IUCrData

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4,7-Dichloro-1*H*-indole-2,3-dione

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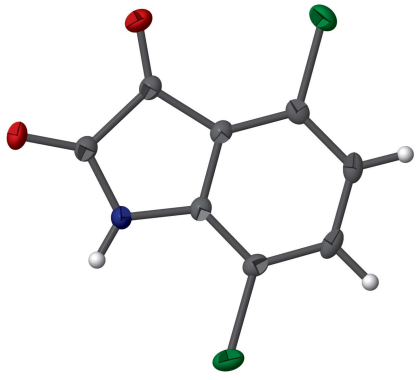
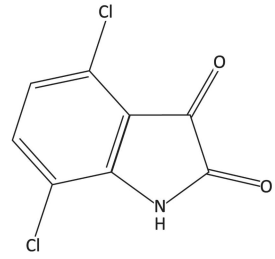
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_3Cl_2NO_2$, has a single near-planar molecule in the asymmetric unit, with the non-H atoms having a mean deviation from planarity of 0.042 Å. In the crystal, the molecules dimerize through two N—H···O hydrogen bonds. The molecules are further linked through slipped π–π interactions that propagate along the *a* axis [inter-centroid distance = 3.8639 (10) Å, interplanar distance = 3.3478 (10) Å and slippage = 1.9292 (15) Å].

3D view



Chemical scheme



Structure description

Herein we report the crystal structure of 4,7-dichloroisatin (Fig. 1). There is a single molecule in the asymmetric unit that has a mean deviation from planarity of only 0.042 Å for the non-H atoms. The bond lengths and angles of the 1*H*-indole-2,3-dione core are similar to those observed in the parent isatin (Goldschmidt & Llewellyn, 1950).

In the crystal, the molecules dimerize through N1—H1···O1 hydrogen bonds (Table 1). The molecular packing of the title compound (Fig. 2) also demonstrates parallel slipped π–π interactions that propagate along the *a* axis [inter-centroid distance = 3.8639 (10) Å, interplanar distance = 3.3478 (10) Å and slippage = 1.9292 (15) Å]. The 4,6-dichloro isomer of this compound does not demonstrate any π–π interactions (Mastrolia *et al.*, 2016). The molecules are further linked through C6—H6···O2 interactions, which are also observed in the monosubstituted 7-chloroisatin (Sun & Cai, 2010). There are C—H···Cl interactions present in the structure of 4-chloroisatin (Juma *et al.*, 2016), though no intermolecular halogen interactions are observed in the title compound.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86 (1)	2.04 (1)	2.8788 (19)	167 (2)
C6—H6···O2^{ii}	0.95	2.43	3.285 (2)	150

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

Synthesis and crystallization

A commercial sample (Matrix Scientific) of 4,7-dichloro-1*H*-indole-2,3-dione was used for the crystallization. Orange block-shaped crystals were grown by slow evaporation from an acetone solution.

