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2-*C*-Methyl-D-allono-1,4-lactone

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.092
Data-to-parameter ratio = 8.7

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

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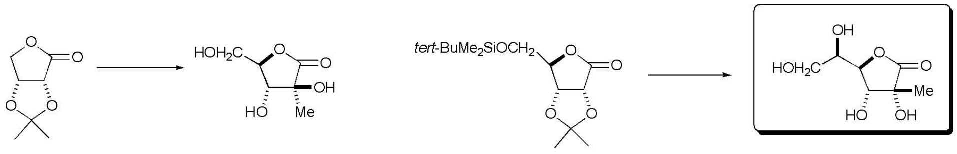
The relative stereochemistry at the quaternary C atom in the title compound, $C_7H_{12}O_6$, a 1,4-lactone formed from a protected D-ribonolactone, is firmly established by X-ray crystallographic analysis.

Comment

The potential of the Kiliani ascension of ketoses to provide readily available branched scaffolds has been recognized (Harding *et al.*, 2005; Hotchkiss *et al.*, 2004; Shallard-Brown *et al.*, 2004); such materials are likely to be of value as a new family of chirons. A further class of branched carbohydrate building blocks may be available from the reaction of cyanide with 1-deoxyketoses, themselves prepared by addition of organometallic reagents to sugar lactones.



(1) (2) (3) (4)

For example, when such a sequence was performed on the acetonide of D-erythronolactone (1), 2-*C*-methyl-D-arabinonolactone (2) was formed (Punzo *et al.*, 2005), in which the 2,3-diol unit is *trans*; none of the epimeric ribonolactone was isolated during the course of the synthesis. When a similar synthetic sequence was applied to the protected D-ribonolactone (3), the crystalline product (4) was isolated, in which the 2,3-diol unit is *cis*. There is no reliable spectroscopic technique to establish the relative stereochemistry in (4), so its relative configuration was unambiguously defined by X-ray crystallographic analysis; the absolute stereochemistry was defined by the use of D-ribonolactone as a starting material.

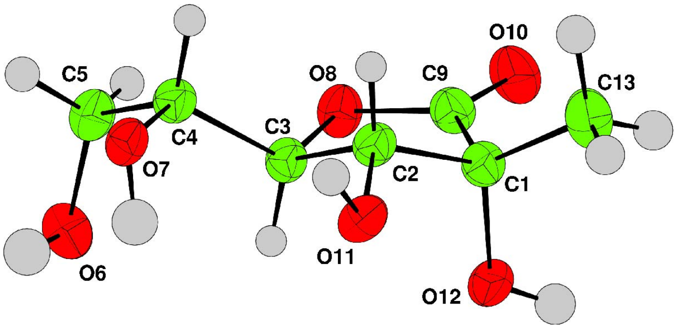


Figure 1
The molecular structure of (4), with displacement ellipsoids drawn at the 50% probability level.

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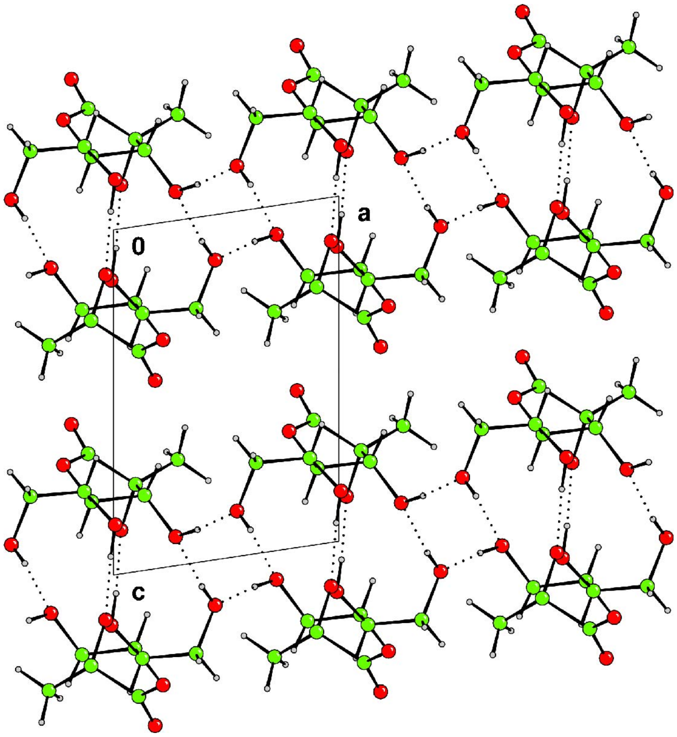

Figure 2
Packing diagram for the title compound, viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

