

# 1-Chloro-3-(6-nitro-1*H*-indazol-1-yl)propan-2-ol

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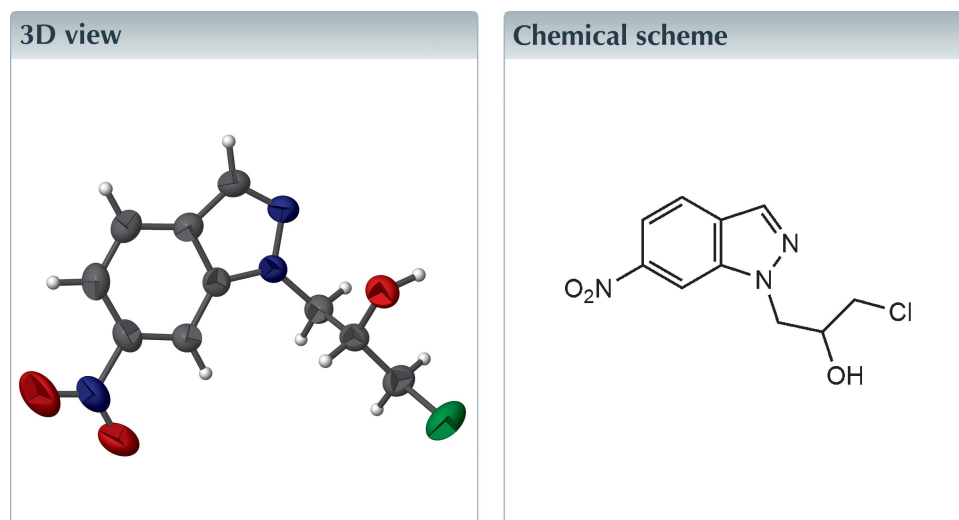
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Keywords: crystal structure; indazole; hydrogen bond.

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Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

In the title compound, C<sub>10</sub>H<sub>10</sub>ClN<sub>3</sub>O<sub>3</sub>, the side chain is oriented nearly perpendicular to the mean plane of the indazole ring system. In the crystal, complementary sets of O—H···N and C—H···O hydrogen bonds form chains of molecules stacked along the *a*-axis direction



## Structure description

Indazoles and their derivatives have gained considerable importance in medicinal chemistry in view of their promising pharmacological properties (Caron & Vazquez, 1999; Yeu *et al.*, 2001). Substituted indazoles are especially important sub-structures and these compounds are present in numerous pharmacophores including antitumor, antiplatelet, anti-viral and anti-microbial agents (Abbassi *et al.*, 2014).

In the title compound (Fig. 1), the indazole moiety is planar with an r.m.s. deviation of 0.0046 Å. The C1—N1—C8—C9 torsion angle of  $-81.0(2)^\circ$  indicates that the side chain is close to perpendicular to the indazole plane. In the crystal, inversion-related O3—H3···N2<sup>i</sup> hydrogen bonds form dimers enclosing  $R_2^2(12)$  rings (Table 1 and Fig. 2). Non-classical C2—H2···O<sup>ii</sup> hydrogen bonds also form inversion dimers and  $R_2^2(10)$  rings. These contacts combine to link molecules in a head-to-tail fashion into chains along the *c*-axis direction. Additional C10—H10A···O3<sup>iii</sup> hydrogen bonds link adjacent chains, stacking molecules along the *a*-axis direction (Table 1 and Fig. 3).

## Synthesis and crystallization

To a solution of 6-nitroindazole (0.01 mol, 0.5 g) in tetrahydrofuran (40 ml), was added potassium bicarbonate (0.02 mol, 2.76 g), 2-(chloromethyl)oxirane (0.02 mol, 1.2 ml) and tetra *n*-butylammonium bromide (0.001 mol, 0.16 g). The reaction mixture was stirred at

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O3-H3\cdots N2^i$	1.00	1.88	2.871 (2)	169
$C2-H2\cdots O1^{ii}$	0.93	2.59	3.492 (3)	163
$C10-H10A\cdots O3^{iii}$	0.97	2.49	3.268 (3)	137

Symmetry codes: (i)  $-x + 1, -y + 1, -z$ ; (ii)  $-x + 1, -y + 1, -z + 1$ ; (iii)  $x - 1, y, z$ .

room temperature for 48 h. The solution was filtered and the the solvent removed under reduced pressure. The residue obtained was chromatographed on a silica-gel column using hexane and ethyl acetate (80/20) as eluents to afford the title compound as yellow plate-like crystals.

