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## Structure Reports

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# (2E)-3-(2-Chlorobenzo[h]quinolin-3-yl)-1-(2-methyl-4-phenylquinolin-3-yl)prop-2-en-1-one

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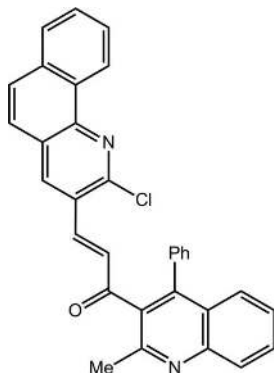
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.034;  $wR$  factor = 0.095; data-to-parameter ratio = 14.9.

In the title compound,  $\text{C}_{32}\text{H}_{21}\text{ClN}_2\text{O}$ , an almost planar (r.m.s. deviation = 0.033 Å) prop-2-en-1-one bridge links quinolinyl and benzoquinolinyl residues; the latter are twisted out of the plane of the bridge [dihedral angles = 75.94 (5) and 20.20 (5)°, respectively]. In the crystal, a three-dimensional architecture arises as a result of  $\text{C}-\text{H}\cdots\text{O}$ ,  $\text{C}-\text{H}\cdots\pi$  and  $\pi-\pi$  [centroid-centroid distances involving pyridine rings = 3.5806 (7)–3.7537 (7) Å] interactions.

## Related literature

For biological applications of quinoline derivatives, see: Jörg *et al.* (2007); Prasath *et al.* (2013a). For a related structure, see: Prasath *et al.* (2013b).



## Experimental

### Crystal data

 $\text{C}_{32}\text{H}_{21}\text{ClN}_2\text{O}$ 
 $M_r = 484.96$ 

Triclinic,  $P\bar{1}$   
 $a = 7.1354$  (3) Å  
 $b = 10.1627$  (5) Å  
 $c = 17.0127$  (8) Å  
 $\alpha = 78.758$  (4)°  
 $\beta = 79.544$  (4)°  
 $\gamma = 84.042$  (4)°

$V = 1186.91$  (9) Å<sup>3</sup>  
 $Z = 2$   
 Cu  $K\alpha$  radiation  
 $\mu = 1.65$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.30 \times 0.25 \times 0.20$  mm

### Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector  
 Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)  
 $T_{\min} = 0.870$ ,  $T_{\max} = 1.000$

8673 measured reflections  
 4849 independent reflections  
 4433 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.095$   
 $S = 1.04$   
 4849 reflections

326 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.24$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.31$  e Å<sup>-3</sup>

## Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the N1-pyridyl ring.

| $D-\text{H}\cdots A$                         | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{C10}-\text{H10C}\cdots\text{O1}^i$    | 0.98         | 2.54               | 3.4116 (18) | 148                  |
| $\text{C15}-\text{H15}\cdots\text{Cg1}^{ii}$ | 0.95         | 2.65               | 3.4695 (15) | 145                  |

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

Data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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, 2012) and *DIAMOND* (Brandenburg, 2006); 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: PV2642).

## References

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## supporting information

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**(2E)-3-(2-Chlorobenzo[*h*]quinolin-3-yl)-1-(2-methyl-4-phenylquinolin-3-yl)prop-2-en-1-one**

**R. Prasath, S. Sarveswari, Seik Weng Ng and Edward R. T. Tiekink**

**S1. Comment**

In connection with the biological potential of quinolinyl derivatives (Jörg *et al.* 2007; Prasath *et al.* 2013a), the title compound (I) was prepared and subjected to a crystallographic study.

The molecular structure of (I) (Fig. 1), sees two somewhat splayed [dihedral angle = 61.60 (3)°] quinolinyl and benzoquinolinyl residues connected by the ends of a planar (r.m.s. deviation = 0.033 Å) prop-2-en-1-one bridge. The quinolinyl, especially, and benzoquinolinyl residues are twisted out of the plane of the prop-2-en-1-one bridge forming dihedral angles of 75.94 (5) and 20.20 (5)°, respectively. The phenyl ring is inclined with respect to the quinolinyl residue to which it is attached, forming a dihedral angle of 73.76 (5)°. Finally, the conformation about the ethylene bond [C18=C19 = 1.3351 (18) Å] is *E*.

A similar conformation was reported recently for a related structure having two quinolinyl residues bridged by a prop-2-en-1-one residue, namely (2*E*)-3-(2-chloro-8-methylquinolin-3-yl)-1-(5,7-dimethylquinolin-6-yl)prop-2-en-1-one (Prasath *et al.*, 2013b) where the dihedral angle between the quinolinyl residues was 83.72 (4)°.

