Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

ent-5*α*,3,15-Dioxodolabr-4(18)-ene-16,18-diol

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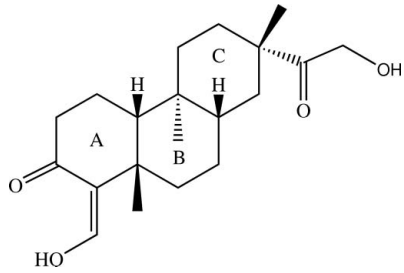
Received 12 October 2010; accepted 17 October 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.052; wR factor = 0.157; data-to-parameter ratio = 12.2.

The title compound, $C_{20}H_{30}O_4$, is a dolabrane diterpenoid isolated from *Ceriops tagal*, in which one of the three fused cyclohexane rings adopts a half-chair conformation and the other two are in the standard chair conformations. The hydroxymethylidene substituent is attached to the half-chair cyclohexane. An intramolecular O—H···O hydrogen bond generate an S(6) ring motif. In the crystal, molecules are arranged into screw chains along the [001] direction. The crystal is stabilized by O—H···O hydrogen bonds and weaker C—H···O interactions.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For ring conformations, see: Cremer & Pople (1975). For background to diterpenoids, see, for example: Hu *et al.* (2010); Zhang *et al.* (2005). For related structures, see: Chantrapromma *et al.* (2007); Fun *et al.* (2006). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).



Experimental

Crystal data

$C_{20}H_{30}O_4$
M_r = 334.44
Orthorhombic, $P2_12_12_1$
a = 7.9633 (3) Å
b = 10.7166 (4) Å
c = 20.8338 (7) Å
V = 1777.95 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.58 × 0.51 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.952, T_{max} = 0.992
20568 measured reflections
2691 independent reflections
2084 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.157
S = 1.09
2691 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1*O2*···O1	0.82	1.69	2.424 (4)	148
O4—H1*O4*···O1^{i}	0.82	2.07	2.841 (3)	156
C1—H1*B*···O2^{ii}	0.97	2.48	3.368 (5)	152
C12—H12*A*···O3	0.97	2.41	2.799 (4)	103
C17—H17*A*···O4^{iii}	0.96	2.53	3.460 (5)	164

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Prince of Songkla University for financial support through the Crystal Materials Research Unit. The authors also thank Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2353).

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supporting information

Acta Cryst. (2010). E**66**, o2897–o2898 [https://doi.org/10.1107/S1600536810042078]

ent-5*α*,3,15-Dioxodolabr-4(18)-ene-16,18-diol

Hoong-Kun Fun, Charoen Pakathirathien, Chatchanok Karalai and Suchada Chantrapromma

S1. Comment

Ceriops tagal (Perr.) C. B. Robinson is a mangrove plant belonging to the Rhizophoraceae family. Diterpenoids and triterpenoids are the main secondary metabolites of *C. tagal* (Chantrapromma *et al.*, 2007; Hu *et al.*, 2010; Zhang *et al.*, 2005). During the course of our studies on the chemical constituents and bioactive compounds from Thai medicinal plants, the title dolabrane diterpenoid compound (I), which is known as Tagalsin S (Hu *et al.*, 2010), was isolated from the the stem barks of *C. tagal*. We have also previously reported the crystal structures of two diterpenoid compounds isolated from the same plant (Chantrapromma *et al.*, 2007; Fun *et al.*, 2006). We herein report the crystal structure of (I).

