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# (4*R*,4a*S*,4b*S*,7*R*,10a*R*)-4-Hydroxy-4a,7-dimethyl-2-(propan-2-yl)-1,4,4a,4b,5,6,-7,8,10,10a-decahydrophenanthren-1-one

**Ignez Caracelli,[a]* Julio Zukerman-Schpector,[b] André T. Lousada Machado,[b] Timothy J. Brocksom,[c] M. Lúcia Ferreira[c] and Edward R. T. Tiekink[d]**

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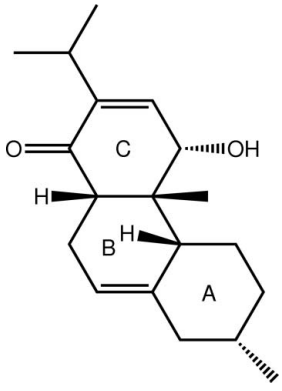
Key indicators: single-crystal X-ray study; $T$ = 290 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.037; $wR$ factor = 0.113; data-to-parameter ratio = 16.6.

In the title compound, $C_{19}H_{28}O_2$, the *A* ring adopts a chair conformation, and each of the *B* and *C* rings adopts a distorted half-chair conformation with the methine C atom in the $CH_2C(H)C(=O)$ residue, common to both rings, lying 0.6397 (19) and 0.6328 (18) Å out of the approximate plane defined by the remaining five C atoms (r.m.s. deviations = 0.0791 and 0.0901 Å for rings *B* and *C*, respectively). Helical supramolecular chains along the *a* axis mediated by hydroxy–carbonyl O—H···O hydrogen bonds feature in the crystal packing.

## Related literature

For background to the biological activity of some diterpene compounds, see: Guo *et al.* (2011); Slusarczyk *et al.* (2011). For the synthesis, see: Ferreira (2002). For conformational analysis, see: Cremer & Pople (1975).




## Experimental

### Crystal data

$C_{19}H_{28}O_2$
$M_r$ = 288.41
Orthorhombic, $P2_12_12_1$
$a$ = 7.3029 (9) Å
$b$ = 13.211 (2) Å
$c$ = 17.224 (3) Å
$V$ = 1661.8 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.07 mm$^{-1}$
$T$ = 290 K
0.12 × 0.08 × 0.07 mm

### Data collection

Enraf–Nonius CAD-4 MACH 3 diffractometer
3684 measured reflections
3243 independent reflections
2253 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031
3 standard reflections every 30 min
intensity decay: 1.1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.113
$S$ = 1.04
3243 reflections
195 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.13 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2*o*···O1$^{i}$ | 0.82 | 2.01 | 2.805 (2) | 162 |

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997), *DIAMOND* (Brandenburg, 2006) and *MarvinSketch* (Chemaxon, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5120).

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# supporting information

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## (4*R*,4a*S*,4b*S*,7*R*,10a*R*)-4-Hydroxy-4a,7-dimethyl-2-(propan-2-yl)-1,4,4a,4b,5,6,7,8,10,10a-decahydrophenanthren-1-one

**Ignez Caracelli, Julio Zukerman-Schpector, André T. Lousada Machado, Timothy J. Brocksom, M. Lúcia Ferreira and Edward R. T. Tiekink**

### S1. Comment

Natural diterpenes exhibit a wide range of biological activities such as neuroprotectives (Guo *et al.* 2011) and as antiplasmodials and antitrypanocidals (Slusarczyk *et al.* 2011). While aiming at the synthesis of some hydrophenanthrene diterpenes, a series of new intermediates were obtained and among them, was the title compound (Ferreira, 2002), (I), which has been characterized crystallographically.

The A ring has a chair conformation. Each of the B and C rings presents a distorted half-chair conformation with atom C7, common to both rings, lying 0.6397 (19) and 0.6328 (18) Å, for B and C, respectively, out of the approximate plane defined by the remaining five C atoms (r.m.s. deviations = 0.0791 and 0.0901, respectively). The ring puckering parameters are: $q_2$ = 0.003 (2), 0.360 (2), 0.363 (2) Å; $q_3$ = 0.570 (2), -0.315 (2), -0.332 (2) Å; QT = 0.570 (2), 0.478 (2), 0.492 (2) Å; and $\theta$ = 1.7 (2), 131.1 (2), 132.5 (2)°, for rings A, B and C, respectively (Cremer & Pople, 1975).