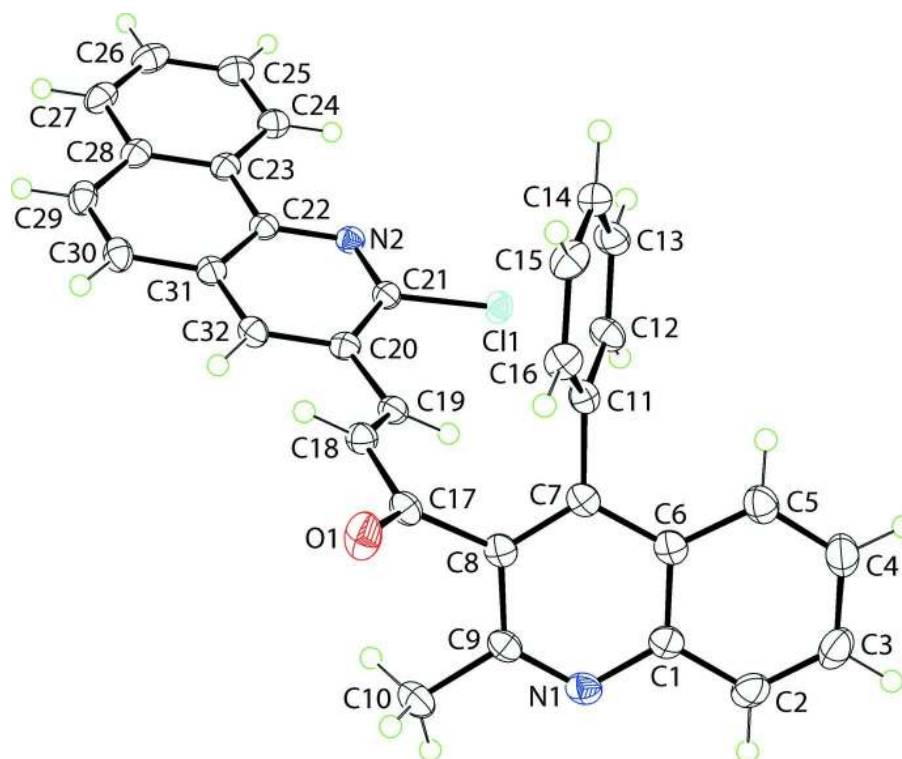
In the crystal packing methyl-C—H $\cdots$ O (carbonyl) interactions (Table 1), link molecules into centrosymmetric dimers which are connected into a three dimensional architecture by phenyl-C—H $\cdots$  $\pi$ (N1-pyridyl) (Table 1), and  $\pi$ – $\pi$  interactions [inter-centroid distances: Cg(N1-pyridyl) $\cdots$ Cg(C1–C6)<sup>i</sup> = 3.7537 (7) Å, Cg(N2-pyridyl) $\cdots$ Cg(C22—C23,C28–C31)<sup>ii,iii</sup> = 3.5806 (7) and 3.7286 (7) Å for *i* - *x*, 1 - *y*, 2 - *z*, *ii* 1 - *x*, -*y*, 1 - *z*, *iii* -*x*, -*y*, 1 - *z*] (Fig. 2).

**S2. Experimental**

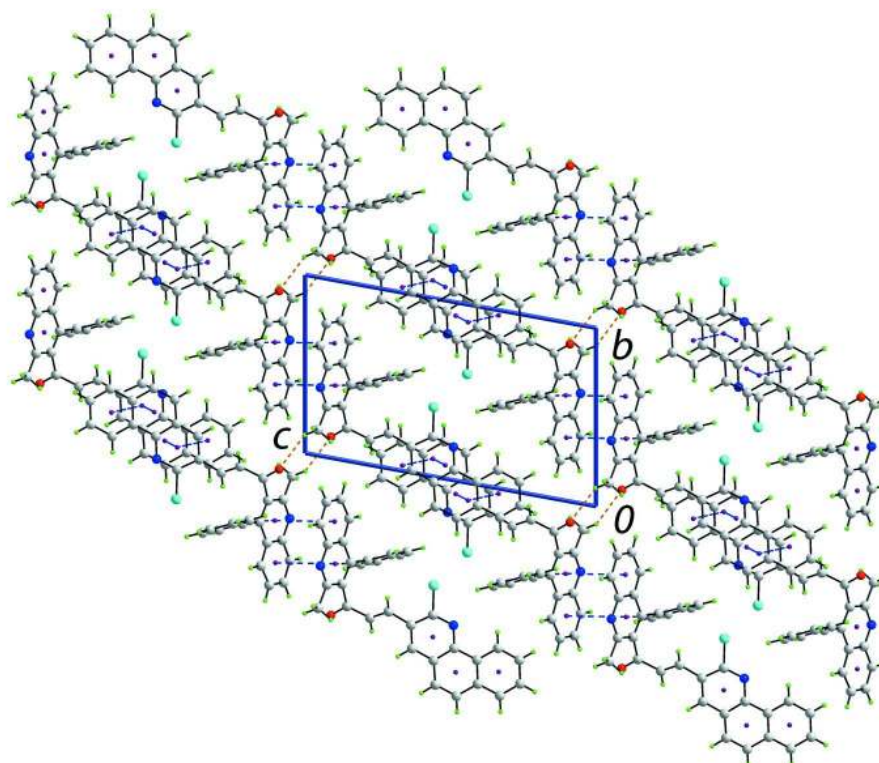
A mixture of 3-acetyl-2-methyl-4-phenylquinoline (260 mg, 0.001 *M*) and 2-chlorobenzoquinoline-3-carbaldehyde (240 mg, 0.001 *M*) in methanol (20 ml) containing potassium hydroxide (0.2 g) was stirred at room temperature for 12 h. After this, the reaction mixture was neutralized with dilute acetic acid and the resultant solid was filtered, dried and purified by column chromatography using an ethyl acetate - hexane (4:1) mixture to afford the title compound, (I). Re-crystallization was by slow evaporation of an acetone solution of (I), which yielded colourless blocks in 80% yield; *M.pt.*: 460–462 K.

