

**Keywords:** crystal structure; hydrogen bonds; paclitaxel; cyclohexane; hydroxy groups

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# Crystal structure of $(\pm)$ -(7*RS*,8*SR*)-7-methyl-1,4-dioxaspiro[4.5]decane-7,8-diol

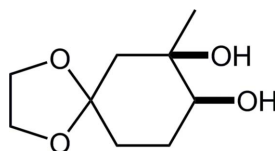
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In the title compound, C<sub>9</sub>H<sub>16</sub>O<sub>4</sub>, the five-membered dioxolane ring adopts a twist conformation; two adjacent C atoms deviate alternately from the mean plane of other atoms by  $-0.297$  (4) and  $0.288$  (4) Å. The spiro-fused cyclohexane ring shows a chair form. The hydroxy group substituted in an axial position makes an intramolecular O—H $\cdots$ O hydrogen bond with one of the O atoms in the cyclic ether, forming an *S*(6) ring motif. In the crystal, the O—H $\cdots$ O hydrogen bond involving the equatorial hydroxy group connects the molecules into a zigzag chain with a *C*(5) motif running along the *c* axis.

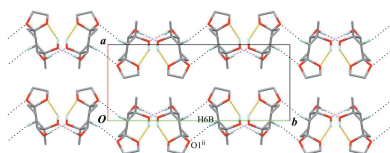
## 1. Chemical context

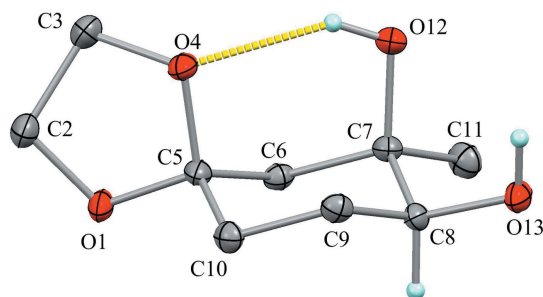
Paclitaxel (systematic name: (1*S*,2*S*,3*R*,4*S*,7*R*,9*S*,10*S*,12*R*,15*S*)-4,12-diacetoxy-1,9-dihydroxy-15-[(2*R*,3*S*)-3-benzoylamino-2-hydroxy-3-phenyl]propanoyloxy-10,14,17,17-tetramethyl-11-oxo-6-oxatetracyclo[11.3.1.0<sup>3,10</sup>.0<sup>4,7</sup>]heptadec-13-en-2-yl benzoate) is a well-known natural diterpenoid with a potent antitumor activity (Wall & Wani, 1995). Its rather complicated structure and significant bioactivity have attracted chemical and medicinal interests. While we recently reported several structures of the compounds (Oishi, Yamaguchi *et al.*, 2015; Oishi, Fukaya *et al.*, 2015*a,b*) obtained in the synthesis of paclitaxel (Fukaya, Tanaka *et al.*, 2015; Fukaya, Kodama *et al.*, 2015), the title compound has been prepared in an efficient synthetic approach to furnish the highly functionalized cyclohexane unit (Fukaya, Sugai *et al.*, 2015). Although the title compound has been reported first with a different synthetic procedure, any stereochemical or conformational assignment was not mentioned (Li *et al.*, 1981).



