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Gibberellin A4 monohydrate

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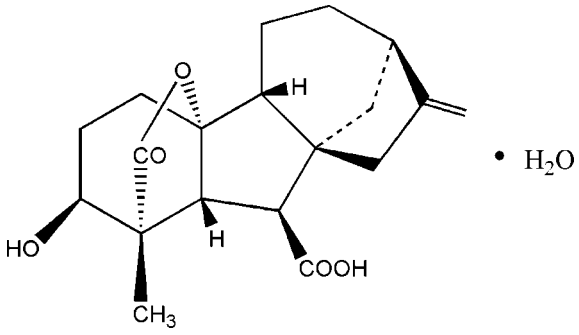
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.011 Å; R factor = 0.077; wR factor = 0.207; data-to-parameter ratio = 6.9.

The title compound, $C_{19}H_{24}O_5\cdot H_2O$, has two gibberellin A4 molecules and two water molecules in the asymmetric unit. The *A* and *B* rings have chair conformations, whereas the *C* and *D* rings have envelope conformations; the two rings which contain the lactone and carbonyl bridge adopt chair and envelope conformations. The crystal structure is established by O—H···O hydrogen bonds and supported by C—H···O hydrogen bonds.

Related literature

For related literature, see: Coggins *et al.* (1969); Ellames *et al.* (1979); Hossain *et al.* (1988); Komoda *et al.* (1968); Kutschabsky & Adam (1983); Thompson *et al.* (2000); Furber *et al.* (1992); Nagata *et al.* (1971); Poling (1991).



Experimental

Crystal data

$C_{19}H_{24}O_5\cdot H_2O$
M_r = 350.40
Monoclinic, $P2_1$
a = 9.6466 (13) Å
b = 18.968 (3) Å
c = 9.4910 (12) Å
β = 89.989 (3)°
V = 1736.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 193 (2) K
0.44 × 0.35 × 0.19 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (*SHELXTL*; Siemens, 1998)
T_{min} = 0.958, T_{max} = 0.981
17165 measured reflections
3281 independent reflections
3080 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.207
S = 1.13
3281 reflections
475 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O11^{i}	0.84	2.01	2.825 (8)	163 (4)
O4—H4···O6^{ii}	0.84	1.83	2.634 (8)	158 (6)
O6—H6*WA*···O8^{iii}	0.82 (5)	2.15 (6)	2.939 (9)	161 (6)
O6—H6*WB*···O4^{iv}	0.82 (5)	1.93 (13)	2.634 (9)	143 (6)
O10—H10···O12	0.83 (7)	1.81 (6)	2.625 (8)	163 (4)
O12—H12*A*···O2	0.82 (9)	2.16 (11)	2.925 (10)	154 (10)
O12—H12*B*···O9^{iv}	0.82 (9)	1.91 (6)	2.727 (10)	167 (2)
C11—H11···O8	1.00	2.54	3.531 (10)	173 (7)
C30—H30···O2^{v}	1.00	2.46	3.442 (9)	166 (7)

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1998); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2480).

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Gibberellin A4 monohydrate

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S1. Comment

The gibberellins are an important family of diterpenoid plant-growth factors. It is used mainly to improve the rind quality of fruit (Coggins *et al.*, 1969; Poling, 1991). Some crystal sructures of gibberellins are reported previously, such as gibberellin A20 (Komoda *et al.*, 1968), gibberellin A4 trmethyl ester (Ellames *et al.*, 1979), gibberellin A3 (Kutschabsky & Adam, 1983), 3-*O*-acetylgiberellin A3 (Hossain *et al.*, 1988), gibberellin C (Thompson *et al.*, 2000), 15-crclogibberellin A9 (Furber *et al.*, 1992), and gibberellin A15 (Nagata *et al.*, 1971). We present here the structure of the title compound, (I).

As shown in Fig. 1, there are two crystallographically independent (I) in the asymmeytric unit cell. The H atom at C2 and C8, the methylat C3, the hdroxyl at C5, the methano (C15) bridge and the carboxyl, all lie on the 'upper' β face of the molecule, and the C=O group also toward the β face. Only the bridge that links the lactone and carbonyl has α stereochemistry. The A ring and the B ring have chair conformations, whereas the C ring and D ring have the envelope conformations, besides those, the two rings which contain the lactone and carbonyl bridge adopt chair and envelope conformations respectively. The H atom of axial hydroxyl group (O10) is aimed away from the ring system toward a water molecule, and the water (O12) hydrogen-bonding relates to the lactone carbonyl (O2). There exists two counter-directional screw-related sets of helices, the hydroxyl and lactone carbonyl in related molecules within a given helix are involved in hydrogen bonds from a single water of hydration (O12—H12A···O2 and O12—H12B···O9), in turn, the oxygen (O12) of the same water accepts a hydrogen bond from the carboxyl group of a third screw-related molecule in an adjacent counter-directionally oriented helix (O10—H10···O12). Thus, water molecule acts both to brace the helix and to bridge it, alternately, to two different screw-related neighboring chains in a complex three-dimensional array.

The water molecules are linked to the GA4 molecules by O—H···O hydrogen bonds (Table 2), more over, weak C—H···O hydrogen bonds are obeserved between the gibberellin molecules to help increasing the stalitiy of the crystal (Fig. 2 & Table 2).

S2. Experimental

Gibberellin A4 (1 mmol, 0.33 g) was dissolved with 10 ml tetrahydrofuran and water (1:1) admixture, then heated to boiling and stirred for ten minutes. The resulting solution was cooled to the room temperature and the colorless crystals were collected after five days.

S3. Refinement

The H atoms attaching to O atoms and the H atoms of water were deduced from difference Fourier maps, and incorporated in refinement. Others were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.98 (methyl), 0.99 (methylene), 1.00Å (methine), with U_{iso}(H) values 1.2 times U_{eq} of the parent atoms. Because of the lack of atoms heavier than oxygen and the short measuring wavelength of Mo radiation, no useful

absolute structure parameter could be refined.

