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

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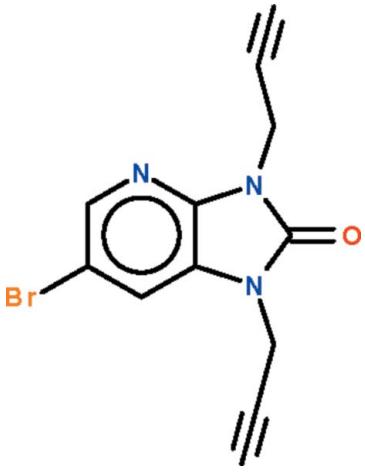
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.085; data-to-parameter ratio = 20.9.

The room-temperature reaction of propargyl bromide and 6-bromo-1,3-dihydroimidazo[4,5-*b*]pyridin-2-one in dimethylformamide yields the title compound, $C_{12}H_8BrN_3O$, which features nitrogen-bound propynyl substituents. The imidazopyridine fused ring is almost planar (r.m.s. deviation = 0.011 Å); the propynyl chains point in opposite directions relative to the fused ring. One acetylenic H atom is hydrogen bonded to the carbonyl O atom of an inversion-related molecule, forming a dimer; adjacent dimers are linked by a second acetylene–pyridine C—H···N interaction, forming a layer motif.

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, $P2_1/c$
a = 9.0725 (3) Å
b = 18.6212 (5) Å
c = 7.0684 (2) Å
β = 102.995 (1)°
V = 1163.56 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.52 mm^{-1}
T = 293 K
0.35 × 0.30 × 0.15 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.372, T_{max} = 0.620
27315 measured reflections
3383 independent reflections
2810 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.085
S = 1.03
3383 reflections
162 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.87 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1^{i}	0.95 (1)	2.53 (2)	3.392 (3)	151 (3)
C12—H12···N1^{ii}	0.94 (1)	2.51 (2)	3.346 (2)	149 (2)

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

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: HG2650).

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supplementary materials

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

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Experimental

To a solution of 6-bromo-1,3-dihydroimidazo[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 stirred 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 yellow 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 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

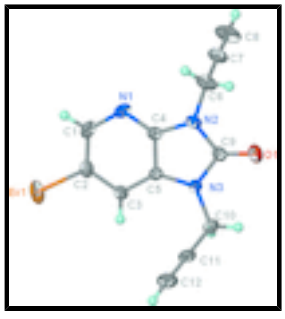

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{12}H_8BrN_3O$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{12}H_8BrN_3O$	$F(000) = 576$
$M_r = 290.12$	$D_x = 1.656$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 9924 reflections
$a = 9.0725$ (3) Å	$\theta = 2.3$–$31.8°$
$b = 18.6212$ (5) Å	$\mu = 3.52$ mm^{-1}
$c = 7.0684$ (2) Å	$T = 293$ K
$\beta = 102.995$ (1)°	Prism, yellow
$V = 1163.56$ (6) Å^3	0.35 × 0.30 × 0.15 mm

$Z = 4$

Data collection

Bruker X8 APEXII diffractometer	3383 independent reflections
Radiation source: fine-focus sealed tube	2810 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.032$
φ and ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12 \rightarrow 12$
$T_{min} = 0.372$, $T_{max} = 0.620$	$k = -26 \rightarrow 26$
27315 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.029$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.085$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.4099P]$ where $P = (F_o^2 + 2F_c^2)/3$
3383 reflections	$(\Delta/\sigma)_{max} = 0.001$
162 parameters	$\Delta\rho_{max} = 0.87$ e Å^{-3}
2 restraints	$\Delta\rho_{min} = -0.80$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.40313 (3)	0.071904 (9)	0.35455 (3)	0.05174 (9)
O1	0.32318 (15)	0.47483 (7)	0.3528 (2)	0.0435 (3)
N1	0.12161 (15)	0.24467 (8)	0.3731 (2)	0.0354 (3)
N2	0.18505 (15)	0.37028 (8)	0.3651 (2)	0.0326 (3)
N3	0.42693 (14)	0.35966 (7)	0.35770 (18)	0.0289 (3)
C1	0.1822 (2)	0.17839 (9)	0.3720 (2)	0.0368 (4)
H1	0.1213	0.1387	0.3786	0.044*
C2	0.3302 (2)	0.16693 (8)	0.3614 (2)	0.0322 (3)
C3	0.43018 (17)	0.22350 (8)	0.3550 (2)	0.0289 (3)
H3	0.5302	0.2161	0.3481	0.035*
C4	0.21635 (17)	0.29782 (8)	0.3662 (2)	0.0279 (3)
C5	0.36927 (16)	0.29091 (8)	0.3596 (2)	0.0251 (3)
C6	0.0418 (2)	0.40256 (12)	0.3799 (3)	0.0457 (4)
H6A	0.0621	0.4471	0.4522	0.055*
H6B	−0.0093	0.3705	0.4524	0.055*
C7	−0.0586 (2)	0.41743 (12)	0.1911 (3)	0.0484 (5)
C8	−0.1389 (3)	0.43130 (16)	0.0410 (4)	0.0699 (8)