**S3. Refinement**

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95–0.98 Å,  $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$ ] and were included in the refinement in the riding model approximation.

**Figure 1**

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 70% probability level.

**Figure 2**

View in projection down the  $a$  axis of the unit-cell contents of (I). The C—H $\cdots$ O, C—H $\cdots$  $\pi$  and  $\pi$ — $\pi$  interactions are shown as orange, purple and blue dashed lines, respectively.

**(2E)-3-(2-Chlorobenzo[*h*]quinolin-3-yl)-1-(2-methyl-4-phenylquinolin-3-yl)prop-2-en-1-one**

*Crystal data*

$C_{32}H_{21}ClN_2O$

$M_r = 484.96$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 7.1354$  (3) Å

$b = 10.1627$  (5) Å

$c = 17.0127$  (8) Å

$\alpha = 78.758$  (4) $^\circ$

$\beta = 79.544$  (4) $^\circ$

$\gamma = 84.042$  (4) $^\circ$

$V = 1186.91$  (9) Å $^3$

$Z = 2$

$F(000) = 504$

$D_x = 1.357$  Mg m $^{-3}$

Cu  $K\alpha$  radiation,  $\lambda = 1.54184$  Å

Cell parameters from 5174 reflections

$\theta = 2.7$ – $76.5^\circ$

$\mu = 1.65$  mm $^{-1}$

$T = 100$  K

Block, colourless

$0.30 \times 0.25 \times 0.20$  mm

*Data collection*

Agilent SuperNova Dual

diffractometer with an Atlas detector

Radiation source: SuperNova (Cu) X-ray

Source

Mirror monochromator

Detector resolution: 10.4041 pixels mm $^{-1}$

$\omega$  scan

Absorption correction: multi-scan

(*CrysAlis PRO*; Agilent, 2013)

$T_{\min} = 0.870$ ,  $T_{\max} = 1.000$

8673 measured reflections

4849 independent reflections

4433 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 76.6^\circ$ ,  $\theta_{\min} = 2.7^\circ$

$h = -7 \rightarrow 8$

$k = -10 \rightarrow 12$

$l = -15 \rightarrow 21$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.095$   
 $S = 1.04$   
 4849 reflections  
 326 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  
 $w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.2928P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.002$   
 $\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

|      | $x$           | $y$          | $z$           | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|--------------|---------------|----------------------------------|
| Cl1  | 0.32862 (4)   | 0.39004 (3)  | 0.557615 (17) | 0.02330 (9)                      |
| O1   | -0.10222 (16) | 0.11751 (11) | 0.91090 (6)   | 0.0351 (3)                       |
| N1   | 0.29529 (16)  | 0.38332 (11) | 0.94697 (6)   | 0.0245 (2)                       |
| N2   | 0.31428 (15)  | 0.18592 (10) | 0.48984 (6)   | 0.0194 (2)                       |
| C1   | 0.22372 (19)  | 0.51192 (13) | 0.92151 (7)   | 0.0228 (3)                       |
| C2   | 0.2945 (2)    | 0.61806 (15) | 0.94820 (8)   | 0.0295 (3)                       |
| H2   | 0.3912        | 0.5989       | 0.9814        | 0.035*                           |
| C3   | 0.2242 (2)    | 0.74768 (15) | 0.92635 (9)   | 0.0348 (3)                       |
| H3   | 0.2719        | 0.8182       | 0.9447        | 0.042*                           |
| C4   | 0.0813 (2)    | 0.77789 (15) | 0.87687 (9)   | 0.0346 (3)                       |
| H4   | 0.0311        | 0.8682       | 0.8634        | 0.042*                           |
| C5   | 0.0143 (2)    | 0.67797 (14) | 0.84801 (8)   | 0.0283 (3)                       |
| H5   | -0.0798       | 0.6997       | 0.8136        | 0.034*                           |
| C6   | 0.08475 (18)  | 0.54237 (13) | 0.86931 (7)   | 0.0217 (3)                       |
| C7   | 0.01900 (18)  | 0.43308 (13) | 0.84298 (7)   | 0.0204 (2)                       |
| C8   | 0.08610 (18)  | 0.30408 (13) | 0.87255 (7)   | 0.0208 (3)                       |
| C9   | 0.22715 (18)  | 0.28343 (13) | 0.92474 (7)   | 0.0225 (3)                       |
| C10  | 0.3081 (2)    | 0.14360 (14) | 0.95496 (9)   | 0.0308 (3)                       |
| H10A | 0.4311        | 0.1487       | 0.9718        | 0.046*                           |
| H10B | 0.3267        | 0.0908       | 0.9114        | 0.046*                           |
| H10C | 0.2192        | 0.1004       | 1.0014        | 0.046*                           |
| C11  | -0.11840 (18) | 0.46154 (12) | 0.78448 (8)   | 0.0210 (2)                       |
| C12  | -0.05441 (19) | 0.51971 (13) | 0.70407 (8)   | 0.0246 (3)                       |
| H12  | 0.0762        | 0.5378       | 0.6873        | 0.029*                           |
| C13  | -0.1793 (2)   | 0.55154 (14) | 0.64831 (8)   | 0.0262 (3)                       |

