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Ethyl 2-(6-nitro-2*H*-indazol-2-yl)acetate

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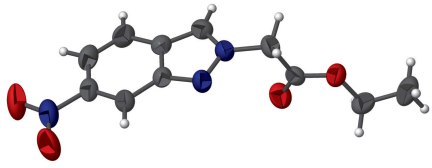
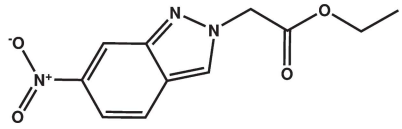
In the title compound, $C_{11}H_{11}N_3O_4$, the indazolyl ring and the nitro group are nearly coplanar, with the greatest deviation from their mean plane being 0.103 (2) Å. The mean plane through the acetate group is almost perpendicular to the indazolyl ring, subtending a dihedral angle of 88.05 (7)°. In the crystal, molecules are linked by C—H···O and C—H···N non-classical hydrogen bonds to form supramolecular layers that stack along the *a* axis.

3D view



Chemical scheme



Structure description

Indazolyl derivatives have been used widely in medicinal chemistry (Gaikwad *et al.*, 2015) and drug discovery (Jennings & Tennant *et al.*, 2007). They exhibit a broad range of biological activities including HIV protease inhibition (Han *et al.*, 1998), anti-inflammatory (Rosati *et al.*, 2007) anti-microbial (Li *et al.*, 2003), antispermatogenic (Takahashi *et al.*, 2011) antiplatelet (Lee *et al.*, 2001) and anticancer activities (Abbassi *et al.*, 2012, 2014).

The molecule of the title compound is built up from an indazolyl ring (C1–C7/N2/N3) linked to a nitro group and to an ethyl acetate groups as shown in Fig. 1. The nitro group and indazolyl cycle are nearly coplanar with the greatest deviation from the mean plane being 0.103 (2) Å for the O1 atom. The mean plan through the acetate moieties is almost perpendicular to the indazolyl ring as indicated by the dihedral angle of 88.05 (7)° between them.

The crystal structure cohesion is ensured by C—H···O and C—H···N hydrogen interactions (Table 1), which form supramolecular layers that stack along the *a* axis.

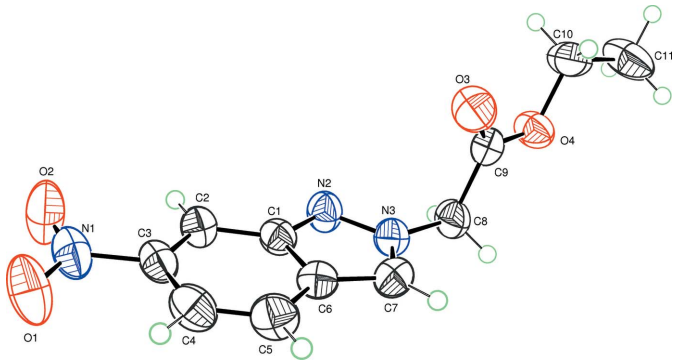


Figure 1
Plot of the molecule of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

Synthesis and crystallization

To a solution of 6-nitroindazole (6.13 mmol) in THF (30 ml) was added K_2CO_3 (9.2 mmol). After 15 min at 298 K, ethyl bromoacetate (6.13 mmol) was added dropwise. The solution was refluxed with stirring for 6 h and 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 evaporated *in vacuo*. The resulting residue was purified by column chromatography (EtOAc/ hexane 3/7). The title compound was recrystallized from ethanol at room temperature giving colourless crystals (m.p. 338 K, yield 65%).

Refinement

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

Acknowledgements

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O3^{i}	0.93	2.54	3.2743 (18)	137
C8—H8A···N2^{ii}	0.97	2.59	3.4650 (19)	149
C8—H8B···O3^{iii}	0.97	2.34	3.2525 (18)	157

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{11}N_3O_4$
M_r	249.23
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	296
a, *b*, *c* (Å)	31.808 (3), 4.6312 (4), 19.0381 (16)
β (°)	122.839 (3)
V (Å^3)	2356.3 (3)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.35 × 0.31 × 0.25
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2015*b*)
T_{min}, T_{max}	0.626, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25389, 3324, 2517
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.694
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.140, 1.03
No. of reflections	3324
No. of parameters	163
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.19

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

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full crystallographic data

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Ethyl 2-(6-nitro-2*H*-indazol-2-yl)acetate

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Ethyl 2-(6-nitro-2*H*-indazol-2-yl)acetate