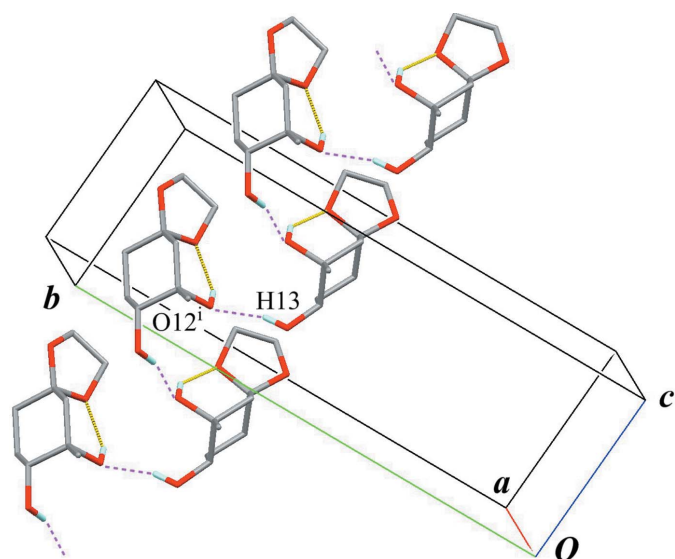
## 2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The dioxolane ring (O1/C2/C3/O4/C5) adopts a twist form with puckering parameters of  $Q(2) = 0.3523$  (16) Å and  $\varphi(2) = 233.8$  (3)°. Atoms C2 and C3 deviate from the mean plane of the other three atoms by  $-0.297$  (4) and  $0.288$  (4) Å, respectively. The cyclohexane ring (C5–C10) adopts a chair form with puckering parameters of  $Q = 0.5560$  (18) Å,  $\theta = 3.32$  (18)°,  $\varphi = 193$  (3)°,  $Q(2) = 0.0323$  (17) Å and  $Q(3) =$

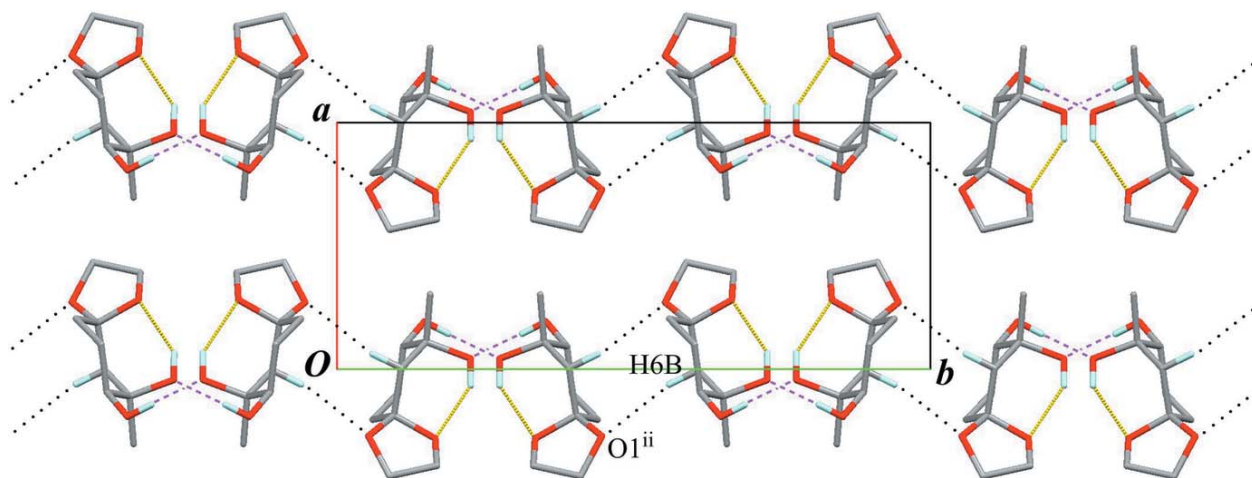




**Figure 1**  
The molecular structure of the title compound, showing the atom labels. Displacement ellipsoids are drawn at the 50% probability level. The yellow dotted line indicates the intramolecular O—H...O hydrogen bond. Only H atoms connected to O and chiral C atoms are shown for clarity.



**Figure 2**  
A partial packing view showing the chain structure. Yellow lines indicate the intramolecular O—H...O hydrogen bonds. Purple dashed lines indicate the intermolecular O—H...O hydrogen bonds. Only H atoms involved in hydrogen bonds are shown for clarity. [Symmetry code: (i)  $x, -y + \frac{3}{2}, z - \frac{1}{2}$ .]



**Figure 3**  
A packing diagram viewed down the  $c$  axis. Black dotted lines indicate the intermolecular C—H...O interactions. Yellow lines and purple dashed lines indicate the intra- and intermolecular O—H...O hydrogen bonds, respectively. Only H atoms involved in hydrogen bonds are shown for clarity. [Symmetry code: (ii)  $-x, -y + 1, -z + 2$ .]

