Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,3-Di-*C*-methyl-D-allono-1,4-lactone

Nigel A. Jones,[a]* Sarah F. Jenkinson,[a] Louise A. Curran,[a] David J. Watkin[b] and George W. J. Fleet[a]

[a]Department of Organic Chemistry, Chemistry Research Laboratory, University of Oxford, 12 Mansfield Road, Oxford OX1 3TA, England, and [b]Department of Chemical Crystallography, Chemistry Research Laboratory, University of Oxford, 12 Mansfield Road, Oxford OX1 3TA, England
Correspondence e-mail: nigel.jones@chem.ox.ac.uk

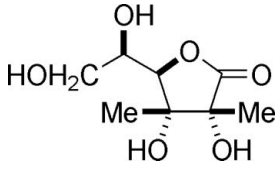
Received 20 June 2007; accepted 22 June 2007

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.024; wR factor = 0.059; data-to-parameter ratio = 9.3.

The relative configuration of the title compound, $C_8H_{14}O_6$, was firmly established by X-ray crystallographic analysis. The absolute configuration was determined by the use of 2-*C*-methyl-D-ribono-1,4-lactone as the starting material. The crystal structure is stabilized by O—H···O hydrogen bonds, with each molecule acting as a donor and an acceptor for five hydrogen bonds.

Related literature

For related literature, see: Booth *et al.* (2007, 2007*a*, 2007*b*, 2007*c*); Görbitz (1999); Jones, Curran *et al.* (2007); Jones, Watkin *et al.* (2007); Lichtenthaler & Peters (2004); Mitchell *et al.* (2007); Hotchkiss *et al.* (2006, 2004); Soengas *et al.* (2005).



Experimental

Crystal data

$C_8H_{14}O_6$
M_r = 206.20
Monoclinic, $P2_1$
a = 6.1387 (2) Å
b = 7.4088 (3) Å
c = 10.8142 (3) Å
β = 94.996 (2)°
V = 489.97 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 150 K
0.30 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.81, T_{max} = 0.99
4427 measured reflections
1185 independent reflections
1151 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.059
S = 0.98
1185 reflections
127 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H4···O8^{i}	0.87	1.81	2.683 (2)	178
O13—H10···O9^{ii}	0.88	1.81	2.675 (2)	169
O8—H11···O13^{iii}	0.83	2.05	2.762 (2)	144
O11—H14···O10^{iv}	0.85	1.90	2.729 (2)	163
O8—H11···O11^{iii}	0.83	2.27	2.954 (2)	140

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

Data collection: *COLLECT* (Nonius, 2001); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2443).

References

Altomare, A., Cascarano, G., Giacovazzo, G., 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, G. W. J. & Watkin, D. J. (2007*a*). *Acta Cryst.* E**63**, o2424–o2426.
Booth, K. V., Jenkinson, S. F., Fleet, G. W. J. & Watkin, D. J. (2007*b*). *Acta Cryst.* E**63**, o2427–o2429.
Booth, K. V., Jenkinson, S. F., Fleet, G. W. J. & Watkin, D. J. (2007*c*). *Acta Cryst.* E**63**, o2204–o2206.
Booth, K. V., Watkin, D. J., Jenkinson, S. F. & Fleet, G. W. J. (2007). *Acta Cryst.* E**63**, o1128–o1130.
Görbitz, C. H. (1999). *Acta Cryst.* B**55**, 1090–1098.
Hotchkiss, D. J., Jenkinson, S. F., Storer, R., Heinz, T. & Fleet, G. W. J. (2006). *Tetrahedron Lett.* **47**, 315–318.
Hotchkiss, D. J., Soengas, R., Simone, M. I., van Ameijde, J., Hunter, S., Cowley, A. R. & Fleet, G. W. J. (2004). *Tetrahedron Lett.* **45**, 9461–9464.
Jones, N. A., Curran, L. A., Wormald, M. R., Dwek, R. A. & Fleet, G. W. J. (2007). In preparation.
Jones, N. A., Watkin, D. J., Curran, L. A., Jenkinson, S. F. & Fleet, G. W. J. (2007). *Acta Cryst.* E**63**, o992–o994.
Lichtenthaler, F. W. & Peters, S. (2004). *C. R. Chim.* **7**, 65–90.
Mitchell, D. A., Jones, N. A., Hunter, S. J., Cook, J. M. D., Jenkinson, S. F., Wormald, M. R., Dwek, R. A. & Fleet, G. W. J. (2007). *Tetrahedron Asymmetry*, **18**. In the press.
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.
Soengas, R., Izumori, K., Simone, M. I., Watkin, D. J., Skytte, U. P., Soetaert, W. & Fleet, G. W. J. (2005). *Tetrahedron Lett.* **46**, 5755–5759.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.

