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4-Chloro-1*H*-indole-2,3-dione

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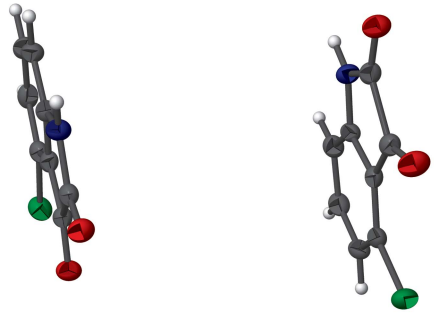
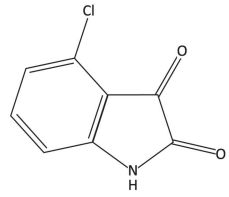
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_4ClNO_2$, has two planar molecules in the asymmetric unit, with the non-H atoms showing a mean deviation from planarity of 0.015 and 0.022 Å, respectively. In the crystal, the molecules are linked through N—H···O hydrogen bonds to form infinite chains along [010]. They are further connected through C—H···Cl close contacts with a donor–acceptor distance of 3.682 (5) Å. No π–π interactions are observed in the structure.

3D view



Chemical scheme



Structure description

As part of a continuing study of halogenated isatins, we report the crystal structure of 4-chloroisatin (4-chloro-1*H*-indole-2,3-dione), Fig. 1. The asymmetric unit of the structure contains two molecules that are nearly planar, with the non-H atoms possessing a mean deviation from planarity of 0.015 and 0.022 Å, respectively. The bond lengths and angles observed in the molecule are similar to those observed in the parent isatin (Goldschmidt & Llewellyn, 1950).

In the crystal, molecules form infinite chains along [010] through N1—H1···O3 and N2—H2···O1 hydrogen bonds (Table 1). These chains are further linked through C5—H5···Cl2 close contacts (Fig. 2). Similar C—H···Cl close contacts were not observed in the other 4-chloroisatin structures that have been reported (Hughes & Fenical, 2010; Wang *et al.*, 2012; Yu *et al.*, 2012). Halogen–oxygen rather than C—H···halogen interactions are observed in the structures of 4-bromoisatin (Huang *et al.*, 2016) and 4-iodoisatin (Golen & Manke, 2016).

Synthesis and crystallization

A commercial sample (Matrix Scientific) of 4-chloro-1*H*-indole-2,3-dione was used for crystallization. A sample suitable for single-crystal X-ray analysis was grown by slow evaporation from an acetone solution.

