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## Structure Reports

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## 1-[(2-Chloro-7-methyl-3-quinoly)-methyl]pyridin-2(1H)-one

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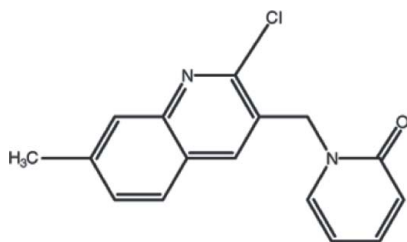
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Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.034;  $wR$  factor = 0.095; data-to-parameter ratio = 14.0.

In the title compound,  $\text{C}_{16}\text{H}_{13}\text{ClN}_2\text{O}$ , the quinoline ring system is essentially planar, with a maximum deviation of 0.021 (2) Å. The pyridone ring is oriented at a dihedral angle of 85.93 (6)° with respect to the quinoline ring system. In the crystal structure, intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules along the  $b$  axis. Weak  $\pi-\pi$  stacking interactions [centroid-centroid distances = 3.7218 (9) and 3.6083 (9) Å] are also observed.

## Related literature

For related structures, see: Arman *et al.* (2009); Clegg & Nichol (2004); Nichol & Clegg (2005). For the synthesis of 2-pyridone derivatives, see: Conreux *et al.* (2005); Roopan & Khan (2009); Roopan *et al.* (2010).



## Experimental

## Crystal data

$\text{C}_{16}\text{H}_{13}\text{ClN}_2\text{O}$   
 $M_r = 284.73$

Monoclinic,  $C2/c$   
 $a = 11.8934$  (3) Å

$b = 11.1092$  (3) Å  
 $c = 21.2858$  (6) Å  
 $\beta = 102.413$  (3)°  
 $V = 2746.67$  (13) Å<sup>3</sup>  
 $Z = 8$

Mo  $K\alpha$  radiation  
 $\mu = 0.27$  mm<sup>-1</sup>  
 $T = 295$  K  
 $0.26 \times 0.21 \times 0.18$  mm

## Data collection

Oxford Xcalibur diffractometer with an Eos (Nova) CCD detector  
Absorption correction: multi-scan (*CrysAlis PRO RED*; Oxford

Diffraction, 2009)  
 $T_{\min} = 0.932$ ,  $T_{\max} = 0.952$   
13638 measured reflections  
2556 independent reflections  
1893 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.095$   
 $S = 1.10$   
2556 reflections

182 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.12$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.22$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                            | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{C13}-\text{H13}\cdots\text{O1}^1$ | 0.93  | 2.37        | 3.299 (2)   | 173           |

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

Data collection: *CrysAlis PRO CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO CCD*; data reduction: *CrysAlis PRO RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

We thank the FIST program for the data collection at SSCU, IISc, Bangalore and Professor T. N. Guru Row, IISc, Bangalore, for his help with the data collection. FNK thanks the DST for Fast Track Proposal funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2533).

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## supporting information

*Acta Cryst.* (2010). E66, o960 [doi:10.1107/S1600536810011177]

## 1-[(2-Chloro-7-methyl-3-quinolyl)methyl]pyridin-2(1H)-one

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### S1. Comment

The pyridone analogues such as naturally occurring mappicine based molecule have been focused of great interest by reason of their diversified biological activities. *N*-alkylated 2-pyridones are important intermediates in the synthesis of alkaloids as illustrated by the recent synthetic approaches toward the mappicine family. Thus, modifications of biologically active mappicine synthons may lead to achieve the highly expected effective drugs (Roopan & Khan, 2009). Having succeeded in developing a practical, alternative synthesis of pyridine (Conreux *et al.*, 2005), we then focused our attention on the general applicability of the *N*-alkylation (Roopan *et al.*, 2010) of pyridones by mean of the *t*-BuOK/THF system In connection with the program of synthesis of 2-pyridone analogues, we report herein the synthesis of 1-[(2-chloro-7-methylquinolin-3yl)-methyl]-pyridine-2(1H)-one.

