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4,6-Dichloro-1*H*-indole-2,3-dione

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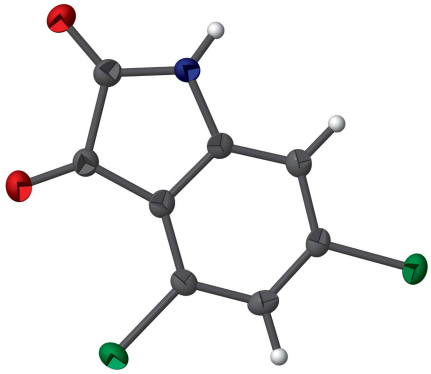
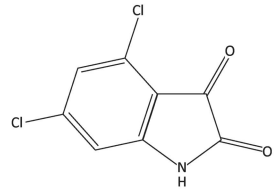
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_3Cl_2NO_2$, has a single, almost planar, molecule in the asymmetric unit, with the non-H atoms having a mean deviation from planarity of 0.027 Å. In the crystal, N—H···O hydrogen bonds form infinite *C*(4) chains along [100]. No π–π interactions were observed in the structure.

3D view



Chemical scheme



Structure description

Herein we report the crystal structure of 4,6-dichloroisatin (Fig. 1), which has a near planar molecule in the asymmetric unit, with non-H atoms possessing a mean deviation from planarity of 0.027 Å. The distances and angles are consistent with those reported for 1*H*-indole-2,3-dione (Goldschmidt & Llewellyn, 1950).

In the crystal, the molecules are linked through N1—H1···O1 hydrogen bonds (Table 1) to form infinite chains along [100] (Fig. 2). No other intermolecular hydrogen bonding or π–π interactions are observed. In addition to N—H···O hydrogen bonding, the monosubstituted 4-chloroisatin possesses C—H···Cl close contacts (Juma *et al.*, 2016) and
6-chloroisatin possesses C—H···O interactions (Golen & Manke, 2016), neither of which are observed in the title compound.

Synthesis and crystallization

A commercial sample (Matrix Scientific) of 4,6-dichloro-1*H*-indole-2,3-dione was used for the crystallization. Orange blocks were grown from the slow evaporation of an acetone solution.

