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# 4-(8-Ethoxy-2,3-dihydro-1*H*-cyclopenta-[*c*]quinolin-4-yl)butane-1-peroxol

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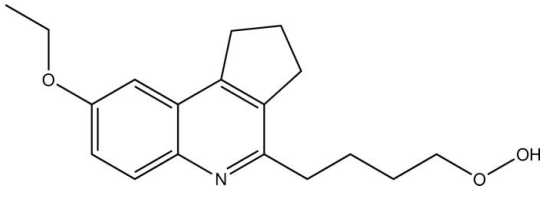
Key indicators: single-crystal X-ray study; $T$ = 90 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.036; $wR$ factor = 0.110; data-to-parameter ratio = 13.9.

In the title molecule, $C_{18}H_{23}NO_3$, the hydroperoxybutyl substituent is nearly fully extended, with the four torsion angles in the range 170.23 (10)–178.71 (9)°. The O—O distance in the hydroperoxide group is 1.4690 (13) Å. This group acts as an intermolecular hydrogen-bond donor to a quinoline N atom. This results in dimeric units about the respective inversion centers, with graph-set notation $R_2^2(18)$.

## Related literature

For a description of the Cambridge Structural Database, see: Allen (2002). For graph-set motifs, see: Etter (1990). For the biological activity of dihydroquinolines, see: Babiak *et al.* (1999); Cracknell *et al.* (1998); Dillard *et al.* (1973); Fotie *et al.* (2010); Lockhart *et al.* (2001); Shah *et al.* (2005); Takahashi *et al.* (2006); Thorisson *et al.* (1992). For related structures, see: Grignon-Dubois *et al.* (1993); Noland *et al.* (1996).




## Experimental

### *Crystal data*

$C_{18}H_{23}NO_3$
$M_r$ = 301.37
Triclinic, $P\overline{1}$
$a$ = 8.0113 (2) Å
$b$ = 8.5091 (2) Å
$c$ = 12.6334 (3) Å
$\alpha$ = 73.605 (1)°
$\beta$ = 74.936 (1)°
$\gamma$ = 78.136 (1)°
$V$ = 789.63 (3) Å$^3$
$Z$ = 2
Cu $K\alpha$ radiation
$\mu$ = 0.69 mm$^{-1}$
$T$ = 90 K
0.19 × 0.17 × 0.15 mm

### *Data collection*

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min}$ = 0.880, $T_{max}$ = 0.904
9369 measured reflections
2798 independent reflections
2400 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.030

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.110
$S$ = 1.08
2798 reflections
202 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.27 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···N1$^{i}$ | 0.84 | 1.93 | 2.7466 (14) | 165 |

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2311).

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# supporting information

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## 4-(8-Ethoxy-2,3-dihydro-1*H*-cyclopenta[*c*]quinolin-4-yl)butane-1-peroxol

**Jean Fotie, Chris F. Fronczek, Kyle A. Burns, Frank R. Fronczek, Cheryl Bain, D. Scott Bohle and Ferdinand P. Poudeu**

### S1. Comment

Dihydroquinolines are mainly known for their antioxidant activity (Thorisson *et al.*, 1992, Lockhart *et al.*, 2001) although they have also been reported to possess anti-inflammatory (Dillard *et al.*, 1973), fungicidal (Cracknell *et al.*, 1998), antiatherosclerotic (Babiak *et al.*, 1999), and hormone receptor modulator (Takahashi *et al.*, 2006) properties. Furthermore, 6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline, also known as ethoxyquin, is a FDA approved antioxidant commonly used as a preservative in the food processing industry (Shah *et al.*, 2005). We have recently reported some dihydroquinoline derivatives with outstanding antitrypanosomal activity (Fotie *et al.*, 2010). In our effort to optimize the trypanocidal activity of this family of compound, we have synthesized the title compound, an unusual hydroperoxybutylquinoline derivative. Here we are reporting the characterization of that compound using $^{1}$H– and $^{13}$C-NMR spectroscopy, mass spectrometry, and single-crystal diffraction.

