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3-(4-Chlorobutyl)-3*H*-indole-5-carbonitrile

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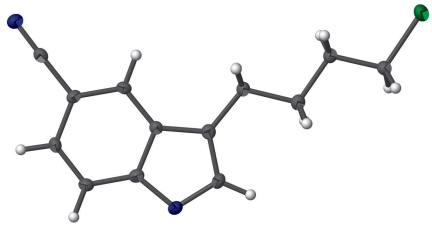
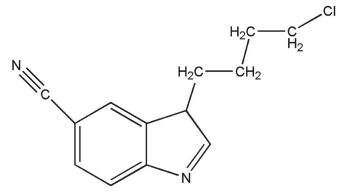
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_{12}ClN_2$, the indole moiety and the chloroalkyl substituent are nearly coplanar, making a dihedral angle of 1.27 (10)°. In the crystal, a supramolecular sheet parallel to the *ab* plane is generated *via* weak C—H···Cl hydrogen bonds, aromatic π–π stacking [centroid–centroid distances of 3.5563 (13) and 3.6792 (13) Å] and C—H···π interactions.

3D view



Chemical scheme



Structure description

The synthesis of indoles currently is of great interest because of their potential in acting as β-blockers, anti-arrhythmic, antiviral, anti-asthmatic, opioid antagonist and also sexual dysfunction drugs (Biswal *et al.*, 2012). The title compound, an indole derivative, is a key intermediate in the synthesis of the antidepressant drug vilazodone hydrochloride (Smith *et al.*, 1981). The present study focuses on its crystal structure and the non-covalent interactions present in it.

An *ORTEP* view of the title compound is shown in Fig. 1. The indole moiety (N1/C2–C9) and the chloroalkyl substituent (C11–C14/Cl1) are nearly coplanar, making a dihedral angle of 1.27 (10)°. The C11—C12—C13—C14 and C12—C13—C14—Cl1 torsion angles are −179.08 (14)° and 178.06 (12)° respectively.

In the crystal, neighboring molecules self-assemble through weak C—H···Cl interactions (Table 1), forming zigzag supramolecular *C*(9) chains extending along the *b*-axis direction, as shown in Fig. 2*a*. π–π stacking interactions are observed between the five- (N1/C2/C3/C9/C8; centroid *Cg*1) and six-membered rings (C4–C9, centroid *Cg*2) of symmetry-related molecules [$Cg1\cdots Cg2^{iv}$ = 3.6792 (13) Å, perpendicular distance = 3.3537 (7) Å, slip angle = 23.4°; $Cg2\cdots Cg2^{iv}$ = 3.5563 (13) Å, perpendicular distance = 3.3648 (7) Å, slip angle = 18.9°; symmetry code: (iv) $-x, 1-y, 1-z$] (Fig. 2*b*). Adjacent

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

*Cg*1 and *Cg*2 are the centroids of the N1/C2/C3/C9/C8 and C4–C9 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···Cl1^{i}	0.95	2.80	3.750 (2)	178
C13—H13*B*···*Cg*2^{ii}	0.99	2.83	3.729 (2)	151
C14—H14*A*···*Cg*1^{iii}	0.99	2.70	3.619 (2)	154

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

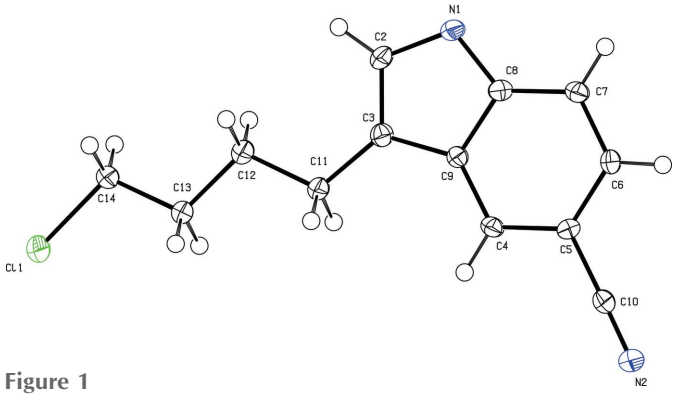


Figure 1
The asymmetric unit, shown in 50% probability displacement ellipsoids.