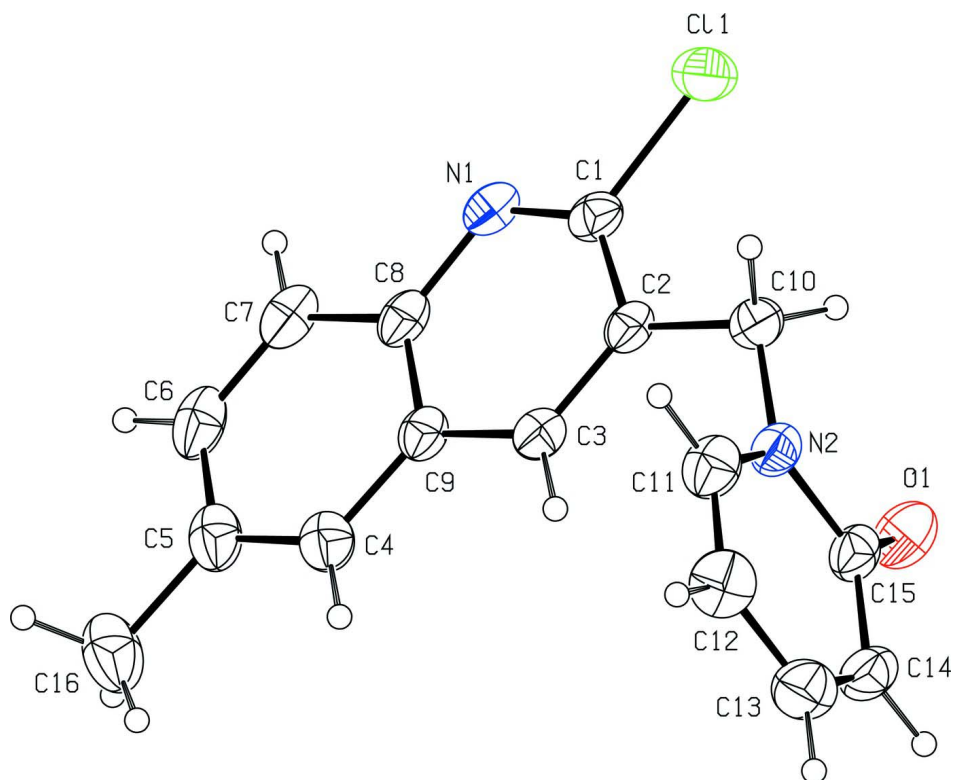
In the title molecule, the quinoline ring system (N1/C1–C9) is almost planar, with maximum deviations of 0.021 (1) Å for N1 and -0.021 (2) Å for C7 (Fig. 1). The pyridone ring (N2/C11–C15) is oriented at a dihedral angle of 85.93 (6)° with respect to the quinoline ring system. In the crystal structure, intermolecular C—H···O hydrogen bonds contribute to the stability of the structure, linking the molecules along the [010] direction (Table 1 and Fig. 2). Weak  $\pi$ – $\pi$  stacking interactions are also observed [ $Cg1 \cdots Cg3(3/2-x, 1/2-y, -z) = 3.7218(9)$ , where  $Cg1$  and  $Cg3$  are the centroids of the N1/C1–C3/C8/C9 and C4–C9 rings, respectively;  $Cg2 \cdots Cg2(2-x, y, 1/2-z) = 3.6083(9)$  Å, where  $Cg2$  is a centroid of the N2/C11–C15 ring].

