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# 11-(4-Methylphenyl)-8,9-dihydro-7*H*-benzo[*f*]cyclopenta[*b*]quinolin-10(11*H*)-one

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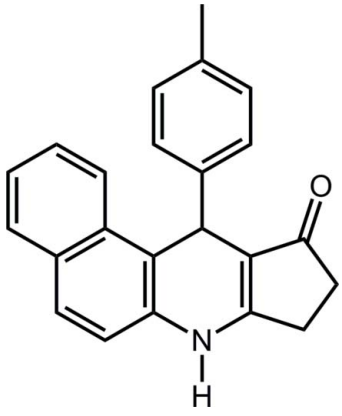
Key indicators: single-crystal X-ray study; $T$ = 113 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.044; $wR$ factor = 0.124; data-to-parameter ratio = 16.9.

In the title compound, $C_{23}H_{19}NO$, the naphthalene ring system and the cyclopent-2-enone ring exhibit planar conformations with maximum deviations of 0.034 (1) and 0.02 (1) Å, respectively. The 1,4-dihydropyridine ring adopts an envelope conformation with the C atom bearing the *p*-tolyl ring as the flap atom. Intermolecular N—H···O hydrogen bonds and C—H···$\pi$ interactions stabilize the crystal packing.

## Related literature

For the medicinal use of quinoline and fused quinoline derivatives, see: Audisio *et al.* (2012); Kurasawa *et al.* (2012); Pokhrel *et al.* (2012). For puckering parameters, see: Cremer & Pople (1975).




## Experimental

### Crystal data

$C_{23}H_{19}NO$ $M_r$ = 325.39
Monoclinic, $P2_1/c$
$a$ = 8.727 (1) Å
$b$ = 11.6820 (14) Å
$c$ = 16.240 (2) Å
$\beta$ = 98.938 (5)°
$V$ = 1635.5 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 113 K
0.24 × 0.20 × 0.18 mm

### Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClearSM Expert*; Rigaku/MSC, 2009)
$T_{min}$ = 0.981, $T_{max}$ = 0.986
15928 measured reflections
3899 independent reflections
2991 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.124
$S$ = 1.07
3899 reflections
231 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$

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

*Cg*1 is the centroid of the C17–C22 ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.902 (17) | 1.983 (17) | 2.8519 (13) | 161.2 (14) |
| C15—H15···*Cg*1$^{ii}$ | 0.95 | 3.00 | 3.8110 (15) | 145 |

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

Data collection: *CrystalClearSM Expert* (Rigaku/MSC, 2009); cell refinement: *CrystalClearSM Expert*; data reduction: *CrystalClearSM Expert*; 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*.

The author is thankful to Professor Youquan Zhu of Nankai University, China, for his kindly help with the collection of the X-ray data set, structure solution and refinement.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5239).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o2746 [doi:10.1107/S1600536812035659]

## 11-(4-Methylphenyl)-8,9-dihydro-7*H*-benzo[*f*]cyclopenta[*b*]quinolin-10(11*H*)-one

**Cao Yang**

### Comment

Many efforts have been devoted to the synthesis of quinoline and fused quinoline derivaties due to their broad spectrum of bioactivities including anti-norovirus activity (Pokhrel *et al.* 2012), antikinetoplastid activity (Audisio *et al.* 2012), antimalarial activity (Kurasawa *et al.* 2012). These work inspired us to assemble various types of fused quinoline skeletons and evaluate their bioactivities to study the relationship between structure and activity (SAR). Determination of the accurate structure is crucial to do SAR research. During our work on the efficient synthesis and biological study of benzo[*f*]cyclopenta[*b*]quinolins, the title compound (I) was isolated and its structure was determined by X-ray diffraction. Herein we report its crystal structure.

