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Crystal structure of *N*-(3-chloro-1-methyl-1*H*-indazol-5-yl)-4-methoxybenzenesulfonamide

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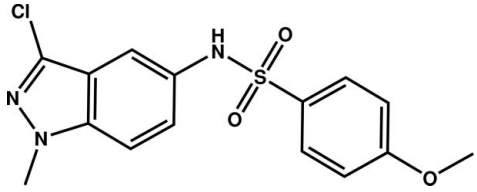
In the title compound, $C_{15}H_{14}ClN_3O_3S$, the dihedral angle between the planes of the indazole ring system (r.m.s. deviation = 0.007 Å) and the benzene ring is 89.05 (7)°. The methoxy C atom deviates from its attached ring by 0.196 (3) Å. In the crystal, inversion dimers linked by pairs of N—H···O hydrogen bonds generate $R_2^2(8)$ loops. The dimers are connected into [010] chains by C—H···O interactions.

Keywords: crystal structure; benzenesulfonamides; hydrogen bonding.

CCDC reference: 1017531

1. Related literature

For the biological activity of sulfonamides, see: El-Sayed *et al.* (2011); Mustafa *et al.* (2012); Scozzafava *et al.* (2003). For similar compounds, see: Abbassi *et al.* (2012, 2013); Chicha *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_{15}H_{14}ClN_3O_3S$
M_r = 351.80
Triclinic, $P\overline{1}$
a = 8.2023 (1) Å
b = 10.6312 (2) Å
c = 10.8957 (2) Å
α = 117.523 (1)°
β = 93.095 (1)°
γ = 103.166 (1)°
V = 806.36 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 296 K
0.40 × 0.36 × 0.31 mm

2.2. Data collection

Bruker X8 APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.693, T_{max} = 0.747
20115 measured reflections
4526 independent reflections
3707 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.116
S = 1.03
4526 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3*N*···O2^{i}	0.89	2.04	2.9286 (17)	175
C5—H5···O3^{ii}	0.93	2.56	3.426 (2)	156

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $x, y-1, z$.

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).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7261).

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supporting information

Acta Cryst. (2014). E**70**, o983–o984 [doi:10.1107/S1600536814017747]

Crystal structure of *N*-(3-chloro-1-methyl-1*H*-indazol-5-yl)-4-methoxybenzene-sulfonamide

Hakima Chicha, El Mostapha Rakib, Ahmed Gamouh, Mohamed Saadi and Lahcen El Ammari

S1. Comment

The sulfonamide functional group is a structure with broad importance, as it is found in numerous medicinal agents (El-Sayed, *et al.*, 2011; Mustafa, *et al.*, 2012; Scozzafava, *et al.*, 2003. Previously, we identified a series of indazoles bearing a sulfonamide moiety with good antiproliferative activities (Abbassi, *et al.*, 2012; Abbassi, *et al.*, 2013; Chicha, *et al.*, 2014).

The molecule of the title compound is built up from two fused five- and six-membered rings (N1 N2 C2 to C8) almost coplanar, with a maximum deviation of 0.010 (2) Å for C5 atom (Fig.1). The dihedral angle between the indazol system and the plane through the benzene ring (C9 to C14) is of 89.05 (7)°.

The cohesion of the crystal structure is ensured by N3–H3N···O2 hydrogen bonds between molecules forming dimers, which are linked together by C5–H5···O3 interaction and forming [010] chains as shown in Fig.2 and Table 1.

S2. Experimental

A mixture of 1-methyl-3-chloro-5-nitroindazole (1.22 mmol) and anhydrous SnCl2 (1.1 g, 6.1 mmol) in 25 mL of absolute ethanol was heated at 60 °C for 6 h. After reduction, the starting material disappeared, and the solution was allowed to cool down. The pH was made slightly basic (pH 7–8) by addition of 5% aqueous potassium bicarbonate before extraction with ethyl acetate. The organic phase was washed with brine and dried over magnesium sulfate. The solvent was removed to afford the amine, which was immediately dissolved in pyridine (5 ml) and then reacted with 4-methoxy-benzenesulfonyl chloride (1.25 mmol) at room temperature for 24 h. After the reaction mixture was concentrated *in vacuo*, the resulting residue was purified by flash chromatography (eluted with Ethyl acetate: Hexane 2:8). The title compound was recrystallized from ethanol solution. Yield: 56%, PF: 140–142°C.