### S2. Experimental

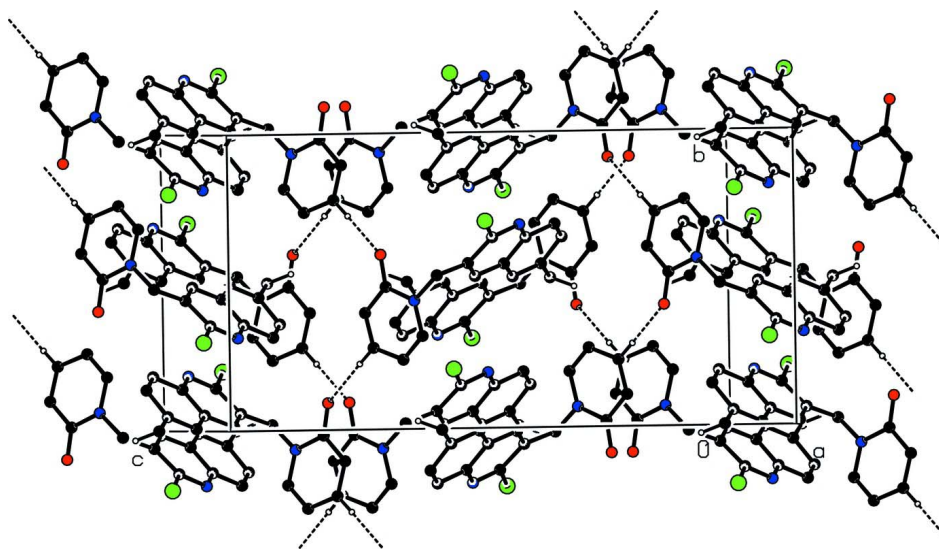
To a mixed well solution of 2-pyridone (95 mg, 1 mmol, in 2 ml of DMF), KO<sup>t</sup>Bu (112 mg, 1 mmol, in 10 ml THF) and 2-chloro-3-(chloromethyl)-7-methylquinoline (226 mg, 1 mmol) were added and the resulting mixture was refluxed at 343 K for 1 h. After the completion of the reaction, cooled and removed the excess of solvent under reduced pressure. Crushed ice was mixed with the residue. White solid was formed, filtered, dried and purified by column chromatography using hexane and ethylacetate as the eluant. Crystals of suitable quality were grown by solvent evaporation from a diethylether solution.

### S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H, respectively, and constrained to ride on their parent atoms, with  $U_{iso}(H) = 1.5U_{eq}(C)$  for methyl H and  $U_{iso}(H) = 1.2U_{eq}(C)$  for all other H atoms.

**Figure 1**

View of the title molecule with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

**Figure 2**

View of the packing diagram and the hydrogen bonding interactions of the title compound down the *a* axis. H atoms not involved in hydrogen bonding have been omitted for clarity.

1-[(2-Chloro-7-methyl-3-quinolyl)methyl]pyridin-2(1*H*)-one

## Crystal data

C<sub>16</sub>H<sub>13</sub>ClN<sub>2</sub>O $M_r = 284.73$ Monoclinic, *C2/c*Hall symbol: -*C* 2yc $a = 11.8934 (3) \text{ \AA}$  $b = 11.1092 (3) \text{ \AA}$  $c = 21.2858 (6) \text{ \AA}$  $\beta = 102.413 (3)^\circ$  $V = 2746.67 (13) \text{ \AA}^3$  $Z = 8$  $F(000) = 1184$  $D_x = 1.377 \text{ Mg m}^{-3}$ Mo *K* $\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$ 

Cell parameters from 985 reflections

 $\theta = 3.4\text{--}25.5^\circ$  $\mu = 0.27 \text{ mm}^{-1}$  $T = 295 \text{ K}$ 

Block, colourless

 $0.26 \times 0.21 \times 0.18 \text{ mm}$ 

## Data collection

Oxford Xcalibur

diffractometer with an Eos (Nova) CCD

detector

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

 $\omega$  scans

Absorption correction: multi-scan

(CrysAlis PRO RED; Oxford Diffraction, 2009)

 $T_{\min} = 0.932, T_{\max} = 0.952$ 

13638 measured reflections

2556 independent reflections

1893 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.025$  $\theta_{\max} = 25.5^\circ, \theta_{\min} = 3.4^\circ$  $h = -14 \rightarrow 14$  $k = -13 \rightarrow 13$  $l = -25 \rightarrow 25$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.034$  $wR(F^2) = 0.095$  $S = 1.10$ 