In the crystal packing, the molecules are linked through O–H···O hydrogen bonds to form supramolecular helical chains along the *a* axis, Fig. 2 and Table 1.

### S2. Experimental

The detailed synthesis of the title compound is described in a Ph.D. thesis (Ferreira, 2002). Crystals were grown by slow evaporation from its hexane solution held at 293 K; *M*.pt: 466.2–468.1 K. $^1$H-NMR ($CDCl_3$, 400 MHz): $\delta$ (p.p.m.): 6.44 (d, 1H, J = 3.8 Hz); 5.26 (s, 1H); 4.58 (s, 1H); 2.81–2.87 (m, 1H); 2.86–2.92 (m, 1H); 2.24 (d, 1H, J = 6.4 Hz); 2.10–2.26 (m, 2H); 1.90–2.10 (m, 2H); 1.82–2.10 (m, 1H); 1.37–1.54 (m, 4H); 1.27 (s, 3H); 1.04 (d, 3H, J = 6.8 Hz); 1.01 (d, 3H, J = 6.8 Hz); 0.73 (d, 3H, J = 6.8 Hz); $\delta$(OH) not obs. $^{13}$C ($CDCl_3$, 100 MHz) $\delta$ (p.p.m.): 197.9; 144.4; 142.7; 135.5; 117.2; 76.1; 49.6; 49.5; 45.9; 44.6; 35.7; 35.6; 33.6; 23.6; 22.3; 21.7; 21.5; 20.2; 20.0. Analysis found: C 78.99, H 9.77%. $C_{19}H_{28}O_2$ requires: C 79.12, H 9.79%.

### S3. Refinement

The H atoms were geometrically placed (C—H = 0.93–0.98 Å; O—H = 0.82 Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C) and $U_{iso}$(H) = 1.5$U_{eq}$(methyl-C,*O*). The absolute structure was based on that of a starting material used in the synthesis (Ferreira, 2002).