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O12—H12...O4	0.84	2.05	2.7838 (16)	146
O13—H13...O12 <sup>i</sup>	0.84	1.99	2.8093 (16)	166
C6—H6B...O1 <sup>ii</sup>	0.99	2.61	3.5631 (19)	162

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

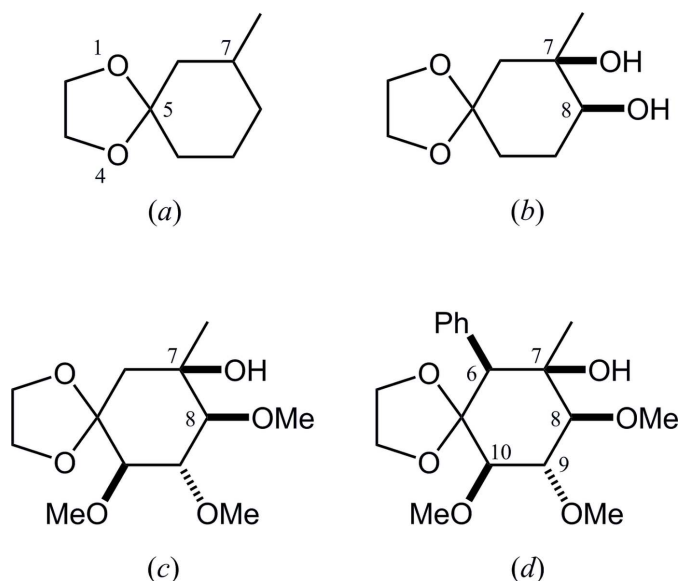
0.5551 (18) Å. The C5—O1, C7—C11 and C8—O13 bonds of equatorially oriented substituents make angles of 68.30 (9), 69.85 (9) and 75.76 (9)°, respectively, with the normal to the Cremer and Pople plane of the cyclohexane ring. The axially oriented hydroxy group forms an intramolecular O—H...O hydrogen bond (O12—H12...O4; Table 1), generating an  $S(6)$  graph-set motif. In this ring motif, five atoms (C5—O4...H12—O12—O7) are nearly coplanar with a maximum deviation of 0.012 (5) Å for atom O4.

### 3. Supramolecular features

The crystal packing features an intermolecular O—H...O hydrogen bond (O13—H13...O12<sup>i</sup>; Table 1) connecting enantiomers related by a glide plane to form a chain structure with a  $C(5)$  graph-set motif running along the  $c$  axis (Fig. 2). An intermolecular C—H...O interaction (C6—H6B...O1<sup>ii</sup>; Table 1) with a slightly longer distance, leading to form a sheet parallel to (100), is also observed (Fig. 3).

### 4. Database survey

In the Cambridge Structural Database (CSD, Version 5.36, November 2014; Groom & Allen, 2014), 266 structures containing a 7-methyl-1,4-dioxaspiro[4.5]decane skeleton, ( $a$ ), are registered (Fig. 4). These include six compounds with 7,8-dioxy-substituents. Two of them (JIQFIY and JIQGAR; Collins *et al.*, 1998), synthesized from *D*-glucose, are closely



**Figure 4**  
 (a) 7-Methyl-1,4-dioxaspiro[4.5]decane; as the core structure for database survey, (b) the title compound, and its (c) 9,10-dimethoxy-8-*O*-methyl and (d) 9,10-dimethoxy-6-phenyl-8-*O*-methyl derivatives.

related to the title compound [(b); racemic,  $P2_1/c$ ], which are its 9,10-dimethoxy-8-*O*-methyl [(c); chiral,  $P2_12_12_1$ ] and 9,10-dimethoxy-6-phenyl-8-*O*-methyl [(d); chiral,  $P2_12_12_1$ ] derivatives. In the crystal structures of (c) and (d), the dioxolane rings adopt twist forms and the cyclohexane rings show chair forms. The intramolecular O—H...O hydrogen bond is also observed in (c), but not in (d).

