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1-Allyl-3-chloro-5-nitro-1*H*-indazole

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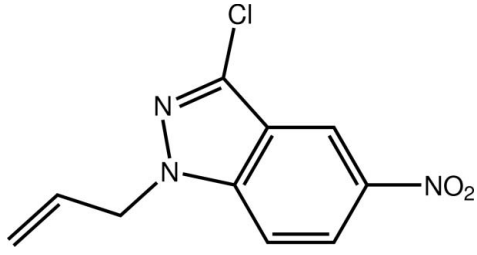
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.136; data-to-parameter ratio = 21.2.

In the title compound, $C_{10}H_8ClN_3O_2$, the indazole ring system makes a dihedral angle of 7.9 (3)° with the plane through the nitro group. The allyl group is rotated out of the plane of the indazole ring system [N—N—C—C torsion angle = 104.28 (19)°]. In the crystal, molecules are linked by C—H···O hydrogen bonds, forming zigzag chains propagating along the b-axis direction.

Related literature

For the pharmacological activity of indazole derivatives, see: Baraldi *et al.* (2001); Rodgers *et al.* (1996); Li *et al.* (2003); Lin *et al.* (2008). For a similar compound, see: El Brahmi *et al.* (2012).



Experimental

Crystal data

$C_{10}H_8ClN_3O_2$
M_r = 237.64
Monoclinic, $P2_1/c$
a = 13.3025 (6) Å
b = 11.2505 (5) Å
c = 7.3092 (3) Å
β = 91.343 (2)°
V = 1093.59 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 296 K
0.41 × 0.34 × 0.22 mm

Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*SADABS*: Sheldrick, 2008)
T_{min} = 0.654, T_{max} = 0.747
14430 measured reflections
3069 independent reflections
1852 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
S = 1.02
3069 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O2^{i}	0.93	2.46	3.274 (2)	146

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

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: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6927).

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supporting information

Acta Cryst. (2013). E**69**, o1410 [doi:10.1107/S1600536813021995]

1-Allyl-3-chloro-5-nitro-1*H*-indazole

Hakima Chicha, El Mostapha Rakib, Domenico Spinelli, Mohamed Saadi and Lahcen El Ammari

S1. Comment

Indazole derivatives are an important class of heterocyclic pharmaceuticals because of their significant and broad spectrum of biological properties, including antitumor, anti-HIV, antimicrobial, anti-inflammatory, and contraceptive activities (Baraldi *et al.*, 2001; Rodgers *et al.*, 1996; Li *et al.*, 2003; Lin *et al.*, 2008). The present work is a contribution to the investigation of indazole derivatives (El Brahmi *et al.*, 2012).

In the molecule of 1-allyl-3-chloro-5-nitro-1*H*-indazole, the dihedral angle between the indazole system and the plan through the atoms forming the nitro group is of 7.9 (3)° and it is nearly perpendicular to the allyl group as indicated by the dihedral angle of 80.8 (3)°.

In the crystal, the molecules are interconnected by C–H···O hydrogen bonds forming zigzag chains running along the *b* axis as shown in Fig. 2 and Table 2.

S2. Experimental

To a solution of 3-chloro-5-nitroindazole (6.13 mmol) in acetone (15 ml) was added potassium hydroxide (6.8 mmol). After 15 mn at 298 K, allyl bromide (12.26 mmol) was added dropwise. Upon disappearance of the starting material as indicated by TLC, the resulting mixture was evaporated. The crude material was dissolved with EtOAc (50 ml), washed with water and brine, dried over $MgSO_4$ and the solvent was evaporated *in vacuo*. The resulting residue was purified by column chromatography (EtOAc/hexane 3/7). The title compound was recrystallized from ethanol.

S3. Refinement

H atoms were located in a difference map and treated as riding with C–H = 0.97 Å, and C–H = 0.93 Å for methylene and aromatic H atoms, respectively. U(H) was set to $1.2U_{iso}$(C).