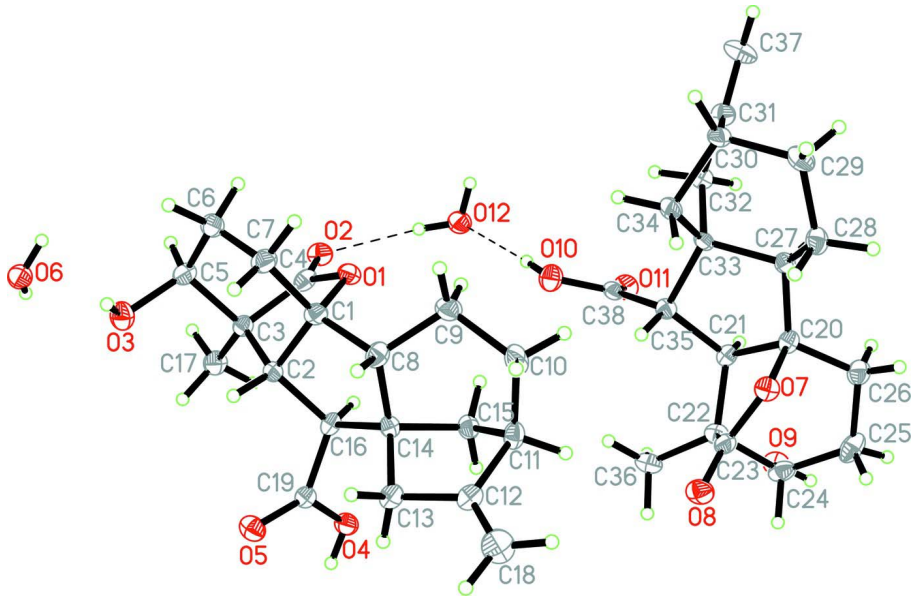

Figure 1

The cell unit of (I) with atom labels, showing 40% probability displacement ellipsoids. The thin lines denote the hydrogen bonds.

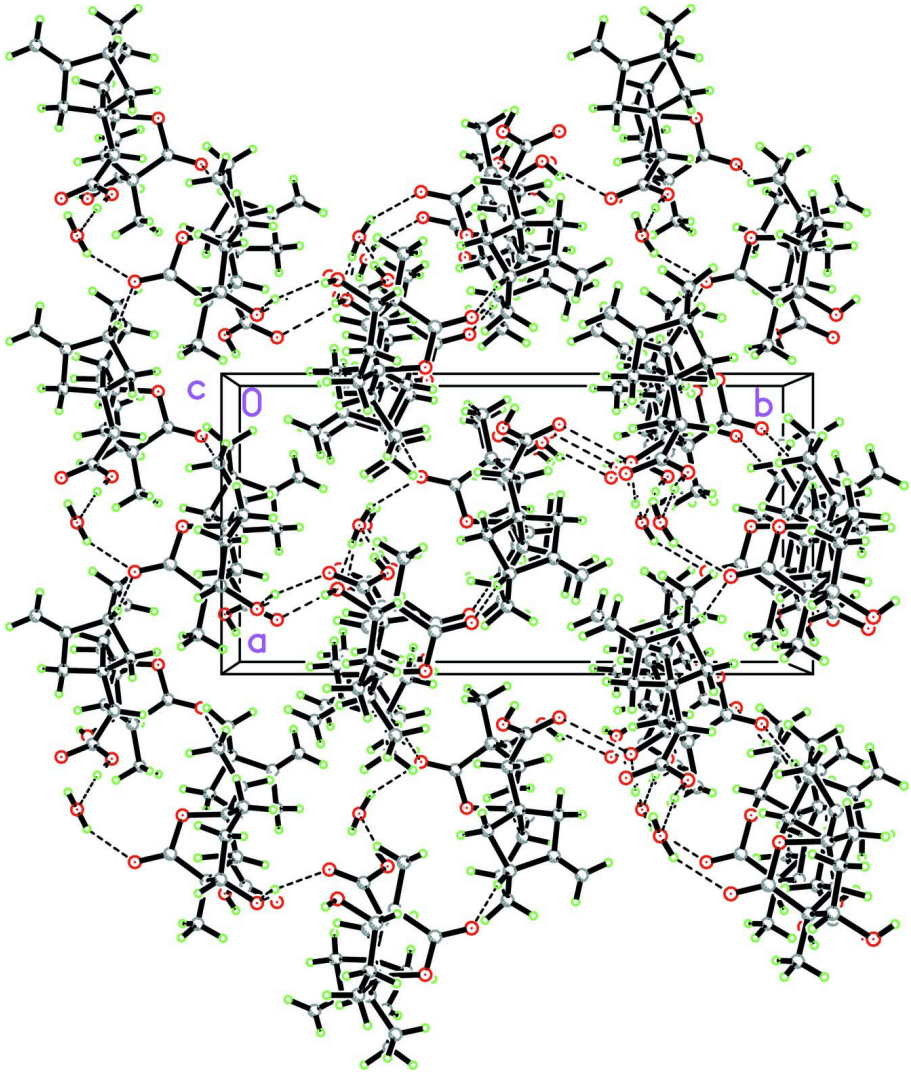

Figure 2

The crystal packing of (I) viewed down along the *c* axis, hydrogen bonds shown as thin lines.

