

# 12,15-Dimethyl-8-oxatetracyclo[8.8.0.0<sup>2,7</sup>.0<sup>11,16</sup>]-octadeca-1(18),2,4,6,11(16),12,14-heptaen-10-ol

Alan J. Lough,<sup>a\*</sup> Samuel Koh<sup>b</sup> and William Tam<sup>b</sup><sup>a</sup>Department of Chemistry, University of Toronto, Toronto, Ontario, M5S 3H6, Canada, and <sup>b</sup>Department of Chemistry, University of Guelph, Guelph, Ontario, N1G 2W1, Canada. \*Correspondence e-mail: alan.lough@utoronto.ca

Received 2 March 2020

Accepted 6 March 2020

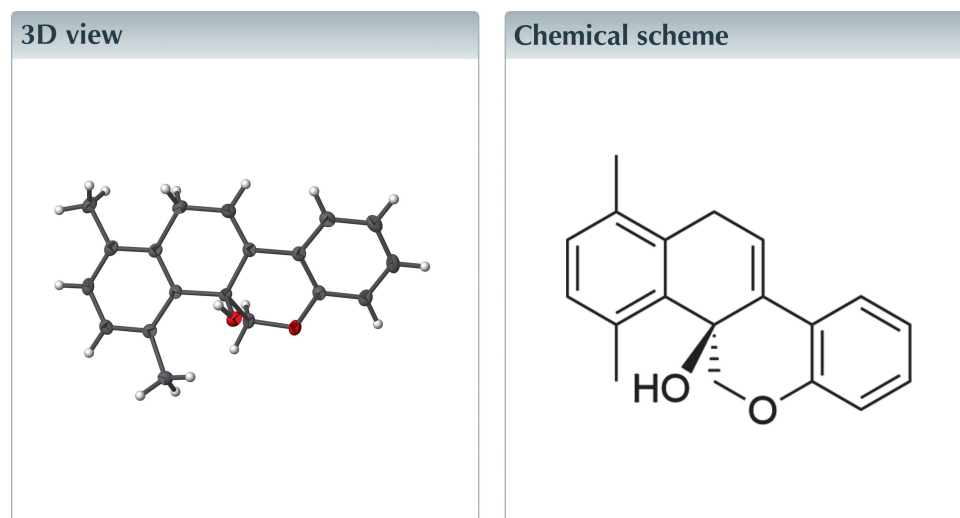
Edited by W. T. A. Harrison, University of Aberdeen, Scotland

Keywords: crystal structure; ring-opening; regioselectivity; hydrogen bonds.

CCDC reference: 1988571

Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

In the title compound, C<sub>19</sub>H<sub>18</sub>O<sub>2</sub>, the pyran ring is in a half-chair conformation. The fused ring system comprising the benzene and cyclohexene rings is essentially planar (r.m.s. deviation = 0.053 Å) and forms a dihedral angle of 27.95 (6)° with the other benzene ring. In the crystal, O—H...O hydrogen bonds connect the molecules into chains propagating along [001].



## Structure description

The ring-opening reaction of oxabenzonorbornadiene (OBD) has been well studied by many groups including our own (Lautens *et al.*, 2003; Rayabarapu & Cheng, 2007; Boutin *et al.*, 2019; Hill *et al.*, 2019; Hill & Tam, 2019). Building on the work of Cheng (Duan & Cheng, 1995), our group has also demonstrated the palladium-catalysed regioselective ring-opening of C<sub>1</sub>-substituted OBDs using aryl iodides (Raheem *et al.*, 2014). However, to the best of our knowledge, intramolecular modes of this reactivity have been left unexplored. Currently, the only known intramolecular transformation of OBD was reported by the Lautens group (Loh *et al.*, 2016) with a similar transformation recently reported by our group on cyclopropanated OBD (Wicks *et al.*, 2019). Based on this, we set out to investigate palladium-catalysed intramolecular ring-openings of OBD with C<sub>1</sub>-tethered aryl halides. The reaction of C<sub>1</sub>-substituted OBD **I** (see Fig. 1) in the presence of PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>, Zn, Et<sub>3</sub>N, and MeCN afforded an expected dehydrated product **II** in 82% yield, as well as an unexpected and yet unreported hydrated product **III** in 14% yield. The structure of the alcohol-containing fused tetracycle **III** was confirmed by single-crystal X-ray analysis.

