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Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of an epoxysterol: 9*α*,11*α*-epoxy-5*α*-cholest-7-ene-3*β*,5,6*α*-triol 3,6-diacetate

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Received 9 September 2017
Accepted 19 September 2017

Edited by C. Rizzoli, Universita degli Studi di Parma, Italy

Keywords: crystal structure; steroids; hydrogen bond.

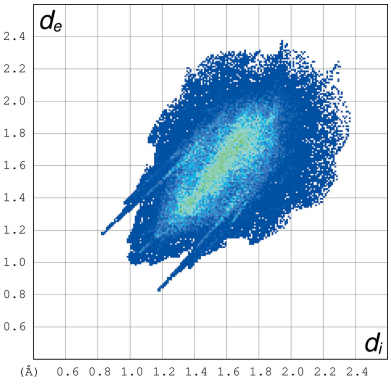
CCDC reference: 1575390

Supporting information: this article has supporting information at journals.iucr.org/e

The title compound, $C_{31}H_{48}O_6$, is a polyoxygenated epoxy steroid obtained by a multi-step synthesis involving oxidation of 7-dehydrocholesterol. It crystallizes in the $P2_12_12_1$ space group; however, the absolute structure of the molecule in the crystal could not be determined by resonant scattering. The configuration at the C5 and C6 positions is in both cases of the α-type, as is that of the C atoms of the epoxy ring. Molecules in the crystal form chains parallel to the *b* axis by hydrogen bonding between O—H donors and carbonyl O-atom acceptors. Some atoms of the alkyl chain are disordered over two orientations, with a refined occupancy ratio of 0.511 (10):0.489 (10).

1. Chemical context

Polyoxygenated steroids (Fig. 1) are metabolites both of terrestrial and marine origin possessing a number of remarkable biological activities (D'Auria *et al.*, 1993). Our previous studies in this field focused on the isolation and synthesis of a number of such substances possessing new nuclear oxygenation patterns (Madaio *et al.*, 1988; Migliuolo *et al.*, 1992). In this context, new ruthenium tetroxide-catalysed oxidation methods (Bifulco *et al.*, 2003*a*,*b*; Piccialli *et al.*, 2007, 2010; Piccialli, 2014) were developed to introduce suitable oxygenated functionalities in the *B*, *C* and *D* rings of the steroid nucleus. Among others, 9,11-epoxysterols have been isolated from various marine organisms (Gunasekera *et al.*, 1983) and display diverse biological activities. In particular, the 3-deacetyl analogue of the title compound (Fig. 1) has shown to inhibit the binding of [I125] IL-8 to the human recombinant IL-8 receptor type A (de Almeida Leone *et al.*, 2000).



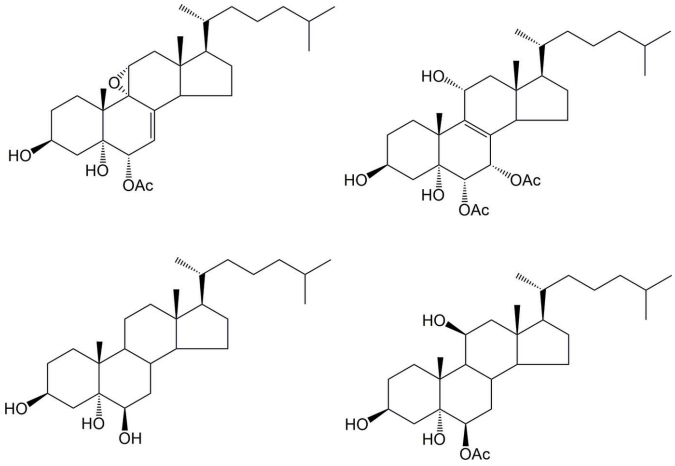


Figure 1
Selected biologically active polyoxygenated steroids of marine origin.

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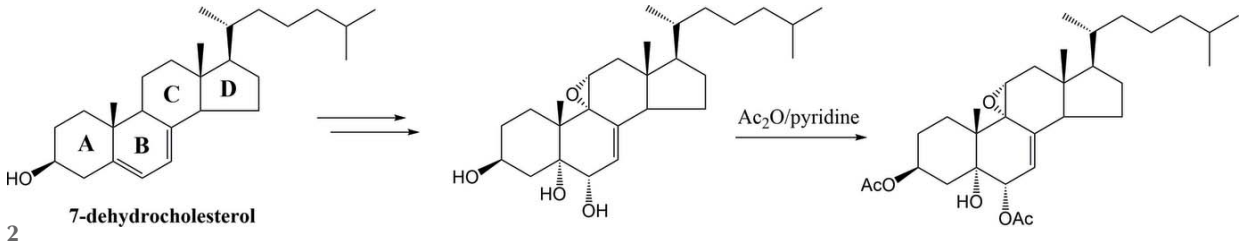
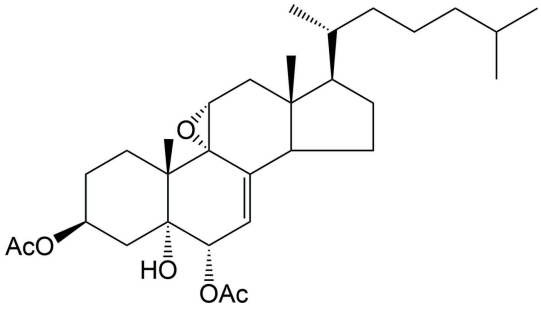


Figure 2
Synthesis of the title compound.

We are carrying out a broad research program aimed at discovering new biologically active substances. In recent years, we have synthesized and studied, among others, purine nucleoside analogues (D'Errico *et al.*, 2011, 2012*a*,*b*; D'Atri *et al.*, 2012; Oliviero *et al.*, 2008, 2010*a*,*b*), cyclic ethers and polyethers (Piccialli *et al.*, 2007, 2009; Piccialli, D'Errico *et al.*, 2013; Piccialli, 2014) and nitrogen-rich fused-ring compounds (Centore *et al.*, 2013). Within this program, and on the basis of the reduced amount of direct structural information available on epoxy steroids, we have synthesized the title compound (**1**), by diacetylation of **3**, in turn obtained from cheap commercially available 7-dehydrocholesterol (**2**) (see Fig. 2), according to a previously reported procedure (Migliuolo *et al.*, 1991). In particular, during the synthesis, two diastereomers, with opposite configuration at C6, were obtained, with predominance of the *trans*-isomer (5α-OH/6β-OH). The structural analysis was performed in order to unambiguously assign the configuration of the title compound.