The molecule of the title compound contains a fused three-ring system *A/B/C* (Fig. 1). The *A/B* ring junction is *cis*-fused and *B/C* is *trans*-fused. The cyclohexane ring *A* adopts half-chair conformation with puckering parameters Q = 0.539 (3) Å, θ = 111.0 (3)° and φ = 92.5 (4)°, rings *B* and *C* are in standard chair conformations (Cremer & Pople 1975). The hydroxylmethylidine substituent is planarly attached to cyclohexane ring *A* at atom C4 as indicated by the torsion angle C3—C4—C18—O2 of 4.4 (5)° and the bond angles around atom C4 are indicative of sp^2 hybridization for this atom. The orientations of the carbonyl and alcohol substituent groups at atom C13 are described by the torsion angles C13–C15—C16—O4 = 166.3 (3)° and O3–C15–C16–O4 = -11.1 (5)°. Intramolecular O2—H1O2···O1 hydrogen bond (Table 1) generates S(6) ring motif (Fig. 1) (Bernstein *et al.*, 1995). The bond distances are of normal values (Allen *et al.*, 1987) and are comparable with the related structures (Chantrapromma *et al.*, 2007; Fun *et al.*, 2006).

In the crystal structure (Fig. 2), the molecules are arranged into screw chains along the [0 0 1] direction and the adjacent chains are further linked by weak C—H···O interactions (Table 1). The crystal packing of (I) is stabilized by intermolecular O—H···O hydrogen bonds and weak C—H···O interactions (Fig. 2 and Table 1).

S2. Experimental

The air-dried and crushed stem barks of *C. tagal* (4.8 kg) were extracted with methylene chloride and then concentrated *in vacuo* to give a residue (17.4 g). This residue was subjected to quick column chromatography over silica gel using solvents of increasing polarity from hexane through 50% acetone/hexane. The eluates were collected and combined, based on TLC, to give 20 fractions (F1—F20). Fraction F14 was further purified by repeated quick column chromatography with CH_2Cl_2/acetone (9:1 *v*/*v*) yielding title compound (30.4 mg). Colorless block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from hexane/CH_2Cl_2 (1:1, *v*/*v*) after several days, Mp. 395–396 K.

S3. Refinement

All H atoms were placed in calculated positions with d(O—H) = 0.82 Å and d(C—H) = 0.93 Å for aromatic and CH, 0.97 for CH_2 and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxy and methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The

highest residual electron density peak is located at 0.18 Å from H1B and the deepest hole is located at 0.50 Å from O2. A total of 2024 Friedel pairs were merged before final refinement as there is no large anomalous dispersion for the determination of the absolute configuration.

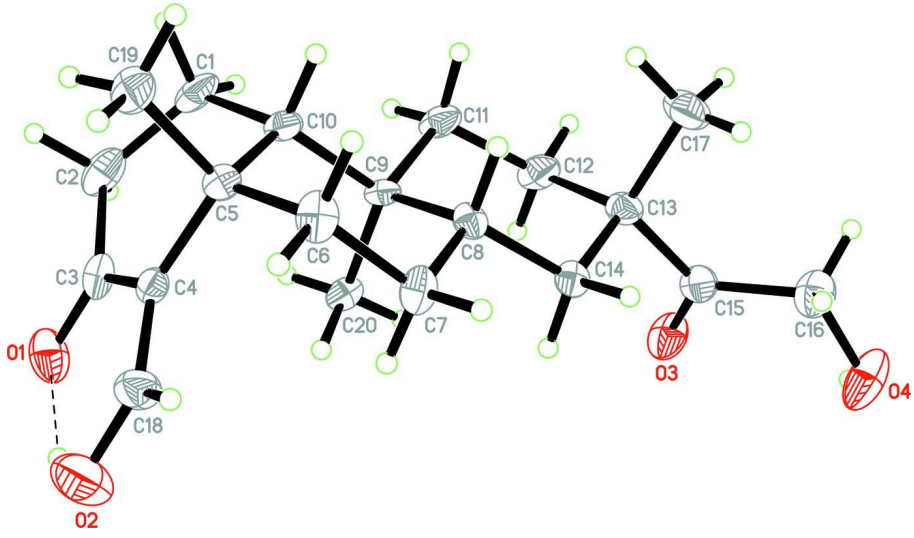

Figure 1

The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds was drawn as dash line.

