organic compounds

Racemic dihydromyricetin dihydrate

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The title compound, $C_{15}H_{12}O_8 \cdot 2H_2O$, crystallizes with two organic molecules and four water molecules in the asymmetric unit. Crystal symmetry generates a racemic mixture of the chiral organic molecules. An extensive network of intra-molecular and intermolecular O—H···O hydrogen bonds helps to establish the structure.

Related literature

or related flavanones, see: Cheng *et al.* (1990).
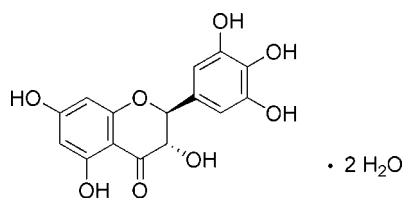
Experimental

Crystal data

$C_{15}H_{12}O_8 \cdot 2H_2O$
$M_r = 356.28$
Monoclinic, $P2_1/c$
$a = 15.391$ (3) Å
$b = 7.9002$ (16) Å
$c = 24.161$ (5) Å
$\beta = 92.57$ (3)°

$V = 2934.8$ (10) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.14$ mm^{-1}
$T = 113$ (2) K
$0.06 \times 0.02 \times 0.02$ mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan
 CrystalClear (Rigaku/MSC, 2005)
$T_{min} = 0.992$, $T_{max} = 0.997$

17668 measured reflections
5189 independent reflections
3433 reflections with $I > 2\sigma(I)$
$R_{int} = 0.077$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.155$
$S = 1.02$
5189 reflections
495 parameters
11 restraints

H atoms treated by a mixture of
 independent and constrained
 refinement
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O19	0.82	1.89	2.706 (3)	179
O3—H3···O4	0.82	1.91	2.639 (3)	147
O5—H5···O17^i	0.82	1.88	2.700 (3)	173
O6—H6···O13	0.82	1.97	2.783 (3)	173
O7—H7···O6	0.82	2.30	2.717 (3)	112
O7—H7···O10^{ii}	0.82	2.00	2.715 (3)	145
O8—H8···O3^{iii}	0.82	1.95	2.749 (3)	164
O8—H8···O7	0.82	2.28	2.722 (3)	115
O10—H10···O20^{iv}	0.82	1.93	2.742 (4)	169
O11—H11···O12	0.82	1.89	2.623 (3)	148
O13—H13···O19^v	0.82	2.00	2.818 (3)	173
O14—H14···O11^{vi}	0.82	1.96	2.744 (3)	160
O14—H14···O15	0.82	2.30	2.743 (3)	114
O15—H15···O18	0.82	1.94	2.746 (3)	166
O15—H15···O16	0.82	2.35	2.770 (3)	112
O16—H16···O5	0.82	1.94	2.739 (3)	165
O17—H17A···O13	0.85 (3)	2.35 (3)	3.042 (3)	140 (3)
O17—H17B···O20^v	0.88 (3)	2.03 (3)	2.896 (3)	170 (4)
O18—H18A···O4	0.852 (10)	2.62 (3)	3.350 (3)	145 (4)
O18—H18B···O2^{vii}	0.855 (10)	1.891 (11)	2.738 (3)	171 (4)
O19—H19A···O14^{viii}	0.853 (10)	1.926 (11)	2.770 (3)	169 (3)
O20—H20A···O8	0.859 (14)	1.984 (16)	2.836 (3)	172 (3)
O20—H20B···O18^{ix}	0.87 (3)	2.04 (4)	2.870 (3)	160 (4)

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

Data collection: *CrystalClear*; cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1997). *SHELXTL.* Bruker AXS Inc., Madison, Wisconsin, USA.
Cheng, M.-C., Cheng, Y.-S., Chang, C.-F. & Wang, Y. (1990). *Acta Cryst.* C**46**, 719–720.
Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure.* Rigaku/MSC,The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97.* University of Göttingen, Germany.

supplementary materials

Racemic dihydromyricetin dihydrate

Z. Xu, B. Liu, Z. Ning and Y. Zhang

Comment

The asymmetric unit of (I) contains of two dihydromyricetin molecules and four water molecules (Fig. 1). In the arbitrarily chosen asymmetric unit, the C1-containing dihydromyricetin molecule has an (*R*, *R*) configuration, and the C16 molecule has an (S, S) configuration. In any event, crystal symmetry generates racemic mixture of molecules. The pyran ring exists as a twisted chair conformation in both molecules. The resorcinol ring makes angles of 64.01 (15)° and 74.42 (13)° with the triol ring in the two molecules.

