



IUCrData

ISSN 2414-3146

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

James A. Golen and David R. Manke*

Department of Chemistry and Biochemistry, University of Massachusetts Dartmouth, 285 Old Westport Road, North Dartmouth, MA 02747, USA. *Correspondence e-mail: dmanke@umassd.edu

Received 19 April 2016

Accepted 23 April 2016

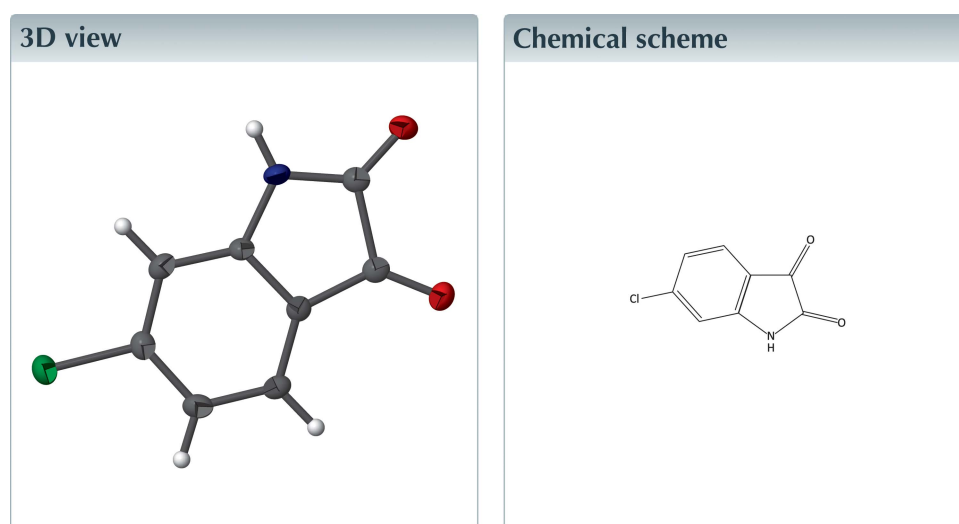
Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

Keywords: crystal structure; isatins; 6-chloroisatin; hydrogen bonding.

CCDC reference: 1476175

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

The molecule of the title compound, C₈H₄ClNO₂, is planar, with the non-H atoms possessing an r.m.s. deviation from planarity of 0.062 Å. In the crystal, molecules are linked through N—H···O hydrogen bonds, forming chains along [010]. The chains are further linked through C—H···O hydrogen bonds, forming layers parallel to (001).



Structure description

Herein, we report on the crystal structure of 6-chloroisatin (Fig. 1). The molecule is almost planar with the non-H atoms possessing an r.m.s. deviation from planarity of 0.062 Å. The bond distances and angles are similar to those reported for 1*H*-indole-2,3-dione (Goldschmidt & Llewellyn, 1950).

In the crystal, molecules are linked together through N1—H1···O1 hydrogen bonds, forming chains along [010]. The chains are connected through C7—H7···O2 hydrogen bonds, forming layers parallel to the *ab* plane (Table 1 and Fig. 2).

The two reported structures of 6-chloroisatin derivatives also demonstrate C—H···O interactions from the carbon on the 7 position of the isatin ring with the oxygen on the 2 position of an isatin ring (Liu *et al.*, 2011, 2012). In contrast, the other reported 6-haloisatins possess intermolecular interactions through their halogen atoms, with 6-fluoroisatin possessing C—H···F close contacts (Golen & Manke, 2016) and 6-bromoisatin possessing Br···O interactions (Turbitt *et al.*, 2016). Both 4-chloroisatin (Juma *et al.*, 2016) and 7-chloroisatin (Sun & Cai, 2010) demonstrate C—H···Cl interactions which are not present in the crystal of the title isomer.