2. Structural commentary

The crystallographically independent molecule is shown in Fig. 3. From the figure it is evident that the two acetyloxy groups have a different stereochemical orientation (3β,6α) and that the stereochemical orientation of the hydroxy group is the same as that of the acetyloxy group at C6 (5α,6α). In addition, the orientation of the epoxy oxygen atom is on the opposite side as compared with the methyl groups C18 and C19 (9α,11α-epoxy) and on the same side of the hydroxy group bonded to C5. The stereoselectivity in the formation of the epoxy ring is probably related to the steric hindrance due to the methyl groups.

We have reported the crystal structure of a steroid closely related to the title compound (Piccialli, Tuzi *et al.*, 2013), in which the two acetyloxy groups, the C18 and C19 methyl groups and the alkyl tail have the same configuration as in the present one, and, moreover, an α hydroxy group at C9 and a keto group at C11 are present. In Fig. 4 the two molecular structures are superimposed. The superposition is very good, apart for a small difference in the torsion angle for the acetyl group at C3.

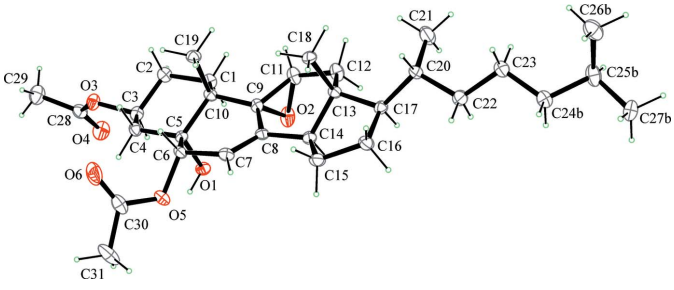


Figure 3
ORTEP view of the molecular structure of the title compound. Displacement ellipsoids are drawn at 30% probability level. Only the most populated orientation of the disordered chain is shown.

3. Supramolecular features

The crystal packing of the title compound is shown in Fig. 5. Molecules in the crystal form chains by hydrogen bonding between the alcohol O1—H donor and the O4 carbonyl acceptor (Table 1). The chains run parallel to the *b* axis and are wrapped around a 2_1 crystallographic screw axes. Adjacent chains along the *a* axis are held by weak hydrogen bonding between C29—H donor and O6 carbonyl acceptor.

In order to detect additional packing features, we have examined the Hirshfeld surface (Spackman & McKinnon, 2002; Wolff *et al.*, 2012). In Fig. 6 the Hirshfeld fingerprint plot of the independent molecule is reported. In the plot, for each

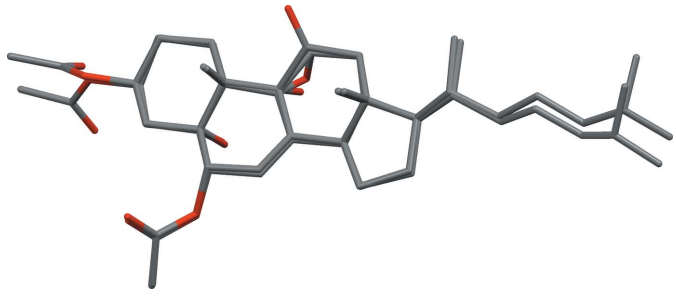


Figure 4
Overlay of the X-ray molecular structure of the title compound with the previously reported 3β,6α-diacetoxy-5,9α-dihydroxy-5α-cholest-7-en-11-one (Piccialli, Tuzi *et al.*, 2013).

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

D—H···A	D—H	H···A	D···A	D—H···A
C29—H29B···O6^{i}	0.98	2.61	3.567 (6)	166
O1—H1O···O4^{ii}	0.81 (3)	2.16 (4)	2.923 (3)	158 (4)

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

point of the Hirshfeld surface enveloping the molecule in the crystal, the distance d_i to the nearest atom inside the surface and the distance d_e to the nearest atom outside the surface are shown. The color of each point in the plot is related to the abundance of that interaction, from blue (low) to green (high) to red (very high).

A distinctive feature of the plot is represented by the two blue spikes at $d_i + d_e = 2.0$ Å, pointing to the lower left of the plot and symmetrically disposed with respect to the diagonal. They correspond to the strong hydrogen bonds present in the packing. Another feature is the central green strip along the diagonal, centered at $d_i + d_e = 3.2$ Å, indicating a large number of loose H···H contacts. As expected, they are the predominant intermolecular contacts in the packing of the title compound. The central green strip ends up in the blue sting at at $d_i = d_e = 1.0$ Å, which reflects points on the Hirshfeld surface that involve nearly head-to-head close H···H contacts.

4. Database survey

A search of the Cambridge Structural Database (CSD version 5.38, last update February 2017; Groom *et al.*, 2016) gave no match for the title compound. We have searched, within steroids with a double bond at C7 (122 hits in total), for an additional epoxy group in any of the *A*, *B*, *C* and *D* rings of the steroid moiety. We found eight hits, with the following refcodes and position of the epoxy ring: EZELAX (4β,5β-epoxy), DIZPUY and FIWYUG (9α,11α-epoxy), RUGDIH (9α,13α-epoxy), POHDEW (9α,14α-epoxy), QULRAS and QULRIA (13α,17α-epoxy), BEXCHO (14α,15α-epoxy).