As well as a number of intramolecular O—H···O links, strong intermolecular O—H···O interactions (Table 1) link the component species into a three-dmiensional network in the crystal of (I).

Experimental

Leaves of Ampelopsis grossedentata (about 200 g) was extracted with 3.5 l boiling water for 1.5 h and then filtered. The filtrate was left for 24 h at ambient temperature and then filtered through a porous filter. The filtered precipitate (about 41.87 g) was collected and purified to yield yellow needles of (I) (about 3.90 g) by recrystallizing 5 times from water.

Refinement

The H atoms of the water molecules were located in difference maps and refined freely, while the other H atoms were positioned geometrically (C—H = 0.93–0.98 Å, O—H = 0.82 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$.
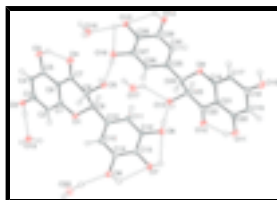
Figures

Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids for the non-hydrogen atoms.

(I)

Crystal data

$C_{15}H_{12}O_8 \cdot 2H_2O$	$F_{000} = 1488$
$M_r = 356.28$	$D_x = 1.613$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å

supplementary materials

Hall symbol: -P 2ybc

$a = 15.391$ (3) Å

$b = 7.9002$ (16) Å

$c = 24.161$ (5) Å

$\beta = 92.57$ (3)°

$V = 2934.8$ (10) Å^3

$Z = 8$

Cell parameters from 4298 reflections

$\theta = 1.7$–27.9°

$\mu = 0.14$ mm^{-1}

$T = 113$ (2) K

Needle, yellow

$0.06 \times 0.02 \times 0.02$ mm

Data collection

Rigaku saturn diffractometer

Radiation source: rotating anode

Monochromator: confocal

$T = 113(2)$ K

ω scans

Absorption correction: multi-scan
CrystalClear (Rigaku/MSC, 2005)

$T_{min} = 0.992$, $T_{max} = 0.997$

17668 measured reflections

5189 independent reflections

3433 reflections with $I > 2\sigma(I)$

$R_{int} = 0.077$

$\theta_{max} = 25.0$°

$\theta_{min} = 1.7$°

$h = -18 \rightarrow 18$

$k = -8 \rightarrow 9$

$l = -28 \rightarrow 28$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.059$

$wR(F^2) = 0.155$

$S = 1.03$

5189 reflections

495 parameters

11 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difmap and geom

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.29$ e Å^{-3}

$\Delta\rho_{min} = -0.29$ e Å^{-3}

Extinction correction: none

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 > 2sigma(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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.48728 (14)	0.6070 (3)	0.21760 (8)	0.0199 (5)
O2	0.28387 (15)	0.4841 (3)	0.07021 (9)	0.0251 (6)
H2	0.2566	0.4476	0.0960	0.038*
O3	0.52593 (15)	0.8135 (3)	0.03286 (9)	0.0251 (6)
H3	0.5704	0.8524	0.0479	0.038*
O4	0.63802 (15)	0.8827 (3)	0.11563 (9)	0.0243 (5)
O5	0.69897 (13)	0.8183 (2)	0.22252 (9)	0.0185 (5)
H5	0.7107	0.9194	0.2213	0.028*
O6	0.75720 (15)	0.4186 (3)	0.39160 (9)	0.0247 (6)
H6	0.7769	0.3522	0.3693	0.037*
O7	0.64803 (14)	0.5739 (3)	0.46089 (8)	0.0232 (5)
H7	0.6861	0.5041	0.4685	0.035*
O8	0.50042 (14)	0.7283 (3)	0.42033 (9)	0.0229 (5)
H8	0.5173	0.7084	0.4523	0.034*
O9	1.04347 (13)	0.4302 (2)	0.32245 (8)	0.0184 (5)
O10	1.23667 (15)	0.5948 (3)	0.47101 (9)	0.0237 (6)
H10	1.2592	0.6436	0.4454	0.036*
O11	1.00809 (15)	0.2216 (3)	0.50702 (9)	0.0230 (5)
H11	0.9676	0.1725	0.4907	0.034*
O12	0.90634 (14)	0.1260 (3)	0.42278 (9)	0.0235 (5)
O13	0.83770 (14)	0.2020 (3)	0.31889 (9)	0.0205 (5)
H13	0.8260	0.1021	0.3238	0.031*
O14	1.02685 (14)	0.2668 (3)	0.12043 (8)	0.0192 (5)
H14	1.0096	0.2804	0.0881	0.029*
O15	0.88587 (14)	0.4432 (3)	0.07755 (8)	0.0204 (5)
H15	0.8415	0.4972	0.0698	0.031*
O16	0.78232 (15)	0.6215 (3)	0.14812 (9)	0.0238 (6)
H16	0.7661	0.6889	0.1713	0.036*
C1	0.4610 (2)	0.6266 (4)	0.16291 (12)	0.0181 (7)
C2	0.3831 (2)	0.5538 (4)	0.14623 (12)	0.0182 (7)
H2A	0.3486	0.5006	0.1716	0.022*
C3	0.3571 (2)	0.5617 (4)	0.09030 (13)	0.0182 (7)
C4	0.4049 (2)	0.6483 (4)	0.05253 (13)	0.0208 (7)
H4	0.3858	0.6544	0.0155	0.025*
C5	0.4813 (2)	0.7257 (4)	0.07043 (13)	0.0198 (7)
C6	0.5117 (2)	0.7172 (4)	0.12640 (12)	0.0158 (7)
C7	0.5897 (2)	0.8035 (4)	0.14589 (13)	0.0172 (7)
C8	0.6089 (2)	0.7946 (4)	0.20853 (13)	0.0177 (7)
H8A	0.5757	0.8834	0.2264	0.021*
C9	0.5800 (2)	0.6225 (4)	0.22990 (12)	0.0174 (7)
H9	0.6106	0.5328	0.2106	0.021*
C10	0.5945 (2)	0.5996 (3)	0.29144 (13)	0.0170 (7)
C11	0.6674 (2)	0.5096 (4)	0.31149 (13)	0.0188 (7)
H11A	0.7033	0.4564	0.2869	0.023*
C12	0.68601 (19)	0.4995 (4)	0.36771 (13)	0.0173 (7)