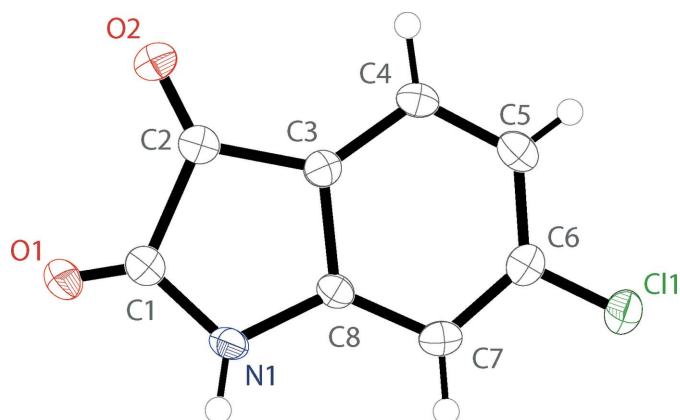


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Synthesis and crystallization

A commercial sample (Matrix Scientific) of 6-chloro-1H-indole-2,3-dione was used for crystallization. A sample suitable for single-crystal X-ray diffraction analysis was grown by slow evaporation from a dimethylsulfoxide solution.

Refinement

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

Acknowledgements

We gratefully acknowledge support from the National Science Foundation (CHE-1429086).

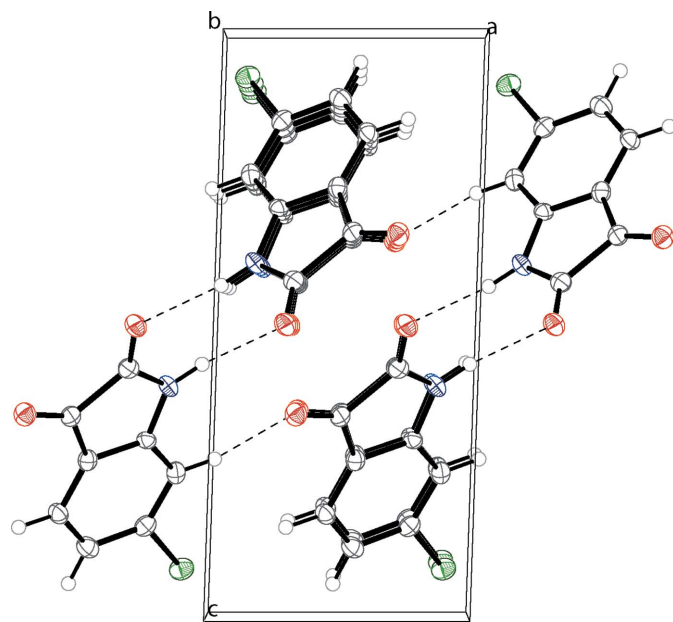


Figure 2
The molecular packing of the title compound viewed along the *b* axis, with the hydrogen bonds shown as dashed lines (see Table 1).

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1···O1 ⁱ	0.86 (1)	2.03 (2)	2.885 (4)	172 (4)
C7—H7···O2 ⁱⁱ	0.95	2.32	3.260 (4)	170

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

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₈ H ₄ ClNO ₂
<i>M_r</i>	181.57
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁
Temperature (K)	120
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.6231 (6), 4.9930 (6), 12.5145 (14)
β (°)	91.916 (7)
<i>V</i> (Å ³)	351.16 (7)
<i>Z</i>	2
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	4.41
Crystal size (mm)	0.22 × 0.06 × 0.04
Data collection	
Diffractometer	Bruker Venture D8 CMOS
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2014)
<i>T</i> _{min} , <i>T</i> _{max}	0.349, 0.467
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	4219, 1262, 1183
<i>R</i> _{int}	0.052
(sin θ / λ) _{max} (Å ⁻¹)	0.603
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.031, 0.070, 1.12
No. of reflections	1262
No. of parameters	114
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.22, -0.20
Absolute structure	Refined as an inversion twin.
Absolute structure parameter	0.09 (3)

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

References

- Bruker (2014). *APEX2*, *SAINT*, and *SADABS*. Bruker AXS Inc. Madison, Wisconsin, USA.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Goldschmidt, G. H. & Llewellyn, F. J. (1950). *Acta Cryst.* **3**, 294–305.
- Golen, J. A. & Manke, D. R. (2016). *IUCrData*, **1**, x160165.
- Juma, R. M., Golen, J. A. & Manke, D. R. (2016). *IUCrData*, **1**, x160689.
- Liu, H., Fan, D., Wang, D. & Ou-yang, P.-K. (2011). *Acta Cryst.* **E67**, o3427.
- Liu, H. Q., Tang, W., Wang, D. C. & Ou-yang, P. K. (2012). *Acta Cryst.* **E68**, o14.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Sun, J. & Cai, Z.-S. (2010). *Acta Cryst.* **E66**, o25.
- Turbitt, J. R., Golen, J. A. & Manke, D. R. (2016). *IUCrData*, **1**, x152434.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2016). **1**, x160690 [doi:10.1107/S2414314616006908]