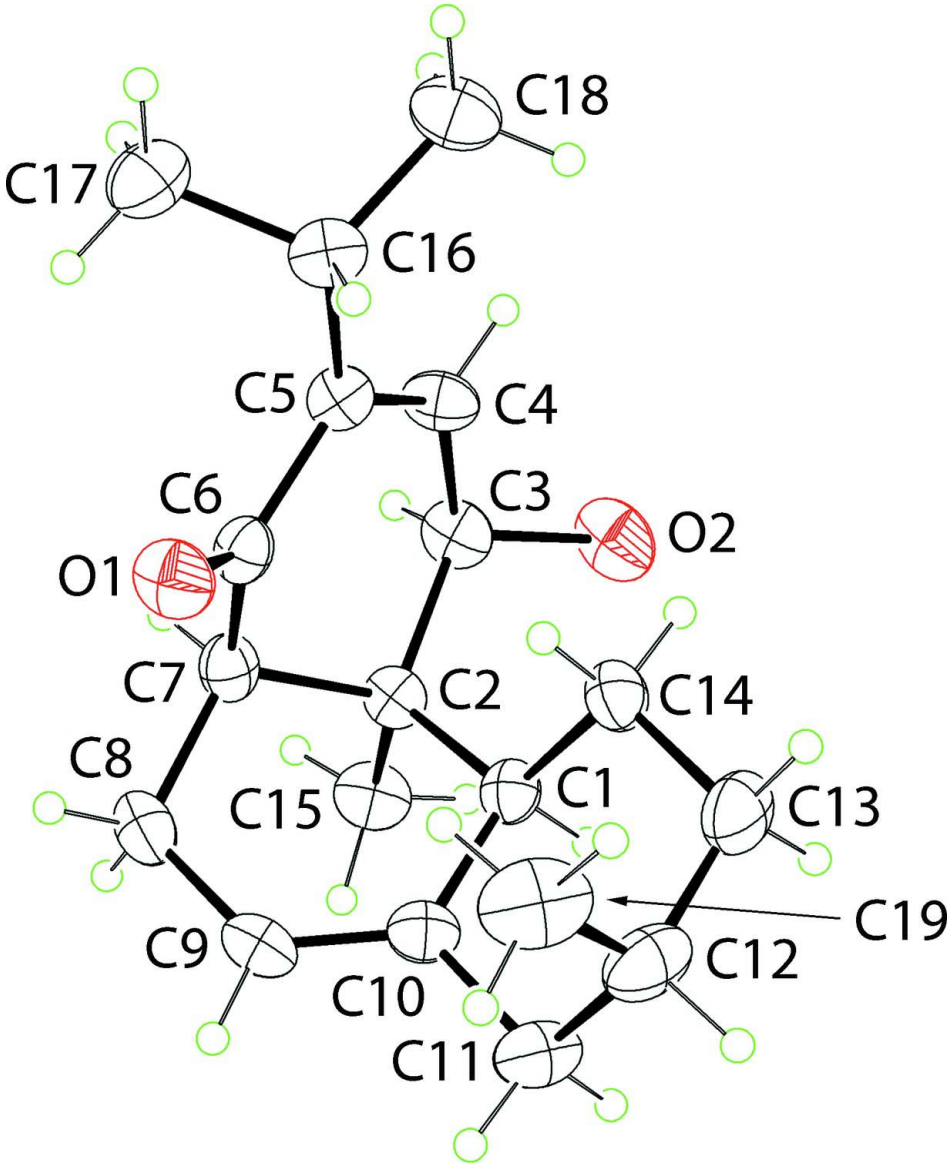


**Figure 1**

The molecular structure of compound (I) showing atom labelling scheme and displacement ellipsoids at the 30% probability level (arbitrary spheres for the H atoms).

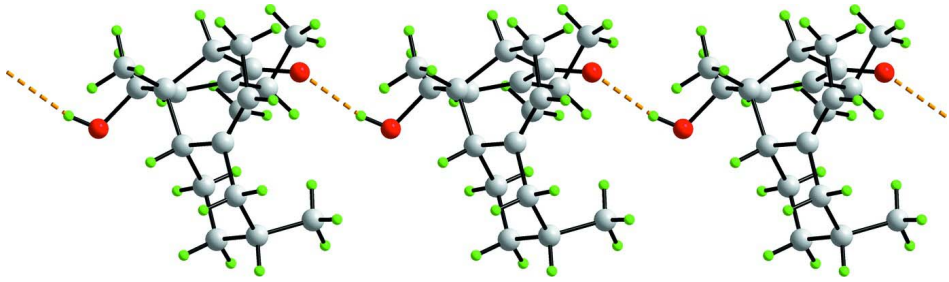

**Figure 2**

A view of the helical supramolecular chain along the *a* axis in (I). The hydroxy-O—H···O(carbonyl) hydrogen bonds are represented by orange dashed lines.

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*Crystal data*

$C_{19}H_{28}O_2$
$M_r = 288.41$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.3029\ (9)$ Å
$b = 13.211\ (2)$ Å
$c = 17.224\ (3)$ Å
$V = 1661.8\ (4)$ Å$^3$
$Z = 4$
$F(000) = 632$
$D_x = 1.153$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.2$–$14.3°$
$\mu = 0.07$ mm$^{-1}$
$T = 290$ K
Irregular, colourless
0.12 × 0.08 × 0.07 mm

*Data collection*

Enraf–Nonius CAD-4 MACH 3 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/-2\theta$ scans
3684 measured reflections
3243 independent reflections
2253 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.9°$
$h = 0\rightarrow8$
$k = 0\rightarrow16$
$l = -21\rightarrow21$
3 standard reflections every 30 min
intensity decay: 1.1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.113$
$S = 1.04$
3243 reflections
195 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
$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 0.0421P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{min} = -0.13$ e Å$^{-3}$

