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

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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. The absolute configuration was determined by the use of 2,4-di-*C*-methyl-L-arabinose as the starting material.

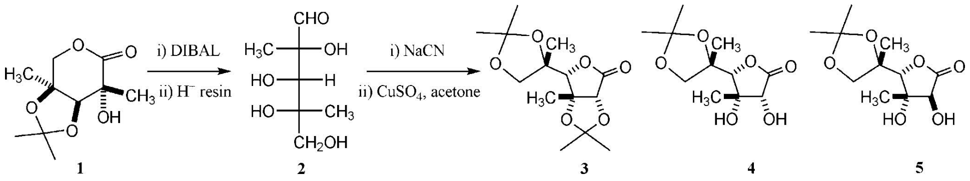
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.075
Data-to-parameter ratio = 10.2

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

Comment

The crystal structure of the title lactone, (4) (Booth *et al.*, 1997), has a three-dimensional network of hydrogen bonding, with each molecule acting as a donor and as an acceptor for two hydrogen bonds (Fig. 2). Two chains of hydrogen bonds can be seen, one from atom O9 to atom O13^{i} running parallel to the b axis (Fig. 3), and a second running from atom O8 to atom O7^{ii} parallel to the a axis (Fig. 4) (symmetry codes as in Table 1).



Experimental

3,5-Di-*C*-methyl-5,6-*O*-isopropylidene-L-mannono-1,4-lactone, (4), was recrystallized by slow evaporation from a mixture of ethyl acetate and cyclohexane until crystals formed (m.p. 433–437 K). $[\alpha]_D^{17}$ −24.7 (c, 1.28 in acetone).

Crystal data

$C_{11}H_{18}O_6$
M_r = 246.26
Orthorhombic, $P2_12_12_1$
a = 7.1286 (2) Å
b = 9.7360 (3) Å
c = 17.1314 (6) Å
V = 1188.99 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 150 K
0.20 × 0.05 × 0.05 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.86, T_{max} = 0.99
7431 measured reflections
1570 independent reflections
1327 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.075
S = 0.93
1570 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

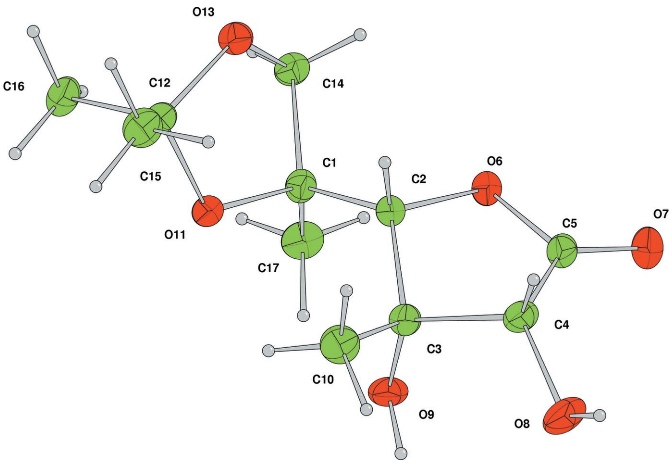


Figure 1
The molecular structure of compound (4), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

