

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 6-Deoxy-3,4-O-isopropylidene-2-C-methyl-L-galactono-1,5-lactone

Sarah F. Jenkinson,<sup>a\*</sup> Loren L. Parry,<sup>a</sup> Francis X. Wilson,<sup>b</sup> George W. J. Fleet<sup>a</sup> and David J. Watkin<sup>c</sup>

<sup>a</sup>Department of Organic Chemistry, Chemistry Research Laboratory, University of Oxford, Oxford OX1 3TA, England, <sup>b</sup>Summit PLC, 91 Milton Park, Abingdon, Oxfordshire OX14 4RY, England, and <sup>c</sup>Department of Chemical Crystallography, Chemistry Research Laboratory, University of Oxford, Oxford OX1 3TA, England  
Correspondence e-mail: sarah.jenkinson@chem.ox.ac.uk

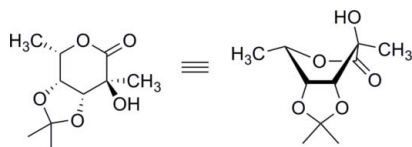
Received 18 August 2011; accepted 25 August 2011

Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.038;  $wR$  factor = 0.090; data-to-parameter ratio = 10.7.

X-ray crystallography unequivocally confirmed the stereochemistry of the 2-*C*-methyl group in the title molecule,  $\text{C}_{10}\text{H}_{16}\text{O}_5$ , in which the 1,5-lactone ring exists in a boat conformation. The absolute stereochemistry was determined by the use of *D*-ribose in the synthesis. The crystal exists as  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonded chains of molecules running parallel to the *a* axis with each molecule acting as a donor and acceptor for one hydrogen bond.

## Related literature

For branched iminosugars, see: Håkansson *et al.* (2007, 2008); Asano *et al.* (2000); da Cruz *et al.* (2011); Best *et al.* (2010) and for branched sugars, see: Booth *et al.* (2008, 2009); da Cruz *et al.* (2008); Hotchkiss *et al.* (2006, 2007); Jenkinson *et al.* (2007); Jones *et al.* (2007, 2008); Rao *et al.* (2008). For conformations of related 1,5-lactones, see: Baird *et al.* (1987); Booth *et al.* (2007*a,b*); Bruce *et al.* (1990); Punzo *et al.* (2005, 2006); Dai *et al.* (2010).



## Experimental

## Crystal data

 $\text{C}_{10}\text{H}_{16}\text{O}_5$  $M_r = 216.23$ Orthorhombic,  $P2_12_12_1$  $a = 6.1132$  (2) Å $b = 12.2963$  (4) Å $c = 14.6367$  (5) Å $V = 1100.24$  (6) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.11$  mm<sup>-1</sup> $T = 150$  K $0.20 \times 0.20 \times 0.04$  mm

## Data collection

Nonius KappaCCD diffractometer

Absorption correction: multi-scan

(DENZO/SCALEPACK;

Otwinowski &amp; Minor, 1997)

 $T_{\min} = 0.95$ ,  $T_{\max} = 1.00$ 

8628 measured reflections

1454 independent reflections

1131 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.067$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.038$  $wR(F^2) = 0.090$  $S = 0.89$ 

1454 reflections

136 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.40$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.29$  e Å<sup>-3</sup>

## Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O11}-\text{H111}\cdots\text{O1}^i$	0.86	1.93	2.793 (3)	172

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

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: LH5321).

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**supplementary materials**

*Acta Cryst.* (2011). E67, o2531-o2532 [ doi:10.1107/S1600536811034957 ]

## 6-Deoxy-3,4-*O*-isopropylidene-2-*C*-methyl-*L*-galactono-1,5-lactone

S. F. Jenkinson, L. L. Parry, F. X. Wilson, G. W. J. Fleet and D. J. Watkin

### Comment

2-*C*-Methyl branched sugars, as well as being chirons for the enantiospecific synthesis of complex targets (Hotchkiss *et al.*, 2006; Hotchkiss *et al.*, 2007; da Cruz *et al.*, 2008; Booth *et al.*, 2009) including 2'-*C*-methyl nucleosides (Jenkinson *et al.*, 2007), are a class of rare sugars with chemotherapeutic potential (Rao *et al.*, 2008; Jones *et al.*, 2008; Booth *et al.*, 2008). Branched iminosugars have also been shown to exhibit interesting biological activity. For example: 6-*C*-methyl-swainsonine is a more potent inhibitor of *L*-rhamnosidase than *L*-swainsonine (Håkansson *et al.*, 2007; Håkansson *et al.*, 2008; Asano *et al.*, 2000); 4-*C*-methylDAB and 4-*C*-methylLAB are both potent and specific  $\alpha$ -glucosidase inhibitors (da Cruz *et al.* 2011) and isoLAB has been shown to partially rescue the defective F508del-CFTR function and therefore may have a role in the study of cystic fibrosis (Best *et al.*, 2010).