In the molecular structure (Fig. 1), the quinoline ring and cyclopent-2-enone ring adopts planar conformations. The atoms with maximal deviation from the the two ring are C9 and ing systr C5 with the distance of 0.034 (1)Å and 0.02 (1) Å, respectively. The dihydropyridine ring is in an envelope (or half boat) conformation, since C1/C2/N1/C4/C5 is coplanar with the largest deviation of 0.040 (1)Å(N1). However, the distance of C3 and the plane consisting of the rest five atoms is 0.212 (1)Å. Cremer & Pople puckering parameters analysis also confirms that the dihydropyridine ring adopts a envelope conformation (Cremer & Pople, 1975). Its puckering amplitude (Q), $\theta$ and $\varphi$ are 0.156 (1)Å, 102.7 (4)° and 0.7 (5)°, respectively. The linkage between *p*-tolyl and dihydropyridine ring can be described by the torsion angle of C2—C3—C17—C22 (45.46 (15)°) and C4—C3—C17—C22(-76.38 (13)°) respectively. The crystal packing is stabilized by intermolecular N—H···O hydrogen bond and C—H··· $\pi$ interactions (Fig. 2, Table 1).

### Experimental

In a dry 50 mL flask, cyclopentane-1,3-dione (0.10 g, 1 mmol), 4-methylbenzaldehyde (0.12 g, 1 mmol), naphthalen-2-amine (0.14 g,1 mmol), *p*-toluenesulfonic acid (0.2 g, 1 mmol) and water (10 mL) were mixed and then stirred at 373 K for 5 h. After completion of the reaction, as indicated by TLC, the solid product was collected by filtration and were purified by flash column chromatography (silica gel, mixtures of ethyl acetate / petroleum ether, 1:3, *v*/*v*) to afford the desired pure product. m. p.: 562-564 K; IR (KBr,*v*, $cm^{-1}$): 3165, 3080, 3013, 2962, 1666, 1632, 1584, 1522, 1468, 1397, 1269, 1239, 1220, 1012, 841, 806, 748, 688, 531; $^1$H NMR (400 MHz, DMSO-$d_6$) ($\delta$, ppm): 10.27 (s, 1H, NH), 7.80 (q, *J* = 8.4 Hz, 3H, ArH), 7.37-7.28 (m, 3H, ArH), 7.05 (d, *J* = 8.0 Hz, 2H, ArH), 6.94 (d, *J* = 8.0 Hz, 2H, ArH), 5.58 (s, 1H, CH), 2.71-2.63 (m, 2H, $CH_2$), 2.34-2.20 (m, 2H, $CH_2$), 2.14 (s, 3H, $CH_3$); HRMS (ESI) m/z: Calcd. for $C_{23}H_{19}NONa$ $[M+Na]^+$ 348.1364, found: 348.1345.The single-crystal suitable for X-ray diffraction was obtained through slow evaporation of the ethanol solution of the title compound.

### Refinement

The hydrogen atom bonded to nitrogen atom was positioned from a Fourier difference map refined freely. All other H atoms were placed in calculated positions, with C—H = 0.95 Å, 0.98 Å, 0.99 Å or 1.00 Å and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$(parent atom).

### Computing details

Data collection: *CrystalClearSM Expert* (Rigaku/MSC, 2009); cell refinement: *CrystalClearSM Expert* (Rigaku/MSC, 2009); data reduction: *CrystalClearSM Expert* (Rigaku/MSC, 2009); 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).