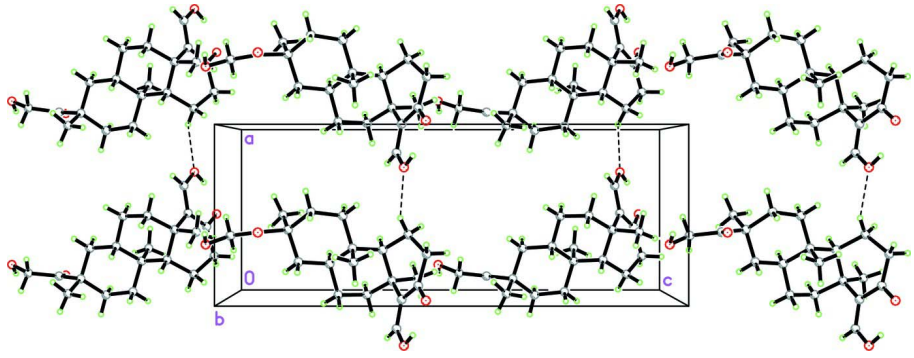

Figure 2

The crystal packing of (I) viewed along the *b* axis, showing one dimensional chains along the [0 0 1] direction. Hydrogen bonds were shown as dashed lines.

ent-5*α*,3,15-Dioxodolabr-4(18)-ene-16,18-diol

Crystal data

$C_{20}H_{30}O_4$
M_r = 334.44
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 7.9633 (3) Å
b = 10.7166 (4) Å
c = 20.8338 (7) Å
V = 1777.95 (11) Å^3
Z = 4
F(000) = 728
D_x = 1.249 Mg m^{-3}
Melting point = 495–496 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2691 reflections
θ = 2.0–29.0°
μ = 0.09 mm^{-1}
T = 100 K
Plate, colourless
0.58 × 0.51 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.952, T_{max} = 0.992
20568 measured reflections
2691 independent reflections
2084 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 29.0°, θ_{min} = 2.0°
h = −10→10
k = −14→10
l = −24→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.157
S = 1.09
2691 reflections
220 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
$w = 1/[\sigma^2(F_o^2) + (0.077P)^2 + 0.7994P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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}
O1	0.9767 (3)	0.4254 (2)	0.05460 (11)	0.0420 (6)
O2	1.2362 (4)	0.3517 (3)	0.10215 (15)	0.0633 (8)
H1O2	1.1724	0.3988	0.0830	0.095*
O3	0.5983 (4)	0.4271 (2)	0.41250 (10)	0.0413 (6)
O4	0.6697 (4)	0.3378 (2)	0.52946 (11)	0.0552 (8)
H1O4	0.6102	0.4000	0.5273	0.083*
C1	0.6196 (4)	0.2200 (3)	0.09423 (15)	0.0360 (8)
H1A	0.5759	0.1550	0.0665	0.043*
H1B	0.5242	0.2658	0.1110	0.043*
C2	0.7243 (4)	0.3092 (3)	0.05327 (15)	0.0379 (8)
H2A	0.7218	0.2792	0.0094	0.045*
H2B	0.6696	0.3900	0.0537	0.045*
C3	0.9026 (4)	0.3276 (3)	0.07172 (13)	0.0294 (7)
C4	0.9885 (3)	0.2325 (3)	0.10845 (12)	0.0221 (5)

