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## Structure Reports

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## 3,4,6-Tri-O-acetyl-1,2-[(S)-ethylidene]- $\beta$ -D-mannopyranose

Henok H. Kinfe, Felix L. Makolo, Zanele Phasha and Alfred Muller\*

Research Center for Synthesis and Catalysis, Department of Chemistry, University of Johannesburg (APK Campus), PO Box 524, Auckland Park, Johannesburg, 2006, South Africa

Correspondence e-mail: mullera@uj.ac.za

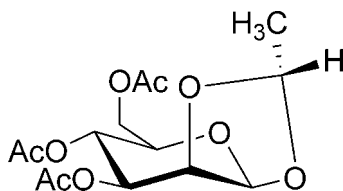
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.026;  $wR$  factor = 0.085; data-to-parameter ratio = 12.7.

In the title compound,  $\text{C}_{14}\text{H}_{20}\text{O}_9$ , the six-membered pyran and the five-membered dioxalane rings adopt chair and twisted conformations, respectively. In the crystal, the molecules are linked by  $\text{C}-\text{H}\cdots\text{O}$  interactions.

### Related literature

For orthogonal protection in carbohydrate chemistry, see: Wuts & Greene (2007); Betaneli *et al.* (1982). For background to the synthetic methodology, see: Doores *et al.* (2010). For ring puckering analysis, see: Cremer & Pople (1975).



### Experimental

#### Crystal data

$\text{C}_{14}\text{H}_{20}\text{O}_9$	$V = 1591.72$ (12) Å <sup>3</sup>
$M_r = 332.3$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Cu $K\alpha$ radiation
$a = 7.0494$ (3) Å	$\mu = 1.01$ mm <sup>-1</sup>
$b = 14.6994$ (7) Å	$T = 100$ K
$c = 15.3608$ (7) Å	$0.16 \times 0.16 \times 0.12$ mm

#### Data collection

Bruker APEX DUO 4K-CCD diffractometer	23454 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2008)	2701 independent reflections
$T_{\min} = 0.856$ , $T_{\max} = 0.889$	2684 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.028$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$	$\Delta\rho_{\text{max}} = 0.26$ e Å <sup>-3</sup>
$wR(F^2) = 0.085$	$\Delta\rho_{\text{min}} = -0.31$ e Å <sup>-3</sup>
$S = 1.20$	Absolute structure: Flack (1983),
2701 reflections	1110 Friedel Pairs
212 parameters	Flack parameter: 0.06 (15)
H-atom parameters constrained	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C1}-\text{H1C}\cdots\text{O3}^{\text{i}}$	0.98	2.55	3.511 (2)	167
$\text{C8}-\text{H8}\cdots\text{O7}^{\text{i}}$	1.00	2.49	3.317 (2)	140
$\text{C12}-\text{H12A}\cdots\text{O4}^{\text{ii}}$	0.98	2.56	3.469 (2)	154
$\text{C12}-\text{H12B}\cdots\text{O1}^{\text{iii}}$	0.98	2.51	3.460 (2)	163
$\text{C14}-\text{H14B}\cdots\text{O9}^{\text{iv}}$	0.98	2.53	3.361 (2)	142

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

Data collection: APEX2 (Bruker, 2011); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT and XPREP (Bruker, 2008); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 2005); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5592).

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## supporting information

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**3,4,6-Tri-*O*-acetyl-1,2-[(*S*)-ethylidene]- $\beta$ -*D*-mannopyranose**

**Henok H. Kinfе, Felix L. Makolo, Zanele Phasha and Alfred Muller**

**S1. Comment**

Ethylidene acetals are important functional groups for orthogonal protection in carbohydrate chemistry (Wuts & Greene, 2007; Betaneli *et al.*, 1982). The title compound is a key intermediate for the preparation of polysaccharides which exhibit strong activity against the HIV-1 virus (Doores *et al.*, 2010). Herein, we report the crystal structure of 3,4,6-*tri-O*-acetyl-1,2-*O*-[*S*-ethylidene]- $\beta$ -*D*-mannopyranoside to confirm its absolute configuration.

The title compound C<sub>14</sub>H<sub>20</sub>O<sub>9</sub> (see Fig. 1, and Scheme 1) crystallizes in the *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub> (*Z* = 4) space group. Puckering analysis confirms the twisted conformation of the five membered dioxalane ring, with puckering parameter values of  $q_2 = 0.347$  (3) Å, and  $\varphi_2 = 61.9$  (4)°; and that of the six membered pyran chair conformation ring as  $q_2 = 0.163$  (3) Å,  $q_3 = -0.526$  (3) Å,  $Q = 0.550$  (3) Å and  $\varphi_2 = 253.3$  (9)° (see Cremer & Pople, 1975). The dioxalane ring is twisted on C6–C7.

