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ISSN 2414-3146

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

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Received 4 March 2016
Accepted 10 March 2016

Edited by K. Fejfarova, Institute of Biotechnology CAS, Czech Republic

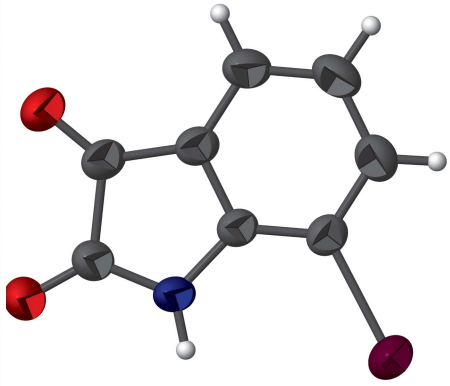
Keywords: crystal structure; hydrogen bonding; isatins; halogen–oxygen interactions.

CCDC reference: 1463777

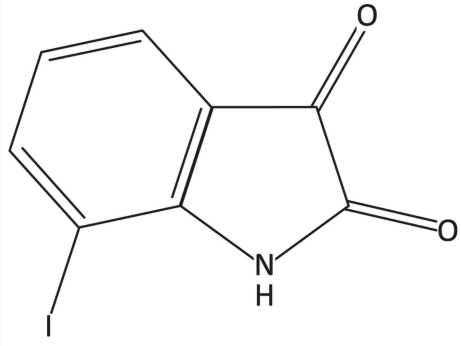
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_4INO_2$, has a single planar molecule in the asymmetric unit, with the non-H atoms possessing a mean deviation from planarity of 0.058 Å. The molecules dimerize in the solid state through N—H···O hydrogen bonds. There are intermolecular I···O close contacts of 3.193 (4) Å that link the molecules into infinite chains along [20$\bar{1}$]. No π–π interactions were observed in the structure.

3D view



Chemical scheme



Structure description

We report herein the crystal structure of 7-iodoisatin (Fig. 1). The molecule is nearly planar, with the non-H atoms possessing a mean deviation from planarity of 0.058 Å, and exhibits bond lengths and angles similar to those observed in isatin (Goldschmidt & Llewellyn, 1950). In the crystal, molecules dimerize through N1—H1···O1^{i} hydrogen bonds (see Table 1 for symmetry code), which are further linked through I1···O2 close contacts [symmetry code: (i) $1 + x, \frac{1}{2} - y, -\frac{1}{2} + z$] of 3.193 (4) Å, leading to infinite chains along [20$\bar{1}$]. Similar I···O interactions are observed in the structures of 4-iodoisatin (Golen & Manke, 2016*a*) and 5-iodoisatin (Garden *et al.*, 2006). The structure of 7-bromoisatin also demonstrates a similar halogen–oxygen interaction (Golen & Manke, 2016*b*). The packing of the title compound, indicating the hydrogen bonding, is shown in Fig. 2.

Synthesis and crystallization

A commercial sample (AK Scientific) of 7-iodo-1*H*-indole-2,3-dione was used for crystallization. A sample suitable for single-crystal X-ray analysis was grown by slow evaporation from an acetone solution.