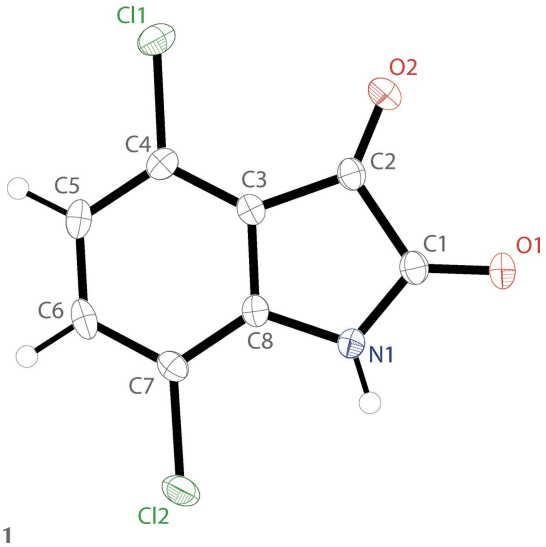


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

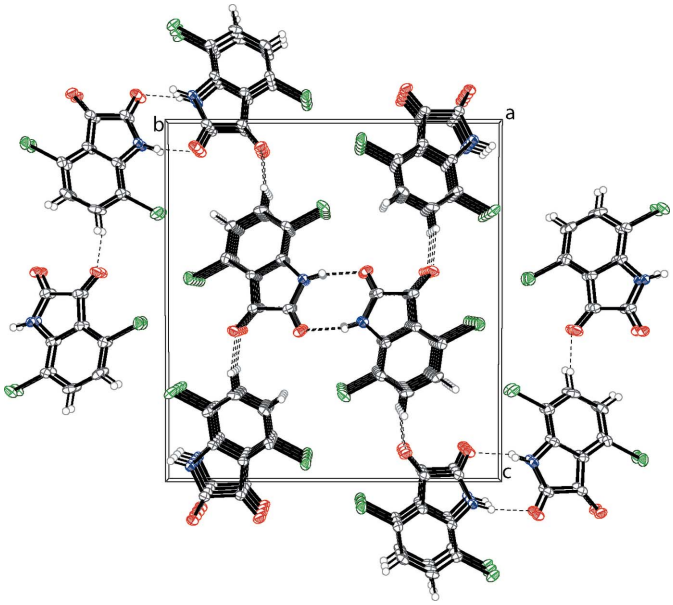


Figure 2
The molecular packing of the title compound along the *a* axis, with hydrogen bonds shown as dashed lines.

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)	3.8639 (10), 13.933 (4), 15.019 (4)
β (°)	93.313 (9)
V (Å^3)	807.2 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.76
Crystal size (mm)	0.22 × 0.2 × 0.1
Data collection	
Diffractometer	Bruker D8 Venture CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.702, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	29667, 1531, 1415
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.610
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.025, 0.061, 1.08
No. of reflections	1531
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})	0.30, −0.24

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**, x161485 [doi:10.1107/S2414314616014851]

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Crystal data

$C_8H_3Cl_2NO_2$
M_r = 216.01
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 3.8639 (10) Å
b = 13.933 (4) Å
c = 15.019 (4) Å
β = 93.313 (9)°
V = 807.2 (4) Å^3
Z = 4
$F(000)$ = 432
D_x = 1.778 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9837 reflections
θ = 3.1–25.7°
μ = 0.76 mm^{-1}
T = 120 K
Block, orange
0.22 × 0.2 × 0.1 mm

Data collection

Bruker D8 Venture CMOS diffractometer
Radiation source: Mo
TRIUMPH monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.702, T_{max} = 0.745
29667 measured reflections
1531 independent reflections
1415 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.7°, θ_{min} = 2.9°
h = −4→4
k = −16→16
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.061
S = 1.08
1531 reflections
121 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0214P)^2 + 0.6864P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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	0.18965 (11)	0.91841 (3)	0.43830 (3)	0.02617 (13)
Cl2	0.46709 (11)	0.52990 (3)	0.24627 (3)	0.02719 (13)
O1	0.9837 (3)	0.59718 (9)	0.58168 (8)	0.0254 (3)
O2	0.6456 (3)	0.78659 (9)	0.58085 (8)	0.0256 (3)
N1	0.7009 (3)	0.58766 (10)	0.44157 (9)	0.0177 (3)
H1	0.778 (5)	0.5327 (7)	0.4261 (12)	0.021*
C1	0.7951 (4)	0.62977 (12)	0.52103 (10)	0.0188 (3)
C2	0.6211 (4)	0.73103 (11)	0.51955 (10)	0.0174 (3)
C3	0.4447 (4)	0.73806 (11)	0.43009 (10)	0.0155 (3)
C4	0.2625 (4)	0.81121 (11)	0.38519 (11)	0.0178 (3)
C5	0.1416 (4)	0.79748 (12)	0.29710 (11)	0.0206 (3)
H5	0.0190	0.8473	0.2658	0.025*
C6	0.2003 (4)	0.71077 (13)	0.25501 (10)	0.0209 (3)
H6	0.1148	0.7017	0.1950	0.025*
C7	0.3826 (4)	0.63658 (11)	0.29914 (10)	0.0176 (3)
C8	0.5044 (4)	0.65116 (11)	0.38626 (10)	0.0152 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0286 (2)	0.0197 (2)	0.0304 (2)	0.00536 (16)	0.00339 (17)	−0.00055 (16)
Cl2	0.0279 (2)	0.0292 (2)	0.0241 (2)	0.00029 (17)	−0.00165 (16)	−0.01124 (17)
O1	0.0297 (6)	0.0260 (6)	0.0195 (6)	0.0056 (5)	−0.0077 (5)	0.0022 (5)
O2	0.0295 (7)	0.0275 (6)	0.0193 (6)	0.0017 (5)	−0.0033 (5)	−0.0058 (5)
N1	0.0194 (7)	0.0164 (7)	0.0169 (6)	0.0016 (5)	−0.0015 (5)	0.0011 (5)
C1	0.0176 (8)	0.0213 (8)	0.0173 (8)	−0.0017 (6)	0.0004 (6)	0.0019 (6)
C2	0.0155 (7)	0.0207 (8)	0.0160 (8)	−0.0017 (6)	0.0001 (6)	0.0011 (6)
C3	0.0133 (7)	0.0188 (8)	0.0145 (7)	−0.0032 (6)	0.0015 (6)	0.0010 (6)
C4	0.0144 (7)	0.0178 (8)	0.0216 (8)	−0.0001 (6)	0.0038 (6)	0.0019 (6)
C5	0.0150 (7)	0.0264 (9)	0.0201 (8)	0.0013 (6)	−0.0008 (6)	0.0077 (7)
C6	0.0153 (7)	0.0338 (9)	0.0133 (7)	−0.0030 (7)	−0.0015 (6)	0.0034 (7)
C7	0.0147 (7)	0.0216 (8)	0.0167 (8)	−0.0032 (6)	0.0015 (6)	−0.0025 (6)
C8	0.0118 (7)	0.0181 (8)	0.0157 (7)	−0.0027 (6)	0.0008 (6)	0.0021 (6)