The molecules are linked by C–H⋯O interactions (see Table 1).

**S2. Experimental**

A solution of 2,3,4,6-*tetra-O*-acetyl- $\alpha$ -*D*-mannopyranosyl bromide (150 mg, 0.36 mmol) in acetonitrile (3 ml) was treated with sodium boron hydride (250 mg, 6.61 mmol) and the reaction mixture was stirred at room temperature for 12 h. The mixture was then diluted with chloroform and washed with water three times. The organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and evaporated *in vacuo* to give an oil. The oily residue was crashed with methanol to afford 70% of the target compound as white crystals.

Analytical data: mp: 108–110 °C (Lit. 113–115 °C; Betaneli *et al.*, 1982); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  5.40–5.10 (m, 4H), 4.31–4.03 (m, 3H), 3.72–3.64 (m, 1H), 2.09 (s, 3H), 2.04 (s, 3H), 2.02 (s, 3H), 1.51 (d, *J* = 4.8 Hz, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  170.7, 170.3, 169.5, 104.8, 96.5, 71.6, 70.6, 66.0, 62.5, 21.6, 20.7, 20.7.

**S3. Refinement**

All hydrogen atoms were positioned in geometrically idealized positions with C–H = 1.00 Å (methine), 0.99 Å (methylene), and 0.98 Å (methyl). All hydrogen atoms were allowed to ride on their parent atoms with  $U_{iso}(H) = 1.2U_{eq}$ , except for the methyl where  $U_{iso}(H) = 1.5U_{eq}$  was utilized. The initial positions of methyl hydrogen atoms were located from a Fourier difference map and refined as a fixed rotor. The *D* enantiomer refined to a final Flack parameter of 0.06 (15). The highest residual electron density of 0.50 e.Å<sup>-3</sup> is 0.93 Å from H14B.

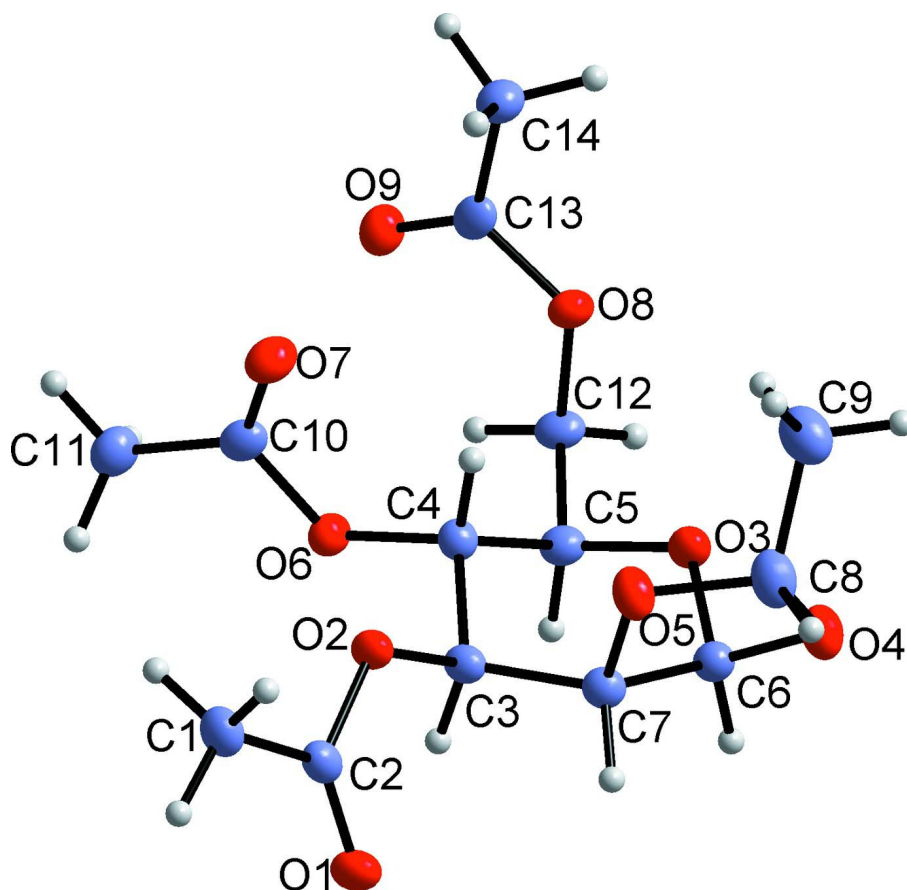


Figure 1

A view of (1). Displacement ellipsoids are drawn at the 50% probability level.

