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5-Nitro-1-(prop-2-en-1-yl)-1*H*-indazole

Mohammed Boulhaoua,[a]* Abdelhanine Essaghouani,[a] Sanae Lahmidi,[a] Mohammed Benchidmi,[a] El Mokhtar Essassi[a] and Joel T. Mague[b]

[a]Laboratoire de Chimie Organique Hétérocyclique, URAC 21, Pôle de Compétence Pharmacochimie, Av Ibn Battouta, BP 1014, Faculté des Sciences, Université Mohammed V, Rabat, Morocco, and [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA. *Correspondence e-mail: mboulhaoua@gmail.com

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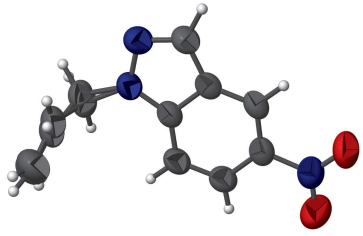
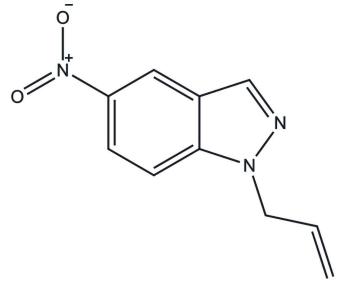
Structural data: full structural data are available from iucrdata.iucr.org

In the crystal, the title molecule, $C_{10}H_9N_3O_2$, packs in layers approximately parallel to (100), which are formed by the association of zigzag chains constructed by weak C—H···O interactions. The allyl group is disordered over two positions, with a ratio of their occupancies close to 70:30.

3D view



Chemical scheme



Structure description

As a continuation of our research work on indazole derivatives (Boulhaoua *et al.*, 2016), we have studied the action of allyl bromide towards 5-nitro-1*H*-indazole under phase-transfer catalysis conditions, by using tetra-*n*-butylammonium bromide (TBAB) as catalyst and potassium carbonate as base. This readily leads to the title compound (Fig. 1) in good yield.

In the crystal, the molecule forms zigzag chains running parallel to the *c* axis through weak intermolecular C7—H7···O1^{i} hydrogen bonds (Table 1 and Fig. 2). The chains pack so as to form layers approximately parallel to (100) aided by weak C9*A*—H9*A*···O1^{ii} interactions (Table 1 and Fig. 3).

Synthesis and crystallization

To a solution of 5-nitro-1*H*-indazole (0.5 g, 3 mmol) in THF (25 ml) was added allyl bromide (0.26 ml, 3 mmol), potassium carbonate (0.83 g, 6 mmol) and a catalytic amount 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 resulting residue was purified by column chromatography (EtOAc/hexane, 2/8). The title compound was obtained as colourless crystals in 70% yield.

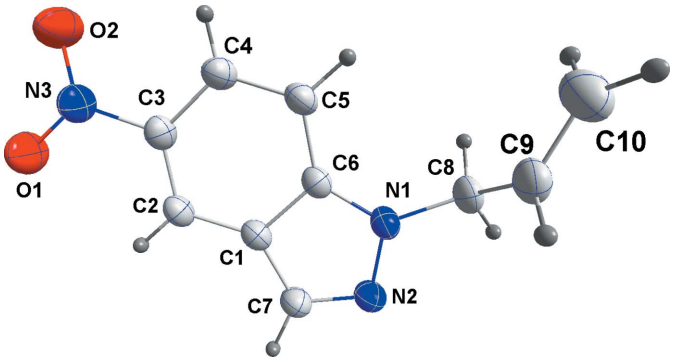


Figure 1
The title molecule with labelling scheme and 50% probability ellipsoids. Only one orientation of the disordered allyl group is shown.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The allyl group is disordered over two partially resolved orientations for which occupancies converged toward 0.702 (12) and 0.298 (12). The two components of the disorder were refined subject to restraints that their geometries be comparable (*SADI* command in *SHELXL;* Sheldrick, 2015*b*) and pairs of C atoms were constrained to have identical displacement parameters (*EADP* command in *SHELXL;* Sheldrick, 2015*b*). The absolute configuration could not be determined with certainty, and was thus assigned arbitrarily.

