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ISSN 2414-3146

2-(3-Bromo-5-nitro-1*H*-indazol-1-yl)-1-phenylethanone

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Received 28 March 2017
Accepted 12 April 2017

Edited by P. Bombicz, Hungarian Academy of Sciences, Hungary

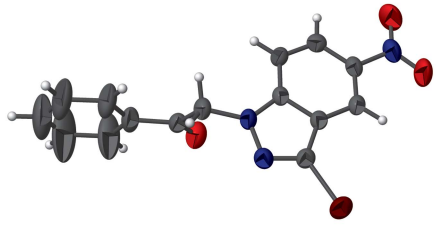
Keywords: crystal structure; 3-bromo-5-nitro-1*H*-indazole; phenylethanone; heterocyclic system; hydrogen bonding; π–π interactions; Br···O interactions.

CCDC reference: 1543758

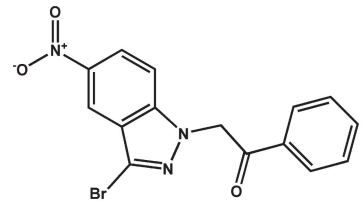
Structural data: full structural data are available from iucrdata.iucr.org

The 5-nitro-1*H*-indazol-1-yl moiety of the title compound, $C_{15}H_{10}BrN_3O_3$, is approximately planar, with the largest deviation from the mean plane being 0.079 (3) Å. The fused-ring system is virtually perpendicular to the mean plane through the 1-phenylethanone group, making a dihedral angle of 89.7 (2)°. In the crystal, pairs of molecules form inversion dimers *via* Br···O interactions [3.211 (2) Å]. The dimers are connected by C—H···O and C—H···N non-classical hydrogen bonds, in addition to π–π interactions [intercentroid distance = 3.6411 (12) Å], forming a three-dimensional network.

3D view



Chemical scheme



Structure description

Recently, pharmacological tests have revealed that indazole derivatives present various biological activities, being potent anti-tumor (Abbassi *et al.*, 2014); anti-microbial (Li *et al.*, 2003); and anti-inflammatory (Schmidt *et al.*, 2008) agents. The crystal structure study of the title compound constitutes a continuation of our previous work on indazole derivatives (Boulhaoua *et al.*, 2015; El Brahmi *et al.*, 2012).

The molecule of the title compound is build up from fused five- and six-membered rings linked to a nitro group and to 1-phenylethanone group as shown in Fig. 1. The highly anisotropic ellipsoids of the phenyl ring are probably due to oscillation of this group. The fused ring system is approximately planar, with the largest deviation from the mean plane being 0.079 (3) Å at O2, and makes a dihedral angle of 89.7 (2)° with the mean plane through the 1-phenylethanone group (O3/C9–C15).

In the crystal, pairs of molecules form inversion dimers *via* Br1···O3 [3.211 (2) Å] interactions. The dimers are linked by C—H···O and C—H···N hydrogen bonds

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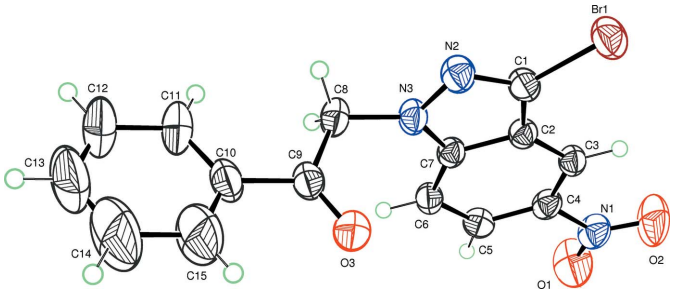

Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small circles of arbitrary radius.

(Table 1) and by π–π interactions [intercentroid distance = 3.6411 (12) Å], forming a three dimensional structure as shown in Fig. 2.

