

Francesco Punzo,<sup>a\*‡</sup> David J. Watkin,<sup>b</sup> David Hotchkiss<sup>c</sup> and George W. J. Fleet<sup>c</sup>

<sup>a</sup>Dipartimento di Scienze Chimiche, Facoltà di Farmacia, Università di Catania, Viale A. Doria 6, 95125 Catania, Italy, <sup>b</sup>Department of Chemical Crystallography, Chemical Research Laboratory, Mansfield Road, Oxford OX1 3TA, England, and <sup>c</sup>Department of Organic Chemistry, Chemical Research Laboratory, Mansfield Road, Oxford OX1 3TA, England

‡ Visiting Scientist at the Department of Chemical Crystallography, Chemical Research Laboratory, Mansfield Road, Oxford OX1 3TA England

Correspondence e-mail: fpunzo@unict.it

#### Key indicators

Single-crystal X-ray study  
 $T = 120\text{ K}$   
 Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.024  
 $wR$  factor = 0.061  
 Data-to-parameter ratio = 10.8

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

## 2-C-Methyl-D-lyxono-1,4-lactone

The title compound,  $\text{C}_6\text{H}_{10}\text{O}_5$ , has been crystallized for the first time, allowing the stereochemistry at C-2 and the ring size of the lactone to be firmly established.

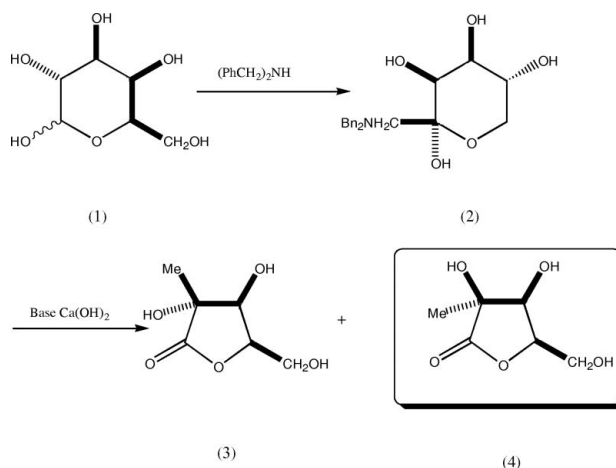
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#### Comment

The Kiliani ascension of ketoses (Hotchkiss *et al.*, 2004; Soengas *et al.*, 2005) provides ready access to a new class of branched carbohydrate scaffolds (Lichtenthaler & Peters, 2004; Bols, 1996) with branched carbon chains. Although saccharinic acids, which are 2-*C*-methyl aldonic acids, are formed in very low yields from treatment of aldoses or ketoses with aqueous calcium hydroxide (Whistler & BeMiller, 1963), it has been shown that significantly higher yields may be obtained from the reaction of lime with ketoses (Hotchkiss *et al.*, 2006) derived from the Amadori rearrangement (Hodge, 1955). *D*-Galactose reacted with dibenzylamine to form the Amadori ketose, (2) (Grunnagel & Haas, 1969), in which the  $\alpha$ -configuration at the anomeric position of the pyranose ring has been proved by X-ray crystallographic analysis (Harding *et al.*, 2005). Treatment of (2) with aqueous calcium hydroxide allowed the isolation of a mixture of two epimeric lactones.



The structure of the minor isomer was confirmed as 2-*C*-methyl-*D*-xylo-1,4-lactone, (3), by an X-ray structure of its 3,5-acetonide (Watkin *et al.*, 2005). The major product, 2-*C*-methyl-*D*-lyxono-1,4-lactone, (4), initially isolated as an oil, slowly crystallized, allowing the relative configuration at C-2 and the ring size of the lyxonolactone to be unambiguously assigned by X-ray crystallographic analysis.

Racemic lactone (4) has only been obtained as an oil (Lopez *et al.*, 1984); the enantiomer of (4) has been prepared in low yield from *L*-sorbose (Ishizu *et al.*, 1972). The absolute configuration of (4) was determined from the use of *D*-galactose (1) as the starting material.