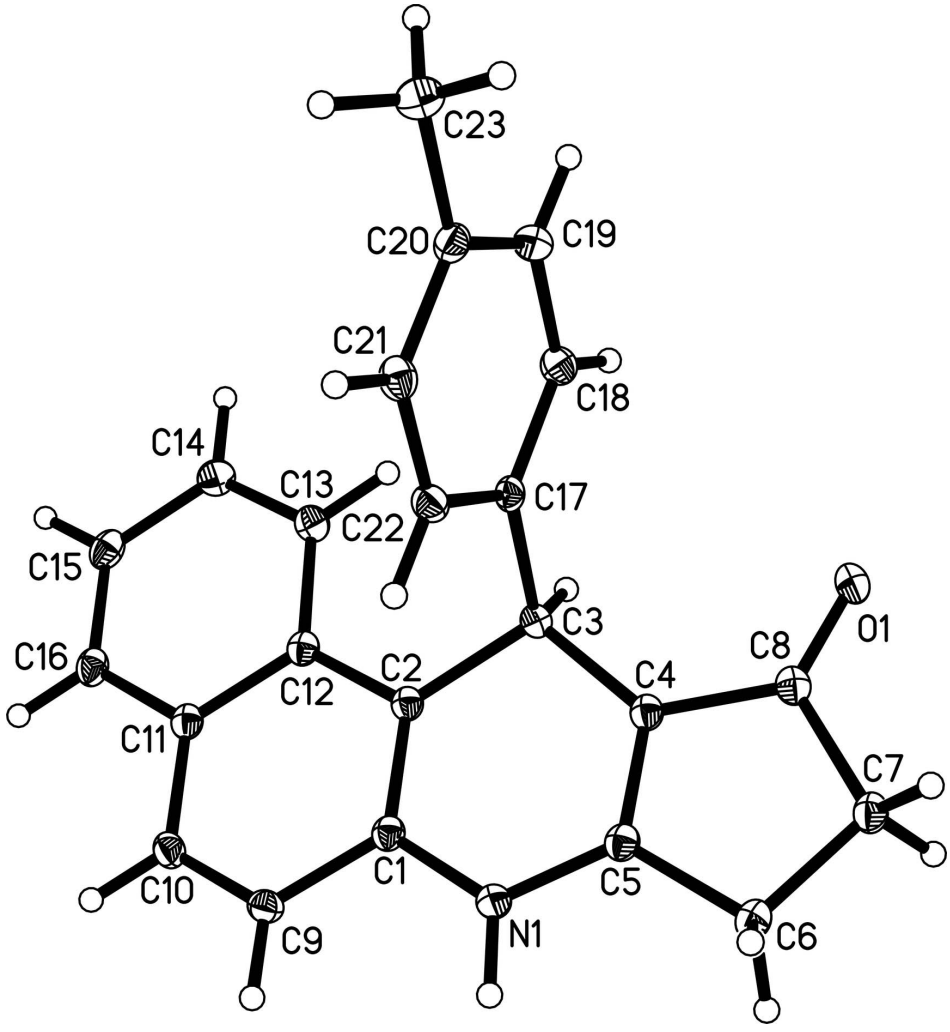


**Figure 1**

The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

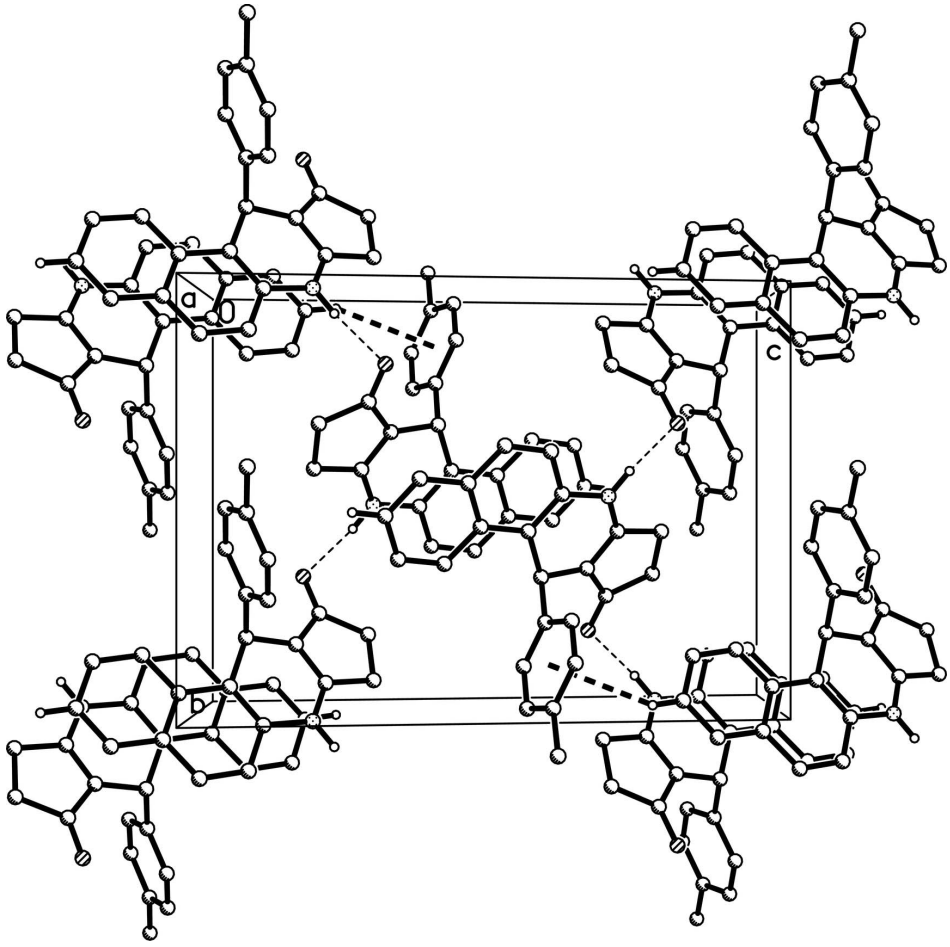


**Figure 2**

The packing diagram of (I), Hydrogen bond represented by the dashed line, *Cg* is the centroid of the ring of C17/C18/C19/C20/C21/C22, Hydrogen atoms not involved in Hydrogen bond were omitted for clarity.

### 11-(4-Methylphenyl)-8,9-dihydro-7*H*- benzo[*f*]cyclopenta[*b*]quinolin-10(11*H*)-one

*Crystal data*

$C_{23}H_{19}NO$
$M_r = 325.39$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.727\ (1)$ Å
$b = 11.6820\ (14)$ Å
$c = 16.240\ (2)$ Å
$\beta = 98.938\ (5)°$
$V = 1635.5\ (3)$ Å$^3$
$Z = 4$
$F(000) = 688$
$D_x = 1.321$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 5530 reflections
$\theta = 1.7–27.9°$
$\mu = 0.08$ mm$^{-1}$
$T = 113$ K
Prism, colorless
0.24 × 0.20 × 0.18 mm

*Data collection*

Rigaku Saturn724 CCD diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.222 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrystalClearSM Expert*; Rigaku/MSC, 2009)

$T_{min}$ = 0.981, $T_{max}$ = 0.986
15928 measured reflections
3899 independent reflections
2991 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031

$\theta_{max}$ = 27.9°, $\theta_{min}$ = 2.2°
$h$ = −11→11
$k$ = −15→15
$l$ = −21→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.124
$S$ = 1.07