Acknowledgements

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References

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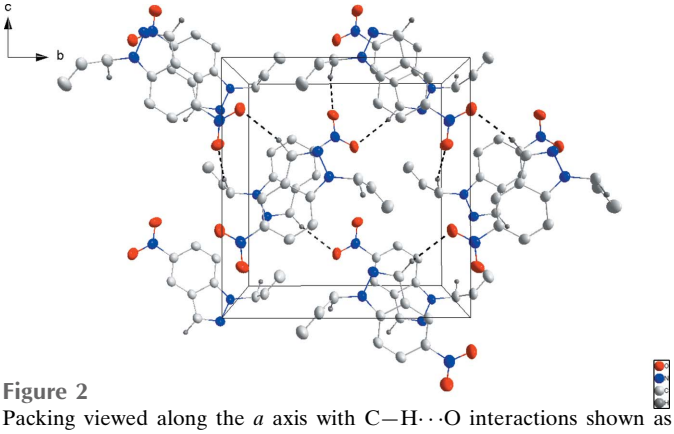

Figure 2
Packing viewed along the *a* axis with C—H···O interactions shown as dotted lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1^{i}	1.00 (2)	2.56 (3)	3.552 (3)	168 (2)
C9*A*—H9*A*···O1^{ii}	0.93	2.55	3.404 (17)	154

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_9N_3O_2$
M_r	203.20
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	298
a, *b*, *c* (Å)	6.9549 (5), 11.6778 (9), 12.2165 (9)
V (Å^3)	992.20 (13)
Z	4
Radiation type	Cu *Kα*
μ (mm^{-1})	0.82
Crystal size (mm)	0.22 × 0.17 × 0.15
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.80, 0.89
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7734, 1937, 1806
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.034, 0.098, 1.05
No. of reflections	1937
No. of parameters	164
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.13, −0.12

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

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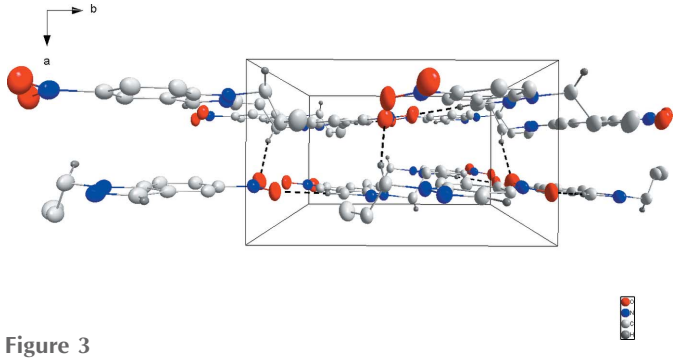

Figure 3
Packing viewed along the *c* axis with C—H···O interactions shown as dotted lines.

full crystallographic data

IUCrData (2017). **2**, x171091 [https://doi.org/10.1107/S2414314617010914]

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Crystal data

$C_{10}H_9N_3O_2$
M_r = 203.20
Orthorhombic, $P2_12_12_1$
a = 6.9549 (5) Å
b = 11.6778 (9) Å
c = 12.2165 (9) Å
V = 992.20 (13) Å^3
Z = 4
$F(000)$ = 424
D_x = 1.360 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 6584 reflections
θ = 3.8–72.3°
μ = 0.82 mm^{-1}
T = 298 K
Block, colourless
0.22 × 0.17 × 0.15 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.80, T_{max} = 0.89
7734 measured reflections
1937 independent reflections
1806 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 72.3°, θ_{min} = 5.2°
h = −8→8
k = −14→14
l = −13→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.098
S = 1.05
1937 reflections
164 parameters
4 restraints
3 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.0423P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015*b*), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Extinction coefficient: 0.013 (2)

