Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*S**,3*S**,3a*S**,6*S**,7a*R**)-3-Hydroxy-2-[(2*R**,3*S**)-3-isopropyloxiran-2-yl]-3,6-dimethyl-3,3a,5,6,7,7a-hexahydro-1-benzofuran-4(2*H*)-one

Mingruo Ding, Qiaoyin Zhang, Lei Chen, Nianyu Huang and Junzhi Wang*

Hubei Key Laboratory of Natural Products Research and Development, College of Chemistry and Life Sciences, China Three Gorges University, Yichang, Hubei 443002, People's Republic of China
Correspondence e-mail: horsedog@163.com

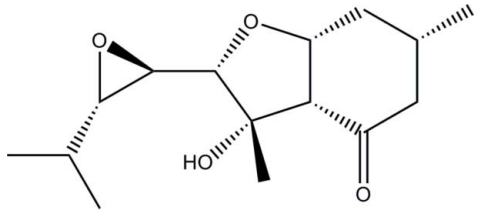
Received 31 August 2012; accepted 7 September 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.006 Å; R factor = 0.067; wR factor = 0.185; data-to-parameter ratio = 11.6.

In the title compound, $C_{15}H_{24}O_4$, the six-membered ring shows a distorted chair conformation and the five-membered ring adopts an envelope conformation with the C atom bearing the methyl and OH groups as the flap. In the crystal, O—H···O hydrogen bonds link the molecules into chains running along the *a*-axis direction.

Related literature

The title compound was synthesized as a potential gastric cytoprotective agent. For background to gastric diseases, see: Palmer *et al.* (2010). For pharmacological uses of bisabolangelone, a sesquiterpene isolated from the roots of *Angelica polymorpha* Maxim, see: Fang & Liao (2006); Muckensturm *et al.* (1981). Huang *et al.* (2012); Wang *et al.* (2009). For the crystal structure of bisabolangelone, see: Wang *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{24}O_4$
M_r = 268.34
Orthorhombic, $P2_12_12_1$
a = 6.616 (7) Å
b = 9.261 (9) Å
c = 25.12 (3) Å
V = 1539 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 K
0.35 × 0.28 × 0.26 mm

Data collection

Rigaku Mercury 375R CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2011)
T_{min} = 0.972, T_{max} = 0.980
16467 measured reflections
2058 independent reflections
1568 reflections with $I > 2\sigma(I)$
R_{int} = 0.176

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.185
S = 1.03
2058 reflections
177 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O4^{i}	0.82	2.02	2.827 (4)	166

Symmetry code: (i) $x-1, y, z$.

Data collection: *CrystalClear* (Rigaku, 2011); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge financial support by the National Natural Science Foundation of China (Nos. 30970296 and 21102084), the Scientific and Technological Research Project of Hubei Provincial Department of Education (No. Q20111210), the Doctoral Startup Foundation of China Three Gorges University (No. KJ2009B046) and the Pre-research Foundation of the College of Chemistry and Life Sciences (No. HY0905).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6831).

References

Fang, Z. & Liao, Z. (2006). *The Medicinal Plants from Enshi of Hubei*. Wuhan, China: The Science and Technological Press of Hubei.
Huang, N. Y., Chen, L., Liao, Z. J., Fang, H. B., Wang, J. Z. & Zou, K. (2012). *Chin. J. Chem.* **30**, 71–76.
Muckensturm, B., Duplay, D., Robert, P. C., Simonis, M. T. & Kienlen, J. C. (1981). *Biochem. Syst. Ecol.* **2**, 289–292.
Palmer, A. M., Chiesa, V., Schmid, A., Muench, G., Grobbel, B., Zimmermann, P. J., Brehm, C., Buhr, W., Simon, W.-A., Kromer, W., Postius, S., Volz, J. & Hess, D. (2010). *J. Med. Chem.* **53**, 3645–3674.
Rigaku (2011). *CrystalClear*. Rigaku Americas, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, J. Z., Zhu, L. B., Zou, K., Cheng, F., Dan, F. J., Guo, Z. Y., Cai, Z. J. & Yang, J. (2009). *J. Ethnopharma.* **123**, 343–346.
Wang, J.-Z., Zou, K., Cheng, F. & Dan, F.-J. (2007). *Acta Cryst.* E**63**, o2706.