The molecular structure of the title compound is shown in Fig. 2. The pyran ring (O1/C1/C2/C11/C12/C17) is in a half-chair conformation with atoms C1 and C2 deviating from the mean-plane of the other four atoms by −0.197 (2) and 0.556 (1) Å, respectively. The fused ring system comprising the benzene (C3–C8) and cyclohexene (C2/C3/C8–C11)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O2-H2O\cdots O1^i$	0.86 (2)	2.03 (2)	2.8805 (14)	171.0 (19)

Symmetry code: (i)  $x, -y + \frac{3}{2}, z - \frac{1}{2}$ .

rings is essentially planar (r.m.s. deviation = 0.053 Å) and forms a dihedral angle of 27.95 (6)° with the other benzene ring (C12–C17). In the arbitrarily chosen asymmetric unit, atom C2 has an *S* configuration but crystal symmetry generates a racemic mixture. In the crystal, O–H···O hydrogen bonds (Table 1) connect the molecules into chains propagating along [001] (Fig. 3).

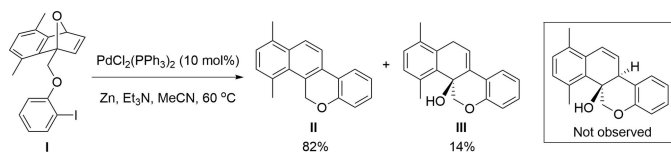
### Synthesis and crystallization

To a 2 dram vial was added oxabenzonorbornadiene **I** (Fig. 1) (67.8 mg, 0.168 mmol), then purged with argon before importing into a glove box under an inert argon atmosphere. The vial was loaded sequentially with Zn (123.3 mg, 1.89 mmol, 11.2 eq.), MeCN (1.5 ml), Et<sub>3</sub>N (0.09 ml, 0.669 mmol, 0.25 eq.) and PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (12.5 mg, 0.0178 mmol, 10.6 mol%), then exported and stirred at 333 K for 1 day. The mixture was cooled to room temperature and stirred in air for 10 minutes before removing the solvent under reduced pressure. The crude mixture was then purified by flash

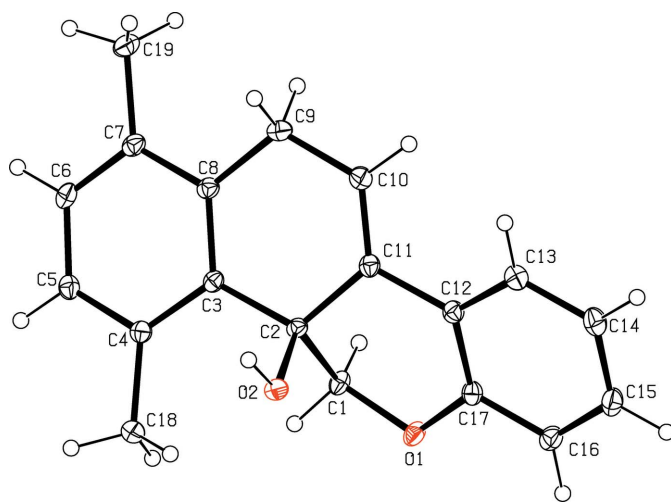
**Table 2**  
Experimental details.