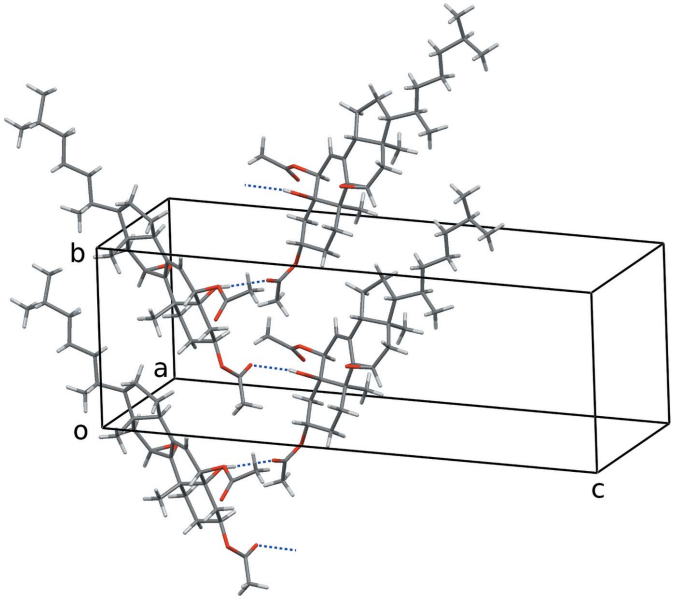


Figure 5
Partial crystal packing of the title compound. Only the most populated orientation of the disordered chain is shown.

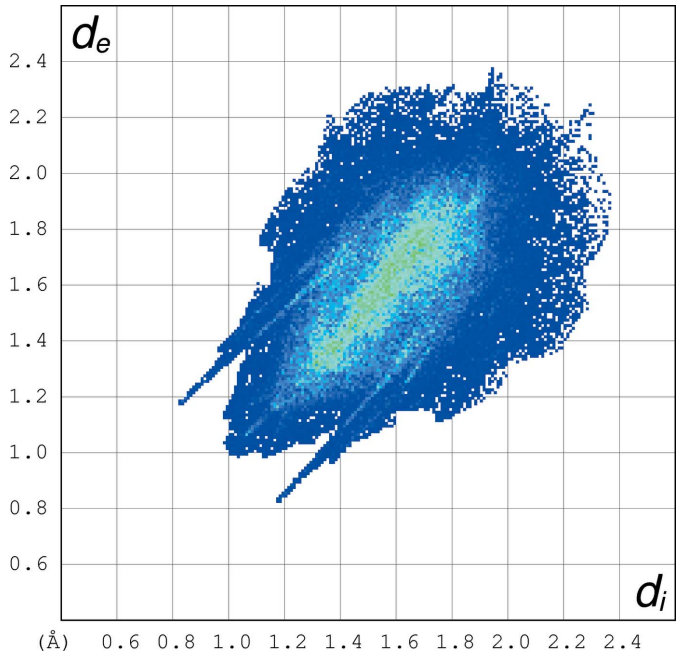


Figure 6
Hirshfeld fingerprint plot of the crystallographically independent molecule of the title compound.

5. Synthesis and crystallization

5α-Cholest-7-ene-3β,5,6α-triol 3,6-diacetate was obtained from 7-dehydrocholesterol as described (Fieser *et al.*, 1953; Migliuolo *et al.*, 1991), followed by acetylation. Mercuric acetate dehydrogenation gave the $\Delta^{7,9(11)}$-analogue. Hydrolytic deacetylation, MnO_2 oxidation at C6 and subsequent *meta*-chloroperbenzoic acid epoxidation at the C9—C11 double bond gave 9α,11α-epoxy-3β,5-dihydroxy-5α-cholest-7-en-6-one. $LiAlH_4$ reduction of the C6 ketone function in the latter, followed by acetylation with Ac_2O/py, furnished the title compound **1** and its C6 epimer, in a 1:4 ratio. The pure title compound was obtained by HPLC separation ($CHCl_3$/MeOH, 96:4 *v*/*v*). The compound was dissolved in a minimal amount of $CHCl_3$ and the solution was left to evaporate slowly at room temperature to give crystals suitable for X-ray diffraction analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms were generated stereochemically and were refined by the riding model. The alcohol H atom was refined freely with U_{iso}(H) =1.2U_{eq}(O). All other H atoms were refined with U_{iso}(H) = 1.2U_{eq}(C) or

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{31}H_{48}O_6$
M_r	516.69
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	173
a, b, c (Å)	9.8990 (13), 10.1030 (16), 28.961 (6)
V (Å^3)	2896.4 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.50 × 0.30 × 0.10
Data collection	
Diffractometer	Bruker–Nonius KappaCCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2001)
T_{min}, T_{max}	0.949, 0.980
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14033, 5983, 3810
R_{int}	0.060
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.058, 0.121, 1.02
No. of reflections	5983
No. of parameters	364
No. of restraints	9
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.19
Absolute structure	Flack x determined using 3518 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−1.6 (8)

Computer programs: *COLLECT* (Nonius, 1999), *DIRAX/LSQ* (Duisenberg *et al.*, 2000), *EVALCCD* (Duisenberg *et al.*, 2003), *SIR97* (Altomare *et al.*, 1999), *SHELXL2016* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006).

1.5U_{eq}(C) for methyl H atoms. A rotating model was used for most methyl groups. The C25 and C26 atoms of the alkyl chain are disordered over two orientations. The two split positions were refined by applying DFIX and SAME restraints on bond lengths. The final refined occupancy factors of the two components of disorder are 0.511 (10) and 0.489 (10).

Acknowledgements

The authors thank the Centro Regionale di Competenza NTAP of Regione Campania (Italy) for the X-ray facility.

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supporting information

Acta Cryst. (2017). E**73**, 1603-1606 [https://doi.org/10.1107/S2056989017013391]

Crystal structure of an epoxysterol: 9*α*,11*α*-epoxy-5*α*-cholest-7-ene-3*β*,5,6*α*-triol 3,6-diacetate

Vincenzo Piccialli, Angela Tuzi and Roberto Centore

Computing details

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

9*α*,11*α*-Epoxy-5*α*-cholest-7-ene-3*β*,5,6*α*-triol 3,6-diacetate

Crystal data

$C_{31}H_{48}O_6$
$M_r = 516.69$
Orthorhombic, $P2_12_12_1$
$a = 9.8990$ (13) Å
$b = 10.1030$ (16) Å
$c = 28.961$ (6) Å
$V = 2896.4$ (8) Å^3
$Z = 4$
$F(000) = 1128$
$D_x = 1.185$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 125 reflections
$\theta = 4.7$–$20.3°$
$\mu = 0.08$ mm^{-1}
$T = 173$ K
Prism, colourless
0.50 × 0.30 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.949$, $T_{max} = 0.980$
14033 measured reflections
5983 independent reflections
3810 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -37 \rightarrow 34$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.121$
$S = 1.02$
5983 reflections
364 parameters
9 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.1235P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Absolute structure: Flack *x* determined using 3518 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −1.6 (8)