## Refinement

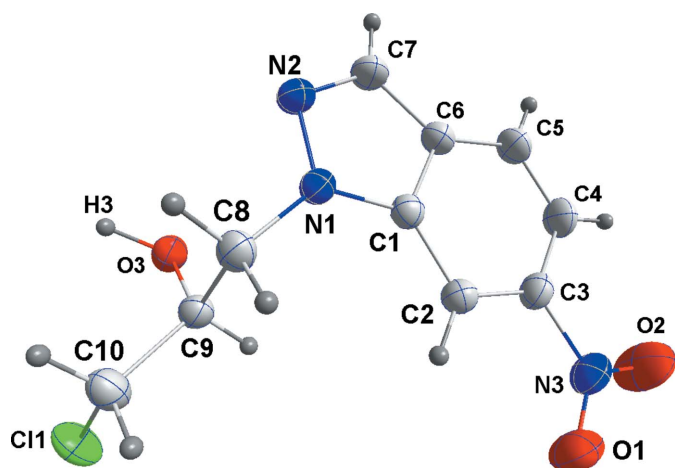
Crystal data, data collection and structure refinement details are summarized in Table 2.

## Acknowledgements

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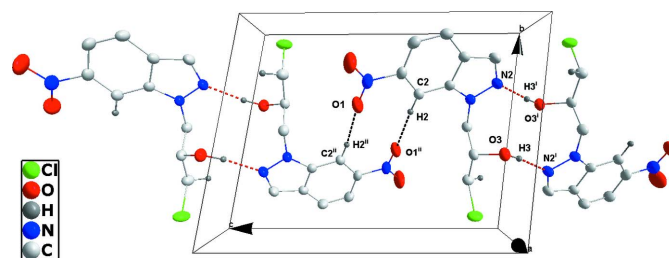


**Figure 1**  
The title molecule with labeling scheme and 30% probability ellipsoids.

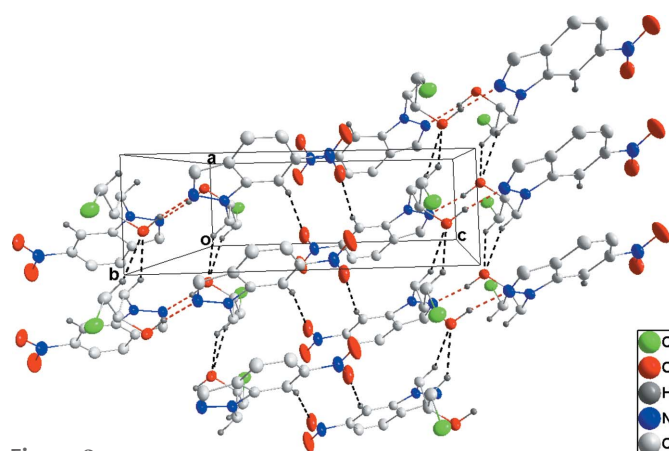
**Table 2**  
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{10}ClN_3O_3$
$M_r$	255.66
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	298
$a, b, c$ (Å)	4.3949 (4), 9.9144 (8), 13.1376 (11)
$\alpha, \beta, \gamma$ (°)	98.481 (1), 90.755 (1), 97.377 (1)
$V$ (Å <sup>3</sup> )	561.19 (8)
$Z$	2
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.34
Crystal size (mm)	0.45 × 0.20 × 0.11
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2016)
$T_{min}, T_{max}$	0.87, 0.96
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	10444, 2790, 1874
$R_{int}$	0.023
$(\sin \theta/\lambda)_{max}$ (Å <sup>-1</sup> )	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.047, 0.151, 1.06
No. of reflections	2790
No. of parameters	156
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.43, -0.19

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



**Figure 2**  
Chains of molecules formed along  $c$  by  $O-H\cdots N$  and  $C-H\cdots O$  hydrogen bonds shown as red and black dotted lines, respectively.