supplementary materials

Acta Cryst. (2012). E**68**, o2949 [doi:10.1107/S1600536812038470]

(2*S**,3*S**,3a*S**,6*S**,7a*R**)-3-Hydroxy-2-[(2*R**,3*S**)-3-isopropyloxiran-2-yl]-3,6-dimethyl-3,3a,5,6,7,7a-hexahydro-1-benzofuran-4(2*H*)-one

Mingruo Ding, Qiaoyin Zhang, Lei Chen, Nianyu Huang and Junzhi Wang

Comment

Acid-related diseases are highly prevalent in the developed world, and the inhibition of the gastric proton pump enzyme (H^+/K^+-ATPase) represents a major approach in the development of drugs against these medical conditions (Palmer *et al.*, 2010). Bisabolangelone, a sesquiterpene isolated from the roots of *Angelica polymorpha* Maxim with the traditional Tujia medicine name of Zijinsha (Fang *et al.*, 2006), displayed attractive bioactivity such as anti-feeding and insecticidal effect (Muckensturm *et al.*, 1981). Recently, we found bisabolangelone and its derivatives also exhibited remarkably preventive and therapeutic action on gastric ulcer, and its anti-ulcer mechanism might be related to inhibition of the H^+/K^+-ATPase and reduction of the secretion of H^+ (Wang *et al.*, 2009). With the aim of studying the relationship between its structure and H^+/K^+-ATPase inhibition activity, the catalytic hydrogenated reduction (Huang *et al.*, 2012) and epoxidation of bisabolangelone were undertaken, and the structure determination of the target compound was conducted by X-ray single-crystal analysis for the first time.

Compared with the crystal structure of bisabolangelone (Wang *et al.*, 2007), most bond lengths in the title compound are in the normal range of single or double bonds. The 6-membered ring C(1)—C(2)—C(3)—C(4)—C(5)—C(6) shows a distorted chair conformation [Φ = 319.3 (9)°, Θ = 146.4 (5)°, puckering amplitude (Q) = 0.485 (4)°]. The 5-membered ring O(2)—C(5)—C(6)—C(8)—C(10) adopts an envelope conformation with C(8) at the flap. Intermolecular O—H···O interactions link the molecules into infinite zigzag chains along the *a* axis, which contribute to the stability of the structure.

Experimental

3-Hydroxy-3,6-dimethyl-2-(3-methylbut- 2-enylidene)-3,3a,7,7a-tetrahydrobenzofuran- 4(2*H*)-one (bisabolangelone, **I**, 1.00 g, 4.0 mmol) and Pd/C (0.10 g, 10% *w*/*w*) was dissolved in MeOH (30 ml) at 10\%C under dry nitrogen atmosphere, then hydrogen gas (99%) was bubbled into the vigorous stirred solution (50 ml/minute) for 2.0 h until the bisabolangelone was consumed. The hydrogen gas was diluted by large amounts of nitrogen and released into air through special pipeline, and the reaction mixture was filtered to recover the catalyst. Removing the solvents at reduced pressure to give white solids, which was purified by column chromatography on silica with ethyl acetate/petroleum ether (1:10, *v*/*v*) as eluent to give the pure intermediates 3-hydroxy-3,6-dimethyl-2-((*E*)-3-methylbut-1-enyl)hexahydrobenzofuran-4(2*H*)-one (**II**) as colorless needles (0.85 g). The *m*-CPBA (0.52 g, 1.5 mmol) and solid $NaHCO_3$ (0.19 g, 2.5 mmol) were added to a solution of the intermediates **II** (0.25 g, 1.0 mmol) in dry CH_2Cl_2 (20 ml) at 0 °C. The solution was stirred for 10 h until complete consumption of the starting material. The reaction was quenched with saturated aqueous sodium thiosulfate solution and extracted with CH_2Cl_2 (3 × 15 ml). The combined organic extracts were washed with saturated aqueous $NaHCO_3$ solution (25 ml) and dried over Na_2SO_4. The solventwas removed *in vacuo* and the residue purified by flash column chromatography on silica gel to give the pure (2*S*,3*S*,3*aS*,6*S*,7*aR*)-3-hy-