*Special details*

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 | 0.6835 (3) | 0.92045 (14) | 0.83208 (11) | 0.0386 (4) |
| H1 | 0.5721 | 0.9042 | 0.8613 | 0.046* |
| C2 | 0.6251 (2) | 0.99993 (14) | 0.77072 (11) | 0.0378 (4) |
| C3 | 0.5391 (2) | 0.95441 (16) | 0.69613 (12) | 0.0457 (5) |
| H3 | 0.4762 | 1.0096 | 0.6691 | 0.055* |
| C4 | 0.6752 (3) | 0.91091 (16) | 0.64072 (11) | 0.0448 (5) |
| H4 | 0.6311 | 0.8659 | 0.6036 | 0.054* |
| C5 | 0.8545 (2) | 0.93064 (15) | 0.63955 (10) | 0.0387 (4) |
| C6 | 0.9268 (2) | 0.99737 (15) | 0.70073 (11) | 0.0382 (4) |
| C7 | 0.7918 (3) | 1.06323 (13) | 0.74402 (11) | 0.0417 (5) |
| H7 | 0.7468 | 1.1144 | 0.7075 | 0.050* |
| C8 | 0.8794 (3) | 1.11916 (16) | 0.81222 (12) | 0.0498 (5) |
| H8A | 0.9985 | 1.1447 | 0.7967 | 0.060* |
| H8B | 0.8036 | 1.1766 | 0.8263 | 0.060* |
| C9 | 0.9011 (3) | 1.05182 (15) | 0.88050 (12) | 0.0471 (5) |
| H9 | 0.9814 | 1.0725 | 0.9193 | 0.056* |
| C10 | 0.8151 (3) | 0.96451 (16) | 0.89061 (11) | 0.0415 (4) |
| C11 | 0.8553 (3) | 0.89695 (17) | 0.95906 (12) | 0.0571 (6) |
| H11A | 0.7435 | 0.8853 | 0.9881 | 0.068* |
| H11B | 0.9424 | 0.9300 | 0.9932 | 0.068* |
| C12 | 0.9340 (4) | 0.79528 (18) | 0.93174 (14) | 0.0613 (7) |
| H12 | 0.9403 | 0.7499 | 0.9767 | 0.074* |
| C13 | 0.8019 (4) | 0.74886 (17) | 0.87258 (13) | 0.0626 (7) |
| H13A | 0.8539 | 0.6862 | 0.8532 | 0.075* |
| H13B | 0.6878 | 0.7324 | 0.8985 | 0.075* |
| C14 | 0.7615 (3) | 0.81779 (14) | 0.80467 (12) | 0.0462 (5) |
| H14A | 0.6739 | 0.7852 | 0.7705 | 0.055* |
| H14B | 0.8731 | 0.8292 | 0.7755 | 0.055* |
| C15 | 0.4816 (3) | 1.06970 (16) | 0.80839 (13) | 0.0520 (5) |
| H15A | 0.4561 | 1.1254 | 0.7743 | 0.078* |
| H15B | 0.5282 | 1.0950 | 0.8568 | 0.078* |
| H15C | 0.3711 | 1.0322 | 0.8176 | 0.078* |
| C16 | 0.9912 (3) | 0.88187 (16) | 0.58484 (11) | 0.0439 (5) |
| H16 | 1.0830 | 0.8479 | 0.6172 | 0.053* |
| C17 | 1.0923 (3) | 0.95967 (18) | 0.53606 (13) | 0.0618 (6) |

