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6-Bromo-1-(1,2-propadienyl)-3-(2-propynyl)-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

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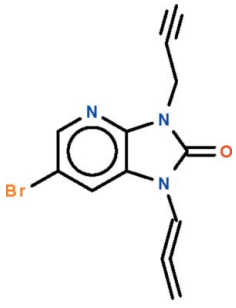
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.087; data-to-parameter ratio = 20.4.

The reaction of propargyl bromide and 6-bromo-1,3-dihydro-imidazo[4,5-*b*]pyridin-2-one in refluxing dimethylformamide yields the title compound, $C_{12}H_8BrN_3O$, which features nitrogen-bound propadienyl and propynyl substituents. The imidazolopyridine fused ring is planar (r.m.s. deviation = 0.012 Å); the propadienyl chain is coplanar with the fused ring as it is conjugated with it, whereas the propynyl chain is not as the nitrogen-bound C atom is a methylene linkage. The acetylenic H atom is hydrogen bonded to the carbonyl O atom of an adjacent molecule, forming a helical chain runnning along the *b* axis.

Related literature

For the crystal structures of other imidazo[4,5-*b*]pyridin-2-ones, see: Kourafalos *et al.* (2002); Meanwell *et al.* (1995).



Experimental

Crystal data

$C_{12}H_8BrN_3O$
M_r = 290.12
Monoclinic, $P2_1/n$
a = 9.6369 (4) Å
b = 9.3086 (4) Å
c = 13.5481 (5) Å
β = 99.123 (2)°
V = 1199.97 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.41 mm^{-1}
T = 293 K
0.55 × 0.35 × 0.30 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.256, T_{max} = 0.428
51411 measured reflections
3393 independent reflections
2577 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.087
S = 1.02
3393 reflections
166 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O1^{i}	0.94 (1)	2.22 (1)	3.161 (3)	173 (2)

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

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: HG2649).

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supplementary materials

Acta Cryst. (2010). E**66**, o755 [**doi:10.1107/S1600536810007695**]

6-Bromo-1-(1,2-propadienyl)-3-(2-propynyl)-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

S. Dahmani, A. Haoudi, F. Capet, E. M. Essassi and S. W. Ng

Experimental

To a solution of 6-bromo-1,3-dihydro-imidazo[4,5-*b*]pyridin-2-one (1 mmol), potassium carbonate (4 mmol) and tetra-*n*-butylammonium bromide (0.1 mmol) in DMF (20 ml) was added propargyl bromide (2.5 mmol). The solution was refluxed for 48 hours. After completion of the reaction (as monitored byTLC), the salt was filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel by using an ethyl acetate/hexane (1/1) mixture as eluent. Slow evaporation of the solvent furnished colorless crystals.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.97 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C). The terminal acetylenic and allenic H-atoms were located in a difference Fourier map, and were refined with a distance restraint of C–H 0.95±0.01 Å; their temperature factors were refined.

Figures

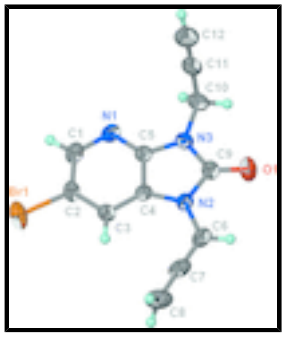

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{12}H_8BrN_3O$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

6-Bromo-1-(1,2-propadienyl)-3-(2-propynyl)-1*H*- imidazo[4,5-*b*]pyridin-2(3*H*)-one

Crystal data

$C_{12}H_8BrN_3O$	$F(000) = 576$
$M_r = 290.12$	$D_x = 1.606$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 9975 reflections
$a = 9.6369$ (4) Å	$\theta = 2.4$–$25.6°$
$b = 9.3086$ (4) Å	$\mu = 3.41$ mm^{-1}
$c = 13.5481$ (5) Å	$T = 293$ K
$\beta = 99.123$ (2)°	Irregular, colorless

V = 1199.97 (8) Å^3	0.55 × 0.35 × 0.30 mm
Z = 4	

Data collection

Bruker X8 APEXII diffractometer	3393 independent reflections
Radiation source: fine-focus sealed tube	2577 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.040
φ and ω scans	θ_{max} = 29.7°, θ_{min} = 2.7°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→13
T_{min} = 0.256, T_{max} = 0.428	k = −12→12
51411 measured reflections	l = −18→18