droxy-2-((2*R*,3*S*)-3-isopropyloxiran-2-yl)-3,6-dimethylhexahydrobenzofuran-4(2*H*)-one **III** (Eluant: ethyl acetate/petroleum ether = 1: 20, *v*/*v*). Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a dilute solution of the title compound **III** in *n*-hexane:ethyl acetate, 10: 1 at room temperature.

Refinement

Due to the absence of anomalous scatterers, the absolute configuration could not be determined and was arbitrarily set. Friedel pairs were merged. All H atoms were geometrically positioned and refined using a riding model with C—H = 0.93–0.97 Å and U_{iso}(methyl H) = 1.5 U_{eq}(C) and 1.2 U_{eq}(C, O) for other H atoms. The methyl and hydroxyl group were allowed to rotate but not to tip.

Computing details

Data collection: *CrystalClear* (Rigaku, 2011); cell refinement: *CrystalClear* (Rigaku, 2011); data reduction: *CrystalClear* (Rigaku, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

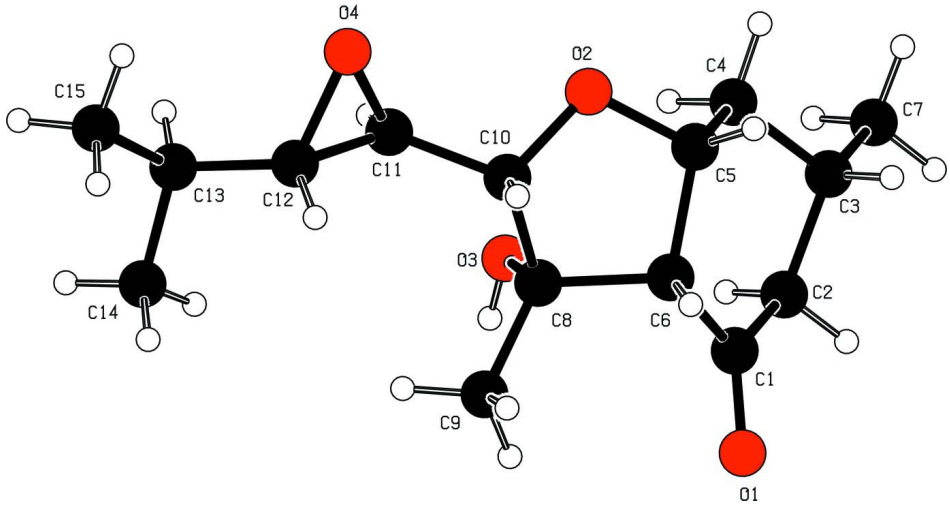


Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atom ise presented as small spheres of arbitrary radius.

