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1,5-Anhydro-2,3,4,6-tetra-*O*-acetyl-D-*lyxo*-hex-1-enitol

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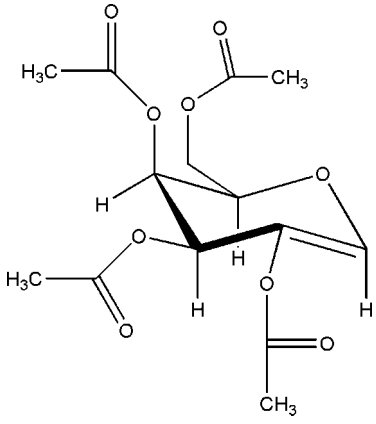
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.105; data-to-parameter ratio = 8.1.

The title compound, $C_{14}H_{18}O_9$, adopt the half-chair conformation $^4H_5$ in the crystalline state. The structure is stabilized by C—H···O hydrogen bonds, in addition to van der Waals forces.

Related literature

For related literature, see: Capozzi *et al.* (2002); Lin *et al.* (2005); Vangehr *et al.* (1979).



Experimental

Crystal data

$C_{14}H_{18}O_9$
M_r = 330.28
Orthorhombic, $P2_12_12_1$
a = 5.5438 (9) Å
b = 12.234 (2) Å
c = 24.312 (4) Å
V = 1649.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.50 × 0.40 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.946, T_{max} = 0.978
8312 measured reflections
1716 independent reflections
1624 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.07
1716 reflections
211 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O9^{i}	0.96	2.56	3.498 (3)	165
C10—H10B···O3^{ii}	0.96	2.56	3.416 (4)	149
C10—H10C···O5^{ii}	0.96	2.59	3.433 (5)	147

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2312).

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supplementary materials

Acta Cryst. (2007). E**63**, o4321 [**doi:10.1107/S1600536807049379**]

1,5-Anhydro-2,3,4,6-tetra-*O*-acetyl-D-*lyxo*-hex-1-enitol

Y. Zhang, J. Jia, L.-N. Pang and J.-W. Wang

Comment

Saccharides are among the most important naturally occurring compounds, and play key roles in the metabolism. Glycal derivatives are a class of important and versatile compounds, which are often derived from saccharides and have found widespread applications in the synthesis of functionalized saccharides with various biological activities (Capozzi *et al.*, 2002; Lin *et al.*, 2005). We herein report the synthesis and crystal structure of a chiral glucal, 1,5-Anhydro-2,3,4,6-tetra-*O*-acetyl-D-*lyxo*-hex-1-entiol, which was synthesized from D-galactose. We report here the crystal structure of (I).

The absolute configuration of the title compound was assigned from a knowledge of the stereochemistry of its synthetic precursor. In (I) (Fig. 1), all bond lengths and angles are normal and in a good agreement with those reported previously (Vangehr *et al.*, 1979). The ring adopt the half-chair conformation $^4H_5$ in the crystalline state. The structure is stabilized by hydrogen bonds of C—H···O type, in addition to van der Waals forces.

Experimental

Acetyl chloride (10 mmol) was added dropwise to the solution of 1,3-bis(4-fluorophenoxy)benzene (10 mmol), aluminium oxide (13 mmol), carbon sulfide (20 ml) and the mixture was heated under reflux for 2 h. Then the mixture was extracted with CS_2 (15 ml) and the organic layer was washed with 50% NaOH solution and water. The excess CS_2 was removed on a water vacuum pump to obtain the final product (80% yield). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

Figures

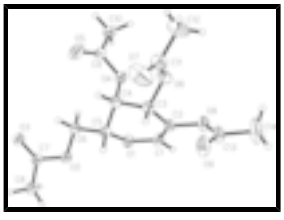

Fig. 1. View of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

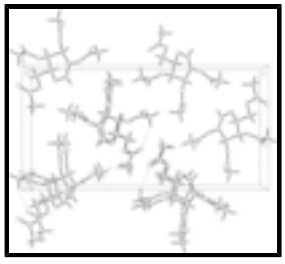

Fig. 2. A packing diagram of the molecule of the title compound. Hydrogen bonds are shown as dashed lines.