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. Some atoms of the alkyl chain are disordered over two orientations. The two split positions were refined by applying DFIX and SAME restraints on bond lengths.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.5882 (4)	0.0382 (4)	0.39740 (12)	0.0315 (9)	
H1A	0.578278	0.020716	0.430868	0.038*	
H1B	0.503001	0.078643	0.386368	0.038*	
C2	0.6100 (4)	−0.0926 (4)	0.37238 (11)	0.0332 (9)	
H2A	0.689079	−0.138333	0.385898	0.040*	
H2B	0.529903	−0.149765	0.376963	0.040*	
C3	0.6330 (3)	−0.0730 (4)	0.32144 (12)	0.0312 (9)	
H3	0.547768	−0.042675	0.306208	0.037*	
C4	0.7455 (4)	0.0246 (3)	0.31225 (11)	0.0292 (8)	
H4A	0.751780	0.040959	0.278617	0.035*	
H4B	0.832304	−0.014254	0.322506	0.035*	
C5	0.7231 (3)	0.1563 (3)	0.33721 (11)	0.0245 (8)	
C6	0.8402 (3)	0.2523 (4)	0.32857 (10)	0.0261 (8)	
H6	0.928232	0.207118	0.334802	0.031*	
C7	0.8297 (4)	0.3746 (4)	0.35694 (11)	0.0284 (8)	
H7	0.881140	0.449469	0.347731	0.034*	
C8	0.7527 (3)	0.3857 (3)	0.39442 (10)	0.0253 (8)	
C9	0.6743 (3)	0.2708 (4)	0.41170 (11)	0.0268 (8)	
C10	0.7045 (3)	0.1369 (4)	0.39006 (11)	0.0253 (8)	
C11	0.6195 (4)	0.2791 (4)	0.45941 (12)	0.0352 (9)	
H11	0.603 (4)	0.188 (4)	0.4759 (11)	0.042*	
C12	0.6379 (4)	0.3972 (4)	0.49048 (12)	0.0365 (10)	
H12A	0.658941	0.365861	0.522048	0.044*	
H12B	0.551775	0.446946	0.491938	0.044*	
C13	0.7498 (3)	0.4907 (3)	0.47455 (10)	0.0246 (8)	
C14	0.7382 (4)	0.5105 (3)	0.42192 (11)	0.0284 (8)	
H14	0.645043	0.544747	0.415891	0.034*	
C15	0.8356 (4)	0.6248 (4)	0.41196 (12)	0.0371 (9)	
H15A	0.806930	0.674854	0.384257	0.045*	
H15B	0.928634	0.591659	0.407143	0.045*	
C16	0.8273 (4)	0.7115 (4)	0.45544 (11)	0.0375 (9)	
H16A	0.785398	0.797861	0.447990	0.045*	
H16B	0.918915	0.727750	0.467961	0.045*	

C17	0.7402 (4)	0.6359 (3)	0.49123 (10)	0.0275 (8)	
H17	0.644573	0.664645	0.486340	0.033*	
C18	0.8863 (3)	0.4285 (4)	0.48704 (11)	0.0306 (9)	
H18A	0.890760	0.414213	0.520477	0.046*	
H18B	0.959417	0.488029	0.477586	0.046*	
H18C	0.895925	0.343564	0.471051	0.046*	
C19	0.8353 (3)	0.0858 (4)	0.41279 (11)	0.0308 (9)	
H19A	0.907044	0.151705	0.408956	0.046*	
H19B	0.862571	0.002624	0.398108	0.046*	
H19C	0.819323	0.070664	0.445769	0.046*	
C20	0.7777 (4)	0.6741 (3)	0.54075 (10)	0.0292 (9)	
H20	0.876547	0.657338	0.544856	0.035*	
C21	0.7026 (5)	0.5940 (4)	0.57697 (13)	0.0500 (12)	
H21A	0.605085	0.605209	0.572699	0.075*	
H21B	0.728257	0.624897	0.607826	0.075*	
H21C	0.726093	0.500211	0.573752	0.075*	
C22	0.7531 (4)	0.8226 (3)	0.54731 (11)	0.0347 (9)	
H22A	0.810582	0.870355	0.524839	0.042*	
H22B	0.657993	0.840976	0.538966	0.042*	
C23	0.7786 (5)	0.8819 (4)	0.59447 (12)	0.0444 (11)	
H23A	0.721681	0.835638	0.617489	0.053*	
H23B	0.874292	0.867234	0.603021	0.053*	
C24A	0.7485 (4)	1.0284 (4)	0.59630 (11)	0.0338 (9)	0.489 (10)
H24A	0.649973	1.040263	0.592132	0.041*	0.489 (10)
H24B	0.793553	1.070903	0.569625	0.041*	0.489 (10)
C25A	0.7905 (9)	1.1033 (6)	0.6400 (2)	0.032 (3)	0.489 (10)
H25A	0.891186	1.107770	0.642078	0.038*	0.489 (10)
C26A	0.7327 (11)	1.0433 (8)	0.6837 (2)	0.053 (3)	0.489 (10)
H26A	0.762730	1.094987	0.710463	0.080*	0.489 (10)
H26B	0.633834	1.044421	0.682156	0.080*	0.489 (10)
H26C	0.764281	0.951815	0.686827	0.080*	0.489 (10)
C27A	0.7307 (5)	1.2442 (4)	0.63715 (13)	0.0569 (12)	0.489 (10)
H27A	0.764570	1.288414	0.609352	0.085*	0.489 (10)
H27B	0.631960	1.238800	0.635799	0.085*	0.489 (10)
H27C	0.757736	1.294718	0.664517	0.085*	0.489 (10)
C24B	0.7485 (4)	1.0284 (4)	0.59630 (11)	0.0338 (9)	0.511 (10)
H24C	0.666532	1.044727	0.577562	0.041*	0.511 (10)
H24D	0.824045	1.075446	0.581051	0.041*	0.511 (10)
C25B	0.7268 (9)	1.0915 (8)	0.6433 (3)	0.045 (3)	0.511 (10)
H25B	0.638104	1.063287	0.656574	0.054*	0.511 (10)
C26B	0.8410 (10)	1.0565 (8)	0.6758 (3)	0.053 (3)	0.511 (10)
H26D	0.825127	1.098150	0.705880	0.080*	0.511 (10)
H26E	0.845221	0.960244	0.679577	0.080*	0.511 (10)
H26F	0.926589	1.088595	0.662989	0.080*	0.511 (10)
C27B	0.7307 (5)	1.2442 (4)	0.63715 (13)	0.0569 (12)	0.511 (10)
H27D	0.658127	1.271625	0.616239	0.085*	0.511 (10)
H27E	0.718428	1.286945	0.667230	0.085*	0.511 (10)
H27F	0.818132	1.270241	0.624124	0.085*	0.511 (10)

