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1-(3-Bromopropyl)indoline-2,3-dione

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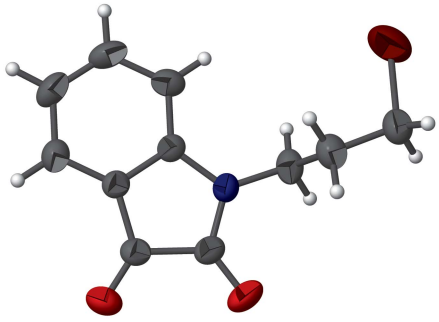
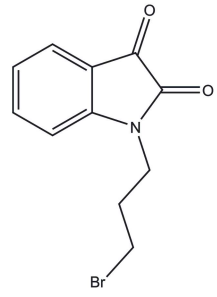
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{11}H_{10}BrNO_2$, the indoline ring system has an r.m.s. deviation of 0.026 Å. The side chain (including the Br atom) has a *trans–gauche* conformation, as indicated by the N—C—C—C and C—C—C—Br torsion angles of −177.5 (3) and 68.1 (3)°, respectively. In the crystal, molecules are linked by weak C—H···O hydrogen bonds, forming a three-dimensional network.

3D view



Chemical scheme



Structure description

Isatin (1*H*-indole-2,3-dione) derivatives possess diverse activities such as antibacterial, antifungal, antiviral, anti-HIV, anti-mycobacterial, anticancer, anti-inflammatory and anticonvulsant activities (Bhrigu *et al.* 2010; Malhotra *et al.* 2011; Ramachandran, 2011; Smitha *et al.* 2008).

As a continuation of our work on the development of isatin derivatives (Mamari *et al.*, 2010), we report here the synthesis of a new indoline-2,3-dione derivative obtained using 1,3-dibromopropane as an alkylating agent.

The title molecule is shown in Fig. 1. The non-H atoms of the indoline core are virtually coplanar [with a maximum deviation of 0.030 (2) Å for N1]. The oxygen atoms O1 and O2 are essentially co-planar with the fused ring system, with the largest deviation from the mean plane being 0.027 (2) Å for O1. The geometric parameters of the title molecule agree well with those reported for similar structures (Mamari *et al.*, 2010). The sum of the angles at N1 (360.1°) is in accordance with sp^2 hybridization for this atom.

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O2^{i}	0.93	2.57	3.232 (3)	129
C9—H9B···O2^{ii}	0.97	2.59	3.444 (4)	146

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

The crystal packing in the title compound is shown in Figs. 2 and 3. The molecules are linked by C—H···O hydrogen-bonding interactions (Table 1), building a three-dimensional network.

Synthesis and crystallization

To a solution of isatin (0.5 g, 3.4 mmol) dissolved in DMF (20 ml) was added potassium carbonate (0.61 g, 4.4 mmol), a catalytic quantity of tetra-*n*-butylammonium bromide (0.1 g, 0.4 mmol) and 1,3-dibromopropane (0.2 ml, 4.1 mmol). The mixture was stirred for 48 h; the reaction was monitored by thin layer chromatography. The mixture was filtered and the solvent removed under vacuum. The solid that was obtained was recrystallized from ethanol solution to afford the title compound as orange crystals in 74% yield.