 doi:10.1107/S1600536807030541

supplementary materials

Acta Cryst. (2007). E**63**, o3415 [**doi:10.1107/S1600536807030541**]

2,3-Di-*C*-methyl-D-allono-1,4-lactone

N. A. Jones, S. F. Jenkinson, L. A. Curran, D. J. Watkin and G. W. J. Fleet

Comment

Carbohydrates are one of the most varied cheap chiral building blocks (Lichtenthaler & Peters, 2004) available. Currently, there are very limited examples of di-branched carbohydrates reported. Examples of sugars that contain two carbon branches in the literature include 2,4-dimethyl-3,4-*O*-isopropylidene-*L*-arabinono-1,5-lactone (Booth *et al.* 2007*a*) and various protected forms of 3,5-Di-*C*-methyl-mannono and glucono lactone (Booth, Watkin *et al.*, 2007; Booth *et al.*, 2007*a*,*b*,*c*).

The Kiliani ascension on ketoses and deoxy ketoses has proved a valuable route towards branched sugars (Hotchkiss *et al.* 2004;2006, Soengas *et al.*, 2005, Jones, Watkin *et al.* 2007). Branched sugars still remains a relatively unstudied class of compounds but recent biological results have indicated they could have a potential use as therapeutics (Mitchell *et al.* 2007).

The crystal structure of the title compound (Fig. 1) exists as a three-dimensionally hydrogen bonded lattice with each molecule acting as a donor and an acceptor for 5 hydrogen bonds. One of the hydrogen bonds, from O8—H11, is bifurcated (Fig. 2).

Experimental

Treatment of 1-deoxy-3-*C*-methyl-D-psicose **1** (see Fig. 3) (Jones, Curran *et al.* 2007) derived from 2-*C*-methyl-D-ribono-1,4-lactone (Hotchkiss *et al.*, 2006), with sodium cyanide, gave a mixture of 2,6-anhydro derivative **2** and lactone **3** Deprotection of lactone **3** gave an equilibrium of 1,5-lactone **4** and 1,4-lactone **5** in which 2,3-*C*-dimethyl-D-allono-lactone exists as 2,3-*C*-dimethyl-D-allono-1,4-lactone **5** in the crystalline state. X-Ray crystallographic analysis shows that the structure is the title compound **5** removing ambiguities as to the stereochemistry at the new C-2 chiral centre. The title compound was crystallized from a mixture of cyclohexane and ethyl acetate; m.p. 426–428 K; $[\alpha]_D^{22}$ +66.8 (*c*, 0.65 in methanol).

Refinement

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

The relatively large ratio of minimum to maximum corrections applied in the multiscan process (1:1.22) reflect 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*/*SCALEPACK*, Otwinowski & Minor, 1997).

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

Figures

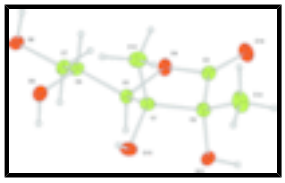


Fig. 1. The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

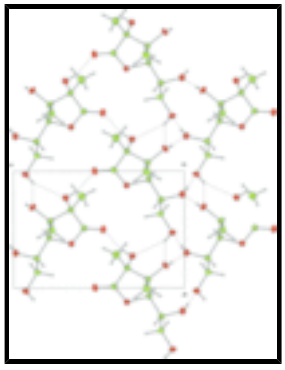


Fig. 2. Packing diagram showing the title compound projected along the *a*-axis.

