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

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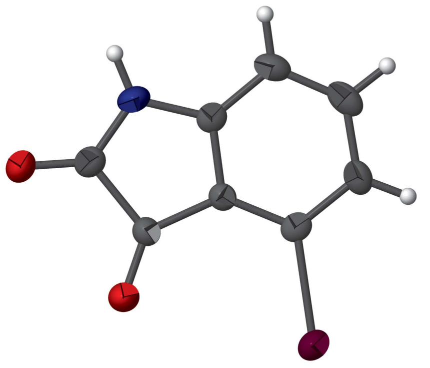
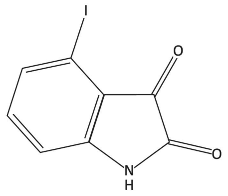
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_4INO_2$, is an almost planar molecule having an r.m.s. deviation of 0.03 Å for all non-H atoms. In the crystal, molecules dimerize through pairs of N—H···O hydrogen bonds. These inversion dimers are linked through pairs of weak I···O interactions [3.184 (4) Å] to form zigzag chains along [010]. The chains are linked in the *c*-axis direction by parallel-slipped π–π interactions [intercentroid distance = 3.623 (3) Å, interplanar distance = 3.423 (2) Å and slippage = 1.667 (5) Å], forming corrugated sheets parallel to (011).

3D view



Chemical scheme



Structure description

Isatin molecules have found widespread use in organic synthesis and in pharmaceutical applications. We have an ongoing study of the solid state structure of halogenated isatins, and report herein on the crystal structure of 4-iodoisatin. The title compound, Fig. 1, exhibits a near planar molecule with the non-H atoms possessing a mean deviation from planarity of 0.03 Å. The observed bond lengths and angles are consistent with the parent isatin (Goldschmidt *et al.*, 1950).

In the crystal, pairs of N—H···O hydrogen bonds link the molecules to form inversion dimers (Table 1 and Fig. 2). The dimers are further linked into zigzag chains along [010] by pairs of weak I···O interactions [$I1\cdots O2^{i}$ = 3.184 (4) Å; symmetry code (i): $-x + 2, -y + 1, -z + 1$]. Similar I···O interactions have been observed in 5-iodoisatin and its derivatives (Abid *et al.*, 2008; Garden *et al.*, 2006; Wang *et al.*, 2014). The closely related 4-bromoisatin also exhibits a weak halogen–oxygen interaction in the solid state (Huang *et al.*, 2016). The chains stack along [001] and are linked *via* parallel slipped π–π interactions [$Cg2\cdots Cg2^{ii}$ = 3.699 (3) Å, where *Cg*2 is the centroid of ring C3–C8, inter-planar distance = 3.428 (2) Å, slippage = 1.383 Å, symmetry code (ii): $x, -y + \frac{3}{2}, z + \frac{1}{2}$], leading to the formation of undulating sheets lying parallel to (011).

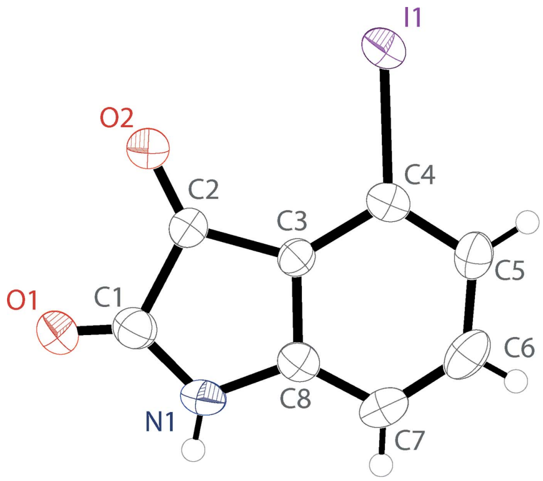


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

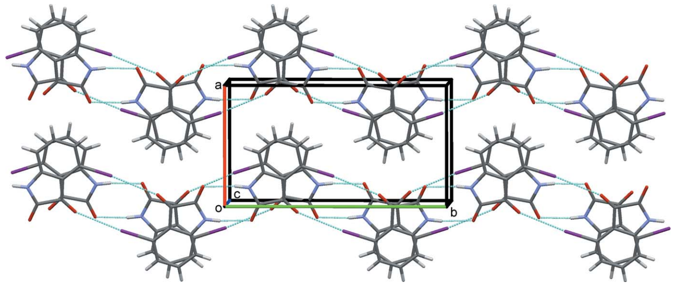


Figure 2
The crystal packing of the title compound, viewed along the *c*-axis direction. The N—H⋯O hydrogen bonds (see Table 1) and I⋯O interactions are shown as dashed lines.