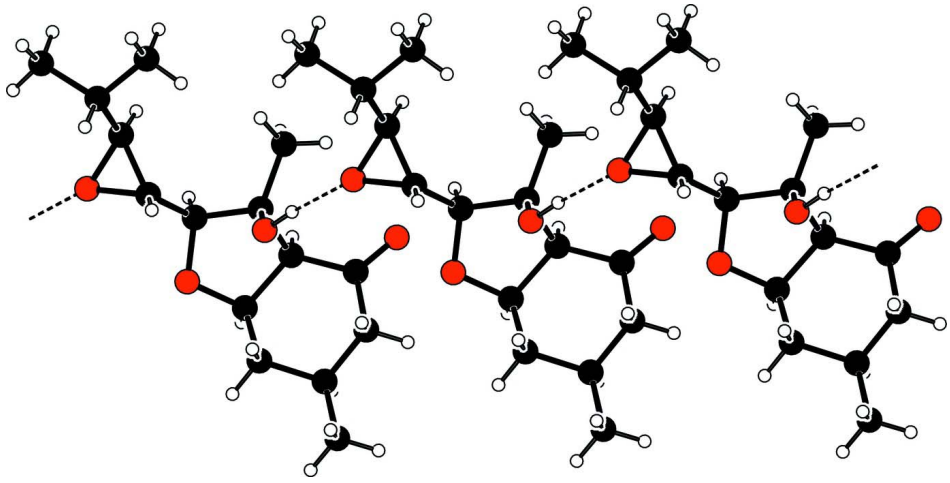


Figure 2

A packing diagram for the title compound showing O—H···O intra-molecular hydrogen bonds (dashed lines).

(2*S,3*S**,3a*S**,6*S**,7a*R**)-3-Hydroxy- 2-[(2*R**,3*S**)-3-isopropyloxiran-2-yl]-3,6-dimethyl- 3,3a,5,6,7,7a-hexahydro-1-benzofuran-4(2*H*)-one**

Crystal data

$C_{15}H_{24}O_4$
$M_r = 268.34$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.616$ (7) Å
$b = 9.261$ (9) Å
$c = 25.12$ (3) Å
$V = 1539$ (3) Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.158$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2058 reflections
$\theta = 2.7$–$27.5°$
$\mu = 0.08$ mm^{-1}
$T = 296$ K
Prism, colorless
0.35 × 0.28 × 0.26 mm

Data collection

Rigaku model name? CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2011)
$T_{min} = 0.972$, $T_{max} = 0.980$
16467 measured reflections
2058 independent reflections
1568 reflections with $I > 2\sigma(I)$
$R_{int} = 0.176$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.7°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.185$
$S = 1.03$
2058 reflections
177 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0887P)^2 + 0.2181P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3330 (6)	0.7529 (5)	0.08201 (17)	0.0809 (11)
C2	0.2584 (5)	0.8871 (5)	0.05651 (16)	0.0771 (11)
H2A	0.2052	0.9501	0.0840	0.093*
H2B	0.1474	0.8623	0.0330	0.093*
C3	0.4150 (6)	0.9702 (5)	0.02476 (14)	0.0788 (11)
H3	0.4605	0.9093	−0.0048	0.095*
C4	0.5947 (6)	1.0012 (5)	0.06053 (14)	0.0793 (11)
H4A	0.6926	1.0586	0.0411	0.095*
H4B	0.5500	1.0572	0.0910	0.095*
C5	0.6938 (5)	0.8657 (5)	0.07967 (14)	0.0748 (11)
H5	0.7693	0.8228	0.0501	0.090*
C6	0.5481 (5)	0.7502 (4)	0.10272 (15)	0.0706 (9)
H6	0.6049	0.6551	0.0944	0.085*
C7	0.3260 (10)	1.1082 (7)	0.0018 (2)	0.1196 (19)
H7A	0.2137	1.0847	−0.0208	0.179*
H7B	0.4273	1.1577	−0.0185	0.179*
H7C	0.2808	1.1692	0.0303	0.179*
C8	0.5676 (5)	0.7732 (3)	0.16381 (14)	0.0626 (8)
C9	0.5010 (8)	0.6450 (4)	0.1976 (2)	0.0937 (14)
H9A	0.5276	0.6650	0.2344	0.141*
H9B	0.5745	0.5605	0.1869	0.141*
H9C	0.3589	0.6288	0.1927	0.141*
C10	0.7945 (5)	0.8060 (4)	0.16665 (15)	0.0658 (9)
H10	0.8702	0.7159	0.1621	0.079*
C11	0.8628 (4)	0.8785 (4)	0.21676 (13)	0.0581 (8)
H11	0.7903	0.9670	0.2264	0.070*
C12	0.9471 (5)	0.7963 (4)	0.26096 (14)	0.0634 (8)
H12	0.9520	0.6916	0.2554	0.076*
C13	0.9347 (5)	0.8420 (4)	0.31751 (14)	0.0690 (9)
H13	0.9259	0.9477	0.3180	0.083*
C14	0.7449 (8)	0.7838 (6)	0.3432 (2)	0.1076 (17)
H14A	0.6293	0.8128	0.3227	0.161*
H14B	0.7335	0.8212	0.3787	0.161*
H14C	0.7514	0.6803	0.3445	0.161*
C15	1.1278 (9)	0.7993 (8)	0.3472 (2)	0.1161 (19)
H15A	1.1347	0.6961	0.3501	0.174*
H15B	1.1262	0.8411	0.3822	0.174*