|     |               |               |             |            |
|-----|---------------|---------------|-------------|------------|
| H13 | -0.1337       | 0.5896        | 0.5934      | 0.031*     |
| C14 | -0.3710 (2)   | 0.52756 (13)  | 0.67300 (8) | 0.0259 (3) |
| H14 | -0.4576       | 0.5513        | 0.6353      | 0.031*     |
| C15 | -0.43578 (19) | 0.46909 (14)  | 0.75253 (9) | 0.0273 (3) |
| H15 | -0.5668       | 0.4523        | 0.7693      | 0.033*     |
| C16 | -0.30955 (19) | 0.43462 (14)  | 0.80842 (8) | 0.0250 (3) |
| H16 | -0.3543       | 0.3929        | 0.8627      | 0.030*     |
| C17 | 0.00319 (19)  | 0.18359 (13)  | 0.85606 (8) | 0.0224 (3) |
| C18 | 0.04774 (18)  | 0.14195 (13)  | 0.77665 (8) | 0.0217 (3) |
| H18 | -0.0178       | 0.0691        | 0.7701      | 0.026*     |
| C19 | 0.17108 (18)  | 0.19583 (12)  | 0.71255 (8) | 0.0202 (2) |
| H19 | 0.2367        | 0.2702        | 0.7168      | 0.024*     |
| C20 | 0.20927 (17)  | 0.14471 (13)  | 0.63586 (7) | 0.0194 (2) |
| C21 | 0.28047 (17)  | 0.22343 (12)  | 0.56031 (8) | 0.0190 (2) |
| C22 | 0.27867 (17)  | 0.05826 (12)  | 0.48717 (7) | 0.0190 (2) |
| C23 | 0.31445 (17)  | 0.01556 (13)  | 0.40911 (8) | 0.0211 (3) |
| C24 | 0.38163 (19)  | 0.10284 (14)  | 0.33679 (8) | 0.0246 (3) |
| H24 | 0.4041        | 0.1927        | 0.3385      | 0.030*     |
| C25 | 0.4152 (2)    | 0.05881 (15)  | 0.26338 (9) | 0.0288 (3) |
| H25 | 0.4604        | 0.1183        | 0.2148      | 0.035*     |
| C26 | 0.3824 (2)    | -0.07406 (16) | 0.26052 (9) | 0.0297 (3) |
| H26 | 0.4060        | -0.1041       | 0.2099      | 0.036*     |
| C27 | 0.31638 (19)  | -0.16072 (15) | 0.33042 (9) | 0.0279 (3) |
| H27 | 0.2949        | -0.2503       | 0.3277      | 0.033*     |
| C28 | 0.28016 (18)  | -0.11821 (13) | 0.40624 (8) | 0.0230 (3) |
| C29 | 0.20946 (19)  | -0.20770 (13) | 0.47966 (9) | 0.0256 (3) |
| H29 | 0.1870        | -0.2972       | 0.4772      | 0.031*     |
| C30 | 0.17442 (18)  | -0.16655 (13) | 0.55216 (9) | 0.0243 (3) |
| H30 | 0.1267        | -0.2272       | 0.5998      | 0.029*     |
| C31 | 0.20836 (17)  | -0.03237 (13) | 0.55800 (8) | 0.0209 (2) |
| C32 | 0.17548 (17)  | 0.01381 (13)  | 0.63202 (8) | 0.0208 (2) |
| H32 | 0.1292        | -0.0454       | 0.6806      | 0.025*     |

Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$      | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|---------------|---------------|
| C11 | 0.03021 (17) | 0.01941 (15) | 0.02004 (15) | -0.00805 (11) | -0.00196 (11) | -0.00167 (11) |
| O1  | 0.0475 (6)   | 0.0300 (5)   | 0.0246 (5)   | -0.0175 (5)   | 0.0085 (4)    | -0.0029 (4)   |
| N1  | 0.0274 (6)   | 0.0271 (6)   | 0.0169 (5)   | -0.0041 (4)   | -0.0017 (4)   | 0.0003 (4)    |
| N2  | 0.0178 (5)   | 0.0207 (5)   | 0.0196 (5)   | -0.0021 (4)   | -0.0038 (4)   | -0.0022 (4)   |
| C1  | 0.0258 (6)   | 0.0256 (6)   | 0.0150 (6)   | -0.0046 (5)   | 0.0015 (5)    | -0.0015 (5)   |
| C2  | 0.0380 (8)   | 0.0324 (7)   | 0.0184 (6)   | -0.0089 (6)   | -0.0022 (5)   | -0.0045 (5)   |
| C3  | 0.0529 (10)  | 0.0289 (7)   | 0.0241 (7)   | -0.0119 (7)   | -0.0008 (6)   | -0.0091 (6)   |
| C4  | 0.0515 (10)  | 0.0225 (7)   | 0.0276 (7)   | 0.0001 (6)    | -0.0015 (6)   | -0.0050 (5)   |
| C5  | 0.0343 (7)   | 0.0245 (7)   | 0.0234 (6)   | 0.0005 (5)    | -0.0012 (5)   | -0.0028 (5)   |
| C6  | 0.0232 (6)   | 0.0226 (6)   | 0.0160 (6)   | -0.0029 (5)   | 0.0032 (5)    | -0.0010 (5)   |
| C7  | 0.0196 (6)   | 0.0223 (6)   | 0.0162 (5)   | -0.0027 (5)   | 0.0026 (4)    | -0.0004 (4)   |
| C8  | 0.0215 (6)   | 0.0222 (6)   | 0.0160 (6)   | -0.0038 (5)   | 0.0024 (4)    | -0.0005 (4)   |

|     |            |            |            |             |             |             |
|-----|------------|------------|------------|-------------|-------------|-------------|
| C9  | 0.0239 (6) | 0.0232 (6) | 0.0170 (6) | -0.0031 (5) | -0.0001 (5) | 0.0021 (5)  |
| C10 | 0.0349 (8) | 0.0256 (7) | 0.0288 (7) | -0.0013 (6) | -0.0074 (6) | 0.0041 (5)  |
| C11 | 0.0222 (6) | 0.0186 (6) | 0.0209 (6) | -0.0011 (5) | -0.0019 (5) | -0.0019 (5) |
| C12 | 0.0224 (6) | 0.0242 (6) | 0.0237 (6) | -0.0028 (5) | -0.0021 (5) | 0.0025 (5)  |
| C13 | 0.0299 (7) | 0.0252 (6) | 0.0215 (6) | -0.0033 (5) | -0.0048 (5) | 0.0017 (5)  |
| C14 | 0.0275 (7) | 0.0239 (6) | 0.0281 (7) | 0.0002 (5)  | -0.0097 (5) | -0.0058 (5) |
| C15 | 0.0211 (6) | 0.0328 (7) | 0.0296 (7) | -0.0053 (5) | -0.0012 (5) | -0.0099 (6) |
| C16 | 0.0240 (7) | 0.0290 (7) | 0.0211 (6) | -0.0045 (5) | 0.0003 (5)  | -0.0047 (5) |
| C17 | 0.0239 (6) | 0.0205 (6) | 0.0200 (6) | -0.0029 (5) | -0.0009 (5) | 0.0014 (5)  |
| C18 | 0.0217 (6) | 0.0216 (6) | 0.0212 (6) | -0.0039 (5) | -0.0039 (5) | -0.0010 (5) |
| C19 | 0.0204 (6) | 0.0198 (6) | 0.0199 (6) | -0.0023 (5) | -0.0051 (5) | -0.0003 (5) |
| C20 | 0.0152 (6) | 0.0227 (6) | 0.0200 (6) | -0.0024 (4) | -0.0032 (4) | -0.0021 (5) |
| C21 | 0.0165 (6) | 0.0192 (6) | 0.0212 (6) | -0.0031 (4) | -0.0041 (4) | -0.0012 (5) |
| C22 | 0.0141 (5) | 0.0215 (6) | 0.0214 (6) | -0.0013 (4) | -0.0039 (4) | -0.0027 (5) |
| C23 | 0.0153 (6) | 0.0240 (6) | 0.0250 (6) | 0.0002 (5)  | -0.0049 (5) | -0.0059 (5) |
| C24 | 0.0230 (6) | 0.0274 (6) | 0.0238 (6) | -0.0003 (5) | -0.0046 (5) | -0.0055 (5) |
| C25 | 0.0259 (7) | 0.0367 (8) | 0.0244 (7) | -0.0003 (6) | -0.0047 (5) | -0.0073 (6) |
| C26 | 0.0239 (7) | 0.0413 (8) | 0.0276 (7) | 0.0011 (6)  | -0.0055 (5) | -0.0157 (6) |
| C27 | 0.0214 (6) | 0.0316 (7) | 0.0350 (7) | 0.0007 (5)  | -0.0081 (5) | -0.0150 (6) |
| C28 | 0.0153 (6) | 0.0261 (6) | 0.0293 (7) | 0.0001 (5)  | -0.0053 (5) | -0.0086 (5) |
| C29 | 0.0201 (6) | 0.0218 (6) | 0.0363 (7) | -0.0024 (5) | -0.0054 (5) | -0.0073 (5) |
| C30 | 0.0205 (6) | 0.0210 (6) | 0.0304 (7) | -0.0037 (5) | -0.0025 (5) | -0.0023 (5) |
| C31 | 0.0162 (6) | 0.0215 (6) | 0.0246 (6) | -0.0014 (5) | -0.0039 (5) | -0.0029 (5) |
| C32 | 0.0179 (6) | 0.0207 (6) | 0.0217 (6) | -0.0038 (5) | -0.0014 (5) | 0.0007 (5)  |