Special details

Refinement. The H-atoms of the disordered allyl group were placed in calculated positions (C—H = 0.93–0.97 Å) while the remainder were refined independently.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.7579 (3)	−0.04269 (14)	0.31969 (17)	0.0971 (7)	
O2	0.7999 (4)	0.05941 (18)	0.17506 (16)	0.1030 (6)	
N1	0.8126 (3)	0.42834 (13)	0.54140 (14)	0.0615 (4)	
N2	0.8021 (3)	0.37982 (15)	0.64367 (15)	0.0675 (5)	
N3	0.7819 (3)	0.04995 (16)	0.27451 (17)	0.0733 (5)	
C1	0.7894 (2)	0.24152 (15)	0.51404 (15)	0.0532 (4)	
C2	0.7815 (3)	0.14109 (14)	0.45294 (16)	0.0554 (4)	
H2	0.775 (3)	0.068 (2)	0.487 (2)	0.066 (6)*	
C3	0.7888 (3)	0.15275 (16)	0.34145 (17)	0.0590 (5)	
C4	0.8024 (3)	0.25941 (17)	0.28784 (18)	0.0641 (5)	
H4	0.812 (4)	0.263 (2)	0.212 (2)	0.079 (7)*	
C5	0.8087 (3)	0.35883 (16)	0.34732 (17)	0.0629 (5)	
H5	0.819 (4)	0.433 (2)	0.313 (2)	0.074 (6)*	
C6	0.8037 (3)	0.34871 (14)	0.46140 (15)	0.0544 (4)	
C7	0.7889 (3)	0.26889 (17)	0.62715 (18)	0.0631 (5)	
H7	0.779 (4)	0.214 (2)	0.690 (2)	0.074 (7)*	
C8	0.8333 (11)	0.5528 (2)	0.5382 (7)	0.0642 (15)	0.702 (12)
H8A	0.942664	0.572027	0.492453	0.077*	0.702 (12)
H8B	0.860253	0.580262	0.611529	0.077*	0.702 (12)
C9	0.6597 (12)	0.6133 (10)	0.4954 (7)	0.0727 (11)	0.702 (12)
H9	0.541851	0.598228	0.528387	0.087*	0.702 (12)
C10	0.6638 (13)	0.6853 (5)	0.4153 (5)	0.1027 (18)	0.702 (12)
H10A	0.779679	0.702051	0.380736	0.123*	0.702 (12)
H10B	0.551014	0.720502	0.392042	0.123*	0.702 (12)
C8A	0.841 (3)	0.5494 (5)	0.516 (2)	0.0642 (15)	0.298 (12)
H8AA	0.898139	0.558371	0.443829	0.077*	0.298 (12)
H8AB	0.924583	0.585220	0.569494	0.077*	0.298 (12)
C9A	0.645 (3)	0.601 (3)	0.5191 (18)	0.0727 (11)	0.298 (12)
H9A	0.570403	0.588830	0.581261	0.087*	0.298 (12)
C10A	0.572 (3)	0.6615 (13)	0.4409 (14)	0.1027 (18)	0.298 (12)
H10C	0.643556	0.675116	0.377733	0.123*	0.298 (12)
H10D	0.449119	0.691462	0.447651	0.123*	0.298 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1346 (18)	0.0541 (8)	0.1026 (13)	0.0013 (10)	0.0014 (12)	−0.0084 (8)
O2	0.1418 (17)	0.0913 (12)	0.0761 (12)	0.0089 (13)	0.0128 (12)	−0.0165 (9)
N1	0.0684 (9)	0.0501 (8)	0.0660 (9)	−0.0022 (7)	−0.0033 (8)	0.0038 (7)
N2	0.0756 (10)	0.0653 (9)	0.0617 (9)	−0.0027 (9)	−0.0005 (8)	0.0062 (7)
N3	0.0759 (11)	0.0638 (10)	0.0803 (12)	0.0082 (9)	0.0040 (10)	−0.0082 (8)

C1	0.0464 (8)	0.0517 (8)	0.0616 (10)	−0.0013 (7)	0.0005 (7)	0.0099 (7)
C2	0.0509 (9)	0.0475 (8)	0.0678 (11)	0.0026 (7)	0.0023 (8)	0.0097 (7)
C3	0.0562 (9)	0.0524 (9)	0.0684 (12)	0.0027 (8)	0.0010 (8)	0.0003 (7)
C4	0.0703 (11)	0.0632 (11)	0.0587 (11)	0.0015 (11)	0.0003 (9)	0.0084 (8)
C5	0.0720 (12)	0.0545 (9)	0.0624 (11)	−0.0032 (10)	−0.0012 (9)	0.0129 (8)
C6	0.0498 (8)	0.0500 (8)	0.0636 (10)	0.0009 (8)	−0.0021 (8)	0.0071 (7)
C7	0.0671 (11)	0.0590 (9)	0.0632 (11)	−0.0014 (9)	0.0022 (9)	0.0103 (8)
C8	0.0749 (14)	0.0489 (9)	0.069 (4)	−0.0057 (9)	0.002 (2)	−0.0027 (11)
C9	0.084 (2)	0.050 (3)	0.084 (4)	0.0044 (18)	−0.002 (2)	−0.001 (2)
C10	0.130 (5)	0.080 (3)	0.099 (4)	0.013 (3)	−0.020 (4)	0.016 (2)
C8A	0.0749 (14)	0.0489 (9)	0.069 (4)	−0.0057 (9)	0.002 (2)	−0.0027 (11)
C9A	0.084 (2)	0.050 (3)	0.084 (4)	0.0044 (18)	−0.002 (2)	−0.001 (2)
C10A	0.130 (5)	0.080 (3)	0.099 (4)	0.013 (3)	−0.020 (4)	0.016 (2)

Geometric parameters (Å, º)