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.031	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.087	H atoms treated by a mixture of independent and constrained refinement
S = 1.02	$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.4635P]$ where $P = (F_o^2 + 2F_c^2)/3$
3393 reflections	$(\Delta/\sigma)_{max}$ = 0.001
166 parameters	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
3 restraints	$\Delta\rho_{min}$ = −0.63 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Br1	0.40648 (3)	0.39876 (3)	0.231050 (17)	0.06785 (11)
O1	0.49655 (17)	0.95432 (17)	0.64205 (11)	0.0577 (4)
N1	0.21863 (16)	0.64109 (19)	0.42594 (13)	0.0453 (4)
N2	0.54742 (15)	0.78896 (16)	0.52363 (11)	0.0395 (3)
N3	0.32605 (16)	0.81244 (17)	0.54674 (11)	0.0417 (3)
C1	0.2477 (2)	0.5495 (2)	0.35479 (15)	0.0490 (4)
H1	0.1744	0.4972	0.3188	0.059*
C2	0.3815 (2)	0.53039 (19)	0.33326 (13)	0.0435 (4)
C3	0.4981 (2)	0.60309 (19)	0.38454 (13)	0.0399 (4)
H3	0.5887	0.5895	0.3708	0.048*
C4	0.46792 (17)	0.69616 (18)	0.45684 (12)	0.0346 (3)
C5	0.32815 (18)	0.71031 (18)	0.47294 (12)	0.0365 (3)
C6	0.6938 (2)	0.8150 (2)	0.53701 (16)	0.0503 (5)
H6	0.7302	0.8828	0.5845	0.060*
C7	0.7806 (2)	0.7510 (2)	0.48776 (17)	0.0529 (5)

C8	0.8709 (3)	0.6872 (4)	0.4413 (2)	0.0750 (8)
H81	0.916 (3)	0.602 (2)	0.467 (3)	0.106 (12)*
H82	0.890 (3)	0.722 (3)	0.3788 (13)	0.089 (10)*
C9	0.4605 (2)	0.8628 (2)	0.57909 (14)	0.0416 (4)
C10	0.2020 (2)	0.8584 (2)	0.58740 (16)	0.0509 (5)
H10A	0.1252	0.8736	0.5328	0.061*
H10B	0.2218	0.9494	0.6217	0.061*
C11	0.1586 (2)	0.7538 (2)	0.65707 (15)	0.0521 (5)
C12	0.1208 (3)	0.6715 (3)	0.71186 (19)	0.0694 (7)
H12	0.093 (3)	0.601 (2)	0.7550 (18)	0.088 (11)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0918 (2)	0.05653 (15)	0.05116 (14)	0.01376 (12)	−0.00107 (12)	−0.01710 (9)
O1	0.0645 (9)	0.0499 (8)	0.0563 (9)	0.0031 (7)	0.0024 (7)	−0.0158 (7)
N1	0.0357 (8)	0.0481 (9)	0.0513 (9)	−0.0027 (7)	0.0046 (7)	0.0022 (7)
N2	0.0367 (7)	0.0396 (8)	0.0417 (7)	−0.0023 (6)	0.0050 (6)	−0.0021 (6)
N3	0.0417 (8)	0.0425 (8)	0.0431 (8)	0.0011 (6)	0.0133 (6)	−0.0014 (6)
C1	0.0469 (10)	0.0461 (10)	0.0504 (10)	−0.0050 (8)	−0.0035 (8)	−0.0017 (8)
C2	0.0555 (11)	0.0369 (9)	0.0362 (8)	0.0034 (8)	0.0016 (7)	−0.0014 (7)
C3	0.0408 (9)	0.0411 (9)	0.0385 (8)	0.0031 (7)	0.0085 (7)	0.0024 (7)
C4	0.0351 (8)	0.0350 (8)	0.0334 (7)	−0.0008 (6)	0.0051 (6)	0.0060 (6)
C5	0.0386 (8)	0.0350 (8)	0.0364 (8)	0.0006 (7)	0.0079 (6)	0.0057 (6)
C6	0.0381 (9)	0.0515 (11)	0.0592 (12)	−0.0078 (8)	0.0015 (8)	−0.0032 (9)
C7	0.0343 (9)	0.0597 (12)	0.0628 (12)	−0.0064 (9)	0.0017 (9)	0.0098 (10)
C8	0.0396 (11)	0.101 (2)	0.0867 (19)	0.0039 (13)	0.0176 (12)	0.0060 (17)
C9	0.0474 (10)	0.0370 (9)	0.0402 (9)	0.0029 (7)	0.0061 (7)	0.0014 (7)
C10	0.0512 (11)	0.0491 (11)	0.0570 (12)	0.0097 (9)	0.0224 (9)	0.0029 (9)
C11	0.0510 (11)	0.0614 (12)	0.0461 (10)	0.0007 (9)	0.0149 (8)	−0.0033 (9)
C12	0.0748 (16)	0.0808 (18)	0.0555 (13)	−0.0120 (14)	0.0197 (12)	0.0087 (12)

