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3-Chloro-1-ethyl-6-nitro-1*H*-indazole

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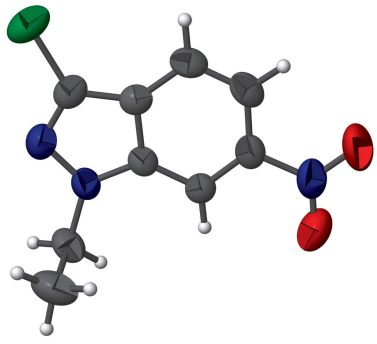
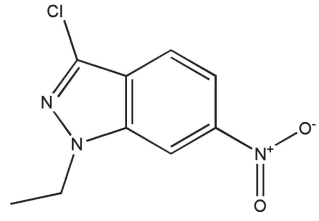
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_9H_8ClN_3O_2$, the terminal C atom of the ethyl group deviates from the indazole ring (r.m.s. deviation = 0.008 Å) by 1.588 (3) Å. The dihedral angle between the ring system and the attached nitro group is 2.8 (3)°. In the crystal, weak C—H···O interactions link the molecules into zigzag chains propagating along [001]. In addition, weak π–π stacking interactions [centroid–centroid separations = 3.6809 (10) and 3.7393 (11) Å] help to consolidate the packing.

3D view



Chemical scheme



Structure description

As a continuation of our studies on indazole derivatives (Mohamed Abdelahi *et al.*, 2017), we report here the synthesis and crystal structure of the title compound, $C_9H_8ClN_3O_2$.

The molecular structure of the title compound is illustrated in Fig. 1. Apart from the terminal carbon atom (C9) of the ethyl moiety, it is essentially planar, as evident from the dihedral angle between the indazole ring plane and nitro group of 2.8 (3)°. Atom C9 deviates from the ring plane by 1.588 (3) Å.

In the crystal, a weak C4—H4···O2^{i} interaction (Table 1) links the molecules into zigzag chains propagating along the *c*-axis direction (Fig. 2). In addition, weak π–π stacking interactions are observed [*Cg*1···*Cg*2^{ii} = 3.6809 (10) Å, *Cg*2···*Cg*2^{ii} = 3.7393 (11) Å, where *Cg*1 is the centroid of the N1/N2/C7/C2/C1 ring and *Cg*2 is the centroid of the C2–C7 ring; symmetry code: (ii) $2-x, -y, 2-z$].

Synthesis and crystallization

To a solution of 6-nitro-1*H*-indazole (0.8 g, 5 mmol) in tetrahydrofuran (30 ml) were added bromoethane (0.8 g, 5 mmol), potassium carbonate (1.24 g, 9 mmol) and a cata-

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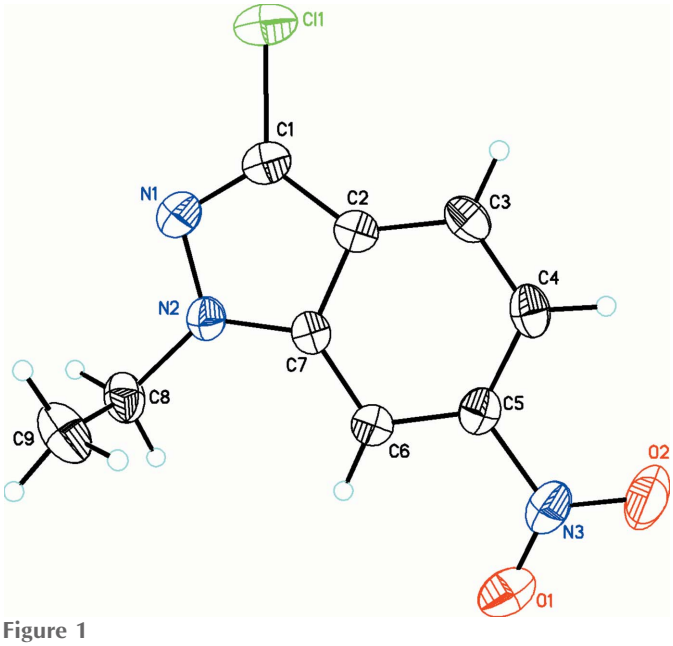

Figure 1
The molecular structure of the title compound, with atom labelling and 30% probability displacement ellipsoids.

