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5,7-Dichloro-1*H*-indole-2,3-dione

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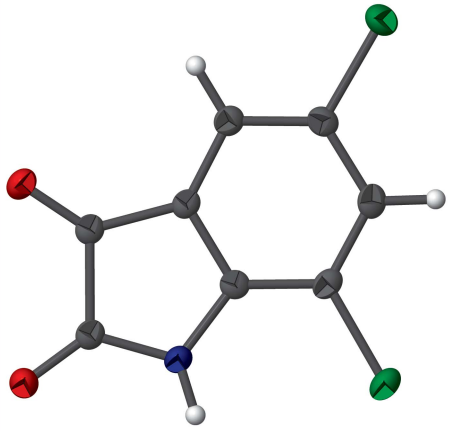
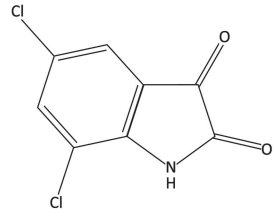
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_3Cl_2NO_2$, has a single molecule in the asymmetric unit that is close to planar, with the non-H atoms having a mean deviation from planarity of 0.035 Å. The molecules dimerize through two N—H···O hydrogen bonds. A weak intermolecular offset π–π interaction is also observed between the five- and six-membered rings, with a centroid–centroid separation of 3.8444 (16) Å.

3D view



Chemical scheme



Structure description

Herein we report the crystal structure of 5,7-dichloroisatin (Fig. 1). There is a single, near-planar molecule in the asymmetric unit that has a mean deviation from planarity for the non-H atoms of 0.035 Å. The bond distances and angles observed for the title compound are consistent with those observed in 1*H*-indole-2,3-dione (Goldschmidt & Llewellyn, 1950).

In the crystal, the molecules dimerize through N1—H1···O1 hydrogen bonds (Table 1) and these dimers are stacked along the *b*-axis direction by a weak π–π contact between the N1/C1–C3/C8 and C3–C8 rings with an inter-centroid distance of 3.8444 (16) Å (Fig. 2). The monosubstituted 5-chloroisatin and 7-chloroisatin demonstrate C—H···O interactions in the solid state (Sun & Cai, 2010; Wei *et al.*, 2010), while no such interaction is observed for the 5,7-disubstituted complex reported here. The 4,7-dichloro isomer of the title compound dimerizes through N—H···O hydrogen bonds in a similar fashion to 5,7-dichloroisatin, but unlike the title compound, it also has C—H···O as well as π–π interactions (Golen & Manke, 2016). The 4,6-dichloro isomer only has N—H···O intermolecular interactions, though these form chains rather than dimers (Mastrolia *et al.*, 2016).

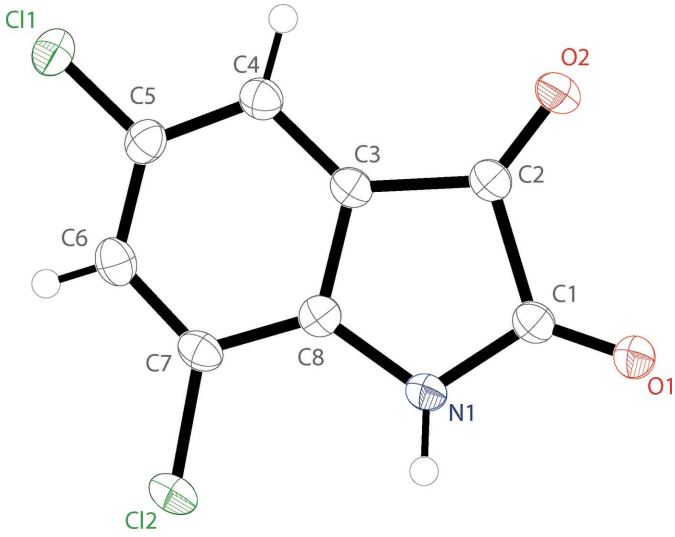


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

Synthesis and crystallization

A commercial sample (Matrix Scientific) of 5,7-dichloroisatin was recrystallized from the slow evaporation of an acetone solution to yield orange blocks suitable for single-crystal diffraction analysis.