Synthesis and crystallization

To a solution of 3-bromo-5-nitro-1*H*-indazole (0.5 g, 1.38 mmol) in DMF (15 ml) was added phenacyl bromide (0.27 g, 1.38 mmol), potassium carbonate (0.38 g, 2.76 mmol) and a catalytic quantity of tetra-*n*-butylammonium bromide. The mixture was stirred at room temperature for 48 h. The solution was filtered and the solvent removed under reduced pressure. The residue was recrystallized from methanol to afford the title compound as yellow crystals (yield: 65%; m.p. = 415 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

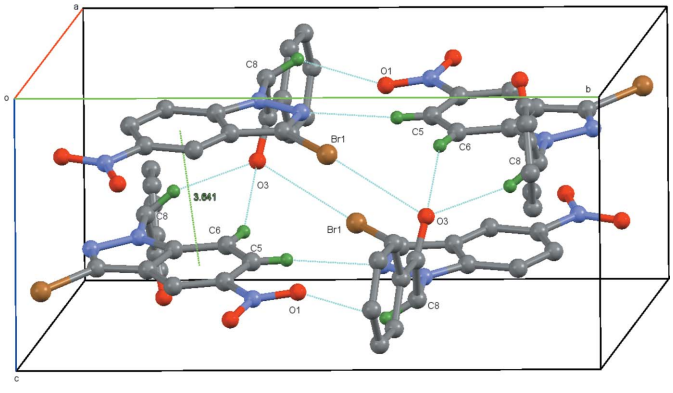

Figure 2
Three-dimensional view of the structure of the title compound, showing molecules linked together by hydrogen bonds (dashed blue lines) and π–π interactions (green line).

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O3^{i}	0.97	2.40	3.315 (3)	157
C8—H8B···O1^{ii}	0.97	2.53	3.244 (3)	131
C5—H5···N2^{iii}	0.93	2.60	3.508 (3)	166
C6—H6···O3^{i}	0.93	2.65	3.502 (3)	152

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{10}BrN_3O_3$
M_r	360.17
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	13.2690 (6), 15.6721 (7), 7.2136 (3)
β (°)	99.029 (2)
V (Å^3)	1481.50 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.79
Crystal size (mm)	0.38 × 0.31 × 0.26
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.547, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	36498, 3823, 2760
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.102, 1.05
No. of reflections	3823
No. of parameters	199
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.41

Computer programs: *APEX2* and *SAINT-Plus* (Bruker, 2009), *SHELXTL2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

Acknowledgements

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements.

Funding information

Funding for this research was provided by: the University Mohammed V, Rabat, Morocco.

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full crystallographic data

IUCrData (2017). **2**, x170559 [https://doi.org/10.1107/S2414314617005594]