Gibberellin A4 monohydrate

Crystal data

$C_{19}H_{24}O_5 \cdot H_2O$
$M_r = 350.40$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 9.6466\ (13)$ Å
$b = 18.968\ (3)$ Å
$c = 9.4910\ (12)$ Å
$\beta = 89.989\ (3)°$
$V = 1736.6\ (4)$ Å^3
$Z = 4$

$F(000) = 752$
$D_x = 1.340$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Cell parameters from 6798 reflections
$\theta = 3.0–25.3°$
$\mu = 0.10$ mm^{-1}
$T = 193$ K
Prism, colourless
0.44 × 0.35 × 0.19 mm

Data collection

Rigaku Mercury diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SHELXTL*; Siemens, 1998)
T_{min} = 0.958, T_{max} = 0.981
17165 measured reflections
3281 independent reflections
3080 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 25.4°, θ_{min} = 3.0°
h = −11→11
k = −20→22
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.207
S = 1.13
3281 reflections
475 parameters
7 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 5.3874P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.030 (4)

Special details

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.4911 (5)	0.4162 (3)	0.4548 (5)	0.0302 (12)
O2	0.3349 (6)	0.3352 (3)	0.3931 (6)	0.0342 (13)
O3	0.2111 (6)	0.5532 (3)	0.2383 (7)	0.0401 (14)
H3	0.2615	0.5851	0.2043	0.060*
O4	0.1765 (6)	0.4775 (3)	0.8495 (6)	0.0346 (13)
H4	0.1107	0.4964	0.8936	0.052*
O5	0.1604 (6)	0.5757 (3)	0.7208 (6)	0.0376 (13)
O6	0.0054 (6)	0.5236 (3)	0.0452 (6)	0.0377 (14)
O7	0.9843 (5)	0.3447 (3)	1.1202 (6)	0.0308 (12)
O8	0.8233 (6)	0.4220 (3)	1.1879 (6)	0.0385 (14)
O9	0.7109 (6)	0.2016 (3)	1.3376 (6)	0.0392 (14)
H9	0.7274	0.1835	1.4166	0.059*
O10	0.6679 (6)	0.2784 (3)	0.7237 (6)	0.0396 (14)
H10	0.6143	0.2548	0.6722	0.059*
O11	0.6654 (6)	0.1799 (3)	0.8549 (6)	0.0371 (14)

O12	0.5048 (6)	0.2267 (4)	0.5279 (6)	0.0382 (14)
C1	0.4931 (8)	0.4951 (4)	0.4695 (8)	0.0287 (17)
C2	0.3418 (7)	0.5094 (4)	0.5070 (7)	0.0256 (16)
H2	0.3155	0.5599	0.4920	0.031*
C3	0.2695 (8)	0.4602 (4)	0.4041 (8)	0.0259 (16)
C4	0.3615 (8)	0.3961 (4)	0.4122 (8)	0.0304 (17)
C5	0.2899 (9)	0.4894 (5)	0.2509 (8)	0.0359 (19)
H5	0.2502	0.4542	0.1837	0.043*
C6	0.4416 (9)	0.4998 (5)	0.2134 (9)	0.039 (2)
H6A	0.4797	0.4541	0.1813	0.046*
H6B	0.4472	0.5331	0.1332	0.046*
C7	0.5301 (9)	0.5267 (5)	0.3279 (8)	0.0359 (19)
H7A	0.5206	0.5786	0.3332	0.043*
H7B	0.6282	0.5158	0.3062	0.043*
C8	0.5786 (8)	0.5146 (5)	0.5987 (8)	0.0329 (18)
H8	0.6024	0.5658	0.5910	0.039*
C9	0.7138 (8)	0.4740 (5)	0.6141 (9)	0.039 (2)
H9A	0.7798	0.4897	0.5406	0.047*
H9B	0.6960	0.4231	0.5999	0.047*
C10	0.7792 (9)	0.4857 (6)	0.7620 (10)	0.044 (2)
H10A	0.8449	0.4469	0.7823	0.052*
H10B	0.8319	0.5305	0.7619	0.052*
C11	0.6678 (8)	0.4884 (4)	0.8784 (9)	0.0328 (18)
H11	0.7037	0.4674	0.9678	0.039*
C12	0.6146 (8)	0.5632 (4)	0.9034 (8)	0.0325 (18)
C13	0.4837 (8)	0.5720 (4)	0.8192 (8)	0.0307 (17)
H13A	0.4881	0.6150	0.7603	0.037*
H13B	0.4019	0.5751	0.8819	0.037*
C14	0.4764 (8)	0.5059 (4)	0.7271 (8)	0.0260 (16)
C15	0.5351 (8)	0.4502 (5)	0.8300 (8)	0.0316 (17)
H15A	0.4712	0.4413	0.9097	0.038*
H15B	0.5563	0.4053	0.7817	0.038*
C16	0.3326 (7)	0.4877 (4)	0.6617 (7)	0.0244 (15)
H16	0.3225	0.4353	0.6643	0.029*
C17	0.1175 (8)	0.4436 (5)	0.4304 (9)	0.0368 (19)
H17A	0.0794	0.4185	0.3488	0.055*
H17B	0.0662	0.4876	0.4448	0.055*
H17C	0.1090	0.4139	0.5145	0.055*
C18	0.6703 (11)	0.6092 (6)	0.9877 (12)	0.054 (3)
H18A	0.6275	0.6538	1.0009	0.064*
H18B	0.7537	0.5982	1.0362	0.064*
C19	0.2140 (8)	0.5185 (4)	0.7452 (8)	0.0267 (16)
C20	0.9918 (8)	0.2658 (4)	1.0997 (8)	0.0274 (16)
C21	0.8411 (8)	0.2487 (4)	1.0648 (7)	0.0253 (15)
H21	0.8199	0.1974	1.0771	0.030*
C22	0.7644 (8)	0.2944 (4)	1.1708 (8)	0.0292 (17)
C23	0.8545 (8)	0.3612 (4)	1.1630 (8)	0.0292 (17)
C24	0.7910 (10)	0.2646 (4)	1.3216 (9)	0.0369 (19)

