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3-Bromo-6-nitro-1-(prop-2-ynyl)-1*H*-indazole

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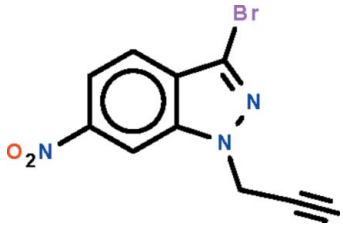
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.079; data-to-parameter ratio = 21.1.

In the title compound, $C_{10}H_6BrN_3O_2$, the indazole fused-ring system is nearly planar (r.m.s. deviation = 0.008 Å); its nitro substituent is nearly coplanar with the fused ring [dihedral angle = 4.5 (2)°]. In the crystal, adjacent molecules are linked by weak acetylene–nitro C—H···O hydrogen bonds, generating a helical chain running along the b axis.

Related literature

For a related compound, 1-allyl-3-chloro-6-nitro-1*H*-indazole, see: El Brahmi *et al.* (2009).



Experimental

Crystal data

$C_{10}H_6BrN_3O_2$ M_r = 280.09
Monoclinic, $P2_1/n$
a = 14.6573 (3) Å
b = 4.1650 (1) Å
c = 17.4566 (3) Å
β = 102.659 (1)°
V = 1039.78 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.94 mm^{-1}
T = 295 K
0.50 × 0.10 × 0.05 mm

Data collection

Bruker APEX DUO diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.243, T_{max} = 0.827
14908 measured reflections
3137 independent reflections
2236 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.079
S = 1.03
3137 reflections
149 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H1···O1^{i}	0.96 (3)	2.45 (3)	3.399 (3)	167 (3)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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).

We thank Université MohammedV-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5386).

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supplementary materials

Acta Cryst. (2011). E**67**, o3260 [**doi:10.1107/S1600536811046927**]

3-Bromo-6-nitro-1-(prop-2-ynyl)-1*H*-indazole

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Comment

We reported 1-allyl-3-chloro-6-nitro-1*H*-indazole, which exists as two independent molecules (El Brahmi *et al.*, 2009). The present 1-propynyl-3-bromo-6-nitro-1*H*-indazole (Scheme I) also has halogen subsituent in the same position but the asymmetric unit consists of one molecule only. The indazole fused-ring is planar; its nitro substituent is nearly coplanar with the fused ring (Fig 1.). Adjacent molecules are linked by a C–$H_{acetylene}$···O_{nitro} hydrogen bond to generate a helical polymer running along the *b*-axis of the monoclinic unit cell (Fig. 2). Weak Br···Br contacts of 3.57 Å are present.

Experimental

3-Bromo-6-nitroindazole (1.2 g, 5 mmol) and propargyl bromide (1.2 g, 10 mmol) were reacted in THF (40 ml) in the presence of potassium carbonate (1.4 g, 10 mmol) and tetra-*n*-butylammonium bromide (0.5 mmol). The mixture was stirred for 24 h, filtered, and the THF removed under vacuum. The product was separated by chromatography on silica gel with a hexane:ethyl acetate (9:1) solvent system. The compound was obtained as yellow crystals.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C). The acetylenic H-atom was located in a difference Fourier map and was refined. The 1 0 1 and -1 0 1 reflections were omitted owing to bad agreement.

Figures

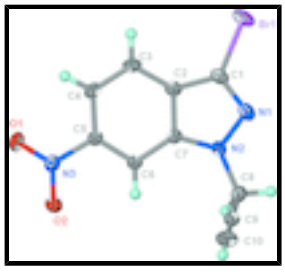

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{10}H_6BrN_3O_2$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

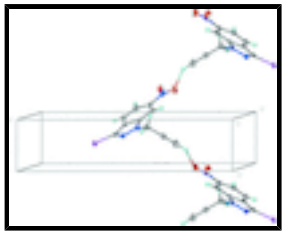

Fig. 2. Helical chain motif.