Crystal data	
Chemical formula	C <sub>19</sub> H <sub>18</sub> O <sub>2</sub>
$M_r$	278.33
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
$a, b, c$ (Å)	12.2712 (7), 11.2934 (6), 10.8984 (7)
$\beta$ (°)	112.565 (2)
$V$ (Å <sup>3</sup> )	1394.71 (14)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.09
Crystal size (mm)	0.25 × 0.19 × 0.11
Data collection	
Diffractometer	Bruker Kappa APEX DUO CCD
Absorption correction	Multi-scan (SADABS; Krause <i>et al.</i> , 2015)
$T_{min}, T_{max}$	0.703, 0.746
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	22859, 3212, 2404
$R_{int}$	0.040
$(\sin \theta/\lambda)_{max}$ (Å <sup>-1</sup> )	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.040, 0.110, 1.04
No. of reflections	3212
No. of parameters	196
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.31, -0.22

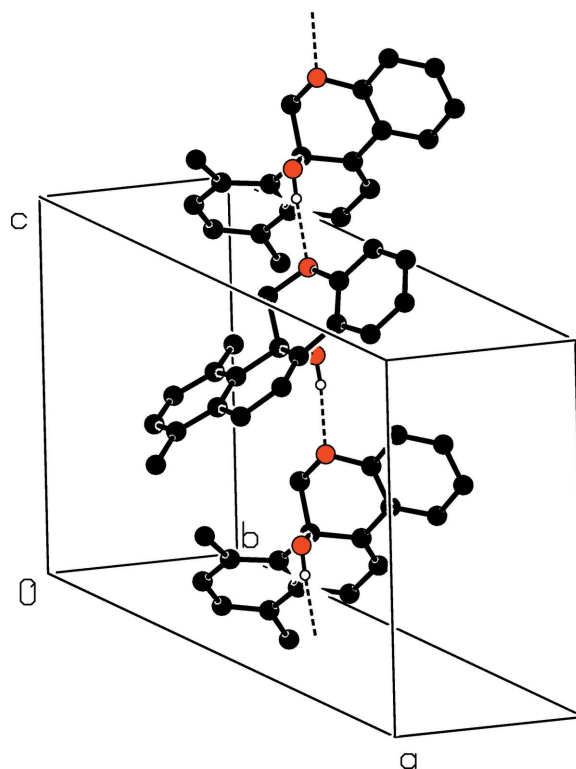
Computer programs: APEX3 (Bruker, 2018), SAINT (Bruker, 2018), SHELXT2014 (Sheldrick, 2015a), SHELXL2018 (Sheldrick, 2015b), PLATON (Spek, 2020), pubCIF (Westrip, 2010).



**Figure 1**  
The reaction scheme



**Figure 2**  
The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.



**Figure 3**  
Part of the crystal structure with O–H···O hydrogen bonds shown as dashed lines.

column chromatography using gradient elution (EtOAc:hexanes 1:9 to EtOAc:hexanes 1:4) to obtain the ring-opened product **II** (35.8 mg, 82%) as a white solid and **III** (6.6 mg, 14%) as a white solid. The product **III** was subsequently crystallized from methylene chloride solution by slow evaporation to give product **III** as colourless crystals with orange specks.

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

### Acknowledgements

The University of Toronto thanks NSERC Canada for funding.

### References

Boutin, R., Koh, S. & Tam, W. (2019). *Curr. Org. Synth.* **16**, 460–484.

- Bruker (2018). *APEX3* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Duan, J. & Cheng, C. H. (1995). *Organometallics*, **14**, 1608–1618.
- Hill, J. & Tam, W. (2019). *J. Org. Chem.* **84**, 8309–8314.
- Hill, J., Wicks, C., Pounder, A. & Tam, W. (2019). *Tetrahedron Lett.* **60**, 150990.
- Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
- Lautens, M., Fagnou, K. & Hiebert, S. (2003). *Acc. Chem. Res.* **36**, 48–58.
- Loh, C. C. J., Schmid, M., Webster, R., Yen, A., Yazdi, S. K., Franke, P. T. & Lautens, M. (2016). *Angew. Chem. Int. Ed.* **55**, 10074–10078.
- Raheem, M. A., Edmunds, M. & Tam, W. (2014). *Can. J. Chem.* **92**, 888–895.
- Rayabarapu, D. K. & Cheng, C.-H. (2007). *Acc. Chem. Res.* **40**, 971–983.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Wicks, C., Koh, S., Pounder, A., Carlson, E. & Tam, W. (2019). *Tetrahedron Lett.* **60**, 151228.

