidene-2-*C*-methyl-D-ribono-1,4-lactone

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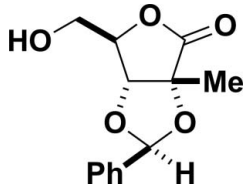
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.075; data-to-parameter ratio = 9.5.

The crystal structure of the title compound, $C_{13}H_{14}O_5$, establishes (i) the (*S*) – rather than (*R*) – configuration at the acetal carbon and (ii) that both the acetal and the lactone form five- rather than six-membered rings; the absolute configuration is determined by the use of 2-*C*-methyl-D-ribono-1,4-lactone as the starting material. The compound consists of hydrogen-bonded chains of molecules running along the *a* axis; there are no unusual packing features. Only classical hydrogen bonding has been considered.

Related literature

For the synthesis of sugar lactones and their use as building blocks, see: Lundt & Madsen (2001); Hotchkiss, Soengas *et al.* (2007); Booth *et al.* (2008, 2009); Jenkinson *et al.* (2007); Hotchkiss, Kato *et al.* (2007); Chen & Joullie (1984); Dho *et al.* (1986); Baird *et al.* (1987). For the structures of benzylidene acetals, see: Baggett *et al.* (1985); Zinner *et al.* (1968).



Experimental

Crystal data

$C_{13}H_{14}O_5$
M_r = 250.25
Orthorhombic, $P2_12_12_1$
a = 8.6170 (2) Å
b = 10.4615 (3) Å
c = 13.2693 (5) Å
V = 1196.18 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 150 K
0.50 × 0.40 × 0.40 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.91, T_{max} = 0.96
8306 measured reflections
1547 independent reflections
1369 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 0.96
1547 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 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
O18—H181···O9^{i}	0.84	2.02	2.801 (3)	153

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

Data collection: *COLLECT* (Nonius, 1997-2001).; 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*.

We would like to thank the Chemical Crystallography Department and ALT at Oxford University for use of the difractometers.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2882).

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supporting information

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2,3-*O*-(*S*)-Benzylidene-2-*C*-methyl-D-ribono-1,4-lactone

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S1. Comment

Lactones have been widely used for the enantiospecific synthesis of complex chiral targets (Lundt & Madsen, 2001). 2-*C*-Methyl-D-ribono-1,4-lactone **3** (Fig. 1) has recently become a readily available starting material (Hotchkiss, Soengas *et al.*, 2007; Booth *et al.*, 2008) and has been used in the synthesis of doubly branched sugars (Booth *et al.*, 2009), 2-*C*-methyl nucleosides (Jenkinson *et al.*, 2007) and complex piperidines (Hotchkiss, Kato *et al.*, 2007). D-Ribono-1,4-lactone **5** with benzaldehyde and concentrated aqueous hydrochloric acid forms a 5 ring benzylidene acetal - 6-ring lactone **6** (Fig. 1). The structure of **6** was established by X-ray crystallographic analysis (Baggett *et al.*, 1985) which corrected the original erroneous 6 ring benzylidene acetal - 5-ring lactone structure proposed (Zinner *et al.*, 1968). The protected 1,5-lactone **6** leaves only the C-2 OH unprotected and has been widely used as a chiron (Chen & Joullie, 1984; Dho *et al.*, 1986; Baird *et al.*, 1987). It was hoped that the analogous reaction with 2-*C*-methyl lactone **3** would form the analogous lactone **4**; however, treatment of **3** with benzaldehyde and concentrated aqueous hydrochloric acid gave as the major product a mixture of epimeric 1,4-lactones **1** and **2**; although it was not possible to separate **1** and **2** by chromatography, suitable crystals of the major component **1** were obtained and the structure of a 5 ring benzylidene acetal - 5-ring lactone, together with the (*S*) stereochemistry at the acetal carbon, was firmly established (Fig. 2).

The compound consists of H—O···H hydrogen bonded chains of molecules running along the *a*-axis (Fig. 3); there are no unusual packing features. Only classical hydrogen bonding has been considered.