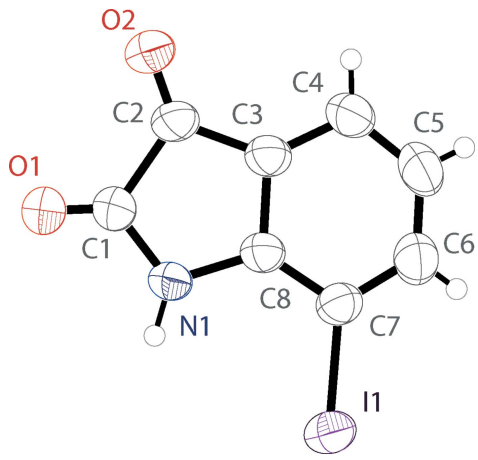


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

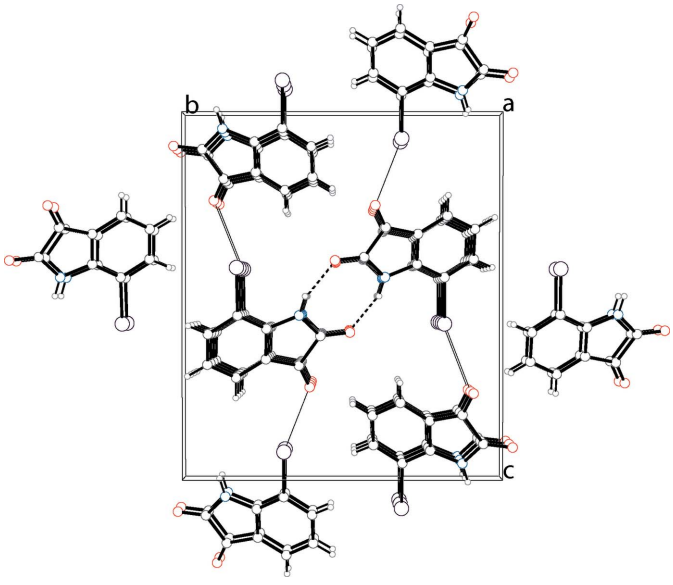


Figure 2
The molecular packing of the title compound, with hydrogen bonding shown as dashed lines and iodine–oxygen interactions shown as thin solid lines.

Refinement

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

Acknowledgements

We greatly 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 (1)	2.08 (2)	2.915 (6)	160 (5)

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

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)	296
a, b, c (Å)	4.0896 (3), 13.2139 (10), 15.1946 (11)
β (°)	92.325 (4)
V (Å^3)	820.43 (10)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	30.33
Crystal size (mm)	0.2 × 0.1 × 0.08
Data collection	
Diffractometer	Bruker D8 Venture CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.037, 0.167
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11737, 1479, 1321
R_{int}	0.061
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.076, 1.12
No. of reflections	1479
No. of parameters	113
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})	0.48, −0.49

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

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full crystallographic data

IUCrData (2016). **1**, x160412 [doi:10.1107/S2414314616004120]

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Crystal data

$C_8H_4INO_2$
M_r = 273.02
Monoclinic, $P2_1/c$
a = 4.0896 (3) Å
b = 13.2139 (10) Å
c = 15.1946 (11) Å
β = 92.325 (4)°
V = 820.43 (10) Å^3
Z = 4
$F(000)$ = 512
D_x = 2.210 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 7596 reflections
θ = 4.4–67.8°
μ = 30.33 mm^{-1}
T = 296 K
BLOCK, red
0.2 × 0.1 × 0.08 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} = 0.037, T_{max} = 0.167
11737 measured reflections
1479 independent reflections
1321 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 68.1°, θ_{min} = 4.4°
h = −4→4
k = −15→15
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.076
S = 1.12
1479 reflections
113 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0197P)^2 + 2.0314P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Special details

Experimental. Absorption correction: *SADABS2014*/4 (Bruker,2014/4) was used for absorption correction. wR2(int) was 0.1330 before and 0.0872 after correction. The Ratio of minimum to maximum transmission is 0.2240. The $\lambda/2$ correction factor is 0.00150.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.19785 (10)	0.18336 (3)	0.57456 (2)	0.05351 (16)
O1	0.7430 (14)	0.5226 (3)	0.4045 (3)	0.0806 (16)
O2	0.9639 (13)	0.3999 (3)	0.2590 (3)	0.0719 (14)
N1	0.5378 (14)	0.3695 (3)	0.4492 (3)	0.0529 (12)
C1	0.6936 (17)	0.4330 (4)	0.3954 (4)	0.0587 (16)
C2	0.8030 (16)	0.3679 (4)	0.3167 (3)	0.0525 (14)
C3	0.6755 (14)	0.2681 (4)	0.3337 (3)	0.0451 (12)
C4	0.6910 (18)	0.1779 (4)	0.2852 (4)	0.0578 (16)
H4	0.7903	0.1760	0.2312	0.069*
C5	0.5550 (17)	0.0925 (4)	0.3200 (4)	0.0617 (16)
H5	0.5622	0.0318	0.2891	0.074*
C6	0.4069 (16)	0.0956 (4)	0.4006 (4)	0.0555 (14)
H6	0.3138	0.0370	0.4224	0.067*
C7	0.3947 (14)	0.1848 (4)	0.4495 (3)	0.0439 (11)
C8	0.5242 (14)	0.2709 (4)	0.4142 (3)	0.0430 (12)
H1	0.463 (13)	0.387 (4)	0.4997 (18)	0.046 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0581 (3)	0.0575 (2)	0.0458 (2)	−0.00171 (17)	0.01231 (16)	0.00592 (15)
O1	0.138 (5)	0.041 (2)	0.065 (3)	−0.007 (2)	0.040 (3)	−0.0021 (18)
O2	0.111 (4)	0.051 (2)	0.056 (2)	0.002 (2)	0.036 (3)	0.0072 (18)
N1	0.085 (4)	0.040 (2)	0.035 (2)	0.004 (2)	0.017 (2)	−0.0001 (18)
C1	0.092 (5)	0.040 (3)	0.045 (3)	0.000 (3)	0.015 (3)	0.000 (2)
C2	0.076 (4)	0.046 (3)	0.036 (3)	0.010 (3)	0.010 (3)	0.009 (2)
C3	0.055 (4)	0.045 (3)	0.036 (2)	0.008 (2)	0.005 (2)	0.003 (2)
C4	0.084 (5)	0.050 (3)	0.040 (3)	0.009 (3)	0.008 (3)	−0.002 (2)
C5	0.083 (5)	0.046 (3)	0.056 (3)	−0.001 (3)	0.001 (3)	−0.013 (3)
C6	0.068 (4)	0.044 (3)	0.054 (3)	−0.006 (3)	−0.001 (3)	−0.002 (2)
C7	0.047 (3)	0.045 (3)	0.040 (2)	0.003 (2)	0.005 (2)	0.005 (2)
C8	0.053 (3)	0.042 (3)	0.034 (2)	0.003 (2)	0.005 (2)	0.002 (2)

