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1-(5-Nitro-1*H*-indazol-1-yl)ethanone

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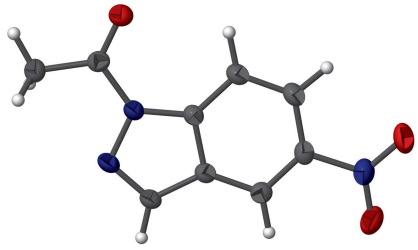
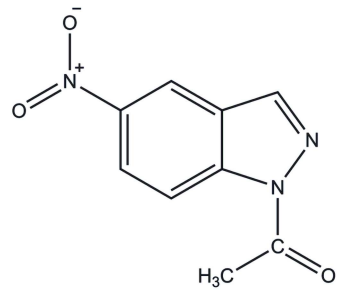
In the title compound, $C_9H_7N_3O_3$, paired ribbons of molecules running in the *a*-axis direction are formed by intermolecular C—H···O and offset π-stacking interactions.

3D view



Chemical scheme



Structure description

Indazole derivatives are important structural fragments in medicinal chemistry having a broad spectrum of pharmacological activities including anti-inflammatory, anti-tumor, or HIV protease inhibition (Stefan *et al.*, 2002; Arán *et al.*, 2005; Boulouard *et al.*, 2007) as well as exhibiting estrogen receptor (Steffan *et al.*, 2004), antifungal and antibacterial activities (Tandon *et al.*, 2005). Here we report the acetylation of 5-nitro-1*H*-indazole using acetic anhydride in the presence of a catalytic amount of acetic acid.

In the crystal structure of the title compound (Fig. 1), weak C3—H3···O1($1 + x$, y, z) hydrogen bonds form ribbons parallel to (010) and running along the *a*-axis direction (Table 1 and Fig. 2). Two such chains are associated *via* offset π-stacking interactions between a six-membered ring in one chain and a five-membered ring in the other with a 3.7361 (9) Å separation (Fig. 2).

Synthesis and crystallization

A mixture of 5-nitro-1*H*-indazole (0.6 g, 3.68 mmol), acetic acid (2 ml) and acetic anhydride (10 ml) were heated under reflux for 24 h, after completion of the reaction (monitored by TLC), the solvent was removed under vacuum. The residue obtained was recrystallized from ethanol to afford the title compound as colourless crystals (yield: 75%; m.p. 429–431 K).

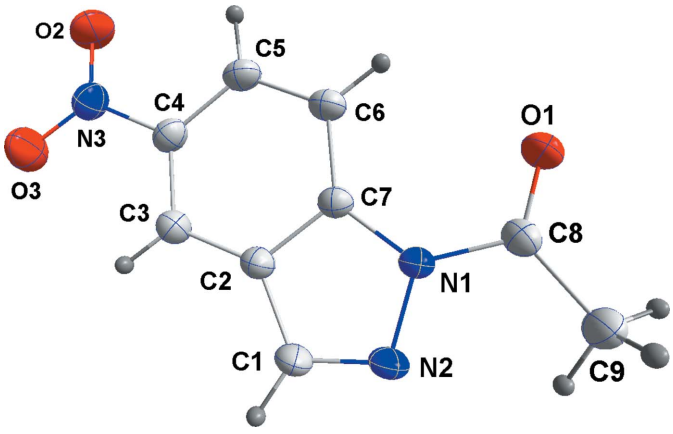


Figure 1
The title molecule with labeling scheme and 50% probability ellipsoids.

Refinement

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

Acknowledgements

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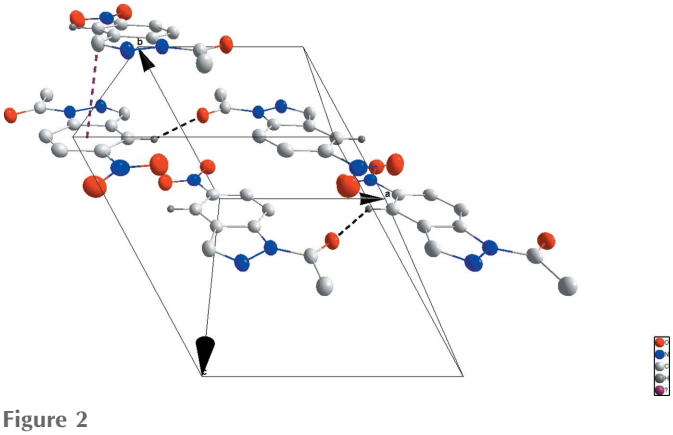

Figure 2
Packing viewed normal to (001). C—H···O interactions are shown by black dotted lines while a representative example of the offset π-stacking is shown by a purple dotted line.

