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5-Bromo-1*H*-indole-2,3-dione

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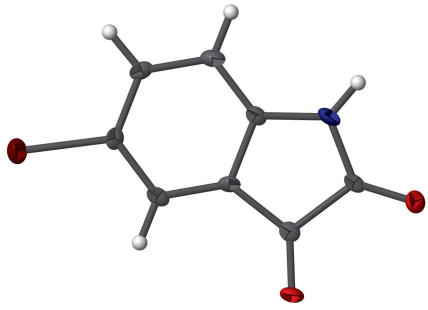
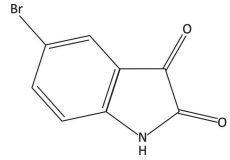
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_4BrNO_2$, has a single, almost planar, planar molecule in the asymmetric unit, with the non-H atoms having a mean deviation from planarity of 0.024 Å. In the crystal, N—H···O hydrogen bonds link the molecules into [001] $C(4)$ chains. C—H···O interactions form [0$\bar{1}$1] chains. These interactions combine to generate sheets along (100). No π–π interactions are observed in the structure.

3D view



Chemical scheme



Structure description

Herein, we report the crystal structure of 5-bromoisatin (Fig. 1), as part of a continuing study on halogenated isatins. The structure exhibits a near planar molecule with the non-hydrogen atoms possessing a mean deviation from planarity of 0.024 Å, with similar bond lengths and angles to those observed in isatin (Goldschmidt *et al.*, 1950). The structure of other bromoisatins report short Br···O contacts (Huang *et al.*, 2016; Turbitt *et al.*, 2016), which are not observed in 5-bromoisatin. The structures of *N*-subsistuted derivatives of 5-bromoisatin have been reported (Kurkin *et al.*, 2008; Maamri *et al.*, 2012) with only one instance of a Br···O interaction being observed (Kharbach *et al.*, 2015).

In the crystals, the molecules form [001] $C(4)$ chains through N1—H1···O1 hydrogen bonds (Table 1). C7—H7···O2 interactions form [0$\bar{1}$1] chains. The combination of these two interactions results i sheets along (100). No π–π interactions are observed in the structure. The packing of the title compound indicating hydrogen bonding is shown in Fig. 2.

Synthesis and crystallization

A commercial sample (Matrix Scientific) of 5-bromo-1*H*-indole-2,3-dione was used for the crystallization. A sample suitable for single-crystal X-ray analysis was grown from the slow evaporation of its acetonitrile solution.

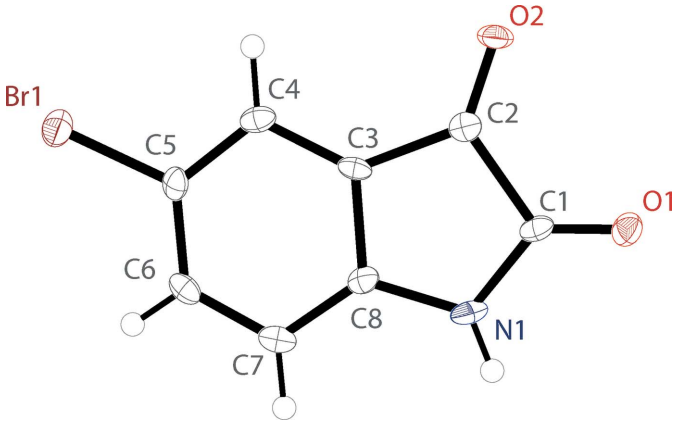


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

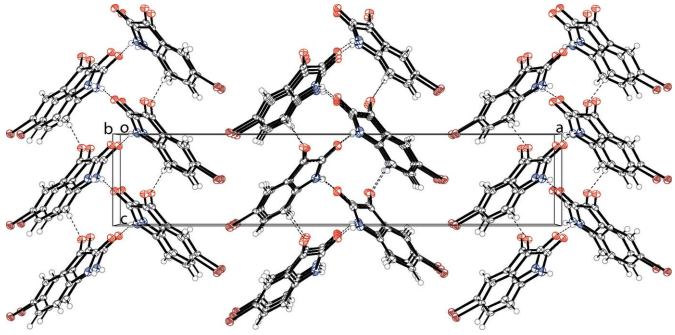


Figure 2
The molecular packing of the title compound along the *b* axis with N—H···O hydrogen bonds shown as dashed lines.

