Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2*C*-Methyl-D-arabinono-1,4-lactone monohydrate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.071
Data-to-parameter ratio = 12.5

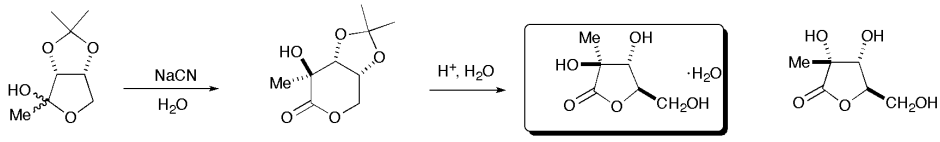
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_6H_{10}O_5\cdot H_2O$, formed by the hydrolysis of a δ-lactone, is shown unequivocally to be a γ-lactone. The diol has a *trans* configuration.

Received 10 December 2004
Accepted 10 January 2005
Online 22 January 2005

Comment

The potential of the Kiliani ascension of ketoses to provide readily available branched scaffolds has been recognized (Hotchkiss *et al.*, 2004). A further class of branched carbohydrate building blocks may be available from the reaction of cyanide on 1-deoxyketoses, themselves prepared by addition of organometallic reagents to sugar lactones. The protected 1-deoxy-D-ribulose, (1), was treated with sodium cyanide and gave a single diastereomeric product, (2), the structure of which was established by X-ray crystallography (Punzo *et al.*, 2005). During the isolation of (2), some loss of the protecting acetonide group afforded an unprotected lactone (3), which was eventually crystallized. NMR and other structural studies on (3) could not firmly determine the size of the lactone ring; X-ray crystallographic analysis established that (3) is a 1,4-lactone (Fig. 1). It is noteworthy that none of the epimeric ribonolactone, (4), was isolated during the course of the synthesis. As usually expected for sugar derivatives, hydrogen bonding (Table 2) occurs between molecules, and the water of crystallization is involved in this network (Fig. 2).



Experimental

Compound (3) was crystallized by dissolving it in diethyl ether, adding a few drops of cyclohexane and allowing the slow competitive evaporation of the two solvents until clear colourless crystals formed. Water was used as solvent during the synthesis of the compound. Moreover the compound was exposed to air before and after crystallization.

Crystal data

$C_6H_{10}O_5\cdot H_2O$
M_r = 180.16
Orthorhombic, $P2_12_12_1$
a = 8.1624 (3) Å
b = 8.5569 (3) Å
c = 11.6000 (5) Å
V = 810.20 (5) Å^3
Z = 4
D_x = 1.477 Mg m^{-3}
Mo Kα radiation
Cell parameters from 1300 reflections
θ = 5–30°
μ = 0.13 mm^{-1}
T = 120 K
Plate, colourless
0.30 × 0.20 × 0.04 mm

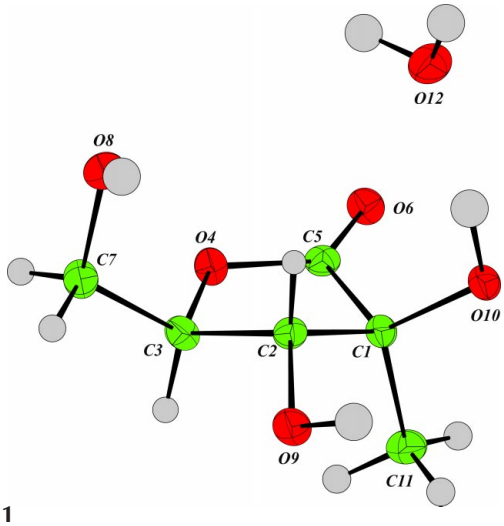


Figure 1
The asymmetric unit of (3), with displacement ellipsoids drawn at the 50% probability level.

Data collection

Nonius KappaCCD diffractometer	1361 independent reflections
ω scans	1201 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)	$R_{int} = 0.013$
	$\theta_{max} = 30.0°$
	$h = -11 \rightarrow 11$
$T_{min} = 0.97$, $T_{max} = 0.99$	$k = -11 \rightarrow 12$
2296 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F^2) + 0.03 + 0.17P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.071$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
$S = 0.98$	$\Delta\rho_{min} = -0.24$ e Å^{-3}
1361 reflections	Absolute structure: see text
109 parameters	
H-atom parameters constrained	

Table 1
Selected bond lengths (Å).

C1—C2	1.537 (2)	C3—O4	1.4695 (18)
C1—C5	1.528 (2)	C3—C7	1.516 (2)
C1—O10	1.4169 (17)	O4—C5	1.3363 (18)
C1—C11	1.526 (2)	C5—O6	1.2106 (17)
C2—C3	1.525 (2)	C7—O8	1.4261 (19)
C2—O9	1.4167 (18)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O10—H5···O12	0.92	1.81	2.7191 (16)	175
O8—H7···O6^{i}	0.97	1.78	2.7235 (15)	163
O9—H9···O8^{i}	0.96	1.76	2.7157 (15)	169
O12—H12···O9^{ii}	0.94	2.01	2.9138 (16)	163
O12—H1···O10^{iii}	0.91	2.00	2.8613 (16)	157

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

In the absence of significant anomalous scattering, Friedel pairs were merged. The absolute configuration was assigned since the starting material was D-erythronolactone with known absolute configuration. H atoms were located in difference density maps. 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 = 0.97–1.01 Å and O—H = 0.91–0.97 Å), after which they were refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) and U_{iso}(H) = 0.05 Å^2 for those bonded to the O atoms.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR*92 (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*.

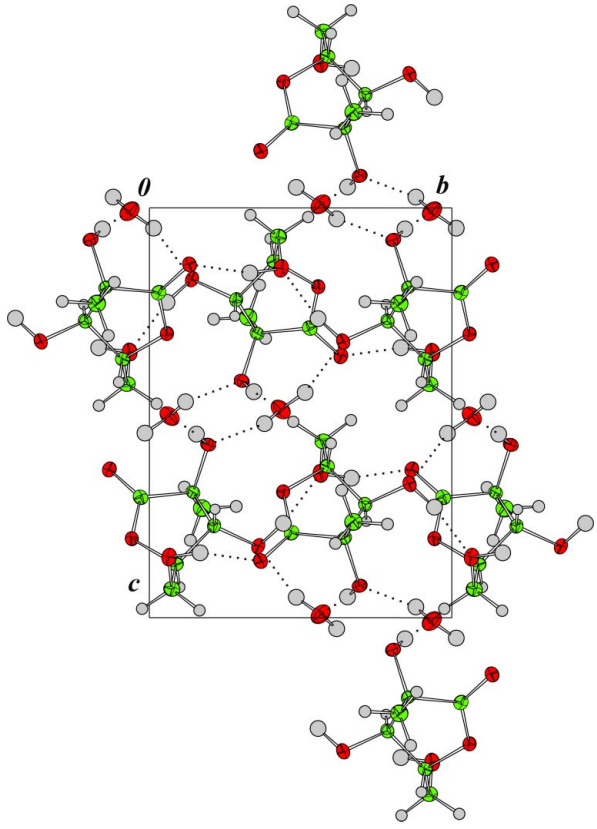


Figure 2
Packing diagram of (3), viewed down the *a* axis. Hydrogen bonds are indicated by dashed lines.

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