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.99 (2)	2.36 (2)	3.1816 (17)	140.0 (16)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_7N_3O_3$
M_r	205.18
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	7.6004 (3), 7.9200 (3), 9.3621 (3)
α, β, γ (°)	113.020 (1), 91.404 (2), 118.350 (2)
V (Å^3)	440.83 (3)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.02
Crystal size (mm)	0.27 × 0.21 × 0.04
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.84, 0.96
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3385, 1641, 1456
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.105, 1.07
No. of reflections	1641
No. of parameters	165
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.22

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

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full crystallographic data

IUCrData (2016). **1**, x160939 [doi:10.1107/S2414314616009391]

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Crystal data

$C_9H_7N_3O_3$
M_r = 205.18
Triclinic, $P\overline{1}$
a = 7.6004 (3) Å
b = 7.9200 (3) Å
c = 9.3621 (3) Å
α = 113.020 (1)°
β = 91.404 (2)°
γ = 118.350 (2)°
V = 440.83 (3) Å^3
Z = 2
$F(000)$ = 212
D_x = 1.546 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 2667 reflections
θ = 5.3–72.3°
μ = 1.02 mm^{-1}
T = 150 K
Thick plate, colourless
0.27 × 0.21 × 0.04 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} = 0.84, T_{max} = 0.96
3385 measured reflections
1641 independent reflections
1456 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 72.3°, θ_{min} = 5.3°
h = −9→9
k = −9→9
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.07
1641 reflections
165 parameters
0 restraints
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.0862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL 2014/7* (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.043 (5)

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}
O1	0.31174 (15)	0.75516 (16)	0.38055 (12)	0.0335 (3)
O2	0.94598 (19)	0.7555 (2)	0.94836 (13)	0.0440 (3)
O3	1.17589 (18)	0.7624 (2)	0.81538 (15)	0.0462 (3)
N1	0.59744 (16)	0.74735 (18)	0.33098 (13)	0.0255 (3)
N2	0.71515 (18)	0.72440 (19)	0.22162 (13)	0.0290 (3)
N3	1.01405 (19)	0.75853 (19)	0.83144 (14)	0.0326 (3)
C1	0.8605 (2)	0.7163 (2)	0.28973 (16)	0.0286 (3)
H1	0.957 (3)	0.697 (3)	0.231 (2)	0.035 (4)*
C2	0.8463 (2)	0.7338 (2)	0.44671 (15)	0.0250 (3)
C3	0.9618 (2)	0.7351 (2)	0.56515 (16)	0.0270 (3)
H3	1.085 (3)	0.724 (3)	0.549 (2)	0.045 (5)*
C4	0.8964 (2)	0.7563 (2)	0.70377 (16)	0.0272 (3)
C5	0.7242 (2)	0.7752 (2)	0.72875 (16)	0.0285 (3)
H5	0.684 (3)	0.786 (3)	0.829 (2)	0.039 (5)*
C6	0.6096 (2)	0.7744 (2)	0.61171 (16)	0.0271 (3)
H6	0.485 (3)	0.784 (3)	0.6265 (19)	0.026 (4)*
C7	0.6741 (2)	0.75373 (19)	0.47014 (15)	0.0236 (3)
C8	0.4213 (2)	0.7508 (2)	0.28895 (16)	0.0268 (3)
C9	0.3841 (2)	0.7473 (3)	0.12989 (17)	0.0326 (4)
H9A	0.366 (3)	0.618 (3)	0.043 (2)	0.050 (5)*
H9B	0.499 (3)	0.865 (3)	0.126 (2)	0.040 (5)*
H9C	0.258 (3)	0.750 (3)	0.113 (2)	0.043 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0311 (5)	0.0428 (6)	0.0336 (5)	0.0228 (5)	0.0142 (4)	0.0190 (5)
O2	0.0520 (7)	0.0565 (7)	0.0307 (6)	0.0286 (6)	0.0132 (5)	0.0264 (5)
O3	0.0394 (6)	0.0649 (8)	0.0470 (7)	0.0302 (6)	0.0103 (5)	0.0335 (6)
N1	0.0258 (6)	0.0300 (6)	0.0226 (5)	0.0162 (5)	0.0090 (4)	0.0117 (4)
N2	0.0299 (6)	0.0354 (6)	0.0245 (6)	0.0193 (5)	0.0135 (5)	0.0133 (5)
N3	0.0335 (7)	0.0320 (6)	0.0296 (6)	0.0144 (5)	0.0047 (5)	0.0156 (5)
C1	0.0285 (7)	0.0352 (7)	0.0259 (6)	0.0195 (6)	0.0125 (5)	0.0139 (6)
C2	0.0248 (6)	0.0253 (6)	0.0245 (6)	0.0132 (5)	0.0086 (5)	0.0108 (5)
C3	0.0262 (6)	0.0263 (6)	0.0285 (7)	0.0137 (6)	0.0081 (5)	0.0128 (5)
C4	0.0292 (7)	0.0254 (6)	0.0243 (6)	0.0120 (6)	0.0054 (5)	0.0121 (5)
C5	0.0305 (7)	0.0271 (7)	0.0241 (6)	0.0127 (6)	0.0097 (5)	0.0114 (5)
C6	0.0272 (7)	0.0267 (6)	0.0257 (7)	0.0135 (6)	0.0105 (5)	0.0110 (5)
C7	0.0248 (6)	0.0221 (6)	0.0219 (6)	0.0115 (5)	0.0072 (5)	0.0092 (5)