S3. Refinement

H atoms were located in a difference map and treated as riding with C–H = 0.96 Å, C–H = 0.93 Å, and N–H = 0.86 Å for methyl, aromatic CH and NH, respectively. All hydrogen with U_{iso}(H) = 1.2 U_{eq} (aromatic, NH) and U_{iso}(H) = 1.5 U_{eq} for methyl.

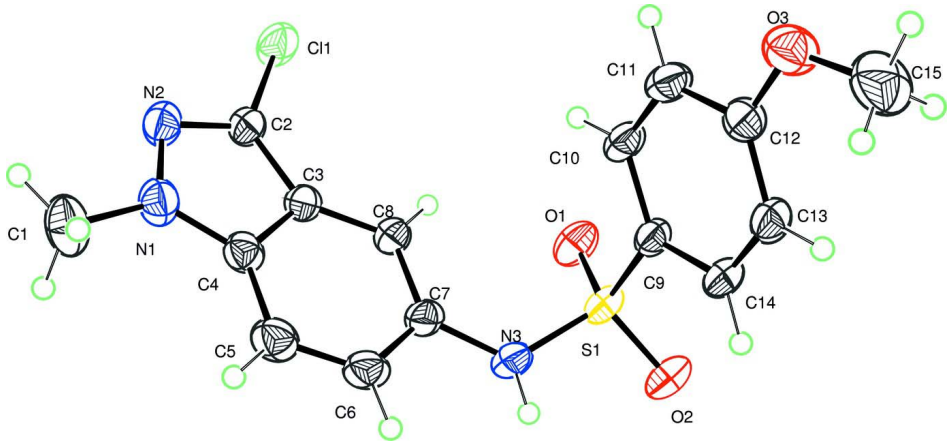

Figure 1

Molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

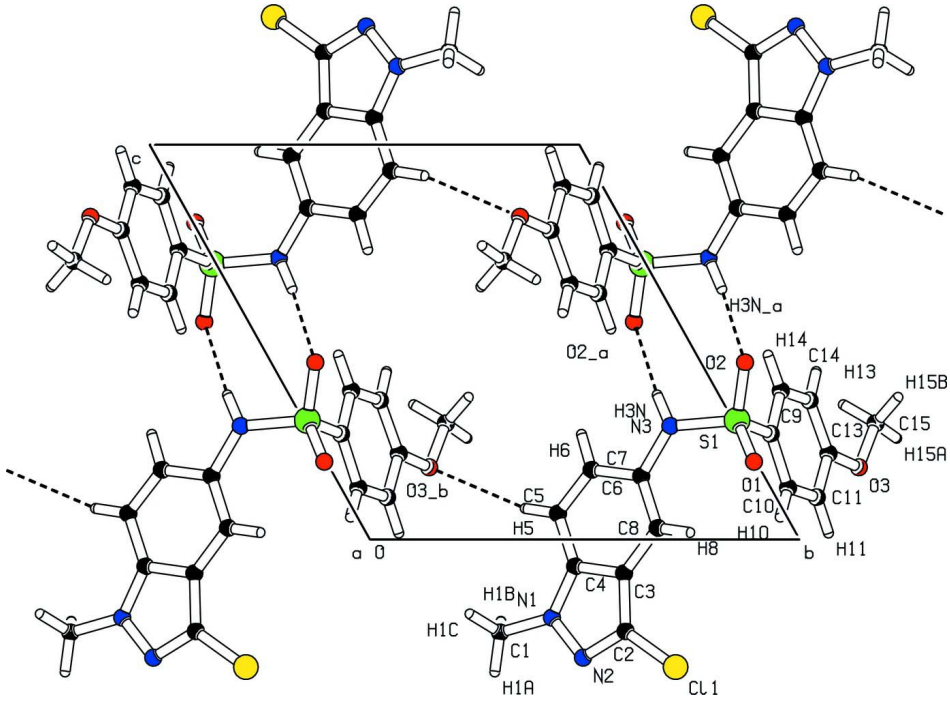

Figure 2

Crystal structure of the title compound, showing molecules linked by N3–H3N···O2 hydrogen bond and forming dimers linked by C5–H5···O3 interaction.

