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# 4-Allyl-6-bromo-2-phenyl-4*H*-imidazo-[4,5-*b*]pyridine monohydrate

**Younès Ouzidan,[a] Youssef Kandri Rodi,[a] Hafid Zouihri,[b] El Mokhtar Essassi[c] and Seik Weng Ng[d]***

[a]Laboratoire de Chimie Organique Appliquée, Faculté des Sciences et Techniques, Université Sidi Mohamed Ben Abdallah, Fès, Morocco, [b]CNRST Division UATRS, Angle Allal Fassi/FAR, BP 8027 Hay Riad, Rabat, Morocco, [c]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, and [d]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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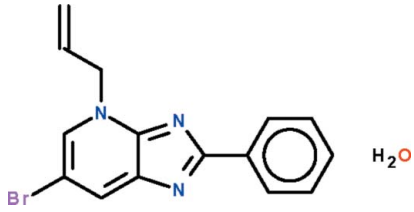
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; disorder in solvent or counterion; $R$ factor = 0.027; $wR$ factor = 0.078; data-to-parameter ratio = 15.6.

In the molecule of the title compound, $C_{15}H_{12}BrN_3\cdot H_2O$, the phenyl ring is coplanar with the imidazopyridine ring system [dihedral angle = 0.4 (1)°]. The water molecule is disordered over two positions with occupancies of 0.58 (1) and 0.42 (1), and it is linked to the main molecule *via* an O—H···N hydrogen bond.

## Related literature

For a related structure, see: Ouzidan *et al.* (2010).




## Experimental

### Crystal data

$C_{15}H_{12}BrN_3\cdot H_2O$ $M_r$ = 332.20
Triclinic, $P\overline{1}$
$a$ = 7.4363 (1) Å
$b$ = 9.4238 (1) Å
$c$ = 11.0829 (2) Å
$\alpha$ = 68.076 (1)°
$\beta$ = 74.637 (1)°
$\gamma$ = 79.736 (1)°
$V$ = 692.02 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 2.97 mm$^{-1}$
$T$ = 293 K
0.20 × 0.20 × 0.15 mm

### Data collection

Bruker X8 APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.588, $T_{max}$ = 0.664
14314 measured reflections
3158 independent reflections
2791 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.078
$S$ = 0.98
3158 reflections
203 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H11···N3 | 0.83 (1) | 2.14 (3) | 2.887 (4) | 149 (5) |

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); 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: CI5117).

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# supporting information

*Acta Cryst.* (2010). E**66**, o1903 [https://doi.org/10.1107/S1600536810025122]

## 4-Allyl-6-bromo-2-phenyl-4*H*-imidazo[4,5-*b*]pyridine monohydrate

**Younès Ouzidan, Youssef Kandri Rodi, Hafid Zouihri, El Mokhtar Essassi and Seik Weng Ng**

### S1. Comment

The imidazo[4,5-*b*]pyridine unit is an important heterocyclic nucleus found in a large number of molecules in medicinal chemistry. Heterocycles derived from such compounds posess useful medicinal properties. Owing to their importance, strategies have been developed for their synthesis. The most popular synthetic approach involves the cyclocondensation of 2,3-pyridinediamine with carboxylic acid derivatives or on condensation with aldehydes. An earlier study reported the crystal structure of 4-benzyl-6-bromo-2-phenyl-4*H*-imidazo[4,5-*b*]pyridine (Ouzidan *et al.*, 2010), which was synthesized by using a much more convenient route. The synthesis is extended to the title compound (Scheme I and Fig. 1).

The imidazopyridine ring system is coplanar with the phenyl ring at the 2-position of the five-membered ring [dihedral angle = 0.4 (1) °].

### S2. Experimental

To a solution 6-bromo-2-phenyl-1*H*-imidazo[4,5-*b*]pyridine (0.3 g, 1.09 mmol), was added a DMF (15 ml) solution of potassium carbonate (0.2 g, 1.42 mmol), tetra-*n*-butylammonium bromide (0.04 g, 0.1 mmol) and allyl bromide (0.11 ml, 1.31 mmol). Stirring was continued at room temperature for 12 h. The mixture was filtered and the solvent removed under reduced pressure. The residue was separated by chromatography on a column of silica gel with ethyl acetate-hexane (2:3) as eluent. Yellow crystals were isolated when the solvent was allowed to evaporate.