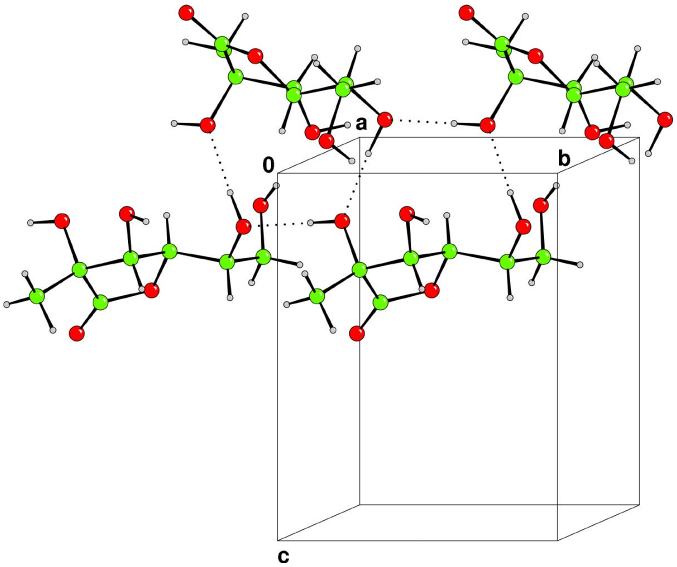

Figure 3
Hydrogen bonding (dashed lines): the zigzag network, forming columns of molecules.

The reactions are being studied further in order to understand the difference in the stereochemical outcome of the two sequences.

The crystal structure is made up of layers of strongly hydrogen-bonded molecules which lie in the *ab* plane. The layers are made up of columns of molecules along the *b* axis held together by a zigzag chain of hydrogen bonds, which are in turn tied together by a helical hydrogen-bonding network (Figs. 3 and 4).

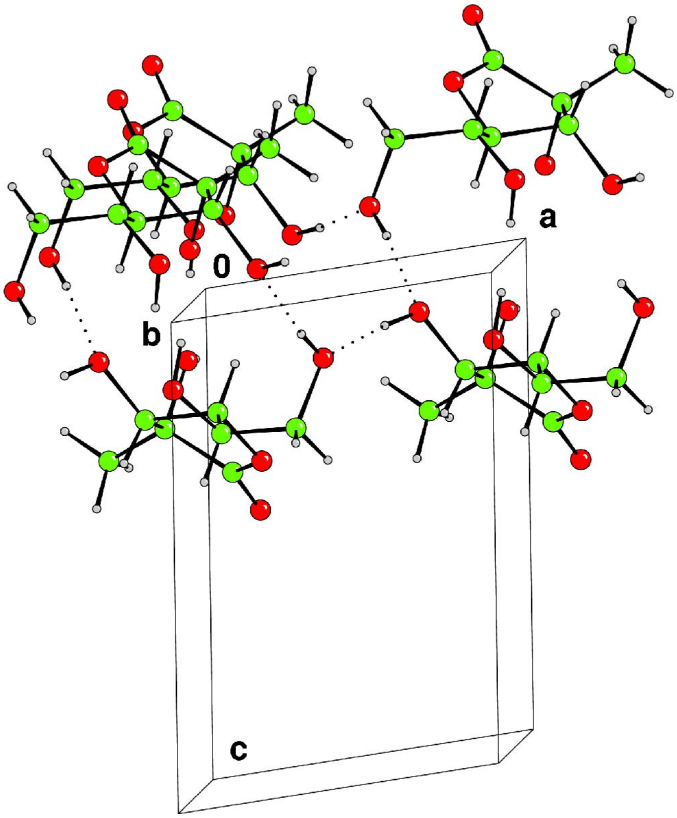

Figure 4
Hydrogen bonding (dashed lines): the helical network which links the columns of molecules together to form a sheet.

Experimental

Crystals of the title compound were obtained by evaporation of a solution in an ethyl acetate/cyclohexane mixture, yielding colourless crystals. The full synthetic procedure will be published separately (Jenkinson *et al.*, 2005).

Crystal data

$C_7H_{12}O_6$
$M_r = 192.17$
Monoclinic, $P2_1$
$a = 6.1521\ (5)$ Å
$b = 7.5495\ (7)$ Å
$c = 9.3055\ (8)$ Å
$\beta = 98.501\ (5)^\circ$
$V = 427.45\ (6)$ Å^3
$Z = 2$
$D_x = 1.493$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1283 reflections
$\theta = 5$–27°
$\mu = 0.13$ mm^{-1}
$T = 190$ K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: none
2940 measured reflections
1025 independent reflections
905 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.026$
$\theta_{\rm max} = 27.5^\circ$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 8$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.092$
$S = 1.05$
1025 reflections
118 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O12—H4$\cdots$O7^{i}	0.88	1.84	2.723 (2)	178
O11—H6$\cdots$O6^{ii}	0.96	1.73	2.681 (2)	175
O6—H8$\cdots$O11^{iii}	0.83	1.95	2.778 (2)	174
O7—H14$\cdots$O12^{iii}	0.96	1.84	2.791 (2)	175

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

All H atoms were observed in a difference electron density map and were refined using slack restraints to optimize their geometry [C—H = 0.98 Å, O—H = 0.82 Å, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) or 1.5$U_{\rm eq}$(O)], then made to ride on their parent atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration is known from the synthesis.

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

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