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

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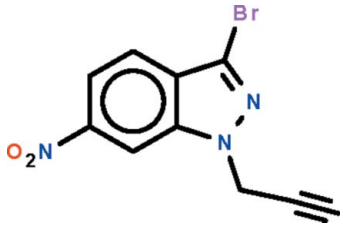
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.028; $wR$ factor = 0.079; data-to-parameter ratio = 21.1.

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
$\beta$ = 102.659 (1)°
$V$ = 1039.78 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 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).

## References

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# supporting information

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

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

**Nabil El Brahmi, Mohamed Benchidmi, El Mokhtar Essassi, Sonia Ladeira and Seik Weng Ng**

### S1. 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.

### S2. 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.

### S3. 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.

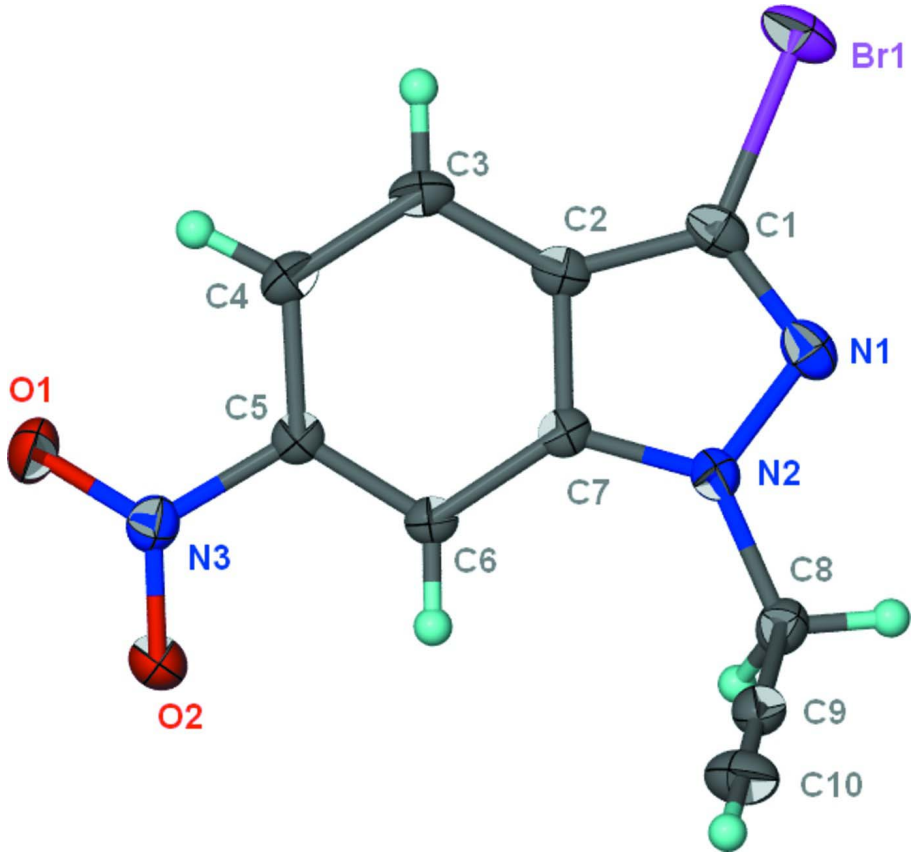


**Figure 1**

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.

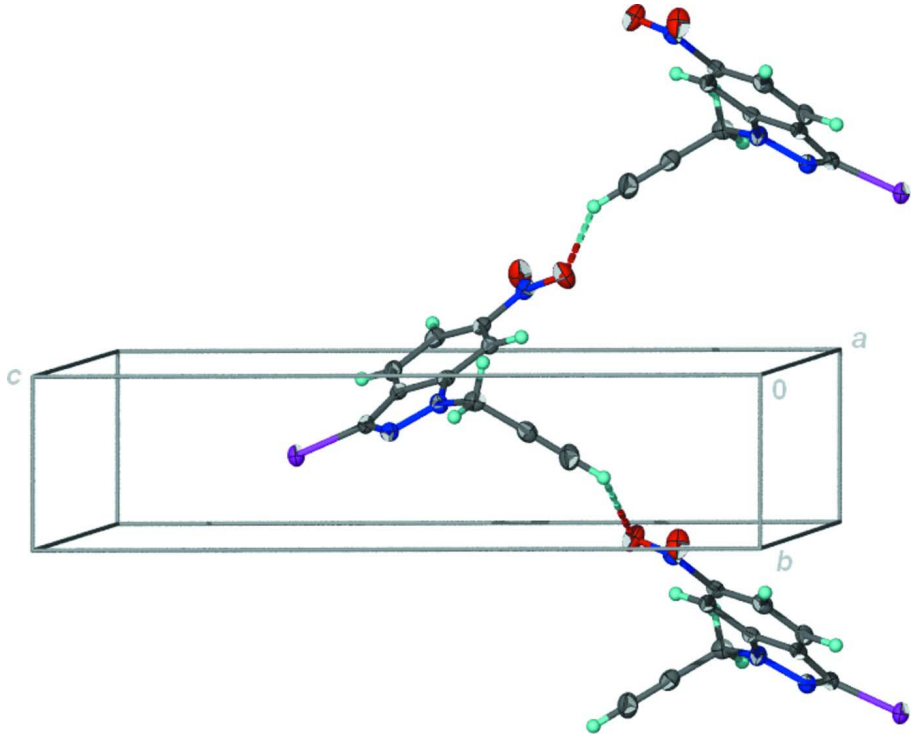


**Figure 2**

Helical chain motif.

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

*Crystal data*

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

*Data collection*

Bruker APEX DUO diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.243$, $T_{max} = 0.827$
14908 measured reflections
3137 independent reflections
2236 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 30.5°$, $\theta_{min} = 2.9°$
$h = -15 \rightarrow 20$
$k = -5 \rightarrow 5$
$l = -24 \rightarrow 24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.079$
$S = 1.03$
3137 reflections
149 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 0.4798P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å$^{-3}$
$\Delta\rho_{min} = -0.76$ e Å$^{-3}$

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

| | *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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *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*+1/2, *y*+3/2, −*z*+1/2.