### S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H = 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with $U$(H) set to 1.2$U_{eq}$(C). The water molecule is disordered over two positions in a 58 (1):42 (1) ratio. The H atoms were located in a difference Fourier map and were refined with distance restraints of O–H = 0.84 (1) Å and H···H 1.37 (1) Å; their $U_{iso}$ values were tied to those of the oxygen atoms by a factor of 1.5.

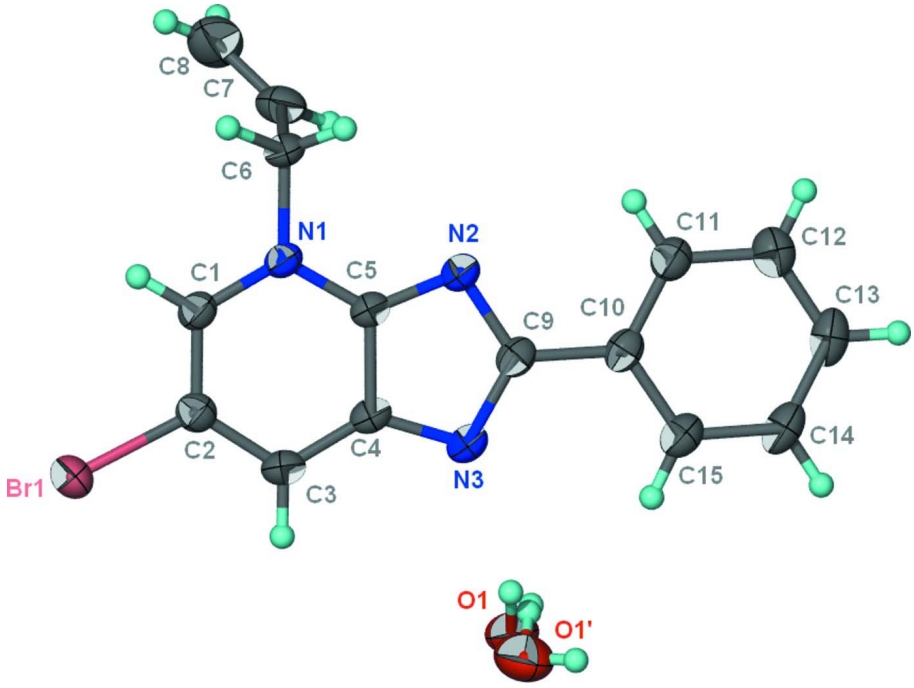


**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of the molecule of $C_{15}H_{12}BrN_3{\cdot}H_2O$ at the 50% probability level. H atoms are shown as spheres of arbitrary radii. The disorder in the water molecule is shown.

### 4-Allyl-6-bromo-2-phenyl-4*H*-imidazo[4,5-*b*]pyridine monohydrate

*Crystal data*

$C_{15}H_{12}BrN_3{\cdot}H_2O$
$M_r = 332.20$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.4363\ (1)$ Å
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$c = 11.0829\ (2)$ Å
$\alpha = 68.076\ (1)°$
$\beta = 74.637\ (1)°$
$\gamma = 79.736\ (1)°$
$V = 692.02\ (2)$ Å$^3$
$Z = 2$
$F(000) = 336$
$D_x = 1.594$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7204 reflections
$\theta = 2.3–27.2°$
$\mu = 2.97$ mm$^{-1}$
$T = 293$ K
Prism, yellow
0.20 × 0.20 × 0.15 mm