H8	−0.212 (3)	0.442 (2)	−0.073 (3)	0.108 (13)*
C9	0.31325 (18)	0.40998 (9)	0.3577 (2)	0.0307 (3)
C10	0.58352 (18)	0.38016 (10)	0.3664 (3)	0.0380 (4)
H10A	0.5957	0.4311	0.3946	0.046*
H10B	0.6068	0.3718	0.2408	0.046*
C11	0.68988 (18)	0.33965 (10)	0.5150 (3)	0.0397 (4)
C12	0.7772 (2)	0.30575 (14)	0.6294 (4)	0.0559 (6)
H12	0.849 (2)	0.2804 (13)	0.721 (3)	0.070 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.07831 (18)	0.02651 (10)	0.04492 (13)	0.00475 (8)	0.00231 (10)	−0.00343 (6)
O1	0.0484 (7)	0.0281 (6)	0.0488 (7)	0.0041 (5)	−0.0001 (6)	0.0039 (5)
N1	0.0277 (6)	0.0436 (8)	0.0344 (7)	−0.0079 (6)	0.0059 (5)	−0.0006 (6)
N2	0.0261 (6)	0.0344 (7)	0.0357 (7)	0.0063 (5)	0.0039 (5)	0.0004 (5)
N3	0.0234 (6)	0.0261 (6)	0.0353 (6)	−0.0012 (4)	0.0023 (5)	0.0030 (5)
C1	0.0402 (9)	0.0356 (8)	0.0330 (8)	−0.0124 (7)	0.0050 (7)	−0.0002 (6)
C2	0.0437 (9)	0.0262 (7)	0.0246 (7)	−0.0018 (6)	0.0036 (6)	−0.0010 (5)
C3	0.0291 (7)	0.0294 (7)	0.0276 (7)	0.0028 (5)	0.0051 (6)	0.0003 (5)
C4	0.0248 (7)	0.0332 (7)	0.0240 (6)	0.0005 (5)	0.0022 (5)	−0.0001 (5)
C5	0.0237 (6)	0.0269 (7)	0.0236 (6)	−0.0015 (5)	0.0034 (5)	0.0005 (5)
C6	0.0346 (9)	0.0554 (11)	0.0481 (10)	0.0163 (8)	0.0114 (8)	−0.0029 (9)
C7	0.0306 (9)	0.0535 (11)	0.0610 (12)	0.0143 (8)	0.0101 (8)	0.0079 (9)
C8	0.0452 (13)	0.093 (2)	0.0672 (16)	0.0256 (12)	0.0040 (11)	0.0159 (13)
C9	0.0304 (7)	0.0294 (7)	0.0286 (7)	0.0028 (6)	−0.0014 (6)	0.0018 (6)
C10	0.0274 (7)	0.0392 (9)	0.0457 (9)	−0.0064 (6)	0.0047 (7)	0.0103 (7)
C11	0.0236 (7)	0.0455 (9)	0.0482 (10)	−0.0043 (6)	0.0042 (7)	0.0049 (8)
C12	0.0307 (9)	0.0659 (14)	0.0661 (14)	0.0016 (9)	0.0002 (9)	0.0171 (11)

Geometric parameters (Å, °)

