data reports

1,2-O-Isopropylidene-β-D-*lyxo*-furanose

Bogdan Doboszewski[a] and Alexander Y. Nazarenko[b]*

[a]Departamento de Química, Universidade Federal Rural de Pernambuco, 52171-900 Recife, PE, Brazil, and [b]Chemistry Department, State University of New York, College at Buffalo, 1300 Elmwood Ave, Buffalo, NY 14222-1095, USA. *Correspondence e-mail: nazareay@buffalostate.edu

In the title compound $C_8H_{14}O_5$, the pentofuranose five-membered ring has a twisted conformation on two carbon atoms while the five-membered ring of the isopropylidene group has an envelope conformation on an oxygen atom. Hydroxy groups are involved an infinite network of O—H$\cdots$O hydrogen bonds that leads to the formation of a layer parallel to the (001) plane. Only weak C—H$\cdots$O contacts exist between neighboring layers.

3D view
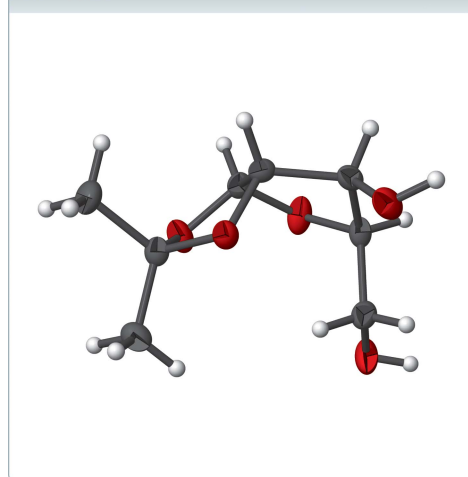
Chemical scheme
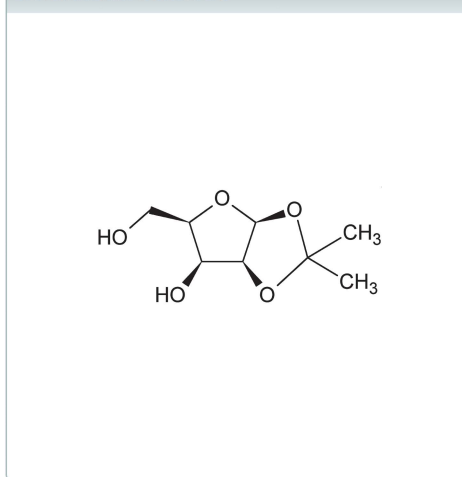
Structure description

The title compound, $C_8H_{14}O_5$, (**1**) together with its enantiomeric *L* form, are relatively rare derivatives and a limited volume of information is available for either of them. Our interest in **1** stems from the possibility of conducting deoxygenation at its C3 position to obtain 3-deoxy-1,2-O-isopropylidene-β-D-*threo*-pentofuranose as a chiral synthon for further synthetic work (Soares *et al.*, 2013). Compound **1** was obtained from the known 1,2-O-isopropylidene-5-O-t-butyldiphenylsilyl-β-D-*arabino*-furanose **2** (Dahlman *et al.*, 1986) *via* oxidation at the C3 position followed by reduction of the intermediate ulose. The reduction proceeded with a total stereoselection from the more accessible *Re* (α) side to furnish 1,2-O-isopropylidene-5-*O*–*t*-butyldiphenylsilyl-β-D-*lyxo*-furanose, whose desilylation gave the target **1**. It should be pointed out that under these isopropyl-idenation conditions, D-lyxose furnished only its α,β-2,3-O-isopropylidenefuranose (Barbat *et al.*, 1991). Compound **1** was previously obtained starting from D-glucose *via* D-gulose (Kuzuhara *et al.*, 1971). The scarcity of any experimental data on **1** prompted us to examine its structure.