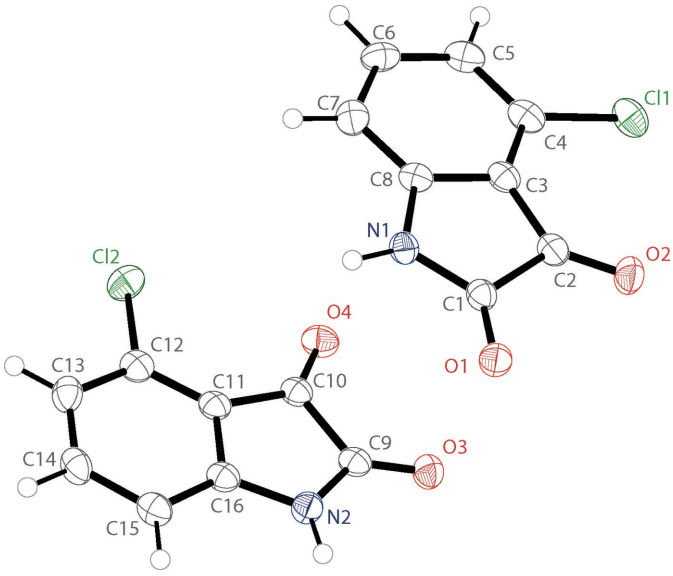


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

Refinement

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

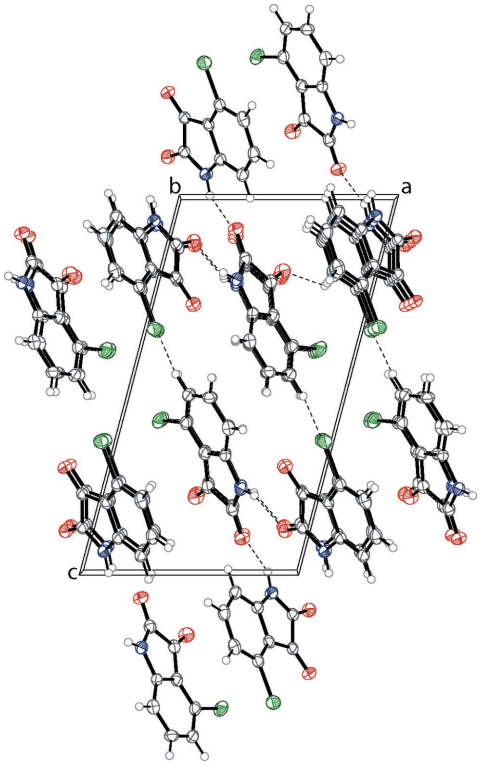


Figure 2
The molecular packing of the title compound viewed along the *b* axis, with hydrogen bonding shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.87 (1)	1.97 (2)	2.768 (5)	152 (4)
N2—H2···O1^{ii}	0.87 (1)	2.11 (2)	2.905 (4)	152 (5)
C5—H5···Cl2^{iii}	0.95	2.91	3.682 (5)	139

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_4ClNO_2$
M_r	181.57
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	120
a, b, c (Å)	8.2745 (8), 6.0367 (6), 14.8609 (14)
β (°)	104.746 (5)
V (Å^3)	717.86 (12)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	4.31
Crystal size (mm)	0.25 × 0.04 × 0.04
Data collection	
Diffractometer	Bruker CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.205, 0.311
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9961, 2579, 2388
R_{int}	0.058
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.603
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.096, 1.04
No. of reflections	2579
No. of parameters	224
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.58, −0.27
Absolute structure	Refined as an inversion twin.
Absolute structure parameter	0.11 (3)

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160689 [doi:10.1107/S2414314616006891]

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Crystal data

$C_8H_4ClNO_2$
M_r = 181.57
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 8.2745 (8) Å
b = 6.0367 (6) Å
c = 14.8609 (14) Å
β = 104.746 (5)°
V = 717.86 (12) Å^3
Z = 4
$F(000)$ = 368
D_x = 1.680 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 6743 reflections
θ = 3.1–68.5°
μ = 4.31 mm^{-1}
T = 120 K
Block, orange
0.25 × 0.04 × 0.04 mm

Data collection

Bruker CMOS diffractometer
Radiation source: Cu
HELIOS MX monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.205, T_{max} = 0.311
9961 measured reflections
2579 independent reflections
2388 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 68.5°, θ_{min} = 3.1°
h = −9→9
k = −7→7
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.096
S = 1.04
2579 reflections
224 parameters
3 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 0.171P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: 0.11 (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.

Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.82671 (11)	−0.28031 (19)	0.40490 (7)	0.0329 (3)
Cl2	1.05021 (12)	1.19333 (19)	0.35368 (6)	0.0317 (3)
O1	0.2975 (4)	−0.0106 (5)	0.09037 (18)	0.0293 (7)
O2	0.5638 (3)	−0.2926 (6)	0.19884 (19)	0.0332 (7)
O3	1.1154 (3)	0.4626 (5)	0.11689 (18)	0.0279 (7)
O4	1.1901 (3)	0.7341 (6)	0.28454 (17)	0.0289 (7)
N1	0.3652 (4)	0.2002 (7)	0.2246 (2)	0.0246 (7)
H1	0.295 (5)	0.309 (6)	0.208 (3)	0.029*
N2	0.9124 (4)	0.7291 (7)	0.0657 (2)	0.0248 (8)
H2	0.868 (5)	0.688 (9)	0.0088 (14)	0.030*
C1	0.3785 (5)	0.0273 (7)	0.1694 (3)	0.0240 (9)
C2	0.5204 (5)	−0.1236 (7)	0.2281 (3)	0.0251 (9)
C3	0.5750 (5)	−0.0089 (7)	0.3177 (3)	0.0235 (9)
C4	0.6994 (5)	−0.0499 (8)	0.3987 (3)	0.0267 (9)
C5	0.7209 (5)	0.0939 (8)	0.4734 (3)	0.0285 (10)
H5	0.8050	0.0660	0.5290	0.034*
C6	0.6195 (5)	0.2785 (8)	0.4667 (3)	0.0285 (10)
H6	0.6340	0.3741	0.5189	0.034*
C7	0.4963 (5)	0.3293 (8)	0.3859 (3)	0.0281 (9)
H7	0.4289	0.4582	0.3815	0.034*
C8	0.4775 (4)	0.1828 (8)	0.3127 (2)	0.0226 (8)
C9	1.0412 (5)	0.6310 (7)	0.1266 (3)	0.0229 (9)
C10	1.0799 (5)	0.7769 (7)	0.2159 (3)	0.0221 (9)
C11	0.9586 (5)	0.9583 (7)	0.1941 (3)	0.0229 (9)
C12	0.9285 (5)	1.1431 (7)	0.2424 (3)	0.0252 (9)
C13	0.8008 (5)	1.2893 (8)	0.2018 (3)	0.0290 (10)
H13	0.7766	1.4130	0.2357	0.035*
C14	0.7089 (5)	1.2512 (8)	0.1105 (3)	0.0297 (10)
H14	0.6232	1.3527	0.0823	0.036*
C15	0.7387 (5)	1.0694 (8)	0.0594 (3)	0.0273 (10)
H15	0.6758	1.0467	−0.0029	0.033*
C16	0.8623 (5)	0.9235 (7)	0.1022 (3)	0.0237 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0260 (5)	0.0331 (6)	0.0362 (5)	0.0057 (5)	0.0014 (4)	0.0081 (5)
Cl2	0.0317 (5)	0.0347 (6)	0.0287 (5)	−0.0035 (5)	0.0076 (4)	−0.0071 (5)
O1	0.0320 (15)	0.0288 (18)	0.0237 (14)	0.0041 (13)	0.0010 (12)	−0.0016 (12)
O2	0.0370 (15)	0.0285 (18)	0.0328 (14)	0.0111 (17)	0.0064 (12)	−0.0021 (16)
O3	0.0281 (14)	0.0259 (17)	0.0290 (14)	0.0051 (14)	0.0061 (12)	0.0007 (13)
O4	0.0260 (13)	0.035 (2)	0.0231 (13)	0.0010 (15)	0.0006 (11)	0.0019 (14)
N1	0.0230 (16)	0.0250 (19)	0.0253 (15)	0.0065 (17)	0.0054 (12)	0.0030 (17)
N2	0.0238 (16)	0.026 (2)	0.0228 (15)	0.0023 (16)	0.0026 (12)	−0.0016 (15)
C1	0.023 (2)	0.023 (2)	0.027 (2)	0.0023 (17)	0.0082 (17)	0.0029 (17)