2556 reflections

182 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

 $w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.1075P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.12 \text{ e \AA}^{-3}$  $\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$ 

## Special details

**Geometry.** Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

**Refinement.** Refinement on  $F^2$  for ALL reflections except those flagged by the user for potential systematic errors. Weighted  $R$ -factors  $wR$  and all goodnesses 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 observed criterion of  $F^2 > \sigma(F^2)$  is used only for calculating  $-R$ -factor-obs 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 ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Cl1 | 1.09208 (3)  | 0.30078 (4)  | 0.05639 (2)  | 0.0632 (2)                       |
| O1  | 0.85807 (10) | 0.41006 (11) | 0.21457 (5)  | 0.0685 (5)                       |
| N1  | 0.89822 (11) | 0.31396 (11) | -0.02677 (6) | 0.0489 (5)                       |

|      |              |              |              |            |
|------|--------------|--------------|--------------|------------|
| N2   | 0.92375 (10) | 0.56671 (11) | 0.16368 (5)  | 0.0467 (4) |
| C1   | 0.95261 (12) | 0.35624 (13) | 0.02798 (7)  | 0.0459 (5) |
| C2   | 0.91154 (12) | 0.44280 (13) | 0.06637 (7)  | 0.0434 (5) |
| C3   | 0.80426 (12) | 0.48690 (13) | 0.04162 (7)  | 0.0460 (5) |
| C4   | 0.62855 (14) | 0.49071 (14) | -0.04533 (8) | 0.0542 (6) |
| C5   | 0.56786 (14) | 0.44843 (16) | -0.10296 (8) | 0.0602 (6) |
| C6   | 0.61864 (16) | 0.35797 (17) | -0.13404 (8) | 0.0648 (7) |
| C7   | 0.72499 (15) | 0.31365 (15) | -0.10943 (7) | 0.0580 (6) |
| C8   | 0.78938 (13) | 0.35771 (14) | -0.05035 (7) | 0.0475 (5) |
| C9   | 0.73932 (12) | 0.44607 (13) | -0.01767 (7) | 0.0447 (5) |
| C10  | 0.98427 (13) | 0.48243 (15) | 0.13024 (7)  | 0.0513 (5) |
| C11  | 0.92446 (14) | 0.68655 (15) | 0.14990 (8)  | 0.0595 (6) |
| C12  | 0.86613 (16) | 0.76605 (17) | 0.17753 (9)  | 0.0705 (7) |
| C13  | 0.80463 (15) | 0.72478 (18) | 0.22262 (8)  | 0.0688 (7) |
| C14  | 0.80364 (13) | 0.60698 (17) | 0.23677 (8)  | 0.0593 (6) |
| C15  | 0.86079 (13) | 0.51932 (16) | 0.20637 (7)  | 0.0503 (6) |
| C16  | 0.44951 (15) | 0.49594 (19) | -0.13245 (9) | 0.0845 (8) |
