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5-Chloro-1-methylindoline-2,3-dione

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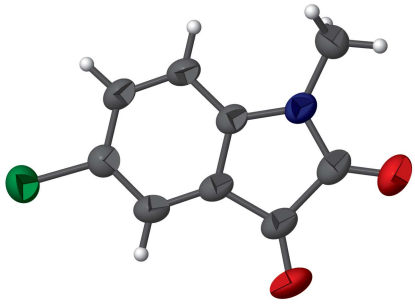
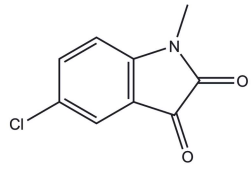
Structural data: full structural data are available from iucrdata.iucr.org

The title molecule, $C_9H_6ClNO_2$, is almost planar, with an r.m.s. deviation of the fitted non-hydrogen atoms of 0.0922 (19) Å. In the crystal, molecules are connected through methyl-C—H···O(carbonyl) interactions into supramolecular helical chains along the *b* axis. Inter-chain π–π interactions lead to layers parallel to the *ab* plane [centroid–centroid distances = 3.4861 (13) to 3.9767 (12) Å]. The crystal studied was a non-merohedral twin with a ratio of the twin components of 0.8612 (12): 0.1388 (12).

3D view



Chemical scheme



Structure description

5-Chloro-1*H*-indole-2,3-dione is a derivative of isatin (1*H*-Indole-2,3-dione) which has several interesting activities such as anti-tubercular (Aboul-Fadl *et al.*, 2010), cytotoxicity (Subba Reddy *et al.*, 2012) anti-inflammatory, (Anisetti *et al.*; 2012) anti-convulsant (Eggadi *et al.*, 2013) anxiolytic (Silva *et al.*, 2013) and anti-depressant (Radhika *et al.*, 2012).

The title compound (Fig. 1) was synthesized by the alkylation method (Kharbach *et al.*, 2015) under phase-transfer catalysis conditions (Bouhfid *et al.*, 2005). The crystal studied was a non-merohedral twin with a ratio of the twin components of 0.8612 (12): 0.1388 (12).

The title molecule, $C_9H_6ClNO_2$, is almost planar, with an r.m.s. deviation of 0.0922 (19) Å. In the crystal, molecules are connected through methyl--carbonyl C—H···O interactions (Table 1) into infinite chains along the *b* axis. The packing (Fig. 2) is also influenced by inter-chain π–π interactions, which form layers parallel to the *ab* plane [centroid–centroid distances = 3.4861 (13) to 3.9767 (12) Å].

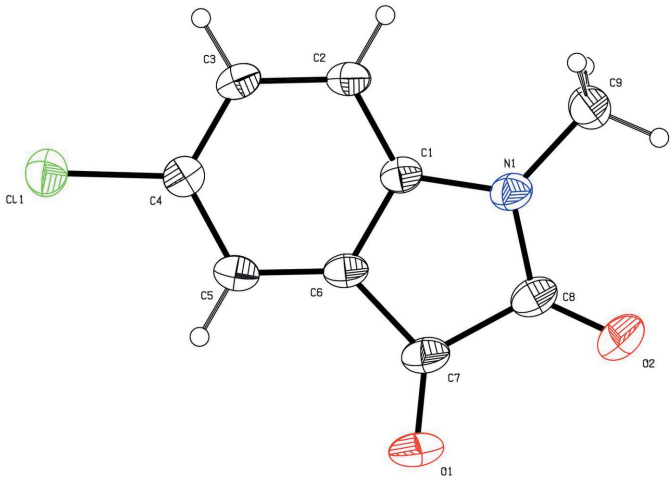


Figure 1
The molecular structure of the title molecule, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Synthesis and crystallization

To a solution of 5-chloro-1*H*-indole-2,3-dione (0.4 g, 2.20 mmol) in DMF (25 ml), were added potassium carbonate (0.5 g, 3.3 mmol), tetra-*n*-butylammonium fluoride (0.1 g, 0,3 mmol) and methyl iodide (0.13 ml, 2.42 mmol). The reaction mixture was stirred at ambient temperature for 48 h. The precipitate was filtered and processed yielding the title compound in a good yield of 89% in the form of red crystals (m.p. 361 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. At the final stage of refinement, an analysis of the data using the TwinRotMat routine in *PLATON* (Spek, 2009) revealed a minor twin (twofold axis around *c**). The twin matrix is (−0.999 0 0.002, 0 − 1 0, 1 0 0.999).