C2	0.022 (2)	0.025 (2)	0.029 (2)	0.0027 (17)	0.0095 (17)	0.0053 (18)
C3	0.0212 (19)	0.024 (2)	0.0264 (19)	−0.0024 (17)	0.0081 (15)	0.0050 (17)
C4	0.0191 (19)	0.031 (3)	0.030 (2)	−0.0020 (18)	0.0067 (16)	0.0081 (18)
C5	0.021 (2)	0.039 (3)	0.024 (2)	−0.0023 (19)	0.0034 (16)	0.0054 (18)
C6	0.030 (2)	0.035 (3)	0.0202 (19)	−0.0077 (19)	0.0056 (17)	−0.0027 (17)
C7	0.025 (2)	0.028 (3)	0.033 (2)	−0.0001 (18)	0.0099 (18)	0.0007 (18)
C8	0.0179 (17)	0.028 (2)	0.0219 (17)	−0.0005 (18)	0.0051 (14)	0.0031 (17)
C9	0.0207 (19)	0.025 (2)	0.0237 (19)	−0.0012 (17)	0.0078 (15)	0.0035 (16)
C10	0.0199 (19)	0.025 (2)	0.0226 (19)	−0.0002 (16)	0.0069 (16)	0.0037 (15)
C11	0.0214 (19)	0.025 (2)	0.0245 (18)	−0.0005 (17)	0.0098 (15)	0.0043 (17)
C12	0.0214 (19)	0.028 (3)	0.028 (2)	−0.0020 (17)	0.0099 (16)	0.0025 (17)
C13	0.029 (2)	0.024 (2)	0.039 (2)	0.0022 (17)	0.0165 (19)	0.0006 (18)
C14	0.0245 (19)	0.028 (3)	0.039 (2)	0.0070 (18)	0.0126 (17)	0.0083 (19)
C15	0.021 (2)	0.032 (3)	0.029 (2)	0.0031 (17)	0.0052 (17)	0.0057 (18)
C16	0.0213 (19)	0.027 (2)	0.0241 (19)	−0.0003 (17)	0.0076 (15)	0.0037 (17)

Geometric parameters (Å, º)

Cl1—C4	1.733 (5)	C5—H5	0.9500
Cl2—C12	1.730 (4)	C5—C6	1.383 (7)
O1—C1	1.217 (5)	C6—H6	0.9500
O2—C2	1.200 (6)	C6—C7	1.398 (6)
O3—C9	1.215 (5)	C7—H7	0.9500
O4—C10	1.210 (5)	C7—C8	1.379 (6)
N1—H1	0.869 (7)	C9—C10	1.557 (6)
N1—C1	1.349 (6)	C10—C11	1.465 (6)
N1—C8	1.404 (4)	C11—C12	1.383 (6)
N2—H2	0.869 (7)	C11—C16	1.410 (6)
N2—C9	1.347 (5)	C12—C13	1.391 (6)
N2—C16	1.399 (6)	C13—H13	0.9500
C1—C2	1.564 (6)	C13—C14	1.395 (6)
C2—C3	1.467 (6)	C14—H14	0.9500
C3—C4	1.393 (6)	C14—C15	1.392 (6)
C3—C8	1.402 (6)	C15—H15	0.9500
C4—C5	1.385 (6)	C15—C16	1.376 (6)
C1—N1—H1	124 (3)	C3—C8—N1	110.3 (4)
C1—N1—C8	111.9 (4)	C7—C8—N1	127.1 (4)
C8—N1—H1	124 (3)	C7—C8—C3	122.7 (4)
C9—N2—H2	127 (4)	O3—C9—N2	128.7 (4)
C9—N2—C16	111.7 (3)	O3—C9—C10	125.0 (4)
C16—N2—H2	122 (4)	N2—C9—C10	106.3 (3)
O1—C1—N1	128.5 (4)	O4—C10—C9	123.0 (4)
O1—C1—C2	125.4 (4)	O4—C10—C11	132.3 (4)
N1—C1—C2	106.1 (3)	C11—C10—C9	104.7 (3)
O2—C2—C1	123.0 (4)	C12—C11—C10	133.8 (4)
O2—C2—C3	132.8 (4)	C12—C11—C16	119.4 (4)
C3—C2—C1	104.2 (3)	C16—C11—C10	106.9 (3)