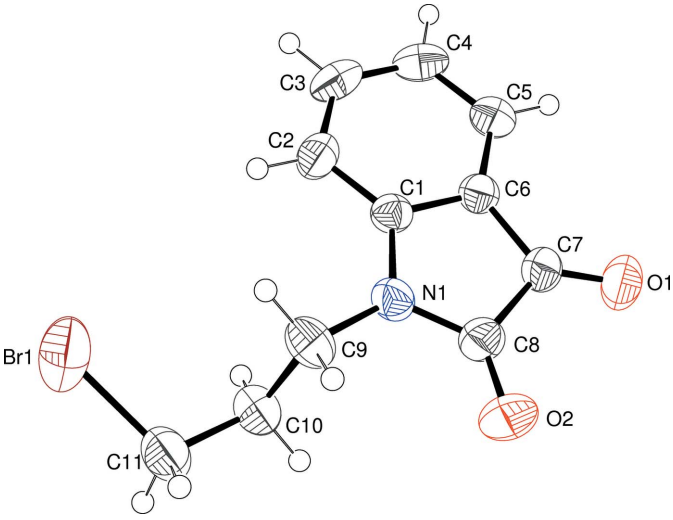


Figure 1
Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

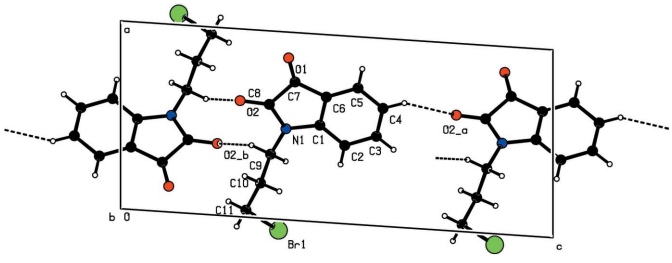


Figure 2
Hydrogen-bond interactions (dashed lines) in the title compound.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{10}BrNO_2$
M_r	268.11
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	7.7113 (2), 8.1375 (2), 17.8089 (4)
β (°)	93.8300 (13)
V (Å^3)	1115.03 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.67
Crystal size (mm)	0.39 × 0.24 × 0.24
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.595, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	39739, 3393, 2360
R_{int}	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.714
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.158, 1.04
No. of reflections	3393
No. of parameters	136
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.10, −0.99

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

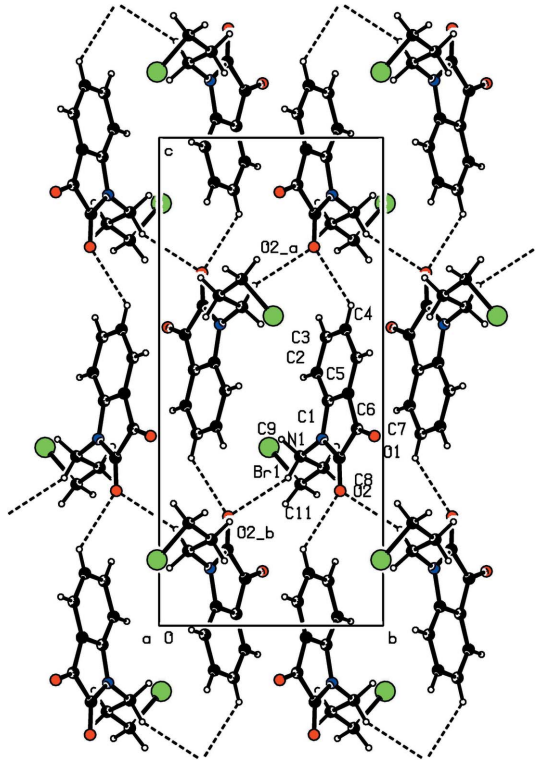


Figure 3
View down the *a* axis of the packing of the title compound. Dashed lines indicate the C—H···O interactions. [Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.]