3-Bromo-6-nitro-1-(prop-2-ynyl)-1*H*-indazole

Crystal data

$C_{10}H_6BrN_3O_2$	$F(000) = 552$
$M_r = 280.09$	$D_x = 1.789$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 5822 reflections
$a = 14.6573$ (3) Å	$\theta = 2.4$–$30.3°$
$b = 4.1650$ (1) Å	$\mu = 3.94$ mm^{-1}
$c = 17.4566$ (3) Å	$T = 295$ K
$\beta = 102.659$ (1)°	Plate, yellow
$V = 1039.78$ (4) Å^3	0.50 × 0.10 × 0.05 mm
$Z = 4$	

Data collection

Bruker APEX DUO diffractometer	3137 independent reflections
Radiation source: fine-focus sealed tube	2236 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.023$
ω scans	$\theta_{max} = 30.5°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -15\rightarrow20$
$T_{min} = 0.243$, $T_{max} = 0.827$	$k = -5\rightarrow5$
14908 measured reflections	$l = -24\rightarrow24$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.028$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.079$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 0.4798P]$ where $P = (F_o^2 + 2F_c^2)/3$
3137 reflections	$(\Delta/\sigma)_{max} = 0.001$
149 parameters	$\Delta\rho_{max} = 0.52$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.76$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.234567 (17)	0.49344 (5)	0.664925 (11)	0.03944 (9)
O1	0.44171 (11)	−0.5183 (4)	0.38032 (10)	0.0431 (4)

O2	0.30475 (11)	−0.5312 (3)	0.30395 (9)	0.0401 (4)
N1	0.11630 (11)	0.3405 (4)	0.52285 (9)	0.0296 (3)
N2	0.11523 (10)	0.1781 (4)	0.45461 (9)	0.0262 (3)
N3	0.35862 (11)	−0.4437 (4)	0.36351 (10)	0.0278 (3)
C1	0.20201 (14)	0.3105 (4)	0.56494 (10)	0.0281 (4)
C2	0.26030 (13)	0.1289 (4)	0.52666 (10)	0.0250 (4)
C3	0.35342 (14)	0.0226 (4)	0.54510 (11)	0.0287 (4)
H3	0.3930	0.0758	0.5927	0.034*
C4	0.38431 (13)	−0.1624 (4)	0.49070 (11)	0.0278 (4)
H4	0.4456	−0.2367	0.5011	0.033*
C5	0.32278 (12)	−0.2386 (4)	0.41932 (10)	0.0237 (3)
C6	0.23102 (12)	−0.1400 (4)	0.39812 (10)	0.0231 (3)
H6	0.1923	−0.1923	0.3501	0.028*
C7	0.20061 (12)	0.0457 (4)	0.45476 (11)	0.0226 (3)
C8	0.02773 (13)	0.1524 (5)	0.39593 (11)	0.0288 (4)
H8A	−0.0229	0.2344	0.4179	0.035*
H8B	0.0150	−0.0722	0.3831	0.035*
C9	0.03026 (13)	0.3299 (5)	0.32386 (11)	0.0293 (4)
C10	0.03171 (17)	0.4760 (5)	0.26605 (13)	0.0403 (5)
H1	0.033 (2)	0.595 (7)	0.2189 (19)	0.069 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.06481 (17)	0.03278 (12)	0.02030 (10)	0.00030 (9)	0.00838 (9)	−0.00163 (7)
O1	0.0298 (8)	0.0620 (11)	0.0371 (8)	0.0170 (7)	0.0068 (6)	−0.0001 (7)
O2	0.0352 (8)	0.0498 (9)	0.0340 (8)	0.0039 (6)	0.0049 (6)	−0.0132 (6)
N1	0.0398 (9)	0.0261 (8)	0.0257 (8)	0.0012 (7)	0.0131 (7)	0.0006 (6)
N2	0.0266 (8)	0.0284 (8)	0.0244 (7)	0.0018 (6)	0.0072 (6)	−0.0018 (6)
N3	0.0270 (8)	0.0301 (8)	0.0269 (8)	0.0028 (6)	0.0071 (6)	0.0046 (6)
C1	0.0415 (11)	0.0236 (8)	0.0199 (8)	−0.0026 (7)	0.0084 (7)	0.0014 (6)
C2	0.0319 (10)	0.0205 (8)	0.0220 (8)	−0.0023 (7)	0.0045 (7)	0.0034 (6)
C3	0.0310 (10)	0.0299 (9)	0.0220 (8)	−0.0045 (7)	−0.0012 (7)	0.0030 (7)
C4	0.0227 (9)	0.0292 (9)	0.0292 (9)	0.0002 (7)	0.0008 (7)	0.0053 (7)
C5	0.0245 (9)	0.0228 (8)	0.0241 (8)	−0.0010 (6)	0.0057 (7)	0.0039 (6)
C6	0.0238 (9)	0.0232 (8)	0.0218 (8)	−0.0023 (7)	0.0036 (6)	0.0012 (6)
C7	0.0235 (8)	0.0204 (8)	0.0236 (8)	−0.0016 (6)	0.0044 (7)	0.0029 (6)
C8	0.0239 (9)	0.0292 (9)	0.0335 (10)	−0.0003 (7)	0.0062 (7)	−0.0008 (7)
C9	0.0246 (9)	0.0323 (10)	0.0298 (9)	−0.0007 (7)	0.0032 (7)	−0.0060 (7)
C10	0.0429 (12)	0.0481 (13)	0.0283 (10)	−0.0086 (10)	0.0042 (9)	−0.0021 (9)