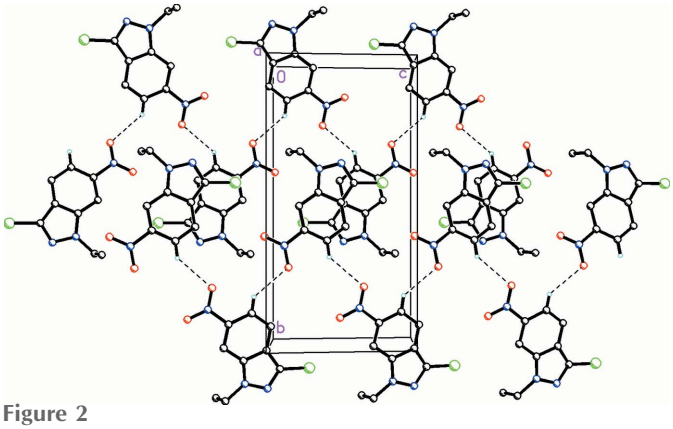

Figure 2
The packing viewed along the *a*-axis direction. Dashed lines indicate weak C—H···O interactions linking the molecules into [001] zigzag chains. H atoms not involved in the hydrogen bonds have been omitted for clarity.

lytic quantity of tetra-*n*-butylammonium iodide. 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 ethanol solution to afford yellow plates of the title compound (yield: 68%).

Refinement

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O2^{i}	0.93	2.52	3.280 (3)	139

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_8ClN_3O_2$
M_r	225.63
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	7.4984 (3), 16.2805 (7), 8.3363 (3)
β (°)	97.403 (4)
V (Å^3)	1009.19 (7)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	3.24
Crystal size (mm)	0.22 × 0.20 × 0.06
Data collection	
Diffractometer	Rigaku Oxford diffraction
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.493, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3567, 1916, 1572
R_{int}	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.116, 1.03
No. of reflections	1916
No. of parameters	137
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.23

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170972 [https://doi.org/10.1107/S2414314617009725]

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Crystal data

$C_9H_8ClN_3O_2$
M_r = 225.63
Monoclinic, $P2_1/c$
a = 7.4984 (3) Å
b = 16.2805 (7) Å
c = 8.3363 (3) Å
β = 97.403 (4)°
V = 1009.19 (7) Å^3
Z = 4
$F(000)$ = 464
D_x = 1.485 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 1252 reflections
θ = 6.0–70.9°
μ = 3.24 mm^{-1}
T = 293 K
Plate, yellow
0.22 × 0.20 × 0.06 mm

Data collection

Rigaku Oxford diffraction diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.493, T_{max} = 1.000
3567 measured reflections
1916 independent reflections
1572 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 71.4°, θ_{min} = 5.4°
h = −7→9
k = −19→16
l = −9→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.116
S = 1.03
1916 reflections
137 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0591P)^2 + 0.080P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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.

Refinement. All the H atoms were placed in calculated positions and refined using the riding model with C—H bond lengths of 0.93 Å (CH), 0.97 Å (CH_2) or 0.96 Å (CH_3). Isotropic displacement parameters for these atoms were set to 1.2 (CH, CH_2) or 1.5 (CH_3) times U_{eq} of the parent atom. The methyl group was allowed to rotate, but not to tip, to best fit the electron density.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.34194 (8)	0.57002 (5)	0.22541 (6)	0.0741 (2)
O1	0.0801 (2)	0.38775 (13)	1.0161 (2)	0.0801 (5)
O2	0.0722 (3)	0.28363 (12)	0.8569 (3)	0.1041 (8)
N1	0.3183 (2)	0.63770 (11)	0.5077 (2)	0.0555 (4)
N2	0.2742 (2)	0.61772 (10)	0.6560 (2)	0.0506 (4)
N3	0.0963 (2)	0.35657 (12)	0.8854 (3)	0.0628 (5)
C1	0.2999 (2)	0.57001 (14)	0.4224 (2)	0.0516 (4)
C2	0.2448 (2)	0.50247 (12)	0.5101 (2)	0.0453 (4)
C3	0.2059 (2)	0.41950 (13)	0.4800 (3)	0.0528 (5)
H3	0.2124	0.3969	0.3785	0.063*
C4	0.1581 (3)	0.37243 (12)	0.6036 (3)	0.0556 (5)
H4	0.1329	0.3169	0.5876	0.067*
C5	0.1473 (2)	0.40879 (12)	0.7548 (2)	0.0487 (4)
C6	0.1812 (2)	0.48984 (12)	0.7903 (2)	0.0459 (4)
H6	0.1710	0.5121	0.8914	0.055*
C7	0.2320 (2)	0.53656 (11)	0.6633 (2)	0.0432 (4)
C8	0.3023 (3)	0.67764 (13)	0.7875 (3)	0.0585 (5)
H8A	0.2237	0.6646	0.8677	0.070*
H8B	0.2704	0.7319	0.7451	0.070*
C9	0.4930 (3)	0.67852 (18)	0.8667 (4)	0.0791 (7)
H9A	0.5700	0.6969	0.7905	0.119*
H9B	0.5276	0.6241	0.9027	0.119*
H9C	0.5038	0.7151	0.9577	0.119*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0669 (4)	0.1084 (5)	0.0482 (3)	0.0074 (3)	0.0124 (2)	0.0114 (3)
O1	0.0843 (12)	0.0871 (13)	0.0730 (11)	0.0025 (10)	0.0251 (9)	0.0224 (10)
O2	0.1397 (19)	0.0585 (11)	0.1091 (17)	−0.0275 (12)	−0.0026 (14)	0.0253 (11)
N1	0.0531 (9)	0.0547 (9)	0.0587 (9)	−0.0002 (7)	0.0078 (7)	0.0092 (8)
N2	0.0570 (9)	0.0410 (8)	0.0542 (9)	−0.0021 (7)	0.0091 (7)	−0.0008 (7)
N3	0.0548 (10)	0.0561 (10)	0.0754 (13)	−0.0035 (8)	0.0001 (8)	0.0195 (9)
C1	0.0413 (9)	0.0646 (12)	0.0490 (10)	0.0044 (8)	0.0054 (7)	0.0071 (9)
C2	0.0378 (8)	0.0516 (10)	0.0458 (9)	0.0060 (7)	0.0027 (7)	−0.0006 (8)
C3	0.0488 (10)	0.0549 (11)	0.0536 (11)	0.0079 (8)	0.0028 (8)	−0.0130 (9)
C4	0.0517 (10)	0.0405 (9)	0.0724 (13)	0.0040 (8)	−0.0001 (9)	−0.0066 (9)
C5	0.0408 (9)	0.0456 (9)	0.0583 (11)	0.0028 (7)	0.0015 (7)	0.0064 (8)
C6	0.0441 (9)	0.0474 (9)	0.0460 (9)	0.0023 (7)	0.0053 (7)	−0.0003 (7)
C7	0.0384 (8)	0.0418 (9)	0.0487 (9)	0.0020 (7)	0.0030 (6)	−0.0022 (7)