The molecular structure of the title compound is illustrated in Fig. 1. The 10-atom quinoline ring system is essentially planar, with mean deviation 0.009 Å and maximum deviation 0.017 (1) Å for both N1 and C11. The five-membered ring has the envelope conformation, with C9 at the flap position, 0.340 (2) Å out of the quinoline plane. The hydroperoxybutyl chain is extended, with torsion angle magnitudes in the range 170.23 (10) to 178.71 (9)°, and the best plane of its four C and two O atoms is approximately perpendicular to the quinoline plane, forming a dihedral angle of 89.53 (3)°. The hydroperoxy O—O distance, 1.4690 (13) Å agrees well with literature values for this group. The mean value of the 135 such distances in the Cambridge Structural Database (version 5.31, Nov. 2009; Allen 2002), after rejecting eight outliers, is 1.462 Å.

The hydroperoxide donates an intermolecular hydrogen bond to quinoline N1, with O···N distance 2.7466 (14) Å, forming discrete dimers having graph set (Etter, 1990) $R^2_2(18)$ about inversion centers, as illustrated in Fig. 2.

### S2. Experimental

The title compound was prepared by heating to reflux for three days, a mixture of *p*-phenitidine (500 mg, 3.6 mmol) and cyclopentanone (10 ml, large excess) in the presence of catalytic amounts of iodine (93 mg) and benzoyl peroxide (8.8 mg). After appropriate work-up, and purification on a silica gel column, crystals were carefully grown at room temperature, in a mixture of hexanes-dichloromethane, over the course of a week.

Mp: 131.3 - 131.6 °C. The melting point was recorded on a MEL-TEMP ELECTROTHERMAL digital melting point apparatus, and is not corrected.

ESIMS *m/z* (%): 316 (90) $[M + CH_3]^+$, 302 (43) $[M + H]^+$, 286 (100) $[M -16]^+$, 284 (94) $[M - H_2O]^+$. These fragment ions are consistent with a molecular formula of $C_{18}H_{23}NO_3$. The ESIMS spectrum was recorded on a Finnigan LCQDUO spectrometer.

NMR data were collected on a Bruker AC 300 Spectrometer. $^{1}$H-NMR (300 MHz, $CDCl_3$) $\delta$: 1.47 (3*H*, t, J = 6.7 Hz), 1.68 (2*H*, m), 1.85 (2*H*, m), 2.23 (2*H*, m), 3.04 (4*H*, t, J = 7.9 Hz), 3.13 (2*H*, t, J = 7.3 Hz), 4.10 (2*H*, t, 6.7 Hz), 4.15 (2*H*, q, J = 6.7 Hz), 6.90 (1*H*, d, J = 2.4 Hz), 7.23 (1*H*, dd, J = 9.2 Hz and 2,4 Hz), 7.83 (1*H*, d, 9.2 Hz), 13.6 (1*H*, brs). $^{13}$C-NMR (75 MHz, $CDCl_3$) $\delta$: 14.9, 24.0, 25.0, 25.9, 31.4, 31.5, 35.0, 63.8, 103.0, 121.1, 125.9, 129.2, 136.0, 141.6, 149.7, 155.9, 156.6. 162.3.

## S3. Refinement

H atoms on C were placed in idealized positions with C—H distances 0.95 - 1.00 Å and thereafter treated as riding. The OH H atom was located from a difference map in the expected circle. Torsional parameters were refined for the methyl and hydroperoxy OH groups. $U_{iso}$ for H were assigned as 1.2 times $U_{eq}$ of the attached atoms (1.5 for methyl and OH).