| | | | | |
|---|---|---|---|---|
| H17A | 1.1538 | 1.0067 | 0.5696 | 0.093* |
| H17B | 1.0064 | 0.9954 | 0.5040 | 0.093* |
| H17C | 1.1806 | 0.9260 | 0.5038 | 0.093* |
| C18 | 0.9070 (3) | 0.80139 (17) | 0.53282 (14) | 0.0597 (6) |
| H18A | 0.8167 | 0.8319 | 0.4998 | 0.090* |
| H18B | 0.8501 | 0.7503 | 0.5643 | 0.090* |
| H18C | 1.0009 | 0.7711 | 0.5015 | 0.090* |
| C19 | 1.1271 (4) | 0.8076 (2) | 0.89947 (16) | 0.0762 (8) |
| H19A | 1.1246 | 0.8534 | 0.8562 | 0.114* |
| H19B | 1.1724 | 0.7430 | 0.8827 | 0.114* |
| H19C | 1.2058 | 0.8343 | 0.9392 | 0.114* |
| O1 | 1.09041 (18) | 1.00004 (12) | 0.71585 (9) | 0.0548 (4) |
| O2 | 0.4059 (2) | 0.87899 (12) | 0.71243 (11) | 0.0637 (5) |
| H2o | 0.3032 | 0.9036 | 0.7089 | 0.096* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0338 (9) | 0.0390 (10) | 0.0431 (10) | 0.0005 (8) | 0.0042 (8) | 0.0009 (8) |
| C2 | 0.0291 (9) | 0.0410 (10) | 0.0433 (10) | 0.0040 (8) | −0.0015 (8) | −0.0020 (9) |
| C3 | 0.0289 (9) | 0.0551 (12) | 0.0531 (11) | 0.0041 (8) | −0.0081 (8) | −0.0028 (10) |
| C4 | 0.0362 (10) | 0.0579 (13) | 0.0403 (10) | 0.0065 (9) | −0.0090 (8) | −0.0087 (10) |
| C5 | 0.0311 (10) | 0.0456 (10) | 0.0395 (10) | 0.0037 (8) | −0.0032 (7) | 0.0031 (9) |
| C6 | 0.0313 (9) | 0.0415 (10) | 0.0419 (10) | −0.0020 (8) | −0.0019 (8) | 0.0072 (9) |
| C7 | 0.0419 (11) | 0.0357 (10) | 0.0476 (11) | 0.0007 (9) | −0.0046 (8) | 0.0046 (9) |
| C8 | 0.0495 (12) | 0.0389 (10) | 0.0611 (13) | −0.0058 (10) | −0.0002 (10) | −0.0079 (10) |
| C9 | 0.0450 (11) | 0.0497 (12) | 0.0465 (11) | 0.0010 (10) | −0.0085 (9) | −0.0142 (9) |
| C10 | 0.0393 (10) | 0.0458 (11) | 0.0393 (10) | 0.0087 (9) | 0.0003 (8) | −0.0045 (9) |
| C11 | 0.0652 (15) | 0.0656 (14) | 0.0404 (11) | 0.0150 (12) | 0.0001 (10) | 0.0004 (10) |
| C12 | 0.0764 (16) | 0.0607 (14) | 0.0467 (12) | 0.0222 (13) | 0.0046 (11) | 0.0148 (11) |
| C13 | 0.0857 (18) | 0.0395 (12) | 0.0626 (14) | 0.0097 (12) | 0.0142 (13) | 0.0084 (11) |
| C14 | 0.0527 (11) | 0.0352 (10) | 0.0507 (11) | −0.0007 (9) | −0.0008 (10) | −0.0012 (9) |
| C15 | 0.0426 (11) | 0.0572 (12) | 0.0563 (12) | 0.0147 (10) | −0.0011 (10) | −0.0066 (11) |
| C16 | 0.0340 (10) | 0.0536 (12) | 0.0440 (11) | 0.0109 (10) | −0.0015 (9) | −0.0001 (9) |
| C17 | 0.0585 (14) | 0.0705 (15) | 0.0565 (13) | 0.0026 (13) | 0.0138 (12) | 0.0031 (12) |
| C18 | 0.0542 (13) | 0.0635 (14) | 0.0615 (14) | 0.0150 (12) | −0.0001 (12) | −0.0155 (11) |
| C19 | 0.0692 (17) | 0.0917 (19) | 0.0677 (15) | 0.0340 (16) | −0.0017 (13) | 0.0006 (15) |
| O1 | 0.0310 (7) | 0.0714 (10) | 0.0622 (10) | −0.0043 (7) | −0.0035 (7) | −0.0086 (8) |
| O2 | 0.0282 (7) | 0.0769 (11) | 0.0861 (12) | −0.0086 (8) | −0.0018 (8) | −0.0182 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C10 | 1.510 (3) | C11—H11B | 0.9700 |
| C1—C14 | 1.545 (3) | C12—C19 | 1.524 (4) |
| C1—C2 | 1.550 (3) | C12—C13 | 1.532 (4) |
| C1—H1 | 0.9800 | C12—H12 | 0.9800 |
| C2—C15 | 1.539 (3) | C13—C14 | 1.511 (3) |
| C2—C7 | 1.547 (3) | C13—H13A | 0.9700 |