3899 reflections
231 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0777P)^2 + 0.041P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 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)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.35410 (9) | 0.68276 (7) | 0.17550 (5) | 0.0239 (2) |
| N1 | 0.67402 (12) | 1.00971 (9) | 0.20388 (6) | 0.0210 (2) |
| C1 | 0.71537 (13) | 1.01482 (10) | 0.12380 (7) | 0.0189 (3) |
| C2 | 0.67622 (12) | 0.92753 (9) | 0.06669 (7) | 0.0181 (2) |
| C3 | 0.59399 (13) | 0.81856 (9) | 0.08934 (7) | 0.0181 (3) |
| H3 | 0.5039 | 0.8036 | 0.0446 | 0.022* |
| C4 | 0.53334 (13) | 0.83755 (10) | 0.17008 (7) | 0.0190 (3) |
| C5 | 0.57787 (13) | 0.92659 (10) | 0.22198 (7) | 0.0198 (3) |
| C6 | 0.50911 (14) | 0.92521 (10) | 0.30110 (8) | 0.0238 (3) |
| H6A | 0.4506 | 0.9964 | 0.3075 | 0.029* |
| H6B | 0.5903 | 0.9155 | 0.3504 | 0.029* |
| C7 | 0.40108 (14) | 0.82132 (10) | 0.28894 (8) | 0.0227 (3) |
| H7A | 0.4289 | 0.7654 | 0.3346 | 0.027* |
| H7B | 0.2919 | 0.8451 | 0.2879 | 0.027* |
| C8 | 0.42339 (13) | 0.76912 (10) | 0.20551 (8) | 0.0204 (3) |
| C9 | 0.79777 (13) | 1.11263 (10) | 0.10358 (8) | 0.0216 (3) |
| H9 | 0.8258 | 1.1702 | 0.1445 | 0.026* |
| C10 | 0.83712 (13) | 1.12502 (10) | 0.02645 (8) | 0.0229 (3) |
| H10 | 0.8904 | 1.1920 | 0.0134 | 0.027* |
| C11 | 0.79946 (13) | 1.03903 (10) | −0.03478 (8) | 0.0199 (3) |