## Experimental

The lactone (4) {m.p. 379–380K,  $[\alpha]_D^{23} +70.4$  ( $c$  0.87 in acetone)} was crystallized by dissolving it in acetone and allowing the slow evaporation of the solvent until colourless block-shaped crystals formed. The multi-scan technique was used to correct for changes in the illuminated volume.

### Crystal data

$C_6H_{10}O_5$	$D_x = 1.549 \text{ Mg m}^{-3}$
$M_r = 162.14$	Mo $K\alpha$ radiation
Monoclinic, $C2$	Cell parameters from 1011 reflections
$a = 18.6680$ (5) Å	$\theta = 5\text{--}30^\circ$
$b = 5.8280$ (2) Å	$\mu = 0.14 \text{ mm}^{-1}$
$c = 6.3943$ (2) Å	$T = 120 \text{ K}$
$\beta = 92.2219$ (14)°	Block, colourless
$V = 695.16$ (4) Å <sup>3</sup>	$0.70 \times 0.60 \times 0.50 \text{ mm}$
$Z = 4$	

### Data collection

Nonius KappaCCD area-detector diffractometer	1087 independent reflections
$\omega$ scans	1073 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (DENZO and SCALEPACK; Otwinowski & Minor, 1997)	$R_{\text{int}} = 0.010$
$T_{\text{min}} = 0.92$ , $T_{\text{max}} = 0.93$	$\theta_{\text{max}} = 30.0^\circ$
1943 measured reflections	$h = -25 \rightarrow 26$
	$k = -7 \rightarrow 8$
	$l = -8 \rightarrow 9$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F^2) + (0.03P)^2 + 0.33P]$
$R[F^2 > 2\sigma(F^2)] = 0.024$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.061$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
1087 reflections	$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
101 parameters	Extinction correction: Larson (1970), equation 22
H-atom parameters constrained	Extinction coefficient: $4.90(3) \times 10^2$

**Table 1**

Selected geometric parameters (Å, °).

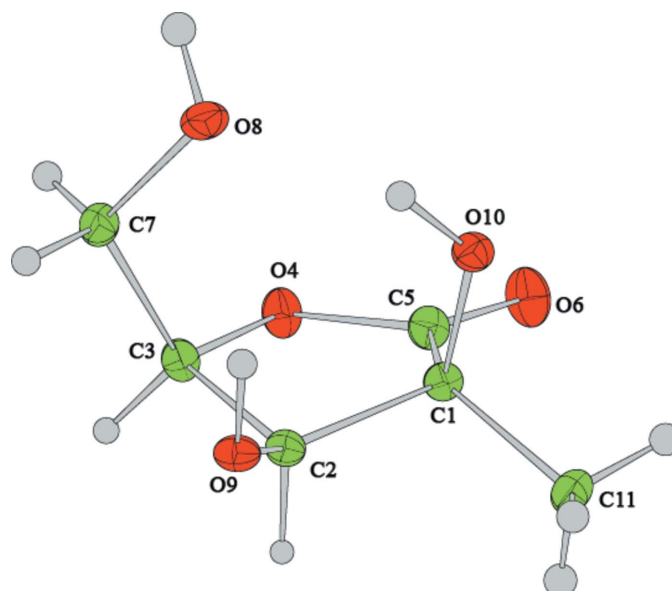
C1–C2	1.5382 (16)	C3–O4	1.4652 (15)
C1–C5	1.5342 (17)	C3–C7	1.5098 (18)
C1–O10	1.4329 (14)	O4–C5	1.3553 (15)
C1–C11	1.5150 (18)	C5–O6	1.2027 (15)
C2–C3	1.5448 (19)	C7–O8	1.4327 (16)
C2–O9	1.4163 (14)		
C2–C1–C5	100.95 (9)	C2–C3–O4	103.36 (9)
C2–C1–O10	112.80 (9)	C2–C3–C7	117.44 (10)
C5–C1–O10	107.55 (10)	O4–C3–C7	109.87 (11)
C2–C1–C11	114.56 (10)	C3–O4–C5	112.01 (10)
C5–C1–C11	113.52 (10)	C1–C5–O4	110.53 (10)
O10–C1–C11	107.29 (9)	C1–C5–O6	128.09 (11)
C1–C2–C3	104.94 (10)	O4–C5–O6	121.36 (12)
C1–C2–O9	115.82 (10)	C3–C7–O8	110.46 (10)
C3–C2–O9	114.94 (10)		

**Table 2**

Hydrogen-bond geometry (Å, °).