Geometric parameters (Å, º)

Cl1—C4	1.7241 (17)	C3—C4	1.391 (2)
Cl2—C7	1.7253 (16)	C3—C8	1.403 (2)
O1—C1	1.2207 (19)	C4—C5	1.391 (2)
O2—C2	1.2024 (19)	C5—H5	0.9500
N1—H1	0.859 (5)	C5—C6	1.388 (2)
N1—C1	1.360 (2)	C6—H6	0.9500
N1—C8	1.406 (2)	C6—C7	1.396 (2)
C1—C2	1.562 (2)	C7—C8	1.380 (2)
C2—C3	1.474 (2)		

C1—N1—H1	122.8 (13)	C5—C4—C3	119.56 (15)
C1—N1—C8	110.78 (13)	C4—C5—H5	120.1
C8—N1—H1	125.8 (13)	C6—C5—C4	119.88 (15)
O1—C1—N1	127.74 (15)	C6—C5—H5	120.1
O1—C1—C2	125.75 (14)	C5—C6—H6	119.4
N1—C1—C2	106.50 (13)	C5—C6—C7	121.28 (15)
O2—C2—C1	123.77 (14)	C7—C6—H6	119.4
O2—C2—C3	131.82 (15)	C6—C7—Cl2	121.53 (12)
C3—C2—C1	104.41 (13)	C8—C7—Cl2	120.01 (13)
C4—C3—C2	133.23 (15)	C8—C7—C6	118.44 (15)
C4—C3—C8	119.81 (14)	C3—C8—N1	111.32 (13)
C8—C3—C2	106.87 (13)	C7—C8—N1	127.64 (14)
C3—C4—Cl1	120.14 (12)	C7—C8—C3	121.03 (14)
C5—C4—Cl1	120.30 (12)		

Cl1—C4—C5—C6	−179.73 (12)	C2—C3—C8—N1	1.18 (17)
Cl2—C7—C8—N1	0.3 (2)	C2—C3—C8—C7	−177.54 (14)
Cl2—C7—C8—C3	178.74 (11)	C3—C4—C5—C6	0.6 (2)
O1—C1—C2—O2	−3.1 (3)	C4—C3—C8—N1	178.20 (13)
O1—C1—C2—C3	175.86 (15)	C4—C3—C8—C7	−0.5 (2)
O2—C2—C3—C4	3.3 (3)	C4—C5—C6—C7	−0.6 (2)
O2—C2—C3—C8	179.75 (17)	C5—C6—C7—Cl2	−178.14 (12)
N1—C1—C2—O2	178.29 (15)	C5—C6—C7—C8	0.0 (2)
N1—C1—C2—C3	−2.77 (16)	C6—C7—C8—N1	−177.96 (14)
C1—N1—C8—C3	−3.17 (17)	C6—C7—C8—C3	0.5 (2)
C1—N1—C8—C7	175.44 (15)	C8—N1—C1—O1	−175.00 (16)
C1—C2—C3—C4	−175.52 (16)	C8—N1—C1—C2	3.59 (16)
C1—C2—C3—C8	0.94 (16)	C8—C3—C4—Cl1	−179.71 (11)
C2—C3—C4—Cl1	−3.6 (2)	C8—C3—C4—C5	−0.1 (2)
C2—C3—C4—C5	176.01 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86 (1)	2.04 (1)	2.8788 (19)	167 (2)
C6—H6···O2^{ii}	0.95	2.43	3.285 (2)	150

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