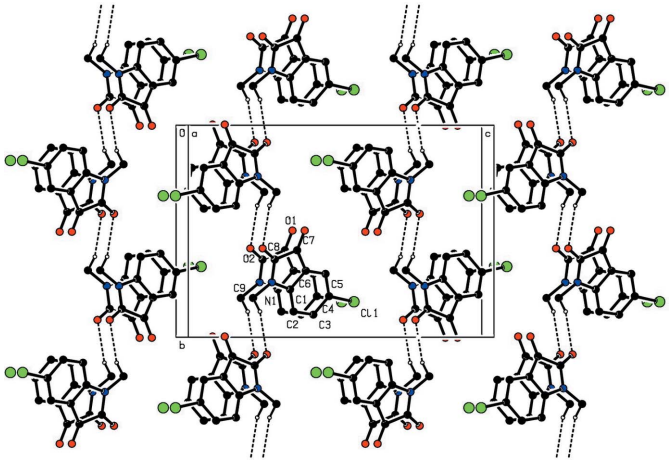


Figure 2
The crystal structure of the title compound, viewed along the *a* axis, showing chains parallel to the *b* axis linked by C—H···O hydrogen bonds (dashed lines). For the sake of clarity, H atoms not involved in the hydrogen bonds have been omitted.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*C*···O2^{i}	0.96	2.52	3.435 (3)	159

Symmetry code: (i) $-x+2, y+\frac{1}{2}, -z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_6ClNO_2$
M_r	195.60
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	300
a, *b*, *c* (Å)	3.9766 (4), 11.9503 (13), 17.947 (2)
β (°)	96.163 (3)
V (Å^3)	847.94 (16)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.41
Crystal size (mm)	0.29 × 0.11 × 0.11
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.697, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12975, 2080, 1705
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.041, 0.103, 1.05
No. of reflections	2080
No. of parameters	120
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.23

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *XT* (Sheldrick, 2015*a*), *SHELXL2015* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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full crystallographic data

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5-Chloro-1-methylindoline-2,3-dione

Crystal data

$C_9H_6ClNO_2$
M_r = 195.60
Monoclinic, $P2_1/c$
a = 3.9766 (4) Å
b = 11.9503 (13) Å
c = 17.947 (2) Å
β = 96.163 (3)°
V = 847.94 (16) Å^3
Z = 4
$F(000)$ = 400
D_x = 1.532 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4975 reflections
θ = 2.9–25.6°
μ = 0.41 mm^{-1}
T = 300 K
Prism, red
0.29 × 0.11 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.697, T_{max} = 0.746
12975 measured reflections
2080 independent reflections
1705 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 28.3°, θ_{min} = 1.7°
h = −5→5
k = −15→15
l = −22→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.05
2080 reflections
120 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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}
Cl1	0.19046 (16)	0.83313 (5)	0.57429 (3)	0.06020 (19)
N1	0.7031 (5)	0.74151 (13)	0.28515 (9)	0.0445 (4)
O2	0.9524 (5)	0.58155 (13)	0.24499 (10)	0.0701 (5)
C1	0.5640 (5)	0.77475 (14)	0.35050 (10)	0.0384 (4)
O1	0.7708 (6)	0.49696 (13)	0.38997 (10)	0.0740 (5)
C2	0.4421 (5)	0.87931 (15)	0.36668 (11)	0.0436 (4)
H2	0.4393	0.9377	0.3324	0.052*
C6	0.5649 (5)	0.68680 (14)	0.40190 (11)	0.0417 (4)
C3	0.3238 (5)	0.89379 (16)	0.43616 (11)	0.0449 (5)
H3	0.2388	0.9631	0.4485	0.054*
C4	0.3301 (5)	0.80689 (16)	0.48740 (11)	0.0439 (4)
C8	0.8099 (6)	0.63366 (16)	0.29043 (12)	0.0509 (5)
C5	0.4483 (5)	0.70138 (16)	0.47102 (12)	0.0459 (5)
H5	0.4490	0.6429	0.5052	0.055*
C9	0.7425 (7)	0.81520 (18)	0.22215 (12)	0.0552 (5)
H9A	0.5238	0.8390	0.2000	0.083*
H9B	0.8562	0.7758	0.1855	0.083*
H9C	0.8736	0.8794	0.2394	0.083*
C7	0.7167 (6)	0.59026 (15)	0.36734 (13)	0.0507 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0628 (4)	0.0650 (4)	0.0544 (3)	0.0064 (3)	0.0138 (3)	−0.0002 (2)
N1	0.0503 (10)	0.0335 (7)	0.0492 (9)	−0.0035 (7)	0.0036 (8)	−0.0052 (7)
O2	0.0883 (13)	0.0505 (9)	0.0740 (10)	0.0077 (9)	0.0199 (11)	−0.0168 (8)
C1	0.0363 (9)	0.0325 (8)	0.0448 (9)	−0.0052 (7)	−0.0027 (8)	−0.0043 (7)
O1	0.1058 (15)	0.0303 (7)	0.0867 (12)	0.0099 (8)	0.0146 (12)	0.0037 (7)
C2	0.0494 (11)	0.0317 (8)	0.0475 (10)	0.0028 (8)	−0.0041 (9)	0.0022 (8)
C6	0.0418 (10)	0.0287 (8)	0.0532 (11)	−0.0032 (7)	−0.0013 (9)	−0.0009 (7)
C3	0.0441 (10)	0.0355 (9)	0.0538 (11)	0.0067 (8)	−0.0014 (10)	−0.0053 (8)
C4	0.0389 (10)	0.0451 (10)	0.0471 (10)	−0.0009 (8)	0.0021 (9)	−0.0017 (8)
C8	0.0550 (12)	0.0362 (9)	0.0605 (12)	−0.0035 (9)	0.0019 (11)	−0.0122 (9)
C5	0.0476 (11)	0.0358 (9)	0.0532 (12)	−0.0024 (8)	0.0005 (10)	0.0064 (8)
C9	0.0688 (15)	0.0495 (11)	0.0478 (11)	−0.0051 (11)	0.0090 (11)	0.0000 (9)
C7	0.0577 (13)	0.0313 (9)	0.0619 (12)	−0.0023 (9)	0.0004 (11)	−0.0037 (9)