chains are linked by weak C—H···π interactions (Fig. 2*c*, Table 1), generating a supramolecular sheet-like architecture parallel to the *ab* plane.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{12}ClN_2$
M_r	231.70
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	8.870 (2), 9.271 (2), 14.498 (3)
β (°)	100.236 (4)
V (Å^3)	1173.3 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.30
Crystal size (mm)	0.18 × 0.17 × 0.15
Data collection	
Diffractometer	Bruker *APEX2*
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2008)
T_{min}, T_{max}	0.618, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7342, 1952, 1632
R_{int}	0.039
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.586
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.114, 1.04
No. of reflections	1952
No. of parameters	145
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.77, −0.26

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2008), *POV-RAY* (Cason, 2004) and *publCIF* (Westrip, 2010).

Synthesis and crystallization

Crystals of the title compound were obtained by dissolving 3-(4-chlorobutyl)-3*H*-indole-5-carbonitrile (purchased from the Tokyo Chemical Industry Co. Ltd; 58.17 mg, 0.25 mmol) in 20 ml of hot DMF, warming the resultant solution over a water bath for half an hour and then allowing it to evaporate slowly.

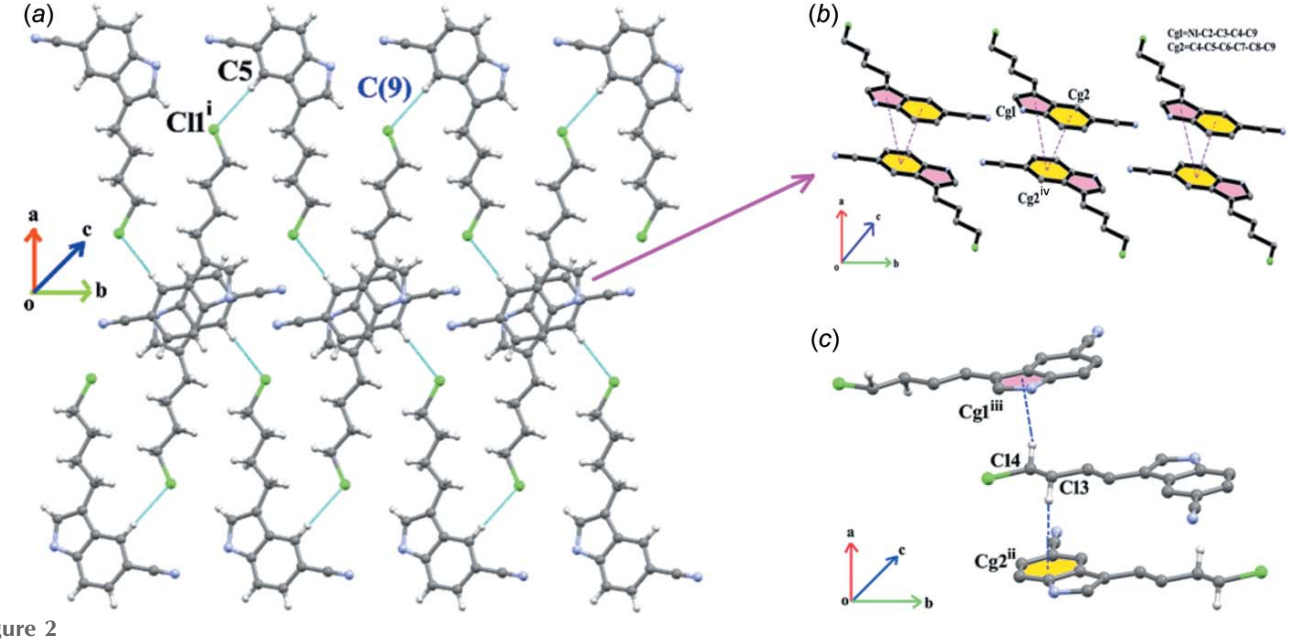


Figure 2
A view of a supramolecular sheet (*a*) generated *via* weak intermolecular C—H···Cl hydrogen bonds, aromatic π–π (*b*) and weak C—H···π interactions (*c*). [Symmetry codes: (i) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (ii) $1-x, 1-y, 1-z$; (iii) $x, \frac{3}{2}-y, -\frac{1}{2}+z$; (iv) $-x, 1-y, 1-z$.]

After a couple of weeks, colourless block-shaped crystals were obtained.

Refinement

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

Acknowledgements

The authors wish to acknowledge the assistance of Dr Matthias Zeller with the data collection and NSF Grant CHE 0087210, Ohio Board of Regents Grant CAP-491, and Youngstown State University for funds to purchase the X-ray diffractometer. RJB wishes to acknowledge NSF award 1205608, Partnership for Reduced Dimensional Materials, for partial funding of this research.

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full crystallographic data

IUCrData (2017). **2**, x170376 [https://doi.org/10.1107/S2414314617003765]

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Crystal data

$C_{13}H_{12}ClN_2$
M_r = 231.70
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.870 (2) Å
b = 9.271 (2) Å
c = 14.498 (3) Å
β = 100.236 (4)°
V = 1173.3 (4) Å^3
Z = 4