Crystal data

$C_{11}H_{11}N_3O_4$
M_r = 249.23
Monoclinic, *C*2/*c*
a = 31.808 (3) Å
b = 4.6312 (4) Å
c = 19.0381 (16) Å
β = 122.839 (3)°
V = 2356.3 (3) Å^3
Z = 8
F(000) = 1040
D_x = 1.405 Mg m^{-3}
Melting point: 338 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3324 reflections
θ = 2.1–29.6°
μ = 0.11 mm^{-1}
T = 296 K
Block, colourless
0.35 × 0.31 × 0.25 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, 2015b)
T_{min} = 0.626, T_{max} = 0.746
25389 measured reflections
3324 independent reflections
2517 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 29.6°, θ_{min} = 2.1°
h = −44→44
k = −6→6
l = −24→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.140
S = 1.03
3324 reflections
163 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 0.8343P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 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	0.33711 (5)	0.4119 (3)	0.46597 (8)	0.0418 (3)
C2	0.37402 (5)	0.6087 (3)	0.52132 (8)	0.0466 (3)
H2	0.3723	0.7029	0.5628	0.056*
C3	0.41254 (5)	0.6530 (3)	0.51054 (8)	0.0472 (3)
C4	0.41682 (5)	0.5172 (4)	0.44835 (10)	0.0570 (4)
H4	0.4443	0.5553	0.4447	0.068*
C5	0.38093 (6)	0.3310 (4)	0.39402 (10)	0.0580 (4)
H5	0.3831	0.2427	0.3522	0.070*
C6	0.34001 (5)	0.2737 (3)	0.40226 (8)	0.0454 (3)
C7	0.29814 (5)	0.0965 (3)	0.36243 (9)	0.0508 (3)
H7	0.2884	−0.0244	0.3171	0.061*
C8	0.22800 (5)	0.0023 (3)	0.38266 (9)	0.0516 (3)
H8A	0.2282	−0.0198	0.4335	0.062*
H8B	0.2252	−0.1883	0.3594	0.062*
C9	0.18351 (5)	0.1819 (3)	0.32093 (8)	0.0432 (3)
C10	0.09600 (6)	0.2351 (4)	0.25660 (11)	0.0614 (4)
H10A	0.0955	0.4089	0.2845	0.074*
H10B	0.0940	0.2908	0.2058	0.074*
C11	0.05359 (6)	0.0510 (5)	0.23640 (13)	0.0792 (6)
H11A	0.0230	0.1540	0.2005	0.119*
H11B	0.0542	−0.1199	0.2084	0.119*
H11C	0.0557	−0.0022	0.2870	0.119*
N1	0.45222 (5)	0.8546 (3)	0.56789 (8)	0.0608 (3)
N2	0.29646 (4)	0.3270 (3)	0.46565 (7)	0.0482 (3)
N3	0.27453 (4)	0.1349 (2)	0.40251 (7)	0.0467 (3)
O1	0.48852 (5)	0.8813 (4)	0.56230 (9)	0.0909 (5)
O2	0.44763 (5)	0.9839 (4)	0.61888 (9)	0.0884 (4)
O3	0.18539 (4)	0.3901 (2)	0.28551 (7)	0.0644 (3)
O4	0.14227 (3)	0.0744 (2)	0.31126 (6)	0.0532 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0379 (6)	0.0444 (6)	0.0440 (6)	0.0005 (5)	0.0229 (5)	0.0021 (5)
C2	0.0426 (6)	0.0519 (7)	0.0444 (6)	−0.0045 (5)	0.0231 (5)	−0.0024 (5)
C3	0.0372 (6)	0.0517 (7)	0.0465 (6)	−0.0025 (5)	0.0186 (5)	0.0082 (5)
C4	0.0456 (7)	0.0720 (9)	0.0616 (8)	0.0004 (7)	0.0345 (7)	0.0075 (7)
C5	0.0575 (8)	0.0695 (9)	0.0609 (8)	0.0013 (7)	0.0411 (7)	−0.0033 (7)
C6	0.0449 (7)	0.0453 (6)	0.0479 (7)	0.0034 (5)	0.0263 (6)	0.0013 (5)
C7	0.0528 (7)	0.0472 (7)	0.0520 (7)	−0.0009 (6)	0.0282 (6)	−0.0068 (6)
C8	0.0432 (7)	0.0446 (7)	0.0594 (8)	−0.0074 (5)	0.0229 (6)	0.0013 (6)
C9	0.0454 (7)	0.0394 (6)	0.0433 (6)	−0.0058 (5)	0.0230 (5)	−0.0063 (5)
C10	0.0529 (8)	0.0542 (8)	0.0715 (10)	0.0136 (7)	0.0301 (7)	0.0105 (7)
C11	0.0435 (8)	0.0896 (13)	0.0937 (13)	0.0143 (8)	0.0302 (9)	0.0275 (11)
N1	0.0453 (7)	0.0706 (8)	0.0555 (7)	−0.0128 (6)	0.0202 (6)	0.0064 (6)