C28	0.5848 (4)	−0.2749 (4)	0.28055 (11)	0.0342 (9)
C29	0.6447 (4)	−0.4026 (4)	0.26568 (15)	0.0505 (12)
H29A	0.622844	−0.471371	0.288356	0.076*
H29B	0.743044	−0.393341	0.263366	0.076*
H29C	0.607876	−0.427560	0.235501	0.076*
C30	0.9279 (4)	0.2477 (5)	0.25108 (13)	0.0432 (11)
C31	0.9098 (5)	0.3112 (5)	0.20457 (13)	0.0621 (14)
H31A	0.925215	0.406701	0.207154	0.093*
H31B	0.817627	0.295117	0.193519	0.093*
H31C	0.974573	0.273129	0.182708	0.093*
O1	0.5995 (2)	0.2137 (2)	0.32194 (8)	0.0315 (6)
H1O	0.603 (4)	0.228 (4)	0.2944 (12)	0.038*
O2	0.5323 (2)	0.3002 (3)	0.42006 (9)	0.0422 (7)
O3	0.6766 (2)	−0.1988 (2)	0.30181 (8)	0.0344 (6)
O4	0.4689 (3)	−0.2405 (3)	0.27494 (9)	0.0496 (7)
O5	0.8345 (2)	0.2948 (2)	0.28049 (7)	0.0348 (6)
O6	1.0105 (3)	0.1650 (4)	0.26133 (10)	0.0613 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0277 (19)	0.034 (2)	0.0332 (19)	−0.0058 (18)	0.0024 (16)	−0.0028 (17)
C2	0.032 (2)	0.033 (2)	0.034 (2)	−0.0058 (19)	−0.0011 (17)	−0.0019 (17)
C3	0.026 (2)	0.032 (2)	0.035 (2)	0.0013 (17)	−0.0072 (16)	−0.0072 (17)
C4	0.0267 (18)	0.035 (2)	0.0264 (18)	0.0015 (18)	−0.0013 (15)	−0.0047 (16)
C5	0.0174 (17)	0.032 (2)	0.0243 (17)	0.0050 (16)	−0.0050 (14)	0.0002 (15)
C6	0.0251 (18)	0.034 (2)	0.0190 (17)	0.0006 (17)	0.0008 (14)	−0.0001 (16)
C7	0.0250 (18)	0.033 (2)	0.0268 (18)	−0.0060 (17)	−0.0078 (15)	0.0021 (16)
C8	0.0210 (18)	0.033 (2)	0.0218 (17)	0.0045 (17)	−0.0086 (15)	0.0004 (15)
C9	0.0146 (16)	0.038 (2)	0.0276 (18)	0.0034 (17)	−0.0025 (14)	−0.0034 (17)
C10	0.0209 (18)	0.032 (2)	0.0227 (17)	−0.0027 (17)	−0.0039 (14)	−0.0008 (15)
C11	0.028 (2)	0.042 (3)	0.035 (2)	−0.0083 (19)	0.0067 (16)	−0.0087 (19)
C12	0.030 (2)	0.043 (2)	0.036 (2)	−0.0077 (19)	0.0080 (16)	−0.0075 (19)
C13	0.0193 (17)	0.032 (2)	0.0228 (17)	0.0012 (17)	−0.0012 (14)	0.0012 (15)
C14	0.0248 (18)	0.033 (2)	0.0276 (18)	0.0048 (17)	−0.0059 (15)	−0.0001 (16)
C15	0.054 (2)	0.030 (2)	0.028 (2)	−0.001 (2)	−0.0026 (18)	0.0053 (17)
C16	0.056 (2)	0.030 (2)	0.0264 (19)	−0.001 (2)	−0.0036 (18)	−0.0008 (17)
C17	0.0255 (19)	0.0276 (19)	0.0293 (18)	0.0029 (18)	−0.0049 (15)	−0.0006 (15)
C18	0.031 (2)	0.031 (2)	0.0293 (18)	0.0057 (19)	−0.0062 (16)	0.0013 (17)
C19	0.0287 (19)	0.036 (2)	0.0280 (19)	0.0028 (19)	−0.0064 (16)	0.0031 (17)
C20	0.032 (2)	0.032 (2)	0.0231 (18)	0.0006 (18)	−0.0029 (15)	−0.0012 (15)
C21	0.074 (3)	0.043 (3)	0.032 (2)	−0.007 (2)	0.009 (2)	−0.0058 (19)
C22	0.037 (2)	0.033 (2)	0.0340 (19)	0.0020 (19)	−0.0040 (18)	−0.0020 (16)
C23	0.066 (3)	0.037 (2)	0.030 (2)	0.004 (2)	−0.0018 (19)	−0.0041 (18)
C24A	0.030 (2)	0.039 (2)	0.033 (2)	−0.0053 (19)	0.0006 (17)	−0.0044 (16)
C25A	0.039 (6)	0.036 (6)	0.019 (5)	0.005 (5)	0.009 (4)	−0.007 (4)
C26A	0.074 (8)	0.054 (6)	0.032 (5)	−0.021 (6)	0.004 (5)	0.003 (4)
C27A	0.080 (4)	0.038 (3)	0.052 (2)	0.017 (3)	0.004 (2)	−0.012 (2)