N-(3-Chloro-1-methyl-1*H*-indazol-5-yl)-4-methoxybenzenesulfonamide

Crystal data

$C_{15}H_{14}ClN_3O_3S$
$M_r = 351.80$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.2023$ (1) Å
$b = 10.6312$ (2) Å
$c = 10.8957$ (2) Å
$\alpha = 117.523$ (1)°
$\beta = 93.095$ (1)°
$\gamma = 103.166$ (1)°
$V = 806.36$ (2) Å^3
$Z = 2$

$F(000) = 364$
$D_x = 1.449$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4526 reflections
$\theta = 2.6–29.6°$
$\mu = 0.38$ mm^{-1}
$T = 296$ K
Block, colourless
0.40 × 0.36 × 0.31 mm

Data collection

Bruker X8 APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.693$, $T_{max} = 0.747$
20115 measured reflections
4526 independent reflections
3707 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 29.6°$, $\theta_{min} = 2.6°$
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.116$
$S = 1.03$
4526 reflections
209 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
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.1878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.5360 (3)	0.1820 (2)	−0.2354 (2)	0.0641 (5)
H1A	0.5166	0.1185	−0.3355	0.096*
H1B	0.4305	0.1986	−0.2072	0.096*
H1C	0.5801	0.1361	−0.1885	0.096*
C2	0.8383 (2)	0.47591 (16)	−0.23208 (15)	0.0391 (3)
C3	0.84060 (18)	0.54753 (16)	−0.08554 (14)	0.0352 (3)
C4	0.72093 (19)	0.44264 (17)	−0.06796 (16)	0.0397 (3)
C5	0.6874 (2)	0.4716 (2)	0.06598 (18)	0.0493 (4)
H5	0.6097	0.4017	0.0787	0.059*
C6	0.7734 (2)	0.60633 (19)	0.17665 (17)	0.0476 (4)
H6	0.7515	0.6289	0.2662	0.057*
C7	0.8951 (2)	0.71369 (17)	0.16058 (14)	0.0385 (3)