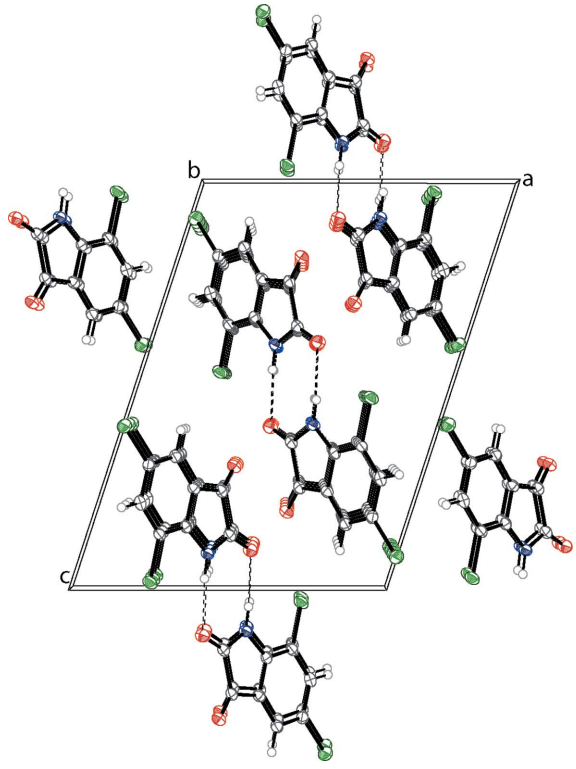


Figure 2
The molecular packing of the title compound along the *b* axis, with hydrogen bonds shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86 (2)	1.98 (2)	2.832 (2)	167 (3)

Symmetry code: (i) $-x+1, -y, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_3Cl_2NO_2$
M_r	216.01
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, b, c (Å)	11.0789 (17), 4.9866 (8), 15.049 (2)
β (°)	108.041 (7)
V (Å^3)	790.5 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	7.08
Crystal size (mm)	0.24 × 0.16 × 0.05
Data collection	
Diffractometer	Bruker D8 Venture CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.288, 0.468
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10168, 1496, 1356
R_{int}	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.611
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.086, 1.06
No. of reflections	1496
No. of parameters	121
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.34

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

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161510 [doi:10.1107/S2414314616015108]

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Crystal data

$C_8H_3Cl_2NO_2$
M_r = 216.01
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.0789 (17) Å
b = 4.9866 (8) Å
c = 15.049 (2) Å
β = 108.041 (7)°
V = 790.5 (2) Å^3
Z = 4
$F(000)$ = 432
D_x = 1.815 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 6137 reflections
θ = 4.2–70.4°
μ = 7.08 mm^{-1}
T = 120 K
Block, orange
0.24 × 0.16 × 0.05 mm

Data collection

Bruker D8 Venture CMOS diffractometer
Radiation source: Cu
HELIOS MX monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.288, T_{max} = 0.468
10168 measured reflections
1496 independent reflections
1356 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 70.4°, θ_{min} = 4.2°
h = −13→13
k = −5→6
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.086
S = 1.06
1496 reflections
121 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.9613P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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	0.97776 (5)	0.75293 (13)	0.90244 (4)	0.02880 (18)
Cl2	0.74527 (5)	0.65413 (12)	0.53083 (4)	0.02535 (17)
O1	0.46893 (16)	−0.1568 (3)	0.60119 (11)	0.0238 (4)
O2	0.60384 (15)	−0.1147 (3)	0.80286 (11)	0.0220 (4)
N1	0.59860 (19)	0.2023 (4)	0.59574 (13)	0.0199 (4)
H1	0.582 (3)	0.213 (6)	0.5359 (12)	0.024*
C1	0.5484 (2)	0.0101 (5)	0.63789 (15)	0.0196 (5)
C2	0.6184 (2)	0.0373 (5)	0.74484 (15)	0.0191 (5)
C3	0.7063 (2)	0.2649 (5)	0.75254 (15)	0.0186 (5)
C4	0.7939 (2)	0.3839 (5)	0.82884 (16)	0.0207 (5)
H4	0.8036	0.3267	0.8909	0.025*
C5	0.8670 (2)	0.5915 (5)	0.81031 (16)	0.0210 (5)
C6	0.8533 (2)	0.6781 (5)	0.71998 (16)	0.0226 (5)
H6	0.9047	0.8202	0.7098	0.027*
C7	0.7645 (2)	0.5570 (5)	0.64436 (15)	0.0203 (5)
C8	0.6913 (2)	0.3505 (5)	0.66125 (15)	0.0184 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0247 (3)	0.0351 (4)	0.0249 (3)	−0.0087 (2)	0.0053 (2)	−0.0035 (2)
Cl2	0.0295 (3)	0.0284 (3)	0.0214 (3)	0.0001 (2)	0.0126 (2)	0.0049 (2)
O1	0.0273 (9)	0.0232 (9)	0.0213 (8)	−0.0055 (7)	0.0082 (7)	−0.0017 (7)
O2	0.0268 (8)	0.0213 (9)	0.0203 (8)	0.0003 (7)	0.0109 (7)	0.0018 (7)
N1	0.0227 (10)	0.0218 (11)	0.0164 (9)	−0.0020 (8)	0.0079 (8)	0.0001 (8)
C1	0.0206 (11)	0.0201 (12)	0.0203 (11)	0.0016 (9)	0.0096 (9)	−0.0003 (9)
C2	0.0186 (10)	0.0199 (12)	0.0207 (11)	0.0019 (9)	0.0090 (9)	−0.0003 (9)
C3	0.0205 (11)	0.0177 (11)	0.0191 (11)	0.0020 (9)	0.0083 (9)	0.0010 (8)
C4	0.0200 (11)	0.0229 (12)	0.0208 (11)	0.0014 (9)	0.0086 (9)	0.0005 (9)
C5	0.0166 (10)	0.0241 (13)	0.0221 (11)	0.0004 (9)	0.0058 (9)	−0.0024 (9)
C6	0.0207 (11)	0.0227 (13)	0.0277 (12)	−0.0002 (9)	0.0124 (10)	0.0000 (10)
C7	0.0206 (11)	0.0228 (12)	0.0200 (10)	0.0035 (9)	0.0102 (9)	0.0030 (9)
C8	0.0173 (10)	0.0192 (11)	0.0199 (10)	0.0028 (9)	0.0077 (9)	−0.0017 (9)