Refinement

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

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full crystallographic data

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Crystal data

$C_{11}H_{10}BrNO_2$
M_r = 268.11
Monoclinic, $P2_1/c$
a = 7.7113 (2) Å
b = 8.1375 (2) Å
c = 17.8089 (4) Å
β = 93.8300 (13)°
V = 1115.03 (5) Å^3
Z = 4
$F(000)$ = 536
D_x = 1.597 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9592 reflections
θ = 2.3–26.6°
μ = 3.67 mm^{-1}
T = 296 K
Irregular parallelepiped, orange
0.39 × 0.24 × 0.24 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.595, T_{max} = 0.746
39739 measured reflections
3393 independent reflections
2360 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 30.5°, θ_{min} = 2.7°
h = −11→11
k = −11→11
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.158
S = 1.04
3393 reflections
136 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2 + 1.1939P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.99 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.05853 (6)	0.49061 (6)	0.36531 (3)	0.0874 (2)
C1	0.5157 (3)	0.7542 (3)	0.46085 (14)	0.0360 (5)
C2	0.4168 (4)	0.7039 (4)	0.51842 (16)	0.0465 (6)
H2	0.3145	0.6446	0.5088	0.056*
C3	0.4765 (4)	0.7455 (4)	0.59154 (16)	0.0529 (7)
H3	0.4116	0.7138	0.6313	0.063*
C4	0.6279 (5)	0.8318 (4)	0.60698 (15)	0.0527 (7)
H4	0.6640	0.8567	0.6565	0.063*
C5	0.7270 (4)	0.8819 (4)	0.54906 (15)	0.0455 (6)
H5	0.8302	0.9394	0.5590	0.055*
C6	0.6679 (3)	0.8439 (3)	0.47589 (13)	0.0367 (5)
C7	0.7371 (3)	0.8840 (3)	0.40349 (15)	0.0417 (5)
C8	0.6087 (4)	0.8045 (4)	0.34400 (14)	0.0441 (6)
C9	0.3438 (4)	0.6304 (4)	0.34693 (17)	0.0476 (6)
H9A	0.3108	0.5462	0.3818	0.057*
H9B	0.3845	0.5760	0.3029	0.057*
C10	0.1865 (4)	0.7331 (4)	0.32359 (18)	0.0503 (7)
H10B	0.2184	0.8142	0.2871	0.060*
H10A	0.1492	0.7914	0.3672	0.060*
C11	0.0372 (4)	0.6315 (5)	0.28988 (18)	0.0600 (8)
H11B	0.0772	0.5641	0.2496	0.072*
H11A	−0.0532	0.7038	0.2686	0.072*
N1	0.4849 (3)	0.7280 (3)	0.38246 (12)	0.0410 (5)
O1	0.8630 (3)	0.9615 (3)	0.38866 (13)	0.0589 (6)
O2	0.6181 (3)	0.8085 (3)	0.27643 (11)	0.0638 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0725 (3)	0.0879 (4)	0.1039 (4)	−0.0261 (2)	0.0208 (2)	0.0013 (2)
C1	0.0370 (11)	0.0371 (12)	0.0340 (11)	0.0042 (9)	0.0024 (9)	0.0005 (9)
C2	0.0462 (14)	0.0456 (14)	0.0489 (15)	−0.0034 (12)	0.0109 (11)	0.0068 (12)
C3	0.071 (2)	0.0511 (16)	0.0378 (13)	0.0049 (14)	0.0166 (13)	0.0112 (12)
C4	0.074 (2)	0.0527 (16)	0.0309 (12)	0.0054 (15)	−0.0026 (12)	0.0021 (11)
C5	0.0463 (14)	0.0494 (15)	0.0400 (13)	0.0006 (12)	−0.0043 (11)	−0.0018 (11)
C6	0.0355 (12)	0.0400 (12)	0.0347 (11)	0.0024 (9)	0.0025 (9)	−0.0003 (9)
C7	0.0364 (12)	0.0486 (14)	0.0407 (12)	0.0017 (11)	0.0076 (10)	−0.0009 (11)
C8	0.0442 (14)	0.0529 (15)	0.0357 (12)	0.0031 (12)	0.0061 (10)	−0.0013 (11)
C9	0.0443 (14)	0.0458 (15)	0.0516 (15)	0.0009 (11)	−0.0054 (11)	−0.0114 (12)
C10	0.0441 (14)	0.0510 (16)	0.0550 (16)	−0.0012 (12)	−0.0031 (12)	−0.0019 (13)
C11	0.0449 (16)	0.080 (2)	0.0541 (17)	−0.0068 (15)	−0.0019 (13)	−0.0093 (16)
N1	0.0372 (10)	0.0508 (13)	0.0346 (10)	−0.0021 (9)	−0.0002 (8)	−0.0049 (9)
O1	0.0470 (12)	0.0733 (14)	0.0581 (13)	−0.0128 (10)	0.0155 (10)	0.0002 (11)
O2	0.0737 (15)	0.0865 (17)	0.0318 (10)	−0.0001 (13)	0.0092 (9)	−0.0027 (10)