| H3   | 0.77340      | 0.54520      | 0.06440      | 0.0550*    |
| H4   | 0.59630      | 0.55000      | -0.02390     | 0.0650*    |
| H6   | 0.57750      | 0.32750      | -0.17290     | 0.0780*    |
| H7   | 0.75570      | 0.25410      | -0.13150     | 0.0700*    |
| H10A | 1.00650      | 0.41230      | 0.15720      | 0.0620*    |
| H10B | 1.05400      | 0.52020      | 0.12310      | 0.0620*    |
| H11  | 0.96660      | 0.71350      | 0.12060      | 0.0710*    |
| H12  | 0.86630      | 0.84730      | 0.16710      | 0.0850*    |
| H13  | 0.76440      | 0.77910      | 0.24280      | 0.0820*    |
| H14  | 0.76400      | 0.58190      | 0.26770      | 0.0710*    |
| H16A | 0.43010      | 0.55940      | -0.10600     | 0.1270*    |
| H16B | 0.39420      | 0.43210      | -0.13540     | 0.1270*    |
| H16C | 0.44880      | 0.52660      | -0.17470     | 0.1270*    |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$   | $U^{23}$    |
|-----|-------------|-------------|-------------|--------------|------------|-------------|
| Cl1 | 0.0530 (3)  | 0.0666 (3)  | 0.0737 (3)  | 0.0066 (2)   | 0.0219 (2) | 0.0046 (2)  |
| O1  | 0.0847 (9)  | 0.0633 (8)  | 0.0632 (8)  | -0.0139 (7)  | 0.0289 (6) | 0.0034 (6)  |
| N1  | 0.0569 (8)  | 0.0505 (8)  | 0.0446 (8)  | -0.0072 (6)  | 0.0228 (6) | -0.0011 (6) |
| N2  | 0.0474 (7)  | 0.0559 (8)  | 0.0375 (7)  | -0.0067 (6)  | 0.0108 (6) | -0.0030 (6) |
| C1  | 0.0485 (9)  | 0.0466 (9)  | 0.0475 (9)  | -0.0040 (7)  | 0.0214 (7) | 0.0056 (7)  |
| C2  | 0.0482 (9)  | 0.0476 (9)  | 0.0372 (8)  | -0.0069 (7)  | 0.0154 (7) | 0.0024 (6)  |
| C3  | 0.0492 (9)  | 0.0486 (9)  | 0.0422 (9)  | -0.0028 (7)  | 0.0143 (7) | -0.0033 (7) |
| C4  | 0.0544 (10) | 0.0542 (10) | 0.0527 (10) | -0.0062 (8)  | 0.0085 (8) | 0.0039 (8)  |
| C5  | 0.0572 (10) | 0.0661 (11) | 0.0532 (11) | -0.0165 (9)  | 0.0030 (8) | 0.0139 (9)  |
| C6  | 0.0747 (12) | 0.0765 (12) | 0.0406 (10) | -0.0307 (10) | 0.0064 (9) | 0.0007 (9)  |
| C7  | 0.0726 (12) | 0.0615 (11) | 0.0436 (10) | -0.0180 (9)  | 0.0205 (9) | -0.0052 (8) |
| C8  | 0.0578 (10) | 0.0490 (9)  | 0.0392 (9)  | -0.0138 (8)  | 0.0183 (7) | 0.0023 (7)  |
| C9  | 0.0495 (9)  | 0.0466 (9)  | 0.0394 (9)  | -0.0080 (7)  | 0.0125 (7) | 0.0033 (7)  |
| C10 | 0.0462 (9)  | 0.0643 (10) | 0.0449 (9)  | -0.0019 (8)  | 0.0131 (7) | -0.0028 (8) |