C13	0.6313 (2)	0.5766 (4)	0.40477 (12)	0.0178 (7)
C14	0.5570 (2)	0.6582 (4)	0.38456 (12)	0.0168 (7)
C15	0.5388 (2)	0.6715 (4)	0.32809 (13)	0.0187 (7)
H15A	0.4892	0.7285	0.3149	0.022*
C16	1.0682 (2)	0.4205 (4)	0.37772 (12)	0.0159 (7)
C17	1.1410 (2)	0.5098 (3)	0.39515 (12)	0.0166 (7)
H17	1.1724	0.5718	0.3701	0.020*
C18	1.1667 (2)	0.5052 (4)	0.45111 (13)	0.0174 (7)
C19	1.1219 (2)	0.4107 (4)	0.48920 (13)	0.0184 (7)
H19	1.1396	0.4105	0.5265	0.022*
C20	1.0511 (2)	0.3177 (4)	0.47055 (13)	0.0196 (7)
C21	1.0212 (2)	0.3215 (3)	0.41395 (12)	0.0158 (7)
C22	0.9487 (2)	0.2199 (4)	0.39350 (13)	0.0185 (7)
C23	0.9280 (2)	0.2292 (4)	0.33132 (13)	0.0189 (7)
H23	0.9617	0.1426	0.3127	0.023*
C24	0.9522 (2)	0.4026 (4)	0.30964 (12)	0.0183 (7)
H24	0.9183	0.4893	0.3281	0.022*
C25	0.9363 (2)	0.4173 (4)	0.24804 (13)	0.0183 (7)
C26	0.8671 (2)	0.5154 (4)	0.22711 (13)	0.0183 (7)
H26	0.8327	0.5740	0.2513	0.022*
C27	0.8491 (2)	0.5263 (4)	0.17056 (13)	0.0179 (7)
C28	0.8997 (2)	0.4385 (4)	0.13371 (12)	0.0159 (7)
C29	0.9709 (2)	0.3463 (3)	0.15507 (13)	0.0164 (7)
C30	0.9886 (2)	0.3341 (3)	0.21165 (13)	0.0175 (7)
H30	1.0356	0.2700	0.2252	0.021*
O17	0.72397 (17)	0.1553 (3)	0.21376 (10)	0.0299 (6)
O18	0.72867 (17)	0.5768 (3)	0.04143 (10)	0.0281 (6)
O19	0.19398 (15)	0.3605 (3)	0.15503 (10)	0.0245 (6)
O20	0.32106 (16)	0.7871 (3)	0.39567 (10)	0.0261 (6)
H17A	0.7710 (15)	0.189 (4)	0.2298 (14)	0.040 (12)*
H17B	0.710 (3)	0.207 (5)	0.1826 (15)	0.061 (15)*
H18A	0.726 (3)	0.677 (2)	0.0545 (15)	0.067 (15)*
H18B	0.723 (4)	0.569 (5)	0.0062 (5)	0.13 (2)*
H19A	0.1449 (11)	0.319 (3)	0.1445 (13)	0.024 (10)*
H19B	0.2265 (18)	0.297 (3)	0.1754 (15)	0.053 (13)*
H20A	0.3757 (8)	0.766 (4)	0.3997 (15)	0.034 (11)*
H20B	0.307 (2)	0.887 (3)	0.407 (2)	0.10 (2)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0192 (12)	0.0287 (12)	0.0117 (12)	−0.0050 (10)	−0.0003 (9)	0.0043 (9)
O2	0.0233 (14)	0.0370 (13)	0.0150 (12)	−0.0092 (11)	0.0005 (10)	0.0004 (10)
O3	0.0229 (14)	0.0361 (13)	0.0164 (13)	−0.0100 (11)	0.0011 (10)	0.0057 (10)
O4	0.0232 (13)	0.0289 (12)	0.0208 (13)	−0.0050 (11)	0.0009 (10)	0.0048 (10)
O5	0.0162 (12)	0.0192 (11)	0.0201 (12)	−0.0022 (9)	−0.0007 (10)	−0.0004 (9)
O6	0.0223 (13)	0.0324 (13)	0.0192 (13)	0.0100 (11)	−0.0016 (11)	−0.0029 (9)
O7	0.0234 (14)	0.0322 (13)	0.0136 (12)	0.0078 (10)	−0.0022 (10)	−0.0003 (9)