| | | | |
|---|---|---|---|
| C2—C3 | 1.551 (3) | C13—H13B | 0.9700 |
| C3—O2 | 1.421 (3) | C14—H14A | 0.9700 |
| C3—C4 | 1.493 (3) | C14—H14B | 0.9700 |
| C3—H3 | 0.9800 | C15—H15A | 0.9600 |
| C4—C5 | 1.335 (3) | C15—H15B | 0.9600 |
| C4—H4 | 0.9300 | C15—H15C | 0.9600 |
| C5—C6 | 1.472 (3) | C16—C17 | 1.519 (3) |
| C5—C16 | 1.517 (3) | C16—C18 | 1.520 (3) |
| C6—O1 | 1.223 (2) | C16—H16 | 0.9800 |
| C6—C7 | 1.512 (3) | C17—H17A | 0.9600 |
| C7—C8 | 1.528 (3) | C17—H17B | 0.9600 |
| C7—H7 | 0.9800 | C17—H17C | 0.9600 |
| C8—C9 | 1.483 (3) | C18—H18A | 0.9600 |
| C8—H8A | 0.9700 | C18—H18B | 0.9600 |
| C8—H8B | 0.9700 | C18—H18C | 0.9600 |
| C9—C10 | 1.325 (3) | C19—H19A | 0.9600 |
| C9—H9 | 0.9300 | C19—H19B | 0.9600 |
| C10—C11 | 1.507 (3) | C19—H19C | 0.9600 |
| C11—C12 | 1.535 (3) | O2—H2o | 0.8200 |
| C11—H11A | 0.9700 | | |

| | | | |
|---|---|---|---|
| C10—C1—C14 | 107.93 (16) | H11A—C11—H11B | 108.1 |
| C10—C1—C2 | 111.68 (15) | C19—C12—C13 | 112.5 (2) |
| C14—C1—C2 | 119.21 (15) | C19—C12—C11 | 111.4 (2) |
| C10—C1—H1 | 105.7 | C13—C12—C11 | 108.6 (2) |
| C14—C1—H1 | 105.7 | C19—C12—H12 | 108.1 |
| C2—C1—H1 | 105.7 | C13—C12—H12 | 108.1 |
| C15—C2—C7 | 109.71 (16) | C11—C12—H12 | 108.1 |
| C15—C2—C1 | 107.80 (15) | C14—C13—C12 | 113.36 (19) |
| C7—C2—C1 | 110.64 (15) | C14—C13—H13A | 108.9 |
| C15—C2—C3 | 107.81 (15) | C12—C13—H13A | 108.9 |
| C7—C2—C3 | 106.38 (15) | C14—C13—H13B | 108.9 |
| C1—C2—C3 | 114.42 (16) | C12—C13—H13B | 108.9 |
| O2—C3—C4 | 108.20 (17) | H13A—C13—H13B | 107.7 |
| O2—C3—C2 | 112.67 (17) | C13—C14—C1 | 111.38 (16) |
| C4—C3—C2 | 114.15 (15) | C13—C14—H14A | 109.4 |
| O2—C3—H3 | 107.1 | C1—C14—H14A | 109.4 |
| C4—C3—H3 | 107.1 | C13—C14—H14B | 109.4 |
| C2—C3—H3 | 107.1 | C1—C14—H14B | 109.4 |
| C5—C4—C3 | 125.97 (18) | H14A—C14—H14B | 108.0 |
| C5—C4—H4 | 117.0 | C2—C15—H15A | 109.5 |
| C3—C4—H4 | 117.0 | C2—C15—H15B | 109.5 |
| C4—C5—C6 | 117.27 (17) | H15A—C15—H15B | 109.5 |
| C4—C5—C16 | 124.90 (18) | C2—C15—H15C | 109.5 |
| C6—C5—C16 | 117.57 (16) | H15A—C15—H15C | 109.5 |
| O1—C6—C5 | 121.36 (18) | H15B—C15—H15C | 109.5 |
| O1—C6—C7 | 121.03 (18) | C5—C16—C17 | 112.10 (17) |
| C5—C6—C7 | 117.61 (15) | C5—C16—C18 | 113.38 (17) |