D-ribose **1** was converted by a number of steps to the lactols **2** (Fig. 1). The reaction of **2** with sodium cyanide in water gave a chain extension to afford a single isolated crystalline product **3** (Fig. 2).

3,4-*O*-Isopropylidene-1,5-lactones, such as **3**, invariably crystallize in boat conformations (Baird *et al.*, 1987; Bruce *et al.*, 1990; Punzo *et al.*, 2005). The diastereoselectivity of the reaction may be rationalized by the formation of the lactone **3** with less steric congestion (Punzo *et al.*, 2006; Booth *et al.*, 2007*a*; Booth *et al.*, 2007*b*; Dai *et al.* 2010) with the smaller hydroxy group rather than the methyl group in the flagpole position (Fig. 2). The structure of **3** was confirmed by the X-ray crystallographic analysis. The absolute configuration was assigned from the use of D-ribose as the starting material. The title compound exists as O—H $\cdots$ O hydrogen bonded chains of molecules running parallel to the *a*-axis (Fig. 3). Each molecule acts as a donor and acceptor for 1 hydrogen bond. Only classical hydrogen bonding has been considered.

### Experimental

The title compound was recrystallized by diffusion from a mixture of ethyl acetate and cyclohexane: m.p. 369–371 K;  $[\alpha]_D^{25}$  -84.6 (*c*, 1.03 in CHCl<sub>3</sub>).

### Refinement

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

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  $U_{\text{iso}}(\text{H})$  (in the range 1.2–1.5 times  $U_{\text{eq}}$  of the parent atom), after which the positions were refined with riding constraints.

## Figures

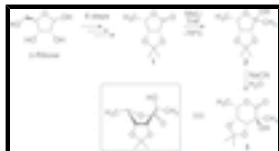


Fig. 1. Synthetic Scheme.

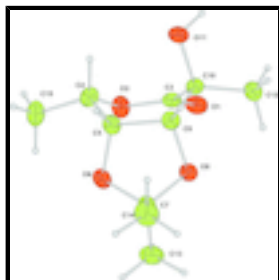


Fig. 2. The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

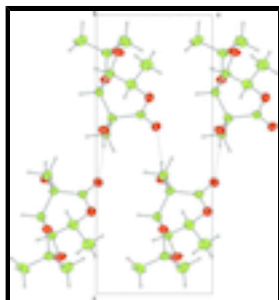


Fig. 3. Packing diagram of the title compound projected along the *b*-axis. Hydrogen bonds are shown by dotted lines.

## 6-Deoxy-3,4-*O*-isopropylidene-2-*C*-methyl-L-galactono-1,5-lactone

### Crystal data

$C_{10}H_{16}O_5$

$M_r = 216.23$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.1132$  (2) Å

$b = 12.2963$  (4) Å

$c = 14.6367$  (5) Å

$V = 1100.24$  (6) Å<sup>3</sup>

$Z = 4$

$F(000) = 464$

$D_x = 1.305$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1433 reflections

$\theta = 5\text{--}27^\circ$

$\mu = 0.11$  mm<sup>-1</sup>

$T = 150$  K

Plate, colourless

$0.20 \times 0.20 \times 0.04$  mm

### Data collection

Nonius KappaCCD  
diffractometer

graphite

$\omega$  scans

Absorption correction: multi-scan

(*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)