C5	0.8927 (4)	0.1186 (3)	0.13108 (13)	0.0230 (6)
C6	0.9782 (4)	0.0532 (3)	0.18765 (13)	0.0301 (7)
H6A	0.9239	−0.0267	0.1947	0.036*
H6B	1.0944	0.0370	0.1765	0.036*
C7	0.9731 (4)	0.1276 (3)	0.24997 (14)	0.0272 (6)
H7A	1.0337	0.2054	0.2446	0.033*
H7B	1.0269	0.0805	0.2840	0.033*
C8	0.7911 (4)	0.1547 (2)	0.26801 (13)	0.0226 (6)
H8A	0.7344	0.0737	0.2708	0.027*
C9	0.6987 (3)	0.2298 (2)	0.21580 (13)	0.0200 (5)
C10	0.7108 (4)	0.1569 (3)	0.15117 (13)	0.0241 (6)
H10A	0.6510	0.0783	0.1585	0.029*
C11	0.5122 (4)	0.2385 (3)	0.23683 (14)	0.0318 (7)
H11A	0.4514	0.2889	0.2059	0.038*
H11B	0.4636	0.1555	0.2363	0.038*
C12	0.4884 (4)	0.2955 (3)	0.30434 (14)	0.0338 (7)
H12A	0.5200	0.3828	0.3030	0.041*
H12B	0.3705	0.2912	0.3157	0.041*
C13	0.5915 (4)	0.2302 (3)	0.35673 (14)	0.0276 (6)
C14	0.7769 (4)	0.2146 (2)	0.33451 (13)	0.0227 (6)
H14A	0.8305	0.2958	0.3336	0.027*
H14B	0.8364	0.1633	0.3654	0.027*
C15	0.6017 (4)	0.3139 (3)	0.41616 (14)	0.0290 (6)
C16	0.6242 (5)	0.2539 (3)	0.48109 (14)	0.0394 (8)
H16A	0.5201	0.2133	0.4932	0.047*
H16B	0.7100	0.1900	0.4778	0.047*
C17	0.5129 (5)	0.1042 (3)	0.37320 (18)	0.0463 (9)
H17A	0.4050	0.1172	0.3926	0.070*
H17B	0.5844	0.0603	0.4026	0.070*
H17C	0.4998	0.0559	0.3347	0.070*
C18	1.1538 (4)	0.2500 (3)	0.11951 (15)	0.0342 (7)
H18A	1.2130	0.1872	0.1404	0.041*
C19	0.8839 (5)	0.0238 (3)	0.07522 (15)	0.0352 (7)
H19A	0.9938	−0.0099	0.0674	0.053*
H19B	0.8441	0.0649	0.0372	0.053*
H19C	0.8085	−0.0427	0.0864	0.053*
C20	0.7677 (4)	0.3630 (2)	0.21044 (13)	0.0236 (6)
H20A	0.7370	0.4095	0.2480	0.035*
H20B	0.7213	0.4026	0.1731	0.035*
H20C	0.8878	0.3602	0.2068	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0564 (16)	0.0328 (12)	0.0369 (12)	0.0036 (12)	0.0098 (12)	0.0126 (10)
O2	0.0435 (16)	0.0705 (19)	0.076 (2)	−0.0151 (15)	0.0071 (15)	0.0049 (17)
O3	0.0643 (16)	0.0281 (11)	0.0314 (11)	0.0050 (12)	−0.0030 (12)	−0.0080 (10)
O4	0.088 (2)	0.0437 (14)	0.0337 (12)	0.0225 (15)	−0.0180 (13)	−0.0096 (11)