H24	0.7535	0.2998	1.3900	0.044*
C25	0.9411 (10)	0.2544 (6)	1.3561 (9)	0.046 (2)
H25A	0.9761	0.2990	1.3973	0.055*
H25B	0.9475	0.2179	1.4302	0.055*
C26	1.0352 (10)	0.2341 (5)	1.2404 (9)	0.039 (2)
H26A	1.0366	0.1821	1.2321	0.047*
H26B	1.1303	0.2500	1.2633	0.047*
C27	1.0758 (8)	0.2501 (4)	0.9691 (8)	0.0271 (16)
H27	1.1051	0.1996	0.9753	0.033*
C28	1.2069 (8)	0.2936 (5)	0.9501 (9)	0.0333 (18)
H28A	1.1839	0.3442	0.9612	0.040*
H28B	1.2740	0.2809	1.0248	0.040*
C29	1.2738 (8)	0.2823 (5)	0.8075 (9)	0.0369 (19)
H29A	1.3313	0.2391	0.8104	0.044*
H29B	1.3356	0.3226	0.7862	0.044*
C30	1.1624 (8)	0.2751 (5)	0.6870 (8)	0.0304 (17)
H30	1.1956	0.2951	0.5956	0.036*
C31	1.1162 (8)	0.1991 (4)	0.6727 (8)	0.0316 (17)
C32	0.9801 (8)	0.1890 (4)	0.7541 (8)	0.0285 (16)
H32A	0.9000	0.1853	0.6893	0.034*
H32B	0.9837	0.1465	0.8145	0.034*
C33	0.9726 (7)	0.2559 (4)	0.8422 (8)	0.0245 (15)
C34	1.0251 (8)	0.3109 (4)	0.7386 (8)	0.0286 (16)
H34A	1.0436	0.3566	0.7856	0.034*
H34B	0.9592	0.3182	0.6601	0.034*
C35	0.8283 (7)	0.2725 (4)	0.9086 (7)	0.0254 (15)
H35	0.8138	0.3247	0.9067	0.031*
C36	0.6119 (9)	0.3093 (5)	1.1440 (10)	0.040 (2)
H36A	0.6011	0.3314	1.0514	0.060*
H36B	0.5765	0.3411	1.2171	0.060*
H36C	0.5598	0.2649	1.1464	0.060*
C37	1.1780 (10)	0.1493 (5)	0.6011 (11)	0.048 (2)
H37A	1.2610	0.1592	0.5510	0.058*
H37B	1.1399	0.1031	0.5992	0.058*
C38	0.7126 (7)	0.2382 (4)	0.8290 (8)	0.0255 (16)
H6WA	−0.060 (5)	0.500 (3)	0.074 (7)	0.060 (18)*
H12B	0.569 (6)	0.213 (5)	0.477 (7)	0.04 (3)*
H12A	0.441 (8)	0.246 (7)	0.485 (8)	0.09 (5)*
H6WB	0.073 (7)	0.501 (4)	0.017 (15)	0.09 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.030 (3)	0.030 (3)	0.032 (3)	0.003 (2)	0.002 (2)	−0.003 (2)
O2	0.042 (3)	0.023 (3)	0.037 (3)	−0.004 (3)	0.005 (2)	0.000 (2)
O3	0.043 (3)	0.033 (3)	0.044 (3)	0.000 (3)	−0.005 (3)	0.003 (3)
O4	0.040 (3)	0.030 (3)	0.034 (3)	0.004 (2)	0.007 (2)	0.004 (2)
O5	0.038 (3)	0.030 (3)	0.045 (3)	0.001 (3)	0.008 (3)	0.006 (3)