N2	0.0430 (6)	0.0540 (6)	0.0507 (6)	−0.0083 (5)	0.0275 (5)	−0.0079 (5)
N3	0.0413 (5)	0.0433 (5)	0.0522 (6)	−0.0040 (4)	0.0231 (5)	−0.0022 (5)
O1	0.0569 (7)	0.1261 (12)	0.0900 (9)	−0.0356 (7)	0.0402 (7)	−0.0075 (8)
O2	0.0756 (8)	0.1017 (10)	0.0873 (9)	−0.0404 (8)	0.0438 (7)	−0.0381 (8)
O3	0.0602 (6)	0.0561 (6)	0.0687 (7)	−0.0068 (5)	0.0296 (6)	0.0132 (5)
O4	0.0417 (5)	0.0527 (5)	0.0620 (6)	0.0017 (4)	0.0260 (5)	0.0095 (4)

Geometric parameters (Å, º)

C1—N2	1.3485 (15)	C8—C9	1.5073 (19)
C1—C2	1.4062 (18)	C8—H8A	0.9700
C1—C6	1.4182 (17)	C8—H8B	0.9700
C2—C3	1.3631 (17)	C9—O3	1.1964 (16)
C2—H2	0.9300	C9—O4	1.3195 (15)
C3—C4	1.411 (2)	C10—C11	1.460 (2)
C3—N1	1.4703 (18)	C10—O4	1.4634 (17)
C4—C5	1.355 (2)	C10—H10A	0.9700
C4—H4	0.9300	C10—H10B	0.9700
C5—C6	1.4177 (18)	C11—H11A	0.9600
C5—H5	0.9300	C11—H11B	0.9600
C6—C7	1.3881 (18)	C11—H11C	0.9600
C7—N3	1.3411 (17)	N1—O2	1.2150 (19)
C7—H7	0.9300	N1—O1	1.2218 (17)
C8—N3	1.4504 (16)	N2—N3	1.3468 (16)
N2—C1—C2	126.96 (11)	C9—C8—H8B	109.4
N2—C1—C6	111.80 (11)	H8A—C8—H8B	108.0
C2—C1—C6	121.23 (11)	O3—C9—O4	125.13 (13)
C3—C2—C1	115.98 (12)	O3—C9—C8	124.84 (12)
C3—C2—H2	122.0	O4—C9—C8	110.03 (11)
C1—C2—H2	122.0	C11—C10—O4	108.64 (13)
C2—C3—C4	124.25 (13)	C11—C10—H10A	110.0
C2—C3—N1	117.61 (13)	O4—C10—H10A	110.0
C4—C3—N1	118.14 (12)	C11—C10—H10B	110.0
C5—C4—C3	119.96 (12)	O4—C10—H10B	110.0
C5—C4—H4	120.0	H10A—C10—H10B	108.3
C3—C4—H4	120.0	C10—C11—H11A	109.5
C4—C5—C6	118.62 (13)	C10—C11—H11B	109.5
C4—C5—H5	120.7	H11A—C11—H11B	109.5
C6—C5—H5	120.7	C10—C11—H11C	109.5
C7—C6—C5	135.79 (13)	H11A—C11—H11C	109.5
C7—C6—C1	104.23 (11)	H11B—C11—H11C	109.5
C5—C6—C1	119.96 (12)	O2—N1—O1	123.26 (14)
N3—C7—C6	106.16 (12)	O2—N1—C3	118.66 (12)
N3—C7—H7	126.9	O1—N1—C3	118.08 (15)
C6—C7—H7	126.9	N3—N2—C1	103.17 (10)
N3—C8—C9	111.32 (11)	C7—N3—N2	114.63 (11)
N3—C8—H8A	109.4	C7—N3—C8	127.07 (12)

C9—C8—H8A	109.4	N2—N3—C8	118.25 (11)
N3—C8—H8B	109.4	C9—O4—C10	116.59 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O3^{i}	0.93	2.54	3.2743 (18)	137
C8—H8*A*···N2^{ii}	0.97	2.59	3.4650 (19)	149
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