|     |             |             |             |              |              |              |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C11 | 0.0643 (11) | 0.0594 (11) | 0.0558 (11) | -0.0124 (9)  | 0.0150 (8)   | 0.0015 (8)   |
| C12 | 0.0798 (13) | 0.0590 (11) | 0.0725 (13) | -0.0027 (10) | 0.0158 (11)  | -0.0053 (9)  |
| C13 | 0.0641 (11) | 0.0784 (14) | 0.0624 (12) | 0.0046 (10)  | 0.0105 (9)   | -0.0214 (10) |
| C14 | 0.0525 (10) | 0.0839 (13) | 0.0437 (9)  | -0.0086 (9)  | 0.0153 (8)   | -0.0137 (9)  |
| C15 | 0.0482 (9)  | 0.0649 (11) | 0.0367 (9)  | -0.0122 (8)  | 0.0069 (7)   | -0.0054 (8)  |
| C16 | 0.0655 (13) | 0.0976 (16) | 0.0791 (14) | -0.0176 (11) | -0.0096 (11) | 0.0184 (12)  |

*Geometric parameters (Å, °)*

|                          |             |                          |           |
|--------------------------|-------------|--------------------------|-----------|
| C11—C1                   | 1.7509 (15) | C11—C12                  | 1.335 (3) |
| O1—C15                   | 1.228 (2)   | C12—C13                  | 1.403 (3) |
| N1—C1                    | 1.2935 (19) | C13—C14                  | 1.344 (3) |
| N1—C8                    | 1.373 (2)   | C14—C15                  | 1.421 (2) |
| N2—C10                   | 1.457 (2)   | C3—H3                    | 0.9300    |
| N2—C11                   | 1.364 (2)   | C4—H4                    | 0.9300    |
| N2—C15                   | 1.3986 (19) | C6—H6                    | 0.9300    |
| C1—C2                    | 1.415 (2)   | C7—H7                    | 0.9300    |
| C2—C3                    | 1.363 (2)   | C10—H10A                 | 0.9700    |
| C2—C10                   | 1.512 (2)   | C10—H10B                 | 0.9700    |
| C3—C9                    | 1.406 (2)   | C11—H11                  | 0.9300    |
| C4—C5                    | 1.366 (2)   | C12—H12                  | 0.9300    |
| C4—C9                    | 1.412 (2)   | C13—H13                  | 0.9300    |
| C5—C6                    | 1.409 (3)   | C14—H14                  | 0.9300    |
| C5—C16                   | 1.508 (3)   | C16—H16A                 | 0.9600    |
| C6—C7                    | 1.354 (3)   | C16—H16B                 | 0.9600    |
| C7—C8                    | 1.412 (2)   | C16—H16C                 | 0.9600    |
| C8—C9                    | 1.407 (2)   |                          |           |
| C11...C9 <sup>i</sup>    | 3.6496 (15) | C15...C10 <sup>iv</sup>  | 3.592 (2) |
| C11...C5 <sup>ii</sup>   | 3.6161 (18) | C15...C15 <sup>iv</sup>  | 3.431 (2) |
| C11...C3 <sup>i</sup>    | 3.5414 (15) | C16...C14 <sup>vi</sup>  | 3.526 (3) |
| C11...H10A               | 2.8500      | C5...H12 <sup>vii</sup>  | 2.8400    |
| C11...H10B               | 2.9100      | C6...H14 <sup>viii</sup> | 3.0600    |
| C11...H16B <sup>ii</sup> | 3.0700      | C7...H14 <sup>viii</sup> | 2.9900    |
| O1...C2                  | 3.3690 (18) | C8...H10B <sup>i</sup>   | 2.9900    |
| O1...C7 <sup>ii</sup>    | 3.348 (2)   | C11...H3                 | 2.7600    |
| O1...C13 <sup>iii</sup>  | 3.299 (2)   | C14...H16B <sup>vi</sup> | 2.8600    |
| O1...H10A                | 2.3500      | C15...H3                 | 2.9900    |
| O1...H13 <sup>iii</sup>  | 2.3700      | C16...H12 <sup>vii</sup> | 3.0100    |
| O1...H10A <sup>iv</sup>  | 2.8600      | H3...N2                  | 2.4700    |
| O1...H7 <sup>ii</sup>    | 2.6900      | H3...C11                 | 2.7600    |
| N2...C15 <sup>iv</sup>   | 3.3835 (19) | H3...C15                 | 2.9900    |
| N1...H11 <sup>i</sup>    | 2.8400      | H3...H4                  | 2.5000    |
| N1...H10B <sup>i</sup>   | 2.9000      | H4...H3                  | 2.5000    |
| N2...H3                  | 2.4700      | H4...H16A                | 2.3400    |
| C1...C6 <sup>ii</sup>    | 3.507 (2)   | H7...O1 <sup>ii</sup>    | 2.6900    |
| C1...C7 <sup>ii</sup>    | 3.550 (2)   | H10A...C11               | 2.8500    |
| C2...O1                  | 3.3690 (18) | H10A...O1                | 2.3500    |