C1	0.0222 (14)	0.056 (2)	0.0301 (15)	0.0036 (14)	−0.0107 (12)	−0.0167 (15)
C2	0.0410 (19)	0.0438 (19)	0.0290 (16)	0.0139 (16)	−0.0147 (14)	−0.0040 (14)
C3	0.0375 (16)	0.0331 (16)	0.0177 (13)	0.0076 (14)	0.0011 (12)	0.0008 (12)
C4	0.0231 (13)	0.0252 (13)	0.0180 (12)	0.0007 (11)	−0.0014 (10)	−0.0005 (10)
C5	0.0233 (13)	0.0212 (13)	0.0245 (13)	0.0021 (11)	−0.0020 (11)	−0.0020 (10)
C6	0.0374 (17)	0.0228 (14)	0.0303 (14)	0.0123 (13)	0.0022 (13)	0.0022 (12)
C7	0.0310 (15)	0.0272 (14)	0.0232 (12)	0.0127 (13)	−0.0015 (12)	0.0032 (11)
C8	0.0289 (14)	0.0133 (11)	0.0254 (13)	0.0001 (11)	0.0041 (11)	0.0011 (10)
C9	0.0154 (11)	0.0198 (12)	0.0248 (13)	−0.0013 (10)	−0.0024 (10)	−0.0051 (11)
C10	0.0212 (13)	0.0241 (14)	0.0269 (14)	−0.0030 (11)	−0.0016 (11)	−0.0068 (12)
C11	0.0190 (13)	0.0440 (18)	0.0325 (16)	−0.0016 (13)	−0.0016 (11)	−0.0148 (14)
C12	0.0187 (14)	0.0483 (18)	0.0345 (15)	0.0004 (14)	0.0012 (12)	−0.0138 (14)
C13	0.0288 (14)	0.0250 (14)	0.0290 (14)	−0.0062 (13)	0.0068 (12)	−0.0070 (12)
C14	0.0293 (14)	0.0145 (12)	0.0242 (13)	0.0027 (11)	0.0002 (11)	0.0009 (10)
C15	0.0265 (14)	0.0320 (15)	0.0286 (15)	0.0014 (13)	0.0044 (12)	−0.0051 (12)
C16	0.050 (2)	0.0347 (17)	0.0333 (18)	0.0056 (17)	0.0005 (15)	−0.0039 (15)
C17	0.057 (2)	0.0367 (18)	0.0451 (19)	−0.0219 (17)	0.0195 (18)	−0.0115 (15)
C18	0.0254 (14)	0.0401 (18)	0.0372 (17)	−0.0031 (14)	−0.0048 (13)	0.0034 (15)
C19	0.0434 (19)	0.0287 (15)	0.0335 (16)	0.0018 (15)	0.0058 (15)	−0.0086 (13)
C20	0.0285 (14)	0.0170 (12)	0.0253 (13)	0.0036 (11)	−0.0081 (11)	−0.0002 (11)

Geometric parameters (Å, º)

O1—C3	1.254 (4)	C9—C20	1.534 (4)
O2—C18	1.323 (4)	C9—C11	1.551 (4)
O2—H1O2	0.8200	C9—C10	1.560 (4)
O3—C15	1.216 (4)	C10—H10A	0.9800
O4—C16	1.398 (4)	C11—C12	1.545 (4)
O4—H1O4	0.8200	C11—H11A	0.9700
C1—C2	1.529 (5)	C11—H11B	0.9700
C1—C10	1.547 (4)	C12—C13	1.535 (4)
C1—H1A	0.9700	C12—H12A	0.9700
C1—H1B	0.9700	C12—H12B	0.9700
C2—C3	1.484 (5)	C13—C17	1.527 (4)
C2—H2A	0.9700	C13—C15	1.531 (4)
C2—H2B	0.9700	C13—C14	1.556 (4)
C3—C4	1.446 (4)	C14—H14A	0.9700
C4—C18	1.350 (4)	C14—H14B	0.9700
C4—C5	1.515 (4)	C15—C16	1.508 (4)
C5—C6	1.531 (4)	C16—H16A	0.9700
C5—C19	1.547 (4)	C16—H16B	0.9700
C5—C10	1.563 (4)	C17—H17A	0.9600
C6—C7	1.525 (4)	C17—H17B	0.9600
C6—H6A	0.9700	C17—H17C	0.9600
C6—H6B	0.9700	C18—H18A	0.9300
C7—C8	1.525 (4)	C19—H19A	0.9600
C7—H7A	0.9700	C19—H19B	0.9600
C7—H7B	0.9700	C19—H19C	0.9600