C24B	0.030 (2)	0.039 (2)	0.033 (2)	−0.0053 (19)	0.0006 (17)	−0.0044 (16)
C25B	0.033 (6)	0.057 (7)	0.045 (6)	0.000 (5)	0.019 (4)	−0.013 (5)
C26B	0.058 (7)	0.058 (6)	0.042 (5)	−0.013 (5)	−0.006 (5)	−0.010 (5)
C27B	0.080 (4)	0.038 (3)	0.052 (2)	0.017 (3)	0.004 (2)	−0.012 (2)
C28	0.037 (2)	0.036 (2)	0.0290 (19)	−0.006 (2)	−0.0048 (17)	−0.0032 (18)
C29	0.048 (3)	0.044 (3)	0.059 (3)	−0.003 (2)	−0.007 (2)	−0.018 (2)
C30	0.043 (3)	0.053 (3)	0.033 (2)	−0.015 (2)	0.0098 (19)	−0.009 (2)
C31	0.077 (3)	0.082 (3)	0.027 (2)	−0.029 (3)	0.013 (2)	−0.004 (2)
O1	0.0238 (12)	0.0406 (16)	0.0301 (13)	0.0059 (12)	−0.0096 (11)	0.0015 (13)
O2	0.0173 (12)	0.0556 (18)	0.0536 (16)	0.0015 (13)	−0.0003 (12)	−0.0183 (14)
O3	0.0298 (13)	0.0327 (14)	0.0408 (14)	0.0015 (13)	−0.0065 (11)	−0.0113 (12)
O4	0.0353 (16)	0.0547 (19)	0.0588 (17)	−0.0004 (15)	−0.0166 (14)	−0.0149 (15)
O5	0.0382 (14)	0.0442 (16)	0.0220 (12)	−0.0035 (14)	0.0021 (11)	0.0003 (12)
O6	0.0466 (18)	0.086 (2)	0.0515 (19)	0.0110 (19)	0.0178 (15)	−0.0129 (18)

Geometric parameters (Å, º)

C1—C2	1.522 (5)	C19—H19C	0.9800
C1—C10	1.537 (5)	C20—C21	1.519 (5)
C1—H1A	0.9900	C20—C22	1.532 (5)
C1—H1B	0.9900	C20—H20	1.0000
C2—C3	1.506 (5)	C21—H21A	0.9800
C2—H2A	0.9900	C21—H21B	0.9800
C2—H2B	0.9900	C21—H21C	0.9800
C3—O3	1.458 (4)	C22—C23	1.513 (5)
C3—C4	1.511 (5)	C22—H22A	0.9900
C3—H3	1.0000	C22—H22B	0.9900
C4—C5	1.530 (5)	C23—C24B	1.511 (5)
C4—H4A	0.9900	C23—C24A	1.511 (5)
C4—H4B	0.9900	C23—H23A	0.9900
C5—O1	1.425 (4)	C23—H23B	0.9900
C5—C6	1.532 (5)	C24A—C25A	1.531 (7)
C5—C10	1.554 (4)	C24A—H24A	0.9900
C6—O5	1.458 (4)	C24A—H24B	0.9900
C6—C7	1.487 (5)	C25A—C26A	1.516 (7)
C6—H6	1.0000	C25A—C27A	1.543 (7)
C7—C8	1.331 (4)	C25A—H25A	1.0000
C7—H7	0.9500	C26A—H26A	0.9800
C8—C9	1.482 (5)	C26A—H26B	0.9800
C8—C14	1.499 (5)	C26A—H26C	0.9800
C9—O2	1.457 (4)	C27A—H27A	0.9800
C9—C11	1.487 (5)	C27A—H27B	0.9800
C9—C10	1.521 (5)	C27A—H27C	0.9800
C10—C19	1.542 (4)	C24B—C25B	1.518 (8)
C11—O2	1.445 (4)	C24B—H24C	0.9900
C11—C12	1.506 (5)	C24B—H24D	0.9900
C11—H11	1.05 (4)	C25B—C26B	1.513 (9)
C12—C13	1.528 (5)	C25B—C27B	1.553 (9)

C12—H12A	0.9900	C25B—H25B	1.0000
C12—H12B	0.9900	C26B—H26D	0.9800
C13—C18	1.533 (5)	C26B—H26E	0.9800
C13—C14	1.542 (4)	C26B—H26F	0.9800
C13—C17	1.547 (5)	C27B—H27D	0.9800
C14—C15	1.532 (5)	C27B—H27E	0.9800
C14—H14	1.0000	C27B—H27F	0.9800
C15—C16	1.536 (5)	C28—O4	1.210 (4)
C15—H15A	0.9900	C28—O3	1.340 (4)
C15—H15B	0.9900	C28—C29	1.484 (5)
C16—C17	1.550 (5)	C29—H29A	0.9800
C16—H16A	0.9900	C29—H29B	0.9800
C16—H16B	0.9900	C29—H29C	0.9800
C17—C20	1.531 (4)	C30—O6	1.206 (5)
C17—H17	1.0000	C30—O5	1.344 (4)
C18—H18A	0.9800	C30—C31	1.503 (6)
C18—H18B	0.9800	C31—H31A	0.9800
C18—H18C	0.9800	C31—H31B	0.9800
C19—H19A	0.9800	C31—H31C	0.9800
C19—H19B	0.9800	O1—H1O	0.81 (3)