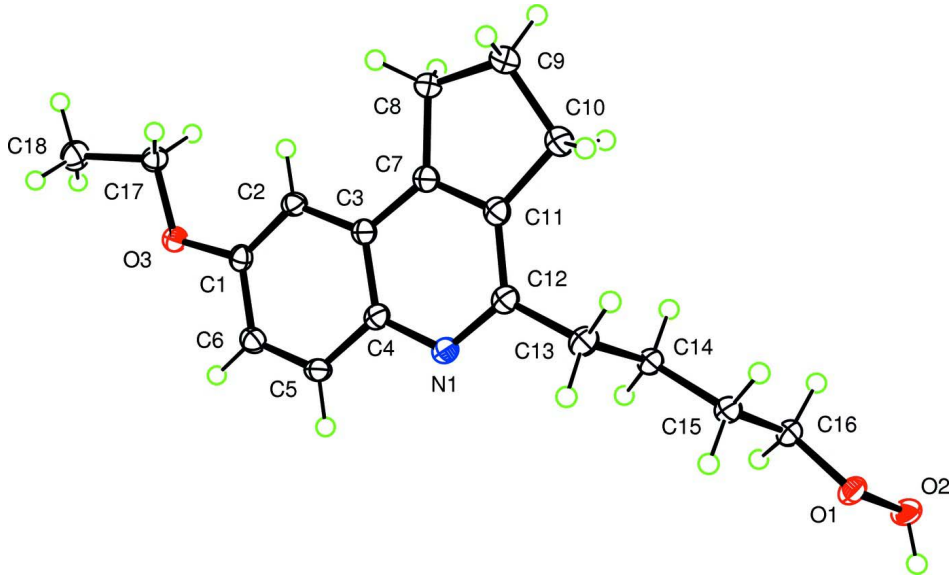


**Figure 1**

Ellipsoids at the 50% level, with H atoms having arbitrary radius.

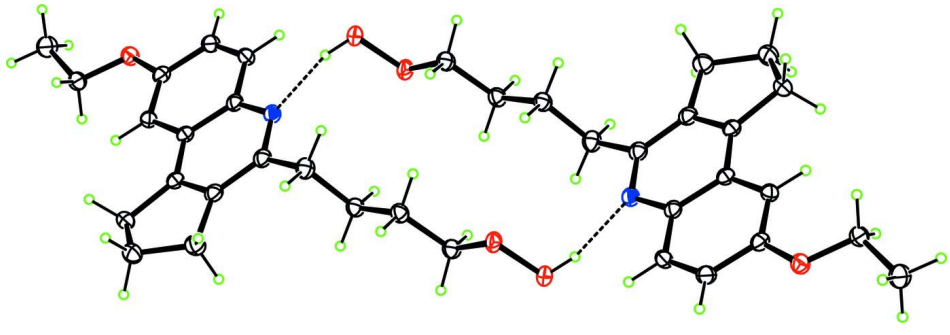

**Figure 2**

The hydrogen-bonded dimer, with graph set $R^2_2(18)$.

### 4-(8-Ethoxy-2,3-dihydro-1*H*-cyclopenta[*c*]quinolin-4-yl)butane- 1-peroxol

*Crystal data*

$C_{18}H_{23}NO_3$
$M_r$ = 301.37

Triclinic, $P\overline{1}$
Hall symbol: -P 1

$a$ = 8.0113 (2) Å
$b$ = 8.5091 (2) Å
$c$ = 12.6334 (3) Å
$\alpha$ = 73.605 (1)°
$\beta$ = 74.936 (1)°
$\gamma$ = 78.136 (1)°
$V$ = 789.63 (3) Å$^3$
$Z$ = 2
$F$(000) = 324

$D_x$ = 1.268 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å
Cell parameters from 4518 reflections
$\theta$ = 3.7–68.3°
$\mu$ = 0.69 mm$^{-1}$
$T$ = 90 K
Prism, colourless
0.19 × 0.17 × 0.15 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min}$ = 0.880, $T_{max}$ = 0.904

9369 measured reflections
2798 independent reflections
2400 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.030
$\theta_{max}$ = 68.8°, $\theta_{min}$ = 3.7°
$h$ = −9→9
$k$ = −9→10
$l$ = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.110
$S$ = 1.08
2798 reflections
202 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
$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2 + 0.1754P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.27 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0023 (7)