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

James A. Golen and David R. Manke

6-Chloro-1*H*-indole-2,3-dione*Crystal data*

$C_8H_4ClNO_2$

$M_r = 181.57$

Monoclinic, $P2_1$

$a = 5.6231$ (6) Å

$b = 4.9930$ (6) Å

$c = 12.5145$ (14) Å

$\beta = 91.916$ (7)°

$V = 351.16$ (7) Å³

$Z = 2$

$F(000) = 184$

$D_x = 1.717$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 3414 reflections

$\theta = 3.5$ – 68.5 °

$\mu = 4.41$ mm⁻¹

$T = 120$ K

Block, orange

$0.22 \times 0.06 \times 0.04$ mm

Data collection

Bruker Venture D8 CMOS
diffractometer

Radiation source: Cu

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2014)

$T_{\min} = 0.349$, $T_{\max} = 0.467$

4219 measured reflections

1262 independent reflections

1183 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.052$

$\theta_{\max} = 68.5$ °, $\theta_{\min} = 3.5$ °

$h = -6 \rightarrow 6$

$k = -6 \rightarrow 6$

$l = -15 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.070$

$S = 1.12$

1262 reflections

114 parameters

2 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0176P)^2 + 0.0693P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.22$ e Å⁻³

$\Delta\rho_{\min} = -0.20$ e Å⁻³

Absolute structure: Refined as an inversion
twin.

Absolute structure parameter: 0.09 (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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.09834 (14)	−0.01835 (18)	0.08160 (7)	0.0243 (2)
O1	0.2777 (4)	1.0175 (6)	0.49908 (19)	0.0232 (6)
O2	0.6762 (4)	1.0073 (6)	0.35185 (18)	0.0235 (5)
N1	0.1575 (5)	0.6515 (6)	0.3975 (2)	0.0202 (7)
H1	0.035 (5)	0.614 (9)	0.434 (3)	0.035 (13)*
C1	0.3027 (6)	0.8549 (8)	0.4278 (3)	0.0192 (7)
C2	0.5114 (6)	0.8531 (8)	0.3481 (3)	0.0190 (7)
C3	0.4503 (6)	0.6351 (7)	0.2732 (3)	0.0185 (7)
C4	0.5569 (6)	0.5441 (7)	0.1822 (3)	0.0206 (8)
H4	0.7019	0.6208	0.1602	0.025*
C5	0.4494 (6)	0.3391 (8)	0.1233 (3)	0.0229 (8)
H5	0.5195	0.2729	0.0605	0.027*
C6	0.2363 (6)	0.2324 (7)	0.1582 (3)	0.0198 (8)
C7	0.1259 (6)	0.3158 (7)	0.2494 (3)	0.0196 (8)
H7	−0.0174	0.2362	0.2721	0.024*
C8	0.2363 (5)	0.5226 (8)	0.3059 (3)	0.0169 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0281 (4)	0.0218 (4)	0.0229 (4)	−0.0025 (4)	−0.0018 (3)	−0.0020 (5)
O1	0.0223 (11)	0.0237 (15)	0.0238 (13)	0.0034 (12)	0.0045 (9)	−0.0029 (13)
O2	0.0222 (11)	0.0225 (13)	0.0257 (13)	−0.0039 (13)	0.0009 (9)	0.0017 (14)
N1	0.0180 (15)	0.0224 (16)	0.0204 (17)	−0.0005 (12)	0.0066 (12)	0.0002 (13)
C1	0.0207 (17)	0.0178 (16)	0.019 (2)	0.0042 (15)	0.0005 (14)	0.0035 (17)
C2	0.0189 (16)	0.0183 (16)	0.0198 (19)	0.0035 (15)	−0.0002 (13)	0.0044 (16)
C3	0.0171 (16)	0.0171 (18)	0.0213 (19)	0.0015 (13)	0.0001 (13)	0.0012 (14)
C4	0.0189 (15)	0.023 (2)	0.0204 (18)	0.0001 (13)	0.0034 (12)	0.0039 (14)
C5	0.0238 (18)	0.0235 (19)	0.022 (2)	0.0046 (15)	0.0050 (14)	0.0011 (17)
C6	0.0217 (17)	0.0165 (17)	0.0209 (19)	0.0015 (13)	−0.0035 (14)	0.0025 (14)
C7	0.0165 (16)	0.019 (2)	0.024 (2)	0.0008 (13)	0.0010 (13)	0.0058 (15)
C8	0.0181 (15)	0.018 (2)	0.0141 (15)	0.0013 (15)	0.0014 (11)	0.0031 (15)