*Geometric parameters (Å, °)*

|         |             |         |             |
|---------|-------------|---------|-------------|
| C11—C21 | 1.7526 (12) | C14—C15 | 1.383 (2)   |
| O1—C17  | 1.2219 (16) | C14—H14 | 0.9500      |
| N1—C9   | 1.3149 (17) | C15—C16 | 1.3976 (19) |
| N1—C1   | 1.3703 (17) | C15—H15 | 0.9500      |
| N2—C21  | 1.3019 (16) | C16—H16 | 0.9500      |
| N2—C22  | 1.3585 (16) | C17—C18 | 1.4643 (18) |
| C1—C2   | 1.4187 (19) | C18—C19 | 1.3351 (18) |
| C1—C6   | 1.4200 (18) | C18—H18 | 0.9500      |
| C2—C3   | 1.366 (2)   | C19—C20 | 1.4671 (17) |
| C2—H2   | 0.9500      | C19—H19 | 0.9500      |
| C3—C4   | 1.410 (2)   | C20—C32 | 1.3923 (17) |
| C3—H3   | 0.9500      | C20—C21 | 1.4130 (17) |
| C4—C5   | 1.372 (2)   | C22—C31 | 1.4127 (18) |
| C4—H4   | 0.9500      | C22—C23 | 1.4477 (17) |
| C5—C6   | 1.4180 (18) | C23—C24 | 1.4067 (19) |
| C5—H5   | 0.9500      | C23—C28 | 1.4176 (18) |
| C6—C7   | 1.4257 (18) | C24—C25 | 1.3801 (19) |
| C7—C8   | 1.3788 (18) | C24—H24 | 0.9500      |
| C7—C11  | 1.4895 (18) | C25—C26 | 1.406 (2)   |
| C8—C9   | 1.4328 (18) | C25—H25 | 0.9500      |
| C8—C17  | 1.5075 (17) | C26—C27 | 1.373 (2)   |