| | | | | |
|---|---|---|---|---|
| C12 | 0.72028 (13) | 0.93887 (10) | −0.01424 (7) | 0.0188 (2) |
| C13 | 0.69057 (13) | 0.85257 (11) | −0.07591 (8) | 0.0227 (3) |
| H13 | 0.6378 | 0.7849 | −0.0639 | 0.027* |
| C14 | 0.73616 (14) | 0.86444 (11) | −0.15246 (8) | 0.0251 (3) |
| H14 | 0.7157 | 0.8048 | −0.1925 | 0.030* |
| C15 | 0.81319 (14) | 0.96438 (11) | −0.17238 (8) | 0.0258 (3) |
| H15 | 0.8441 | 0.9724 | −0.2257 | 0.031* |
| C16 | 0.84296 (13) | 1.04920 (10) | −0.11474 (8) | 0.0240 (3) |
| H16 | 0.8941 | 1.1167 | −0.1285 | 0.029* |
| C17 | 0.70253 (13) | 0.71503 (10) | 0.09513 (7) | 0.0183 (2) |
| C18 | 0.65617 (14) | 0.61097 (10) | 0.05873 (7) | 0.0232 (3) |
| H18 | 0.5536 | 0.6029 | 0.0296 | 0.028* |
| C19 | 0.75686 (14) | 0.51809 (11) | 0.06393 (8) | 0.0253 (3) |
| H19 | 0.7220 | 0.4476 | 0.0385 | 0.030* |
| C20 | 0.90795 (14) | 0.52715 (10) | 0.10585 (8) | 0.0230 (3) |
| C21 | 0.95452 (14) | 0.63162 (11) | 0.14195 (8) | 0.0239 (3) |
| H21 | 1.0576 | 0.6403 | 0.1702 | 0.029* |
| C22 | 0.85309 (14) | 0.72368 (10) | 0.13750 (8) | 0.0218 (3) |
| H22 | 0.8872 | 0.7938 | 0.1639 | 0.026* |
| C23 | 1.01741 (15) | 0.42637 (11) | 0.11254 (9) | 0.0319 (3) |
| H23A | 1.1244 | 0.4540 | 0.1171 | 0.038* |
| H23B | 0.9930 | 0.3783 | 0.0628 | 0.038* |
| H23C | 1.0059 | 0.3813 | 0.1621 | 0.038* |
| H1 | 0.6867 (18) | 1.0689 (14) | 0.2399 (10) | 0.045 (5)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0220 (4) | 0.0226 (5) | 0.0270 (5) | −0.0030 (3) | 0.0040 (4) | 0.0034 (3) |
| N1 | 0.0233 (5) | 0.0206 (5) | 0.0197 (5) | −0.0025 (4) | 0.0054 (4) | −0.0022 (4) |
| C1 | 0.0162 (5) | 0.0199 (6) | 0.0208 (6) | 0.0016 (4) | 0.0033 (5) | 0.0012 (5) |
| C2 | 0.0160 (5) | 0.0189 (6) | 0.0192 (6) | 0.0008 (4) | 0.0023 (4) | 0.0015 (4) |
| C3 | 0.0175 (5) | 0.0181 (6) | 0.0185 (6) | −0.0013 (4) | 0.0024 (5) | 0.0007 (4) |
| C4 | 0.0171 (5) | 0.0194 (6) | 0.0208 (6) | 0.0011 (4) | 0.0034 (5) | 0.0026 (5) |
| C5 | 0.0175 (5) | 0.0212 (6) | 0.0208 (6) | 0.0023 (4) | 0.0033 (5) | 0.0019 (5) |
| C6 | 0.0252 (6) | 0.0265 (7) | 0.0209 (7) | −0.0007 (5) | 0.0075 (5) | 0.0002 (5) |
| C7 | 0.0220 (6) | 0.0233 (6) | 0.0237 (7) | 0.0015 (4) | 0.0066 (5) | 0.0044 (5) |
| C8 | 0.0176 (5) | 0.0199 (6) | 0.0232 (6) | 0.0032 (4) | 0.0019 (5) | 0.0058 (5) |
| C9 | 0.0206 (6) | 0.0179 (6) | 0.0262 (7) | 0.0000 (4) | 0.0036 (5) | −0.0021 (5) |
| C10 | 0.0205 (6) | 0.0177 (6) | 0.0313 (7) | −0.0003 (4) | 0.0071 (5) | 0.0033 (5) |
| C11 | 0.0170 (5) | 0.0198 (6) | 0.0233 (6) | 0.0030 (4) | 0.0037 (5) | 0.0044 (5) |
| C12 | 0.0162 (5) | 0.0204 (6) | 0.0196 (6) | 0.0025 (4) | 0.0023 (5) | 0.0030 (4) |
| C13 | 0.0222 (6) | 0.0237 (6) | 0.0217 (6) | −0.0018 (5) | 0.0015 (5) | 0.0007 (5) |
| C14 | 0.0271 (6) | 0.0273 (6) | 0.0202 (6) | 0.0007 (5) | 0.0020 (5) | −0.0009 (5) |
| C15 | 0.0257 (6) | 0.0310 (7) | 0.0221 (7) | 0.0059 (5) | 0.0077 (5) | 0.0059 (5) |