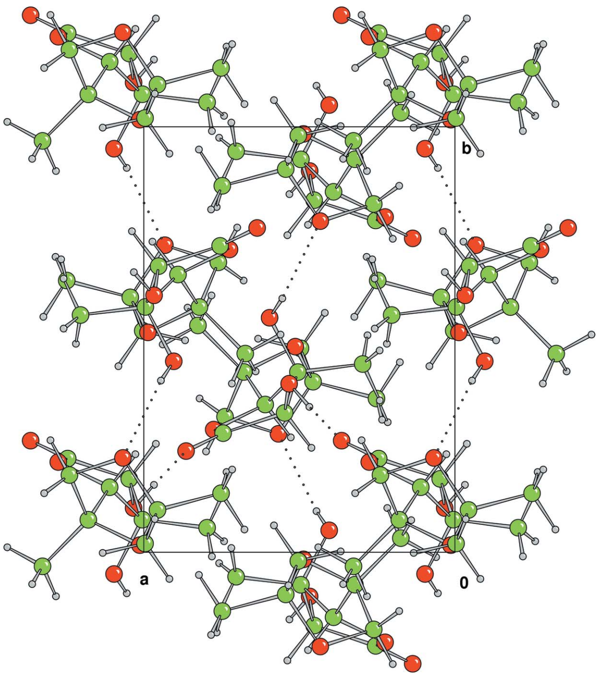


Figure 2
The packing of compound (4), projected along the *c* axis. Dotted lines indicate hydrogen bonds.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O9—H12$\cdots$O13^{i}	0.84	2.10	2.857 (2)	149
O8—H13$\cdots$O7^{ii}	0.84	1.95	2.752 (2)	157

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

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assigned from the starting material.

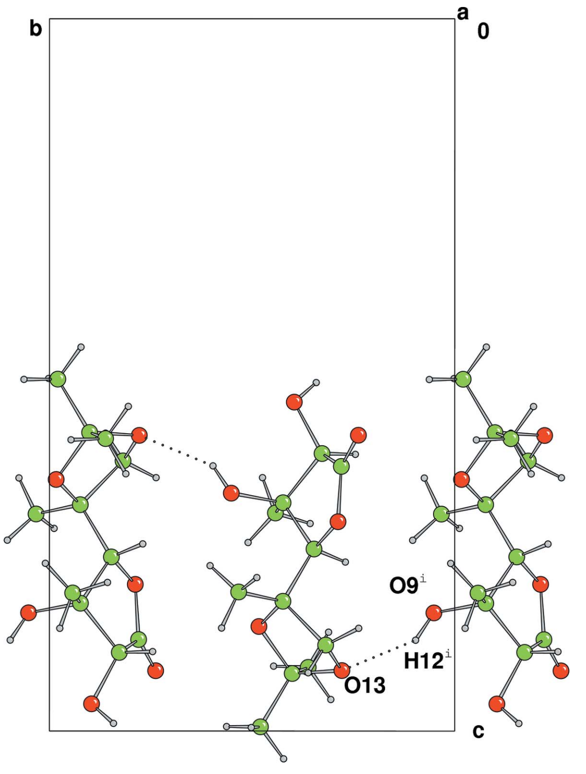


Figure 3
A hydrogen-bonded (dotted lines) chain running parallel to *b*. [Symmetry code: (i) $1-x, -\frac{1}{2}+y, \frac{3}{2}-z$.]

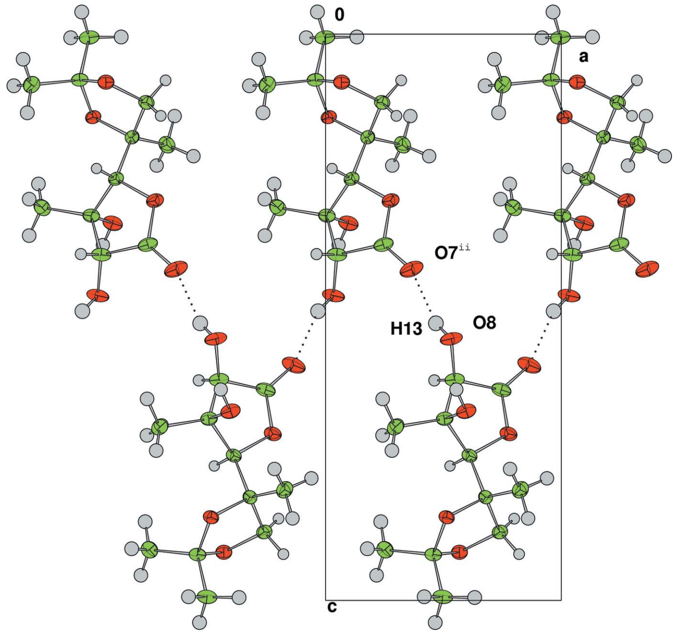


Figure 4
A hydrogen-bonded (dotted lines) chain running parallel to *a*. [Symmetry code: (ii) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$.]

The relatively large ratio of minimum to maximum corrections applied in the multi-scan process (1:1.16) 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).

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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 or1.5 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*.

References

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

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

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

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.

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