S2. Experimental

The title compound was recrystallized from a mixture of diethyl ether and petrol by slow evaporation: m.p. 369–372 K; $[\alpha]_D^{18}$ -38.7 (*c*, 0.86 in $CHCl_3$).

S3. Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged.

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, 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.

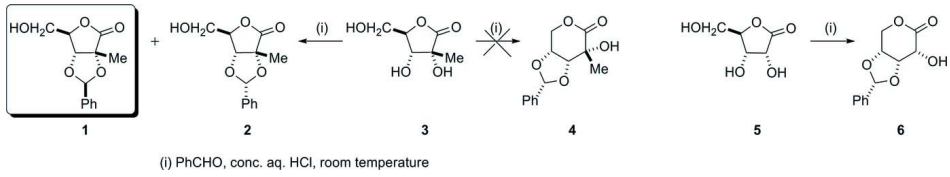

Figure 1

Synthetic Scheme

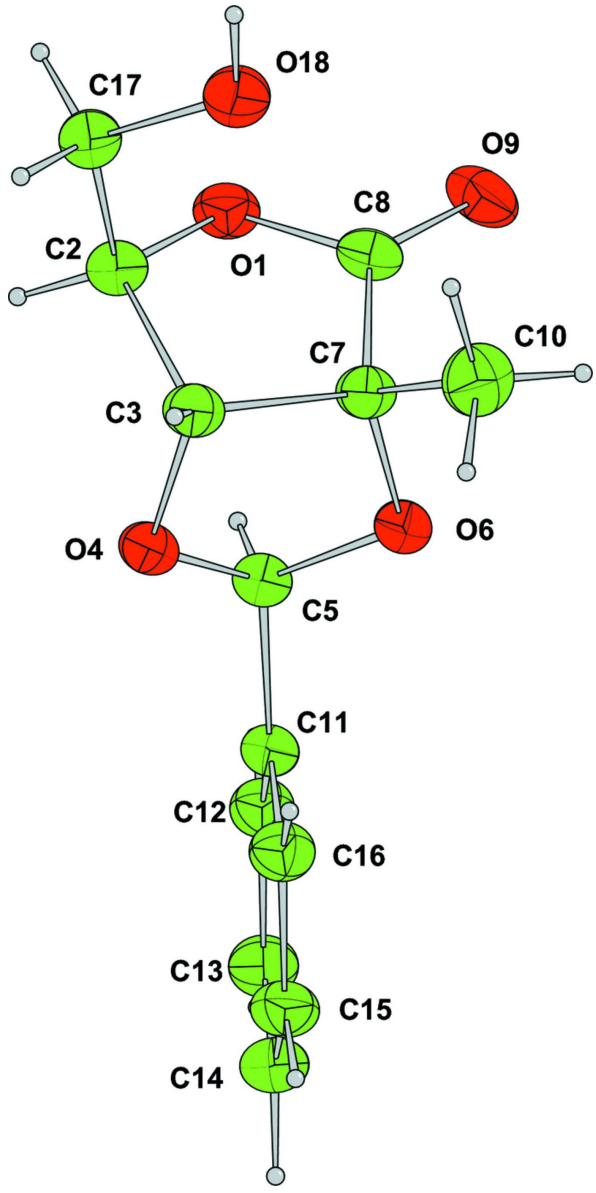

Figure 2

The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

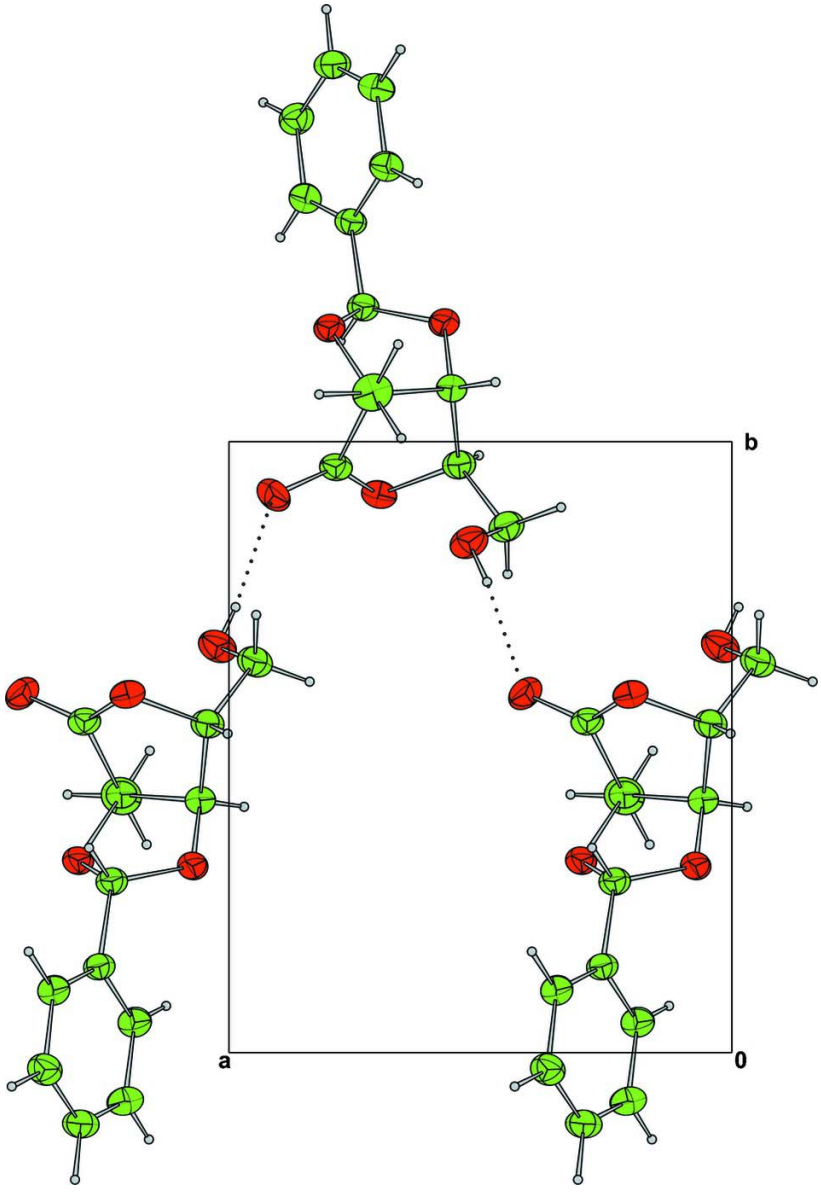

Figure 3

Packing diagram for the title compound projected along the *c*-axis. Hydrogen bonds are indicated by dotted lines.

(I)

Crystal data

$C_{13}H_{14}O_5$
$M_r = 250.25$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.6170\ (2)$ Å
$b = 10.4615\ (3)$ Å
$c = 13.2693\ (5)$ Å
$V = 1196.18\ (6)$ Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.390$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1493 reflections
$\theta = 5$–$27°$
$\mu = 0.11$ mm^{-1}
$T = 150$ K
Block, colourless
0.50 × 0.40 × 0.40 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.91, T_{max} = 0.96
8306 measured reflections
1547 independent reflections
1369 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.4°, θ_{min} = 5.1°
h = −11→11
k = −13→13
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 0.96