Geometric parameters (Å, º)

Cl1—C4	1.739 (2)	C6—C5	1.381 (3)
N1—C1	1.407 (3)	C6—C7	1.470 (3)
N1—C8	1.357 (3)	C3—H3	0.9300
N1—C9	1.455 (3)	C3—C4	1.386 (3)
O2—C8	1.214 (3)	C4—C5	1.388 (3)
C1—C2	1.382 (3)	C8—C7	1.556 (3)

C1—C6	1.398 (3)	C5—H5	0.9300
O1—C7	1.198 (2)	C9—H9A	0.9600
C2—H2	0.9300	C9—H9B	0.9600
C2—C3	1.390 (3)	C9—H9C	0.9600
C1—N1—C9	124.18 (16)	C5—C4—Cl1	120.11 (16)
C8—N1—C1	110.98 (17)	N1—C8—C7	106.02 (17)
C8—N1—C9	124.77 (18)	O2—C8—N1	127.3 (2)
C2—C1—N1	127.50 (17)	O2—C8—C7	126.71 (19)
C2—C1—C6	121.13 (18)	C6—C5—C4	117.31 (18)
C6—C1—N1	111.37 (16)	C6—C5—H5	121.3
C1—C2—H2	121.3	C4—C5—H5	121.3
C1—C2—C3	117.46 (17)	N1—C9—H9A	109.5
C3—C2—H2	121.3	N1—C9—H9B	109.5
C1—C6—C7	106.48 (17)	N1—C9—H9C	109.5
C5—C6—C1	121.36 (17)	H9A—C9—H9B	109.5
C5—C6—C7	132.12 (18)	H9A—C9—H9C	109.5
C2—C3—H3	119.4	H9B—C9—H9C	109.5
C4—C3—C2	121.22 (17)	O1—C7—C6	130.9 (2)
C4—C3—H3	119.4	O1—C7—C8	124.0 (2)
C3—C4—Cl1	118.38 (15)	C6—C7—C8	105.09 (15)
C3—C4—C5	121.51 (19)		
Cl1—C4—C5—C6	−178.13 (16)	C2—C1—C6—C7	−178.79 (18)
N1—C1—C2—C3	−178.3 (2)	C2—C3—C4—Cl1	177.88 (16)
N1—C1—C6—C5	178.22 (19)	C2—C3—C4—C5	−1.3 (3)
N1—C1—C6—C7	0.3 (2)	C6—C1—C2—C3	0.6 (3)
N1—C8—C7—O1	179.0 (2)	C3—C4—C5—C6	1.0 (3)
N1—C8—C7—C6	−2.1 (2)	C8—N1—C1—C2	177.2 (2)
O2—C8—C7—O1	−1.8 (4)	C8—N1—C1—C6	−1.7 (2)
O2—C8—C7—C6	177.2 (2)	C5—C6—C7—O1	2.3 (4)
C1—N1—C8—O2	−177.0 (2)	C5—C6—C7—C8	−176.6 (2)
C1—N1—C8—C7	2.3 (2)	C9—N1—C1—C2	0.2 (3)
C1—C2—C3—C4	0.4 (3)	C9—N1—C1—C6	−178.75 (19)
C1—C6—C5—C4	0.0 (3)	C9—N1—C8—O2	0.0 (4)
C1—C6—C7—O1	179.9 (3)	C9—N1—C8—C7	179.3 (2)
C1—C6—C7—C8	1.1 (2)	C7—C6—C5—C4	177.4 (2)
C2—C1—C6—C5	−0.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*C*···O2^{i}	0.96	2.52	3.435 (3)	159

Symmetry code: (i) −*x*+2, *y*+1/2, −*z*+1/2.