O1—N3	1.226 (3)	C5—H5	0.96 (3)
O2—N3	1.226 (3)	C7—H7	1.00 (2)
N1—C6	1.351 (3)	C8—C9	1.493 (4)
N1—N2	1.374 (3)	C8—H8A	0.9700
N1—C8	1.461 (3)	C8—H8B	0.9700
N1—C8A	1.461 (3)	C9—C10	1.291 (5)
N2—C7	1.314 (3)	C9—H9	0.9300
N3—C3	1.453 (3)	C10—H10A	0.9300
C1—C2	1.391 (3)	C10—H10B	0.9300
C1—C6	1.411 (2)	C8A—C9A	1.493 (5)
C1—C7	1.418 (3)	C8A—H8AA	0.9700
C2—C3	1.370 (3)	C8A—H8AB	0.9700
C2—H2	0.95 (2)	C9A—C10A	1.291 (6)
C3—C4	1.410 (3)	C9A—H9A	0.9300
C4—C5	1.370 (3)	C10A—H10C	0.9300
C4—H4	0.93 (3)	C10A—H10D	0.9300
C5—C6	1.399 (3)		
C6—N1—N2	111.81 (15)	N2—C7—C1	111.81 (17)
C6—N1—C8	132.1 (4)	N2—C7—H7	120.9 (15)
N2—N1—C8	116.1 (4)	C1—C7—H7	127.2 (15)
C6—N1—C8A	121.2 (11)	N1—C8—C9	113.6 (6)
N2—N1—C8A	126.9 (11)	N1—C8—H8A	108.8
C7—N2—N1	105.70 (18)	C9—C8—H8A	108.8
O1—N3—O2	122.6 (2)	N1—C8—H8B	108.8
O1—N3—C3	118.70 (19)	C9—C8—H8B	108.8
O2—N3—C3	118.7 (2)	H8A—C8—H8B	107.7
C2—C1—C6	120.41 (17)	C10—C9—C8	123.7 (4)
C2—C1—C7	135.45 (17)	C10—C9—H9	118.1
C6—C1—C7	104.14 (16)	C8—C9—H9	118.1
C3—C2—C1	116.64 (16)	C9—C10—H10A	120.0
C3—C2—H2	121.9 (15)	C9—C10—H10B	120.0

C1—C2—H2	121.4 (15)	H10A—C10—H10B	120.0
C2—C3—C4	123.50 (19)	N1—C8A—C9A	105.1 (14)
C2—C3—N3	118.44 (17)	N1—C8A—H8AA	110.7
C4—C3—N3	118.06 (19)	C9A—C8A—H8AA	110.7
C5—C4—C3	120.27 (19)	N1—C8A—H8AB	110.7
C5—C4—H4	119.3 (16)	C9A—C8A—H8AB	110.7
C3—C4—H4	120.4 (17)	H8AA—C8A—H8AB	108.8
C4—C5—C6	117.12 (17)	C10A—C9A—C8A	123.9 (8)
C4—C5—H5	122.1 (15)	C10A—C9A—H9A	118.0
C6—C5—H5	120.8 (15)	C8A—C9A—H9A	118.0
N1—C6—C5	131.41 (17)	C9A—C10A—H10C	120.0
N1—C6—C1	106.52 (16)	C9A—C10A—H10D	120.0
C5—C6—C1	122.06 (17)	H10C—C10A—H10D	120.0
C6—N1—N2—C7	0.8 (2)	C8—N1—C6—C1	178.2 (4)
C8—N1—N2—C7	−178.5 (4)	C8A—N1—C6—C1	175.9 (9)
C8A—N1—N2—C7	−175.8 (9)	C4—C5—C6—N1	178.0 (2)
C6—C1—C2—C3	0.1 (2)	C4—C5—C6—C1	−1.0 (3)
C7—C1—C2—C3	−179.0 (2)	C2—C1—C6—N1	−178.67 (16)
C1—C2—C3—C4	−0.4 (3)	C7—C1—C6—N1	0.7 (2)
C1—C2—C3—N3	179.75 (16)	C2—C1—C6—C5	0.6 (3)
O1—N3—C3—C2	4.4 (3)	C7—C1—C6—C5	179.95 (18)
O2—N3—C3—C2	−175.7 (2)	N1—N2—C7—C1	−0.3 (3)
O1—N3—C3—C4	−175.5 (2)	C2—C1—C7—N2	179.0 (2)
O2—N3—C3—C4	4.5 (3)	C6—C1—C7—N2	−0.2 (2)
C2—C3—C4—C5	−0.1 (3)	C6—N1—C8—C9	70.0 (8)
N3—C3—C4—C5	179.8 (2)	N2—N1—C8—C9	−110.9 (6)
C3—C4—C5—C6	0.8 (3)	N1—C8—C9—C10	−126.0 (11)
N2—N1—C6—C5	179.9 (2)	C6—N1—C8A—C9A	98.4 (17)
C8—N1—C6—C5	−1.0 (5)	N2—N1—C8A—C9A	−85.3 (16)
C8A—N1—C6—C5	−3.3 (9)	N1—C8A—C9A—C10A	−128 (3)
N2—N1—C6—C1	−0.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1^{i}	1.00 (2)	2.56 (3)	3.552 (3)	168 (2)
C9*A*—H9*A*···O1^{ii}	0.93	2.55	3.404 (17)	154

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