|                         |             |                           |             |
|-------------------------|-------------|---------------------------|-------------|
| C2...C7 <sup>ii</sup>   | 3.498 (2)   | H10A...O1 <sup>iv</sup>   | 2.8600      |
| C3...C11                | 3.295 (2)   | H10B...C11                | 2.9100      |
| C3...C15                | 3.445 (2)   | H10B...H11                | 2.3800      |
| C3...C11 <sup>i</sup>   | 3.5414 (15) | H10B...N1 <sup>i</sup>    | 2.9000      |
| C5...C11 <sup>ii</sup>  | 3.6161 (18) | H10B...C8 <sup>i</sup>    | 2.9900      |
| C6...C1 <sup>ii</sup>   | 3.507 (2)   | H11...H10B                | 2.3800      |
| C7...C2 <sup>ii</sup>   | 3.498 (2)   | H11...N1 <sup>i</sup>     | 2.8400      |
| C7...O1 <sup>ii</sup>   | 3.348 (2)   | H12...C5 <sup>vii</sup>   | 2.8400      |
| C7...C1 <sup>ii</sup>   | 3.550 (2)   | H12...C16 <sup>vii</sup>  | 3.0100      |
| C8...C8 <sup>ii</sup>   | 3.473 (2)   | H12...H16C <sup>vii</sup> | 2.5800      |
| C9...C11 <sup>i</sup>   | 3.6496 (15) | H13...O1 <sup>v</sup>     | 2.3700      |
| C10...C15 <sup>iv</sup> | 3.592 (2)   | H14...C6 <sup>ix</sup>    | 3.0600      |
| C11...C3                | 3.295 (2)   | H14...C7 <sup>ix</sup>    | 2.9900      |
| C13...O1 <sup>v</sup>   | 3.299 (2)   | H16A...H4                 | 2.3400      |
| C14...C16 <sup>vi</sup> | 3.526 (3)   | H16B...C14 <sup>vi</sup>  | 2.8600      |
| C15...N2 <sup>iv</sup>  | 3.3835 (19) | H16B...C11 <sup>ii</sup>  | 3.0700      |
| C15...C3                | 3.445 (2)   | H16C...H12 <sup>vii</sup> | 2.5800      |
|                         |             |                           |             |
| C1—N1—C8                | 116.78 (13) | O1—C15—C14                | 125.67 (15) |
| C10—N2—C11              | 119.73 (12) | N2—C15—C14                | 114.41 (15) |
| C10—N2—C15              | 117.73 (13) | C2—C3—H3                  | 119.00      |
| C11—N2—C15              | 122.42 (13) | C9—C3—H3                  | 119.00      |
| C11—C1—N1               | 115.89 (11) | C5—C4—H4                  | 119.00      |
| C11—C1—C2               | 117.30 (11) | C9—C4—H4                  | 119.00      |
| N1—C1—C2                | 126.81 (14) | C5—C6—H6                  | 119.00      |
| C1—C2—C3                | 115.58 (13) | C7—C6—H6                  | 119.00      |
| C1—C2—C10               | 121.03 (13) | C6—C7—H7                  | 120.00      |
| C3—C2—C10               | 123.39 (13) | C8—C7—H7                  | 120.00      |
| C2—C3—C9                | 121.28 (14) | N2—C10—H10A               | 109.00      |
| C5—C4—C9                | 121.27 (15) | N2—C10—H10B               | 109.00      |
| C4—C5—C6                | 118.03 (16) | C2—C10—H10A               | 109.00      |
| C4—C5—C16               | 121.28 (16) | C2—C10—H10B               | 109.00      |
| C6—C5—C16               | 120.68 (16) | H10A—C10—H10B             | 108.00      |
| C5—C6—C7                | 122.49 (16) | N2—C11—H11                | 119.00      |