## full crystallographic data

*IUCrData* (2020). 5, x200315 [https://doi.org/10.1107/S2414314620003156]

## 12,15-Dimethyl-8-oxatetracyclo- [8.8.0.0<sup>2,7</sup>.0<sup>11,16</sup>]octadeca-1(18),2,4,6,11(16),12,14-heptaen-10-ol

Alan J. Lough, Samuel Koh and William Tam

12,15-Dimethyl-8-oxatetracyclo[8.8.0.0<sup>2,7</sup>.0<sup>11,16</sup>]octadeca-1(18),2,4,6,11(16),12,14-heptaen-10-ol

### Crystal data

C<sub>19</sub>H<sub>18</sub>O<sub>2</sub>

$M_r = 278.33$

Monoclinic,  $P2_1/c$

$a = 12.2712$  (7) Å

$b = 11.2934$  (6) Å

$c = 10.8984$  (7) Å

$\beta = 112.565$  (2)°

$V = 1394.71$  (14) Å<sup>3</sup>

$Z = 4$

$F(000) = 592$

$D_x = 1.326$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 6674 reflections

$\theta = 2.6$ – $27.5$ °

$\mu = 0.09$  mm<sup>-1</sup>

$T = 150$  K

Shard, colourless

0.25 × 0.19 × 0.11 mm

### Data collection

Bruker Kappa APEX DUO CCD  
diffractometer

Radiation source: sealed tube with Bruker  
Triumph monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.703$ ,  $T_{\max} = 0.746$

22859 measured reflections

3212 independent reflections

2404 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\max} = 27.5$ °,  $\theta_{\min} = 1.8$ °

$h = -15$ → $15$

$k = -14$ → $12$

$l = -14$ → $14$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.110$

$S = 1.03$

3212 reflections

196 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.6316P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.31$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.21$  e Å<sup>-3</sup>

### 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.38362 (9)	0.71483 (9)	0.94095 (10)	0.0227 (2)
O2	0.35317 (9)	0.79851 (9)	0.68983 (10)	0.0195 (2)
H2O	0.3639 (17)	0.8028 (17)	0.616 (2)	0.041 (6)*
C1	0.28530 (12)	0.67697 (13)	0.82436 (13)	0.0194 (3)
H1A	0.216856	0.729079	0.811291	0.023*
H1B	0.263028	0.595336	0.838378	0.023*
C2	0.31342 (12)	0.67990 (12)	0.69883 (13)	0.0165 (3)
C3	0.20305 (12)	0.64875 (12)	0.57662 (13)	0.0168 (3)
C4	0.10115 (12)	0.72098 (12)	0.54010 (14)	0.0183 (3)
C5	-0.00046 (13)	0.68454 (13)	0.43491 (14)	0.0207 (3)
H5A	-0.070186	0.730804	0.411431	0.025*
C6	-0.00220 (13)	0.58267 (13)	0.36392 (14)	0.0217 (3)
H6A	-0.073619	0.558560	0.294864	0.026*
C7	0.09851 (13)	0.51547 (13)	0.39205 (14)	0.0204 (3)
C8	0.20197 (12)	0.54850 (12)	0.49950 (14)	0.0185 (3)
C9	0.31027 (13)	0.47357 (14)	0.52591 (15)	0.0266 (3)
H9A	0.289357	0.389843	0.532701	0.032*
H9B	0.333308	0.479937	0.448491	0.032*
C10	0.41426 (13)	0.50446 (14)	0.64767 (15)	0.0230 (3)
H10A	0.482918	0.456604	0.670766	0.028*
C11	0.41632 (12)	0.59543 (12)	0.72595 (13)	0.0182 (3)
C12	0.51602 (12)	0.61900 (12)	0.85341 (14)	0.0188 (3)
C13	0.63234 (13)	0.58519 (13)	0.87853 (15)	0.0235 (3)
H13A	0.649773	0.547125	0.810468	0.028*
C14	0.72254 (13)	0.60601 (14)	1.00035 (16)	0.0266 (3)
H14A	0.800938	0.582302	1.015531	0.032*
C15	0.69775 (13)	0.66181 (14)	1.10041 (16)	0.0258 (3)
H15A	0.759337	0.675389	1.184465	0.031*
C16	0.58418 (13)	0.69762 (13)	1.07837 (15)	0.0231 (3)
H16A	0.567555	0.736584	1.146479	0.028*
C17	0.49430 (12)	0.67609 (13)	0.95542 (14)	0.0192 (3)
C18	0.09345 (14)	0.83818 (14)	0.60380 (16)	0.0255 (3)
H18A	0.172468	0.872790	0.644921	0.038*
H18B	0.060772	0.825342	0.671871	0.038*
H18C	0.042111	0.892264	0.535837	0.038*
C19	0.09555 (14)	0.40895 (14)	0.30771 (15)	0.0271 (3)
H19A	0.154520	0.418330	0.268138	0.041*
H19B	0.016948	0.401618	0.237010	0.041*
H19C	0.113242	0.337581	0.363080	0.041*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0194 (5)	0.0335 (6)	0.0143 (5)	0.0017 (4)	0.0056 (4)	-0.0035 (4)
O2	0.0245 (5)	0.0175 (5)	0.0182 (5)	-0.0023 (4)	0.0100 (4)	-0.0002 (4)