$T_{\min} = 0.95$ ,  $T_{\max} = 1.00$

1131 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.067$

$\theta_{\text{max}} = 27.5^\circ$ ,  $\theta_{\text{min}} = 5.2^\circ$

$h = -7 \rightarrow 7$

$k = -15 \rightarrow 15$

8628 measured reflections  
1454 independent reflections

$l = -18 \rightarrow 19$

*Refinement*

Refinement on  $F^2$

Primary atom site location: structure-invariant direct methods

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.038$

H-atom parameters constrained

$wR(F^2) = 0.090$

Method = Modified Sheldrick  $w = 1/[\sigma^2(F^2) + (0.06P)^2 + 0.14P]$ ,

where  $P = [\max(F_o^2, 0) + 2F_c^2]/3$

$S = 0.89$

$(\Delta/\sigma)_{\max} = 0.000259$

1454 reflections

$\Delta\rho_{\max} = 0.40 \text{ e } \text{\AA}^{-3}$

136 parameters

$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

0 restraints

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.0165 (3)	0.80556 (12)	0.89995 (10)	0.0290
C2	0.1164 (4)	0.75275 (16)	0.85712 (12)	0.0226
O3	0.0426 (3)	0.68163 (11)	0.79561 (10)	0.0287
C4	0.1986 (4)	0.60986 (16)	0.74843 (14)	0.0289
C5	0.3861 (4)	0.67578 (16)	0.71001 (14)	0.0271
O6	0.3108 (3)	0.73556 (12)	0.63264 (9)	0.0355
C7	0.4068 (4)	0.84132 (17)	0.63637 (13)	0.0292
O8	0.4222 (3)	0.86365 (10)	0.73198 (9)	0.0261
C9	0.4744 (4)	0.76299 (15)	0.77626 (13)	0.0240
C10	0.3643 (4)	0.76166 (16)	0.86972 (13)	0.0221
O11	0.4337 (3)	0.66259 (11)	0.91228 (10)	0.0301
C12	0.4222 (4)	0.86099 (16)	0.92581 (13)	0.0277
C13	0.2506 (5)	0.92177 (19)	0.59465 (16)	0.0401
C14	0.6306 (5)	0.8415 (2)	0.59246 (16)	0.0422
C15	0.0664 (5)	0.55020 (19)	0.67809 (16)	0.0402
H41	0.2554	0.5572	0.7948	0.0338*
H51	0.5068	0.6260	0.6926	0.0337*
H91	0.6348	0.7543	0.7832	0.0290*
H121	0.5803	0.8627	0.9363	0.0432*
H122	0.3490	0.8570	0.9864	0.0426*
H123	0.3742	0.9272	0.8933	0.0431*
H131	0.3082	0.9961	0.6014	0.0583*
H132	0.2349	0.9048	0.5289	0.0582*
H133	0.1099	0.9171	0.6262	0.0586*
H141	0.6868	0.9161	0.5934	0.0627*
H142	0.7280	0.7929	0.6268	0.0622*
H143	0.6166	0.8179	0.5275	0.0623*

## supplementary materials

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H153	0.1616	0.4999	0.6465	0.0610*
H152	-0.0508	0.5092	0.7077	0.0613*
H151	0.0062	0.6022	0.6340	0.0612*
H111	0.4361	0.6716	0.9708	0.0460*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0284 (9)	0.0329 (8)	0.0257 (7)	0.0061 (7)	0.0025 (7)	0.0035 (7)
C2	0.0264 (11)	0.0224 (10)	0.0191 (8)	0.0006 (10)	0.0006 (9)	0.0049 (9)
O3	0.0263 (8)	0.0302 (8)	0.0295 (8)	-0.0029 (7)	0.0011 (6)	-0.0051 (6)
C4	0.0344 (13)	0.0224 (10)	0.0299 (11)	0.0027 (9)	0.0008 (10)	-0.0023 (9)
C5	0.0315 (12)	0.0276 (10)	0.0222 (9)	0.0034 (10)	0.0012 (10)	-0.0035 (9)
O6	0.0500 (11)	0.0332 (8)	0.0234 (7)	-0.0145 (8)	-0.0068 (7)	0.0026 (7)
C7	0.0354 (13)	0.0305 (12)	0.0216 (10)	-0.0092 (11)	-0.0011 (10)	-0.0005 (9)
O8	0.0326 (8)	0.0250 (7)	0.0208 (7)	-0.0002 (7)	0.0006 (6)	0.0007 (6)
C9	0.0220 (11)	0.0253 (10)	0.0248 (9)	0.0032 (9)	0.0000 (9)	-0.0009 (9)
C10	0.0246 (11)	0.0198 (10)	0.0220 (9)	0.0042 (9)	-0.0017 (9)	0.0009 (8)
O11	0.0405 (9)	0.0267 (7)	0.0233 (7)	0.0089 (7)	-0.0047 (7)	0.0023 (6)
C12	0.0300 (12)	0.0288 (11)	0.0243 (10)	0.0005 (10)	-0.0028 (10)	-0.0022 (9)
C13	0.0453 (16)	0.0394 (13)	0.0355 (12)	-0.0026 (13)	-0.0080 (13)	0.0110 (12)
C14	0.0470 (16)	0.0480 (14)	0.0316 (11)	-0.0057 (14)	0.0137 (12)	-0.0004 (12)
C15	0.0509 (17)	0.0330 (12)	0.0368 (12)	-0.0104 (12)	-0.0011 (13)	-0.0088 (10)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