*Data collection*

Bruker X8 APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.588$, $T_{max} = 0.664$
14314 measured reflections
3158 independent reflections
2791 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.5°$
$h = -9\rightarrow9$
$k = -12\rightarrow12$
$l = -13\rightarrow14$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.078$
$S = 0.98$
3158 reflections
203 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.1091P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.39$ e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Br1 | 0.78351 (3) | 0.73753 (2) | 0.425170 (19) | 0.05007 (9) | |
| O1 | 1.1389 (9) | 0.1942 (6) | 0.8855 (4) | 0.0635 (16) | 0.582 (14) |
| H11 | 1.028 (3) | 0.229 (7) | 0.903 (7) | 0.095* | 0.582 (14) |
| H12 | 1.208 (6) | 0.237 (6) | 0.908 (6) | 0.095* | 0.582 (14) |
| O1′ | 1.0501 (14) | 0.1260 (12) | 0.8987 (6) | 0.081 (3) | 0.418 (14) |
| H13 | 1.089 (17) | 0.191 (10) | 0.917 (11) | 0.122* | 0.418 (14) |
| H14 | 1.001 (16) | 0.059 (9) | 0.969 (6) | 0.122* | 0.418 (14) |
| N1 | 0.61186 (19) | 0.78390 (15) | 0.79149 (15) | 0.0323 (3) | |
| N2 | 0.66816 (19) | 0.61173 (15) | 1.00435 (15) | 0.0347 (3) | |
| N3 | 0.82874 (19) | 0.41411 (16) | 0.93153 (16) | 0.0358 (3) | |
| C1 | 0.6432 (2) | 0.80592 (19) | 0.66074 (18) | 0.0350 (3) | |
| H1 | 0.5976 | 0.8977 | 0.6035 | 0.042* | |
| C2 | 0.7415 (2) | 0.6947 (2) | 0.61055 (19) | 0.0368 (4) | |
| C3 | 0.8123 (2) | 0.55420 (19) | 0.69201 (19) | 0.0371 (4) | |
| H3 | 0.8780 | 0.4791 | 0.6576 | 0.044* | |
| C4 | 0.7798 (2) | 0.53257 (18) | 0.82533 (18) | 0.0331 (3) | |
| C5 | 0.6797 (2) | 0.65118 (18) | 0.87503 (17) | 0.0313 (3) | |
| C6 | 0.5091 (2) | 0.90590 (19) | 0.84527 (19) | 0.0381 (4) | |
| H6A | 0.4210 | 0.9672 | 0.7908 | 0.046* | |
| H6B | 0.4386 | 0.8587 | 0.9352 | 0.046* | |
| C7 | 0.6408 (3) | 1.0068 (2) | 0.8464 (2) | 0.0481 (5) | |
| H7 | 0.7367 | 0.9607 | 0.8917 | 0.058* | |
| C8 | 0.6310 (5) | 1.1541 (3) | 0.7888 (3) | 0.0711 (7) | |
| H8A | 0.5368 | 1.2037 | 0.7427 | 0.085* | |
| H8B | 0.7180 | 1.2105 | 0.7933 | 0.085* | |
| C9 | 0.7604 (2) | 0.46733 (18) | 1.03315 (18) | 0.0339 (3) | |
| C10 | 0.7799 (2) | 0.37743 (19) | 1.17031 (18) | 0.0354 (4) | |
| C11 | 0.7034 (3) | 0.4364 (2) | 1.2724 (2) | 0.0422 (4) | |
| H11A | 0.6407 | 0.5342 | 1.2535 | 0.051* | |
| C12 | 0.7194 (3) | 0.3511 (3) | 1.4024 (2) | 0.0498 (5) | |
| H12A | 0.6678 | 0.3919 | 1.4699 | 0.060* | |
| C13 | 0.8118 (3) | 0.2058 (3) | 1.4313 (2) | 0.0522 (5) | |
| H13A | 0.8219 | 0.1481 | 1.5184 | 0.063* | |