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Crystal data

$C_{15}H_{10}BrN_3O_3$
M_r = 360.17
Monoclinic, $P2_1/c$
a = 13.2690 (6) Å
b = 15.6721 (7) Å
c = 7.2136 (3) Å
β = 99.029 (2)°
V = 1481.50 (11) Å^3
Z = 4
$F(000)$ = 720
D_x = 1.615 Mg m^{-3}
Melting point: 415 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3823 reflections
θ = 2.6–28.7°
μ = 2.79 mm^{-1}
T = 296 K
Block, yellow
0.38 × 0.31 × 0.26 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.547, T_{max} = 0.746
36498 measured reflections
3823 independent reflections
2760 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.7°, θ_{min} = 2.6°
h = −13→17
k = −21→21
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.05
3823 reflections
199 parameters
0 restraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.5228P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.58530 (16)	0.58951 (14)	0.7404 (3)	0.0394 (5)
C2	0.60236 (15)	0.67859 (13)	0.7387 (3)	0.0344 (4)
C3	0.68138 (15)	0.73149 (14)	0.7045 (3)	0.0372 (4)
H3	0.7425	0.7099	0.6763	0.045*
C4	0.66406 (15)	0.81759 (15)	0.7149 (3)	0.0393 (5)
C5	0.57322 (17)	0.85329 (14)	0.7571 (3)	0.0407 (5)
H5	0.5660	0.9123	0.7613	0.049*
C6	0.49523 (16)	0.80152 (14)	0.7922 (3)	0.0383 (5)
H6	0.4346	0.8238	0.8212	0.046*
C7	0.51122 (14)	0.71293 (14)	0.7824 (3)	0.0339 (4)
C8	0.34793 (17)	0.64587 (16)	0.8557 (3)	0.0457 (5)
H8A	0.3415	0.6932	0.9396	0.055*
H8B	0.3376	0.5935	0.9220	0.055*
C9	0.26590 (16)	0.65326 (14)	0.6852 (3)	0.0424 (5)
C10	0.15864 (17)	0.64096 (17)	0.7165 (4)	0.0569 (7)
C11	0.1326 (2)	0.6316 (3)	0.8921 (5)	0.0850 (10)
H11	0.1833	0.6344	0.9967	0.102*
C12	0.0327 (3)	0.6182 (3)	0.9170 (8)	0.1154 (16)
H12	0.0160	0.6125	1.0369	0.138*
C13	−0.0393 (3)	0.6136 (4)	0.7660 (11)	0.139 (2)
H13	−0.1065	0.6035	0.7820	0.167*
C14	−0.0177 (3)	0.6229 (5)	0.5930 (11)	0.187 (3)
H14	−0.0698	0.6206	0.4905	0.225*
C15	0.0850 (3)	0.6367 (4)	0.5643 (7)	0.1321 (19)
H15	0.1010	0.6425	0.4439	0.158*
N1	0.74533 (16)	0.87569 (14)	0.6780 (3)	0.0524 (5)
N2	0.49530 (14)	0.56952 (12)	0.7801 (3)	0.0442 (4)
N3	0.44972 (13)	0.64583 (12)	0.8077 (3)	0.0406 (4)
O1	0.73418 (17)	0.95111 (13)	0.6986 (4)	0.0893 (7)
O2	0.82021 (17)	0.84566 (15)	0.6268 (4)	0.0855 (7)
O3	0.28825 (13)	0.66909 (13)	0.5328 (3)	0.0609 (5)
Br1	0.67373 (2)	0.50413 (2)	0.68364 (4)	0.05714 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0357 (11)	0.0396 (11)	0.0430 (12)	0.0034 (9)	0.0066 (9)	0.0003 (9)
C2	0.0313 (10)	0.0390 (10)	0.0328 (10)	−0.0007 (8)	0.0047 (8)	0.0004 (8)
C3	0.0302 (10)	0.0454 (11)	0.0365 (11)	0.0004 (8)	0.0065 (8)	0.0003 (9)
C4	0.0357 (11)	0.0434 (12)	0.0389 (12)	−0.0082 (9)	0.0062 (9)	0.0030 (9)
C5	0.0427 (12)	0.0375 (11)	0.0412 (12)	0.0004 (9)	0.0045 (9)	−0.0010 (9)
C6	0.0336 (10)	0.0444 (11)	0.0377 (12)	0.0025 (9)	0.0078 (9)	−0.0023 (9)
C7	0.0292 (9)	0.0409 (11)	0.0317 (10)	−0.0026 (8)	0.0047 (8)	−0.0001 (8)
C8	0.0364 (11)	0.0502 (13)	0.0536 (14)	−0.0052 (9)	0.0169 (10)	−0.0009 (10)
C9	0.0358 (11)	0.0364 (11)	0.0567 (14)	0.0013 (9)	0.0123 (10)	0.0001 (10)

C10	0.0331 (12)	0.0498 (14)	0.089 (2)	0.0041 (10)	0.0141 (12)	0.0091 (13)
C11	0.0444 (16)	0.116 (3)	0.102 (3)	0.0016 (17)	0.0338 (17)	0.014 (2)
C12	0.055 (2)	0.144 (4)	0.158 (4)	0.005 (2)	0.052 (3)	0.032 (3)
C13	0.043 (2)	0.164 (5)	0.213 (6)	0.000 (2)	0.031 (3)	0.046 (4)
C14	0.045 (2)	0.344 (11)	0.163 (6)	−0.003 (4)	−0.016 (3)	0.048 (7)
C15	0.0454 (19)	0.233 (6)	0.112 (3)	−0.008 (3)	−0.006 (2)	0.034 (4)
N1	0.0450 (12)	0.0539 (13)	0.0596 (13)	−0.0143 (10)	0.0119 (10)	0.0035 (10)
N2	0.0395 (10)	0.0397 (10)	0.0541 (12)	−0.0036 (8)	0.0099 (8)	−0.0005 (8)
N3	0.0315 (9)	0.0421 (10)	0.0499 (11)	−0.0028 (7)	0.0114 (8)	−0.0003 (8)
O1	0.0766 (14)	0.0484 (12)	0.150 (2)	−0.0197 (10)	0.0394 (14)	0.0016 (13)
O2	0.0570 (12)	0.0768 (14)	0.134 (2)	−0.0170 (11)	0.0492 (13)	−0.0045 (14)
O3	0.0487 (10)	0.0787 (13)	0.0566 (11)	0.0044 (9)	0.0123 (8)	0.0108 (9)
Br1	0.05290 (17)	0.04388 (16)	0.0780 (2)	0.00958 (10)	0.02077 (14)	−0.00060 (12)