Refinement

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

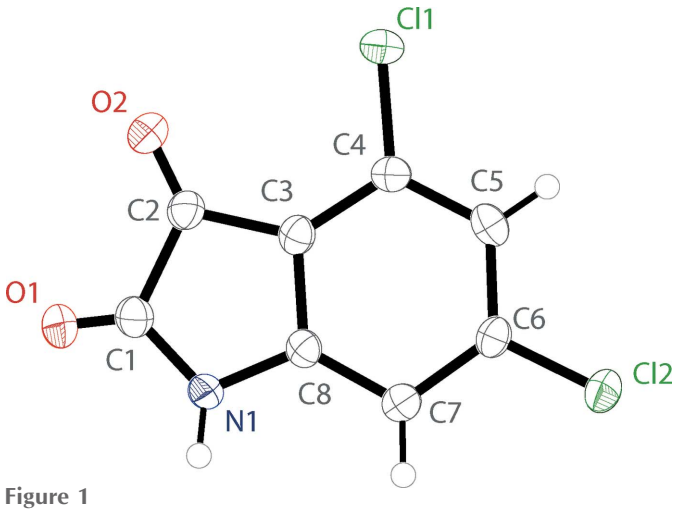


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius

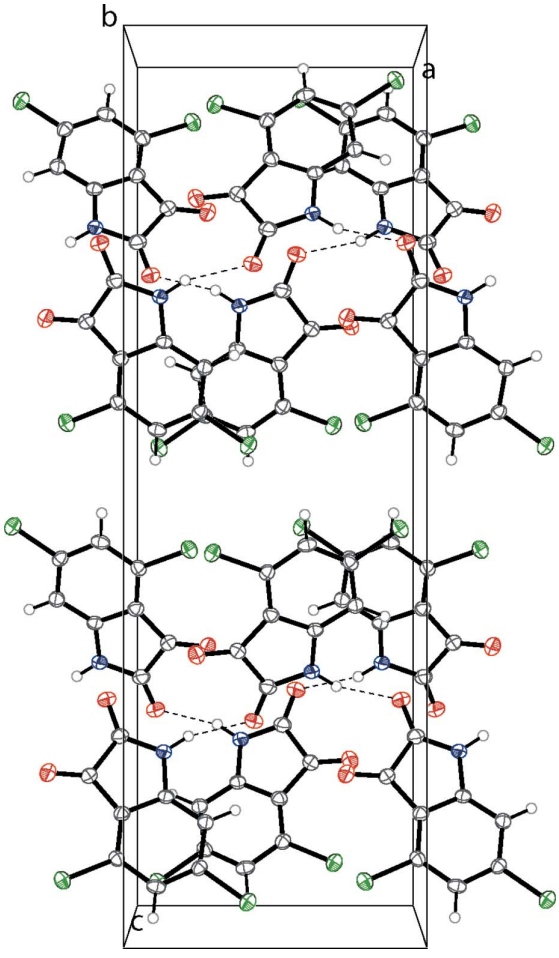


Figure 2
Molecular packing of the title compound along the *b* axis, with hydrogen bonding shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.87 (2)	1.98 (2)	2.809 (3)	161 (3)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_3Cl_2NO_2$
M_r	216.01
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	120
a, b, c (Å)	8.6253 (19), 7.1250 (16), 26.289 (6)
V (Å^3)	1615.6 (6)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	6.92
Crystal size (mm)	0.25 × 0.2 × 0.1
Data collection	
Diffractometer	Bruker Venture D8 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.190, 0.386
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13414, 1604, 1531
R_{int}	0.057
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.619
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.098, 1.10
No. of reflections	1604
No. of parameters	122
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})	0.34, −0.26

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160695 [doi:10.1107/S2414314616006957]

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Crystal data

$C_8H_3Cl_2NO_2$
M_r = 216.01
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 8.6253 (19) Å
b = 7.1250 (16) Å
c = 26.289 (6) Å
V = 1615.6 (6) Å^3
Z = 8
F(000) = 864
D_x = 1.776 Mg m^{-3}
Cu *Kα* radiation, λ = 1.54178 Å
Cell parameters from 8528 reflections
θ = 3.4–72.4°
μ = 6.92 mm^{-1}
T = 120 K
Block, orange
0.25 × 0.2 × 0.1 mm

Data collection

Bruker Venture D8 CMOS diffractometer
Radiation source: Cu
HELIOS MX monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.190, T_{max} = 0.386
13414 measured reflections
1604 independent reflections
1531 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 72.7°, θ_{min} = 3.4°
h = −10→10
k = −8→8
l = −32→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.098
S = 1.10
1604 reflections
122 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 2.2094P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0017 (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	0.29514 (7)	0.91432 (9)	0.57109 (2)	0.0280 (2)
Cl2	0.90946 (7)	0.85524 (8)	0.54527 (2)	0.0266 (2)
O1	0.4268 (2)	0.5417 (3)	0.76169 (6)	0.0307 (4)
O2	0.2373 (2)	0.7287 (3)	0.68422 (6)	0.0340 (5)
N1	0.6227 (2)	0.6103 (3)	0.70490 (7)	0.0233 (4)
H1	0.706 (2)	0.586 (4)	0.7219 (10)	0.028*
C8	0.6327 (3)	0.6988 (3)	0.65726 (8)	0.0199 (5)
C4	0.4730 (3)	0.8391 (3)	0.59277 (9)	0.0211 (5)
C3	0.4861 (3)	0.7546 (3)	0.64011 (8)	0.0216 (5)
C7	0.7662 (3)	0.7288 (3)	0.62975 (8)	0.0219 (5)
H7	0.8654	0.6946	0.6424	0.026*
C6	0.7471 (3)	0.8124 (3)	0.58219 (8)	0.0208 (5)
C5	0.6037 (3)	0.8662 (3)	0.56284 (9)	0.0225 (5)
H5	0.5954	0.9202	0.5299	0.027*
C2	0.3748 (3)	0.7047 (4)	0.68032 (9)	0.0240 (5)
C1	0.4750 (3)	0.6071 (4)	0.72214 (9)	0.0255 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0227 (3)	0.0336 (4)	0.0277 (3)	0.0046 (2)	−0.0042 (2)	0.0030 (2)
Cl2	0.0233 (3)	0.0291 (3)	0.0273 (3)	−0.0025 (2)	0.0057 (2)	0.0015 (2)
O1	0.0293 (9)	0.0422 (11)	0.0205 (8)	0.0017 (8)	0.0032 (7)	0.0037 (8)
O2	0.0229 (9)	0.0498 (12)	0.0291 (9)	0.0048 (8)	0.0039 (7)	0.0043 (8)
N1	0.0212 (10)	0.0314 (11)	0.0172 (9)	0.0039 (8)	−0.0006 (7)	0.0013 (8)
C8	0.0226 (11)	0.0206 (11)	0.0165 (10)	−0.0002 (9)	−0.0002 (8)	−0.0037 (8)
C4	0.0211 (11)	0.0206 (11)	0.0215 (11)	0.0019 (9)	−0.0024 (9)	−0.0029 (9)
C3	0.0223 (11)	0.0226 (11)	0.0198 (10)	−0.0017 (9)	0.0013 (9)	−0.0030 (9)
C7	0.0210 (11)	0.0227 (11)	0.0220 (10)	−0.0005 (9)	−0.0005 (9)	−0.0041 (9)
C6	0.0217 (11)	0.0198 (11)	0.0210 (10)	−0.0020 (9)	0.0036 (9)	−0.0034 (8)
C5	0.0307 (13)	0.0187 (11)	0.0181 (10)	−0.0037 (10)	−0.0028 (9)	−0.0006 (8)
C2	0.0225 (12)	0.0286 (12)	0.0208 (11)	0.0019 (10)	0.0014 (9)	−0.0022 (9)
C1	0.0273 (12)	0.0296 (13)	0.0195 (11)	0.0030 (10)	0.0010 (9)	−0.0023 (9)