C4—C3—C2	133.7 (4)	C11—C12—Cl2	120.0 (3)
C4—C3—C8	118.8 (4)	C11—C12—C13	120.3 (4)
C8—C3—C2	107.5 (3)	C13—C12—Cl2	119.7 (3)
C3—C4—Cl1	119.6 (3)	C12—C13—H13	120.6
C5—C4—Cl1	120.6 (3)	C12—C13—C14	118.9 (4)
C5—C4—C3	119.8 (4)	C14—C13—H13	120.6
C4—C5—H5	120.2	C13—C14—H14	118.9
C6—C5—C4	119.7 (4)	C15—C14—C13	122.1 (4)
C6—C5—H5	120.2	C15—C14—H14	118.9
C5—C6—H6	118.8	C14—C15—H15	121.1
C5—C6—C7	122.4 (4)	C16—C15—C14	117.8 (4)
C7—C6—H6	118.8	C16—C15—H15	121.1
C6—C7—H7	121.7	N2—C16—C11	110.5 (3)
C8—C7—C6	116.6 (4)	C15—C16—N2	128.0 (4)
C8—C7—H7	121.7	C15—C16—C11	121.5 (4)
Cl1—C4—C5—C6	179.2 (3)	C5—C6—C7—C8	1.4 (6)
Cl2—C12—C13—C14	177.2 (3)	C6—C7—C8—N1	−178.8 (4)
O1—C1—C2—O2	1.0 (7)	C6—C7—C8—C3	0.3 (6)
O1—C1—C2—C3	−178.8 (4)	C8—N1—C1—O1	178.1 (4)
O2—C2—C3—C4	2.8 (8)	C8—N1—C1—C2	−1.0 (4)
O2—C2—C3—C8	−179.3 (4)	C8—C3—C4—Cl1	−177.6 (3)
O3—C9—C10—O4	0.3 (6)	C8—C3—C4—C5	2.1 (6)
O3—C9—C10—C11	−179.9 (4)	C9—N2—C16—C11	−1.7 (4)
O4—C10—C11—C12	1.0 (8)	C9—N2—C16—C15	178.1 (4)
O4—C10—C11—C16	179.0 (4)	C9—C10—C11—C12	−178.7 (4)
N1—C1—C2—O2	−179.9 (4)	C9—C10—C11—C16	−0.8 (4)
N1—C1—C2—C3	0.3 (4)	C10—C11—C12—Cl2	0.0 (6)
N2—C9—C10—O4	−180.0 (4)	C10—C11—C12—C13	179.9 (4)
N2—C9—C10—C11	−0.2 (4)	C10—C11—C16—N2	1.5 (4)
C1—N1—C8—C3	1.3 (5)	C10—C11—C16—C15	−178.4 (4)
C1—N1—C8—C7	−179.6 (4)	C11—C12—C13—C14	−2.7 (6)
C1—C2—C3—C4	−177.4 (4)	C12—C11—C16—N2	179.8 (3)
C1—C2—C3—C8	0.4 (4)	C12—C11—C16—C15	0.0 (6)
C2—C3—C4—Cl1	0.0 (6)	C12—C13—C14—C15	1.3 (6)
C2—C3—C4—C5	179.7 (4)	C13—C14—C15—C16	0.7 (6)
C2—C3—C8—N1	−1.0 (4)	C14—C15—C16—N2	178.9 (4)
C2—C3—C8—C7	179.8 (4)	C14—C15—C16—C11	−1.3 (6)
C3—C4—C5—C6	−0.5 (6)	C16—N2—C9—O3	−179.2 (4)
C4—C3—C8—N1	177.2 (3)	C16—N2—C9—C10	1.1 (4)
C4—C3—C8—C7	−2.0 (6)	C16—C11—C12—Cl2	−177.8 (3)
C4—C5—C6—C7	−1.3 (6)	C16—C11—C12—C13	2.1 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.87 (1)	1.97 (2)	2.768 (5)	152 (4)

N2—H2···O1^{ii}	0.87 (1)	2.11 (2)	2.905 (4)	152 (5)
C5—H5···Cl2^{iii}	0.95	2.91	3.682 (5)	139

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