## 5. Synthesis and crystallization

The title compound was afforded in an improved synthetic approach of paclitaxel from 3-methylanisole (Fukaya, Sugai *et al.*, 2015). Purification was carried out by silica gel column chromatography, and colorless crystals were obtained from an ethyl acetate solution by slow evaporation at ambient temperature. M.p. 359–360 K. HRMS (ESI)  $m/z$  calculated for  $C_9H_{16}O_4Na^+$  [ $M + Na$ ] $^+$ : 211.0946; found: 211.0936. Analysis calculated for  $C_9H_{16}O_4$ : C 57.43, H 8.57%; found: C 57.51, H 8.50%. It is noted that the crystals grown from a diethyl ether solution under a pentane-saturated atmosphere were non-merohedral twins, and the structure is essentially the same as that reported here.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were positioned geometrically with C—H = 0.98–1.00 Å, and constrained to ride on their parent atoms with  $U_{iso}(H) = 1.2U_{eq}(C)$  or  $1.5U_{eq}(methyl\ C)$ . The hydroxy H atoms were placed guided by difference maps, with O—H = 0.84 Å and with  $U_{iso}(H) = 1.5U_{eq}(O)$ .

**Table 2**  
 Experimental details.

Crystal data	
Chemical formula	$C_9H_{16}O_4$
$M_r$	188.22
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	90
$a, b, c$ (Å)	7.7403 (5), 18.1498 (11), 6.7335 (5)
$\beta$ (°)	103.281 (2)
$V$ (Å <sup>3</sup> )	920.66 (11)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.11
Crystal size (mm)	0.28 × 0.27 × 0.25
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (SADABS; Bruker, 2014)
$T_{min}$ , $T_{max}$	0.97, 0.97
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	8165, 1612, 1205
$R_{int}$	0.037
$(\sin \theta/\lambda)_{max}$ (Å <sup>-1</sup> )	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.036, 0.092, 1.01
No. of reflections	1612
No. of parameters	121
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$ , $\Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.25, -0.27

Computer programs: APEX2 and SAINT (Bruker, 2014), SHELXS2013 (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015), Mercury (Macrae *et al.*, 2006), publCIF (Westrip, 2010) and PLATON (Spek, 2009).

## Acknowledgements

This research was partially supported by the Keio Gijuku Fukuzawa Memorial Fund for the Advancement of Education and Research. We also thank Professor S. Ohba (Keio University, Japan) for his valuable advice.

## References

- Bruker (2014). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Collins, D. J., Hibberd, A. I., Skelton, B. W. & White, A. H. (1998). *Aust. J. Chem.* **51**, 681–694.
- Fukaya, K., Kodama, K., Tanaka, Y., Yamazaki, H., Sugai, T., Yamaguchi, Y., Watanabe, A., Oishi, T., Sato, T. & Chida, N. (2015). *Org. Lett.* **17**, 2574–2577.
- Fukaya, K., Sugai, T., Sugai, T., Yamaguchi, Y., Watanabe, A., Yamamoto, H., Sato, T. & Chida, N. (2015). In preparation.
- Fukaya, K., Tanaka, Y., Sato, A. C., Kodama, K., Yamazaki, H., Ishimoto, T., Nozaki, Y., Iwaki, Y. M., Yuki, Y., Umei, K., Sugai, T., Yamaguchi, Y., Watanabe, A., Oishi, T., Sato, T. & Chida, N. (2015). *Org. Lett.* **17**, 2570–2573.
- Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
- Li, Y.-L., Pan, X.-F., Huang, W.-K., Wang, Y.-K. & Li, Y.-C. (1981). *Acta Chim. Sin.* **39**, 937–939.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Oishi, T., Fukaya, K., Yamaguchi, Y., Sugai, T., Watanabe, A., Sato, T. & Chida, N. (2015a). *Acta Cryst.* **E71**, 466–472.
- Oishi, T., Fukaya, K., Yamaguchi, Y., Sugai, T., Watanabe, A., Sato, T. & Chida, N. (2015b). *Acta Cryst.* **E71**, 490–493.

Oishi, T., Yamaguchi, Y., Fukaya, K., Sugai, T., Watanabe, A., Sato, T. & Chida, N. (2015). *Acta Cryst.* **E71**, 8–11.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.

Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.  
Wall, M. E. & Wani, M. C. (1995). *ACS Symp. Ser.* **583**, 18–30.  
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

## supporting information

*Acta Cryst.* (2015). E71, 1181-1184 [doi:10.1107/S2056989015016783]

## Crystal structure of (±)-(7*RS*,8*SR*)-7-methyl-1,4-dioxaspiro[4.5]decane-7,8-diol

Takeshi Oishi, Hiroaki Yamamoto, Tomoya Sugai, Keisuke Fukaya, Yu Yamaguchi, Ami Watanabe, Takaaki Sato and Noritaka Chida

### Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

### (±)-(7*RS*,8*SR*)-7-Methyl-1,4-dioxaspiro[4.5]decane-7,8-diol

#### Crystal data

C<sub>9</sub>H<sub>16</sub>O<sub>4</sub>

$M_r = 188.22$

Monoclinic,  $P2_1/c$

$a = 7.7403$  (5) Å

$b = 18.1498$  (11) Å

$c = 6.7335$  (5) Å

$\beta = 103.281$  (2)°

$V = 920.66$  (11) Å<sup>3</sup>

$Z = 4$

$F(000) = 408$

$D_x = 1.358$  Mg m<sup>-3</sup>

Melting point: 360.2 K

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

Cell parameters from 2733 reflections

$\theta = 2.7$ – $24.7$ °

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

$T = 90$  K

Prism, colorless

$0.28 \times 0.27 \times 0.25$  mm

#### Data collection

Bruker D8 Venture  
diffractometer

Radiation source: fine-focus sealed tube

Multilayered confocal mirror monochromator

Detector resolution: 10.4167 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2014)

$T_{\min} = 0.97$ ,  $T_{\max} = 0.97$

8165 measured reflections

1612 independent reflections

1205 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 25.0$ °,  $\theta_{\min} = 2.7$ °

$h = -9 \rightarrow 8$

$k = -21 \rightarrow 21$

$l = -8 \rightarrow 7$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.092$

$S = 1.01$

1612 reflections

121 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

$$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.4103P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.008$

$$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$$

### Special details

**Experimental.** IR (KBr) 3476, 3398, 2986, 2950, 2931, 2895, 1448, 1419, 1397, 1356, 1229, 1120, 1083, 1060, 1013, 952, 840, 696  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  (p.p.m.) 4.02–3.91 (m, 4H), 3.73 (s, 1H), 3.33 (ddd,  $J = 10.7, 10.6, 4.9$  Hz, 1H), 2.03 (d,  $J = 10.6$  Hz, 1H), 1.94–1.86 (m, 2H), 1.78–1.56 (m, 4H), 1.25 (d,  $J = 0.9$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  (p.p.m.) 108.7 (C), 74.0 (CH), 72.5 (C), 64.7 ( $\text{CH}_2$ ), 64.4 ( $\text{CH}_2$ ), 44.1 ( $\text{CH}_2$ ), 33.2 ( $\text{CH}_2$ ), 28.4 ( $\text{CH}_2$ ), 26.2 ( $\text{CH}_3$ ).

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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  $F^2 > \sigma(F^2)$  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. Problematic one reflection with  $|I(\text{obs})-I(\text{calc})|/\sigma W(I)$  greater than 10 (0 2 0) has been omitted in the final refinement.