$F(000)$ = 484
D_x = 1.312 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1952 reflections
θ = 2.6–24.6°
μ = 0.30 mm^{-1}
T = 100 K
Block, colourless
0.18 × 0.17 × 0.15 mm

Data collection

Bruker APEX2 diffractometer
Detector resolution: 18.4 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2008)
T_{min} = 0.618, T_{max} = 0.745
7342 measured reflections
1952 independent reflections
1632 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 24.6°, θ_{min} = 2.6°
h = −10→10
k = −10→10
l = −17→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.114
S = 1.04
1952 reflections
145 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$W = 1/[\Sigma^2(FO^2) + (0.0787P)^2 + 0.103P]$ where $P = (FO^2 + 2FC^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs 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}
Cl1	0.64037 (6)	0.62791 (5)	0.10556 (3)	0.0374 (2)
N1	0.17148 (16)	0.77777 (15)	0.56156 (10)	0.0201 (5)
N2	0.11010 (18)	0.07016 (16)	0.63996 (10)	0.0253 (5)
C2	0.24695 (19)	0.77015 (18)	0.48563 (12)	0.0196 (5)
C3	0.27340 (19)	0.62955 (18)	0.46458 (12)	0.0180 (5)
C4	0.19954 (19)	0.39639 (19)	0.54477 (12)	0.0167 (5)
C5	0.12932 (19)	0.34712 (18)	0.61728 (12)	0.0171 (5)
C6	0.07011 (19)	0.44444 (18)	0.67723 (11)	0.0185 (5)
C7	0.0791 (2)	0.59138 (18)	0.66467 (12)	0.0191 (5)
C8	0.14883 (19)	0.63995 (18)	0.59107 (12)	0.0176 (5)
C9	0.21102 (18)	0.54455 (19)	0.53132 (11)	0.0153 (5)
C10	0.11668 (19)	0.19390 (19)	0.63026 (12)	0.0191 (5)
C11	0.34870 (18)	0.56955 (19)	0.38841 (11)	0.0182 (5)
C12	0.4092 (2)	0.68144 (18)	0.32641 (12)	0.0203 (5)
C13	0.4845 (2)	0.60776 (18)	0.25126 (11)	0.0190 (5)
C14	0.5469 (2)	0.71641 (19)	0.19066 (12)	0.0215 (5)
H2	0.27617	0.85126	0.45284	0.0240*
H4	0.23902	0.33015	0.50508	0.0200*
H6	0.02363	0.40800	0.72665	0.0220*
H7	0.03952	0.65724	0.70453	0.0230*
H11A	0.27405	0.50708	0.34793	0.0220*
H11B	0.43526	0.50774	0.41744	0.0220*
H12A	0.32352	0.74294	0.29572	0.0240*
H12B	0.48507	0.74416	0.36575	0.0240*
H13A	0.40796	0.54622	0.21144	0.0230*
H13B	0.56877	0.54487	0.28208	0.0230*
H14A	0.46219	0.77712	0.15794	0.0260*
H14B	0.62104	0.78010	0.23056	0.0260*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0600 (4)	0.0245 (3)	0.0365 (3)	0.0068 (2)	0.0330 (3)	0.0054 (2)
N1	0.0249 (8)	0.0132 (8)	0.0242 (8)	−0.0009 (6)	0.0095 (6)	−0.0002 (6)
N2	0.0355 (9)	0.0163 (9)	0.0265 (9)	0.0014 (7)	0.0123 (7)	0.0007 (6)
C2	0.0229 (9)	0.0149 (9)	0.0223 (9)	−0.0021 (7)	0.0074 (7)	0.0028 (7)
C3	0.0170 (9)	0.0180 (10)	0.0196 (9)	−0.0001 (7)	0.0053 (7)	0.0007 (7)
C4	0.0177 (9)	0.0154 (9)	0.0178 (9)	0.0020 (7)	0.0053 (7)	−0.0023 (7)
C5	0.0203 (9)	0.0125 (9)	0.0186 (9)	0.0001 (7)	0.0038 (7)	0.0006 (6)