In the title compound (Fig. 1), the pentofuranose five-membered ring has twisted conformation on atoms C6 and C9 [Q = 0.3175 (12) Å, φ = 117.6 (2)°]. The five-membered ring of the isopropylidene group has an envelope conformation on atom O1 [$Q(2)$ = 0.3192 (11) Å, φ = 187.1 (2)°]. Puckering parameters (Cremer & Pople, 1975)
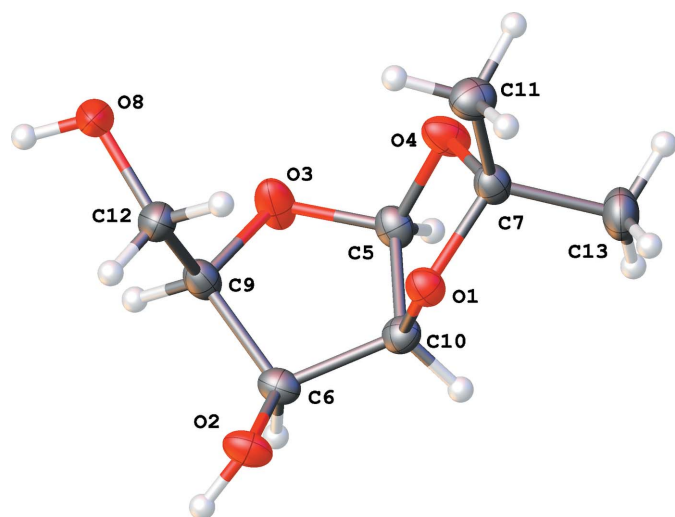
Figure 1
The title compound with the atom-numbering scheme and 50% probability displacement ellipsoids

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2—H2⋯O8[i]	0.88 (2)	1.72 (2)	2.5946 (15)	169 (2)
O8—H8⋯O2[ii]	0.84 (2)	1.86 (2)	2.6567 (16)	158 (2)
C12—H12B⋯O1	0.95 (2)	2.54 (2)	3.1908 (15)	126.1 (14)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{14}O_5$
M_r	190.19
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	173
a, b, c (Å)	5.9196 (3), 7.3562 (4), 21.1126 (12)
V (Å^3)	919.36 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.12
Crystal size (mm)	$0.45 \times 0.43 \times 0.37$
Data collection	
Diffractometer	Bruker PHOTON-100 CMOS
Absorption correction	Multi-scan (SADABS; Krause *et al.*, 2015)
T_{min}, T_{max}	0.944, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20693, 4938, 4094
R_{int}	0.029
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.862
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.109, 1.03
No. of reflections	4938
No. of parameters	174
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.41, −0.30
Absolute structure	Flack x determined using 1496 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.3 (2) **su large for Mo Kα?**

Computer programs: APEX2 and SAINT (Bruker, 2013), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), OLEX2 (Dolomanov *et al.*, 2009) and Mercury (Macrae *et al.*, 2020).

were calculated using *PLATON* (Spek, 2020). We have observed the same conformation of the isopropyledene fragment in other carbohydrates (Doboszewski & Nazarenko, 2003; Doboszewski *et al.*, 2010).

In the crystal, the two hydroxy groups form an infinite network of O—H⋯O hydrogen bonds that leads to the formation of a layer parallel to the (001) plane (Table 1, Fig. 2).
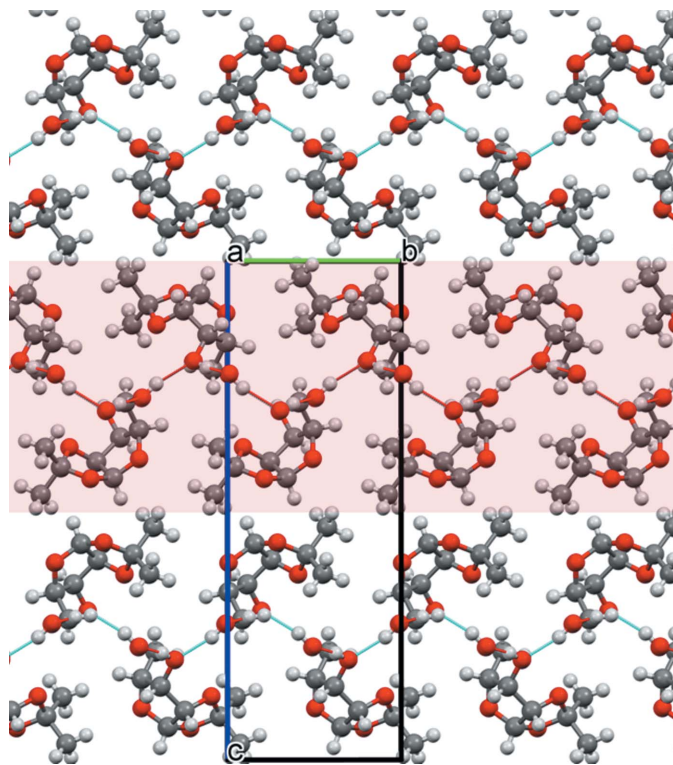
Only weak C—H⋯O (Fig. 3) contacts exist between neighboring layers; the C5⋯O4($-\frac{1}{2} + x, \frac{3}{2} - y, -z$) distance is 3.389 (2) Å. Similar hydrogen bonds have been observed in various carbohydrates (Desiraju & Steiner, 1999). A short intramolecular contact between oxygen O1 and the H12B atom of a neighboring methylene group (Table 1) may addi-
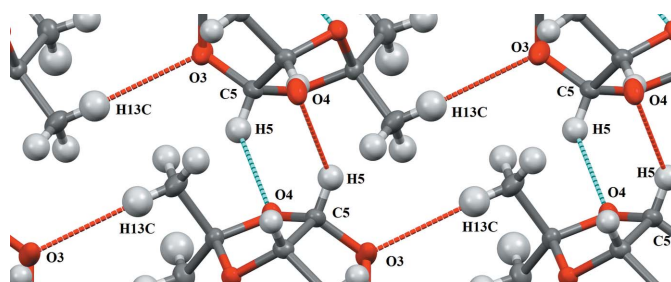