C1	0.0176 (7)	0.0263 (8)	0.0140 (7)	-0.0015 (6)	0.0059 (5)	-0.0003 (5)
C2	0.0197 (7)	0.0162 (7)	0.0141 (6)	-0.0010 (5)	0.0071 (5)	-0.0006 (5)
C3	0.0191 (7)	0.0186 (7)	0.0136 (6)	-0.0012 (5)	0.0073 (5)	0.0013 (5)
C4	0.0223 (7)	0.0181 (7)	0.0162 (7)	0.0000 (6)	0.0091 (6)	0.0019 (5)
C5	0.0203 (7)	0.0231 (8)	0.0182 (7)	0.0023 (6)	0.0068 (6)	0.0035 (6)
C6	0.0220 (7)	0.0255 (8)	0.0146 (7)	-0.0036 (6)	0.0036 (6)	0.0013 (6)
C7	0.0264 (7)	0.0195 (7)	0.0155 (7)	-0.0032 (6)	0.0083 (6)	0.0001 (5)
C8	0.0223 (7)	0.0183 (7)	0.0160 (7)	-0.0010 (5)	0.0085 (6)	0.0000 (5)
C9	0.0279 (8)	0.0252 (8)	0.0239 (8)	0.0039 (6)	0.0068 (6)	-0.0078 (6)
C10	0.0225 (7)	0.0247 (8)	0.0214 (7)	0.0041 (6)	0.0079 (6)	-0.0007 (6)
C11	0.0192 (7)	0.0202 (7)	0.0159 (7)	0.0005 (5)	0.0076 (5)	0.0024 (5)
C12	0.0206 (7)	0.0179 (7)	0.0176 (7)	-0.0003 (6)	0.0071 (6)	0.0021 (5)
C13	0.0231 (7)	0.0234 (8)	0.0243 (8)	0.0018 (6)	0.0094 (6)	0.0022 (6)
C14	0.0194 (7)	0.0278 (8)	0.0298 (8)	0.0025 (6)	0.0065 (6)	0.0041 (7)
C15	0.0229 (8)	0.0256 (8)	0.0221 (8)	-0.0028 (6)	0.0010 (6)	0.0033 (6)
C16	0.0263 (8)	0.0242 (8)	0.0177 (7)	-0.0018 (6)	0.0073 (6)	0.0003 (6)
C17	0.0189 (7)	0.0203 (7)	0.0180 (7)	-0.0001 (5)	0.0064 (6)	0.0032 (6)
C18	0.0250 (8)	0.0242 (8)	0.0245 (8)	0.0053 (6)	0.0063 (6)	-0.0030 (6)
C19	0.0320 (9)	0.0247 (8)	0.0215 (8)	-0.0029 (6)	0.0070 (6)	-0.0061 (6)

