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

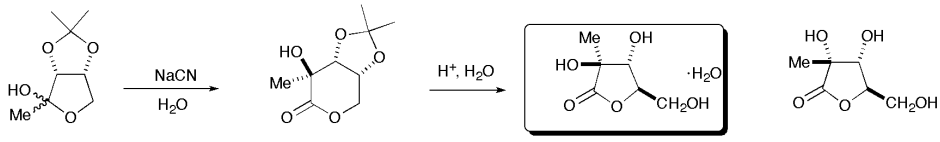
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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

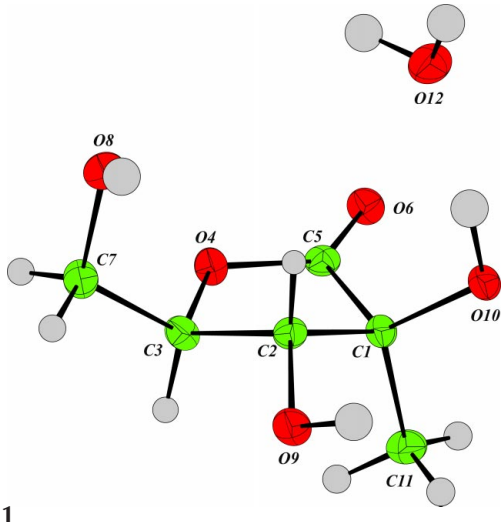


Figure 1
The asymmetric unit of (3), with displacement ellipsoids drawn at the 50% probability level.

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*.

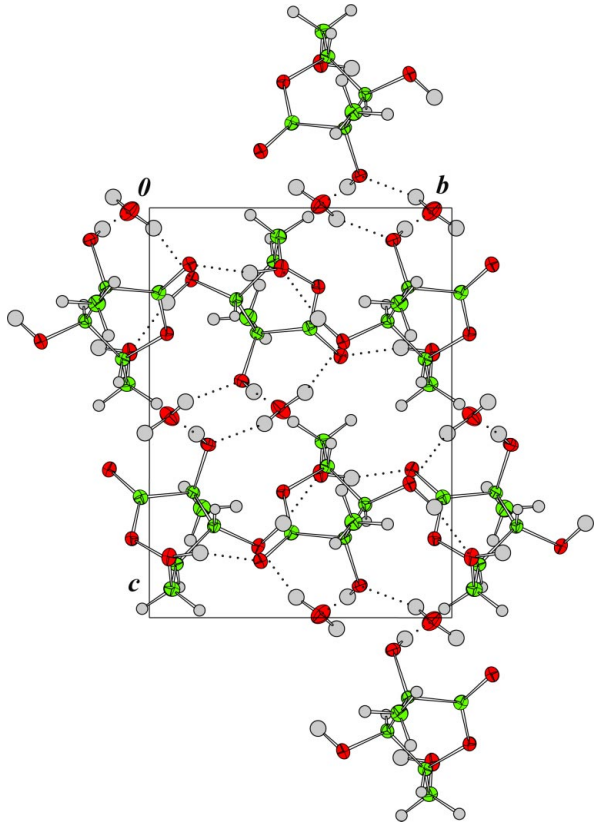


Figure 2
Packing diagram of (3), viewed down the *a* axis. Hydrogen bonds are indicated by dashed lines.

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$D_x = 1.477$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1300 reflections
$\theta = 5$–30°
$\mu = 0.13$ mm^{-1}
$T = 120$ K
Plate, colourless
0.30 × 0.20 × 0.04 mm

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Graphite monochromator
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Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)
$T_{min} = 0.97$, $T_{max} = 0.99$
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1201 reflections with $I > 2u(I)$
$R_{int} = 0.013$
$\theta_{max} = 30.0°$, $\theta_{min} = 5.3°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.071$
$S = 0.98$
1361 reflections
109 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + 0.03 + 0.17P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.000246$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Absolute structure: see text

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.77461 (19)	0.14456 (17)	0.69407 (12)	0.0165
C2	0.64570 (18)	0.21334 (16)	0.77623 (13)	0.0168
C3	0.63386 (19)	0.08861 (18)	0.86979 (13)	0.0181
O4	0.66325 (13)	−0.05809 (12)	0.80726 (9)	0.0179