C8	0.0593 (12)	0.0447 (10)	0.0732 (13)	0.0006 (9)	0.0148 (9)	−0.0122 (9)
C9	0.0680 (14)	0.0823 (17)	0.0850 (17)	−0.0009 (13)	0.0020 (12)	−0.0333 (14)

Geometric parameters (Å, º)

Cl1—C1	1.712 (2)	C3—C4	1.369 (3)
O1—N3	1.222 (3)	C4—H4	0.9300
O2—N3	1.220 (3)	C4—C5	1.404 (3)
N1—N2	1.360 (2)	C5—C6	1.369 (3)
N1—C1	1.309 (3)	C6—H6	0.9300
N2—C7	1.362 (2)	C6—C7	1.396 (3)
N2—C8	1.462 (3)	C8—H8A	0.9700
N3—C5	1.470 (3)	C8—H8B	0.9700
C1—C2	1.411 (3)	C8—C9	1.496 (3)
C2—C3	1.398 (3)	C9—H9A	0.9600
C2—C7	1.407 (2)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C1—N1—N2	105.75 (17)	C6—C5—N3	117.14 (18)
N1—N2—C7	110.95 (16)	C6—C5—C4	124.74 (19)
N1—N2—C8	119.57 (17)	C5—C6—H6	122.5
C7—N2—C8	128.75 (17)	C5—C6—C7	115.00 (17)
O1—N3—C5	119.02 (19)	C7—C6—H6	122.5
O2—N3—O1	123.3 (2)	N2—C7—C2	107.41 (16)
O2—N3—C5	117.7 (2)	N2—C7—C6	130.55 (17)
N1—C1—Cl1	120.10 (17)	C6—C7—C2	122.03 (17)
N1—C1—C2	113.08 (18)	N2—C8—H8A	109.2
C2—C1—Cl1	126.83 (17)	N2—C8—H8B	109.2
C3—C2—C1	136.70 (19)	N2—C8—C9	112.00 (18)
C3—C2—C7	120.51 (18)	H8A—C8—H8B	107.9
C7—C2—C1	102.79 (17)	C9—C8—H8A	109.2
C2—C3—H3	120.8	C9—C8—H8B	109.2
C4—C3—C2	118.32 (18)	C8—C9—H9A	109.5
C4—C3—H3	120.8	C8—C9—H9B	109.5
C3—C4—H4	120.3	C8—C9—H9C	109.5
C3—C4—C5	119.39 (18)	H9A—C9—H9B	109.5
C5—C4—H4	120.3	H9A—C9—H9C	109.5
C4—C5—N3	118.12 (19)	H9B—C9—H9C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.93	2.52	3.280 (3)	139

Symmetry code: (i) x, $-y+1/2$, $z-1/2$.