1547 reflections
163 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.33P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.000267
$\Delta\rho_{max}$ = 0.21 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}
O1	0.20180 (16)	0.58639 (12)	0.35659 (10)	0.0315
C2	0.0428 (2)	0.53792 (18)	0.36504 (15)	0.0308
C3	0.0572 (2)	0.41273 (17)	0.42367 (14)	0.0290
O4	0.07235 (15)	0.30513 (13)	0.35853 (12)	0.0348
C5	0.2332 (2)	0.27989 (17)	0.34757 (14)	0.0282
O6	0.30017 (15)	0.31402 (12)	0.44177 (10)	0.0292
C7	0.2137 (2)	0.42186 (16)	0.47876 (13)	0.0271
C8	0.2876 (2)	0.54172 (17)	0.43333 (14)	0.0284
O9	0.41116 (16)	0.58753 (14)	0.45530 (11)	0.0382
C10	0.2143 (3)	0.4195 (2)	0.59243 (14)	0.0403
C11	0.2581 (2)	0.14034 (17)	0.32652 (14)	0.0278
C12	0.3486 (2)	0.10164 (19)	0.24540 (15)	0.0314
C13	0.3662 (2)	−0.0280 (2)	0.22427 (16)	0.0349
C14	0.2945 (3)	−0.11716 (19)	0.28505 (15)	0.0358
C15	0.2066 (2)	−0.07955 (18)	0.36660 (15)	0.0345
C16	0.1874 (2)	0.04930 (19)	0.38769 (15)	0.0314
C17	−0.0516 (2)	0.63951 (19)	0.41789 (16)	0.0359
O18	0.02305 (17)	0.66472 (14)	0.51139 (11)	0.0385
H21	0.0028	0.5221	0.2934	0.0368*
H31	−0.0301	0.4024	0.4712	0.0369*
H51	0.2780	0.3352	0.2917	0.0355*
H101	0.3207	0.4225	0.6174	0.0626*
H103	0.1573	0.4942	0.6146	0.0621*
H102	0.1635	0.3404	0.6145	0.0620*
H121	0.3978	0.1652	0.2045	0.0380*
H131	0.4316	−0.0556	0.1653	0.0416*