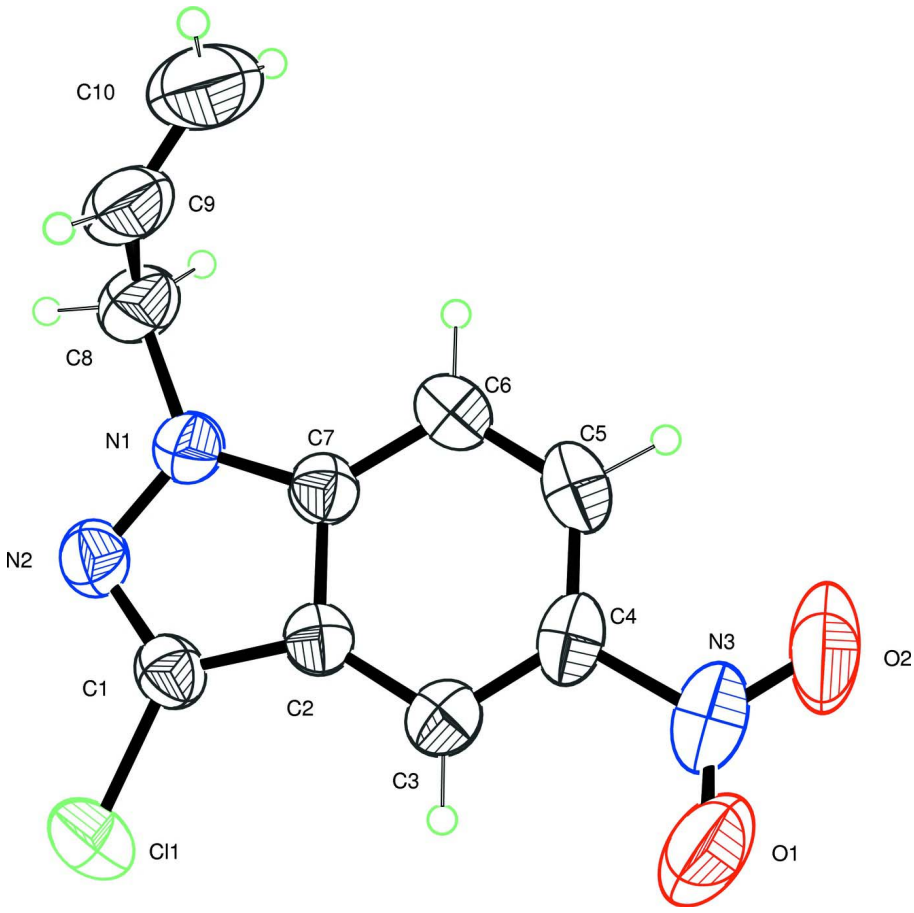

Figure 1

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

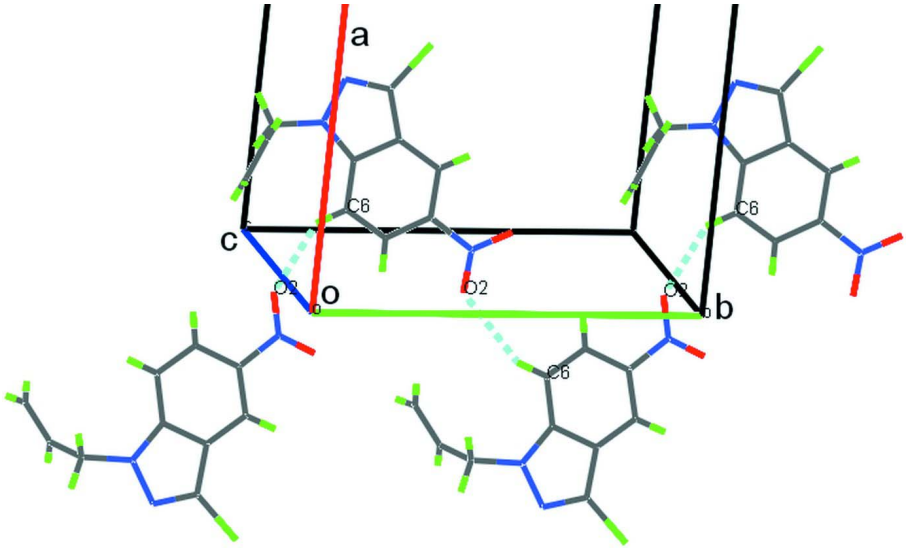

Figure 2

Partial crystal packing for the title compound showing C–H···O hydrogen bonds as dashed lines, showing a zigzag chain running along the *b* axis.