Geometric parameters (Å, º)

C1—N2	1.309 (3)	C8—H8B	0.9700
C1—C2	1.415 (3)	C9—O3	1.209 (3)
C1—Br1	1.867 (2)	C9—C10	1.488 (3)
C2—C3	1.389 (3)	C10—C15	1.352 (5)
C2—C7	1.404 (3)	C10—C11	1.372 (4)
C3—C4	1.373 (3)	C11—C12	1.381 (4)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.405 (3)	C12—C13	1.333 (7)
C4—N1	1.467 (3)	C12—H12	0.9300
C5—C6	1.369 (3)	C13—C14	1.332 (8)
C5—H5	0.9300	C13—H13	0.9300
C6—C7	1.408 (3)	C14—C15	1.427 (7)
C6—H6	0.9300	C14—H14	0.9300
C7—N3	1.361 (3)	C15—H15	0.9300
C8—N3	1.446 (3)	N1—O1	1.203 (3)
C8—C9	1.513 (3)	N1—O2	1.208 (3)
C8—H8A	0.9700	N2—N3	1.369 (3)
N2—C1—C2	112.99 (18)	O3—C9—C8	120.5 (2)
N2—C1—Br1	120.19 (16)	C10—C9—C8	116.7 (2)
C2—C1—Br1	126.77 (16)	C15—C10—C11	119.3 (3)
C3—C2—C7	120.79 (19)	C15—C10—C9	118.0 (3)
C3—C2—C1	135.80 (19)	C11—C10—C9	122.6 (3)
C7—C2—C1	103.41 (17)	C10—C11—C12	121.5 (4)
C4—C3—C2	116.08 (18)	C10—C11—H11	119.2
C4—C3—H3	122.0	C12—C11—H11	119.2
C2—C3—H3	122.0	C13—C12—C11	118.8 (4)
C3—C4—C5	124.05 (19)	C13—C12—H12	120.6
C3—C4—N1	117.8 (2)	C11—C12—H12	120.6
C5—C4—N1	118.2 (2)	C14—C13—C12	121.8 (4)
C6—C5—C4	120.2 (2)	C14—C13—H13	119.1
C6—C5—H5	119.9	C12—C13—H13	119.1

C4—C5—H5	119.9	C13—C14—C15	120.2 (5)
C5—C6—C7	116.8 (2)	C13—C14—H14	119.9
C5—C6—H6	121.6	C15—C14—H14	119.9
C7—C6—H6	121.6	C10—C15—C14	118.3 (5)
N3—C7—C2	106.83 (18)	C10—C15—H15	120.8
N3—C7—C6	131.08 (19)	C14—C15—H15	120.8
C2—C7—C6	122.09 (19)	O1—N1—O2	122.9 (2)
N3—C8—C9	112.68 (19)	O1—N1—C4	118.6 (2)
N3—C8—H8A	109.1	O2—N1—C4	118.5 (2)
C9—C8—H8A	109.1	C1—N2—N3	105.19 (17)
N3—C8—H8B	109.1	C7—N3—N2	111.57 (16)
C9—C8—H8B	109.1	C7—N3—C8	129.35 (19)
H8A—C8—H8B	107.8	N2—N3—C8	119.08 (18)
O3—C9—C10	122.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O3^{i}	0.97	2.40	3.315 (3)	157
C8—H8*B*···O1^{ii}	0.97	2.53	3.244 (3)	131
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