Fig. 3. The reaction scheme.

2,3-*C*-Dimethyl-D-allono-1,4-lactone

Crystal data

$C_8H_{14}O_6$	F_{000} = 220
M_r = 206.20	D_x = 1.398 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 1103 reflections
a = 6.1387 (2) Å	θ = 5–27º
b = 7.4088 (3) Å	μ = 0.12 mm^{-1}
c = 10.8142 (3) Å	*T* = 150 K
β = 94.996 (2)º	Plate, colourless
V = 489.97 (3) Å^3	0.30 × 0.20 × 0.10 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	1151 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 150 K	θ_{max} = 27.5º
ω scans	θ_{min} = 5.5º
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)	*h* = −7→7
T_{min} = 0.81, T_{max} = 0.99	*k* = −9→8

4427 measured reflections
1185 independent reflections

$l = -14 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.059$

$S = 0.98$

1185 reflections

127 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F^2) + 0.11P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$

$(\Delta/\sigma)_{max} = 0.0003$

$\Delta\rho_{max} = 0.22$ e Å^{-3}

$\Delta\rho_{min} = -0.15$ e Å^{-3}

Extinction correction: None

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8416 (2)	0.4263 (2)	0.78654 (13)	0.0150
C2	0.7563 (2)	0.3296 (2)	0.66461 (13)	0.0162
C3	0.6629 (2)	0.4883 (2)	0.58761 (13)	0.0167
O4	0.60598 (17)	0.62177 (17)	0.66215 (9)	0.0191
C5	0.6621 (2)	0.5717 (2)	0.79221 (12)	0.0142
C6	0.7150 (2)	0.7452 (2)	0.86458 (13)	0.0167
C7	0.5125 (2)	0.8654 (2)	0.86070 (13)	0.0183
O8	0.55256 (18)	1.02775 (17)	0.92964 (10)	0.0217
O9	0.79185 (17)	0.69964 (18)	0.98862 (9)	0.0202
O10	0.63373 (19)	0.49872 (19)	0.47585 (9)	0.0248
O11	0.57031 (17)	0.22808 (17)	0.69455 (9)	0.0201
C12	0.9154 (3)	0.2152 (3)	0.60015 (15)	0.0271
O13	0.83413 (16)	0.30793 (17)	0.88832 (9)	0.0184
C14	1.0685 (2)	0.5052 (2)	0.77945 (14)	0.0213
H51	0.5313	0.5108	0.8209	0.0186*
H61	0.8324	0.8105	0.8255	0.0186*
H71	0.4019	0.7962	0.9000	0.0210*
H72	0.4647	0.8908	0.7734	0.0213*
H121	0.8351	0.1608	0.5271	0.0417*
H122	0.9737	0.1226	0.6570	0.0412*
H123	1.0318	0.2899	0.5717	0.0413*
H141	1.1107	0.5686	0.8566	0.0333*
H142	1.1713	0.4101	0.7692	0.0338*
H143	1.0615	0.5863	0.7091	0.0340*
H4	0.6813	0.6440	1.0171	0.0323*
H10	0.9644	0.2852	0.9245	0.0307*