| C16 | 0.0232 (6) | 0.0238 (6) | 0.0264 (7) | 0.0029 (5) | 0.0086 (5) | 0.0072 (5) |
| C17 | 0.0195 (5) | 0.0201 (6) | 0.0163 (6) | −0.0023 (4) | 0.0060 (5) | 0.0015 (4) |
| C18 | 0.0199 (6) | 0.0244 (6) | 0.0249 (7) | −0.0032 (5) | 0.0020 (5) | −0.0024 (5) |
| C19 | 0.0270 (6) | 0.0213 (6) | 0.0284 (7) | −0.0025 (5) | 0.0066 (5) | −0.0043 (5) |
| C20 | 0.0239 (6) | 0.0240 (6) | 0.0228 (6) | 0.0016 (5) | 0.0090 (5) | 0.0013 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C21 | 0.0189 (6) | 0.0261 (6) | 0.0261 (7) | −0.0015 (5) | 0.0016 (5) | 0.0030 (5) |
| C22 | 0.0225 (6) | 0.0208 (6) | 0.0222 (6) | −0.0029 (4) | 0.0033 (5) | −0.0011 (5) |
| C23 | 0.0312 (7) | 0.0278 (7) | 0.0376 (8) | 0.0055 (5) | 0.0081 (6) | −0.0010 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C8 | 1.2366 (14) | C11—C16 | 1.4134 (17) |
| N1—C5 | 1.3454 (15) | C11—C12 | 1.4245 (15) |
| N1—C1 | 1.4039 (16) | C12—C13 | 1.4163 (17) |
| N1—H1 | 0.902 (17) | C13—C14 | 1.3700 (17) |
| C1—C2 | 1.3849 (16) | C13—H13 | 0.9500 |
| C1—C9 | 1.4156 (15) | C14—C15 | 1.4097 (17) |
| C2—C12 | 1.4318 (16) | C14—H14 | 0.9500 |
| C2—C3 | 1.5338 (15) | C15—C16 | 1.3601 (18) |
| C3—C4 | 1.5049 (16) | C15—H15 | 0.9500 |
| C3—C17 | 1.5301 (15) | C16—H16 | 0.9500 |
| C3—H3 | 1.0000 | C17—C18 | 1.3844 (16) |
| C4—C5 | 1.3571 (16) | C17—C22 | 1.3889 (17) |
| C4—C8 | 1.4360 (16) | C18—C19 | 1.3904 (16) |
| C5—C6 | 1.5002 (16) | C18—H18 | 0.9500 |
| C6—C7 | 1.5308 (16) | C19—C20 | 1.3908 (18) |
| C6—H6A | 0.9900 | C19—H19 | 0.9500 |
| C6—H6B | 0.9900 | C20—C21 | 1.3872 (17) |
| C7—C8 | 1.5257 (17) | C20—C23 | 1.5093 (16) |
| C7—H7A | 0.9900 | C21—C22 | 1.3876 (16) |
| C7—H7B | 0.9900 | C21—H21 | 0.9500 |
| C9—C10 | 1.3571 (17) | C22—H22 | 0.9500 |
| C9—H9 | 0.9500 | C23—H23A | 0.9800 |
| C10—C11 | 1.4158 (17) | C23—H23B | 0.9800 |
| C10—H10 | 0.9500 | C23—H23C | 0.9800 |
| | | | |
| C5—N1—C1 | 119.46 (10) | C16—C11—C10 | 121.29 (11) |
| C5—N1—H1 | 115.4 (10) | C16—C11—C12 | 119.57 (11) |
| C1—N1—H1 | 123.1 (10) | C10—C11—C12 | 119.11 (11) |
| C2—C1—N1 | 121.12 (10) | C13—C12—C11 | 117.47 (11) |
| C2—C1—C9 | 121.35 (11) | C13—C12—C2 | 122.52 (11) |
| N1—C1—C9 | 117.52 (10) | C11—C12—C2 | 120.01 (11) |
| C1—C2—C12 | 118.24 (10) | C14—C13—C12 | 121.50 (11) |
| C1—C2—C3 | 121.76 (10) | C14—C13—H13 | 119.2 |
| C12—C2—C3 | 119.95 (10) | C12—C13—H13 | 119.2 |
| C4—C3—C17 | 111.17 (9) | C13—C14—C15 | 120.54 (12) |
| C4—C3—C2 | 109.12 (9) | C13—C14—H14 | 119.7 |
| C17—C3—C2 | 111.26 (9) | C15—C14—H14 | 119.7 |
| C4—C3—H3 | 108.4 | C16—C15—C14 | 119.50 (12) |
| C17—C3—H3 | 108.4 | C16—C15—H15 | 120.3 |
| C2—C3—H3 | 108.4 | C14—C15—H15 | 120.3 |
| C5—C4—C8 | 108.82 (11) | C15—C16—C11 | 121.41 (11) |
| C5—C4—C3 | 123.27 (10) | C15—C16—H16 | 119.3 |
| C8—C4—C3 | 127.91 (11) | C11—C16—H16 | 119.3 |
| N1—C5—C4 | 123.07 (11) | C18—C17—C22 | 117.82 (11) |