O6	0.027 (3)	0.048 (4)	0.038 (3)	0.000 (3)	0.003 (3)	0.003 (3)
O7	0.031 (3)	0.026 (3)	0.035 (3)	−0.006 (2)	0.000 (2)	−0.002 (2)
O8	0.051 (4)	0.024 (3)	0.040 (3)	0.004 (3)	0.003 (3)	−0.001 (2)
O9	0.043 (3)	0.032 (3)	0.043 (3)	−0.002 (3)	0.008 (3)	0.002 (3)
O10	0.039 (3)	0.040 (3)	0.041 (3)	−0.003 (3)	−0.006 (3)	0.001 (3)
O11	0.039 (3)	0.028 (3)	0.045 (3)	−0.006 (3)	0.000 (3)	0.000 (3)
O12	0.028 (3)	0.049 (4)	0.038 (3)	0.001 (3)	0.003 (3)	−0.001 (3)
C1	0.038 (4)	0.021 (4)	0.027 (4)	−0.001 (3)	0.001 (3)	−0.002 (3)
C2	0.028 (4)	0.024 (4)	0.026 (4)	0.004 (3)	−0.001 (3)	−0.001 (3)
C3	0.029 (4)	0.024 (4)	0.024 (4)	−0.002 (3)	−0.002 (3)	0.000 (3)
C4	0.033 (4)	0.035 (5)	0.023 (4)	−0.007 (3)	0.000 (3)	0.003 (3)
C5	0.045 (5)	0.037 (5)	0.026 (4)	0.005 (4)	0.001 (3)	−0.001 (3)
C6	0.048 (5)	0.036 (5)	0.032 (4)	0.012 (4)	0.011 (4)	0.012 (4)
C7	0.037 (5)	0.042 (5)	0.029 (4)	0.000 (4)	0.004 (3)	0.001 (4)
C8	0.030 (4)	0.038 (4)	0.030 (4)	−0.006 (3)	−0.004 (3)	0.001 (3)
C9	0.028 (4)	0.050 (6)	0.038 (5)	0.003 (4)	0.001 (3)	−0.008 (4)
C10	0.026 (4)	0.051 (5)	0.055 (5)	0.004 (4)	−0.017 (4)	−0.011 (5)
C11	0.038 (4)	0.027 (4)	0.034 (4)	−0.001 (3)	−0.010 (3)	0.004 (3)
C12	0.036 (4)	0.032 (4)	0.029 (4)	−0.007 (4)	0.000 (3)	0.000 (3)
C13	0.038 (4)	0.026 (4)	0.029 (4)	−0.006 (3)	−0.003 (3)	0.001 (3)
C14	0.026 (4)	0.024 (4)	0.028 (4)	0.002 (3)	−0.001 (3)	0.001 (3)
C15	0.030 (4)	0.032 (4)	0.033 (4)	0.003 (3)	0.000 (3)	0.006 (3)
C16	0.027 (4)	0.023 (4)	0.023 (3)	−0.010 (3)	−0.005 (3)	0.002 (3)
C17	0.031 (4)	0.042 (5)	0.038 (4)	−0.004 (4)	−0.004 (3)	−0.004 (4)
C18	0.050 (6)	0.042 (5)	0.068 (7)	−0.009 (5)	−0.012 (5)	−0.011 (5)
C19	0.027 (4)	0.026 (4)	0.028 (4)	−0.003 (3)	−0.001 (3)	−0.001 (3)
C20	0.030 (4)	0.026 (4)	0.026 (4)	−0.004 (3)	−0.003 (3)	0.001 (3)
C21	0.031 (4)	0.022 (4)	0.023 (4)	−0.004 (3)	−0.002 (3)	0.002 (3)
C22	0.030 (4)	0.027 (4)	0.031 (4)	−0.003 (3)	0.008 (3)	0.002 (3)
C23	0.037 (4)	0.028 (4)	0.023 (4)	0.005 (3)	−0.003 (3)	−0.004 (3)
C24	0.056 (5)	0.026 (4)	0.029 (4)	0.001 (4)	0.003 (4)	−0.005 (3)
C25	0.055 (6)	0.056 (6)	0.027 (4)	−0.014 (5)	−0.004 (4)	0.010 (4)
C26	0.048 (5)	0.038 (5)	0.032 (4)	0.008 (4)	−0.004 (4)	0.006 (4)
C27	0.031 (4)	0.023 (4)	0.028 (4)	0.005 (3)	0.000 (3)	0.001 (3)
C28	0.025 (4)	0.038 (5)	0.037 (4)	−0.006 (3)	−0.005 (3)	0.003 (4)
C29	0.026 (4)	0.036 (5)	0.049 (5)	−0.001 (3)	0.008 (3)	0.002 (4)
C30	0.028 (4)	0.036 (4)	0.028 (4)	0.003 (3)	0.007 (3)	0.003 (3)
C31	0.037 (4)	0.025 (4)	0.033 (4)	0.002 (3)	0.004 (3)	0.002 (3)
C32	0.027 (4)	0.024 (4)	0.034 (4)	−0.003 (3)	0.002 (3)	−0.002 (3)
C33	0.020 (3)	0.026 (4)	0.027 (4)	0.005 (3)	0.003 (3)	0.000 (3)
C34	0.030 (4)	0.020 (4)	0.035 (4)	0.005 (3)	0.002 (3)	0.002 (3)
C35	0.022 (3)	0.027 (4)	0.027 (4)	−0.002 (3)	0.005 (3)	0.003 (3)
C36	0.039 (5)	0.039 (5)	0.042 (5)	0.006 (4)	0.010 (4)	−0.006 (4)
C37	0.041 (5)	0.039 (5)	0.065 (6)	0.007 (4)	0.020 (5)	−0.009 (5)
C38	0.020 (3)	0.029 (4)	0.028 (4)	0.002 (3)	0.004 (3)	−0.001 (3)

Geometric parameters (Å, º)

O1—C4	1.368 (9)	C14—C16	1.558 (10)
O1—C1	1.503 (9)	C15—H15A	0.9900
O2—C4	1.198 (10)	C15—H15B	0.9900
O3—C5	1.434 (10)	C16—C19	1.509 (10)
O3—H3	0.8400	C16—H16	1.0000
O4—C19	1.309 (9)	C17—H17A	0.9800
O4—H4	0.8400	C17—H17B	0.9800
O5—C19	1.224 (10)	C17—H17C	0.9800
O6—H6WA	0.82 (2)	C18—H18A	0.9500
O6—H6WB	0.82 (2)	C18—H18B	0.9500
O7—C23	1.353 (9)	C20—C27	1.511 (10)
O7—C20	1.509 (9)	C20—C26	1.523 (11)
O8—C23	1.216 (9)	C20—C21	1.526 (10)
O9—C24	1.432 (10)	C21—C22	1.521 (10)
O9—H9	0.8400	C21—C35	1.555 (10)
O10—C38	1.328 (9)	C21—H21	1.0000
O10—H10	0.8400	C22—C36	1.519 (11)
O11—C38	1.221 (9)	C22—C23	1.538 (11)
O12—H12B	0.83 (2)	C22—C24	1.560 (11)
O12—H12A	0.83 (2)	C24—C25	1.497 (13)
C1—C7	1.514 (11)	C24—H24	1.0000
C1—C8	1.523 (11)	C25—C26	1.476 (13)
C1—C2	1.526 (11)	C25—H25A	0.9900
C2—C3	1.520 (10)	C25—H25B	0.9900
C2—C16	1.527 (10)	C26—H26A	0.9900
C2—H2	1.0000	C26—H26B	0.9900
C3—C4	1.508 (11)	C27—C28	1.521 (11)
C3—C17	1.520 (11)	C27—C33	1.567 (10)
C3—C5	1.569 (10)	C27—H27	1.0000
C5—C6	1.518 (12)	C28—C29	1.515 (11)
C5—H5	1.0000	C28—H28A	0.9900
C6—C7	1.473 (12)	C28—H28B	0.9900
C6—H6A	0.9900	C29—C30	1.576 (11)
C6—H6B	0.9900	C29—H29A	0.9900
C7—H7A	0.9900	C29—H29B	0.9900
C7—H7B	0.9900	C30—C31	1.514 (11)
C8—C9	1.521 (12)	C30—C34	1.566 (10)
C8—C14	1.576 (10)	C30—H30	1.0000
C8—H8	1.0000	C31—C37	1.308 (12)
C9—C10	1.555 (12)	C31—C32	1.536 (10)
C9—H9A	0.9900	C32—C33	1.521 (11)
C9—H9B	0.9900	C32—H32A	0.9900
C10—C11	1.542 (13)	C32—H32B	0.9900
C10—H10A	0.9900	C33—C34	1.520 (10)
C10—H10B	0.9900	C33—C35	1.560 (9)
C11—C12	1.528 (11)	C34—H34A	0.9900