C2—C1—C10	113.0 (3)	C10—C19—H19A	109.5
C2—C1—H1A	109.0	C10—C19—H19B	109.5
C10—C1—H1A	109.0	H19A—C19—H19B	109.5
C2—C1—H1B	109.0	C10—C19—H19C	109.5
C10—C1—H1B	109.0	H19A—C19—H19C	109.5
H1A—C1—H1B	107.8	H19B—C19—H19C	109.5
C3—C2—C1	112.0 (3)	C21—C20—C17	113.2 (3)
C3—C2—H2A	109.2	C21—C20—C22	111.0 (3)
C1—C2—H2A	109.2	C17—C20—C22	108.9 (3)
C3—C2—H2B	109.2	C21—C20—H20	107.8
C1—C2—H2B	109.2	C17—C20—H20	107.8
H2A—C2—H2B	107.9	C22—C20—H20	107.8
O3—C3—C2	108.2 (3)	C20—C21—H21A	109.5
O3—C3—C4	106.4 (3)	C20—C21—H21B	109.5
C2—C3—C4	111.7 (3)	H21A—C21—H21B	109.5
O3—C3—H3	110.2	C20—C21—H21C	109.5
C2—C3—H3	110.2	H21A—C21—H21C	109.5
C4—C3—H3	110.2	H21B—C21—H21C	109.5
C3—C4—C5	112.2 (3)	C23—C22—C20	118.3 (3)
C3—C4—H4A	109.2	C23—C22—H22A	107.7
C5—C4—H4A	109.2	C20—C22—H22A	107.7
C3—C4—H4B	109.2	C23—C22—H22B	107.7
C5—C4—H4B	109.2	C20—C22—H22B	107.7
H4A—C4—H4B	107.9	H22A—C22—H22B	107.1
O1—C5—C4	109.4 (3)	C24B—C23—C22	112.8 (3)
O1—C5—C6	110.0 (3)	C24A—C23—C22	112.8 (3)
C4—C5—C6	111.3 (3)	C24A—C23—H23A	109.0

O1—C5—C10	104.8 (2)	C22—C23—H23A	109.0
C4—C5—C10	111.9 (3)	C24A—C23—H23B	109.0
C6—C5—C10	109.3 (2)	C22—C23—H23B	109.0
O5—C6—C7	106.3 (3)	H23A—C23—H23B	107.8
O5—C6—C5	108.2 (2)	C23—C24A—C25A	117.3 (4)
C7—C6—C5	112.5 (3)	C23—C24A—H24A	108.0
O5—C6—H6	109.9	C25A—C24A—H24A	108.0
C7—C6—H6	109.9	C23—C24A—H24B	108.0
C5—C6—H6	109.9	C25A—C24A—H24B	108.0
C8—C7—C6	124.1 (3)	H24A—C24A—H24B	107.2
C8—C7—H7	118.0	C26A—C25A—C24A	113.0 (6)
C6—C7—H7	118.0	C26A—C25A—C27A	105.6 (6)
C7—C8—C9	120.6 (3)	C24A—C25A—C27A	107.9 (5)
C7—C8—C14	124.0 (3)	C26A—C25A—H25A	110.1
C9—C8—C14	115.4 (3)	C24A—C25A—H25A	110.1
O2—C9—C8	113.7 (3)	C27A—C25A—H25A	110.1
O2—C9—C11	58.8 (2)	C25A—C26A—H26A	109.5
C8—C9—C11	117.4 (3)	C25A—C26A—H26B	109.5
O2—C9—C10	116.0 (3)	H26A—C26A—H26B	109.5
C8—C9—C10	117.0 (3)	C25A—C26A—H26C	109.5
C11—C9—C10	120.3 (3)	H26A—C26A—H26C	109.5
C9—C10—C1	111.9 (3)	H26B—C26A—H26C	109.5
C9—C10—C19	106.6 (3)	C25A—C27A—H27A	109.5
C1—C10—C19	110.7 (3)	C25A—C27A—H27B	109.5
C9—C10—C5	108.5 (3)	H27A—C27A—H27B	109.5
C1—C10—C5	107.9 (3)	C25A—C27A—H27C	109.5
C19—C10—C5	111.3 (3)	H27A—C27A—H27C	109.5
O2—C11—C9	59.6 (2)	H27B—C27A—H27C	109.5
O2—C11—C12	115.2 (3)	C23—C24B—C25B	118.1 (4)
C9—C11—C12	123.8 (3)	C23—C24B—H24C	107.8
O2—C11—H11	113 (2)	C25B—C24B—H24C	107.8
C9—C11—H11	115.4 (19)	C23—C24B—H24D	107.8
C12—C11—H11	116.3 (19)	C25B—C24B—H24D	107.8
C11—C12—C13	113.4 (3)	H24C—C24B—H24D	107.1
C11—C12—H12A	108.9	C26B—C25B—C24B	110.7 (6)
C13—C12—H12A	108.9	C26B—C25B—C27B	106.5 (6)
C11—C12—H12B	108.9	C24B—C25B—C27B	108.1 (5)
C13—C12—H12B	108.9	C26B—C25B—H25B	110.5
H12A—C12—H12B	107.7	C24B—C25B—H25B	110.5
C12—C13—C18	108.3 (3)	C27B—C25B—H25B	110.5
C12—C13—C14	108.9 (3)	C25B—C26B—H26D	109.5
C18—C13—C14	110.6 (3)	C25B—C26B—H26E	109.5
C12—C13—C17	116.6 (3)	H26D—C26B—H26E	109.5
C18—C13—C17	111.7 (3)	C25B—C26B—H26F	109.5
C14—C13—C17	100.4 (3)	H26D—C26B—H26F	109.5
C8—C14—C15	118.3 (3)	H26E—C26B—H26F	109.5
C8—C14—C13	114.2 (3)	C25B—C27B—H27D	109.5
C15—C14—C13	103.7 (3)	C25B—C27B—H27E	109.5

C8—C14—H14	106.7	H27D—C27B—H27E	109.5
C15—C14—H14	106.7	C25B—C27B—H27F	109.5
C13—C14—H14	106.7	H27D—C27B—H27F	109.5
C14—C15—C16	104.0 (3)	H27E—C27B—H27F	109.5
C14—C15—H15A	111.0	O4—C28—O3	122.7 (4)
C16—C15—H15A	111.0	O4—C28—C29	126.2 (3)
C14—C15—H15B	111.0	O3—C28—C29	111.2 (3)
C16—C15—H15B	111.0	C28—C29—H29A	109.5
H15A—C15—H15B	109.0	C28—C29—H29B	109.5
C15—C16—C17	107.3 (3)	H29A—C29—H29B	109.5
C15—C16—H16A	110.3	C28—C29—H29C	109.5
C17—C16—H16A	110.3	H29A—C29—H29C	109.5
C15—C16—H16B	110.3	H29B—C29—H29C	109.5
C17—C16—H16B	110.3	O6—C30—O5	123.8 (4)
H16A—C16—H16B	108.5	O6—C30—C31	126.7 (4)
C20—C17—C13	121.1 (3)	O5—C30—C31	109.5 (4)
C20—C17—C16	111.6 (3)	C30—C31—H31A	109.5
C13—C17—C16	102.9 (3)	C30—C31—H31B	109.5
C20—C17—H17	106.8	H31A—C31—H31B	109.5
C13—C17—H17	106.8	C30—C31—H31C	109.5
C16—C17—H17	106.8	H31A—C31—H31C	109.5
C13—C18—H18A	109.5	H31B—C31—H31C	109.5
C13—C18—H18B	109.5	C5—O1—H1O	110 (3)
H18A—C18—H18B	109.5	C11—O2—C9	61.6 (2)
C13—C18—H18C	109.5	C28—O3—C3	118.6 (3)
H18A—C18—H18C	109.5	C30—O5—C6	118.3 (3)
H18B—C18—H18C	109.5		