*Geometric parameters (Å, °)*

O1—C17	1.3771 (17)	C9—H9A	0.9900
O1—C1	1.4410 (17)	C9—H9B	0.9900
O2—C2	1.4417 (17)	C10—C11	1.329 (2)
O2—H2O	0.86 (2)	C10—H10A	0.9500
C1—C2	1.5343 (18)	C11—C12	1.4809 (19)
C1—H1A	0.9900	C12—C17	1.397 (2)
C1—H1B	0.9900	C12—C13	1.399 (2)
C2—C11	1.5184 (19)	C13—C14	1.384 (2)
C2—C3	1.5322 (19)	C13—H13A	0.9500
C3—C8	1.4071 (19)	C14—C15	1.390 (2)
C3—C4	1.4162 (19)	C14—H14A	0.9500
C4—C5	1.393 (2)	C15—C16	1.381 (2)
C4—C18	1.514 (2)	C15—H15A	0.9500
C5—C6	1.382 (2)	C16—C17	1.392 (2)
C5—H5A	0.9500	C16—H16A	0.9500
C6—C7	1.381 (2)	C18—H18A	0.9800
C6—H6A	0.9500	C18—H18B	0.9800
C7—C8	1.408 (2)	C18—H18C	0.9800
C7—C19	1.506 (2)	C19—H19A	0.9800
C8—C9	1.507 (2)	C19—H19B	0.9800
C9—C10	1.487 (2)	C19—H19C	0.9800
C17—O1—C1	117.52 (11)	H9A—C9—H9B	107.5
C2—O2—H2O	107.0 (13)	C11—C10—C9	123.53 (14)
O1—C1—C2	112.41 (11)	C11—C10—H10A	118.2
O1—C1—H1A	109.1	C9—C10—H10A	118.2