O1—C2	1.214 (2)	C9—H91	0.992
C2—O3	1.334 (2)	C10—O11	1.433 (2)
C2—C10	1.530 (3)	C10—C12	1.514 (3)
O3—C4	1.472 (3)	O11—H111	0.864
C4—C5	1.512 (3)	C12—H121	0.979
C4—C15	1.501 (3)	C12—H122	0.995
C4—H41	1.000	C12—H123	0.987
C5—O6	1.426 (2)	C13—H131	0.985
C5—C9	1.543 (3)	C13—H132	0.989
C5—H51	0.992	C13—H133	0.978
O6—C7	1.428 (3)	C14—H141	0.979
C7—O8	1.429 (2)	C14—H142	0.982
C7—C13	1.505 (3)	C14—H143	0.997
C7—C14	1.512 (3)	C15—H153	0.967
O8—C9	1.433 (2)	C15—H152	0.977
C9—C10	1.525 (3)	C15—H151	0.980
O1—C2—O3	118.2 (2)	C2—C10—O11	106.55 (17)
O1—C2—C10	124.18 (18)	C9—C10—O11	105.58 (16)
O3—C2—C10	117.60 (18)	C2—C10—C12	110.78 (18)
C2—O3—C4	119.38 (17)	C9—C10—C12	112.01 (17)
O3—C4—C5	110.13 (15)	O11—C10—C12	112.40 (15)
O3—C4—C15	105.41 (19)	C10—O11—H111	109.1

C5—C4—C15	114.54 (18)	C10—C12—H121	109.5
O3—C4—H41	107.1	C10—C12—H122	109.8
C5—C4—H41	109.7	H121—C12—H122	107.8
C15—C4—H41	109.6	C10—C12—H123	109.5
C4—C5—O6	109.09 (18)	H121—C12—H123	110.5
C4—C5—C9	113.83 (17)	H122—C12—H123	109.7
O6—C5—C9	104.69 (15)	C7—C13—H131	110.0
C4—C5—H51	109.2	C7—C13—H132	108.5
O6—C5—H51	110.7	H131—C13—H132	109.2
C9—C5—H51	109.3	C7—C13—H133	109.2
C5—O6—C7	107.85 (16)	H131—C13—H133	108.7
O6—C7—O8	103.85 (15)	H132—C13—H133	111.2
O6—C7—C13	108.81 (18)	C7—C14—H141	108.2
O8—C7—C13	108.23 (18)	C7—C14—H142	109.3
O6—C7—C14	110.92 (18)	H141—C14—H142	110.5
O8—C7—C14	110.89 (19)	C7—C14—H143	109.1
C13—C7—C14	113.65 (18)	H141—C14—H143	108.4
C7—O8—C9	106.95 (14)	H142—C14—H143	111.3
C5—C9—O8	103.77 (15)	C4—C15—H153	108.4
C5—C9—C10	113.70 (17)	C4—C15—H152	110.1
O8—C9—C10	108.47 (15)	H153—C15—H152	108.9
C5—C9—H91	109.6	C4—C15—H151	109.6
O8—C9—H91	111.1	H153—C15—H151	109.1
C10—C9—H91	110.1	H152—C15—H151	110.7
C2—C10—C9	109.25 (16)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C13—H131 $\cdots$ O11 <sup>i</sup>	0.99	2.59	3.536 (3)	161
O11—H111 $\cdots$ O1 <sup>ii</sup>	0.86	1.93	2.793 (3)	172

Symmetry codes: (i)  $-x+1, y+1/2, -z+3/2$ ; (ii)  $x+1/2, -y+3/2, -z+2$ .