O8	0.0224 (13)	0.0339 (12)	0.0125 (12)	0.0081 (10)	0.0011 (10)	−0.0020 (10)
O9	0.0185 (12)	0.0233 (11)	0.0133 (12)	−0.0033 (9)	−0.0008 (9)	0.0028 (9)
O10	0.0232 (13)	0.0286 (13)	0.0188 (13)	−0.0056 (10)	−0.0051 (11)	0.0048 (9)
O11	0.0270 (14)	0.0273 (12)	0.0146 (12)	−0.0068 (10)	0.0014 (10)	0.0032 (9)
O12	0.0258 (14)	0.0281 (12)	0.0167 (12)	−0.0047 (10)	0.0010 (10)	0.0058 (9)
O13	0.0180 (12)	0.0213 (11)	0.0221 (13)	−0.0034 (9)	−0.0019 (10)	0.0027 (9)
O14	0.0225 (13)	0.0223 (11)	0.0126 (12)	0.0026 (10)	0.0000 (10)	−0.0006 (9)
O15	0.0226 (13)	0.0265 (12)	0.0118 (12)	0.0038 (10)	−0.0020 (10)	0.0010 (9)
O16	0.0222 (13)	0.0287 (13)	0.0202 (13)	0.0073 (10)	−0.0004 (11)	−0.0030 (9)
C1	0.0196 (18)	0.0197 (15)	0.0149 (17)	0.0021 (14)	0.0009 (14)	0.0010 (12)
C2	0.0195 (18)	0.0220 (15)	0.0133 (16)	0.0017 (14)	0.0020 (13)	0.0040 (12)
C3	0.0155 (17)	0.0210 (15)	0.0181 (17)	0.0010 (14)	−0.0009 (14)	−0.0007 (13)
C4	0.0201 (18)	0.0293 (17)	0.0130 (17)	−0.0013 (14)	0.0003 (14)	−0.0026 (13)
C5	0.0207 (18)	0.0239 (16)	0.0148 (17)	0.0021 (14)	0.0010 (14)	0.0024 (13)
C6	0.0173 (17)	0.0184 (15)	0.0117 (16)	−0.0008 (13)	0.0006 (13)	−0.0008 (12)
C7	0.0210 (18)	0.0173 (15)	0.0133 (17)	0.0047 (14)	0.0020 (14)	0.0013 (12)
C8	0.0171 (17)	0.0190 (15)	0.0169 (17)	0.0012 (13)	0.0000 (14)	0.0007 (12)
C9	0.0174 (17)	0.0228 (16)	0.0120 (16)	0.0034 (14)	0.0002 (13)	0.0010 (12)
C10	0.0177 (18)	0.0165 (15)	0.0168 (17)	−0.0024 (13)	0.0018 (14)	−0.0011 (12)
C11	0.0167 (17)	0.0211 (15)	0.0187 (17)	−0.0006 (14)	0.0031 (14)	0.0001 (12)
C12	0.0128 (17)	0.0195 (15)	0.0195 (17)	−0.0012 (13)	−0.0002 (13)	−0.0011 (12)
C13	0.0213 (18)	0.0189 (15)	0.0130 (17)	−0.0010 (13)	−0.0017 (14)	0.0015 (12)
C14	0.0167 (18)	0.0178 (15)	0.0164 (17)	−0.0011 (13)	0.0048 (14)	−0.0013 (12)
C15	0.0173 (17)	0.0186 (15)	0.0197 (18)	0.0019 (13)	−0.0027 (14)	0.0025 (12)
C16	0.0179 (17)	0.0162 (14)	0.0137 (16)	0.0035 (13)	0.0016 (13)	−0.0016 (11)
C17	0.0187 (17)	0.0169 (14)	0.0140 (16)	0.0011 (13)	0.0005 (14)	0.0042 (12)
C18	0.0149 (17)	0.0157 (14)	0.0214 (18)	0.0003 (13)	−0.0017 (14)	−0.0015 (12)
C19	0.0203 (18)	0.0214 (16)	0.0132 (16)	0.0033 (14)	−0.0032 (14)	0.0011 (12)
C20	0.0205 (18)	0.0193 (15)	0.0193 (18)	0.0032 (14)	0.0045 (14)	0.0023 (12)
C21	0.0181 (17)	0.0169 (14)	0.0125 (16)	0.0020 (13)	0.0017 (13)	−0.0010 (11)
C22	0.0189 (18)	0.0164 (15)	0.0205 (18)	0.0026 (13)	0.0029 (14)	0.0035 (13)
C23	0.0149 (17)	0.0240 (16)	0.0175 (17)	−0.0021 (14)	−0.0021 (14)	−0.0001 (13)
C24	0.0157 (17)	0.0240 (16)	0.0151 (17)	−0.0008 (14)	−0.0013 (14)	−0.0010 (12)
C25	0.0209 (18)	0.0184 (15)	0.0153 (17)	−0.0052 (14)	−0.0015 (14)	0.0020 (12)
C26	0.0191 (18)	0.0202 (15)	0.0162 (17)	−0.0031 (13)	0.0053 (14)	−0.0034 (12)
C27	0.0171 (17)	0.0174 (15)	0.0190 (17)	0.0000 (13)	−0.0008 (14)	−0.0018 (12)
C28	0.0182 (17)	0.0178 (14)	0.0117 (16)	−0.0054 (13)	−0.0004 (13)	−0.0009 (12)
C29	0.0174 (17)	0.0137 (14)	0.0182 (17)	−0.0024 (13)	0.0011 (14)	−0.0024 (12)
C30	0.0188 (17)	0.0149 (14)	0.0186 (17)	−0.0035 (13)	−0.0027 (14)	0.0016 (12)
O17	0.0335 (16)	0.0297 (13)	0.0261 (15)	−0.0047 (12)	−0.0009 (13)	−0.0019 (11)
O18	0.0337 (15)	0.0293 (13)	0.0207 (14)	−0.0005 (12)	−0.0041 (12)	−0.0009 (10)
O19	0.0188 (13)	0.0254 (12)	0.0292 (14)	−0.0023 (11)	−0.0009 (11)	0.0034 (10)
O20	0.0216 (14)	0.0259 (13)	0.0309 (15)	0.0022 (11)	0.0020 (11)	−0.0007 (10)