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.54940 (12) | 0.44045 (11) | 1.21141 (7) | 0.0202 (2) |
| O2 | 0.43514 (12) | 0.35072 (12) | 1.31138 (8) | 0.0233 (3) |
| H2 | 0.3429 | 0.4140 | 1.3292 | 0.035* |
| O3 | 0.98882 (12) | 0.17854 (11) | 0.27498 (8) | 0.0201 (2) |
| N1 | 0.84064 (14) | 0.40032 (13) | 0.66551 (9) | 0.0180 (3) |
| C1 | 0.96537 (17) | 0.22269 (16) | 0.37459 (11) | 0.0177 (3) |

| | | | | |
|---|---|---|---|---|
| C2 | 1.08402 (17) | 0.17863 (16) | 0.44285 (11) | 0.0175 (3) |
| H2A | 1.1914 | 0.1104 | 0.4233 | 0.021* |
| C3 | 1.04475 (17) | 0.23589 (15) | 0.54275 (11) | 0.0166 (3) |
| C4 | 0.88489 (17) | 0.33756 (16) | 0.57160 (11) | 0.0171 (3) |
| C5 | 0.76525 (17) | 0.37924 (16) | 0.49902 (11) | 0.0184 (3) |
| H5 | 0.6573 | 0.4473 | 0.5172 | 0.022* |
| C6 | 0.80419 (17) | 0.32234 (16) | 0.40359 (11) | 0.0198 (3) |
| H6 | 0.7225 | 0.3498 | 0.3562 | 0.024* |
| C7 | 1.15889 (17) | 0.19861 (16) | 0.61825 (11) | 0.0173 (3) |
| C8 | 1.33845 (17) | 0.09920 (17) | 0.60806 (11) | 0.0203 (3) |
| H8A | 1.4123 | 0.1362 | 0.5321 | 0.024* |
| H8B | 1.3323 | −0.0203 | 0.6226 | 0.024* |
| C9 | 1.40947 (18) | 0.13488 (18) | 0.69997 (12) | 0.0235 (3) |
| H9A | 1.4726 | 0.0323 | 0.7404 | 0.028* |
| H9B | 1.4909 | 0.2177 | 0.6656 | 0.028* |
| C10 | 1.25038 (18) | 0.20199 (18) | 0.78213 (12) | 0.0229 (3) |
| H10A | 1.2155 | 0.1143 | 0.8507 | 0.027* |
| H10B | 1.2753 | 0.2956 | 0.8047 | 0.027* |
| C11 | 1.11048 (17) | 0.25808 (16) | 0.71348 (11) | 0.0188 (3) |
| C12 | 0.95014 (17) | 0.36144 (16) | 0.73497 (11) | 0.0184 (3) |
| C13 | 0.89624 (18) | 0.43704 (17) | 0.83518 (11) | 0.0209 (3) |
| H13A | 0.8268 | 0.5471 | 0.8139 | 0.025* |
| H13B | 1.0025 | 0.4540 | 0.8543 | 0.025* |
| C14 | 0.78889 (17) | 0.33287 (16) | 0.94029 (11) | 0.0186 (3) |
| H14A | 0.6851 | 0.3105 | 0.9213 | 0.022* |
| H14B | 0.8601 | 0.2254 | 0.9657 | 0.022* |
| C15 | 0.73048 (17) | 0.42275 (16) | 1.03551 (11) | 0.0190 (3) |
| H15A | 0.8351 | 0.4403 | 1.0561 | 0.023* |
| H15B | 0.6655 | 0.5329 | 1.0079 | 0.023* |
| C16 | 0.61601 (18) | 0.32967 (16) | 1.13985 (11) | 0.0194 (3) |
| H16A | 0.5194 | 0.2962 | 1.1196 | 0.023* |
| H16B | 0.6850 | 0.2291 | 1.1779 | 0.023* |
| C17 | 1.15683 (18) | 0.09386 (16) | 0.23380 (11) | 0.0201 (3) |
| H17A | 1.1735 | −0.0203 | 0.2812 | 0.024* |
| H17B | 1.2503 | 0.1526 | 0.2357 | 0.024* |
| C18 | 1.1631 (2) | 0.09065 (18) | 0.11403 (12) | 0.0256 (3) |
| H18A | 1.0682 | 0.0347 | 0.1132 | 0.038* |
| H18B | 1.2755 | 0.0309 | 0.0835 | 0.038* |
| H18C | 1.1496 | 0.2043 | 0.0676 | 0.038* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0200 (5) | 0.0214 (5) | 0.0171 (5) | −0.0013 (4) | 0.0010 (4) | −0.0070 (4) |
| O2 | 0.0197 (5) | 0.0255 (5) | 0.0176 (5) | 0.0011 (4) | 0.0027 (4) | −0.0033 (4) |
| O3 | 0.0201 (5) | 0.0229 (5) | 0.0180 (5) | 0.0001 (4) | −0.0048 (4) | −0.0076 (4) |
| N1 | 0.0179 (6) | 0.0168 (6) | 0.0179 (6) | −0.0021 (4) | −0.0017 (4) | −0.0044 (4) |
