Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

3,5-Di-*C*-methyl-5,6-*O*-isopropylidene-L-glucono-1,4-lactone

Kathrine V. Booth,[a]* Sarah F. Jenkinson,[a] George W. J. Fleet[a] and David J. Watkin[b]

[a]Department of Organic Chemistry, Chemistry Research Laboratory, University of Oxford, Mansfield Road, Oxford OX1 3TA, England, and [b]Department of Chemical Chrystallography, Chemistry Research Laboratory, University of Oxford, Mansfield Road, Oxford OX1 3TA, England

Correspondence e-mail:
victoria.booth@chem.ox.ac.uk

Received 23 March 2007
Accepted 12 April 2007

The relative configuration at position C-2 of the title lactone, $C_{11}H_{18}O_6$, which exists in the five-membered ring form, was unequivocally established by X-ray crystallographic analysis. There are two molecules present in the asymmetric unit (Z' = 2). The absolute configuration was determined by the use of 2,4-di-*C*-methyl-L-arabinose as the starting material.

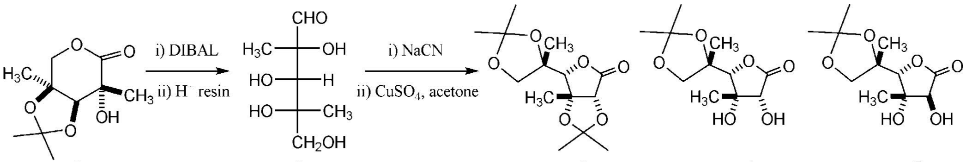
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.070
Data-to-parameter ratio = 9.7

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

Comment

In the title compound, (5) (Booth *et al.*, 2007), there are two molecules in the asymmetric unit (Z' = 2). The crystal structure exists as layers of these two molecules lying parallel to the *b* axis, with each molecule acting as an acceptor and donor for two hydrogen bonds (Fig. 3).



1 2 3 4 5

The main difference between the two independent molecules is in the orientation of the C1—C2 bond between the lactone ring and the acetonide [torsion angles O6—C2—C1—O11 = 175.3 (2)° and O106—C102—C101—O111 = −64.6 (2)°)] (Figs. 1 and 2).

The main residues are very similar. After least-squares fitting of the lactone residues against each other, the r.m.s. positional discrepancy is 0.097 Å, the r.m.s. bond length discrepancy is 0.0055° and the r.m.s. torsion angle deviation is 7.23° (Fig. 4). The five-membered rings of the acetonide groups match less well, as the envelopes pucker in the opposite sense (Fig. 5, Table 2).

Experimental

3,5-Di-*C*-methyl-5,6-*O*-isopropylidene-L-glucono-1,4-lactone was recrystallized by slow evaporation from a mixture of ethyl acetate and cyclohexane until crystals formed (m.p. 370–375 K). $[\alpha]_D^{18}$ −40.4 (*c*, 1.04 in $CHCl_3$).

Crystal data

$C_{11}H_{18}O_6$
M_r = 246.26
Monoclinic, $P2_1$
a = 8.7635 (3) Å
b = 10.6004 (3) Å
c = 13.7678 (4) Å
β = 99.4044 (14)°
V = 1261.79 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 150 K
1.00 × 0.40 × 0.10 mm

doi:10.1107/S1600536807018314

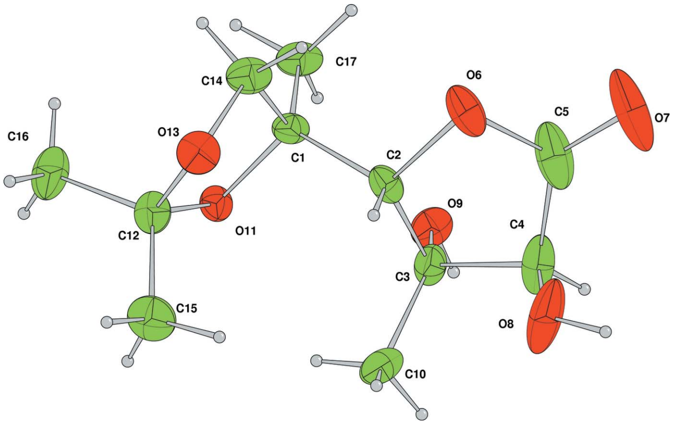


Figure 1
Molecule 1 of compound (5), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