### 3,4,6-Tri-*O*-acetyl-1,2-[(*S*)-ethylidene]- $\beta$ -*D*-mannopyranose

#### Crystal data

$C_{14}H_{20}O_9$

$M_r = 332.3$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.0494$  (3) Å

$b = 14.6994$  (7) Å

$c = 15.3608$  (7) Å

$V = 1591.72$  (12) Å<sup>3</sup>

$Z = 4$

$F(000) = 704$

$D_x = 1.387$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å

Cell parameters from 9727 reflections

$\theta = 6.5$ – $65.8^\circ$

$\mu = 1.01$  mm<sup>-1</sup>

$T = 100$  K

Cube, colourless

$0.16 \times 0.16 \times 0.12$  mm

#### Data collection

Bruker APEX DUO 4K-CCD  
diffractometer

Radiation source: Incoatec  $I\mu$ S microfocus X-  
ray source

Incoatec Quazar Multilayer Mirror  
monochromator

Detector resolution: 8.4 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2008)

$T_{\min} = 0.856$ ,  $T_{\max} = 0.889$

23454 measured reflections

2701 independent reflections

2684 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 66.2^\circ$ ,  $\theta_{\min} = 4.2^\circ$

$h = -8 \rightarrow 8$   
 $k = -16 \rightarrow 14$

$l = -18 \rightarrow 17$

### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.085$   
 $S = 1.20$   
 2701 reflections  
 212 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods  
 Secondary atom site location: difference Fourier  
 map

Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.3436P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$   
 Absolute structure: Flack (1983), 1110 Friedel  
 Pairs  
 Absolute structure parameter: 0.06 (15)

### Special details

**Experimental.** The intensity data was collected on a Bruker Apex DUO 4 K CCD diffractometer using an exposure time of 5 s/frame. A total of 4548 frames were collected with a frame width of  $1^\circ$  covering up to  $\theta = 66.21^\circ$  with 97.7% completeness accomplished.

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

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

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1916 (3)	0.55884 (11)	0.36854 (11)	0.0238 (4)
H1A	0.0905	0.5131	0.3687	0.036*
H1B	0.2086	0.5828	0.4276	0.036*
H1C	0.3102	0.5309	0.3487	0.036*
C2	0.1392 (2)	0.63445 (10)	0.30873 (10)	0.0191 (3)
C3	0.2253 (2)	0.78379 (10)	0.26420 (10)	0.0182 (3)
H3	0.085	0.7938	0.2623	0.022*
C4	0.3175 (2)	0.86302 (10)	0.30997 (10)	0.0181 (3)
H4	0.4551	0.8504	0.3202	0.022*
C5	0.2937 (2)	0.94827 (11)	0.25454 (10)	0.0199 (3)
H5	0.156	0.9571	0.2413	0.024*
C6	0.3204 (2)	0.86280 (11)	0.12195 (10)	0.0200 (3)
H6	0.1988	0.8811	0.0933	0.024*
C7	0.2989 (2)	0.77323 (10)	0.17178 (10)	0.0195 (3)
H7	0.2167	0.7302	0.1384	0.023*
C8	0.5782 (3)	0.76982 (12)	0.09524 (12)	0.0275 (4)
H8	0.5843	0.7181	0.053	0.033*
C9	0.7739 (3)	0.80202 (14)	0.11606 (13)	0.0346 (4)