|               |             |             |             |
|---------------|-------------|-------------|-------------|
| C9—C10        | 1.5067 (18) | C26—H26     | 0.9500      |
| C10—H10A      | 0.9800      | C27—C28     | 1.4108 (19) |
| C10—H10B      | 0.9800      | C27—H27     | 0.9500      |
| C10—H10C      | 0.9800      | C28—C29     | 1.437 (2)   |
| C11—C16       | 1.3905 (18) | C29—C30     | 1.351 (2)   |
| C11—C12       | 1.3940 (18) | C29—H29     | 0.9500      |
| C12—C13       | 1.3877 (19) | C30—C31     | 1.4347 (17) |
| C12—H12       | 0.9500      | C30—H30     | 0.9500      |
| C13—C14       | 1.389 (2)   | C31—C32     | 1.4005 (18) |
| C13—H13       | 0.9500      | C32—H32     | 0.9500      |
|               |             |             |             |
| C9—N1—C1      | 118.65 (12) | C11—C16—H16 | 120.1       |
| C21—N2—C22    | 117.81 (11) | C15—C16—H16 | 120.1       |
| N1—C1—C2      | 117.88 (12) | O1—C17—C18  | 118.97 (12) |
| N1—C1—C6      | 122.75 (12) | O1—C17—C8   | 119.05 (12) |
| C2—C1—C6      | 119.37 (12) | C18—C17—C8  | 121.98 (11) |
| C3—C2—C1      | 120.19 (14) | C19—C18—C17 | 126.68 (12) |
| C3—C2—H2      | 119.9       | C19—C18—H18 | 116.7       |
| C1—C2—H2      | 119.9       | C17—C18—H18 | 116.7       |
| C2—C3—C4      | 120.63 (13) | C18—C19—C20 | 122.60 (11) |
| C2—C3—H3      | 119.7       | C18—C19—H19 | 118.7       |
| C4—C3—H3      | 119.7       | C20—C19—H19 | 118.7       |
| C5—C4—C3      | 120.53 (14) | C32—C20—C21 | 114.63 (11) |
| C5—C4—H4      | 119.7       | C32—C20—C19 | 122.08 (11) |
| C3—C4—H4      | 119.7       | C21—C20—C19 | 123.29 (11) |
| C4—C5—C6      | 120.27 (14) | N2—C21—C20  | 126.73 (11) |
| C4—C5—H5      | 119.9       | N2—C21—C11  | 114.54 (9)  |
| C6—C5—H5      | 119.9       | C20—C21—C11 | 118.72 (9)  |
| C5—C6—C1      | 118.95 (12) | N2—C22—C31  | 121.81 (11) |
| C5—C6—C7      | 123.36 (12) | N2—C22—C23  | 118.37 (11) |
| C1—C6—C7      | 117.66 (12) | C31—C22—C23 | 119.83 (11) |
| C8—C7—C6      | 118.52 (12) | C24—C23—C28 | 119.61 (12) |
| C8—C7—C11     | 122.21 (11) | C24—C23—C22 | 121.87 (12) |
| C6—C7—C11     | 119.26 (11) | C28—C23—C22 | 118.53 (12) |
| C7—C8—C9      | 119.66 (12) | C25—C24—C23 | 120.44 (13) |
| C7—C8—C17     | 121.10 (12) | C25—C24—H24 | 119.8       |
| C9—C8—C17     | 119.07 (11) | C23—C24—H24 | 119.8       |
| N1—C9—C8      | 122.61 (12) | C24—C25—C26 | 120.01 (14) |
| N1—C9—C10     | 116.89 (12) | C24—C25—H25 | 120.0       |
| C8—C9—C10     | 120.48 (12) | C26—C25—H25 | 120.0       |
| C9—C10—H10A   | 109.5       | C27—C26—C25 | 120.43 (13) |
| C9—C10—H10B   | 109.5       | C27—C26—H26 | 119.8       |
| H10A—C10—H10B | 109.5       | C25—C26—H26 | 119.8       |
| C9—C10—H10C   | 109.5       | C26—C27—C28 | 120.74 (13) |
| H10A—C10—H10C | 109.5       | C26—C27—H27 | 119.6       |
| H10B—C10—H10C | 109.5       | C28—C27—H27 | 119.6       |
| C16—C11—C12   | 119.22 (12) | C27—C28—C23 | 118.77 (13) |
| C16—C11—C7    | 121.74 (11) | C27—C28—C29 | 121.20 (12) |