Fig. 1

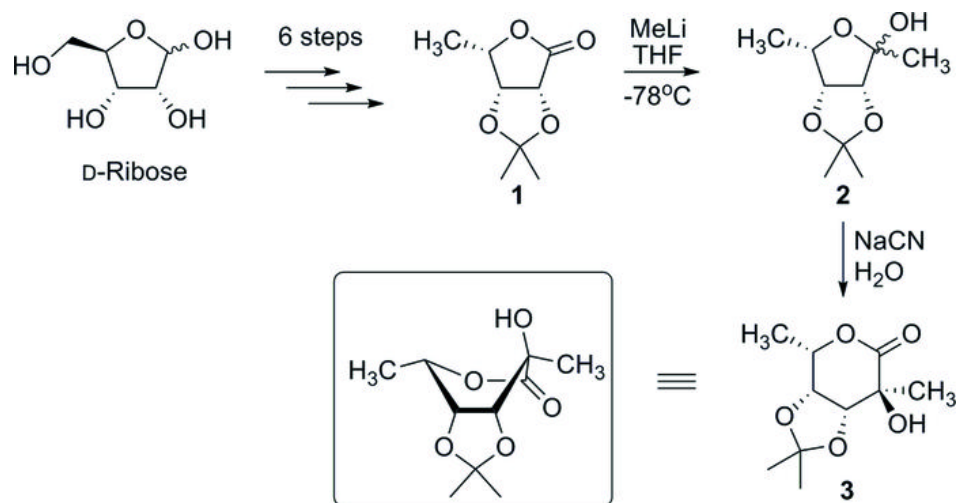


Fig. 2

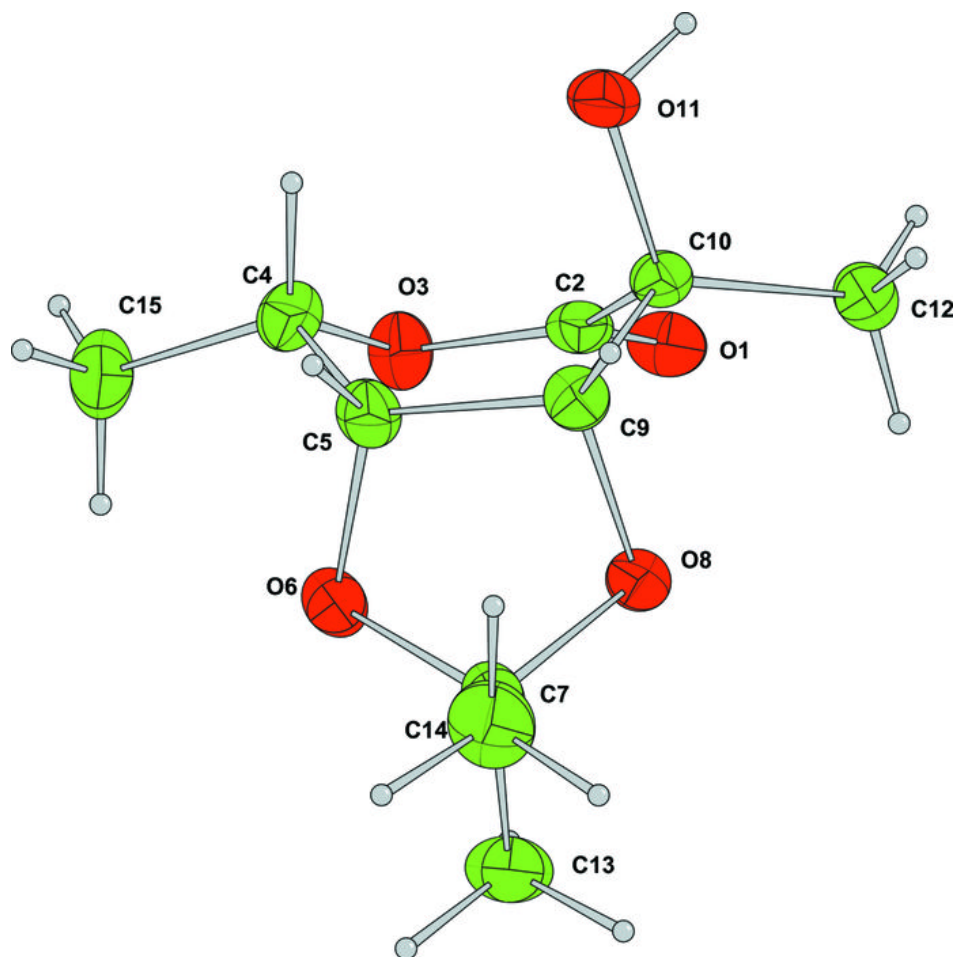


Fig. 3