C6	0.0202 (9)	0.0190 (9)	0.0182 (9)	−0.0007 (7)	0.0087 (7)	0.0012 (7)
C7	0.0219 (9)	0.0157 (9)	0.0213 (9)	0.0011 (7)	0.0079 (7)	−0.0024 (7)
C8	0.0182 (9)	0.0144 (10)	0.0203 (9)	0.0005 (7)	0.0036 (7)	−0.0007 (7)
C9	0.0153 (8)	0.0152 (9)	0.0160 (8)	0.0005 (6)	0.0043 (7)	−0.0001 (7)
C10	0.0206 (9)	0.0197 (10)	0.0187 (9)	0.0018 (7)	0.0078 (7)	−0.0003 (7)
C11	0.0209 (9)	0.0158 (9)	0.0194 (9)	0.0002 (7)	0.0074 (7)	0.0018 (7)
C12	0.0235 (10)	0.0179 (9)	0.0207 (9)	0.0004 (8)	0.0074 (7)	0.0027 (7)
C13	0.0236 (9)	0.0162 (9)	0.0186 (9)	−0.0006 (7)	0.0072 (7)	0.0009 (7)
C14	0.0279 (10)	0.0173 (9)	0.0218 (9)	0.0009 (7)	0.0113 (8)	−0.0002 (7)

Geometric parameters (Å, º)