H15C	1.2434	0.8341	0.3280	0.174*
O1	0.2253 (6)	0.6483 (5)	0.08748 (18)	0.1301 (15)
O2	0.8347 (3)	0.8990 (3)	0.12242 (9)	0.0749 (7)
O3	0.4646 (3)	0.9014 (2)	0.17901 (9)	0.0613 (6)
H3A	0.3551	0.8803	0.1922	0.092*
O4	1.0785 (3)	0.8795 (4)	0.22651 (11)	0.0802 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.074 (2)	0.087 (3)	0.081 (2)	−0.021 (2)	−0.003 (2)	−0.014 (2)
C2	0.0604 (18)	0.100 (3)	0.071 (2)	−0.010 (2)	0.0036 (16)	−0.015 (2)
C3	0.080 (2)	0.102 (3)	0.0548 (17)	−0.005 (2)	0.0107 (18)	−0.0113 (19)
C4	0.081 (2)	0.093 (3)	0.0630 (19)	−0.022 (2)	0.0109 (19)	0.004 (2)
C5	0.0617 (17)	0.106 (3)	0.0564 (17)	−0.009 (2)	0.0154 (16)	−0.022 (2)
C6	0.070 (2)	0.0609 (18)	0.081 (2)	−0.0009 (17)	0.0061 (18)	−0.0212 (17)
C7	0.133 (4)	0.135 (5)	0.090 (3)	−0.002 (4)	−0.015 (3)	0.025 (3)
C8	0.0639 (18)	0.0495 (15)	0.075 (2)	−0.0059 (15)	0.0072 (16)	−0.0049 (15)
C9	0.095 (3)	0.063 (2)	0.123 (4)	−0.020 (2)	−0.008 (3)	0.020 (2)
C10	0.0593 (17)	0.0631 (19)	0.075 (2)	0.0080 (16)	0.0098 (17)	−0.0040 (18)
C11	0.0485 (15)	0.0563 (17)	0.0695 (18)	0.0006 (14)	0.0097 (14)	0.0032 (15)
C12	0.0545 (16)	0.0559 (16)	0.080 (2)	0.0060 (15)	0.0053 (16)	0.0029 (16)
C13	0.074 (2)	0.0594 (17)	0.073 (2)	−0.0010 (17)	0.0024 (18)	0.0082 (16)
C14	0.129 (4)	0.096 (3)	0.098 (3)	−0.018 (3)	0.036 (3)	0.012 (3)
C15	0.121 (4)	0.128 (5)	0.099 (3)	0.023 (4)	−0.028 (3)	0.006 (3)
O1	0.124 (3)	0.114 (3)	0.153 (3)	−0.058 (2)	−0.045 (3)	0.012 (3)
O2	0.0550 (12)	0.104 (2)	0.0660 (13)	−0.0143 (14)	0.0110 (11)	−0.0024 (13)
O3	0.0545 (11)	0.0597 (12)	0.0696 (13)	−0.0028 (10)	0.0165 (10)	−0.0034 (11)
O4	0.0488 (11)	0.110 (2)	0.0821 (16)	−0.0093 (15)	0.0106 (11)	0.0071 (16)