**Figure 3**  
Molecules stacked along the  $a$ -axis direction by  $O-H\cdots N$  and  $C-H\cdots O$  hydrogen bonds shown as red and black dashed lines, respectively.

## full crystallographic data

*IUCrData* (2017). **2**, x170637 [https://doi.org/10.1107/S241431461700637X]

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1-Chloro-3-(6-nitro-1*H*-indazol-1-yl)propan-2-ol*Crystal data*

$C_{10}H_{10}ClN_3O_3$

$M_r = 255.66$

Triclinic,  $P\bar{1}$

$a = 4.3949$  (4) Å

$b = 9.9144$  (8) Å

$c = 13.1376$  (11) Å

$\alpha = 98.481$  (1)°

$\beta = 90.755$  (1)°

$\gamma = 97.377$  (1)°

$V = 561.19$  (8) Å<sup>3</sup>

$Z = 2$

$F(000) = 264$

$D_x = 1.513$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 3584 reflections

$\theta = 2.4$ – $27.0$ °

$\mu = 0.34$  mm<sup>-1</sup>

$T = 298$  K

Plate, pale yellow

$0.45 \times 0.20 \times 0.11$  mm

*Data collection*

Bruker SMART APEX CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3333 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2016)

$T_{\min} = 0.87$ ,  $T_{\max} = 0.96$

10444 measured reflections

2790 independent reflections

1874 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 28.4$ °,  $\theta_{\min} = 1.6$ °

$h = -5 \rightarrow 5$

$k = -13 \rightarrow 13$

$l = -17 \rightarrow 17$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.151$

$S = 1.06$

2790 reflections

156 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0916P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.43$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.19$  e Å<sup>-3</sup>

*Special details*

**Experimental.** The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in  $\omega$ , collected at  $\varphi = 0.00$ , 90.00 and 180.00° and 2 sets of 800 frames, each of width 0.45° in  $\varphi$ , collected at  $\omega = -30.00$  and 210.00°. The scan time was 20 sec/frame.

**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.** 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  $F^2 > 2\sigma(F^2)$  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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å) while that attached to oxygen was placed in a location derived from a difference map and its coordinates adjusted to give O—H = 1.00 %A. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.46205 (13)	0.09914 (5)	0.12647 (5)	0.0763 (2)
O1	0.8217 (5)	0.6041 (2)	0.57705 (12)	0.0975 (6)
O2	1.1879 (6)	0.7675 (3)	0.61324 (14)	0.1311 (9)
O3	0.7011 (3)	0.38489 (14)	0.07195 (10)	0.0562 (4)
H3	0.562	0.362	0.0091	0.084*
N1	0.5788 (3)	0.63286 (15)	0.19817 (10)	0.0462 (4)
N2	0.6434 (4)	0.70386 (16)	0.11859 (11)	0.0544 (4)
N3	0.9933 (5)	0.6999 (2)	0.55419 (13)	0.0732 (5)
C1	0.7535 (4)	0.69161 (17)	0.28323 (12)	0.0428 (4)
C2	0.7652 (4)	0.65277 (18)	0.38148 (13)	0.0480 (4)
H2	0.6441	0.5758	0.3985	0.058*
C3	0.9679 (5)	0.7365 (2)	0.45025 (13)	0.0530 (5)
C4	1.1529 (5)	0.8533 (2)	0.42777 (15)	0.0617 (5)
H4	1.2851	0.9064	0.4784	0.074*
C5	1.1401 (5)	0.8896 (2)	0.33168 (15)	0.0590 (5)
H5	1.2633	0.9666	0.3157	0.071*
C6	0.9353 (4)	0.80712 (18)	0.25730 (13)	0.0479 (4)
C7	0.8535 (5)	0.80725 (19)	0.15292 (14)	0.0573 (5)
H7	0.9381	0.8724	0.1136	0.069*
C8	0.3623 (4)	0.50762 (18)	0.18429 (13)	0.0472 (4)
H8A	0.2181	0.5100	0.1283	0.057*
H8B	0.2472	0.5025	0.2464	0.057*
C9	0.5248 (4)	0.37983 (17)	0.16051 (12)	0.0454 (4)
H9	0.6651	0.3783	0.2188	0.054*
C10	0.2891 (4)	0.25374 (19)	0.15235 (15)	0.0558 (5)
H10A	0.1403	0.2559	0.0976	0.067*
H10B	0.1810	0.2545	0.2163	0.067*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.0755 (4)	0.0445 (3)	0.1061 (5)	0.0009 (2)	0.0158 (3)	0.0074 (3)
O1	0.1276 (16)	0.1079 (15)	0.0554 (9)	-0.0111 (12)	0.0029 (9)	0.0302 (9)
O2	0.155 (2)	0.160 (2)	0.0647 (11)	-0.0362 (17)	-0.0454 (12)	0.0256 (12)