Geometric parameters (Å, °)

O1—C1	1.373 (3)	C8—H8A	0.9800
O1—C9	1.450 (4)	C9—C10	1.505 (4)
O2—C3	1.354 (3)	C9—H9	0.9800

O2—H2	0.8200	C10—C15	1.383 (5)
O3—C5	1.353 (4)	C10—C11	1.396 (4)
O3—H3	0.8200	C11—C12	1.378 (4)
O4—C7	1.236 (4)	C11—H11A	0.9300
O5—C8	1.424 (3)	C12—C13	1.396 (5)
O5—H5	0.8200	C13—C14	1.382 (4)
O6—C12	1.373 (3)	C14—C15	1.385 (4)
O6—H6	0.8200	C15—H15A	0.9300
O7—C13	1.369 (3)	C16—C17	1.374 (4)
O7—H7	0.8200	C16—C21	1.399 (4)
O8—C14	1.371 (4)	C17—C18	1.392 (4)
O8—H8	0.8200	C17—H17	0.9300
O9—C16	1.374 (3)	C18—C19	1.392 (5)
O9—C24	1.441 (3)	C19—C20	1.373 (4)
O10—C18	1.359 (3)	C19—H19	0.9300
O10—H10	0.8200	C20—C21	1.423 (4)
O11—C20	1.358 (4)	C21—C22	1.444 (4)
O11—H11	0.8200	C22—C23	1.523 (4)
O12—C22	1.232 (4)	C23—C24	1.519 (4)
O13—C23	1.425 (3)	C23—H23	0.9800
O13—H13	0.8200	C24—C25	1.502 (4)
O14—C29	1.379 (4)	C24—H24	0.9800
O14—H14	0.8200	C25—C30	1.384 (5)
O15—C28	1.364 (3)	C25—C26	1.394 (4)
O15—H15	0.8200	C26—C27	1.385 (4)
O16—C27	1.366 (3)	C26—H26	0.9300
O16—H16	0.8200	C27—C28	1.394 (4)
C1—C2	1.374 (4)	C28—C29	1.395 (4)
C1—C6	1.400 (4)	C29—C30	1.385 (4)
C2—C3	1.393 (4)	C30—H30	0.9300
C2—H2A	0.9300	O17—H17A	0.847 (10)
C3—C4	1.379 (5)	O17—H17B	0.88 (3)
C4—C5	1.378 (4)	O18—H18A	0.852 (10)
C4—H4	0.9300	O18—H18B	0.855 (10)
C5—C6	1.412 (4)	O19—H19A	0.853 (10)
C6—C7	1.442 (4)	O19—H19B	0.849 (10)
C7—C8	1.530 (4)	O20—H20A	0.859 (10)
C8—C9	1.528 (4)	O20—H20B	0.862 (10)
C1—O1—C9	115.6 (2)	O8—C14—C15	118.9 (3)
C3—O2—H2	109.5	C13—C14—C15	120.8 (3)
C5—O3—H3	109.5	C10—C15—C14	119.7 (3)
C8—O5—H5	109.5	C10—C15—H15A	120.2
C12—O6—H6	109.5	C14—C15—H15A	120.2
C13—O7—H7	109.5	C17—C16—O9	117.1 (3)
C14—O8—H8	109.5	C17—C16—C21	122.2 (3)
C16—O9—C24	115.2 (2)	O9—C16—C21	120.7 (3)
C18—O10—H10	109.5	C16—C17—C18	118.4 (3)
C20—O11—H11	109.5	C16—C17—H17	120.8
C23—O13—H13	109.5	C18—C17—H17	120.8

C29—O14—H14	109.5	O10—C18—C19	117.0 (3)
C28—O15—H15	109.5	O10—C18—C17	121.1 (3)