C11—C15	1.540 (11)	C34—H34B	0.9900
C11—H11	1.0000	C35—C38	1.496 (10)
C12—C18	1.300 (12)	C35—H35	1.0000
C12—C13	1.504 (11)	C36—H36A	0.9800
C13—C14	1.530 (11)	C36—H36B	0.9800
C13—H13A	0.9900	C36—H36C	0.9800
C13—H13B	0.9900	C37—H37A	0.9500
C14—C15	1.546 (10)	C37—H37B	0.9500

C4—O1—C1	108.5 (6)	H18A—C18—H18B	120.0
C5—O3—H3	109.5	O5—C19—O4	123.5 (7)
C19—O4—H4	109.5	O5—C19—C16	124.3 (7)
H6WA—O6—H6WB	116 (7)	O4—C19—C16	112.2 (6)
C23—O7—C20	108.2 (6)	O7—C20—C27	109.1 (6)
C24—O9—H9	109.5	O7—C20—C26	106.9 (6)
C38—O10—H10	109.5	C27—C20—C26	119.6 (7)
H12B—O12—H12A	114 (8)	O7—C20—C21	101.2 (6)
O1—C1—C7	108.4 (6)	C27—C20—C21	106.9 (6)
O1—C1—C8	108.8 (6)	C26—C20—C21	111.6 (6)
C7—C1—C8	119.3 (7)	C22—C21—C20	101.5 (6)
O1—C1—C2	100.8 (6)	C22—C21—C35	115.2 (6)
C7—C1—C2	111.2 (6)	C20—C21—C35	102.8 (6)
C8—C1—C2	106.7 (6)	C22—C21—H21	112.2
C3—C2—C1	100.4 (6)	C20—C21—H21	112.2
C3—C2—C16	115.2 (6)	C35—C21—H21	112.2
C1—C2—C16	103.4 (6)	C36—C22—C21	117.8 (7)
C3—C2—H2	112.3	C36—C22—C23	112.7 (7)
C1—C2—H2	112.3	C21—C22—C23	99.4 (6)
C16—C2—H2	112.3	C36—C22—C24	112.4 (7)
C4—C3—C2	101.1 (6)	C21—C22—C24	108.6 (6)
C4—C3—C17	113.1 (7)	C23—C22—C24	104.4 (6)
C2—C3—C17	117.7 (6)	O8—C23—O7	120.5 (7)
C4—C3—C5	105.0 (6)	O8—C23—C22	129.2 (7)
C2—C3—C5	108.7 (6)	O7—C23—C22	110.3 (6)
C17—C3—C5	110.3 (6)	O9—C24—C25	113.1 (7)
O2—C4—O1	120.7 (7)	O9—C24—C22	108.1 (6)
O2—C4—C3	130.1 (7)	C25—C24—C22	114.0 (7)
O1—C4—C3	109.1 (7)	O9—C24—H24	107.1
O3—C5—C6	112.4 (7)	C25—C24—H24	107.1
O3—C5—C3	108.0 (6)	C22—C24—H24	107.1
C6—C5—C3	112.6 (7)	C26—C25—C24	117.7 (7)
O3—C5—H5	107.9	C26—C25—H25A	107.9
C6—C5—H5	107.9	C24—C25—H25A	107.9
C3—C5—H5	107.9	C26—C25—H25B	107.9
C7—C6—C5	115.5 (7)	C24—C25—H25B	107.9
C7—C6—H6A	108.4	H25A—C25—H25B	107.2
C5—C6—H6A	108.4	C25—C26—C20	112.3 (7)
C7—C6—H6B	108.4	C25—C26—H26A	109.1

C5—C6—H6B	108.4	C20—C26—H26A	109.1
H6A—C6—H6B	107.5	C25—C26—H26B	109.1
C6—C7—C1	112.4 (7)	C20—C26—H26B	109.1
C6—C7—H7A	109.1	H26A—C26—H26B	107.9
C1—C7—H7A	109.1	C20—C27—C28	115.8 (6)
C6—C7—H7B	109.1	C20—C27—C33	106.0 (6)
C1—C7—H7B	109.1	C28—C27—C33	113.5 (6)
H7A—C7—H7B	107.9	C20—C27—H27	107.0
C9—C8—C1	114.8 (7)	C28—C27—H27	107.0
C9—C8—C14	114.1 (6)	C33—C27—H27	107.0
C1—C8—C14	104.9 (6)	C29—C28—C27	112.5 (7)
C9—C8—H8	107.5	C29—C28—H28A	109.1
C1—C8—H8	107.5	C27—C28—H28A	109.1
C14—C8—H8	107.5	C29—C28—H28B	109.1
C8—C9—C10	111.3 (7)	C27—C28—H28B	109.1
C8—C9—H9A	109.4	H28A—C28—H28B	107.8
C10—C9—H9A	109.4	C28—C29—C30	111.7 (6)
C8—C9—H9B	109.4	C28—C29—H29A	109.3
C10—C9—H9B	109.4	C30—C29—H29A	109.3
H9A—C9—H9B	108.0	C28—C29—H29B	109.3
C11—C10—C9	111.6 (7)	C30—C29—H29B	109.3
C11—C10—H10A	109.3	H29A—C29—H29B	107.9
C9—C10—H10A	109.3	C31—C30—C34	101.1 (6)
C11—C10—H10B	109.3	C31—C30—C29	110.4 (7)
C9—C10—H10B	109.3	C34—C30—C29	108.2 (6)
H10A—C10—H10B	108.0	C31—C30—H30	112.2
C12—C11—C15	101.7 (6)	C34—C30—H30	112.2
C12—C11—C10	112.1 (7)	C29—C30—H30	112.2
C15—C11—C10	110.5 (7)	C37—C31—C30	126.9 (8)
C12—C11—H11	110.7	C37—C31—C32	124.1 (8)
C15—C11—H11	110.7	C30—C31—C32	108.9 (6)
C10—C11—H11	110.7	C33—C32—C31	102.3 (6)
C18—C12—C13	126.8 (9)	C33—C32—H32A	111.3
C18—C12—C11	125.5 (8)	C31—C32—H32A	111.3
C13—C12—C11	107.6 (6)	C33—C32—H32B	111.3
C12—C13—C14	104.6 (6)	C31—C32—H32B	111.3
C12—C13—H13A	110.8	H32A—C32—H32B	109.2
C14—C13—H13A	110.8	C34—C33—C32	101.6 (6)
C12—C13—H13B	110.8	C34—C33—C35	114.9 (6)
C14—C13—H13B	110.8	C32—C33—C35	115.6 (6)
H13A—C13—H13B	108.9	C34—C33—C27	109.5 (6)
C13—C14—C15	100.4 (6)	C32—C33—C27	109.4 (6)
C13—C14—C16	116.7 (6)	C35—C33—C27	105.7 (6)
C15—C14—C16	115.3 (6)	C33—C34—C30	100.7 (6)
C13—C14—C8	109.1 (6)	C33—C34—H34A	111.6
C15—C14—C8	109.3 (6)	C30—C34—H34A	111.6
C16—C14—C8	105.8 (6)	C33—C34—H34B	111.6
C11—C15—C14	99.9 (6)	C30—C34—H34B	111.6