C8—C14	1.531 (4)	C20—H20A	0.9600
C8—C9	1.540 (4)	C20—H20B	0.9600
C8—H8A	0.9800	C20—H20C	0.9600
C18—O2—H1O2	109.5	C5—C10—H10A	105.5
C16—O4—H1O4	109.5	C12—C11—C9	113.5 (2)
C2—C1—C10	116.4 (3)	C12—C11—H11A	108.9
C2—C1—H1A	108.2	C9—C11—H11A	108.9
C10—C1—H1A	108.2	C12—C11—H11B	108.9
C2—C1—H1B	108.2	C9—C11—H11B	108.9
C10—C1—H1B	108.2	H11A—C11—H11B	107.7
H1A—C1—H1B	107.3	C13—C12—C11	113.7 (3)
C3—C2—C1	117.4 (3)	C13—C12—H12A	108.8
C3—C2—H2A	107.9	C11—C12—H12A	108.8
C1—C2—H2A	107.9	C13—C12—H12B	108.8
C3—C2—H2B	107.9	C11—C12—H12B	108.8
C1—C2—H2B	107.9	H12A—C12—H12B	107.7
H2A—C2—H2B	107.2	C17—C13—C15	111.0 (2)
O1—C3—C4	121.1 (3)	C17—C13—C12	110.1 (3)
O1—C3—C2	119.2 (3)	C15—C13—C12	109.7 (2)
C4—C3—C2	119.7 (3)	C17—C13—C14	111.2 (3)
C18—C4—C3	117.0 (3)	C15—C13—C14	104.7 (2)
C18—C4—C5	123.4 (3)	C12—C13—C14	110.2 (2)
C3—C4—C5	119.6 (3)	C8—C14—C13	112.6 (2)
C4—C5—C6	112.6 (2)	C8—C14—H14A	109.1
C4—C5—C19	108.5 (2)	C13—C14—H14A	109.1
C6—C5—C19	107.4 (2)	C8—C14—H14B	109.1
C4—C5—C10	109.8 (2)	C13—C14—H14B	109.1
C6—C5—C10	109.0 (2)	H14A—C14—H14B	107.8
C19—C5—C10	109.4 (2)	O3—C15—C16	118.9 (3)
C7—C6—C5	113.8 (2)	O3—C15—C13	122.2 (3)
C7—C6—H6A	108.8	C16—C15—C13	118.8 (3)
C5—C6—H6A	108.8	O4—C16—C15	113.8 (3)
C7—C6—H6B	108.8	O4—C16—H16A	108.8
C5—C6—H6B	108.8	C15—C16—H16A	108.8
H6A—C6—H6B	107.7	O4—C16—H16B	108.8
C6—C7—C8	109.5 (3)	C15—C16—H16B	108.8
C6—C7—H7A	109.8	H16A—C16—H16B	107.7
C8—C7—H7A	109.8	C13—C17—H17A	109.5
C6—C7—H7B	109.8	C13—C17—H17B	109.5
C8—C7—H7B	109.8	H17A—C17—H17B	109.5
H7A—C7—H7B	108.2	C13—C17—H17C	109.5
C7—C8—C14	111.9 (2)	H17A—C17—H17C	109.5
C7—C8—C9	112.3 (2)	H17B—C17—H17C	109.5
C14—C8—C9	112.6 (2)	O2—C18—C4	123.4 (3)
C7—C8—H8A	106.5	O2—C18—H18A	118.3
C14—C8—H8A	106.5	C4—C18—H18A	118.3
C9—C8—H8A	106.5	C5—C19—H19A	109.5

C20—C9—C8	111.5 (2)	C5—C19—H19B	109.5
C20—C9—C11	107.9 (2)	H19A—C19—H19B	109.5
C8—C9—C11	106.8 (2)	C5—C19—H19C	109.5
C20—C9—C10	112.4 (2)	H19A—C19—H19C	109.5
C8—C9—C10	108.6 (2)	H19B—C19—H19C	109.5
C11—C9—C10	109.5 (2)	C9—C20—H20A	109.5
C1—C10—C9	114.5 (2)	C9—C20—H20B	109.5
C1—C10—C5	110.2 (2)	H20A—C20—H20B	109.5
C9—C10—C5	114.9 (2)	C9—C20—H20C	109.5
C1—C10—H10A	105.5	H20A—C20—H20C	109.5
C9—C10—H10A	105.5	H20B—C20—H20C	109.5