| | | | | |
|---|---|---|---|---|
| C14 | 0.8890 (3) | 0.1467 (2) | 1.3309 (2) | 0.0509 (5) |
| H14A | 0.9520 | 0.0490 | 1.3505 | 0.061* |
| C15 | 0.8741 (3) | 0.2306 (2) | 1.2012 (2) | 0.0436 (4) |
| H15 | 0.9269 | 0.1892 | 1.1342 | 0.052* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.06412 (15) | 0.05036 (14) | 0.03761 (13) | 0.00253 (9) | −0.01310 (9) | −0.01926 (9) |
| O1 | 0.076 (3) | 0.056 (2) | 0.068 (2) | 0.020 (2) | −0.0289 (19) | −0.0341 (17) |
| O1′ | 0.091 (5) | 0.084 (5) | 0.074 (3) | 0.022 (4) | −0.021 (3) | −0.044 (3) |
| N1 | 0.0350 (7) | 0.0260 (6) | 0.0359 (8) | 0.0023 (5) | −0.0108 (6) | −0.0108 (5) |
| N2 | 0.0360 (7) | 0.0294 (7) | 0.0370 (8) | 0.0013 (5) | −0.0094 (6) | −0.0104 (6) |
| N3 | 0.0352 (7) | 0.0288 (7) | 0.0422 (8) | 0.0013 (5) | −0.0112 (6) | −0.0107 (6) |
| C1 | 0.0373 (8) | 0.0307 (8) | 0.0371 (9) | 0.0006 (6) | −0.0122 (7) | −0.0106 (7) |
| C2 | 0.0394 (8) | 0.0368 (9) | 0.0373 (9) | −0.0032 (7) | −0.0104 (7) | −0.0148 (7) |
| C3 | 0.0382 (8) | 0.0321 (8) | 0.0435 (10) | 0.0008 (6) | −0.0084 (7) | −0.0181 (7) |
| C4 | 0.0307 (7) | 0.0276 (7) | 0.0418 (9) | 0.0004 (6) | −0.0095 (7) | −0.0129 (7) |
| C5 | 0.0302 (7) | 0.0274 (7) | 0.0368 (9) | −0.0010 (6) | −0.0083 (6) | −0.0116 (6) |
| C6 | 0.0422 (9) | 0.0305 (8) | 0.0394 (9) | 0.0086 (7) | −0.0097 (7) | −0.0143 (7) |
| C7 | 0.0513 (11) | 0.0487 (11) | 0.0530 (12) | 0.0017 (8) | −0.0112 (9) | −0.0303 (9) |
| C8 | 0.101 (2) | 0.0508 (13) | 0.0645 (16) | −0.0212 (13) | −0.0088 (14) | −0.0227 (12) |
| C9 | 0.0293 (7) | 0.0299 (8) | 0.0402 (9) | −0.0027 (6) | −0.0087 (6) | −0.0085 (7) |
| C10 | 0.0318 (8) | 0.0307 (8) | 0.0407 (9) | −0.0048 (6) | −0.0107 (7) | −0.0058 (7) |
| C11 | 0.0443 (9) | 0.0376 (9) | 0.0419 (10) | −0.0011 (7) | −0.0118 (8) | −0.0101 (7) |
| C12 | 0.0521 (11) | 0.0543 (12) | 0.0419 (11) | −0.0061 (9) | −0.0123 (9) | −0.0132 (9) |
| C13 | 0.0524 (11) | 0.0521 (12) | 0.0427 (11) | −0.0107 (9) | −0.0178 (9) | 0.0021 (9) |
| C14 | 0.0495 (11) | 0.0370 (10) | 0.0561 (13) | −0.0005 (8) | −0.0199 (9) | −0.0006 (9) |
| C15 | 0.0432 (9) | 0.0348 (9) | 0.0482 (11) | 0.0000 (7) | −0.0120 (8) | −0.0093 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C2 | 1.8887 (19) | C6—C7 | 1.487 (3) |
| O1—H11 | 0.834 (10) | C6—H6A | 0.97 |
| O1—H12 | 0.838 (10) | C6—H6B | 0.97 |
| O1—H13 | 0.43 (12) | C7—C8 | 1.291 (3) |
| O1′—H11 | 0.97 (5) | C7—H7 | 0.93 |
| O1′—H13 | 0.836 (10) | C8—H8A | 0.93 |
| O1′—H14 | 0.838 (10) | C8—H8B | 0.93 |
| N1—C1 | 1.346 (2) | C9—C10 | 1.470 (3) |
| N1—C5 | 1.354 (2) | C10—C11 | 1.389 (3) |
| N1—C6 | 1.489 (2) | C10—C15 | 1.396 (2) |
| N2—C5 | 1.322 (2) | C11—C12 | 1.388 (3) |
| N2—C9 | 1.372 (2) | C11—H11A | 0.93 |
| N3—C9 | 1.344 (2) | C12—C13 | 1.379 (3) |
| N3—C4 | 1.365 (2) | C12—H12A | 0.93 |
| C1—C2 | 1.375 (2) | C13—C14 | 1.374 (3) |
| C1—H1 | 0.93 | C13—H13A | 0.93 |

| | | | |
|---|---|---|---|
| C2—C3 | 1.398 (2) | C14—C15 | 1.381 (3) |
| C3—C4 | 1.374 (3) | C14—H14A | 0.93 |
| C3—H3 | 0.93 | C15—H15 | 0.93 |
| C4—C5 | 1.433 (2) | | |