Figure 2
Packing diagram of the title compound; view along [100] vector. Highlighted are the layers of molecules connected *via* O—H⋯O hydrogen bonds.

Figure 3
Detail view of the intermolecular C—H⋯O interactions.

tionally stabilize the conformation of the molecule. Therefore, all oxygen atoms of the title molecule participate in O—H$\cdots$O or C—H$\cdots$O interactions.

Synthesis and crystallization

The synthesis of the title compound is described in Kuzuhara *et al.* (1971) and Soares *et al.* (2013).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. An additional dataset was collected using Cu $K\alpha$ radiation, resulting in a Flack parameter of 0.09 (13) and a probability of the absolute configuration being correct of 1.000.

Funding information

References

Barbat, J., Gelas, J. & Horton, D. (1991). *Carbohydr. Res.* **219**, 115–121.

Bruker (2013). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Dahlman, O., Garegg, P. J., Mayer, H. & Schramek, S. (1986). *Acta Chim. Scand. B* **40**, 15-20.

Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*. New York: Oxford University Press Inc.

Doboszewski, B. & Nazarenko, A. Y. (2003). *Acta Cryst.* E**59**, 158–160.

Doboszewski, B., Silva, P. R., Nazarenko, A. Y. & Nemykin, V. N. (2010). *Acta Cryst.* E**66**, o3217–o3218.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.

Kuzuhara, H., Terayama, H., Ohrui, H. & Emoto, S. (1971). *Carbohydr. Res.* **20**, 165–169.

Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Sheldrick, G. M. (2015a). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015b). *Acta Cryst.* C**71**, 3–8.

Soares, F. da Paixão, Silva, M. J. & Doboszewski, B. (2013). *Carbohydr. Res.* **380**, 143–148.

Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.

full crystallographic data

1,2-*O*-Isopropylidene-*β*-D-*lyxo*-furanose

Bogdan Doboszewski and Alexander Y. Nazarenko

1,2-*O*-Isopropylidene-*β*-D-*lyxo*-furanose

Crystal data

$C_8H_{14}O_5$
$M_r = 190.19$
Orthorhombic, $P2_12_12_1$
$a = 5.9196 (3)$ Å
$b = 7.3562 (4)$ Å
$c = 21.1126 (12)$ Å
$V = 919.36 (9)$ Å^3
$Z = 4$
$F(000) = 408$

$D_x = 1.374$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9980 reflections
$\theta = 2.9$–$37.3°$
$\mu = 0.12$ mm^{-1}
$T = 173$ K
Block, colourless
$0.45 \times 0.43 \times 0.37$ mm

Data collection

Bruker PHOTON-100 CMOS diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 10.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min} = 0.944$, $T_{max} = 1.000$

20693 measured reflections
4938 independent reflections
4094 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 37.8°$, $\theta_{min} = 2.9°$
$h = -9 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 36$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.109$
$S = 1.03$
4938 reflections
174 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2 + 0.0319P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}
Absolute structure: Flack x determined using 1496 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.3 (2)