Geometric parameters (Å, °)

Br1—C1	1.8682 (17)	C3—H3	0.9300
O1—N3	1.228 (2)	C4—C5	1.405 (2)
O2—N3	1.216 (2)	C4—H4	0.9300
N1—C1	1.315 (2)	C5—C6	1.377 (2)
N1—N2	1.367 (2)	C6—C7	1.403 (2)
N2—C7	1.367 (2)	C6—H6	0.9300

N2—C8	1.459 (2)	C8—C9	1.467 (3)
N3—C5	1.476 (2)	C8—H8A	0.9700
C1—C2	1.414 (3)	C8—H8B	0.9700
C2—C7	1.407 (2)	C9—C10	1.183 (3)
C2—C3	1.403 (3)	C10—H1	0.96 (3)
C3—C4	1.374 (3)		
C1—N1—N2	105.49 (15)	C5—C4—H4	120.2
N1—N2—C7	111.24 (15)	C6—C5—C4	124.76 (17)
N1—N2—C8	119.25 (15)	C6—C5—N3	117.59 (15)
C7—N2—C8	129.42 (15)	C4—C5—N3	117.65 (16)
O2—N3—O1	123.55 (17)	C5—C6—C7	114.68 (16)
O2—N3—C5	118.66 (15)	C5—C6—H6	122.7
O1—N3—C5	117.79 (16)	C7—C6—H6	122.7
N1—C1—C2	112.90 (15)	N2—C7—C6	130.79 (16)
N1—C1—Br1	120.03 (14)	N2—C7—C2	106.99 (16)
C2—C1—Br1	127.07 (14)	C6—C7—C2	122.22 (16)
C7—C2—C3	120.67 (17)	N2—C8—C9	112.42 (15)
C7—C2—C1	103.38 (16)	N2—C8—H8A	109.1
C3—C2—C1	135.92 (17)	C9—C8—H8A	109.1
C4—C3—C2	118.00 (17)	N2—C8—H8B	109.1
C4—C3—H3	121.0	C9—C8—H8B	109.1
C2—C3—H3	121.0	H8A—C8—H8B	107.9
C3—C4—C5	119.66 (17)	C10—C9—C8	179.2 (2)
C3—C4—H4	120.2	C9—C10—H1	180 (2)
C1—N1—N2—C7	0.35 (19)	O1—N3—C5—C4	−4.7 (2)
C1—N1—N2—C8	177.07 (15)	C4—C5—C6—C7	−0.9 (3)
N2—N1—C1—C2	−0.1 (2)	N3—C5—C6—C7	178.09 (15)
N2—N1—C1—Br1	−179.08 (12)	N1—N2—C7—C6	179.58 (17)
N1—C1—C2—C7	−0.2 (2)	C8—N2—C7—C6	3.3 (3)
Br1—C1—C2—C7	178.71 (13)	N1—N2—C7—C2	−0.49 (19)
N1—C1—C2—C3	−178.39 (19)	C8—N2—C7—C2	−176.79 (17)
Br1—C1—C2—C3	0.5 (3)	C5—C6—C7—N2	−178.79 (17)
C7—C2—C3—C4	0.4 (3)	C5—C6—C7—C2	1.3 (2)
C1—C2—C3—C4	178.4 (2)	C3—C2—C7—N2	178.93 (16)
C2—C3—C4—C5	0.0 (3)	C1—C2—C7—N2	0.41 (18)
C3—C4—C5—C6	0.2 (3)	C3—C2—C7—C6	−1.1 (3)
C3—C4—C5—N3	−178.71 (16)	C1—C2—C7—C6	−179.65 (16)
O2—N3—C5—C6	−4.2 (2)	N1—N2—C8—C9	112.94 (18)
O1—N3—C5—C6	176.26 (16)	C7—N2—C8—C9	−71.0 (2)
O2—N3—C5—C4	174.85 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H1···O1^{i}	0.96 (3)	2.45 (3)	3.399 (3)	167 (3)