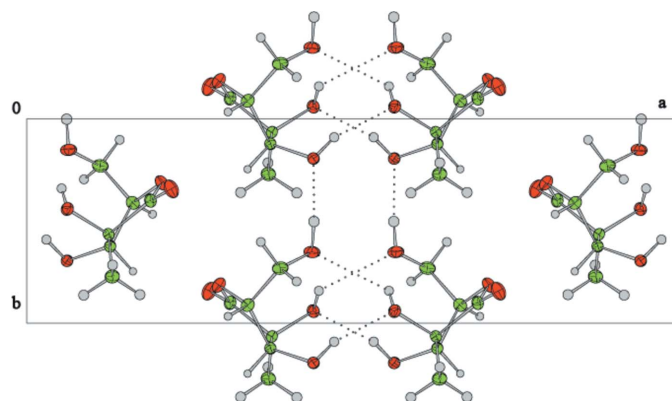
$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O10–H10 <sup>i</sup> ⋯O8	0.81	1.90	2.6770 (13)	159
O8–H8 <sup>j</sup> ⋯O9 <sup>l</sup>	0.88	1.82	2.6906 (14)	172
O9–H9 <sup>k</sup> ⋯O10 <sup>ii</sup>	0.86	1.93	2.7547 (13)	160

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



**Figure 1**

The molecular structure of (4), with displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitrary.



**Figure 2**

Packing diagram of (4), viewed down the  $c$  axis. Hydrogen bonds are displayed with dashed lines.

In the absence of significant anomalous scattering, Friedel pairs were merged. H atoms were located in a difference density map. Those attached to C atoms were repositioned geometrically. H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry ( $C\text{--}H = 0.93\text{--}0.98 \text{ \AA}$  and  $O\text{--}H = 0.82 \text{ \AA}$ ) and isotropic displacement parameters [ $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{parent atom})$ ], after which their positions were refined with riding constraints.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *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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## supporting information

*Acta Cryst.* (2006). E62, o98–o100 [doi:10.1107/S1600536805040456]

## 2-C-Methyl-D-lyxono-1,4-lactone

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### S1. Comment

The Kiliani ascension of ketoses (Hotchkiss *et al.*, 2004; Soengas *et al.*, 2005) provides ready access to a new class of branched carbohydrate scaffolds (Lichtenthaler & Peters, 2004; Bols, 1996) with branched carbon chains. Although saccharinic acids - which are 2-*C*-methyl aldonic acids - are formed in very low yields from treatment of aldoses or ketoses with aqueous calcium hydroxide (Whistler & BeMiller, 1963), it has been shown that significantly higher yields may be obtained from the reaction of lime with ketoses (Hotchkiss *et al.*, 2006) derived from the Amadori rearrangement (Hodge, 1955). *D*-Galactose reacted with dibenzylamine to form the Amadori ketose (2) (Grunnagel & Haas, 1969), in which the  $\alpha$ -configuration at the anomeric position of the pyranose ring has been proved by X-ray crystallographic analysis (Harding *et al.*, 2005). Treatment of (2) with aqueous calcium hydroxide allowed the isolation of a mixture of two epimeric lactones.

Table 1.

The structure of the minor isomer was confirmed as 2-*C*-methyl-*D*-xylo-1,4-lactone (3) by an X-ray structure of its 3,5-acetonide (Watkin *et al.*, 2005). The major product 2-*C*-methyl-*D*-lyxono-1,4-lactone (4), initially isolated as an oil, slowly crystallized allowing the relative configuration at C-2 and the ring size of the lyxonolactone to be unambiguously assigned by X-ray crystallographic analysis.

Figure 1.

Racemic lactone (4) has only been obtained as an oil (Lopez *et al.*, 1984); the enantiomer of (4) has been prepared in low yield from *l*-sorbose (Ishizu *et al.*, 1982 or 1972). The absolute configuration of (4) is determined from the use of *D*-galactose (1) as the starting material.