C27—O16—H16	109.5	C19—C18—C17	121.9 (3)
O1—C1—C2	116.8 (3)	C20—C19—C18	118.6 (3)
O1—C1—C6	121.0 (3)	C20—C19—H19	120.7
C2—C1—C6	122.2 (3)	C18—C19—H19	120.7
C1—C2—C3	118.3 (3)	O11—C20—C19	119.3 (3)
C1—C2—H2A	120.8	O11—C20—C21	119.3 (3)
C3—C2—H2A	120.8	C19—C20—C21	121.4 (3)
O2—C3—C4	116.6 (3)	C16—C21—C20	117.3 (3)
O2—C3—C2	121.7 (3)	C16—C21—C22	120.7 (3)
C4—C3—C2	121.7 (3)	C20—C21—C22	121.9 (3)
C5—C4—C3	119.0 (3)	O12—C22—C21	124.0 (3)
C5—C4—H4	120.5	O12—C22—C23	120.2 (3)
C3—C4—H4	120.5	C21—C22—C23	115.8 (3)
O3—C5—C4	117.9 (3)	O13—C23—C24	108.4 (2)
O3—C5—C6	120.7 (3)	O13—C23—C22	111.1 (3)
C4—C5—C6	121.4 (3)	C24—C23—C22	109.9 (2)
C1—C6—C5	117.3 (3)	O13—C23—H23	109.2
C1—C6—C7	121.0 (3)	C24—C23—H23	109.2
C5—C6—C7	121.7 (3)	C22—C23—H23	109.2
O4—C7—C6	124.1 (3)	O9—C24—C25	108.3 (3)
O4—C7—C8	120.9 (3)	O9—C24—C23	108.3 (2)
C6—C7—C8	115.0 (3)	C25—C24—C23	112.3 (2)
O5—C8—C9	109.4 (2)	O9—C24—H24	109.3
O5—C8—C7	111.8 (3)	C25—C24—H24	109.3
C9—C8—C7	109.2 (2)	C23—C24—H24	109.3
O5—C8—H8A	108.8	C30—C25—C26	119.4 (3)
C9—C8—H8A	108.8	C30—C25—C24	121.3 (3)
C7—C8—H8A	108.8	C26—C25—C24	119.3 (3)
O1—C9—C10	107.1 (3)	C27—C26—C25	120.6 (3)
O1—C9—C8	107.7 (2)	C27—C26—H26	119.7
C10—C9—C8	114.1 (2)	C25—C26—H26	119.7
O1—C9—H9	109.3	O16—C27—C26	122.7 (3)
C10—C9—H9	109.3	O16—C27—C28	116.9 (3)
C8—C9—H9	109.3	C26—C27—C28	120.4 (3)
C15—C10—C11	120.0 (3)	O15—C28—C27	123.7 (3)
C15—C10—C9	120.9 (3)	O15—C28—C29	117.8 (3)
C11—C10—C9	119.1 (3)	C27—C28—C29	118.4 (3)
C12—C11—C10	120.0 (3)	O14—C29—C30	117.8 (3)
C12—C11—H11A	120.0	O14—C29—C28	121.0 (3)
C10—C11—H11A	120.0	C30—C29—C28	121.2 (3)
O6—C12—C11	124.6 (3)	C25—C30—C29	119.9 (3)
O6—C12—C13	115.2 (3)	C25—C30—H30	120.0
C11—C12—C13	120.1 (3)	C29—C30—H30	120.0
O7—C13—C14	118.2 (3)	H17A—O17—H17B	115 (3)
O7—C13—C12	122.4 (3)	H18A—O18—H18B	115.6 (19)
C14—C13—C12	119.3 (3)	H19A—O19—H19B	115.8 (18)
O8—C14—C13	120.3 (3)	H20A—O20—H20B	113.4 (18)