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| C6—C7—C8 | 112.58 (17) | C17—C16—C18 | 110.12 (18) |
| C6—C7—C2 | 110.42 (15) | C5—C16—H16 | 107.0 |
| C8—C7—C2 | 111.26 (16) | C17—C16—H16 | 107.0 |
| C6—C7—H7 | 107.4 | C18—C16—H16 | 107.0 |
| C8—C7—H7 | 107.4 | C16—C17—H17A | 109.5 |
| C2—C7—H7 | 107.4 | C16—C17—H17B | 109.5 |
| C9—C8—C7 | 111.36 (16) | H17A—C17—H17B | 109.5 |
| C9—C8—H8A | 109.4 | C16—C17—H17C | 109.5 |
| C7—C8—H8A | 109.4 | H17A—C17—H17C | 109.5 |
| C9—C8—H8B | 109.4 | H17B—C17—H17C | 109.5 |
| C7—C8—H8B | 109.4 | C16—C18—H18A | 109.5 |
| H8A—C8—H8B | 108.0 | C16—C18—H18B | 109.5 |
| C10—C9—C8 | 125.16 (18) | H18A—C18—H18B | 109.5 |
| C10—C9—H9 | 117.4 | C16—C18—H18C | 109.5 |
| C8—C9—H9 | 117.4 | H18A—C18—H18C | 109.5 |
| C9—C10—C11 | 121.7 (2) | H18B—C18—H18C | 109.5 |
| C9—C10—C1 | 123.35 (18) | C12—C19—H19A | 109.5 |
| C11—C10—C1 | 114.70 (18) | C12—C19—H19B | 109.5 |
| C10—C11—C12 | 110.57 (17) | H19A—C19—H19B | 109.5 |
| C10—C11—H11A | 109.5 | C12—C19—H19C | 109.5 |
| C12—C11—H11A | 109.5 | H19A—C19—H19C | 109.5 |
| C10—C11—H11B | 109.5 | H19B—C19—H19C | 109.5 |
| C12—C11—H11B | 109.5 | C3—O2—H2o | 109.5 |

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| C10—C1—C2—C15 | −77.5 (2) | C3—C2—C7—C6 | −58.80 (19) |
| C14—C1—C2—C15 | 155.54 (17) | C15—C2—C7—C8 | 59.1 (2) |
| C10—C1—C2—C7 | 42.5 (2) | C1—C2—C7—C8 | −59.7 (2) |
| C14—C1—C2—C7 | −84.5 (2) | C3—C2—C7—C8 | 175.44 (16) |
| C10—C1—C2—C3 | 162.63 (15) | C6—C7—C8—C9 | −79.0 (2) |
| C14—C1—C2—C3 | 35.6 (2) | C2—C7—C8—C9 | 45.5 (2) |
| C15—C2—C3—O2 | −73.6 (2) | C7—C8—C9—C10 | −17.0 (3) |
| C7—C2—C3—O2 | 168.73 (15) | C8—C9—C10—C11 | 175.48 (19) |
| C1—C2—C3—O2 | 46.3 (2) | C8—C9—C10—C1 | 1.1 (3) |
| C15—C2—C3—C4 | 162.42 (17) | C14—C1—C10—C9 | 118.6 (2) |
| C7—C2—C3—C4 | 44.8 (2) | C2—C1—C10—C9 | −14.3 (3) |
| C1—C2—C3—C4 | −77.7 (2) | C14—C1—C10—C11 | −56.2 (2) |
| O2—C3—C4—C5 | −144.8 (2) | C2—C1—C10—C11 | 170.94 (16) |
| C2—C3—C4—C5 | −18.5 (3) | C9—C10—C11—C12 | −116.8 (2) |
| C3—C4—C5—C6 | 3.3 (3) | C1—C10—C11—C12 | 58.0 (3) |
| C3—C4—C5—C16 | 177.17 (19) | C10—C11—C12—C19 | 70.1 (3) |
| C4—C5—C6—O1 | 162.0 (2) | C10—C11—C12—C13 | −54.3 (3) |
| C16—C5—C6—O1 | −12.4 (3) | C19—C12—C13—C14 | −68.0 (3) |
| C4—C5—C6—C7 | −18.4 (3) | C11—C12—C13—C14 | 55.8 (3) |
| C16—C5—C6—C7 | 167.19 (17) | C12—C13—C14—C1 | −56.9 (3) |
| O1—C6—C7—C8 | −7.3 (3) | C10—C1—C14—C13 | 54.0 (2) |
| C5—C6—C7—C8 | 173.06 (16) | C2—C1—C14—C13 | −177.28 (19) |
| O1—C6—C7—C2 | −132.3 (2) | C4—C5—C16—C17 | 121.9 (2) |
| C5—C6—C7—C2 | 48.0 (2) | C6—C5—C16—C17 | −64.2 (2) |

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| C15—C2—C7—C6 | −175.15 (16) | C4—C5—C16—C18 | −3.5 (3) |
| C1—C2—C7—C6 | 66.04 (19) | C6—C5—C16—C18 | 170.39 (18) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2*o*···O1$^{i}$ | 0.82 | 2.01 | 2.805 (2) | 162 |

Symmetry code: (i) *x*−1, *y*, *z*.