Cl1—C14	1.8018 (19)	C12—C13	1.536 (2)
N1—C2	1.388 (2)	C13—C14	1.505 (2)
N1—C8	1.374 (2)	C2—H2	0.9500
N2—C10	1.159 (2)	C4—H4	0.9500
C2—C3	1.369 (2)	C6—H6	0.9500
C3—C9	1.433 (2)	C7—H7	0.9500
C3—C11	1.496 (2)	C11—H11A	0.9900
C4—C5	1.391 (2)	C11—H11B	0.9900
C4—C9	1.394 (3)	C12—H12A	0.9900
C5—C6	1.417 (2)	C12—H12B	0.9900
C5—C10	1.440 (2)	C13—H13A	0.9900
C6—C7	1.379 (2)	C13—H13B	0.9900
C7—C8	1.400 (2)	C14—H14A	0.9900
C8—C9	1.417 (2)	C14—H14B	0.9900
C11—C12	1.531 (2)		
C2—N1—C8	108.51 (14)	C9—C4—H4	121.00
N1—C2—C3	110.57 (15)	C5—C6—H6	120.00
C2—C3—C9	105.73 (15)	C7—C6—H6	120.00
C2—C3—C11	129.47 (16)	C6—C7—H7	121.00
C9—C3—C11	124.80 (15)	C8—C7—H7	121.00
C5—C4—C9	118.89 (16)	C3—C11—H11A	108.00
C4—C5—C6	121.27 (15)	C3—C11—H11B	108.00
C4—C5—C10	118.59 (15)	C12—C11—H11A	108.00
C6—C5—C10	120.14 (15)	C12—C11—H11B	108.00
C5—C6—C7	120.78 (15)	H11A—C11—H11B	107.00
C6—C7—C8	117.55 (15)	C11—C12—H12A	109.00
N1—C8—C7	130.22 (16)	C11—C12—H12B	109.00
N1—C8—C9	107.21 (15)	C13—C12—H12A	109.00
C7—C8—C9	122.57 (15)	C13—C12—H12B	109.00
C3—C9—C4	133.08 (16)	H12A—C12—H12B	108.00
C3—C9—C8	107.97 (15)	C12—C13—H13A	109.00
C4—C9—C8	118.93 (15)	C12—C13—H13B	109.00
N2—C10—C5	178.24 (19)	C14—C13—H13A	109.00
C3—C11—C12	115.51 (14)	C14—C13—H13B	109.00
C11—C12—C13	110.93 (14)	H13A—C13—H13B	108.00

C12—C13—C14	111.58 (14)	Cl1—C14—H14A	109.00
Cl1—C14—C13	110.89 (12)	Cl1—C14—H14B	109.00
N1—C2—H2	125.00	C13—C14—H14A	109.00
C3—C2—H2	125.00	C13—C14—H14B	109.00
C5—C4—H4	121.00	H14A—C14—H14B	108.00

C8—N1—C2—C3	0.3 (2)	C5—C4—C9—C8	−0.9 (2)
C2—N1—C8—C7	179.71 (18)	C4—C5—C6—C7	0.7 (3)
C2—N1—C8—C9	−0.42 (19)	C10—C5—C6—C7	−178.91 (16)
N1—C2—C3—C9	−0.1 (2)	C5—C6—C7—C8	−0.2 (3)
N1—C2—C3—C11	179.14 (16)	C6—C7—C8—N1	178.92 (17)
C2—C3—C9—C4	178.35 (18)	C6—C7—C8—C9	−0.9 (3)
C2—C3—C9—C8	−0.18 (19)	N1—C8—C9—C3	0.37 (19)
C11—C3—C9—C4	−0.9 (3)	N1—C8—C9—C4	−178.40 (15)
C11—C3—C9—C8	−179.45 (16)	C7—C8—C9—C3	−179.75 (16)
C2—C3—C11—C12	1.9 (3)	C7—C8—C9—C4	1.5 (3)
C9—C3—C11—C12	−179.01 (15)	C3—C11—C12—C13	179.74 (14)
C9—C4—C5—C6	−0.1 (3)	C11—C12—C13—C14	−179.08 (14)
C9—C4—C5—C10	179.47 (16)	C12—C13—C14—Cl1	178.06 (12)
C5—C4—C9—C3	−179.31 (18)		

Hydrogen-bond geometry (Å, º)

*Cg*1 and *Cg*2 are the centroids of the N1/C2/C3/C9/C8 and C4–C9 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···Cl1^{i}	0.95	2.80	3.750 (2)	178
C13—H13*B*···*Cg*2^{ii}	0.99	2.83	3.729 (2)	151
C14—H14*A*···*Cg*1^{iii}	0.99	2.70	3.619 (2)	154

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*, −*y*+3/2, *z*−1/2.