1-Allyl-3-chloro-5-nitro-1*H*-indazole

Crystal data

$C_{10}H_8ClN_3O_2$
M_r = 237.64
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.3025 (6) Å
b = 11.2505 (5) Å
c = 7.3092 (3) Å
β = 91.343 (2)°
V = 1093.59 (8) Å^3
Z = 4
F(000) = 488
D_x = 1.443 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3069 reflections
θ = 2.4–29.6°
μ = 0.34 mm^{-1}
T = 296 K
Block, colourless
0.41 × 0.34 × 0.22 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*: Sheldrick, 2008)
T_{min} = 0.654, T_{max} = 0.747
14430 measured reflections
3069 independent reflections
1852 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 29.6°, θ_{min} = 2.4°
$h = -18 \rightarrow 18$
$k = -10 \rightarrow 15$
$l = -9 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
S = 1.02
3069 reflections
145 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 0.0517P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against all reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on all data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.39644 (11)	0.25338 (16)	1.0420 (2)	0.0451 (4)
C2	0.29681 (11)	0.22952 (14)	0.97697 (19)	0.0415 (4)
C3	0.24119 (12)	0.12846 (15)	0.9339 (2)	0.0462 (4)
H3	0.2681	0.0525	0.9450	0.055*
C4	0.14423 (13)	0.14758 (16)	0.8741 (2)	0.0517 (4)
C5	0.10115 (12)	0.26115 (18)	0.8562 (2)	0.0549 (5)
H5	0.0346	0.2685	0.8160	0.066*
C6	0.15549 (13)	0.36043 (16)	0.8970 (2)	0.0508 (4)
H6	0.1278	0.4360	0.8848	0.061*
C7	0.25499 (12)	0.34380 (14)	0.9583 (2)	0.0428 (4)
C8	0.32262 (15)	0.55248 (16)	1.0068 (3)	0.0596 (5)
H8A	0.3794	0.5846	1.0763	0.071*
H8B	0.2616	0.5785	1.0650	0.071*
C9	0.32387 (17)	0.59986 (17)	0.8194 (3)	0.0666 (5)
H9	0.3814	0.5866	0.7526	0.080*
C10	0.2520 (2)	0.6582 (2)	0.7404 (5)	0.1088 (10)
H10A	0.1931	0.6734	0.8025	0.131*
H10B	0.2588	0.6852	0.6210	0.131*
N1	0.32739 (10)	0.42252 (13)	1.01153 (19)	0.0492 (4)
N2	0.41482 (10)	0.36715 (13)	1.06173 (19)	0.0503 (4)
N3	0.08324 (14)	0.04395 (18)	0.8226 (2)	0.0720 (5)
O1	0.12321 (14)	−0.05345 (16)	0.8167 (3)	0.0968 (6)
O2	−0.00516 (13)	0.06089 (18)	0.7844 (3)	0.1118 (7)
Cl1	0.48743 (3)	0.15046 (5)	1.08995 (7)	0.0677 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0412 (8)	0.0522 (10)	0.0417 (8)	0.0014 (7)	−0.0009 (6)	0.0049 (7)
C2	0.0419 (8)	0.0462 (9)	0.0364 (7)	−0.0014 (7)	0.0006 (6)	0.0022 (7)
C3	0.0520 (10)	0.0463 (9)	0.0404 (8)	−0.0039 (7)	0.0034 (7)	0.0008 (7)
C4	0.0497 (10)	0.0619 (12)	0.0435 (8)	−0.0181 (8)	0.0021 (7)	−0.0055 (8)