### 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	0.27151 (15)	0.55328 (6)	1.12431 (17)	0.0191 (3)
C2	0.4342 (2)	0.58896 (9)	1.2186 (3)	0.0209 (4)
H2A	0.5326	0.5725	1.158	0.025*
H2B	0.4662	0.5796	1.3675	0.025*
C3	0.3914 (2)	0.66886 (9)	1.1734 (3)	0.0204 (4)
H3A	0.327	0.6902	1.271	0.025*
H3B	0.5002	0.698	1.1772	0.025*
O4	0.28135 (15)	0.66576 (6)	0.97209 (18)	0.0177 (3)
C5	0.1875 (2)	0.59633 (9)	0.9519 (2)	0.0154 (4)
C6	-0.0045 (2)	0.60871 (9)	0.9585 (2)	0.0139 (4)
H6A	-0.0085	0.6365	1.0839	0.017*
H6B	-0.0617	0.5603	0.9663	0.017*
C7	-0.1103 (2)	0.65073 (8)	0.7741 (3)	0.0139 (4)
C8	-0.0926 (2)	0.61203 (9)	0.5774 (2)	0.0141 (4)
H8	-0.1427	0.5614	0.5804	0.017*
C9	0.1009 (2)	0.60306 (9)	0.5692 (3)	0.0161 (4)
H9A	0.1551	0.6523	0.5649	0.019*
H9B	0.1079	0.5764	0.443	0.019*
C10	0.2047 (2)	0.56059 (9)	0.7546 (3)	0.0160 (4)
H10A	0.1598	0.5094	0.7496	0.019*
H10B	0.3315	0.5586	0.7499	0.019*
C11	-0.3033 (2)	0.65735 (10)	0.7850 (3)	0.0209 (4)
H11A	-0.3108	0.6838	0.9097	0.031*
H11B	-0.3544	0.608	0.7868	0.031*
H11C	-0.3692	0.6845	0.6658	0.031*
O12	-0.04355 (15)	0.72524 (6)	0.77185 (18)	0.0170 (3)
H12	0.0652	0.726	0.828	0.026*

O13	-0.19319 (15)	0.64738 (6)	0.40008 (17)	0.0179 (3)
H13	-0.1446	0.6875	0.3825	0.027*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0174 (6)	0.0186 (7)	0.0174 (7)	-0.0029 (5)	-0.0040 (5)	0.0046 (5)
C2	0.0167 (9)	0.0220 (10)	0.0206 (10)	-0.0018 (8)	-0.0024 (7)	-0.0001 (8)
C3	0.0197 (10)	0.0203 (10)	0.0186 (10)	-0.0022 (8)	-0.0012 (8)	-0.0022 (8)
O4	0.0167 (6)	0.0158 (6)	0.0178 (7)	-0.0057 (5)	-0.0021 (5)	0.0019 (5)
C5	0.0157 (9)	0.0124 (8)	0.0162 (9)	-0.0029 (7)	-0.0001 (7)	0.0041 (7)
C6	0.0165 (9)	0.0130 (9)	0.0130 (9)	-0.0021 (7)	0.0048 (7)	-0.0011 (7)
C7	0.0154 (9)	0.0099 (8)	0.0166 (10)	-0.0008 (7)	0.0042 (7)	0.0003 (7)
C8	0.0160 (9)	0.0114 (9)	0.0132 (9)	-0.0010 (7)	-0.0002 (7)	0.0010 (7)
C9	0.0178 (9)	0.0162 (9)	0.0147 (9)	-0.0001 (7)	0.0045 (7)	-0.0020 (7)
C10	0.0135 (9)	0.0164 (9)	0.0188 (10)	0.0001 (7)	0.0051 (7)	-0.0005 (7)
C11	0.0181 (9)	0.0223 (10)	0.0233 (10)	0.0017 (8)	0.0071 (8)	0.0005 (8)
O12	0.0176 (6)	0.0129 (6)	0.0195 (7)	-0.0011 (5)	0.0021 (5)	-0.0007 (5)
O13	0.0192 (7)	0.0171 (6)	0.0145 (7)	-0.0021 (5)	-0.0019 (5)	0.0036 (5)