Br1—C2	1.8935 (16)	C3—C5	1.375 (2)
O1—C9	1.212 (2)	C3—H3	0.9300
N1—C4	1.319 (2)	C4—C5	1.405 (2)
N1—C1	1.352 (2)	C6—C7	1.463 (3)
N2—C4	1.378 (2)	C6—H6A	0.9700
N2—C9	1.389 (2)	C6—H6B	0.9700
N2—C6	1.457 (2)	C7—C8	1.173 (3)
N3—C5	1.3842 (19)	C8—H8	0.948 (10)
N3—C9	1.394 (2)	C10—C11	1.466 (2)
N3—C10	1.459 (2)	C10—H10A	0.9700
C1—C2	1.379 (3)	C10—H10B	0.9700
C1—H1	0.9300	C11—C12	1.179 (3)
C2—C3	1.397 (2)	C12—H12	0.939 (10)
C4—N1—C1	114.56 (14)	N3—C5—C4	107.09 (13)
C4—N2—C9	110.37 (13)	N2—C6—C7	113.27 (16)
C4—N2—C6	126.11 (16)	N2—C6—H6A	108.9

C9—N2—C6	123.44 (15)	C7—C6—H6A	108.9
C5—N3—C9	109.91 (13)	N2—C6—H6B	108.9
C5—N3—C10	127.46 (14)	C7—C6—H6B	108.9
C9—N3—C10	122.53 (14)	H6A—C6—H6B	107.7
N1—C1—C2	122.96 (15)	C8—C7—C6	178.2 (3)
N1—C1—H1	118.5	C7—C8—H8	174 (2)
C2—C1—H1	118.5	O1—C9—N2	126.84 (15)
C1—C2—C3	122.16 (15)	O1—C9—N3	127.61 (16)
C1—C2—Br1	119.74 (12)	N2—C9—N3	105.55 (13)
C3—C2—Br1	118.10 (13)	N3—C10—C11	111.93 (14)
C5—C3—C2	114.86 (14)	N3—C10—H10A	109.2
C5—C3—H3	122.6	C11—C10—H10A	109.2
C2—C3—H3	122.6	N3—C10—H10B	109.2
N1—C4—N2	126.85 (15)	C11—C10—H10B	109.2
N1—C4—C5	126.10 (15)	H10A—C10—H10B	107.9
N2—C4—C5	107.05 (13)	C12—C11—C10	177.6 (2)
C3—C5—N3	133.58 (14)	C11—C12—H12	177.7 (17)
C3—C5—C4	119.33 (14)		
C4—N1—C1—C2	−1.0 (2)	N1—C4—C5—C3	1.7 (2)
N1—C1—C2—C3	1.4 (3)	N2—C4—C5—C3	−178.61 (13)
N1—C1—C2—Br1	−178.53 (12)	N1—C4—C5—N3	−178.81 (14)
C1—C2—C3—C5	−0.2 (2)	N2—C4—C5—N3	0.92 (16)
Br1—C2—C3—C5	179.73 (10)	C4—N2—C6—C7	−93.3 (2)
C1—N1—C4—N2	179.82 (15)	C9—N2—C6—C7	90.2 (2)
C1—N1—C4—C5	−0.5 (2)	C4—N2—C9—O1	179.15 (16)
C9—N2—C4—N1	179.78 (15)	C6—N2—C9—O1	−3.9 (3)
C6—N2—C4—N1	2.9 (3)	C4—N2—C9—N3	−0.99 (17)
C9—N2—C4—C5	0.05 (17)	C6—N2—C9—N3	175.99 (15)
C6—N2—C4—C5	−176.84 (15)	C5—N3—C9—O1	−178.56 (16)
C2—C3—C5—N3	179.41 (15)	C10—N3—C9—O1	4.8 (3)
C2—C3—C5—C4	−1.2 (2)	C5—N3—C9—N2	1.58 (17)
C9—N3—C5—C3	177.86 (16)	C10—N3—C9—N2	−175.03 (14)
C10—N3—C5—C3	−5.7 (3)	C5—N3—C10—C11	−45.0 (2)
C9—N3—C5—C4	−1.58 (16)	C9—N3—C10—C11	131.00 (17)
C10—N3—C5—C4	174.83 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.95 (1)	2.53 (2)	3.392 (3)	151 (3)
C12—H12···N1^{ii}	0.94 (1)	2.51 (2)	3.346 (2)	149 (2)

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

Fig. 1