Special details

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

Refinement. All hydrogen atoms are refined in isotropic approximation.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.94830 (15)	0.59218 (11)	0.12758 (4)	0.02153 (15)
O2	0.66312 (15)	0.80371 (13)	0.20056 (4)	0.02597 (18)
H2	0.556 (4)	0.878 (3)	0.2138 (10)	0.044 (6)*
O3	1.1047 (2)	0.99300 (12)	0.10435 (5)	0.0312 (2)
O4	1.21881 (17)	0.71302 (12)	0.06429 (5)	0.02782 (19)
O8	1.31943 (18)	1.00933 (18)	0.22671 (6)	0.0398 (3)
H8	1.294 (4)	1.090 (3)	0.2540 (11)	0.044 (6)*
C5	1.0438 (2)	0.84268 (15)	0.06643 (5)	0.02228 (19)
H5	1.021 (4)	0.887 (3)	0.0247 (9)	0.034 (5)*
C6	0.7654 (2)	0.88480 (15)	0.14745 (5)	0.02169 (19)
H6	0.661 (3)	0.966 (3)	0.1270 (9)	0.029 (5)*
C7	1.1331 (2)	0.54276 (15)	0.08756 (5)	0.02213 (19)
C9	0.9802 (2)	0.99245 (15)	0.16300 (5)	0.0234 (2)
H9	0.946 (3)	1.122 (2)	0.1724 (8)	0.021 (4)*
C10	0.8456 (2)	0.74699 (15)	0.09853 (5)	0.02134 (18)
H10	0.728 (4)	0.710 (3)	0.0702 (8)	0.029 (5)*
C11	1.3131 (2)	0.45089 (18)	0.12639 (7)	0.0291 (2)
H11A	1.446 (5)	0.431 (4)	0.0981 (11)	0.057 (7)*
H11B	1.367 (4)	0.524 (3)	0.1593 (11)	0.039 (5)*
H11C	1.264 (4)	0.337 (3)	0.1401 (9)	0.040 (5)*
C12	1.1131 (2)	0.91394 (18)	0.21779 (6)	0.0272 (2)
H12A	1.027 (4)	0.919 (3)	0.2536 (9)	0.027 (4)*
H12B	1.143 (3)	0.789 (3)	0.2097 (9)	0.030 (5)*
C13	1.0527 (3)	0.4265 (2)	0.03239 (7)	0.0335 (3)
H13A	0.942 (4)	0.487 (3)	0.0070 (10)	0.038 (5)*
H13B	1.178 (4)	0.399 (3)	0.0052 (11)	0.039 (5)*
H13C	0.997 (4)	0.314 (3)	0.0495 (11)	0.048 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0226 (4)	0.0202 (3)	0.0218 (3)	−0.0006 (3)	0.0036 (3)	0.0012 (3)
O2	0.0209 (4)	0.0287 (4)	0.0283 (4)	0.0024 (3)	0.0084 (3)	0.0056 (3)
O3	0.0406 (5)	0.0230 (4)	0.0302 (4)	−0.0094 (4)	0.0161 (4)	−0.0036 (3)
O4	0.0254 (4)	0.0216 (3)	0.0364 (4)	0.0009 (3)	0.0104 (3)	0.0034 (3)
O8	0.0203 (4)	0.0466 (6)	0.0527 (6)	0.0001 (4)	0.0012 (4)	−0.0295 (5)
C5	0.0271 (5)	0.0219 (4)	0.0178 (4)	0.0010 (4)	0.0032 (4)	0.0018 (3)
C6	0.0197 (4)	0.0237 (4)	0.0216 (4)	0.0030 (4)	0.0022 (3)	0.0031 (4)
C7	0.0245 (5)	0.0190 (4)	0.0229 (4)	−0.0015 (3)	0.0036 (4)	−0.0009 (3)
C9	0.0254 (5)	0.0200 (4)	0.0248 (4)	−0.0010 (4)	0.0074 (4)	−0.0021 (4)
C10	0.0204 (4)	0.0242 (4)	0.0194 (4)	−0.0005 (3)	−0.0017 (3)	0.0006 (3)
C11	0.0261 (5)	0.0259 (5)	0.0351 (6)	0.0023 (4)	−0.0018 (5)	0.0018 (5)
C12	0.0228 (5)	0.0307 (5)	0.0280 (5)	−0.0011 (4)	−0.0006 (4)	−0.0080 (4)
C13	0.0401 (7)	0.0305 (6)	0.0298 (5)	0.0008 (5)	−0.0020 (5)	−0.0109 (5)

Geometric parameters (Å, º)