1,5-Anhydro-2,3,4,6-tetra-*O*-acetyl-D-*lyxo*-hex-1-enitol

Crystal data

$C_{14}H_{18}O_9$	F_{000} = 696
M_r = 330.28	D_x = 1.330 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 1048 reflections
a = 5.5438 (9) Å	θ = 2.3–22.3º
b = 12.234 (2) Å	μ = 0.11 mm^{-1}
c = 24.312 (4) Å	T = 298 (2) K
V = 1649.0 (5) Å^3	Block, colourless
Z = 4	0.50 × 0.40 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1716 independent reflections
Radiation source: fine-focus sealed tube	1624 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.040
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	h = −6→6
T_{min} = 0.946, T_{max} = 0.978	k = −12→14
8312 measured reflections	l = −20→28

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.2628P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.105	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
1716 reflections	Extinction correction: none
211 parameters	

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.1546 (3)	0.13015 (13)	0.14145 (7)	0.0438 (4)
O2	0.5874 (4)	0.19719 (13)	0.09274 (7)	0.0449 (5)
O3	0.8227 (4)	0.18316 (14)	0.01826 (7)	0.0491 (5)
O4	0.0478 (3)	−0.05072 (13)	0.07124 (7)	0.0425 (4)
O5	0.2868 (5)	−0.1341 (3)	0.01074 (10)	0.0927 (9)
O6	−0.0527 (4)	−0.19092 (14)	0.15321 (8)	0.0509 (5)
O7	0.2425 (6)	−0.3095 (2)	0.16694 (17)	0.1114 (12)
O8	−0.2334 (3)	−0.03845 (15)	0.22552 (7)	0.0504 (5)
O9	0.0155 (4)	−0.1276 (2)	0.28190 (9)	0.0762 (8)
C1	−0.0111 (5)	0.0920 (2)	0.17801 (9)	0.0422 (6)
H1A	−0.1102	0.1428	0.1954	0.051*
C2	−0.0403 (5)	−0.0118 (2)	0.19047 (10)	0.0401 (6)
C3	0.1064 (5)	−0.1017 (2)	0.16562 (10)	0.0410 (6)
H3A	0.2289	−0.1258	0.1920	0.049*
C4	0.2294 (5)	−0.05934 (19)	0.11368 (10)	0.0400 (6)
H4A	0.3565	−0.1101	0.1022	0.048*
C5	0.3366 (5)	0.05263 (19)	0.12544 (9)	0.0391 (5)
H5A	0.4521	0.0453	0.1557	0.047*
C6	0.4671 (6)	0.0983 (2)	0.07650 (9)	0.0461 (6)
H6A	0.5839	0.0457	0.0631	0.055*
H6B	0.3534	0.1136	0.0472	0.055*
C7	0.7630 (5)	0.23214 (19)	0.05877 (9)	0.0389 (6)
C8	0.8643 (7)	0.3370 (2)	0.07797 (10)	0.0561 (8)
H8A	1.0223	0.3469	0.0626	0.084*
H8B	0.8750	0.3365	0.1174	0.084*
H8C	0.7615	0.3959	0.0664	0.084*
C9	0.0957 (5)	−0.0947 (2)	0.02159 (11)	0.0489 (7)
C10	−0.1142 (6)	−0.0863 (3)	−0.01573 (12)	0.0622 (8)

