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

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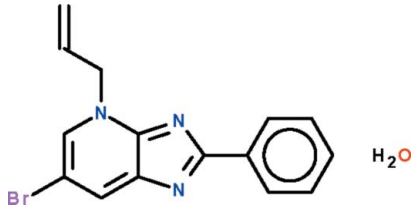
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in solvent or counterion; R factor = 0.027; wR factor = 0.078; data-to-parameter ratio = 15.6.

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
α = 68.076 (1)°
β = 74.637 (1)°
γ = 79.736 (1)°
V = 692.02 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 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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supplementary materials

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

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

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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) °].

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.

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 1.2U_{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.

Figures

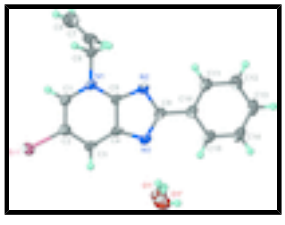

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of the molecule of $C_{15}H_{12}BrN_3 \cdot H_2O$ at the 50% probability level. H atoms are shown as spheres of arbitrary radii. The disorder in the water molecule is shown.

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

Crystal data

$C_{15}H_{12}BrN_3 \cdot H_2O$	$Z = 2$
$M_r = 332.20$	$F(000) = 336$
Triclinic, $P\overline{1}$	$D_x = 1.594$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.4363$ (1) Å	Cell parameters from 7204 reflections
$b = 9.4238$ (1) Å	$\theta = 2.3$–$27.2°$
$c = 11.0829$ (2) Å	$\mu = 2.97$ mm^{-1}
$\alpha = 68.076$ (1)°	$T = 293$ K
$\beta = 74.637$ (1)°	Prism, yellow
$\gamma = 79.736$ (1)°	0.20 × 0.20 × 0.15 mm
$V = 692.02$ (2) Å^3	

Data collection

Bruker X8 APEXII area-detector diffractometer	3158 independent reflections
Radiation source: fine-focus sealed tube	2791 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.029$
φ and ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.5°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{min} = 0.588$, $T_{max} = 0.664$	$k = -12 \rightarrow 12$
14314 measured reflections	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.027$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.078$	H atoms treated by a mixture of independent and constrained refinement
$S = 0.98$	$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.1091P]$ where $P = (F_o^2 + 2F_c^2)/3$
3158 reflections	$(\Delta/\sigma)_{max} = 0.001$
203 parameters	$\Delta\rho_{max} = 0.25$ e Å^{-3}
6 restraints	$\Delta\rho_{min} = -0.39$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	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 (Å^2)

	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, °)

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, °)

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 codes: (i) −*x*+2, −*y*+1, −*z*+2.

Fig. 1