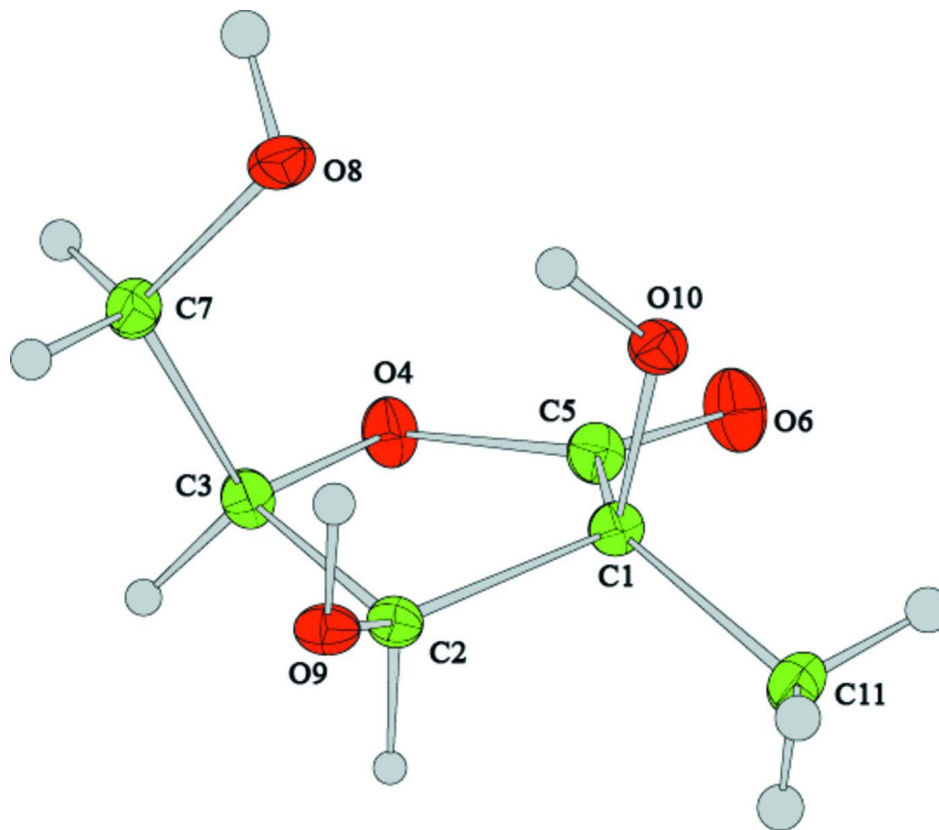
Figure 2.

### S2. Experimental

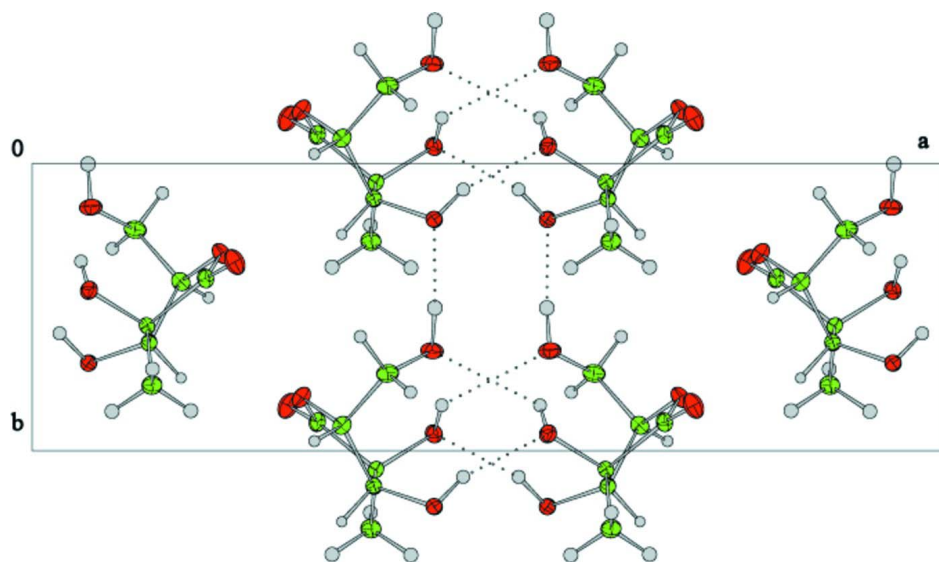
The lactone (4) (m.p. 379–380 K,  $[\alpha]_{\text{D}}^{23} +70.4$  ( $c$  0.87 in acetone)) was crystallized by dissolving it in acetone and allowing the slow evaporation of the solvent until colourless block-shaped crystals formed. The multi-scan technique was used to correct for changes in the illuminated volume.