C8	0.93096 (19)	0.68456 (16)	0.02938 (14)	0.0375 (3)
H8	1.0120	0.7532	0.0177	0.045*
C9	0.88896 (18)	1.06974 (16)	0.26748 (13)	0.0364 (3)
C10	0.83288 (19)	1.04012 (17)	0.13088 (14)	0.0407 (3)
H10	0.8815	0.9839	0.0568	0.049*
C11	0.7053 (2)	1.09469 (18)	0.10689 (15)	0.0441 (3)
H11	0.6677	1.0754	0.0162	0.053*
C12	0.6321 (2)	1.17864 (17)	0.21742 (16)	0.0413 (3)
C13	0.6836 (2)	1.20357 (17)	0.35215 (16)	0.0435 (3)
H13	0.6319	1.2566	0.4254	0.052*
C14	0.8118 (2)	1.14935 (17)	0.37674 (14)	0.0413 (3)
H14	0.8469	1.1661	0.4670	0.050*
C15	0.4427 (3)	1.3276 (3)	0.2942 (3)	0.0797 (7)
H15A	0.3597	1.3573	0.2564	0.120*
H15B	0.5324	1.4136	0.3600	0.120*
H15C	0.3895	1.2767	0.3414	0.120*
N1	0.65760 (18)	0.32210 (15)	−0.19782 (15)	0.0478 (3)
N2	0.72912 (18)	0.34244 (15)	−0.29918 (14)	0.0456 (3)
N3	0.98123 (19)	0.84656 (15)	0.28741 (13)	0.0459 (3)
H3N	0.9455	0.8563	0.3661	0.057 (5)*
O1	1.16088 (15)	0.99481 (14)	0.19715 (12)	0.0520 (3)
O2	1.13503 (15)	1.10282 (14)	0.44851 (11)	0.0535 (3)
O3	0.51174 (17)	1.23162 (16)	0.18293 (14)	0.0575 (3)
S1	1.05777 (5)	1.00891 (4)	0.30176 (3)	0.04069 (12)
Cl1	0.95605 (6)	0.54559 (5)	−0.32303 (4)	0.05614 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0617 (11)	0.0421 (9)	0.0786 (13)	0.0032 (8)	0.0160 (10)	0.0262 (9)
C2	0.0482 (8)	0.0355 (7)	0.0327 (6)	0.0140 (6)	0.0112 (6)	0.0147 (6)
C3	0.0424 (7)	0.0354 (7)	0.0325 (6)	0.0176 (6)	0.0115 (5)	0.0170 (5)
C4	0.0443 (7)	0.0386 (8)	0.0420 (7)	0.0166 (6)	0.0133 (6)	0.0215 (6)
C5	0.0585 (10)	0.0506 (9)	0.0507 (9)	0.0187 (8)	0.0230 (7)	0.0315 (8)
C6	0.0631 (10)	0.0545 (10)	0.0396 (7)	0.0273 (8)	0.0224 (7)	0.0285 (7)
C7	0.0492 (8)	0.0416 (8)	0.0314 (6)	0.0243 (6)	0.0126 (6)	0.0174 (6)
C8	0.0468 (7)	0.0363 (7)	0.0308 (6)	0.0157 (6)	0.0108 (5)	0.0154 (5)
C9	0.0443 (7)	0.0328 (7)	0.0264 (6)	0.0081 (5)	0.0090 (5)	0.0110 (5)
C10	0.0462 (8)	0.0430 (8)	0.0263 (6)	0.0092 (6)	0.0109 (5)	0.0128 (6)
C11	0.0498 (8)	0.0489 (9)	0.0311 (6)	0.0088 (7)	0.0072 (6)	0.0197 (6)
C12	0.0446 (8)	0.0372 (7)	0.0413 (7)	0.0073 (6)	0.0085 (6)	0.0203 (6)
C13	0.0540 (9)	0.0394 (8)	0.0344 (7)	0.0165 (7)	0.0153 (6)	0.0137 (6)
C14	0.0535 (8)	0.0406 (8)	0.0256 (6)	0.0146 (6)	0.0103 (6)	0.0120 (5)
C15	0.0883 (16)	0.0877 (17)	0.0909 (17)	0.0560 (14)	0.0333 (14)	0.0502 (14)
N1	0.0523 (8)	0.0374 (7)	0.0485 (7)	0.0079 (6)	0.0134 (6)	0.0187 (6)
N2	0.0536 (8)	0.0367 (7)	0.0393 (6)	0.0111 (6)	0.0103 (6)	0.0134 (5)
N3	0.0649 (8)	0.0476 (8)	0.0272 (5)	0.0231 (6)	0.0116 (5)	0.0165 (5)
O1	0.0485 (6)	0.0604 (8)	0.0370 (5)	0.0149 (5)	0.0163 (5)	0.0151 (5)

O2	0.0533 (7)	0.0610 (8)	0.0281 (5)	0.0122 (6)	0.0018 (4)	0.0097 (5)
O3	0.0610 (7)	0.0654 (8)	0.0594 (7)	0.0281 (6)	0.0141 (6)	0.0362 (7)
S1	0.0437 (2)	0.0454 (2)	0.02480 (16)	0.01330 (15)	0.00761 (13)	0.01019 (14)
Cl1	0.0743 (3)	0.0487 (2)	0.0361 (2)	0.0060 (2)	0.01902 (18)	0.01758 (17)

Geometric parameters (Å, º)