*Geometric parameters (Å, °)*

O1—C5	1.4265 (19)	C7—C11	1.517 (2)
O1—C2	1.428 (2)	C7—C8	1.532 (2)
C2—C3	1.503 (2)	C8—O13	1.4200 (19)
C2—H2A	0.99	C8—C9	1.520 (2)
C2—H2B	0.99	C8—H8	1.0
C3—O4	1.427 (2)	C9—C10	1.529 (2)
C3—H3A	0.99	C9—H9A	0.99
C3—H3B	0.99	C9—H9B	0.99
O4—C5	1.4453 (19)	C10—H10A	0.99
C5—C10	1.512 (2)	C10—H10B	0.99
C5—C6	1.514 (2)	C11—H11A	0.98
C6—C7	1.526 (2)	C11—H11B	0.98
C6—H6A	0.99	C11—H11C	0.98
C6—H6B	0.99	O12—H12	0.84
C7—O12	1.4491 (19)	O13—H13	0.84
C5—O1—C2	107.70 (12)	O12—C7—C8	108.41 (13)
O1—C2—C3	102.52 (13)	C11—C7—C8	111.28 (14)
O1—C2—H2A	111.3	C6—C7—C8	109.66 (13)
C3—C2—H2A	111.3	O13—C8—C9	111.85 (13)
O1—C2—H2B	111.3	O13—C8—C7	112.27 (13)
C3—C2—H2B	111.3	C9—C8—C7	111.41 (13)
H2A—C2—H2B	109.2	O13—C8—H8	107.0
O4—C3—C2	102.13 (13)	C9—C8—H8	107.0
O4—C3—H3A	111.3	C7—C8—H8	107.0
C2—C3—H3A	111.3	C8—C9—C10	111.21 (14)

O4—C3—H3B	111.3	C8—C9—H9A	109.4
C2—C3—H3B	111.3	C10—C9—H9A	109.4
H3A—C3—H3B	109.2	C8—C9—H9B	109.4
C3—O4—C5	107.54 (12)	C10—C9—H9B	109.4
O1—C5—O4	105.99 (12)	H9A—C9—H9B	108.0
O1—C5—C10	111.37 (13)	C5—C10—C9	111.42 (13)
O4—C5—C10	108.24 (13)	C5—C10—H10A	109.3
O1—C5—C6	108.93 (13)	C9—C10—H10A	109.3
O4—C5—C6	110.10 (13)	C5—C10—H10B	109.3
C10—C5—C6	112.02 (13)	C9—C10—H10B	109.3
C5—C6—C7	113.40 (13)	H10A—C10—H10B	108.0
C5—C6—H6A	108.9	C7—C11—H11A	109.5
C7—C6—H6A	108.9	C7—C11—H11B	109.5
C5—C6—H6B	108.9	H11A—C11—H11B	109.5
C7—C6—H6B	108.9	C7—C11—H11C	109.5
H6A—C6—H6B	107.7	H11A—C11—H11C	109.5
O12—C7—C11	106.50 (13)	H11B—C11—H11C	109.5
O12—C7—C6	110.42 (13)	C7—O12—H12	109.5
C11—C7—C6	110.50 (14)	C8—O13—H13	109.5
C5—O1—C2—C3	30.97 (17)	C5—C6—C7—C8	53.80 (17)
O1—C2—C3—O4	-37.36 (16)	O12—C7—C8—O13	-61.28 (17)
C2—C3—O4—C5	30.55 (17)	C11—C7—C8—O13	55.53 (17)
C2—O1—C5—O4	-12.62 (16)	C6—C7—C8—O13	178.10 (12)
C2—O1—C5—C10	104.89 (15)	O12—C7—C8—C9	65.06 (16)
C2—O1—C5—C6	-131.06 (14)	C11—C7—C8—C9	-178.13 (13)
C3—O4—C5—O1	-12.23 (16)	C6—C7—C8—C9	-55.56 (17)
C3—O4—C5—C10	-131.81 (14)	O13—C8—C9—C10	-176.36 (12)
C3—O4—C5—C6	105.44 (15)	C7—C8—C9—C10	57.07 (18)
O1—C5—C6—C7	-176.78 (12)	O1—C5—C10—C9	174.98 (12)
O4—C5—C6—C7	67.39 (16)	O4—C5—C10—C9	-68.89 (16)
C10—C5—C6—C7	-53.12 (18)	C6—C5—C10—C9	52.69 (18)
C5—C6—C7—O12	-65.60 (17)	C8—C9—C10—C5	-55.09 (18)
C5—C6—C7—C11	176.83 (13)		

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>
O12—H12...O4	0.84	2.05	2.7838 (16)	146
O13—H13...O12 <sup>i</sup>	0.84	1.99	2.8093 (16)	166
C6—H6B...O1 <sup>ii</sup>	0.99	2.61	3.5631 (19)	162

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