Geometric parameters (\AA , $^\circ$)

Cl1—C6	1.743 (4)	C3—C8	1.401 (5)
O1—C1	1.218 (4)	C4—H4	0.9500
O2—C2	1.204 (4)	C4—C5	1.388 (5)
N1—H1	0.859 (14)	C5—H5	0.9500
N1—C1	1.350 (5)	C5—C6	1.395 (5)
N1—C8	1.400 (5)	C6—C7	1.382 (5)
C1—C2	1.565 (5)	C7—H7	0.9500
C2—C3	1.470 (5)	C7—C8	1.386 (5)
C3—C4	1.382 (5)		

C1—N1—H1	120 (3)	C5—C4—H4	120.4
C1—N1—C8	111.8 (3)	C4—C5—H5	120.7
C8—N1—H1	128 (3)	C4—C5—C6	118.6 (3)
O1—C1—N1	128.6 (3)	C6—C5—H5	120.7
O1—C1—C2	125.3 (3)	C5—C6—C11	118.3 (3)
N1—C1—C2	106.1 (3)	C7—C6—C11	117.8 (3)
O2—C2—C1	124.4 (3)	C7—C6—C5	123.9 (3)
O2—C2—C3	131.4 (3)	C6—C7—H7	122.0
C3—C2—C1	104.3 (3)	C6—C7—C8	116.1 (3)
C4—C3—C2	132.1 (3)	C8—C7—H7	122.0
C4—C3—C8	120.6 (3)	N1—C8—C3	110.7 (3)
C8—C3—C2	107.2 (3)	C7—C8—N1	127.7 (3)
C3—C4—H4	120.4	C7—C8—C3	121.6 (3)
C3—C4—C5	119.2 (3)		
C11—C6—C7—C8	-177.6 (3)	C2—C3—C8—C7	177.9 (3)
O1—C1—C2—O2	3.3 (6)	C3—C4—C5—C6	0.2 (5)
O1—C1—C2—C3	-175.3 (3)	C4—C3—C8—N1	-177.9 (3)
O2—C2—C3—C4	-2.5 (7)	C4—C3—C8—C7	0.7 (5)
O2—C2—C3—C8	-179.3 (4)	C4—C5—C6—C11	178.2 (3)
N1—C1—C2—O2	-179.4 (3)	C4—C5—C6—C7	-1.0 (5)
N1—C1—C2—C3	2.0 (4)	C5—C6—C7—C8	1.6 (5)
C1—N1—C8—C3	2.2 (4)	C6—C7—C8—N1	177.0 (3)
C1—N1—C8—C7	-176.4 (3)	C6—C7—C8—C3	-1.4 (5)
C1—C2—C3—C4	176.0 (3)	C8—N1—C1—O1	174.7 (4)
C1—C2—C3—C8	-0.7 (4)	C8—N1—C1—C2	-2.5 (4)
C2—C3—C4—C5	-176.4 (4)	C8—C3—C4—C5	0.0 (5)
C2—C3—C8—N1	-0.7 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O1 ⁱ	0.86 (1)	2.03 (2)	2.885 (4)	172 (4)
C7—H7 \cdots O2 ⁱⁱ	0.95	2.32	3.260 (4)	170

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