C5	0.0388 (9)	0.0757 (13)	0.0500 (10)	−0.0016 (9)	−0.0047 (7)	−0.0016 (9)
C6	0.0458 (9)	0.0567 (11)	0.0498 (9)	0.0071 (8)	−0.0024 (7)	0.0017 (8)
C7	0.0428 (8)	0.0458 (9)	0.0398 (8)	−0.0012 (7)	−0.0008 (6)	0.0017 (7)
C8	0.0678 (12)	0.0427 (10)	0.0679 (12)	−0.0045 (8)	−0.0037 (9)	−0.0069 (8)
C9	0.0795 (14)	0.0424 (11)	0.0778 (13)	−0.0081 (10)	−0.0029 (11)	0.0029 (10)
C10	0.139 (3)	0.0582 (14)	0.127 (2)	−0.0137 (14)	−0.056 (2)	0.0176 (14)
N1	0.0485 (8)	0.0440 (8)	0.0548 (8)	−0.0036 (6)	−0.0071 (6)	0.0020 (6)
N2	0.0438 (8)	0.0544 (9)	0.0523 (8)	−0.0044 (6)	−0.0057 (6)	0.0045 (7)
N3	0.0667 (11)	0.0798 (13)	0.0694 (11)	−0.0279 (10)	0.0027 (9)	−0.0118 (10)
O1	0.0979 (12)	0.0673 (11)	0.1254 (15)	−0.0298 (10)	0.0049 (11)	−0.0242 (10)
O2	0.0639 (10)	0.1174 (15)	0.1528 (18)	−0.0334 (10)	−0.0243 (10)	−0.0240 (12)
Cl1	0.0516 (3)	0.0709 (4)	0.0803 (4)	0.0142 (2)	−0.0084 (2)	0.0080 (2)

Geometric parameters (Å, º)

C1—N2	1.310 (2)	C7—N1	1.359 (2)
C1—C2	1.423 (2)	C8—N1	1.464 (2)
C1—Cl1	1.7056 (17)	C8—C9	1.470 (3)
C2—C3	1.389 (2)	C8—H8A	0.9700
C2—C7	1.406 (2)	C8—H8B	0.9700
C3—C4	1.369 (2)	C9—C10	1.286 (3)
C3—H3	0.9300	C9—H9	0.9300
C4—C5	1.405 (3)	C10—H10A	0.9300
C4—N3	1.465 (2)	C10—H10B	0.9300
C5—C6	1.360 (2)	N1—N2	1.3621 (19)
C5—H5	0.9300	N3—O2	1.217 (2)
C6—C7	1.400 (2)	N3—O1	1.219 (2)
C6—H6	0.9300		
N2—C1—C2	113.02 (14)	C6—C7—C2	121.48 (15)
N2—C1—Cl1	120.71 (12)	N1—C8—C9	112.52 (16)
C2—C1—Cl1	126.27 (14)	N1—C8—H8A	109.1
C3—C2—C7	121.26 (15)	C9—C8—H8A	109.1
C3—C2—C1	135.84 (16)	N1—C8—H8B	109.1
C7—C2—C1	102.90 (14)	C9—C8—H8B	109.1
C4—C3—C2	115.89 (16)	H8A—C8—H8B	107.8
C4—C3—H3	122.1	C10—C9—C8	125.4 (3)
C2—C3—H3	122.1	C10—C9—H9	117.3
C3—C4—C5	123.49 (16)	C8—C9—H9	117.3
C3—C4—N3	117.96 (18)	C9—C10—H10A	120.0
C5—C4—N3	118.54 (17)	C9—C10—H10B	120.0
C6—C5—C4	120.86 (16)	H10A—C10—H10B	120.0
C6—C5—H5	119.6	C7—N1—N2	111.97 (13)
C4—C5—H5	119.6	C7—N1—C8	127.90 (15)
C5—C6—C7	117.01 (16)	N2—N1—C8	119.96 (14)
C5—C6—H6	121.5	C1—N2—N1	105.13 (13)
C7—C6—H6	121.5	O2—N3—O1	123.54 (19)
N1—C7—C6	131.54 (16)	O2—N3—C4	117.4 (2)

N1—C7—C2	106.97 (14)	O1—N3—C4	119.02 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.93	2.46	3.274 (2)	146

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