Synthesis and crystallization

A commercial sample (Matrix Scientific) of 4-iodo-1*H*-indole-2,3-dione was used for crystallization. Orange rod-like crystals, suitable for X-ray diffraction analysis, were grown by slow evaporation of a solution in acetone.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The NH H atom was refined with $U_{iso}(H) = 1.2U_{eq}(N)$.

Acknowledgements

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

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)	2.05 (2)	2.910 (6)	173 (7)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_4INO_2$
M_r	273.02
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	298
a, *b*, *c* (Å)	7.6138 (9), 13.9658 (17), 7.3866 (10)
β (°)	91.249 (5)
V (Å^3)	785.25 (17)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	4.03
Crystal size (mm)	0.25 × 0.1 × 0.08
Data collection	
Diffractometer	Bruker D8 Venture CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.182, 0.259
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16058, 1491, 1268
R_{int}	0.050
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.611
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.082, 1.19
No. of reflections	1491
No. of parameters	112
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.95, −0.83

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

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full crystallographic data

IUCrData (2016). **1**, x160215 [https://doi.org/10.1107/S2414314616002157]

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Crystal data

$C_8H_4INO_2$
$M_r = 273.02$
Monoclinic, $P2_1/c$
$a = 7.6138\ (9)$ Å
$b = 13.9658\ (17)$ Å
$c = 7.3866\ (10)$ Å
$\beta = 91.249\ (5)°$
$V = 785.25\ (17)$ Å^3
$Z = 4$

$F(000) = 512$
$D_x = 2.309$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7683 reflections
$\theta = 2.9–25.7°$
$\mu = 4.03$ mm^{-1}
$T = 298$ K
Rod, orange
0.25 × 0.1 × 0.08 mm

Data collection

Bruker D8 Venture CMOS diffractometer
Radiation source: Mo
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
$T_{\min} = 0.182$, $T_{\max} = 0.259$
16058 measured reflections

1491 independent reflections
1268 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.050$
$\theta_{\max} = 25.7°$, $\theta_{\min} = 2.9°$
$h = -9 \rightarrow 9$
$k = -17 \rightarrow 17$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.082$
$S = 1.19$
1491 reflections
112 parameters
1 restraint

Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 3.7223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.95$ e Å^{-3}
$\Delta\rho_{\min} = -0.83$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
I1	0.76809 (5)	0.52256 (3)	0.58699 (6)	0.03698 (15)
O1	1.1415 (6)	0.8920 (3)	0.4389 (7)	0.0440 (11)
O2	1.0723 (6)	0.6869 (3)	0.4540 (7)	0.0438 (11)
N1	0.8656 (7)	0.8997 (3)	0.5598 (7)	0.0349 (11)
H1	0.854 (9)	0.9613 (16)	0.561 (9)	0.042*
C1	1.0079 (8)	0.8563 (4)	0.4961 (8)	0.0334 (13)
C2	0.9695 (7)	0.7460 (4)	0.5078 (7)	0.0284 (12)
C3	0.7944 (7)	0.7395 (4)	0.5841 (7)	0.0242 (11)
C4	0.6850 (7)	0.6629 (4)	0.6254 (7)	0.0265 (11)
C5	0.5197 (7)	0.6816 (4)	0.6915 (8)	0.0320 (13)
H5	0.4451	0.6315	0.7211	0.038*
C6	0.4664 (8)	0.7758 (5)	0.7130 (8)	0.0369 (14)
H6	0.3549	0.7873	0.7571	0.044*
C7	0.5710 (8)	0.8532 (4)	0.6722 (8)	0.0350 (13)
H7	0.5316	0.9157	0.6859	0.042*
C8	0.7365 (7)	0.8335 (4)	0.6102 (7)	0.0268 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0378 (2)	0.0255 (2)	0.0478 (3)	−0.00292 (17)	0.00400 (16)	0.00133 (19)
O1	0.039 (2)	0.029 (2)	0.064 (3)	−0.0036 (19)	0.013 (2)	0.003 (2)
O2	0.037 (2)	0.028 (2)	0.067 (3)	0.0010 (18)	0.015 (2)	0.000 (2)
N1	0.041 (3)	0.021 (2)	0.043 (3)	0.002 (2)	0.006 (2)	−0.001 (2)
C1	0.037 (3)	0.029 (3)	0.035 (3)	−0.001 (2)	0.003 (2)	0.001 (2)
C2	0.026 (3)	0.028 (3)	0.031 (3)	0.000 (2)	0.003 (2)	−0.001 (2)
C3	0.025 (3)	0.026 (3)	0.022 (3)	0.001 (2)	0.001 (2)	0.000 (2)
C4	0.034 (3)	0.026 (3)	0.019 (3)	−0.001 (2)	−0.003 (2)	0.000 (2)
C5	0.027 (3)	0.041 (3)	0.028 (3)	−0.003 (2)	0.003 (2)	−0.003 (3)
C6	0.032 (3)	0.053 (4)	0.026 (3)	0.008 (3)	0.004 (2)	−0.006 (3)
C7	0.035 (3)	0.035 (3)	0.034 (3)	0.010 (3)	0.001 (3)	−0.006 (3)
C8	0.032 (3)	0.028 (3)	0.021 (3)	−0.002 (2)	−0.002 (2)	−0.002 (2)