C9—O1—C1—C2	156.6 (3)	C24—O9—C16—C17	−153.9 (3)
C9—O1—C1—C6	−23.6 (4)	C24—O9—C16—C21	27.0 (4)
O1—C1—C2—C3	−175.9 (3)	O9—C16—C17—C18	178.7 (3)
C6—C1—C2—C3	4.3 (5)	C21—C16—C17—C18	−2.2 (4)
C1—C2—C3—O2	176.0 (3)	C16—C17—C18—O10	−178.1 (3)
C1—C2—C3—C4	−3.8 (5)	C16—C17—C18—C19	1.3 (4)
O2—C3—C4—C5	−178.2 (3)	O10—C18—C19—C20	−179.6 (3)
C2—C3—C4—C5	1.5 (5)	C17—C18—C19—C20	0.9 (5)
C3—C4—C5—O3	−178.4 (3)	C18—C19—C20—O11	178.4 (3)
C3—C4—C5—C6	0.4 (5)	C18—C19—C20—C21	−2.3 (5)
O1—C1—C6—C5	177.7 (3)	C17—C16—C21—C20	0.8 (4)
C2—C1—C6—C5	−2.5 (5)	O9—C16—C21—C20	179.9 (3)
O1—C1—C6—C7	−4.7 (4)	C17—C16—C21—C22	−175.6 (3)
C2—C1—C6—C7	175.1 (3)	O9—C16—C21—C22	3.5 (4)
O3—C5—C6—C1	178.8 (3)	O11—C20—C21—C16	−179.2 (3)
C4—C5—C6—C1	0.1 (5)	C19—C20—C21—C16	1.5 (4)
O3—C5—C6—C7	1.3 (5)	O11—C20—C21—C22	−2.8 (4)
C4—C5—C6—C7	−177.5 (3)	C19—C20—C21—C22	177.9 (3)
C1—C6—C7—O4	178.6 (3)	C16—C21—C22—O12	177.7 (3)
C5—C6—C7—O4	−3.9 (5)	C20—C21—C22—O12	1.4 (5)
C1—C6—C7—C8	−2.9 (4)	C16—C21—C22—C23	−0.1 (4)
C5—C6—C7—C8	174.6 (3)	C20—C21—C22—C23	−176.3 (3)
O4—C7—C8—O5	−25.1 (4)	O12—C22—C23—O13	31.7 (4)
C6—C7—C8—O5	156.3 (2)	C21—C22—C23—O13	−150.5 (3)
O4—C7—C8—C9	−146.2 (3)	O12—C22—C23—C24	151.5 (3)
C6—C7—C8—C9	35.1 (4)	C21—C22—C23—C24	−30.6 (4)
C1—O1—C9—C10	179.2 (2)	C16—O9—C24—C25	−179.9 (2)
C1—O1—C9—C8	56.0 (3)	C16—O9—C24—C23	−57.8 (3)
O5—C8—C9—O1	176.9 (2)	O13—C23—C24—O9	179.8 (2)
C7—C8—C9—O1	−60.5 (3)	C22—C23—C24—O9	58.3 (3)
O5—C8—C9—C10	58.2 (4)	O13—C23—C24—C25	−60.6 (3)
C7—C8—C9—C10	−179.2 (3)	C22—C23—C24—C25	177.9 (3)
O1—C9—C10—C15	−41.0 (4)	O9—C24—C25—C30	47.9 (4)
C8—C9—C10—C15	78.1 (4)	C23—C24—C25—C30	−71.7 (4)
O1—C9—C10—C11	141.4 (3)	O9—C24—C25—C26	−132.7 (3)
C8—C9—C10—C11	−99.4 (3)	C23—C24—C25—C26	107.7 (3)
C15—C10—C11—C12	−3.2 (4)	C30—C25—C26—C27	1.7 (4)
C9—C10—C11—C12	174.4 (3)	C24—C25—C26—C27	−177.7 (3)
C10—C11—C12—O6	−178.0 (3)	C25—C26—C27—O16	−179.6 (3)
C10—C11—C12—C13	1.2 (4)	C25—C26—C27—C28	0.4 (4)
O6—C12—C13—O7	1.0 (4)	O16—C27—C28—O15	0.0 (4)
C11—C12—C13—O7	−178.3 (3)	C26—C27—C28—O15	−180.0 (3)
O6—C12—C13—C14	−178.7 (3)	O16—C27—C28—C29	177.1 (3)
C11—C12—C13—C14	2.0 (4)	C26—C27—C28—C29	−2.9 (4)
O7—C13—C14—O8	−2.6 (4)	O15—C28—C29—O14	1.9 (4)
C12—C13—C14—O8	177.2 (3)	C27—C28—C29—O14	−175.4 (3)
O7—C13—C14—C15	177.1 (3)	O15—C28—C29—C30	−179.3 (3)
C12—C13—C14—C15	−3.2 (4)	C27—C28—C29—C30	3.4 (4)
C11—C10—C15—C14	2.0 (4)	C26—C25—C30—C29	−1.2 (4)