C10—C1—C2—C3	−0.2 (4)	C8—C9—C10—C5	−52.4 (3)
C1—C2—C3—O1	−157.0 (3)	C11—C9—C10—C5	−168.7 (3)
C1—C2—C3—C4	23.7 (4)	C4—C5—C10—C1	57.8 (3)
O1—C3—C4—C18	−4.4 (4)	C6—C5—C10—C1	−178.4 (2)
C2—C3—C4—C18	175.0 (3)	C19—C5—C10—C1	−61.2 (3)
O1—C3—C4—C5	177.0 (2)	C4—C5—C10—C9	−73.3 (3)
C2—C3—C4—C5	−3.6 (4)	C6—C5—C10—C9	50.6 (3)
C18—C4—C5—C6	22.3 (4)	C19—C5—C10—C9	167.7 (2)
C3—C4—C5—C6	−159.2 (2)	C20—C9—C11—C12	−64.2 (3)
C18—C4—C5—C19	−96.4 (3)	C8—C9—C11—C12	55.8 (3)
C3—C4—C5—C19	82.1 (3)	C10—C9—C11—C12	173.2 (3)
C18—C4—C5—C10	144.0 (3)	C9—C11—C12—C13	−53.6 (4)
C3—C4—C5—C10	−37.5 (3)	C11—C12—C13—C17	−74.3 (3)
C4—C5—C6—C7	69.2 (3)	C11—C12—C13—C15	163.3 (3)
C19—C5—C6—C7	−171.3 (3)	C11—C12—C13—C14	48.6 (3)
C10—C5—C6—C7	−52.9 (3)	C7—C8—C14—C13	−174.0 (2)
C5—C6—C7—C8	58.1 (3)	C9—C8—C14—C13	58.3 (3)
C6—C7—C8—C14	172.7 (2)	C17—C13—C14—C8	71.3 (3)
C6—C7—C8—C9	−59.5 (3)	C15—C13—C14—C8	−168.8 (2)
C7—C8—C9—C20	−68.1 (3)	C12—C13—C14—C8	−51.0 (3)
C14—C8—C9—C20	59.3 (3)	C17—C13—C15—O3	−151.8 (4)
C7—C8—C9—C11	174.2 (2)	C12—C13—C15—O3	−30.0 (4)
C14—C8—C9—C11	−58.4 (3)	C14—C13—C15—O3	88.2 (4)
C7—C8—C9—C10	56.2 (3)	C17—C13—C15—C16	31.0 (4)
C14—C8—C9—C10	−176.4 (2)	C12—C13—C15—C16	152.8 (3)
C2—C1—C10—C9	91.3 (3)	C14—C13—C15—C16	−89.0 (3)
C2—C1—C10—C5	−40.0 (3)	O3—C15—C16—O4	−11.1 (5)
C20—C9—C10—C1	−57.5 (3)	C13—C15—C16—O4	166.3 (3)
C8—C9—C10—C1	178.7 (2)	C3—C4—C18—O2	4.4 (5)
C11—C9—C10—C1	62.4 (3)	C5—C4—C18—O2	−177.1 (3)
C20—C9—C10—C5	71.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1*O2*···O1	0.82	1.69	2.424 (4)	148

O4—H1*O*4···O1^{i}	0.82	2.07	2.841 (3)	156
C1—H1*B*···O2^{ii}	0.97	2.48	3.368 (5)	152
C12—H12*A*···O3	0.97	2.41	2.799 (4)	103
C17—H17*A*···O4^{iii}	0.96	2.53	3.460 (5)	164

Symmetry codes: (i) $-x+3/2, -y+1, z+1/2$; (ii) $x-1, y, z$; (iii) $x-1/2, -y+1/2, -z+1$.