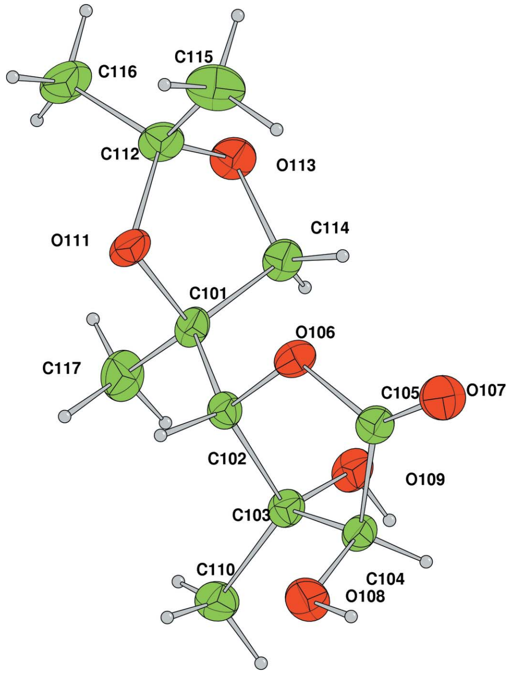


Figure 2
Molecule 2 of compound (5), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.67$, $T_{\max} = 0.99$

10042 measured reflections
2992 independent reflections
2521 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.070$
$S = 0.93$
2992 reflections
307 parameters

1 restraint
H-atom parameters constrained
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

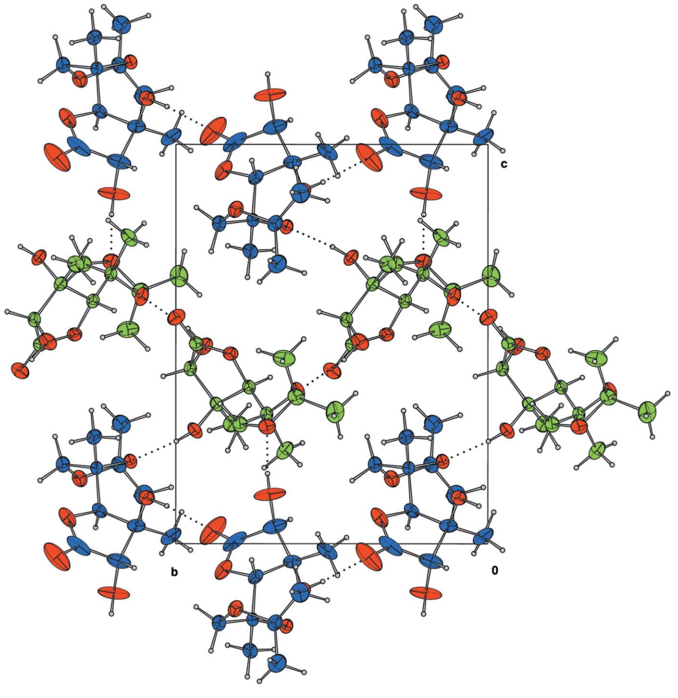


Figure 3
The packing diagram for (5), projected along the *a* axis. The crystal structure contains layers of molecule 1 (blue C atoms) and molecule 2 (green C atoms) running along the *b* direction. Hydrogen bonds are shown as dotted lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O108—H10···O111$^{\rm i}$	0.83	2.01	2.827 (2)	165
O109—H16···O11$^{\rm ii}$	0.85	2.03	2.876 (2)	174
O9—H37···O7$^{\rm iii}$	0.82	2.01	2.825 (2)	175
O8—H38···O113$^{\rm iv}$	0.84	1.93	2.766 (2)	175

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

Table 2
Torsion angles in the five-membered ring of the acetonide moiety of (5) (°).