C11—C15—H15A	111.8	H34A—C34—H34B	109.4
C14—C15—H15A	111.8	C38—C35—C21	114.5 (6)
C11—C15—H15B	111.8	C38—C35—C33	112.0 (6)
C14—C15—H15B	111.8	C21—C35—C33	104.8 (6)
H15A—C15—H15B	109.5	C38—C35—H35	108.4
C19—C16—C2	116.4 (6)	C21—C35—H35	108.4
C19—C16—C14	112.4 (6)	C33—C35—H35	108.4
C2—C16—C14	105.8 (6)	C22—C36—H36A	109.5
C19—C16—H16	107.3	C22—C36—H36B	109.5
C2—C16—H16	107.3	H36A—C36—H36B	109.5
C14—C16—H16	107.3	C22—C36—H36C	109.5
C3—C17—H17A	109.5	H36A—C36—H36C	109.5
C3—C17—H17B	109.5	H36B—C36—H36C	109.5
H17A—C17—H17B	109.5	C31—C37—H37A	120.0
C3—C17—H17C	109.5	C31—C37—H37B	120.0
H17A—C17—H17C	109.5	H37A—C37—H37B	120.0
H17B—C17—H17C	109.5	O11—C38—O10	123.4 (7)
C12—C18—H18A	120.0	O11—C38—C35	124.7 (7)
C12—C18—H18B	120.0	O10—C38—C35	111.9 (6)

C4—O1—C1—C7	−87.7 (7)	C23—O7—C20—C27	139.9 (6)
C4—O1—C1—C8	141.1 (6)	C23—O7—C20—C26	−89.5 (7)
C4—O1—C1—C2	29.1 (7)	C23—O7—C20—C21	27.4 (7)
O1—C1—C2—C3	−42.7 (7)	O7—C20—C21—C22	−42.2 (6)
C7—C1—C2—C3	72.0 (7)	C27—C20—C21—C22	−156.4 (6)
C8—C1—C2—C3	−156.3 (6)	C26—C20—C21—C22	71.2 (8)
O1—C1—C2—C16	76.5 (6)	O7—C20—C21—C35	77.2 (6)
C7—C1—C2—C16	−168.8 (6)	C27—C20—C21—C35	−36.9 (7)
C8—C1—C2—C16	−37.1 (7)	C26—C20—C21—C35	−169.4 (6)
C1—C2—C3—C4	40.9 (7)	C20—C21—C22—C36	162.4 (7)
C16—C2—C3—C4	−69.3 (7)	C35—C21—C22—C36	52.2 (9)
C1—C2—C3—C17	164.6 (7)	C20—C21—C22—C23	40.4 (7)
C16—C2—C3—C17	54.3 (9)	C35—C21—C22—C23	−69.8 (7)
C1—C2—C3—C5	−69.2 (7)	C20—C21—C22—C24	−68.4 (7)
C16—C2—C3—C5	−179.4 (6)	C35—C21—C22—C24	−178.6 (6)
C1—O1—C4—O2	179.9 (7)	C20—O7—C23—O8	179.3 (7)
C1—O1—C4—C3	−3.0 (8)	C20—O7—C23—C22	−1.4 (8)
C2—C3—C4—O2	152.2 (8)	C36—C22—C23—O8	28.5 (11)
C17—C3—C4—O2	25.5 (11)	C21—C22—C23—O8	154.1 (8)
C5—C3—C4—O2	−94.7 (10)	C24—C22—C23—O8	−93.8 (9)
C2—C3—C4—O1	−24.5 (7)	C36—C22—C23—O7	−150.7 (6)
C17—C3—C4—O1	−151.3 (6)	C21—C22—C23—O7	−25.1 (7)
C5—C3—C4—O1	88.5 (7)	C24—C22—C23—O7	87.0 (7)
C4—C3—C5—O3	−176.7 (6)	C36—C22—C24—O9	57.8 (9)
C2—C3—C5—O3	−69.2 (8)	C21—C22—C24—O9	−74.4 (8)
C17—C3—C5—O3	61.3 (8)	C23—C22—C24—O9	−179.7 (6)
C4—C3—C5—C6	−52.0 (9)	C36—C22—C24—C25	−175.5 (8)
C2—C3—C5—C6	55.5 (9)	C21—C22—C24—C25	52.3 (9)