Geometric parameters (Å, º)

I1—C7	2.094 (5)	C3—C8	1.395 (7)
O1—C1	1.207 (7)	C4—H4	0.9300
O2—C2	1.196 (6)	C4—C5	1.373 (8)
N1—C1	1.350 (7)	C5—H5	0.9300
N1—C8	1.408 (6)	C5—C6	1.389 (8)
N1—H1	0.869 (5)	C6—H6	0.9300
C1—C2	1.553 (7)	C6—C7	1.395 (7)
C2—C3	1.445 (8)	C7—C8	1.372 (7)
C3—C4	1.404 (7)		

C1—N1—C8	111.0 (4)	C5—C4—H4	121.0
C1—N1—H1	124 (4)	C4—C5—H5	119.6
C8—N1—H1	125 (4)	C4—C5—C6	120.9 (5)
O1—C1—N1	128.3 (5)	C6—C5—H5	119.6
O1—C1—C2	125.4 (5)	C5—C6—H6	119.3
N1—C1—C2	106.2 (4)	C5—C6—C7	121.4 (5)
O2—C2—C1	123.5 (5)	C7—C6—H6	119.3
O2—C2—C3	131.9 (5)	C6—C7—I1	119.9 (4)
C3—C2—C1	104.6 (4)	C8—C7—I1	122.0 (4)
C4—C3—C2	131.1 (5)	C8—C7—C6	118.0 (5)
C8—C3—C2	108.0 (4)	C3—C8—N1	110.1 (4)
C8—C3—C4	121.0 (5)	C7—C8—N1	129.0 (4)
C3—C4—H4	121.0	C7—C8—C3	120.8 (5)
C5—C4—C3	117.9 (5)		

I1—C7—C8—N1	−3.2 (9)	C2—C3—C8—N1	2.1 (6)
I1—C7—C8—C3	175.2 (4)	C2—C3—C8—C7	−176.6 (5)
O1—C1—C2—O2	4.0 (11)	C3—C4—C5—C6	0.0 (10)
O1—C1—C2—C3	−177.5 (7)	C4—C3—C8—N1	−179.4 (6)
O2—C2—C3—C4	−2.8 (12)	C4—C3—C8—C7	1.9 (9)
O2—C2—C3—C8	175.5 (7)	C4—C5—C6—C7	−0.9 (10)
N1—C1—C2—O2	−175.9 (6)	C5—C6—C7—I1	−175.7 (5)
N1—C1—C2—C3	2.7 (7)	C5—C6—C7—C8	2.2 (9)
C1—N1—C8—C3	−0.2 (7)	C6—C7—C8—N1	178.9 (5)
C1—N1—C8—C7	178.3 (6)	C6—C7—C8—C3	−2.6 (9)
C1—C2—C3—C4	178.9 (7)	C8—N1—C1—O1	178.6 (7)
C1—C2—C3—C8	−2.8 (6)	C8—N1—C1—C2	−1.5 (7)
C2—C3—C4—C5	177.6 (6)	C8—C3—C4—C5	−0.5 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.87 (1)	2.08 (2)	2.915 (6)	160 (5)

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