H10A	−0.0748	−0.1187	−0.0505	0.093*
H10B	−0.1547	−0.0107	−0.0210	0.093*
H10C	−0.2492	−0.1239	0.0001	0.093*
C11	0.0378 (7)	−0.2922 (2)	0.15502 (15)	0.0621 (8)
C12	−0.1472 (9)	−0.3745 (3)	0.14133 (18)	0.0938 (13)
H12A	−0.0894	−0.4459	0.1510	0.141*
H12B	−0.1804	−0.3721	0.1026	0.141*
H12C	−0.2923	−0.3591	0.1615	0.141*
C13	−0.1823 (5)	−0.0986 (3)	0.27069 (11)	0.0563 (7)
C14	−0.4027 (7)	−0.1204 (5)	0.30325 (17)	0.1029 (17)
H14A	−0.3586	−0.1389	0.3403	0.154*
H14B	−0.4899	−0.1801	0.2871	0.154*
H14C	−0.5027	−0.0564	0.3035	0.154*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0481 (10)	0.0396 (8)	0.0436 (9)	0.0013 (8)	0.0102 (9)	0.0063 (7)
O2	0.0519 (11)	0.0457 (9)	0.0372 (8)	−0.0086 (9)	0.0101 (8)	−0.0016 (7)
O3	0.0548 (12)	0.0531 (10)	0.0395 (9)	−0.0080 (10)	0.0113 (9)	−0.0041 (8)
O4	0.0427 (10)	0.0493 (9)	0.0356 (8)	0.0061 (9)	−0.0012 (8)	−0.0032 (7)
O5	0.0618 (16)	0.142 (3)	0.0743 (15)	0.0261 (18)	0.0004 (13)	−0.0500 (16)
O6	0.0466 (11)	0.0427 (9)	0.0634 (11)	−0.0056 (9)	−0.0067 (10)	0.0089 (8)
O7	0.0763 (19)	0.0560 (14)	0.202 (4)	0.0119 (15)	−0.019 (2)	−0.0055 (18)
O8	0.0378 (10)	0.0674 (11)	0.0460 (10)	0.0031 (10)	0.0053 (9)	0.0194 (9)
O9	0.0431 (12)	0.132 (2)	0.0534 (12)	0.0056 (15)	−0.0001 (10)	0.0421 (14)
C1	0.0428 (15)	0.0515 (13)	0.0324 (11)	0.0037 (12)	0.0048 (11)	0.0036 (10)
C2	0.0355 (13)	0.0512 (13)	0.0338 (12)	−0.0007 (12)	0.0005 (11)	0.0076 (10)
C3	0.0371 (14)	0.0431 (12)	0.0426 (13)	−0.0028 (12)	−0.0045 (11)	0.0088 (10)
C4	0.0361 (13)	0.0401 (12)	0.0439 (12)	0.0044 (11)	−0.0010 (11)	0.0041 (10)
C5	0.0369 (13)	0.0446 (12)	0.0359 (11)	0.0009 (11)	−0.0015 (11)	0.0050 (10)
C6	0.0491 (15)	0.0527 (13)	0.0364 (12)	−0.0108 (13)	0.0061 (12)	−0.0028 (11)
C7	0.0433 (14)	0.0428 (12)	0.0305 (11)	−0.0013 (12)	0.0013 (11)	0.0075 (10)
C8	0.074 (2)	0.0502 (14)	0.0438 (14)	−0.0173 (16)	0.0089 (15)	−0.0015 (11)
C9	0.0456 (17)	0.0523 (14)	0.0487 (14)	−0.0064 (13)	0.0074 (13)	−0.0148 (12)
C10	0.0591 (19)	0.0769 (19)	0.0506 (15)	−0.0012 (17)	−0.0038 (15)	−0.0203 (15)
C11	0.065 (2)	0.0491 (16)	0.072 (2)	−0.0006 (16)	−0.0018 (18)	0.0035 (14)
C12	0.102 (3)	0.0527 (18)	0.127 (3)	−0.016 (2)	−0.015 (3)	−0.003 (2)
C13	0.0398 (16)	0.0832 (19)	0.0461 (14)	−0.0016 (15)	0.0015 (13)	0.0234 (15)
C14	0.052 (2)	0.160 (4)	0.096 (3)	0.009 (3)	0.021 (2)	0.072 (3)

Geometric parameters (Å, °)

O1—C1	1.361 (3)	C5—C6	1.501 (3)
O1—C5	1.438 (3)	C5—H5A	0.9800
O2—C7	1.346 (3)	C6—H6A	0.9700
O2—C6	1.436 (3)	C6—H6B	0.9700
O3—C7	1.200 (3)	C7—C8	1.477 (4)
O4—C9	1.348 (3)	C8—H8A	0.9600