H11	0.6169	1.0978	0.8849	0.0359*
H14	0.5063	0.1748	0.6320	0.0313*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0152 (6)	0.0146 (7)	0.0149 (6)	0.0006 (6)	0.0000 (5)	0.0030 (5)
C2	0.0178 (7)	0.0149 (7)	0.0159 (6)	0.0015 (6)	0.0015 (5)	0.0011 (5)
C3	0.0181 (7)	0.0155 (7)	0.0163 (6)	−0.0009 (6)	−0.0004 (5)	0.0002 (6)
O4	0.0258 (5)	0.0159 (5)	0.0145 (5)	0.0043 (4)	−0.0041 (4)	0.0000 (4)
C5	0.0155 (6)	0.0141 (7)	0.0124 (6)	0.0003 (5)	−0.0023 (5)	0.0007 (5)
C6	0.0180 (6)	0.0155 (7)	0.0163 (6)	−0.0042 (6)	0.0004 (5)	−0.0007 (6)
C7	0.0238 (7)	0.0128 (7)	0.0181 (6)	−0.0004 (6)	0.0012 (5)	−0.0017 (6)
O8	0.0309 (6)	0.0132 (5)	0.0220 (5)	−0.0040 (5)	0.0076 (4)	−0.0024 (4)
O9	0.0211 (5)	0.0222 (6)	0.0162 (5)	−0.0057 (5)	−0.0046 (4)	−0.0002 (4)
O10	0.0349 (6)	0.0247 (6)	0.0141 (5)	0.0031 (5)	−0.0022 (4)	0.0021 (5)
O11	0.0243 (5)	0.0180 (5)	0.0176 (5)	−0.0074 (5)	−0.0006 (4)	−0.0012 (4)
C12	0.0317 (8)	0.0262 (9)	0.0238 (7)	0.0093 (7)	0.0051 (6)	−0.0030 (7)
O13	0.0171 (5)	0.0197 (5)	0.0177 (5)	−0.0014 (4)	−0.0030 (4)	0.0074 (4)
C14	0.0149 (7)	0.0248 (8)	0.0240 (7)	−0.0026 (6)	0.0003 (5)	0.0029 (7)

Geometric parameters (Å, °)

C1—C2	1.5509 (19)	C6—H61	0.992
C1—C5	1.547 (2)	C7—O8	1.4253 (17)
C1—O13	1.4110 (17)	C7—H71	0.977
C1—C14	1.518 (2)	C7—H72	0.982
C2—C3	1.523 (2)	O8—H11	0.832
C2—O11	1.4277 (17)	O9—H4	0.873
C2—C12	1.509 (2)	O11—H14	0.850
C3—O4	1.3413 (18)	C12—H121	0.981
C3—O10	1.2087 (17)	C12—H122	0.970
O4—C5	1.4663 (16)	C12—H123	0.974
C5—C6	1.5249 (19)	O13—H10	0.876
C5—H51	0.994	C14—H141	0.972
C6—C7	1.527 (2)	C14—H142	0.958
C6—O9	1.4232 (17)	C14—H143	0.968
C2—C1—C5	99.57 (11)	C5—C6—H61	108.8
C2—C1—O13	110.17 (12)	C7—C6—H61	108.9
C5—C1—O13	109.28 (11)	O9—C6—H61	108.9
C2—C1—C14	112.23 (12)	C6—C7—O8	112.03 (12)
C5—C1—C14	113.17 (13)	C6—C7—H71	106.1
O13—C1—C14	111.78 (11)	O8—C7—H71	108.0
C1—C2—C3	101.06 (12)	C6—C7—H72	108.2
C1—C2—O11	105.55 (10)	O8—C7—H72	111.4
C3—C2—O11	105.10 (11)	H71—C7—H72	111.0
C1—C2—C12	117.88 (13)	C7—O8—H11	106.9
C3—C2—C12	114.12 (12)	C6—O9—H4	103.9

O11—C2—C12	111.80 (14)	C2—O11—H14	112.8
C2—C3—O4	110.23 (11)	C2—C12—H121	107.6
C2—C3—O10	127.72 (14)	C2—C12—H122	108.8
O4—C3—O10	122.01 (14)	H121—C12—H122	110.6
C3—O4—C5	109.77 (11)	C2—C12—H123	110.4
C1—C5—O4	104.29 (10)	H121—C12—H123	108.1
C1—C5—C6	119.14 (12)	H122—C12—H123	111.2
O4—C5—C6	107.52 (11)	C1—O13—H10	112.2
C1—C5—H51	107.1	C1—C14—H141	108.7
O4—C5—H51	106.6	C1—C14—H142	109.8
C6—C5—H51	111.4	H141—C14—H142	108.9
C5—C6—C7	109.92 (11)	C1—C14—H143	107.5
C5—C6—O9	108.80 (11)	H141—C14—H143	111.4
C7—C6—O9	111.49 (11)	H142—C14—H143	110.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9—H4···O8^{i}	0.87	1.81	2.683 (2)	178
O13—H10···O9^{ii}	0.88	1.81	2.675 (2)	169
O8—H11···O13^{iii}	0.83	2.05	2.762 (2)	144
O11—H14···O10^{iv}	0.85	1.90	2.729 (2)	163
O8—H11···O11^{iii}	0.83	2.27	2.954 (2)	140