H9A	0.7676	0.8491	0.1611	0.052*
H9B	0.8325	0.8273	0.0635	0.052*
H9C	0.85	0.7508	0.1372	0.052*
C10	0.3704 (2)	1.03347 (11)	0.29613 (11)	0.0214 (4)
H10A	0.3591	1.0852	0.2552	0.026*
H10B	0.2967	1.048	0.3492	0.026*
C11	0.6193 (2)	1.03193 (10)	0.40213 (11)	0.0203 (4)
C12	0.8252 (3)	1.01153 (13)	0.41372 (11)	0.0258 (4)
H12A	0.8696	1.038	0.4686	0.039*
H12B	0.8971	1.0378	0.3652	0.039*
H12C	0.8441	0.9455	0.4149	0.039*
C13	0.3177 (2)	0.85697 (10)	0.46588 (11)	0.0208 (3)
C14	0.1957 (3)	0.87231 (12)	0.54383 (11)	0.0274 (4)
H14A	0.267	0.857	0.5966	0.041*
H14B	0.0827	0.8337	0.54	0.041*
H14C	0.1573	0.9363	0.5461	0.041*
O1	0.00432 (18)	0.63631 (8)	0.26050 (8)	0.0251 (3)
O2	0.26442 (16)	0.70374 (7)	0.31509 (7)	0.0192 (2)
O3	0.39448 (16)	0.93390 (8)	0.17474 (8)	0.0213 (3)
O4	0.46022 (18)	0.84164 (8)	0.06055 (8)	0.0251 (3)
O5	0.48923 (17)	0.74080 (7)	0.17374 (8)	0.0234 (3)
O6	0.56740 (16)	1.01897 (7)	0.31844 (7)	0.0202 (3)
O7	0.51158 (18)	1.05559 (8)	0.45868 (8)	0.0249 (3)
O8	0.22067 (16)	0.87679 (7)	0.39160 (7)	0.0201 (3)
O9	0.47936 (18)	0.83119 (8)	0.46649 (8)	0.0265 (3)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0263 (9)	0.0187 (8)	0.0263 (8)	-0.0010 (7)	0.0026 (7)	0.0042 (7)
C2	0.0200 (8)	0.0172 (8)	0.0200 (8)	-0.0019 (6)	0.0034 (7)	-0.0019 (6)
C3	0.0178 (7)	0.0158 (7)	0.0210 (7)	-0.0010 (6)	-0.0022 (6)	0.0033 (6)
C4	0.0179 (7)	0.0177 (8)	0.0186 (7)	0.0006 (6)	0.0005 (6)	0.0012 (6)
C5	0.0192 (7)	0.0181 (8)	0.0224 (8)	0.0014 (6)	-0.0036 (6)	0.0007 (6)
C6	0.0210 (8)	0.0197 (8)	0.0192 (8)	-0.0022 (6)	-0.0015 (6)	0.0008 (6)
C7	0.0197 (8)	0.0170 (7)	0.0216 (7)	0.0000 (6)	-0.0008 (7)	0.0005 (6)
C8	0.0314 (9)	0.0222 (8)	0.0290 (9)	0.0041 (7)	0.0065 (8)	0.0050 (7)
C9	0.0309 (10)	0.0329 (10)	0.0399 (10)	0.0006 (8)	0.0032 (8)	0.0091 (8)
C10	0.0198 (8)	0.0186 (8)	0.0259 (8)	0.0014 (6)	-0.0065 (7)	0.0008 (7)
C11	0.0275 (9)	0.0128 (7)	0.0207 (8)	-0.0017 (7)	-0.0031 (7)	0.0031 (7)
C12	0.0243 (9)	0.0293 (9)	0.0238 (8)	-0.0009 (7)	-0.0044 (7)	0.0016 (7)
C13	0.0247 (9)	0.0147 (8)	0.0229 (8)	-0.0006 (6)	-0.0043 (7)	-0.0007 (6)
C14	0.0315 (9)	0.0283 (9)	0.0222 (8)	0.0003 (8)	-0.0005 (7)	-0.0040 (7)
O1	0.0234 (6)	0.0225 (6)	0.0294 (6)	-0.0049 (5)	-0.0047 (5)	0.0021 (5)
O2	0.0208 (6)	0.0158 (5)	0.0210 (5)	-0.0030 (4)	-0.0030 (4)	0.0039 (4)
O3	0.0246 (6)	0.0186 (5)	0.0208 (5)	-0.0046 (5)	-0.0017 (5)	0.0019 (5)
O4	0.0271 (6)	0.0229 (6)	0.0254 (6)	0.0029 (5)	0.0053 (5)	0.0057 (5)
O5	0.0241 (6)	0.0202 (5)	0.0259 (6)	0.0051 (5)	0.0051 (5)	0.0060 (5)

O6	0.0199 (6)	0.0203 (5)	0.0204 (5)	-0.0003 (4)	-0.0039 (5)	-0.0014 (5)
O7	0.0286 (6)	0.0242 (6)	0.0219 (6)	0.0031 (5)	0.0011 (5)	-0.0005 (5)
O8	0.0200 (6)	0.0218 (5)	0.0186 (5)	0.0022 (5)	-0.0007 (5)	-0.0005 (4)
O9	0.0257 (7)	0.0287 (6)	0.0250 (6)	0.0050 (5)	-0.0056 (5)	-0.0003 (5)