| C1 | 0.0208 (7) | 0.0161 (7) | 0.0153 (6) | −0.0040 (5) | −0.0026 (5) | −0.0027 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.0164 (7) | 0.0150 (6) | 0.0183 (7) | −0.0013 (5) | −0.0018 (5) | −0.0023 (5) |
| C3 | 0.0171 (7) | 0.0141 (6) | 0.0166 (7) | −0.0038 (5) | −0.0023 (5) | −0.0006 (5) |
| C4 | 0.0181 (7) | 0.0141 (6) | 0.0168 (7) | −0.0033 (5) | −0.0004 (5) | −0.0022 (5) |
| C5 | 0.0151 (7) | 0.0165 (7) | 0.0210 (7) | 0.0002 (5) | −0.0027 (5) | −0.0029 (5) |
| C6 | 0.0187 (7) | 0.0193 (7) | 0.0199 (7) | −0.0030 (5) | −0.0051 (5) | −0.0017 (5) |
| C7 | 0.0173 (7) | 0.0148 (6) | 0.0175 (7) | −0.0039 (5) | −0.0023 (5) | −0.0003 (5) |
| C8 | 0.0176 (7) | 0.0211 (7) | 0.0201 (7) | 0.0016 (5) | −0.0045 (5) | −0.0045 (5) |
| C9 | 0.0183 (7) | 0.0282 (8) | 0.0235 (7) | −0.0008 (6) | −0.0065 (6) | −0.0056 (6) |
| C10 | 0.0222 (7) | 0.0259 (8) | 0.0219 (7) | −0.0011 (6) | −0.0075 (6) | −0.0071 (6) |
| C11 | 0.0194 (7) | 0.0180 (7) | 0.0175 (7) | −0.0052 (5) | −0.0029 (5) | −0.0012 (5) |
| C12 | 0.0193 (7) | 0.0164 (7) | 0.0182 (7) | −0.0043 (5) | −0.0017 (5) | −0.0032 (5) |
| C13 | 0.0215 (7) | 0.0203 (7) | 0.0212 (7) | −0.0037 (5) | −0.0020 (6) | −0.0075 (6) |
| C14 | 0.0194 (7) | 0.0188 (7) | 0.0182 (7) | −0.0005 (5) | −0.0043 (5) | −0.0067 (5) |
| C15 | 0.0181 (7) | 0.0203 (7) | 0.0192 (7) | −0.0006 (5) | −0.0041 (5) | −0.0072 (5) |
| C16 | 0.0213 (7) | 0.0195 (7) | 0.0179 (7) | 0.0005 (5) | −0.0043 (5) | −0.0075 (5) |
| C17 | 0.0214 (7) | 0.0180 (7) | 0.0197 (7) | −0.0001 (5) | −0.0029 (5) | −0.0058 (5) |
| C18 | 0.0307 (8) | 0.0246 (8) | 0.0226 (7) | −0.0005 (6) | −0.0046 (6) | −0.0106 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C16 | 1.4193 (15) | C9—H9B | 0.9900 |
| O1—O2 | 1.4690 (13) | C10—C11 | 1.5125 (18) |
| O2—H2 | 0.8400 | C10—H10A | 0.9900 |
| O3—C1 | 1.3693 (15) | C10—H10B | 0.9900 |
| O3—C17 | 1.4319 (16) | C11—C12 | 1.4094 (19) |
| N1—C12 | 1.3288 (17) | C12—C13 | 1.5048 (18) |
| N1—C4 | 1.3709 (17) | C13—C14 | 1.5310 (18) |
| C1—C2 | 1.3729 (18) | C13—H13A | 0.9900 |
| C1—C6 | 1.4142 (19) | C13—H13B | 0.9900 |
| C2—C3 | 1.4166 (18) | C14—C15 | 1.5266 (17) |
| C2—H2A | 0.9500 | C14—H14A | 0.9900 |
| C3—C4 | 1.4147 (18) | C14—H14B | 0.9900 |
| C3—C7 | 1.4176 (18) | C15—C16 | 1.5122 (18) |
| C4—C5 | 1.4208 (18) | C15—H15A | 0.9900 |
| C5—C6 | 1.3628 (19) | C15—H15B | 0.9900 |
| C5—H5 | 0.9500 | C16—H16A | 0.9900 |
| C6—H6 | 0.9500 | C16—H16B | 0.9900 |
| C7—C11 | 1.3691 (19) | C17—C18 | 1.5087 (18) |
| C7—C8 | 1.5054 (18) | C17—H17A | 0.9900 |
| C8—C9 | 1.5420 (19) | C17—H17B | 0.9900 |
| C8—H8A | 0.9900 | C18—H18A | 0.9800 |
| C8—H8B | 0.9900 | C18—H18B | 0.9800 |
| C9—C10 | 1.5413 (19) | C18—H18C | 0.9800 |
| C9—H9A | 0.9900 | | |
| | | | |
| C16—O1—O2 | 105.80 (9) | C7—C11—C12 | 120.33 (12) |
| O1—O2—H2 | 109.5 | C7—C11—C10 | 111.14 (12) |
| C1—O3—C17 | 117.05 (10) | C12—C11—C10 | 128.50 (12) |