Geometric parameters (Å, º)

C1—O1	1.210 (6)	C9—H9A	0.9600
C1—C2	1.482 (7)	C9—H9B	0.9600
C1—C6	1.515 (6)	C9—H9C	0.9600
C2—C3	1.517 (6)	C10—O2	1.431 (4)
C2—H2A	0.9700	C10—C11	1.497 (5)
C2—H2B	0.9700	C10—H10	0.9800
C3—C4	1.518 (5)	C11—O4	1.448 (4)
C3—C7	1.520 (8)	C11—C12	1.457 (5)
C3—H3	0.9800	C11—H11	0.9800
C4—C5	1.495 (7)	C12—O4	1.448 (4)
C4—H4A	0.9700	C12—C13	1.484 (5)
C4—H4B	0.9700	C12—H12	0.9800
C5—O2	1.455 (4)	C13—C14	1.512 (6)
C5—C6	1.552 (6)	C13—C15	1.531 (6)
C5—H5	0.9800	C13—H13	0.9800
C6—C8	1.554 (5)	C14—H14A	0.9600
C6—H6	0.9800	C14—H14B	0.9600
C7—H7A	0.9600	C14—H14C	0.9600

C7—H7B	0.9600	C15—H15A	0.9600
C7—H7C	0.9600	C15—H15B	0.9600
C8—O3	1.420 (4)	C15—H15C	0.9600
C8—C9	1.525 (5)	O3—H3A	0.8200
C8—C10	1.533 (5)		

O1—C1—C2	121.6 (4)	C8—C9—H9A	109.5
O1—C1—C6	120.0 (5)	C8—C9—H9B	109.5
C2—C1—C6	118.4 (4)	H9A—C9—H9B	109.5
C1—C2—C3	115.2 (4)	C8—C9—H9C	109.5
C1—C2—H2A	108.5	H9A—C9—H9C	109.5
C3—C2—H2A	108.5	H9B—C9—H9C	109.5
C1—C2—H2B	108.5	O2—C10—C11	109.1 (3)
C3—C2—H2B	108.5	O2—C10—C8	105.4 (3)
H2A—C2—H2B	107.5	C11—C10—C8	115.1 (3)
C4—C3—C7	111.7 (4)	O2—C10—H10	109.1
C4—C3—C2	108.6 (3)	C11—C10—H10	109.1
C7—C3—C2	111.2 (4)	C8—C10—H10	109.1
C4—C3—H3	108.4	O4—C11—C12	59.8 (2)
C7—C3—H3	108.4	O4—C11—C10	116.3 (3)
C2—C3—H3	108.4	C12—C11—C10	121.5 (3)
C5—C4—C3	112.1 (4)	O4—C11—H11	115.8
C5—C4—H4A	109.2	C12—C11—H11	115.8
C3—C4—H4A	109.2	C10—C11—H11	115.8
C5—C4—H4B	109.2	O4—C12—C11	59.8 (2)
C3—C4—H4B	109.2	O4—C12—C13	116.9 (3)
H4A—C4—H4B	107.9	C11—C12—C13	124.0 (3)
O2—C5—C4	109.9 (3)	O4—C12—H12	114.9
O2—C5—C6	105.6 (3)	C11—C12—H12	114.9
C4—C5—C6	115.2 (3)	C13—C12—H12	114.9
O2—C5—H5	108.7	C12—C13—C14	110.6 (4)
C4—C5—H5	108.7	C12—C13—C15	110.3 (3)
C6—C5—H5	108.7	C14—C13—C15	113.1 (4)
C1—C6—C5	116.3 (4)	C12—C13—H13	107.5
C1—C6—C8	114.5 (3)	C14—C13—H13	107.5
C5—C6—C8	102.8 (3)	C15—C13—H13	107.5
C1—C6—H6	107.6	C13—C14—H14A	109.5
C5—C6—H6	107.6	C13—C14—H14B	109.5
C8—C6—H6	107.6	H14A—C14—H14B	109.5
C3—C7—H7A	109.5	C13—C14—H14C	109.5
C3—C7—H7B	109.5	H14A—C14—H14C	109.5
H7A—C7—H7B	109.5	H14B—C14—H14C	109.5
C3—C7—H7C	109.5	C13—C15—H15A	109.5
H7A—C7—H7C	109.5	C13—C15—H15B	109.5
H7B—C7—H7C	109.5	H15A—C15—H15B	109.5
O3—C8—C9	111.2 (3)	C13—C15—H15C	109.5
O3—C8—C10	107.0 (3)	H15A—C15—H15C	109.5
C9—C8—C10	114.3 (3)	H15B—C15—H15C	109.5
O3—C8—C6	109.9 (3)	C10—O2—C5	109.0 (3)