C1—N1	1.448 (2)	C9—S1	1.7465 (16)
C1—H1A	0.9600	C10—C11	1.374 (2)
C1—H1B	0.9600	C10—H10	0.9300
C1—H1C	0.9600	C11—C12	1.392 (2)
C2—N2	1.320 (2)	C11—H11	0.9300
C2—C3	1.4129 (18)	C12—O3	1.357 (2)
C2—Cl1	1.7085 (15)	C12—C13	1.388 (2)
C3—C8	1.401 (2)	C13—C14	1.378 (2)
C3—C4	1.401 (2)	C13—H13	0.9300
C4—N1	1.362 (2)	C14—H14	0.9300
C4—C5	1.403 (2)	C15—O3	1.423 (3)
C5—C6	1.362 (2)	C15—H15A	0.9600
C5—H5	0.9300	C15—H15B	0.9600
C6—C7	1.419 (2)	C15—H15C	0.9600
C6—H6	0.9300	N1—N2	1.3577 (19)
C7—C8	1.3796 (19)	N3—S1	1.6260 (15)
C7—N3	1.4283 (19)	N3—H3N	0.8897
C8—H8	0.9300	O1—S1	1.4286 (11)
C9—C14	1.3884 (19)	O2—S1	1.4429 (11)
C9—C10	1.3974 (18)		

N1—C1—H1A	109.5	C10—C11—C12	120.42 (13)
N1—C1—H1B	109.5	C10—C11—H11	119.8
H1A—C1—H1B	109.5	C12—C11—H11	119.8
N1—C1—H1C	109.5	O3—C12—C13	124.39 (14)
H1A—C1—H1C	109.5	O3—C12—C11	115.54 (14)
H1B—C1—H1C	109.5	C13—C12—C11	120.08 (15)
N2—C2—C3	112.74 (13)	C14—C13—C12	119.65 (14)
N2—C2—Cl1	120.30 (11)	C14—C13—H13	120.2
C3—C2—Cl1	126.95 (12)	C12—C13—H13	120.2
C8—C3—C4	121.43 (13)	C13—C14—C9	120.33 (13)
C8—C3—C2	135.32 (14)	C13—C14—H14	119.8
C4—C3—C2	103.24 (13)	C9—C14—H14	119.8
N1—C4—C3	107.20 (13)	O3—C15—H15A	109.5
N1—C4—C5	131.85 (15)	O3—C15—H15B	109.5
C3—C4—C5	120.95 (14)	H15A—C15—H15B	109.5
C6—C5—C4	117.13 (15)	O3—C15—H15C	109.5
C6—C5—H5	121.4	H15A—C15—H15C	109.5
C4—C5—H5	121.4	H15B—C15—H15C	109.5
C5—C6—C7	122.58 (14)	N2—N1—C4	111.53 (13)
C5—C6—H6	118.7	N2—N1—C1	119.65 (15)

C7—C6—H6	118.7	C4—N1—C1	128.76 (15)
C8—C7—C6	120.47 (14)	C2—N2—N1	105.29 (12)
C8—C7—N3	123.47 (14)	C7—N3—S1	125.87 (10)
C6—C7—N3	115.98 (13)	C7—N3—H3N	116.4
C7—C8—C3	117.42 (14)	S1—N3—H3N	109.8
C7—C8—H8	121.3	C12—O3—C15	117.58 (15)
C3—C8—H8	121.3	O1—S1—O2	119.16 (7)
C14—C9—C10	120.00 (14)	O1—S1—N3	108.43 (7)
C14—C9—S1	119.80 (11)	O2—S1—N3	104.11 (7)
C10—C9—S1	120.20 (11)	O1—S1—C9	107.91 (7)
C11—C10—C9	119.45 (13)	O2—S1—C9	108.05 (7)
C11—C10—H10	120.3	N3—S1—C9	108.82 (7)
C9—C10—H10	120.3		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O2^{i}	0.89	2.04	2.9286 (17)	175
C5—H5···O3^{ii}	0.93	2.56	3.426 (2)	156

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+1; (ii) *x*, *y*−1, *z*.