Refinement

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

Acknowledgements

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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)	2.05 (3)	2.886 (6)	166 (9)
C7—H7···O2^{ii}	0.95	2.38	3.312 (7)	167

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_4BrNO_2$
M_r	226.03
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	120
a, b, c (Å)	25.1411 (18), 5.6851 (4), 5.1593 (3)
V (Å^3)	737.42 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	5.52
Crystal size (mm)	0.2 × 0.12 × 0.1
Data collection	
Diffractometer	Bruker D8 Venture CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.109, 0.148
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8877, 1334, 1232
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.603
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.025, 0.054, 1.12
No. of reflections	1334
No. of parameters	113
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.79
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.02 (2)

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

full crystallographic data

IUCrData (2016). **1**, x160177 [https://doi.org/10.1107/S2414314616001772]

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5-Bromo-1*H*-indole-2,3-dione

Crystal data

$C_8H_4BrNO_2$
M_r = 226.03
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
a = 25.1411 (18) Å
b = 5.6851 (4) Å
c = 5.1593 (3) Å
V = 737.42 (9) Å^3
Z = 4
$F(000)$ = 440
D_x = 2.036 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5354 reflections
θ = 3.2–25.4°
μ = 5.52 mm^{-1}
T = 120 K
Block, orange
0.2 × 0.12 × 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} = 0.109, T_{max} = 0.148
8877 measured reflections
1334 independent reflections
1232 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.4°, θ_{min} = 3.2°
h = −30→30
k = −6→6
l = −6→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.054
S = 1.12
1334 reflections
113 parameters
2 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + 1.6735P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.79 e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.02 (2)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.26549 (2)	0.64837 (9)	0.99780 (18)	0.02277 (16)
O1	0.50004 (16)	0.2784 (7)	0.1243 (9)	0.0188 (9)
O2	0.42674 (16)	0.6864 (6)	0.1519 (8)	0.0185 (9)
N1	0.45090 (16)	0.1622 (7)	0.4830 (18)	0.0185 (10)
H1	0.4664 (18)	0.028 (5)	0.496 (19)	0.022*
C1	0.4649 (2)	0.3068 (9)	0.2839 (11)	0.0151 (12)
C2	0.4258 (2)	0.5187 (9)	0.2971 (12)	0.0144 (12)
C3	0.38981 (18)	0.4647 (8)	0.5154 (17)	0.0146 (11)
C4	0.3472 (2)	0.5867 (10)	0.6209 (12)	0.0156 (12)
H4	0.3359	0.7335	0.5523	0.019*
C5	0.3218 (2)	0.4844 (10)	0.8311 (11)	0.0148 (12)
C6	0.3381 (2)	0.2689 (10)	0.9336 (10)	0.0151 (14)
H6	0.3195	0.2040	1.0771	0.018*
C7	0.3812 (2)	0.1476 (11)	0.8286 (11)	0.0176 (13)
H7	0.3925	0.0011	0.8980	0.021*
C8	0.4068 (2)	0.2485 (10)	0.6199 (11)	0.0138 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0220 (3)	0.0254 (3)	0.0210 (3)	0.0056 (2)	0.0035 (5)	−0.0021 (5)
O1	0.021 (2)	0.016 (2)	0.020 (2)	0.0018 (17)	0.0033 (18)	−0.0030 (17)
O2	0.026 (2)	0.012 (2)	0.017 (2)	0.0028 (17)	−0.0013 (18)	0.0046 (18)
N1	0.020 (2)	0.0111 (19)	0.024 (3)	0.0047 (18)	−0.002 (4)	0.003 (3)
C1	0.018 (3)	0.009 (3)	0.018 (3)	0.002 (2)	−0.003 (3)	−0.002 (2)
C2	0.016 (3)	0.013 (3)	0.014 (3)	−0.001 (2)	−0.004 (2)	−0.003 (2)
C3	0.017 (2)	0.015 (2)	0.012 (3)	0.0009 (19)	−0.004 (4)	0.004 (4)
C4	0.018 (3)	0.013 (3)	0.016 (3)	0.002 (2)	−0.006 (2)	−0.001 (2)
C5	0.016 (3)	0.019 (3)	0.010 (3)	0.001 (2)	0.000 (2)	−0.004 (2)
C6	0.020 (3)	0.016 (3)	0.009 (4)	−0.005 (2)	−0.002 (2)	0.000 (2)
C7	0.021 (3)	0.014 (3)	0.018 (3)	−0.001 (3)	−0.004 (2)	0.004 (3)
C8	0.017 (3)	0.011 (3)	0.013 (3)	0.001 (2)	−0.004 (2)	−0.003 (3)