C1—C14—O13—C12	−38.4 (2)	C101—C114—O113—C112	36.8 (2)
C14—O13—C12—O11	29.2 (2)	C114—O113—C112—O111	−27.9 (2)
O13—C12—O11—C1	−7.9 (2)	O113—C112—O111—C101	7.1 (2)
C12—O11—C1—C14	−14.6 (2)	C112—O111—C101—C114	14.8 (2)
O11—C1—C14—O13	31.9 (2)	O111—C101—C114—O113	−30.5 (2)

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 (1:1.49) reflects changes in the illuminated volume of the crystal. Changes in illuminated volume were kept to a minimum, and were taken into account (Görbitz, 1999) by the multi-scan inter-frame scaling (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997).

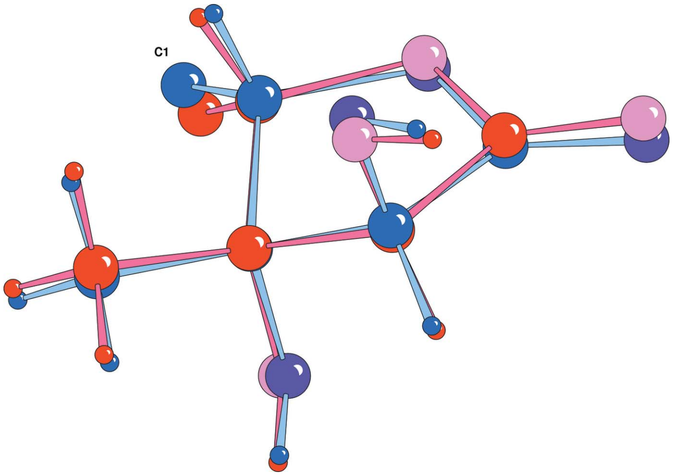


Figure 4
Overlay of the lactone moiety. Molecule 1 is blue with O atoms shown in purple, and molecule 2 is red with O atoms shown in pink. The acetonide portion would be connected at C1.

The H atoms were all located in a difference map, but those attached to C 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 Å and O—H = 0.82 Å) and $U_{\rm iso}$(H) (in the range 1.2–1.5 times $U_{\rm eq}$ of the parent atom), after which the positions were refined with riding constraints.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *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*.

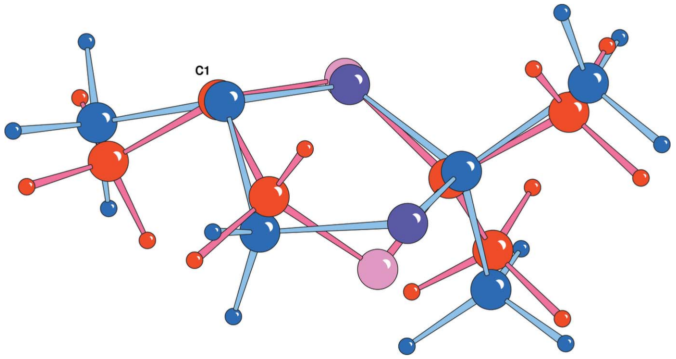


Figure 5
Overlay of the five-membered rings of the acetonide moiety; molecule 1 is blue with O atoms shown in purple, and molecule 2 is red with O atoms shown in pink. The remaining lactone portion would be connected at C1.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.

Booth, K. V., Jenkinson, S. F., Fleet, W. J. & Watkin, D. J. (2007). *Acta Cryst.* E**63**, o2204–o2206.

Görbitz, C. H. (1999). *Acta Cryst.* B**55**, 1090–1098.

Nonius (2001). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.