O4—C4	1.445 (3)	C8—H8B	0.9600
O5—C9	1.193 (4)	C8—H8C	0.9600
O6—C11	1.337 (4)	C9—C10	1.479 (4)
O6—C3	1.436 (3)	C10—H10A	0.9600
O7—C11	1.190 (4)	C10—H10B	0.9600
O8—C13	1.352 (3)	C10—H10C	0.9600
O8—C2	1.407 (3)	C11—C12	1.475 (5)
O9—C13	1.184 (4)	C12—H12A	0.9600
C1—C2	1.316 (4)	C12—H12B	0.9600
C1—H1A	0.9300	C12—H12C	0.9600
C2—C3	1.495 (4)	C13—C14	1.480 (4)
C3—C4	1.526 (3)	C14—H14A	0.9600
C3—H3A	0.9800	C14—H14B	0.9600
C4—C5	1.520 (3)	C14—H14C	0.9600
C4—H4A	0.9800		
C1—O1—C5	115.12 (18)	O3—C7—C8	126.1 (2)
C7—O2—C6	115.74 (18)	O2—C7—C8	110.9 (2)
C9—O4—C4	118.2 (2)	C7—C8—H8A	109.5
C11—O6—C3	117.9 (2)	C7—C8—H8B	109.5
C13—O8—C2	117.3 (2)	H8A—C8—H8B	109.5
C2—C1—O1	124.3 (2)	C7—C8—H8C	109.5
C2—C1—H1A	117.8	H8A—C8—H8C	109.5
O1—C1—H1A	117.8	H8B—C8—H8C	109.5
C1—C2—O8	117.2 (2)	O5—C9—O4	122.3 (3)
C1—C2—C3	123.4 (2)	O5—C9—C10	126.2 (3)
O8—C2—C3	119.2 (2)	O4—C9—C10	111.5 (2)
O6—C3—C2	108.1 (2)	C9—C10—H10A	109.5
O6—C3—C4	111.0 (2)	C9—C10—H10B	109.5
C2—C3—C4	109.14 (19)	H10A—C10—H10B	109.5
O6—C3—H3A	109.5	C9—C10—H10C	109.5
C2—C3—H3A	109.5	H10A—C10—H10C	109.5
C4—C3—H3A	109.5	H10B—C10—H10C	109.5
O4—C4—C5	109.92 (18)	O7—C11—O6	122.0 (3)
O4—C4—C3	107.7 (2)	O7—C11—C12	126.6 (3)
C5—C4—C3	109.0 (2)	O6—C11—C12	111.4 (3)
O4—C4—H4A	110.1	C11—C12—H12A	109.5
C5—C4—H4A	110.1	C11—C12—H12B	109.5
C3—C4—H4A	110.1	H12A—C12—H12B	109.5
O1—C5—C6	107.9 (2)	C11—C12—H12C	109.5
O1—C5—C4	111.8 (2)	H12A—C12—H12C	109.5
C6—C5—C4	112.0 (2)	H12B—C12—H12C	109.5
O1—C5—H5A	108.3	O9—C13—O8	122.9 (3)
C6—C5—H5A	108.3	O9—C13—C14	126.0 (3)
C4—C5—H5A	108.3	O8—C13—C14	111.1 (3)
O2—C6—C5	108.64 (19)	C13—C14—H14A	109.5
O2—C6—H6A	110.0	C13—C14—H14B	109.5
C5—C6—H6A	110.0	H14A—C14—H14B	109.5
O2—C6—H6B	110.0	C13—C14—H14C	109.5
C5—C6—H6B	110.0	H14A—C14—H14C	109.5