|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C12—C11—C7      | 119.03 (11)  | C23—C28—C29     | 120.03 (12)  |
| C13—C12—C11     | 120.78 (12)  | C30—C29—C28     | 121.05 (12)  |
| C13—C12—H12     | 119.6        | C30—C29—H29     | 119.5        |
| C11—C12—H12     | 119.6        | C28—C29—H29     | 119.5        |
| C12—C13—C14     | 119.79 (13)  | C29—C30—C31     | 120.81 (13)  |
| C12—C13—H13     | 120.1        | C29—C30—H30     | 119.6        |
| C14—C13—H13     | 120.1        | C31—C30—H30     | 119.6        |
| C15—C14—C13     | 119.88 (13)  | C32—C31—C22     | 117.68 (11)  |
| C15—C14—H14     | 120.1        | C32—C31—C30     | 122.58 (12)  |
| C13—C14—H14     | 120.1        | C22—C31—C30     | 119.74 (12)  |
| C14—C15—C16     | 120.41 (12)  | C20—C32—C31     | 121.34 (12)  |
| C14—C15—H15     | 119.8        | C20—C32—H32     | 119.3        |
| C16—C15—H15     | 119.8        | C31—C32—H32     | 119.3        |
| C11—C16—C15     | 119.90 (12)  |                 |              |
|                 |              |                 |              |
| C9—N1—C1—C2     | 178.03 (12)  | C9—C8—C17—C18   | -109.35 (14) |
| C9—N1—C1—C6     | -2.71 (18)   | O1—C17—C18—C19  | -174.33 (14) |
| N1—C1—C2—C3     | -178.22 (12) | C8—C17—C18—C19  | 4.6 (2)      |
| C6—C1—C2—C3     | 2.5 (2)      | C17—C18—C19—C20 | 178.53 (12)  |
| C1—C2—C3—C4     | -0.3 (2)     | C18—C19—C20—C32 | -22.79 (19)  |
| C2—C3—C4—C5     | -1.8 (2)     | C18—C19—C20—C21 | 156.45 (12)  |
| C3—C4—C5—C6     | 1.5 (2)      | C22—N2—C21—C20  | -0.07 (19)   |
| C4—C5—C6—C1     | 0.7 (2)      | C22—N2—C21—C11  | -179.46 (9)  |
| C4—C5—C6—C7     | 178.69 (13)  | C32—C20—C21—N2  | 0.69 (19)    |
| N1—C1—C6—C5     | 178.07 (12)  | C19—C20—C21—N2  | -178.60 (11) |
| C2—C1—C6—C5     | -2.69 (18)   | C32—C20—C21—C11 | -179.95 (9)  |
| N1—C1—C6—C7     | -0.04 (18)   | C19—C20—C21—C11 | 0.76 (16)    |
| C2—C1—C6—C7     | 179.20 (12)  | C21—N2—C22—C31  | -0.37 (17)   |
| C5—C6—C7—C8     | -174.86 (12) | C21—N2—C22—C23  | 179.59 (11)  |
| C1—C6—C7—C8     | 3.16 (17)    | N2—C22—C23—C24  | -0.97 (18)   |
| C5—C6—C7—C11    | 5.43 (19)    | C31—C22—C23—C24 | 179.00 (11)  |
| C1—C6—C7—C11    | -176.55 (11) | N2—C22—C23—C28  | 179.08 (11)  |
| C6—C7—C8—C9     | -3.58 (18)   | C31—C22—C23—C28 | -0.95 (17)   |
| C11—C7—C8—C9    | 176.12 (11)  | C28—C23—C24—C25 | -0.35 (19)   |
| C6—C7—C8—C17    | 171.82 (11)  | C22—C23—C24—C25 | 179.70 (12)  |
| C11—C7—C8—C17   | -8.49 (18)   | C23—C24—C25—C26 | -0.1 (2)     |
| C1—N1—C9—C8     | 2.33 (19)    | C24—C25—C26—C27 | 0.2 (2)      |
| C1—N1—C9—C10    | -179.25 (11) | C25—C26—C27—C28 | 0.1 (2)      |
| C7—C8—C9—N1     | 0.84 (19)    | C26—C27—C28—C23 | -0.46 (19)   |
| C17—C8—C9—N1    | -174.65 (11) | C26—C27—C28—C29 | 179.43 (12)  |
| C7—C8—C9—C10    | -177.52 (12) | C24—C23—C28—C27 | 0.59 (18)    |
| C17—C8—C9—C10   | 6.99 (18)    | C22—C23—C28—C27 | -179.45 (11) |
| C8—C7—C11—C16   | 73.14 (17)   | C24—C23—C28—C29 | -179.29 (12) |
| C6—C7—C11—C16   | -107.17 (14) | C22—C23—C28—C29 | 0.66 (18)    |
| C8—C7—C11—C12   | -108.44 (14) | C27—C28—C29—C30 | -179.82 (12) |
| C6—C7—C11—C12   | 71.26 (16)   | C23—C28—C29—C30 | 0.07 (19)    |
| C16—C11—C12—C13 | 0.4 (2)      | C28—C29—C30—C31 | -0.5 (2)     |
| C7—C11—C12—C13  | -178.03 (12) | N2—C22—C31—C32  | 0.16 (18)    |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C11—C12—C13—C14 | 1.2 (2)      | C23—C22—C31—C32 | -179.81 (11) |
| C12—C13—C14—C15 | -1.6 (2)     | N2—C22—C31—C30  | -179.50 (11) |
| C13—C14—C15—C16 | 0.4 (2)      | C23—C22—C31—C30 | 0.53 (18)    |
| C12—C11—C16—C15 | -1.64 (19)   | C29—C30—C31—C32 | -179.43 (12) |
| C7—C11—C16—C15  | 176.78 (12)  | C29—C30—C31—C22 | 0.21 (19)    |
| C14—C15—C16—C11 | 1.3 (2)      | C21—C20—C32—C31 | -0.87 (18)   |
| C7—C8—C17—O1    | -105.83 (15) | C19—C20—C32—C31 | 178.43 (11)  |
| C9—C8—C17—O1    | 69.59 (17)   | C22—C31—C32—C20 | 0.50 (18)    |
| C7—C8—C17—C18   | 75.23 (16)   | C30—C31—C32—C20 | -179.85 (12) |

*Hydrogen-bond geometry (Å, °)*

Cg1 is the centroid of the N1-pyridyl ring.

| <i>D</i> —H... <i>A</i>     | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| C10—H10C...O1 <sup>i</sup>  | 0.98        | 2.54          | 3.4116 (18)           | 148                     |
| C15—H15...Cg1 <sup>ii</sup> | 0.95        | 2.65          | 3.4695 (15)           | 145                     |

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