### S3. Refinement

Because the data were collected with molybdenum radiation, there were no measurable anomalous differences, as a consequence of which it was admissible to merge Friedel pairs of reflections. H atoms were seen in a difference density synthesis. 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 = 0.96–0.98, O—H = 0.81–0.88 Å), after which they were refined as riding, with  $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for those bonded to carbon, and  $U(\text{H}) = 0.05 \text{ \AA}^2$  for the hydroxy group.

**Figure 1**

The asymmetric unit of (4), with displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitrary.

**Figure 2**

Packing diagram of (4), viewed down the *c* axis. Hydrogen bonds are displayed with dashed lines.

## 2-C-Methyl-D-lyxono-1,4-lactone

## Crystal data

C<sub>6</sub>H<sub>10</sub>O<sub>5</sub> $M_r = 162.14$ Monoclinic, *C*2Hall symbol: *C* 2y $a = 18.6680$  (5) Å $b = 5.8280$  (2) Å $c = 6.3943$  (2) Å $\beta = 92.2219$  (14)° $V = 695.16$  (4) Å<sup>3</sup> $Z = 4$  $F(000) = 344$  $D_x = 1.549$  Mg m<sup>-3</sup>Mo *K* $\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1011 reflections

 $\theta = 5\text{--}30^\circ$  $\mu = 0.14$  mm<sup>-1</sup> $T = 120$  K

Block, colourless

0.70 × 0.60 × 0.50 mm

## Data collection

Nonius KappaCCD

diffractometer

Graphite monochromator

 $\omega$  scans

Absorption correction: multi-scan

*(DENZO/SCALEPACK; Otwinowski & Minor, 1997)* $T_{\min} = 0.92$ ,  $T_{\max} = 0.93$ 

1943 measured reflections

1087 independent reflections

1073 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.010$  $\theta_{\max} = 30.0^\circ$ ,  $\theta_{\min} = 5.3^\circ$  $h = -25 \rightarrow 26$  $k = -7 \rightarrow 8$  $l = -8 \rightarrow 9$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.024$  $wR(F^2) = 0.061$  $S = 1.04$ 

1087 reflections

101 parameters

1 restraint

Primary atom site location: structure-invariant

direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F^2) + (0.03P)^2 + 0.33P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.000363$  $\Delta\rho_{\max} = 0.22$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.16$  e Å<sup>-3</sup>

Extinction correction: Larson (1970), equation

22

Extinction coefficient: 490 (30)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.62534 (6)	-0.0653 (2)	0.62746 (18)	0.0118
C2	0.62752 (6)	-0.1232 (2)	0.39310 (18)	0.0118
C3	0.66213 (6)	0.0885 (2)	0.29245 (19)	0.0134
O4	0.70639 (5)	0.18637 (17)	0.46441 (13)	0.0153
C5	0.68912 (6)	0.0998 (2)	0.65261 (19)	0.0139
O6	0.72080 (5)	0.1571 (2)	0.81146 (15)	0.0203
C7	0.61246 (7)	0.2698 (2)	0.20044 (19)	0.0160
O8	0.56415 (5)	0.34812 (16)	0.35384 (15)	0.0177
O9	0.56175 (5)	-0.19459 (17)	0.29616 (14)	0.0136
O10	0.56216 (4)	0.05895 (18)	0.67885 (13)	0.0138
C11	0.63081 (7)	-0.2712 (2)	0.7718 (2)	0.0161
H21	0.6625	-0.2465	0.3726	0.0110*
H31	0.6924	0.0333	0.1831	0.0132*