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| C12—N1—C4 | 119.06 (11) | N1—C12—C11 | 121.27 (12) |
| O3—C1—C2 | 125.10 (12) | N1—C12—C13 | 116.62 (11) |
| O3—C1—C6 | 114.06 (11) | C11—C12—C13 | 122.09 (12) |
| C2—C1—C6 | 120.84 (12) | C12—C13—C14 | 114.07 (11) |
| C1—C2—C3 | 119.32 (12) | C12—C13—H13A | 108.7 |
| C1—C2—H2A | 120.3 | C14—C13—H13A | 108.7 |
| C3—C2—H2A | 120.3 | C12—C13—H13B | 108.7 |
| C4—C3—C2 | 120.22 (12) | C14—C13—H13B | 108.7 |
| C4—C3—C7 | 116.10 (12) | H13A—C13—H13B | 107.6 |
| C2—C3—C7 | 123.68 (12) | C15—C14—C13 | 110.73 (11) |
| N1—C4—C3 | 123.16 (12) | C15—C14—H14A | 109.5 |
| N1—C4—C5 | 118.20 (12) | C13—C14—H14A | 109.5 |
| C3—C4—C5 | 118.63 (12) | C15—C14—H14B | 109.5 |
| C6—C5—C4 | 120.53 (12) | C13—C14—H14B | 109.5 |
| C6—C5—H5 | 119.7 | H14A—C14—H14B | 108.1 |
| C4—C5—H5 | 119.7 | C16—C15—C14 | 113.20 (11) |
| C5—C6—C1 | 120.44 (12) | C16—C15—H15A | 108.9 |
| C5—C6—H6 | 119.8 | C14—C15—H15A | 108.9 |
| C1—C6—H6 | 119.8 | C16—C15—H15B | 108.9 |
| C11—C7—C3 | 120.02 (12) | C14—C15—H15B | 108.9 |
| C11—C7—C8 | 111.83 (11) | H15A—C15—H15B | 107.8 |
| C3—C7—C8 | 128.14 (12) | O1—C16—C15 | 106.06 (11) |
| C7—C8—C9 | 103.20 (11) | O1—C16—H16A | 110.5 |
| C7—C8—H8A | 111.1 | C15—C16—H16A | 110.5 |
| C9—C8—H8A | 111.1 | O1—C16—H16B | 110.5 |
| C7—C8—H8B | 111.1 | C15—C16—H16B | 110.5 |
| C9—C8—H8B | 111.1 | H16A—C16—H16B | 108.7 |
| H8A—C8—H8B | 109.1 | O3—C17—C18 | 107.10 (11) |
| C10—C9—C8 | 106.77 (11) | O3—C17—H17A | 110.3 |
| C10—C9—H9A | 110.4 | C18—C17—H17A | 110.3 |
| C8—C9—H9A | 110.4 | O3—C17—H17B | 110.3 |
| C10—C9—H9B | 110.4 | C18—C17—H17B | 110.3 |
| C8—C9—H9B | 110.4 | H17A—C17—H17B | 108.6 |
| H9A—C9—H9B | 108.6 | C17—C18—H18A | 109.5 |
| C11—C10—C9 | 103.12 (11) | C17—C18—H18B | 109.5 |
| C11—C10—H10A | 111.1 | H18A—C18—H18B | 109.5 |
| C9—C10—H10A | 111.1 | C17—C18—H18C | 109.5 |
| C11—C10—H10B | 111.1 | H18A—C18—H18C | 109.5 |
| C9—C10—H10B | 111.1 | H18B—C18—H18C | 109.5 |
| H10A—C10—H10B | 109.1 | | |