H141	0.3061	−0.2081	0.2710	0.0428*
H151	0.1588	−0.1422	0.4102	0.0424*
H161	0.1248	0.0765	0.4455	0.0371*
H172	−0.0541	0.7167	0.3741	0.0456*
H171	−0.1605	0.6071	0.4292	0.0454*
H181	−0.0132	0.7294	0.5413	0.0598*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0387 (7)	0.0245 (6)	0.0313 (7)	−0.0037 (6)	0.0002 (6)	0.0025 (5)
C2	0.0337 (9)	0.0244 (9)	0.0343 (10)	0.0014 (8)	−0.0037 (8)	−0.0015 (8)
C3	0.0286 (9)	0.0233 (9)	0.0352 (10)	−0.0005 (8)	−0.0006 (8)	−0.0020 (8)
O4	0.0290 (7)	0.0240 (7)	0.0513 (9)	0.0010 (6)	−0.0099 (7)	−0.0079 (6)
C5	0.0307 (9)	0.0232 (9)	0.0306 (10)	−0.0008 (7)	−0.0024 (8)	−0.0019 (7)
O6	0.0303 (6)	0.0239 (6)	0.0335 (7)	0.0014 (6)	−0.0054 (6)	−0.0044 (5)
C7	0.0296 (9)	0.0226 (8)	0.0290 (9)	−0.0003 (8)	0.0000 (8)	−0.0009 (7)
C8	0.0335 (10)	0.0226 (8)	0.0292 (9)	−0.0013 (8)	0.0028 (8)	−0.0053 (7)
O9	0.0341 (7)	0.0322 (7)	0.0482 (8)	−0.0095 (6)	−0.0005 (7)	−0.0095 (7)
C10	0.0500 (12)	0.0428 (12)	0.0282 (10)	0.0028 (11)	0.0006 (9)	0.0014 (9)
C11	0.0312 (9)	0.0217 (8)	0.0307 (9)	−0.0016 (7)	−0.0040 (8)	−0.0008 (7)
C12	0.0320 (9)	0.0276 (9)	0.0345 (10)	−0.0016 (8)	0.0004 (8)	0.0022 (8)
C13	0.0372 (10)	0.0314 (10)	0.0360 (11)	0.0031 (9)	0.0022 (9)	−0.0041 (8)
C14	0.0418 (11)	0.0245 (9)	0.0411 (11)	0.0017 (9)	−0.0031 (10)	−0.0014 (8)
C15	0.0414 (10)	0.0251 (9)	0.0370 (10)	−0.0042 (9)	−0.0013 (9)	0.0017 (8)
C16	0.0350 (10)	0.0283 (9)	0.0308 (9)	−0.0015 (8)	0.0014 (8)	0.0013 (8)
C17	0.0382 (10)	0.0291 (10)	0.0406 (11)	0.0050 (9)	−0.0079 (10)	−0.0051 (9)
O18	0.0442 (8)	0.0326 (7)	0.0385 (8)	0.0078 (6)	−0.0058 (7)	−0.0091 (6)