H71	0.6421	0.3972	0.1576	0.0166*
H72	0.5877	0.2039	0.0787	0.0164*
H111	0.6312	-0.2152	0.9153	0.0203*
H112	0.6729	-0.3606	0.7480	0.0211*
H113	0.5871	-0.3629	0.7458	0.0206*
H10	0.5535	0.1590	0.5935	0.0169*
H8	0.5610	0.4976	0.3450	0.0229*
H9	0.5301	-0.0909	0.3159	0.0174*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0105 (5)	0.0117 (6)	0.0132 (5)	-0.0010 (4)	0.0008 (4)	0.0004 (4)
C2	0.0109 (5)	0.0117 (5)	0.0129 (5)	0.0003 (4)	0.0003 (4)	-0.0008 (5)
C3	0.0143 (5)	0.0141 (6)	0.0118 (5)	-0.0018 (5)	0.0012 (4)	-0.0007 (5)
O4	0.0148 (4)	0.0181 (5)	0.0132 (4)	-0.0051 (4)	0.0012 (3)	0.0002 (4)
C5	0.0116 (5)	0.0155 (6)	0.0147 (5)	-0.0011 (5)	0.0021 (4)	0.0006 (5)
O6	0.0181 (4)	0.0271 (6)	0.0154 (4)	-0.0064 (4)	-0.0016 (3)	-0.0016 (4)
C7	0.0210 (6)	0.0135 (6)	0.0134 (5)	-0.0012 (5)	0.0015 (4)	0.0010 (5)
O8	0.0217 (5)	0.0109 (5)	0.0209 (4)	0.0011 (4)	0.0054 (3)	0.0021 (4)
O9	0.0116 (4)	0.0115 (4)	0.0174 (4)	0.0003 (3)	-0.0015 (3)	-0.0023 (3)
O10	0.0124 (4)	0.0140 (4)	0.0152 (4)	0.0011 (4)	0.0026 (3)	0.0005 (4)
C11	0.0176 (6)	0.0147 (6)	0.0160 (5)	0.0006 (5)	0.0007 (4)	0.0039 (5)

*Geometric parameters (Å, °)*

C1—C2	1.5382 (16)	C5—O6	1.2027 (15)
C1—C5	1.5342 (17)	C7—O8	1.4327 (16)
C1—O10	1.4329 (14)	C7—H71	0.972
C1—C11	1.5150 (18)	C7—H72	0.969
C2—C3	1.5448 (19)	O8—H8	0.875
C2—O9	1.4163 (14)	O9—H9	0.858
C2—H21	0.983	O10—H10	0.811
C3—O4	1.4652 (15)	C11—H111	0.974
C3—C7	1.5098 (18)	C11—H112	0.960
C3—H31	0.971	C11—H113	0.984
O4—C5	1.3553 (15)		
C2—C1—C5	100.95 (9)	C1—C5—O4	110.53 (10)
C2—C1—O10	112.80 (9)	C1—C5—O6	128.09 (11)
C5—C1—O10	107.55 (10)	O4—C5—O6	121.36 (12)
C2—C1—C11	114.56 (10)	C3—C7—O8	110.46 (10)
C5—C1—C11	113.52 (10)	C3—C7—H71	107.3
O10—C1—C11	107.29 (9)	O8—C7—H71	109.2
C1—C2—C3	104.94 (10)	C3—C7—H72	107.6
C1—C2—O9	115.82 (10)	O8—C7—H72	112.5
C3—C2—O9	114.94 (10)	H71—C7—H72	109.6
C1—C2—H21	109.5	C7—O8—H8	108.4

C3—C2—H21	103.8	C2—O9—H9	108.6
O9—C2—H21	107.2	C1—O10—H10	110.7
C2—C3—O4	103.36 (9)	C1—C11—H111	107.8
C2—C3—C7	117.44 (10)	C1—C11—H112	111.7
O4—C3—C7	109.87 (11)	H111—C11—H112	110.7
C2—C3—H31	107.5	C1—C11—H113	106.9
O4—C3—H31	110.0	H111—C11—H113	108.5
C7—C3—H31	108.5	H112—C11—H113	110.9
C3—O4—C5	112.01 (10)		

*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>
O10—H10 $\cdots$ O8	0.81	1.90	2.6770 (13)	159
O8—H8 $\cdots$ O9 <sup>i</sup>	0.88	1.82	2.6906 (14)	172
O9—H9 $\cdots$ O10 <sup>ii</sup>	0.86	1.93	2.7547 (13)	160

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