C17—C3—C5—C6	−174.1 (7)	C23—C22—C24—C25	−53.0 (9)
O3—C5—C6—C7	84.0 (9)	O9—C24—C25—C26	90.8 (10)
C3—C5—C6—C7	−38.2 (10)	C22—C24—C25—C26	−33.3 (12)
C5—C6—C7—C1	38.8 (10)	C24—C25—C26—C20	33.1 (12)
O1—C1—C7—C6	52.0 (9)	O7—C20—C26—C25	56.0 (9)
C8—C1—C7—C6	177.3 (7)	C27—C20—C26—C25	−179.5 (7)
C2—C1—C7—C6	−57.9 (9)	C21—C20—C26—C25	−53.8 (10)
O1—C1—C8—C9	43.9 (9)	O7—C20—C27—C28	42.7 (8)
C7—C1—C8—C9	−81.1 (10)	C26—C20—C27—C28	−80.8 (9)
C2—C1—C8—C9	151.9 (7)	C21—C20—C27—C28	151.3 (6)
O1—C1—C8—C14	−82.2 (7)	O7—C20—C27—C33	−84.2 (7)
C7—C1—C8—C14	152.7 (7)	C26—C20—C27—C33	152.3 (7)
C2—C1—C8—C14	25.7 (8)	C21—C20—C27—C33	24.5 (8)
C1—C8—C9—C10	−168.4 (7)	C20—C27—C28—C29	−171.7 (7)
C14—C8—C9—C10	−47.2 (10)	C33—C27—C28—C29	−48.7 (9)
C8—C9—C10—C11	38.4 (11)	C27—C28—C29—C30	38.6 (10)
C9—C10—C11—C12	−89.9 (9)	C28—C29—C30—C31	−87.4 (8)
C9—C10—C11—C15	22.8 (10)	C28—C29—C30—C34	22.4 (9)
C15—C11—C12—C18	155.2 (9)	C34—C30—C31—C37	161.8 (9)
C10—C11—C12—C18	−86.7 (11)	C29—C30—C31—C37	−83.8 (11)
C15—C11—C12—C13	−22.0 (8)	C34—C30—C31—C32	−16.8 (8)
C10—C11—C12—C13	96.1 (8)	C29—C30—C31—C32	97.5 (7)
C18—C12—C13—C14	173.7 (9)	C37—C31—C32—C33	167.8 (9)
C11—C12—C13—C14	−9.2 (8)	C30—C31—C32—C33	−13.5 (8)
C12—C13—C14—C15	36.5 (7)	C31—C32—C33—C34	39.4 (7)
C12—C13—C14—C16	161.9 (6)	C31—C32—C33—C35	164.5 (6)
C12—C13—C14—C8	−78.3 (7)	C31—C32—C33—C27	−76.3 (7)
C9—C8—C14—C13	102.4 (8)	C20—C27—C33—C34	122.1 (6)
C1—C8—C14—C13	−131.0 (7)	C28—C27—C33—C34	−6.1 (8)
C9—C8—C14—C15	−6.5 (9)	C20—C27—C33—C32	−127.3 (6)
C1—C8—C14—C15	120.0 (7)	C28—C27—C33—C32	104.4 (7)
C9—C8—C14—C16	−131.2 (7)	C20—C27—C33—C35	−2.2 (8)
C1—C8—C14—C16	−4.7 (8)	C28—C27—C33—C35	−130.4 (7)
C12—C11—C15—C14	44.0 (7)	C32—C33—C34—C30	−50.6 (7)
C10—C11—C15—C14	−75.2 (8)	C35—C33—C34—C30	−176.1 (6)
C13—C14—C15—C11	−49.9 (7)	C27—C33—C34—C30	65.1 (7)
C16—C14—C15—C11	−176.2 (6)	C31—C30—C34—C33	40.9 (7)
C8—C14—C15—C11	64.8 (7)	C29—C30—C34—C33	−75.1 (7)
C3—C2—C16—C19	−92.6 (8)	C22—C21—C35—C38	−92.9 (8)
C1—C2—C16—C19	158.9 (6)	C20—C21—C35—C38	157.7 (6)
C3—C2—C16—C14	141.8 (6)	C22—C21—C35—C33	144.0 (6)
C1—C2—C16—C14	33.4 (7)	C20—C21—C35—C33	34.6 (7)
C13—C14—C16—C19	−24.3 (9)	C34—C33—C35—C38	94.3 (8)
C15—C14—C16—C19	93.2 (8)	C32—C33—C35—C38	−23.7 (9)
C8—C14—C16—C19	−145.9 (6)	C27—C33—C35—C38	−144.9 (6)
C13—C14—C16—C2	103.7 (7)	C34—C33—C35—C21	−141.0 (7)
C15—C14—C16—C2	−138.8 (7)	C32—C33—C35—C21	101.1 (7)
C8—C14—C16—C2	−17.8 (8)	C27—C33—C35—C21	−20.2 (8)

C2—C16—C19—O5	−29.1 (10)	C21—C35—C38—O11	−27.7 (10)
C14—C16—C19—O5	93.1 (9)	C33—C35—C38—O11	91.4 (9)
C2—C16—C19—O4	152.4 (6)	C21—C35—C38—O10	153.4 (6)
C14—C16—C19—O4	−85.5 (8)	C33—C35—C38—O10	−87.4 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O11^{i}	0.84	2.01	2.825 (8)	163 (4)
O4—H4···O6^{ii}	0.84	1.83	2.634 (8)	158 (6)
O6—H6*WA*···O8^{iii}	0.82 (5)	2.15 (6)	2.939 (9)	161 (6)
O6—H6*WB*···O4^{iv}	0.82 (5)	1.93 (13)	2.634 (9)	143 (6)
O10—H10···O12	0.83 (7)	1.81 (6)	2.625 (8)	163 (4)
O12—H12*A*···O2	0.82 (9)	2.16 (11)	2.925 (10)	154 (10)
O12—H12*B*···O9^{iv}	0.82 (9)	1.91 (6)	2.727 (10)	167 (2)
C11—H11···O8	1.00	2.54	3.531 (10)	173 (7)
C30—H30···O2^{v}	1.00	2.46	3.442 (9)	166 (7)

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