| | | | |
|---|---|---|---|
| H11—O1—H12 | 110.6 (18) | H6A—C6—H6B | 108.0 |
| H12—O1—H13 | 101 (6) | C8—C7—C6 | 124.3 (2) |
| H11—O1′—H14 | 114 (9) | C8—C7—H7 | 117.8 |
| H13—O1′—H14 | 110.2 (19) | C6—C7—H7 | 117.8 |
| C1—N1—C5 | 119.78 (14) | C7—C8—H8A | 120.0 |
| C1—N1—C6 | 120.89 (14) | C7—C8—H8B | 120.0 |
| C5—N1—C6 | 119.29 (14) | H8A—C8—H8B | 120.0 |
| C5—N2—C9 | 101.46 (14) | N3—C9—N2 | 117.02 (16) |
| C9—N3—C4 | 103.09 (13) | N3—C9—C10 | 122.81 (15) |
| N1—C1—C2 | 120.95 (16) | N2—C9—C10 | 120.17 (16) |
| N1—C1—H1 | 119.5 | C11—C10—C15 | 118.49 (18) |
| C2—C1—H1 | 119.5 | C11—C10—C9 | 120.61 (16) |
| C1—C2—C3 | 122.00 (17) | C15—C10—C9 | 120.90 (17) |
| C1—C2—Br1 | 117.99 (14) | C12—C11—C10 | 120.79 (18) |
| C3—C2—Br1 | 120.01 (13) | C12—C11—H11A | 119.6 |
| C4—C3—C2 | 116.73 (15) | C10—C11—H11A | 119.6 |
| C4—C3—H3 | 121.6 | C13—C12—C11 | 120.0 (2) |
| C2—C3—H3 | 121.6 | C13—C12—H12A | 120.0 |
| N3—C4—C3 | 132.84 (15) | C11—C12—H12A | 120.0 |
| N3—C4—C5 | 106.85 (15) | C14—C13—C12 | 119.7 (2) |
| C3—C4—C5 | 120.29 (15) | C14—C13—H13A | 120.1 |
| N2—C5—N1 | 128.19 (15) | C12—C13—H13A | 120.1 |
| N2—C5—C4 | 111.57 (14) | C13—C14—C15 | 120.79 (19) |
| N1—C5—C4 | 120.24 (16) | C13—C14—H14A | 119.6 |
| C7—C6—N1 | 110.95 (14) | C15—C14—H14A | 119.6 |
| C7—C6—H6A | 109.4 | C14—C15—C10 | 120.2 (2) |
| N1—C6—H6A | 109.4 | C14—C15—H15 | 119.9 |
| C7—C6—H6B | 109.4 | C10—C15—H15 | 119.9 |
| N1—C6—H6B | 109.4 | | |

| | | | |
|---|---|---|---|
| C5—N1—C1—C2 | 0.8 (2) | C1—N1—C6—C7 | −91.40 (19) |
| C6—N1—C1—C2 | 178.59 (16) | C5—N1—C6—C7 | 86.45 (19) |
| N1—C1—C2—C3 | 0.4 (3) | N1—C6—C7—C8 | 124.1 (2) |
| N1—C1—C2—Br1 | −178.60 (12) | C4—N3—C9—N2 | 0.46 (19) |
| C1—C2—C3—C4 | −0.4 (3) | C4—N3—C9—C10 | 179.96 (15) |
| Br1—C2—C3—C4 | 178.57 (12) | C5—N2—C9—N3 | 0.11 (19) |
| C9—N3—C4—C3 | 177.66 (18) | C5—N2—C9—C10 | −179.41 (14) |
| C9—N3—C4—C5 | −0.79 (17) | N3—C9—C10—C11 | −178.67 (16) |
| C2—C3—C4—N3 | −178.94 (17) | N2—C9—C10—C11 | 0.8 (2) |
| C2—C3—C4—C5 | −0.7 (2) | N3—C9—C10—C15 | 0.8 (2) |
| C9—N2—C5—N1 | 179.89 (16) | N2—C9—C10—C15 | −179.71 (15) |
| C9—N2—C5—C4 | −0.63 (17) | C15—C10—C11—C12 | −0.3 (3) |
| C1—N1—C5—N2 | 177.63 (16) | C9—C10—C11—C12 | 179.18 (17) |

| | | | |
|---|---|---|---|
| C6—N1—C5—N2 | −0.2 (3) | C10—C11—C12—C13 | −0.1 (3) |
| C1—N1—C5—C4 | −1.8 (2) | C11—C12—C13—C14 | 0.5 (3) |
| C6—N1—C5—C4 | −179.69 (15) | C12—C13—C14—C15 | −0.4 (3) |
| N3—C4—C5—N2 | 0.95 (19) | C13—C14—C15—C10 | 0.0 (3) |
| C3—C4—C5—N2 | −177.74 (14) | C11—C10—C15—C14 | 0.4 (3) |
| N3—C4—C5—N1 | −179.52 (14) | C9—C10—C15—C14 | −179.12 (16) |
| C3—C4—C5—N1 | 1.8 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H11···N3 | 0.83 (1) | 2.14 (3) | 2.887 (4) | 149 (5) |
| O1—H12···N2$^{i}$ | 0.84 (1) | 2.41 (2) | 3.229 (7) | 165 (5) |

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