O1—C7	1.4291 (14)	C6—C10	1.5230 (16)
O1—C10	1.4292 (14)	C7—C11	1.5049 (18)
O2—H2	0.88 (3)	C7—C13	1.5214 (17)
O2—C6	1.4071 (14)	C9—H9	0.993 (17)
O3—C5	1.4120 (14)	C9—C12	1.5135 (18)
O3—C9	1.4409 (14)	C10—H10	0.95 (2)
O4—C5	1.4090 (15)	C11—H11A	1.00 (3)
O4—C7	1.4380 (14)	C11—H11B	0.94 (2)
O8—H8	0.84 (3)	C11—H11C	0.93 (3)
O8—C12	1.4210 (17)	C12—H12A	0.91 (2)
C5—H5	0.947 (19)	C12—H12B	0.95 (2)
C5—C10	1.5267 (16)	C13—H13A	0.96 (2)
C6—H6	0.96 (2)	C13—H13B	0.96 (2)
C6—C9	1.5334 (17)	C13—H13C	0.96 (3)
C10—O1—C7	105.93 (8)	C12—C9—C6	113.43 (10)
C6—O2—H2	107.3 (14)	C12—C9—H9	108.6 (10)
C5—O3—C9	110.76 (9)	O1—C10—C5	103.36 (9)
C5—O4—C7	108.61 (9)	O1—C10—C6	111.84 (8)
C12—O8—H8	106.6 (17)	O1—C10—H10	110.7 (12)
O3—C5—H5	107.2 (12)	C5—C10—H10	114.3 (11)
O3—C5—C10	107.79 (9)	C6—C10—C5	103.51 (9)
O4—C5—O3	111.14 (10)	C6—C10—H10	112.6 (12)
O4—C5—H5	107.7 (13)	C7—C11—H11A	107.4 (15)
O4—C5—C10	105.50 (9)	C7—C11—H11B	112.8 (14)
C10—C5—H5	117.5 (14)	C7—C11—H11C	110.7 (13)
O2—C6—H6	110.1 (12)	H11A—C11—H11B	105 (2)
O2—C6—C9	113.91 (9)	H11A—C11—H11C	107 (2)
O2—C6—C10	113.10 (9)	H11B—C11—H11C	113.2 (18)
C9—C6—H6	107.9 (12)	O8—C12—C9	111.08 (11)
C10—C6—H6	108.1 (12)	O8—C12—H12A	110.5 (13)
C10—C6—C9	103.33 (9)	O8—C12—H12B	110.0 (12)
O1—C7—O4	104.51 (8)	C9—C12—H12A	109.2 (12)
O1—C7—C11	109.53 (10)	C9—C12—H12B	109.1 (12)
O1—C7—C13	110.88 (10)	H12A—C12—H12B	106.9 (16)
O4—C7—C11	109.11 (10)	C7—C13—H13A	112.5 (13)
O4—C7—C13	109.77 (10)	C7—C13—H13B	109.5 (14)
C11—C7—C13	112.71 (11)	C7—C13—H13C	107.8 (13)
O3—C9—C6	103.98 (9)	H13A—C13—H13B	107.1 (18)
O3—C9—H9	105.9 (10)	H13A—C13—H13C	112 (2)
O3—C9—C12	113.08 (11)	H13B—C13—H13C	108 (2)
C6—C9—H9	111.5 (11)		
O2—C6—C9—O3	155.65 (9)	C6—C9—C12—O8	175.05 (9)
O2—C6—C9—C12	32.41 (13)	C7—O1—C10—C5	−31.62 (10)
O2—C6—C10—O1	−41.18 (13)	C7—O1—C10—C6	−142.34 (9)

O2—C6—C10—C5	−151.81 (9)	C7—O4—C5—O3	121.62 (10)
O3—C5—C10—O1	−102.57 (10)	C7—O4—C5—C10	5.06 (12)
O3—C5—C10—C6	14.19 (12)	C9—O3—C5—O4	−108.31 (11)
O3—C9—C12—O8	56.97 (13)	C9—O3—C5—C10	6.84 (13)
O4—C5—C10—O1	16.25 (11)	C9—C6—C10—O1	82.45 (11)
O4—C5—C10—C6	133.01 (9)	C9—C6—C10—C5	−28.18 (11)
C5—O3—C9—C6	−24.89 (13)	C10—O1—C7—O4	35.34 (11)
C5—O3—C9—C12	98.58 (12)	C10—O1—C7—C11	152.12 (9)
C5—O4—C7—O1	−24.63 (12)	C10—O1—C7—C13	−82.87 (11)
C5—O4—C7—C11	−141.71 (10)	C10—C6—C9—O3	32.55 (11)
C5—O4—C7—C13	94.33 (12)	C10—C6—C9—C12	−90.68 (11)

Hydrogen-bond geometry (Å, º)

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2$\cdots$O8[i]	0.88 (2)	1.72 (2)	2.5946 (15)	169 (2)
O8—H8$\cdots$O2[ii]	0.84 (2)	1.86 (2)	2.6567 (16)	158 (2)
C12—H12*B*$\cdots$O1	0.95 (2)	2.54 (2)	3.1908 (15)	126.1 (14)

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