O3	0.0470 (7)	0.0696 (9)	0.0496 (7)	-0.0046 (6)	0.0041 (6)	0.0116 (6)
N1	0.0480 (8)	0.0474 (8)	0.0427 (7)	0.0006 (6)	-0.0045 (6)	0.0104 (6)
N2	0.0634 (10)	0.0521 (9)	0.0482 (8)	-0.0007 (7)	-0.0047 (7)	0.0170 (7)
N3	0.0903 (14)	0.0835 (14)	0.0437 (9)	0.0053 (11)	-0.0071 (9)	0.0086 (9)
C1	0.0427 (9)	0.0418 (8)	0.0436 (9)	0.0066 (7)	0.0001 (7)	0.0048 (7)
C2	0.0517 (10)	0.0478 (9)	0.0445 (9)	0.0037 (8)	0.0037 (7)	0.0088 (7)
C3	0.0599 (11)	0.0582 (11)	0.0402 (9)	0.0097 (9)	-0.0023 (8)	0.0034 (8)
C4	0.0656 (13)	0.0588 (12)	0.0537 (11)	-0.0032 (9)	-0.0113 (9)	-0.0031 (9)
C5	0.0633 (12)	0.0480 (10)	0.0610 (11)	-0.0078 (9)	-0.0014 (9)	0.0060 (9)
C6	0.0517 (10)	0.0420 (9)	0.0497 (9)	0.0027 (7)	0.0008 (8)	0.0090 (7)
C7	0.0695 (13)	0.0491 (10)	0.0534 (10)	-0.0038 (9)	-0.0020 (9)	0.0176 (8)
C8	0.0400 (9)	0.0514 (10)	0.0482 (9)	-0.0030 (7)	-0.0009 (7)	0.0090 (7)
C9	0.0427 (9)	0.0503 (9)	0.0421 (8)	-0.0029 (7)	0.0003 (7)	0.0115 (7)
C10	0.0515 (11)	0.0519 (10)	0.0613 (11)	-0.0042 (8)	0.0070 (9)	0.0092 (8)