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

Fig. 1

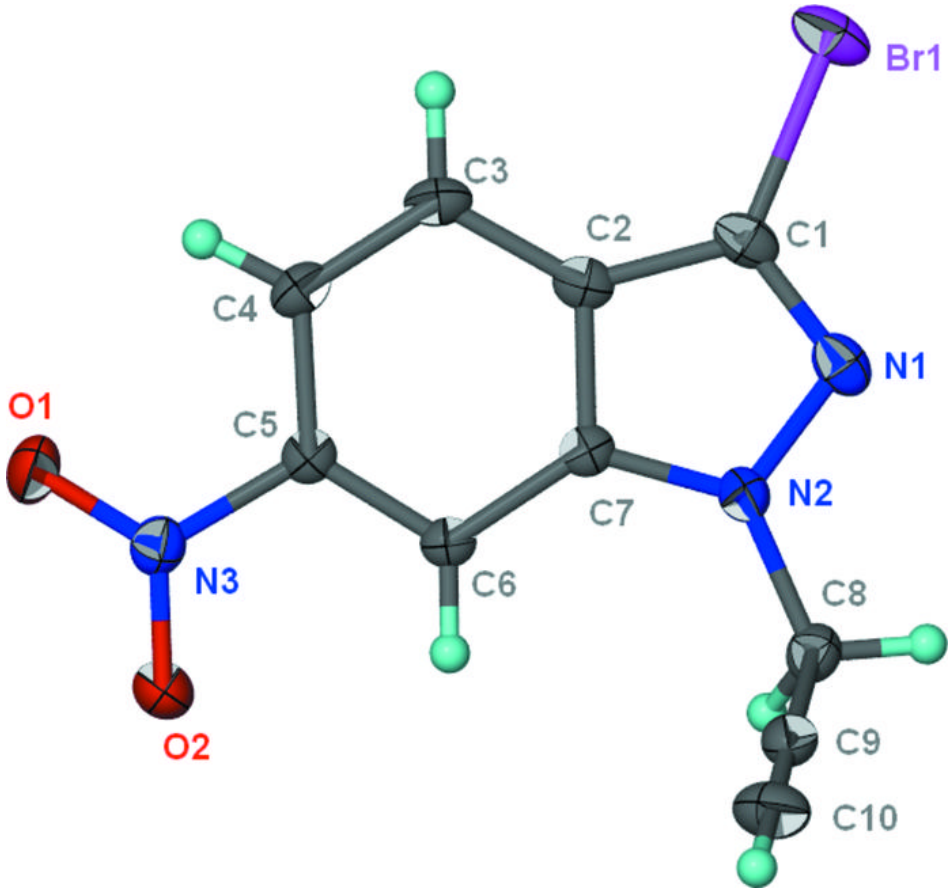

Fig. 2