Geometric parameters (Å, º)

O1—C2	1.465 (2)	C10—H103	0.968
O1—C8	1.342 (2)	C10—H102	0.981
C2—C3	1.528 (3)	C11—C12	1.390 (3)
C2—C17	1.511 (3)	C11—C16	1.392 (3)
C2—H21	1.025	C12—C13	1.394 (3)
C3—O4	1.425 (2)	C12—H121	0.957
C3—C7	1.537 (3)	C13—C14	1.379 (3)
C3—H31	0.988	C13—H131	1.007
O4—C5	1.418 (2)	C14—C15	1.379 (3)
C5—O6	1.422 (2)	C14—H141	0.974
C5—C11	1.502 (2)	C15—C16	1.387 (3)
C5—H51	1.016	C15—H151	0.967
O6—C7	1.438 (2)	C16—H161	0.981
C7—C8	1.530 (2)	C17—O18	1.422 (2)
C7—C10	1.508 (2)	C17—H172	0.995
C8—O9	1.203 (2)	C17—H171	1.009
C10—H101	0.975	O18—H181	0.844

C2—O1—C8	109.66 (14)	C7—C10—H103	106.8
O1—C2—C3	105.05 (15)	H101—C10—H103	110.3
O1—C2—C17	107.19 (15)	C7—C10—H102	108.1
C3—C2—C17	114.22 (17)	H101—C10—H102	110.2
O1—C2—H21	107.4	H103—C10—H102	111.3
C3—C2—H21	111.2	C5—C11—C12	120.49 (16)
C17—C2—H21	111.3	C5—C11—C16	119.63 (17)
C2—C3—O4	112.06 (15)	C12—C11—C16	119.86 (17)
C2—C3—C7	105.08 (15)	C11—C12—C13	120.03 (18)
O4—C3—C7	104.91 (14)	C11—C12—H121	119.0
C2—C3—H31	110.9	C13—C12—H121	120.9
O4—C3—H31	111.8	C12—C13—C14	119.47 (19)
C7—C3—H31	111.8	C12—C13—H131	119.8
C3—O4—C5	107.37 (13)	C14—C13—H131	120.8
O4—C5—O6	105.05 (15)	C13—C14—C15	120.82 (19)
O4—C5—C11	109.85 (15)	C13—C14—H141	120.1
O6—C5—C11	110.45 (15)	C15—C14—H141	119.0
O4—C5—H51	109.9	C14—C15—C16	120.09 (18)
O6—C5—H51	110.1	C14—C15—H151	120.7
C11—C5—H51	111.3	C16—C15—H151	119.2
C5—O6—C7	106.65 (13)	C11—C16—C15	119.71 (18)
C3—C7—O6	104.08 (14)	C11—C16—H161	119.9
C3—C7—C8	103.21 (15)	C15—C16—H161	120.4
O6—C7—C8	107.03 (14)	C2—C17—O18	106.96 (16)
C3—C7—C10	118.52 (17)	C2—C17—H172	108.2
O6—C7—C10	109.08 (16)	O18—C17—H172	111.6
C8—C7—C10	113.95 (16)	C2—C17—H171	109.5
C7—C8—O1	110.80 (15)	O18—C17—H171	110.7
C7—C8—O9	126.80 (18)	H172—C17—H171	109.8
O1—C8—O9	122.17 (17)	C17—O18—H181	113.1
C7—C10—H101	110.0		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H103···O18	0.97	2.53	3.233 (3)	130
C13—H131···O9^{i}	1.01	2.58	3.289 (3)	128
C14—H141···O1^{ii}	0.97	2.59	3.340 (3)	134
O18—H181···O9^{iii}	0.84	2.02	2.801 (3)	153

Symmetry codes: (i) $-x+1, y-1/2, -z+1/2$; (ii) $x, y-1, z$; (iii) $x-1/2, -y+3/2, -z+1$.