Geometric parameters (Å, º)

Cl1—C5	1.738 (2)	C3—C4	1.386 (3)
Cl2—C7	1.724 (2)	C3—C8	1.399 (3)
O1—C1	1.213 (3)	C4—H4	0.9500
O2—C2	1.204 (3)	C4—C5	1.395 (3)
N1—H1	0.864 (17)	C5—C6	1.389 (3)
N1—C1	1.360 (3)	C6—H6	0.9500
N1—C8	1.395 (3)	C6—C7	1.391 (3)
C1—C2	1.561 (3)	C7—C8	1.382 (3)
C2—C3	1.477 (3)		

C1—N1—H1	123.2 (19)	C5—C4—H4	121.5
C1—N1—C8	111.21 (19)	C4—C5—Cl1	119.63 (17)
C8—N1—H1	125.0 (19)	C6—C5—Cl1	118.20 (18)
O1—C1—N1	128.0 (2)	C6—C5—C4	122.2 (2)
O1—C1—C2	126.0 (2)	C5—C6—H6	120.0
N1—C1—C2	105.96 (19)	C5—C6—C7	120.0 (2)
O2—C2—C1	123.9 (2)	C7—C6—H6	120.0
O2—C2—C3	131.3 (2)	C6—C7—Cl2	121.84 (18)
C3—C2—C1	104.65 (18)	C8—C7—Cl2	119.48 (18)
C4—C3—C2	132.0 (2)	C8—C7—C6	118.7 (2)
C4—C3—C8	121.5 (2)	N1—C8—C3	111.7 (2)
C8—C3—C2	106.41 (19)	C7—C8—N1	127.6 (2)
C3—C4—H4	121.5	C7—C8—C3	120.7 (2)
C3—C4—C5	116.9 (2)		

Cl1—C5—C6—C7	−179.08 (18)	C2—C3—C8—N1	1.7 (3)
Cl2—C7—C8—N1	−0.3 (3)	C2—C3—C8—C7	−177.7 (2)
Cl2—C7—C8—C3	179.01 (17)	C3—C4—C5—Cl1	179.36 (17)
O1—C1—C2—O2	3.1 (4)	C3—C4—C5—C6	0.3 (3)
O1—C1—C2—C3	178.8 (2)	C4—C3—C8—N1	179.8 (2)
O2—C2—C3—C4	−4.3 (4)	C4—C3—C8—C7	0.3 (3)
O2—C2—C3—C8	173.5 (2)	C4—C5—C6—C7	0.0 (4)
N1—C1—C2—O2	−174.5 (2)	C5—C6—C7—Cl2	−179.15 (18)
N1—C1—C2—C3	1.2 (2)	C5—C6—C7—C8	−0.1 (3)
C1—N1—C8—C3	−1.0 (3)	C6—C7—C8—N1	−179.4 (2)
C1—N1—C8—C7	178.4 (2)	C6—C7—C8—C3	0.0 (3)
C1—C2—C3—C4	−179.5 (2)	C8—N1—C1—O1	−177.7 (2)
C1—C2—C3—C8	−1.7 (2)	C8—N1—C1—C2	−0.2 (2)
C2—C3—C4—C5	177.0 (2)	C8—C3—C4—C5	−0.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86 (2)	1.98 (2)	2.832 (2)	167 (3)

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