Geometric parameters (Å, º)

Br1—C5	1.901 (5)	C3—C8	1.409 (8)
O1—C1	1.219 (7)	C4—H4	0.9500
O2—C2	1.213 (7)	C4—C5	1.386 (8)
N1—H1	0.858 (14)	C5—C6	1.396 (8)
N1—C1	1.362 (9)	C6—H6	0.9500
N1—C8	1.403 (8)	C6—C7	1.394 (8)
C1—C2	1.555 (7)	C7—H7	0.9500
C2—C3	1.478 (9)	C7—C8	1.380 (8)
C3—C4	1.387 (8)		

C1—N1—H1	119 (6)	C5—C4—H4	121.5
C1—N1—C8	111.9 (4)	C4—C5—Br1	119.4 (4)
C8—N1—H1	129 (5)	C4—C5—C6	122.0 (5)
O1—C1—N1	128.1 (5)	C6—C5—Br1	118.5 (4)
O1—C1—C2	126.2 (5)	C5—C6—H6	119.5
N1—C1—C2	105.8 (5)	C7—C6—C5	121.0 (5)
O2—C2—C1	124.7 (5)	C7—C6—H6	119.5
O2—C2—C3	130.2 (5)	C6—C7—H7	121.3
C3—C2—C1	105.0 (4)	C8—C7—C6	117.4 (5)
C4—C3—C2	132.0 (5)	C8—C7—H7	121.3
C4—C3—C8	121.3 (6)	N1—C8—C3	110.6 (5)
C8—C3—C2	106.7 (5)	C7—C8—N1	128.1 (5)
C3—C4—H4	121.5	C7—C8—C3	121.3 (5)
C5—C4—C3	116.9 (5)		

Br1—C5—C6—C7	−176.2 (4)	C2—C3—C8—C7	−180.0 (5)
O1—C1—C2—O2	−2.1 (9)	C3—C4—C5—Br1	176.7 (5)
O1—C1—C2—C3	177.5 (6)	C3—C4—C5—C6	0.1 (8)
O2—C2—C3—C4	−0.6 (11)	C4—C3—C8—N1	−179.0 (6)
O2—C2—C3—C8	−179.4 (6)	C4—C3—C8—C7	1.1 (9)
N1—C1—C2—O2	178.7 (6)	C4—C5—C6—C7	0.4 (8)
N1—C1—C2—C3	−1.7 (6)	C5—C6—C7—C8	−0.2 (8)
C1—N1—C8—C3	−1.2 (7)	C6—C7—C8—N1	179.6 (6)
C1—N1—C8—C7	178.7 (6)	C6—C7—C8—C3	−0.6 (8)
C1—C2—C3—C4	179.9 (7)	C8—N1—C1—O1	−177.4 (6)
C1—C2—C3—C8	1.1 (6)	C8—N1—C1—C2	1.8 (7)
C2—C3—C4—C5	−179.5 (6)	C8—C3—C4—C5	−0.8 (9)
C2—C3—C8—N1	−0.1 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86 (1)	2.05 (3)	2.886 (6)	166 (9)
C7—H7···O2^{ii}	0.95	2.38	3.312 (7)	167

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