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+2; (ii) −*x*+2, *y*−1/2, −*z*+2; (iii) *x*, *y*+1, *z*; (iv) −*x*+1, *y*−1/2, −*z*+1.

Fig. 1

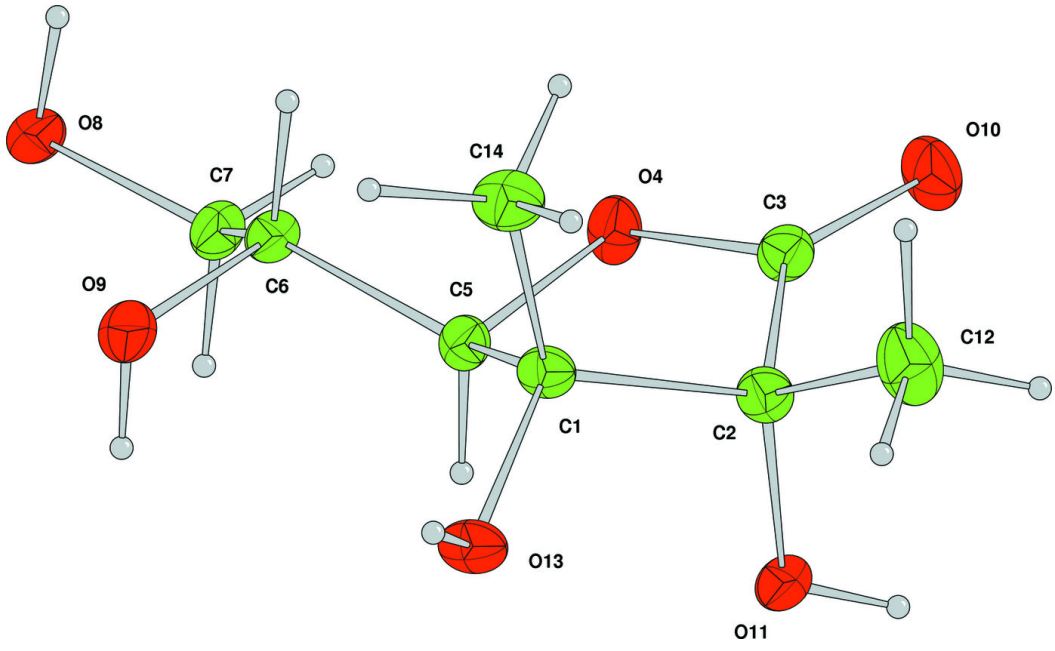


Fig. 2

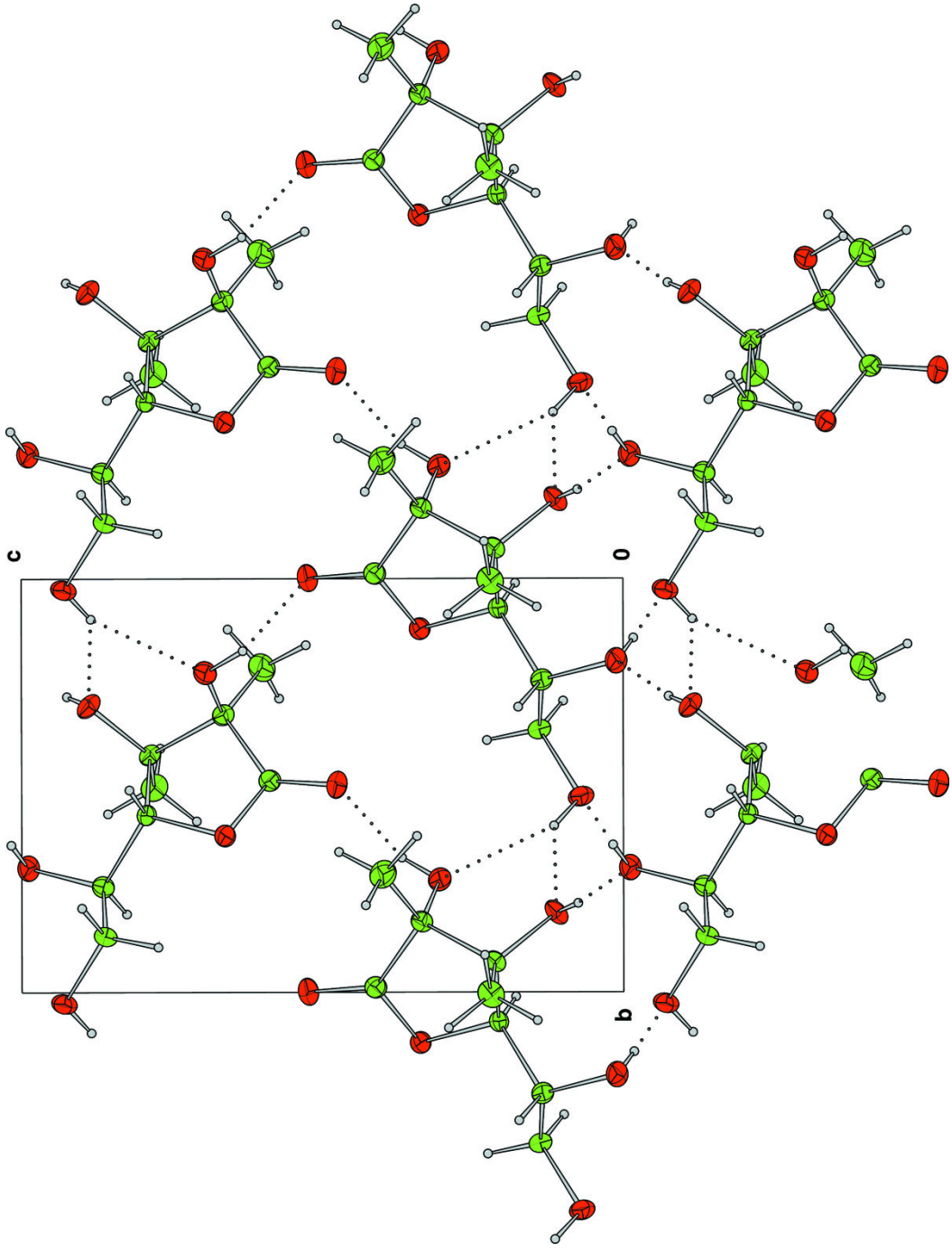
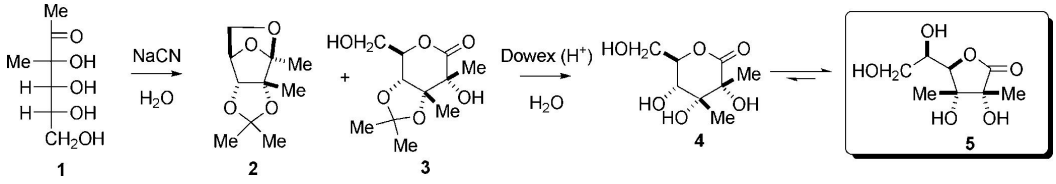


Fig. 3


Me
O
Me
OH
H
OH
H
OH
CH2OH
1
NaCN
H2O
O
O
Me
Me
O
O
Me
Me
2
HOH2C
O
O
Me
+
O
OH
Me
O
Me
Me
3
Dowex (H+)
H2O
HOH2C
O
O
Me
HO
HO
OH
Me
4
OH
HOH2C
O
O
Me
Me
HO
OH
5