C10—C1—C2—C3	−55.8 (4)	C9—C11—C12—C13	−16.5 (5)
C1—C2—C3—O3	170.0 (3)	C11—C12—C13—C18	−76.6 (4)
C1—C2—C3—C4	53.2 (4)	C11—C12—C13—C14	43.8 (4)
O3—C3—C4—C5	−171.5 (3)	C11—C12—C13—C17	156.5 (3)
C2—C3—C4—C5	−53.7 (4)	C7—C8—C14—C15	−11.4 (5)
C3—C4—C5—O1	−59.9 (3)	C9—C8—C14—C15	168.5 (3)
C3—C4—C5—C6	178.4 (3)	C7—C8—C14—C13	−133.8 (3)
C3—C4—C5—C10	55.7 (4)	C9—C8—C14—C13	46.1 (4)
O1—C5—C6—O5	−51.4 (3)	C12—C13—C14—C8	−60.6 (4)
C4—C5—C6—O5	69.9 (3)	C18—C13—C14—C8	58.3 (4)
C10—C5—C6—O5	−165.9 (3)	C17—C13—C14—C8	176.4 (3)
O1—C5—C6—C7	65.7 (3)	C12—C13—C14—C15	169.3 (3)
C4—C5—C6—C7	−172.9 (3)	C18—C13—C14—C15	−71.8 (3)
C10—C5—C6—C7	−48.8 (4)	C17—C13—C14—C15	46.3 (3)
O5—C6—C7—C8	136.3 (3)	C8—C14—C15—C16	−160.6 (3)
C5—C6—C7—C8	18.0 (5)	C13—C14—C15—C16	−33.1 (4)
C6—C7—C8—C9	2.5 (5)	C14—C15—C16—C17	7.0 (4)
C6—C7—C8—C14	−177.6 (3)	C12—C13—C17—C20	76.3 (4)
C7—C8—C9—O2	−129.4 (3)	C18—C13—C17—C20	−49.0 (4)
C14—C8—C9—O2	50.7 (4)	C14—C13—C17—C20	−166.3 (3)

C7—C8—C9—C11	164.8 (3)	C12—C13—C17—C16	−158.4 (3)
C14—C8—C9—C11	−15.1 (4)	C18—C13—C17—C16	76.4 (3)
C7—C8—C9—C10	10.2 (5)	C14—C13—C17—C16	−40.9 (3)
C14—C8—C9—C10	−169.7 (3)	C15—C16—C17—C20	152.8 (3)
O2—C9—C10—C1	−21.3 (4)	C15—C16—C17—C13	21.4 (4)
C8—C9—C10—C1	−159.9 (3)	C13—C17—C20—C21	−54.0 (4)
C11—C9—C10—C1	46.2 (4)	C16—C17—C20—C21	−175.3 (3)
O2—C9—C10—C19	−142.4 (3)	C13—C17—C20—C22	−178.0 (3)
C8—C9—C10—C19	79.0 (3)	C16—C17—C20—C22	60.7 (4)
C11—C9—C10—C19	−74.9 (4)	C21—C20—C22—C23	52.5 (5)
O2—C9—C10—C5	97.7 (3)	C17—C20—C22—C23	177.8 (3)
C8—C9—C10—C5	−41.0 (4)	C20—C22—C23—C24B	−179.2 (4)
C11—C9—C10—C5	165.2 (3)	C20—C22—C23—C24A	−179.2 (4)
C2—C1—C10—C9	174.8 (3)	C22—C23—C24A—C25A	−171.0 (5)
C2—C1—C10—C19	−66.5 (4)	C23—C24A—C25A—C26A	−55.2 (8)
C2—C1—C10—C5	55.5 (4)	C23—C24A—C25A—C27A	−171.5 (5)
O1—C5—C10—C9	−58.2 (3)	C22—C23—C24B—C25B	161.2 (5)
C4—C5—C10—C9	−176.6 (3)	C23—C24B—C25B—C26B	50.8 (9)
C6—C5—C10—C9	59.6 (3)	C23—C24B—C25B—C27B	167.2 (5)
O1—C5—C10—C1	63.2 (3)	C12—C11—O2—C9	115.8 (3)
C4—C5—C10—C1	−55.2 (4)	C8—C9—O2—C11	−108.8 (3)
C6—C5—C10—C1	−179.0 (3)	C10—C9—O2—C11	111.1 (3)
O1—C5—C10—C19	−175.2 (3)	O4—C28—O3—C3	3.8 (5)
C4—C5—C10—C19	66.4 (4)	C29—C28—O3—C3	−176.0 (3)
C6—C5—C10—C19	−57.4 (4)	C2—C3—O3—C28	98.5 (3)
C8—C9—C11—O2	102.4 (3)	C4—C3—O3—C28	−141.4 (3)
C10—C9—C11—O2	−103.9 (3)	O6—C30—O5—C6	5.5 (5)
O2—C9—C11—C12	−101.7 (4)	C31—C30—O5—C6	−175.9 (3)
C8—C9—C11—C12	0.7 (5)	C7—C6—O5—C30	131.4 (3)
C10—C9—C11—C12	154.4 (4)	C5—C6—O5—C30	−107.5 (3)
O2—C11—C12—C13	−85.5 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C29—H29*B*···O6^{i}	0.98	2.61	3.567 (6)	166
O1—H1*O*···O4^{ii}	0.81 (3)	2.16 (4)	2.923 (3)	158 (4)

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