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| C17—O3—C1—C2 | −6.48 (18) | C3—C7—C8—C9 | 168.11 (13) |
| C17—O3—C1—C6 | 173.01 (11) | C7—C8—C9—C10 | 18.58 (14) |
| O3—C1—C2—C3 | 178.62 (11) | C8—C9—C10—C11 | −19.42 (14) |
| C6—C1—C2—C3 | −0.8 (2) | C3—C7—C11—C12 | −2.3 (2) |
| C1—C2—C3—C4 | −0.07 (19) | C8—C7—C11—C12 | 176.70 (12) |
| C1—C2—C3—C7 | −179.45 (12) | C3—C7—C11—C10 | 179.39 (11) |
| C12—N1—C4—C3 | −1.71 (19) | C8—C7—C11—C10 | −1.59 (16) |

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| C12—N1—C4—C5 | 179.37 (11) | C9—C10—C11—C7 | 13.27 (15) |
| C2—C3—C4—N1 | −178.41 (11) | C9—C10—C11—C12 | −164.84 (13) |
| C7—C3—C4—N1 | 1.02 (19) | C4—N1—C12—C11 | 0.34 (19) |
| C2—C3—C4—C5 | 0.51 (19) | C4—N1—C12—C13 | 178.70 (11) |
| C7—C3—C4—C5 | 179.94 (11) | C7—C11—C12—N1 | 1.7 (2) |
| N1—C4—C5—C6 | 178.92 (11) | C10—C11—C12—N1 | 179.63 (12) |
| C3—C4—C5—C6 | −0.05 (19) | C7—C11—C12—C13 | −176.59 (12) |
| C4—C5—C6—C1 | −0.8 (2) | C10—C11—C12—C13 | 1.4 (2) |
| O3—C1—C6—C5 | −178.21 (11) | N1—C12—C13—C14 | 89.79 (14) |
| C2—C1—C6—C5 | 1.3 (2) | C11—C12—C13—C14 | −91.87 (15) |
| C4—C3—C7—C11 | 1.01 (19) | C12—C13—C14—C15 | −176.62 (11) |
| C2—C3—C7—C11 | −179.59 (12) | C13—C14—C15—C16 | 177.09 (10) |
| C4—C3—C7—C8 | −177.84 (12) | O2—O1—C16—C15 | 178.71 (9) |
| C2—C3—C7—C8 | 1.6 (2) | C14—C15—C16—O1 | −170.23 (10) |
| C11—C7—C8—C9 | −10.82 (15) | C1—O3—C17—C18 | −168.40 (11) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···N1$^{i}$ | 0.84 | 1.93 | 2.7466 (14) | 165 |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+2.