C8	0.0243 (6)	0.0262 (6)	0.0280 (7)	0.0134 (6)	0.0065 (5)	0.0104 (5)
C9	0.0331 (7)	0.0410 (8)	0.0279 (7)	0.0217 (7)	0.0088 (6)	0.0166 (6)

Geometric parameters (Å, º)

O1—C8	1.2119 (17)	C3—C4	1.3787 (18)
O2—N3	1.2259 (16)	C3—H3	0.99 (2)
O3—N3	1.2302 (18)	C4—C5	1.401 (2)
N1—C7	1.3866 (16)	C5—C6	1.379 (2)
N1—N2	1.3912 (15)	C5—H5	0.981 (18)
N1—C8	1.4020 (17)	C6—C7	1.4005 (17)
N2—C1	1.3018 (19)	C6—H6	0.994 (18)
N3—C4	1.4646 (17)	C8—C9	1.4959 (19)
C1—C2	1.4331 (18)	C9—H9A	0.97 (2)
C1—H1	0.962 (18)	C9—H9B	0.941 (19)
C2—C3	1.3915 (19)	C9—H9C	0.98 (2)
C2—C7	1.4032 (19)		
C7—N1—N2	111.10 (10)	C6—C5—C4	120.29 (12)
C7—N1—C8	129.03 (11)	C6—C5—H5	120.1 (11)
N2—N1—C8	119.79 (11)	C4—C5—H5	119.6 (11)
C1—N2—N1	106.12 (11)	C5—C6—C7	116.86 (13)
O2—N3—O3	123.36 (13)	C5—C6—H6	121.2 (9)
O2—N3—C4	118.42 (12)	C7—C6—H6	121.9 (9)
O3—N3—C4	118.21 (12)	N1—C7—C6	132.17 (12)
N2—C1—C2	111.95 (12)	N1—C7—C2	105.71 (11)
N2—C1—H1	119.0 (11)	C6—C7—C2	122.12 (13)
C2—C1—H1	129.0 (11)	O1—C8—N1	119.14 (12)
C3—C2—C7	120.90 (12)	O1—C8—C9	125.17 (13)
C3—C2—C1	133.98 (13)	N1—C8—C9	115.70 (12)
C7—C2—C1	105.12 (12)	C8—C9—H9A	111.2 (12)
C4—C3—C2	116.08 (13)	C8—C9—H9B	110.3 (11)
C4—C3—H3	123.1 (12)	H9A—C9—H9B	106.8 (16)
C2—C3—H3	120.8 (12)	C8—C9—H9C	108.4 (11)
C3—C4—C5	123.74 (13)	H9A—C9—H9C	108.9 (16)
C3—C4—N3	117.76 (13)	H9B—C9—H9C	111.3 (16)
C5—C4—N3	118.51 (12)		
C7—N1—N2—C1	0.09 (15)	C4—C5—C6—C7	0.1 (2)
C8—N1—N2—C1	−177.03 (12)	N2—N1—C7—C6	−179.93 (13)
N1—N2—C1—C2	−0.08 (16)	C8—N1—C7—C6	−3.1 (2)
N2—C1—C2—C3	−179.60 (14)	N2—N1—C7—C2	−0.06 (14)
N2—C1—C2—C7	0.05 (16)	C8—N1—C7—C2	176.72 (12)
C7—C2—C3—C4	0.16 (19)	C5—C6—C7—N1	−179.88 (13)
C1—C2—C3—C4	179.76 (14)	C5—C6—C7—C2	0.27 (19)
C2—C3—C4—C5	0.2 (2)	C3—C2—C7—N1	179.71 (11)
C2—C3—C4—N3	−179.96 (11)	C1—C2—C7—N1	0.01 (14)
O2—N3—C4—C3	−172.80 (12)	C3—C2—C7—C6	−0.4 (2)

O3—N3—C4—C3	6.87 (19)	C1—C2—C7—C6	179.89 (12)
O2—N3—C4—C5	7.06 (19)	C7—N1—C8—O1	−1.8 (2)
O3—N3—C4—C5	−173.27 (13)	N2—N1—C8—O1	174.72 (11)
C3—C4—C5—C6	−0.3 (2)	C7—N1—C8—C9	178.53 (12)
N3—C4—C5—C6	179.82 (11)	N2—N1—C8—C9	−4.93 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.99 (2)	2.36 (2)	3.1816 (17)	140.0 (16)

Symmetry code: (i) *x*+1, *y*, *z*.