C2—C1—H1A	109.1	C10—C11—C12	123.11 (13)
O1—C1—H1B	109.1	C10—C11—C2	123.44 (13)
C2—C1—H1B	109.1	C12—C11—C2	113.31 (12)
H1A—C1—H1B	107.9	C17—C12—C13	117.53 (13)
O2—C2—C11	108.61 (11)	C17—C12—C11	119.23 (12)
O2—C2—C3	111.32 (11)	C13—C12—C11	123.23 (13)
C11—C2—C3	114.62 (11)	C14—C13—C12	121.44 (14)
O2—C2—C1	106.17 (11)	C14—C13—H13A	119.3
C11—C2—C1	105.53 (11)	C12—C13—H13A	119.3
C3—C2—C1	110.11 (11)	C13—C14—C15	119.62 (14)
C8—C3—C4	119.39 (13)	C13—C14—H14A	120.2
C8—C3—C2	120.44 (12)	C15—C14—H14A	120.2
C4—C3—C2	120.18 (12)	C16—C15—C14	120.41 (14)
C5—C4—C3	118.42 (13)	C16—C15—H15A	119.8
C5—C4—C18	116.22 (13)	C14—C15—H15A	119.8
C3—C4—C18	125.34 (13)	C15—C16—C17	119.37 (14)
C6—C5—C4	121.55 (13)	C15—C16—H16A	120.3
C6—C5—H5A	119.2	C17—C16—H16A	120.3
C4—C5—H5A	119.2	O1—C17—C16	115.93 (13)
C7—C6—C5	120.87 (13)	O1—C17—C12	122.45 (12)
C7—C6—H6A	119.6	C16—C17—C12	121.62 (13)
C5—C6—H6A	119.6	C4—C18—H18A	109.5
C6—C7—C8	118.93 (13)	C4—C18—H18B	109.5
C6—C7—C19	119.58 (13)	H18A—C18—H18B	109.5
C8—C7—C19	121.49 (13)	C4—C18—H18C	109.5
C3—C8—C7	120.59 (13)	H18A—C18—H18C	109.5
C3—C8—C9	122.15 (13)	H18B—C18—H18C	109.5
C7—C8—C9	117.25 (13)	C7—C19—H19A	109.5
C10—C9—C8	115.51 (12)	C7—C19—H19B	109.5
C10—C9—H9A	108.4	H19A—C19—H19B	109.5
C8—C9—H9A	108.4	C7—C19—H19C	109.5
C10—C9—H9B	108.4	H19A—C19—H19C	109.5
C8—C9—H9B	108.4	H19B—C19—H19C	109.5
C17—O1—C1—C2	-41.25 (17)	C7—C8—C9—C10	-174.41 (13)
O1—C1—C2—O2	-54.75 (14)	C8—C9—C10—C11	-3.5 (2)
O1—C1—C2—C11	60.43 (14)	C9—C10—C11—C12	174.19 (14)
O1—C1—C2—C3	-175.37 (11)	C9—C10—C11—C2	-1.2 (2)
O2—C2—C3—C8	123.95 (13)	O2—C2—C11—C10	-122.26 (14)
C11—C2—C3—C8	0.19 (18)	C3—C2—C11—C10	2.93 (19)
C1—C2—C3—C8	-118.58 (14)	C1—C2—C11—C10	124.26 (15)
O2—C2—C3—C4	-55.88 (16)	O2—C2—C11—C12	61.99 (14)
C11—C2—C3—C4	-179.64 (11)	C3—C2—C11—C12	-172.82 (11)
C1—C2—C3—C4	61.59 (16)	C1—C2—C11—C12	-51.50 (14)
C8—C3—C4—C5	5.28 (19)	C10—C11—C12—C17	-150.86 (15)
C2—C3—C4—C5	-174.89 (12)	C2—C11—C12—C17	24.91 (18)
C8—C3—C4—C18	-172.91 (13)	C10—C11—C12—C13	28.1 (2)
C2—C3—C4—C18	6.9 (2)	C2—C11—C12—C13	-156.10 (13)

C3—C4—C5—C6	-2.2 (2)	C17—C12—C13—C14	0.8 (2)
C18—C4—C5—C6	176.20 (13)	C11—C12—C13—C14	-178.20 (14)
C4—C5—C6—C7	-2.3 (2)	C12—C13—C14—C15	-0.1 (2)
C5—C6—C7—C8	3.6 (2)	C13—C14—C15—C16	-0.7 (2)
C5—C6—C7—C19	-176.41 (13)	C14—C15—C16—C17	0.8 (2)
C4—C3—C8—C7	-4.1 (2)	C1—O1—C17—C16	-169.75 (12)
C2—C3—C8—C7	176.07 (12)	C1—O1—C17—C12	10.5 (2)
C4—C3—C8—C9	174.87 (13)	C15—C16—C17—O1	-179.75 (13)
C2—C3—C8—C9	-5.0 (2)	C15—C16—C17—C12	0.0 (2)
C6—C7—C8—C3	-0.3 (2)	C13—C12—C17—O1	178.97 (13)
C19—C7—C8—C3	179.64 (13)	C11—C12—C17—O1	-2.0 (2)
C6—C7—C8—C9	-179.36 (13)	C13—C12—C17—C16	-0.8 (2)
C19—C7—C8—C9	0.6 (2)	C11—C12—C17—C16	178.27 (13)
C3—C8—C9—C10	6.6 (2)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2O...O1 <sup>i</sup>	0.86 (2)	2.03 (2)	2.8805 (14)	171.0 (19)

Symmetry code: (i)  $x, -y+3/2, z-1/2$ .