Geometric parameters (Å, º)

I1—C4	2.081 (5)	C3—C8	1.399 (8)
O1—C1	1.216 (7)	C4—C5	1.385 (8)
O2—C2	1.211 (7)	C5—H5	0.9300
N1—H1	0.87 (2)	C5—C6	1.386 (9)
N1—C1	1.336 (8)	C6—H6	0.9300
N1—C8	1.406 (7)	C6—C7	1.380 (9)
C1—C2	1.571 (8)	C7—H7	0.9300
C2—C3	1.462 (7)	C7—C8	1.378 (8)
C3—C4	1.393 (8)		

C1—N1—H1	123 (5)	C5—C4—C3	119.0 (5)
C1—N1—C8	111.8 (5)	C4—C5—H5	120.3
C8—N1—H1	125 (5)	C4—C5—C6	119.3 (5)
O1—C1—N1	128.8 (6)	C6—C5—H5	120.3
O1—C1—C2	125.4 (5)	C5—C6—H6	118.4
N1—C1—C2	105.8 (5)	C7—C6—C5	123.1 (6)
O2—C2—C1	121.9 (5)	C7—C6—H6	118.4
O2—C2—C3	133.3 (5)	C6—C7—H7	121.6
C3—C2—C1	104.8 (4)	C8—C7—C6	116.9 (5)
C4—C3—C2	133.4 (5)	C8—C7—H7	121.6
C4—C3—C8	119.9 (5)	C3—C8—N1	110.9 (5)
C8—C3—C2	106.7 (5)	C7—C8—N1	127.3 (5)
C3—C4—I1	120.5 (4)	C7—C8—C3	121.8 (5)
C5—C4—I1	120.5 (4)		

I1—C4—C5—C6	−179.0 (4)	C2—C3—C8—N1	−1.5 (6)
O1—C1—C2—O2	−2.2 (10)	C2—C3—C8—C7	176.6 (5)
O1—C1—C2—C3	−179.8 (6)	C3—C4—C5—C6	0.7 (8)
O2—C2—C3—C4	1.6 (11)	C4—C3—C8—N1	−179.7 (5)
O2—C2—C3—C8	−176.3 (7)	C4—C3—C8—C7	−1.6 (8)
N1—C1—C2—O2	177.5 (6)	C4—C5—C6—C7	−0.2 (9)
N1—C1—C2—C3	−0.1 (6)	C5—C6—C7—C8	−1.1 (9)
C1—N1—C8—C3	1.5 (7)	C6—C7—C8—N1	179.8 (6)
C1—N1—C8—C7	−176.5 (6)	C6—C7—C8—C3	2.0 (8)
C1—C2—C3—C4	178.8 (6)	C8—N1—C1—O1	178.9 (6)
C1—C2—C3—C8	1.0 (6)	C8—N1—C1—C2	−0.8 (7)
C2—C3—C4—I1	2.2 (8)	C8—C3—C4—I1	179.8 (4)
C2—C3—C4—C5	−177.4 (6)	C8—C3—C4—C5	0.2 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.87 (2)	2.05 (2)	2.910 (6)	173 (7)

Symmetry code: (i) −*x*+2, −*y*+2, −*z*+1.