| C6—C7—C8                | 120.10 (15) | C12—C11—H11               | 119.00      |
| N1—C8—C7                | 119.42 (14) | C11—C12—H12               | 121.00      |
| N1—C8—C9                | 122.15 (13) | C13—C12—H12               | 121.00      |
| C7—C8—C9                | 118.43 (14) | C12—C13—H13               | 120.00      |
| C3—C9—C4                | 122.97 (14) | C14—C13—H13               | 120.00      |
| C3—C9—C8                | 117.37 (13) | C13—C14—H14               | 119.00      |
| C4—C9—C8                | 119.65 (14) | C15—C14—H14               | 119.00      |
| N2—C10—C2               | 112.30 (12) | C5—C16—H16A               | 109.00      |
| N2—C11—C12              | 121.55 (16) | C5—C16—H16B               | 109.00      |
| C11—C12—C13             | 118.81 (17) | C5—C16—H16C               | 109.00      |
| C12—C13—C14             | 120.18 (17) | H16A—C16—H16B             | 109.00      |
| C13—C14—C15             | 122.51 (16) | H16A—C16—H16C             | 109.00      |
| O1—C15—N2               | 119.91 (14) | H16B—C16—H16C             | 109.00      |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C8—N1—C1—C11   | -179.03 (11) | C2—C3—C9—C4     | -179.11 (15) |
| C8—N1—C1—C2    | 0.2 (2)      | C2—C3—C9—C8     | 0.1 (2)      |
| C1—N1—C8—C7    | -179.03 (14) | C9—C4—C5—C6     | 0.5 (2)      |
| C1—N1—C8—C9    | 1.3 (2)      | C9—C4—C5—C16    | 179.90 (16)  |
| C11—N2—C10—C2  | -85.11 (17)  | C5—C4—C9—C3     | -179.74 (15) |
| C15—N2—C10—C2  | 91.08 (15)   | C5—C4—C9—C8     | 1.0 (2)      |
| C10—N2—C11—C12 | 177.19 (16)  | C4—C5—C6—C7     | -1.1 (3)     |
| C15—N2—C11—C12 | 1.2 (2)      | C16—C5—C6—C7    | 179.45 (17)  |
| C10—N2—C15—O1  | 0.1 (2)      | C5—C6—C7—C8     | 0.2 (3)      |
| C10—N2—C15—C14 | -179.61 (13) | C6—C7—C8—N1     | -178.43 (15) |
| C11—N2—C15—O1  | 176.14 (14)  | C6—C7—C8—C9     | 1.3 (2)      |
| C11—N2—C15—C14 | -3.5 (2)     | N1—C8—C9—C3     | -1.5 (2)     |
| C11—C1—C2—C3   | 177.80 (11)  | N1—C8—C9—C4     | 177.83 (14)  |
| C11—C1—C2—C10  | -1.85 (19)   | C7—C8—C9—C3     | 178.85 (14)  |
| N1—C1—C2—C3    | -1.4 (2)     | C7—C8—C9—C4     | -1.9 (2)     |
| N1—C1—C2—C10   | 178.91 (14)  | N2—C11—C12—C13  | 1.2 (3)      |
| C1—C2—C3—C9    | 1.2 (2)      | C11—C12—C13—C14 | -0.9 (3)     |
| C10—C2—C3—C9   | -179.19 (14) | C12—C13—C14—C15 | -1.7 (3)     |
| C1—C2—C10—N2   | -176.75 (13) | C13—C14—C15—O1  | -175.85 (16) |
| C3—C2—C10—N2   | 3.6 (2)      | C13—C14—C15—N2  | 3.8 (2)      |

Symmetry codes: (i)  $-x+2, -y+1, -z$ ; (ii)  $-x+3/2, -y+1/2, -z$ ; (iii)  $-x+3/2, y-1/2, -z+1/2$ ; (iv)  $-x+2, y, -z+1/2$ ; (v)  $-x+3/2, y+1/2, -z+1/2$ ; (vi)  $-x+1, -y+1, -z$ ; (vii)  $-x+3/2, -y+3/2, -z$ ; (viii)  $x, -y+1, z-1/2$ ; (ix)  $x, -y+1, z+1/2$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

| $D-H\cdots A$                    | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|-------|-------------|-------------|---------------|
| C13—H13 $\cdots$ O1 <sup>v</sup> | 0.93  | 2.37        | 3.299 (2)   | 173           |

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