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| N1—C5—C6 | 122.75 (11) | C18—C17—C3 | 122.06 (10) |
| C4—C5—C6 | 114.18 (11) | C22—C17—C3 | 120.12 (10) |
| C5—C6—C7 | 102.61 (10) | C17—C18—C19 | 121.28 (11) |
| C5—C6—H6A | 111.2 | C17—C18—H18 | 119.4 |
| C7—C6—H6A | 111.2 | C19—C18—H18 | 119.4 |
| C5—C6—H6B | 111.2 | C18—C19—C20 | 120.83 (11) |
| C7—C6—H6B | 111.2 | C18—C19—H19 | 119.6 |
| H6A—C6—H6B | 109.2 | C20—C19—H19 | 119.6 |
| C8—C7—C6 | 105.69 (9) | C21—C20—C19 | 117.84 (11) |
| C8—C7—H7A | 110.6 | C21—C20—C23 | 121.01 (11) |
| C6—C7—H7A | 110.6 | C19—C20—C23 | 121.15 (11) |
| C8—C7—H7B | 110.6 | C20—C21—C22 | 121.15 (11) |
| C6—C7—H7B | 110.6 | C20—C21—H21 | 119.4 |
| H7A—C7—H7B | 108.7 | C22—C21—H21 | 119.4 |
| O1—C8—C4 | 127.77 (12) | C21—C22—C17 | 121.07 (11) |
| O1—C8—C7 | 123.64 (10) | C21—C22—H22 | 119.5 |
| C4—C8—C7 | 108.59 (10) | C17—C22—H22 | 119.5 |
| C10—C9—C1 | 120.71 (11) | C20—C23—H23A | 109.5 |
| C10—C9—H9 | 119.6 | C20—C23—H23B | 109.5 |
| C1—C9—H9 | 119.6 | H23A—C23—H23B | 109.5 |
| C9—C10—C11 | 120.54 (11) | C20—C23—H23C | 109.5 |
| C9—C10—H10 | 119.7 | H23A—C23—H23C | 109.5 |
| C11—C10—H10 | 119.7 | H23B—C23—H23C | 109.5 |