H6A—C6—H6B	108.3	H14B—C14—H14C	109.5
O3—C7—O2	123.0 (2)		
C5—O1—C1—C2	−13.4 (4)	C1—O1—C5—C6	167.4 (2)
O1—C1—C2—O8	−174.7 (2)	C1—O1—C5—C4	43.8 (3)
O1—C1—C2—C3	−0.1 (4)	O4—C4—C5—O1	57.5 (2)
C13—O8—C2—C1	−127.1 (3)	C3—C4—C5—O1	−60.4 (2)
C13—O8—C2—C3	58.0 (3)	O4—C4—C5—C6	−63.8 (3)
C11—O6—C3—C2	−149.9 (2)	C3—C4—C5—C6	178.4 (2)
C11—O6—C3—C4	90.5 (3)	C7—O2—C6—C5	162.6 (2)
C1—C2—C3—O6	−138.1 (3)	O1—C5—C6—O2	63.5 (3)
O8—C2—C3—O6	36.4 (3)	C4—C5—C6—O2	−173.1 (2)
C1—C2—C3—C4	−17.3 (4)	C6—O2—C7—O3	−2.2 (4)
O8—C2—C3—C4	157.2 (2)	C6—O2—C7—C8	176.9 (2)
C9—O4—C4—C5	110.5 (2)	C4—O4—C9—O5	−4.7 (4)
C9—O4—C4—C3	−130.9 (2)	C4—O4—C9—C10	175.8 (2)
O6—C3—C4—O4	44.7 (3)	C3—O6—C11—O7	0.7 (5)
C2—C3—C4—O4	−74.3 (2)	C3—O6—C11—C12	180.0 (3)
O6—C3—C4—C5	164.0 (2)	C2—O8—C13—O9	1.2 (5)
C2—C3—C4—C5	44.9 (3)	C2—O8—C13—C14	−179.7 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8B···O9^{i}	0.96	2.56	3.498 (3)	165
C10—H10B···O3^{ii}	0.96	2.56	3.416 (4)	149
C10—H10C···O5^{ii}	0.96	2.59	3.433 (5)	147

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

Fig. 1

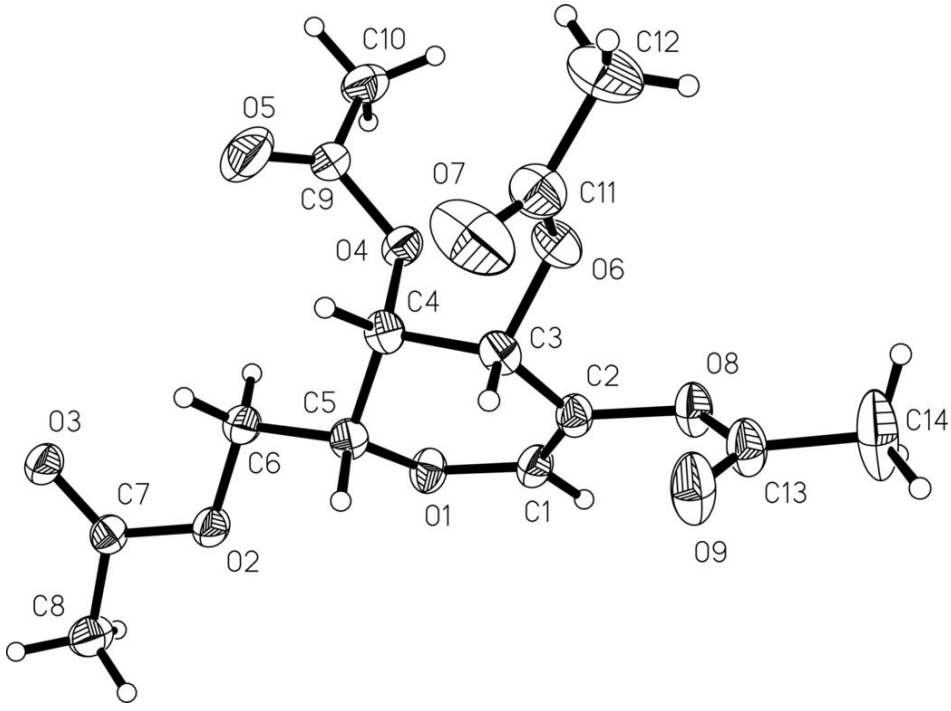
C10
C12
O5
C9
O7
C11
O4
O6
C4
C3
C2
O8
C14
C5
O3
C6
C7
O1
C1
C13
O2
O9
C8

Fig. 2