Geometric parameters (Å, °)

Br1—C2	1.8928 (18)	C3—C4	1.373 (2)
O1—C9	1.216 (2)	C3—H3	0.9300
N1—C5	1.312 (2)	C4—C5	1.404 (2)
N1—C1	1.349 (3)	C6—C7	1.295 (3)
N2—C4	1.390 (2)	C6—H6	0.9300
N2—C9	1.393 (2)	C7—C8	1.296 (4)
N2—C6	1.414 (2)	C8—H81	0.95 (1)
N3—C5	1.382 (2)	C8—H82	0.95 (1)
N3—C9	1.382 (2)	C10—C11	1.463 (3)
N3—C10	1.458 (2)	C10—H10A	0.9700
C1—C2	1.378 (3)	C10—H10B	0.9700
C1—H1	0.9300	C11—C12	1.164 (3)
C2—C3	1.398 (3)	C12—H12	0.94 (1)
C5—N1—C1	114.50 (16)	N1—C5—C4	126.59 (17)

C4—N2—C9	109.96 (14)	N3—C5—C4	107.53 (15)
C4—N2—C6	128.72 (16)	C7—C6—N2	124.62 (19)
C9—N2—C6	121.29 (16)	C7—C6—H6	117.7
C5—N3—C9	110.03 (15)	N2—C6—H6	117.7
C5—N3—C10	125.63 (16)	C8—C7—C6	178.0 (3)
C9—N3—C10	124.33 (16)	C7—C8—H81	121 (2)
N1—C1—C2	122.72 (18)	C7—C8—H82	120.7 (19)
N1—C1—H1	118.6	H81—C8—H82	118 (3)
C2—C1—H1	118.6	O1—C9—N3	127.55 (18)
C1—C2—C3	122.49 (17)	O1—C9—N2	126.48 (18)
C1—C2—Br1	118.07 (14)	N3—C9—N2	105.97 (15)
C3—C2—Br1	119.43 (14)	N3—C10—C11	112.56 (17)
C4—C3—C2	114.56 (16)	N3—C10—H10A	109.1
C4—C3—H3	122.7	C11—C10—H10A	109.1
C2—C3—H3	122.7	N3—C10—H10B	109.1
C3—C4—N2	134.39 (16)	C11—C10—H10B	109.1
C3—C4—C5	119.12 (16)	H10A—C10—H10B	107.8
N2—C4—C5	106.49 (15)	C12—C11—C10	178.4 (3)
N1—C5—N3	125.87 (16)	C11—C12—H12	177.2 (19)
C5—N1—C1—C2	0.0 (3)	C3—C4—C5—N1	0.9 (3)
N1—C1—C2—C3	0.9 (3)	N2—C4—C5—N1	−179.58 (17)
N1—C1—C2—Br1	−179.76 (15)	C3—C4—C5—N3	−178.29 (15)
C1—C2—C3—C4	−0.8 (3)	N2—C4—C5—N3	1.26 (18)
Br1—C2—C3—C4	179.81 (12)	C4—N2—C6—C7	−1.4 (3)
C2—C3—C4—N2	−179.36 (18)	C9—N2—C6—C7	−179.6 (2)
C2—C3—C4—C5	0.0 (2)	C5—N3—C9—O1	178.90 (19)
C9—N2—C4—C3	178.04 (18)	C10—N3—C9—O1	−2.7 (3)
C6—N2—C4—C3	−0.3 (3)	C5—N3—C9—N2	−0.2 (2)
C9—N2—C4—C5	−1.41 (19)	C10—N3—C9—N2	178.17 (16)
C6—N2—C4—C5	−179.75 (17)	C4—N2—C9—O1	−178.10 (19)
C1—N1—C5—N3	178.16 (17)	C6—N2—C9—O1	0.4 (3)
C1—N1—C5—C4	−0.9 (3)	C4—N2—C9—N3	1.00 (19)
C9—N3—C5—N1	−179.84 (17)	C6—N2—C9—N3	179.49 (16)
C10—N3—C5—N1	1.8 (3)	C5—N3—C10—C11	76.4 (2)
C9—N3—C5—C4	−0.67 (19)	C9—N3—C10—C11	−101.7 (2)
C10—N3—C5—C4	−179.00 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1^{i}	0.94 (1)	2.22 (1)	3.161 (3)	173 (2)

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+3/2.

Fig. 1