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| C5—N1—C1—C2 | −8.76 (16) | C9—C10—C11—C16 | −178.39 (11) |
| C5—N1—C1—C9 | 170.95 (10) | C9—C10—C11—C12 | −0.31 (17) |
| N1—C1—C2—C12 | 179.22 (10) | C16—C11—C12—C13 | 0.79 (16) |
| C9—C1—C2—C12 | −0.47 (16) | C10—C11—C12—C13 | −177.32 (10) |
| N1—C1—C2—C3 | −3.34 (16) | C16—C11—C12—C2 | 179.77 (10) |
| C9—C1—C2—C3 | 176.96 (10) | C10—C11—C12—C2 | 1.65 (16) |
| C1—C2—C3—C4 | 13.56 (14) | C1—C2—C12—C13 | 177.68 (10) |
| C12—C2—C3—C4 | −169.04 (10) | C3—C2—C12—C13 | 0.19 (16) |
| C1—C2—C3—C17 | −109.46 (12) | C1—C2—C12—C11 | −1.24 (16) |
| C12—C2—C3—C17 | 67.93 (13) | C3—C2—C12—C11 | −178.73 (9) |
| C17—C3—C4—C5 | 109.07 (12) | C11—C12—C13—C14 | 0.08 (17) |
| C2—C3—C4—C5 | −14.00 (15) | C2—C12—C13—C14 | −178.86 (11) |
| C17—C3—C4—C8 | −70.90 (14) | C12—C13—C14—C15 | −0.67 (18) |
| C2—C3—C4—C8 | 166.03 (11) | C13—C14—C15—C16 | 0.35 (18) |
| C1—N1—C5—C4 | 8.74 (17) | C14—C15—C16—C11 | 0.55 (18) |
| C1—N1—C5—C6 | −170.53 (10) | C10—C11—C16—C15 | 176.95 (11) |
| C8—C4—C5—N1 | −176.27 (10) | C12—C11—C16—C15 | −1.12 (17) |
| C3—C4—C5—N1 | 3.75 (17) | C4—C3—C17—C18 | 103.97 (13) |
| C8—C4—C5—C6 | 3.06 (14) | C2—C3—C17—C18 | −134.18 (11) |
| C3—C4—C5—C6 | −176.92 (10) | C4—C3—C17—C22 | −76.38 (13) |
| N1—C5—C6—C7 | 175.76 (10) | C2—C3—C17—C22 | 45.46 (15) |
| C4—C5—C6—C7 | −3.57 (13) | C22—C17—C18—C19 | −0.29 (18) |
| C5—C6—C7—C8 | 2.56 (12) | C3—C17—C18—C19 | 179.36 (11) |
| C5—C4—C8—O1 | 178.03 (11) | C17—C18—C19—C20 | −0.19 (19) |
| C3—C4—C8—O1 | −2.0 (2) | C18—C19—C20—C21 | −0.13 (19) |

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| C5—C4—C8—C7 | −1.15 (13) | C18—C19—C20—C23 | 179.35 (12) |
| C3—C4—C8—C7 | 178.82 (11) | C19—C20—C21—C22 | 0.94 (18) |
| C6—C7—C8—O1 | 179.74 (11) | C23—C20—C21—C22 | −178.54 (11) |
| C6—C7—C8—C4 | −1.04 (12) | C20—C21—C22—C17 | −1.46 (19) |
| C2—C1—C9—C10 | 1.84 (17) | C18—C17—C22—C21 | 1.10 (18) |
| N1—C1—C9—C10 | −177.87 (10) | C3—C17—C22—C21 | −178.56 (11) |
| C1—C9—C10—C11 | −1.41 (17) | | |

*Hydrogen-bond geometry (Å, º)*

Cg1 is the centroid of the C17–C22 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.902 (17) | 1.983 (17) | 2.8519 (13) | 161.2 (14) |
| C15—H15···*Cg*1$^{ii}$ | 0.95 | 3.00 | 3.8110 (15) | 145 |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*, −*y*+2, −*z*.