Geometric parameters (Å, º)

Cl1—C4	1.722 (2)	C4—C3	1.387 (3)
Cl2—C6	1.731 (2)	C4—C5	1.388 (3)
O1—C1	1.213 (3)	C3—C2	1.471 (3)
O2—C2	1.202 (3)	C7—H7	0.9500
N1—H1	0.865 (17)	C7—C6	1.395 (3)
N1—C8	1.405 (3)	C6—C5	1.391 (3)
N1—C1	1.352 (3)	C5—H5	0.9500
C8—C3	1.400 (3)	C2—C1	1.562 (3)
C8—C7	1.377 (3)		

C8—N1—H1	120 (2)	C6—C7—H7	122.0
C1—N1—H1	127.4 (19)	C7—C6—Cl2	118.82 (19)
C1—N1—C8	111.3 (2)	C5—C6—Cl2	117.76 (17)
C3—C8—N1	111.1 (2)	C5—C6—C7	123.4 (2)
C7—C8—N1	126.1 (2)	C4—C5—C6	118.4 (2)
C7—C8—C3	122.8 (2)	C4—C5—H5	120.8
C3—C4—Cl1	120.31 (18)	C6—C5—H5	120.8
C3—C4—C5	120.2 (2)	O2—C2—C3	132.1 (2)
C5—C4—Cl1	119.49 (18)	O2—C2—C1	123.3 (2)
C8—C3—C2	106.8 (2)	C3—C2—C1	104.6 (2)
C4—C3—C8	119.1 (2)	O1—C1—N1	128.1 (2)
C4—C3—C2	134.1 (2)	O1—C1—C2	125.8 (2)
C8—C7—H7	122.0	N1—C1—C2	106.14 (19)
C8—C7—C6	116.0 (2)		

Cl1—C4—C3—C8	−178.52 (17)	C8—C7—C6—C5	0.8 (3)
Cl1—C4—C3—C2	1.4 (4)	C4—C3—C2—O2	−2.5 (5)
Cl1—C4—C5—C6	176.95 (17)	C4—C3—C2—C1	178.3 (3)
Cl2—C6—C5—C4	−177.68 (17)	C3—C8—C7—C6	−2.5 (3)
O2—C2—C1—O1	1.9 (4)	C3—C4—C5—C6	−2.1 (3)
O2—C2—C1—N1	−178.5 (3)	C3—C2—C1—O1	−178.7 (2)
N1—C8—C3—C4	−177.9 (2)	C3—C2—C1—N1	0.8 (3)
N1—C8—C3—C2	2.2 (3)	C7—C8—C3—C4	1.9 (4)
N1—C8—C7—C6	177.3 (2)	C7—C8—C3—C2	−178.0 (2)
C8—N1—C1—O1	−180.0 (2)	C7—C6—C5—C4	1.5 (4)
C8—N1—C1—C2	0.5 (3)	C5—C4—C3—C8	0.5 (3)
C8—C3—C2—O2	177.5 (3)	C5—C4—C3—C2	−179.6 (2)
C8—C3—C2—C1	−1.8 (3)	C1—N1—C8—C3	−1.7 (3)
C8—C7—C6—Cl2	179.91 (17)	C1—N1—C8—C7	178.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.87 (2)	1.98 (2)	2.809 (3)	161 (3)

Symmetry code: (i) $x+1/2, y, -z+3/2$.