C5	0.73681 (18)	−0.02955 (17)	0.70652 (13)	0.0165
O6	0.76709 (13)	−0.13251 (12)	0.63819 (9)	0.0197
C7	0.4700 (2)	0.0739 (2)	0.93035 (13)	0.0213
O8	0.33569 (13)	0.06498 (13)	0.85204 (10)	0.0220
O9	0.68542 (15)	0.35975 (12)	0.82592 (9)	0.0220
O10	0.76159 (13)	0.19458 (12)	0.57800 (8)	0.0194
C11	0.95038 (18)	0.1741 (2)	0.73335 (14)	0.0230
O12	0.47338 (14)	0.06611 (15)	0.50844 (10)	0.0282
H21	0.5406	0.2174	0.7354	0.0207*
H31	0.7252	0.1002	0.9253	0.0238*
H71	0.4532	0.1665	0.9824	0.0260*
H72	0.4727	−0.0243	0.9782	0.0260*
H111	1.0219	0.1159	0.6815	0.0286*
H112	0.9699	0.2875	0.7284	0.0286*
H113	0.9610	0.1360	0.8114	0.0286*
H5	0.6643	0.1552	0.5515	0.0500*
H7	0.2885	0.1680	0.8411	0.0500*
H9	0.6767	0.4424	0.7698	0.0500*
H12	0.4229	−0.0145	0.5497	0.0500*
H1	0.3899	0.1193	0.4735	0.0500*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0184 (7)	0.0167 (6)	0.0144 (6)	−0.0014 (6)	−0.0008 (6)	0.0019 (6)
C2	0.0178 (7)	0.0151 (6)	0.0175 (6)	−0.0019 (6)	0.0000 (6)	−0.0012 (6)
C3	0.0218 (7)	0.0172 (7)	0.0153 (7)	0.0006 (6)	−0.0013 (6)	−0.0010 (6)
O4	0.0221 (5)	0.0148 (5)	0.0169 (5)	0.0015 (4)	0.0016 (4)	0.0004 (4)
C5	0.0129 (6)	0.0182 (7)	0.0184 (7)	0.0009 (6)	−0.0036 (6)	0.0017 (5)
O6	0.0227 (5)	0.0167 (5)	0.0198 (5)	0.0013 (5)	0.0001 (5)	−0.0019 (4)
C7	0.0232 (7)	0.0222 (7)	0.0185 (7)	−0.0007 (7)	0.0014 (6)	0.0007 (7)
O8	0.0203 (5)	0.0186 (5)	0.0270 (6)	0.0006 (5)	−0.0003 (5)	−0.0015 (5)
O9	0.0304 (6)	0.0151 (5)	0.0205 (5)	−0.0022 (5)	0.0002 (5)	−0.0031 (4)
O10	0.0236 (5)	0.0196 (5)	0.0150 (5)	−0.0026 (5)	−0.0009 (4)	0.0019 (4)
C11	0.0190 (7)	0.0258 (8)	0.0242 (8)	−0.0028 (7)	−0.0033 (6)	0.0003 (7)
O12	0.0221 (5)	0.0318 (6)	0.0307 (6)	−0.0026 (6)	−0.0065 (5)	0.0094 (6)

Geometric parameters (Å, º)

C1—C2	1.537 (2)	C7—O8	1.4261 (19)
C1—C5	1.528 (2)	C7—H71	1.006
C1—O10	1.4169 (17)	C7—H72	1.007
C1—C11	1.526 (2)	O8—H7	0.970
C2—C3	1.525 (2)	O9—H9	0.964
C2—O9	1.4167 (18)	O10—H5	0.916
C2—H21	0.980	C11—H111	0.975
C3—O4	1.4695 (18)	C11—H112	0.985
C3—C7	1.516 (2)	C11—H113	0.967

C3—H31	0.990	O12—H12	0.935
O4—C5	1.3363 (18)	O12—H1	0.914
C5—O6	1.2106 (17)		

C2—C1—C5	100.17 (12)	C1—C5—O4	110.58 (12)
C2—C1—O10	114.98 (12)	C1—C5—O6	127.33 (14)
C5—C1—O10	111.66 (12)	O4—C5—O6	122.09 (13)
C2—C1—C11	113.27 (12)	C3—C7—O8	112.81 (12)
C5—C1—C11	108.84 (13)	C3—C7—H71	109.5
O10—C1—C11	107.71 (12)	O8—C7—H71	108.6
C1—C2—C3	102.51 (12)	C3—C7—H72	107.8
C1—C2—O9	115.74 (12)	O8—C7—H72	108.8
C3—C2—O9	110.11 (12)	H71—C7—H72	109.3
C1—C2—H21	108.3	C7—O8—H7	109.9
C3—C2—H21	108.2	C2—O9—H9	110.9
O9—C2—H21	111.4	C1—O10—H5	105.8
C2—C3—O4	103.66 (11)	C1—C11—H111	107.1
C2—C3—C7	116.35 (13)	C1—C11—H112	107.3
O4—C3—C7	107.56 (12)	H111—C11—H112	111.8
C2—C3—H31	110.2	C1—C11—H113	107.9
O4—C3—H31	106.5	H111—C11—H113	110.6
C7—C3—H31	111.8	H112—C11—H113	111.9
C3—O4—C5	110.42 (11)	H12—O12—H1	105.4

Hydrogen-bond geometry (Å, º)

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
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