*Geometric parameters (Å, °)*

C11—C10	1.787 (2)	C3—C4	1.398 (3)
O1—N3	1.211 (3)	C4—C5	1.365 (3)
O2—N3	1.210 (3)	C4—H4	0.9300
O3—C9	1.4098 (19)	C5—C6	1.409 (3)
O3—H3	1.0000	C5—H5	0.9300
N1—N2	1.357 (2)	C6—C7	1.413 (2)
N1—C1	1.360 (2)	C7—H7	0.9300
N1—C8	1.451 (2)	C8—C9	1.528 (3)
N2—C7	1.310 (2)	C8—H8A	0.9700
N3—C3	1.470 (3)	C8—H8B	0.9700
C1—C6	1.398 (2)	C9—C10	1.507 (2)
C1—C2	1.403 (2)	C9—H9	0.9800
C2—C3	1.366 (3)	C10—H10A	0.9700
C2—H2	0.9300	C10—H10B	0.9700
C9—O3—H3	109.5	C1—C6—C7	104.02 (15)
N2—N1—C1	110.65 (14)	C5—C6—C7	136.27 (17)
N2—N1—C8	120.76 (13)	N2—C7—C6	111.41 (15)
C1—N1—C8	128.49 (14)	N2—C7—H7	124.3
C7—N2—N1	106.79 (14)	C6—C7—H7	124.3
O2—N3—O1	122.9 (2)	N1—C8—C9	111.61 (14)
O2—N3—C3	117.9 (2)	N1—C8—H8A	109.3
O1—N3—C3	119.21 (19)	C9—C8—H8A	109.3
N1—C1—C6	107.13 (14)	N1—C8—H8B	109.3
N1—C1—C2	130.19 (16)	C9—C8—H8B	109.3
C6—C1—C2	122.68 (16)	H8A—C8—H8B	108.0
C3—C2—C1	114.95 (17)	O3—C9—C10	113.23 (14)
C3—C2—H2	122.5	O3—C9—C8	111.05 (13)
C1—C2—H2	122.5	C10—C9—C8	108.81 (15)
C2—C3—C4	124.27 (17)	O3—C9—H9	107.9
C2—C3—N3	117.44 (18)	C10—C9—H9	107.9

C4—C3—N3	118.29 (17)	C8—C9—H9	107.9
C5—C4—C3	120.15 (17)	C9—C10—C11	111.62 (14)
C5—C4—H4	119.9	C9—C10—H10A	109.3
C3—C4—H4	119.9	C11—C10—H10A	109.3
C4—C5—C6	118.24 (18)	C9—C10—H10B	109.3
C4—C5—H5	120.9	C11—C10—H10B	109.3
C6—C5—H5	120.9	H10A—C10—H10B	108.0
C1—C6—C5	119.71 (16)		
C1—N1—N2—C7	-0.7 (2)	C3—C4—C5—C6	0.5 (3)
C8—N1—N2—C7	-177.30 (17)	N1—C1—C6—C5	179.55 (16)
N2—N1—C1—C6	1.0 (2)	C2—C1—C6—C5	-0.1 (3)
C8—N1—C1—C6	177.20 (16)	N1—C1—C6—C7	-0.8 (2)
N2—N1—C1—C2	-179.43 (17)	C2—C1—C6—C7	179.58 (16)
C8—N1—C1—C2	-3.2 (3)	C4—C5—C6—C1	-0.2 (3)
N1—C1—C2—C3	-179.49 (18)	C4—C5—C6—C7	-179.7 (2)
C6—C1—C2—C3	0.0 (3)	N1—N2—C7—C6	0.2 (2)
C1—C2—C3—C4	0.3 (3)	C1—C6—C7—N2	0.4 (2)
C1—C2—C3—N3	-179.21 (16)	C5—C6—C7—N2	179.9 (2)
O2—N3—C3—C2	174.7 (2)	N2—N1—C8—C9	94.92 (18)
O1—N3—C3—C2	-5.0 (3)	C1—N1—C8—C9	-81.0 (2)
O2—N3—C3—C4	-4.8 (3)	N1—C8—C9—O3	-57.54 (17)
O1—N3—C3—C4	175.5 (2)	N1—C8—C9—C10	177.18 (13)
C2—C3—C4—C5	-0.6 (3)	O3—C9—C10—C11	56.88 (18)
N3—C3—C4—C5	178.9 (2)	C8—C9—C10—C11	-179.12 (11)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O3—H3...N2 <sup>i</sup>	1.00	1.88	2.871 (2)	169
C2—H2...O1 <sup>ii</sup>	0.93	2.59	3.492 (3)	163
C10—H10A...O3 <sup>iii</sup>	0.97	2.49	3.268 (3)	137

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