C9—C8—C6	114.7 (3)	C8—O3—H3A	109.5
C10—C8—C6	98.9 (3)	C11—O4—C12	60.4 (2)
O1—C1—C2—C3	−146.8 (5)	C9—C8—C10—O2	164.2 (3)
C6—C1—C2—C3	34.8 (5)	C6—C8—C10—O2	41.9 (3)
C1—C2—C3—C4	−54.8 (5)	O3—C8—C10—C11	48.0 (4)
C1—C2—C3—C7	−178.1 (4)	C9—C8—C10—C11	−75.6 (4)
C7—C3—C4—C5	−175.3 (4)	C6—C8—C10—C11	162.1 (3)
C2—C3—C4—C5	61.8 (4)	O2—C10—C11—O4	−76.3 (4)
C3—C4—C5—O2	−167.6 (3)	C8—C10—C11—O4	165.6 (3)
C3—C4—C5—C6	−48.5 (4)	O2—C10—C11—C12	−145.5 (3)
O1—C1—C6—C5	161.9 (4)	C8—C10—C11—C12	96.4 (4)
C2—C1—C6—C5	−19.6 (5)	C10—C11—C12—O4	104.2 (3)
O1—C1—C6—C8	−78.2 (6)	O4—C11—C12—C13	103.9 (4)
C2—C1—C6—C8	100.2 (4)	C10—C11—C12—C13	−152.0 (3)
O2—C5—C6—C1	148.0 (3)	O4—C12—C13—C14	160.0 (3)
C4—C5—C6—C1	26.6 (5)	C11—C12—C13—C14	89.8 (4)
O2—C5—C6—C8	22.0 (4)	O4—C12—C13—C15	−74.1 (4)
C4—C5—C6—C8	−99.4 (3)	C11—C12—C13—C15	−144.3 (4)
C1—C6—C8—O3	−53.2 (4)	C11—C10—O2—C5	−154.0 (3)
C5—C6—C8—O3	74.0 (3)	C8—C10—O2—C5	−29.9 (4)
C1—C6—C8—C9	73.1 (5)	C4—C5—O2—C10	129.2 (3)
C5—C6—C8—C9	−159.8 (3)	C6—C5—O2—C10	4.4 (4)
C1—C6—C8—C10	−164.9 (3)	C10—C11—O4—C12	−112.7 (4)
C5—C6—C8—C10	−37.8 (3)	C13—C12—O4—C11	−115.5 (3)
O3—C8—C10—O2	−72.2 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O4^{i}	0.82	2.02	2.827 (4)	166

Symmetry code: (i) *x*−1, *y*, *z*.