C9—C10—C15—C14	−175.5 (3)	C24—C25—C30—C29	178.2 (3)
O8—C14—C15—C10	−179.1 (3)	O14—C29—C30—C25	177.5 (3)
C13—C14—C15—C10	1.2 (4)	C28—C29—C30—C25	−1.4 (4)

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O2—H2⋯O19	0.82	1.89	2.706 (3)	179
O3—H3⋯O4	0.82	1.91	2.639 (3)	147
O5—H5⋯O17[i]	0.82	1.88	2.700 (3)	173
O6—H6⋯O13	0.82	1.97	2.783 (3)	173
O7—H7⋯O6	0.82	2.30	2.717 (3)	112
O7—H7⋯O10[ii]	0.82	2.00	2.715 (3)	145
O8—H8⋯O3[iii]	0.82	1.95	2.749 (3)	164
O8—H8⋯O7	0.82	2.28	2.722 (3)	115
O10—H10⋯O20[iv]	0.82	1.93	2.742 (4)	169
O11—H11⋯O12	0.82	1.89	2.623 (3)	148
O13—H13⋯O19[v]	0.82	2.00	2.818 (3)	173
O14—H14⋯O11[vi]	0.82	1.96	2.744 (3)	160
O14—H14⋯O15	0.82	2.30	2.743 (3)	114
O15—H15⋯O18	0.82	1.94	2.746 (3)	166
O15—H15⋯O16	0.82	2.35	2.770 (3)	112
O16—H16⋯O5	0.82	1.94	2.739 (3)	165
O17—H17A⋯O13	0.85 (3)	2.35 (3)	3.042 (3)	140 (3)
O17—H17B⋯O20[v]	0.88 (3)	2.03 (3)	2.896 (3)	170 (4)
O18—H18A⋯O4	0.852 (10)	2.62 (3)	3.350 (3)	145 (4)
O18—H18B⋯O2[vii]	0.855 (10)	1.891 (11)	2.738 (3)	171 (4)
O19—H19A⋯O14[viii]	0.853 (10)	1.926 (11)	2.770 (3)	169 (3)
O20—H20A⋯O8	0.859 (14)	1.984 (16)	2.836 (3)	172 (3)
O20—H20B⋯O18[ix]	0.87 (3)	2.04 (4)	2.870 (3)	160 (4)

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

Fig. 1