*Geometric parameters (Å, °)*

C1—C2	1.489 (2)	C8—O5	1.425 (2)
C1—H1A	0.98	C8—O4	1.446 (2)
C1—H1B	0.98	C8—C9	1.493 (3)
C1—H1C	0.98	C8—H8	1
C2—O1	1.206 (2)	C9—H9A	0.98
C2—O2	1.3514 (19)	C9—H9B	0.98
C3—O2	1.4394 (18)	C9—H9C	0.98
C3—C4	1.508 (2)	C10—O6	1.4461 (19)
C3—C7	1.520 (2)	C10—H10A	0.99
C3—H3	1	C10—H10B	0.99
C4—O8	1.4420 (19)	C11—O7	1.205 (2)
C4—C5	1.524 (2)	C11—O6	1.350 (2)
C4—H4	1	C11—C12	1.493 (2)
C5—O3	1.433 (2)	C12—H12A	0.98
C5—C10	1.506 (2)	C12—H12B	0.98
C5—H5	1	C12—H12C	0.98
C6—O4	1.399 (2)	C13—O9	1.201 (2)
C6—O3	1.422 (2)	C13—O8	1.3620 (19)
C6—C7	1.530 (2)	C13—C14	1.492 (2)
C6—H6	1	C14—H14A	0.98
C7—O5	1.424 (2)	C14—H14B	0.98
C7—H7	1	C14—H14C	0.98
C2—C1—H1A	109.5	O4—C8—C9	112.28 (15)
C2—C1—H1B	109.5	O5—C8—H8	109.9
H1A—C1—H1B	109.5	O4—C8—H8	109.9
C2—C1—H1C	109.5	C9—C8—H8	109.9
H1A—C1—H1C	109.5	C8—C9—H9A	109.5
H1B—C1—H1C	109.5	C8—C9—H9B	109.5
O1—C2—O2	122.85 (14)	H9A—C9—H9B	109.5
O1—C2—C1	126.29 (15)	C8—C9—H9C	109.5
O2—C2—C1	110.86 (13)	H9A—C9—H9C	109.5
O2—C3—C4	107.19 (12)	H9B—C9—H9C	109.5
O2—C3—C7	110.99 (12)	O6—C10—C5	108.84 (13)
C4—C3—C7	111.54 (13)	O6—C10—H10A	109.9
O2—C3—H3	109	C5—C10—H10A	109.9
C4—C3—H3	109	O6—C10—H10B	109.9
C7—C3—H3	109	C5—C10—H10B	109.9
O8—C4—C3	108.04 (12)	H10A—C10—H10B	108.3
O8—C4—C5	108.55 (12)	O7—C11—O6	123.81 (15)
C3—C4—C5	109.08 (12)	O7—C11—C12	125.78 (16)

O8—C4—H4	110.4	O6—C11—C12	110.40 (14)
C3—C4—H4	110.4	C11—C12—H12A	109.5
C5—C4—H4	110.4	C11—C12—H12B	109.5
O3—C5—C10	107.89 (13)	H12A—C12—H12B	109.5
O3—C5—C4	107.58 (12)	C11—C12—H12C	109.5
C10—C5—C4	114.01 (13)	H12A—C12—H12C	109.5
O3—C5—H5	109.1	H12B—C12—H12C	109.5
C10—C5—H5	109.1	O9—C13—O8	123.42 (15)
C4—C5—H5	109.1	O9—C13—C14	126.07 (16)
O4—C6—O3	106.79 (13)	O8—C13—C14	110.51 (14)
O4—C6—C7	102.44 (12)	C13—C14—H14A	109.5
O3—C6—C7	112.54 (12)	C13—C14—H14B	109.5
O4—C6—H6	111.5	H14A—C14—H14B	109.5
O3—C6—H6	111.5	C13—C14—H14C	109.5
C7—C6—H6	111.5	H14A—C14—H14C	109.5
O5—C7—C3	109.66 (13)	H14B—C14—H14C	109.5
O5—C7—C6	101.86 (12)	C2—O2—C3	116.85 (12)
C3—C7—C6	114.39 (13)	C6—O3—C5	114.45 (12)
O5—C7—H7	110.2	C6—O4—C8	108.62 (12)
C3—C7—H7	110.2	C7—O5—C8	107.27 (12)
C6—C7—H7	110.2	C11—O6—C10	117.71 (13)
O5—C8—O4	106.08 (13)	C13—O8—C4	117.42 (12)
O5—C8—C9	108.69 (15)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C1—H1C...O3 <sup>i</sup>	0.98	2.55	3.511 (2)	167
C8—H8...O7 <sup>i</sup>	1.00	2.49	3.317 (2)	140
C12—H12A...O4 <sup>ii</sup>	0.98	2.56	3.469 (2)	154
C12—H12B...O1 <sup>iii</sup>	0.98	2.51	3.460 (2)	163
C14—H14B...O9 <sup>iv</sup>	0.98	2.53	3.361 (2)	142

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