Geometric parameters (Å, º)

C11—Br1	1.948 (4)	C7—O1	1.202 (3)
C1—C2	1.380 (4)	C7—C8	1.543 (4)
C1—C6	1.393 (4)	C8—O2	1.211 (3)
C1—N1	1.417 (3)	C8—N1	1.363 (3)
C2—C3	1.394 (4)	C9—N1	1.457 (3)
C2—H2	0.9300	C9—C10	1.508 (4)
C3—C4	1.375 (5)	C9—H9A	0.9700
C3—H3	0.9300	C9—H9B	0.9700
C4—C5	1.385 (4)	C10—C11	1.510 (4)
C4—H4	0.9300	C10—H10B	0.9700
C5—C6	1.386 (4)	C10—H10A	0.9700
C5—H5	0.9300	C11—H11B	0.9700
C6—C7	1.465 (3)	C11—H11A	0.9700
C2—C1—C6	120.9 (2)	N1—C8—C7	106.6 (2)
C2—C1—N1	128.5 (2)	N1—C9—C10	112.4 (2)
C6—C1—N1	110.6 (2)	N1—C9—H9A	109.1
C1—C2—C3	117.2 (3)	C10—C9—H9A	109.1
C1—C2—H2	121.4	N1—C9—H9B	109.1
C3—C2—H2	121.4	C10—C9—H9B	109.1
C4—C3—C2	122.3 (3)	H9A—C9—H9B	107.9
C4—C3—H3	118.9	C9—C10—C11	112.6 (3)
C2—C3—H3	118.9	C9—C10—H10B	109.1
C3—C4—C5	120.3 (3)	C11—C10—H10B	109.1
C3—C4—H4	119.8	C9—C10—H10A	109.1
C5—C4—H4	119.8	C11—C10—H10A	109.1
C4—C5—C6	118.1 (3)	H10B—C10—H10A	107.8
C4—C5—H5	120.9	C10—C11—Br1	111.2 (2)
C6—C5—H5	120.9	C10—C11—H11B	109.4
C5—C6—C1	121.1 (2)	Br1—C11—H11B	109.4
C5—C6—C7	131.4 (2)	C10—C11—H11A	109.4
C1—C6—C7	107.4 (2)	Br1—C11—H11A	109.4
O1—C7—C6	131.1 (3)	H11B—C11—H11A	108.0
O1—C7—C8	124.0 (2)	C8—N1—C1	110.5 (2)
C6—C7—C8	104.9 (2)	C8—N1—C9	124.0 (2)
O2—C8—N1	127.0 (3)	C1—N1—C9	125.6 (2)
O2—C8—C7	126.4 (3)		
C6—C1—C2—C3	−0.4 (4)	C6—C7—C8—O2	−180.0 (3)
N1—C1—C2—C3	179.8 (3)	O1—C7—C8—N1	179.8 (3)
C1—C2—C3—C4	−0.5 (4)	C6—C7—C8—N1	−0.7 (3)
C2—C3—C4—C5	0.4 (5)	N1—C9—C10—C11	−177.5 (3)
C3—C4—C5—C6	0.6 (4)	C9—C10—C11—Br1	68.1 (3)
C4—C5—C6—C1	−1.6 (4)	O2—C8—N1—C1	−178.6 (3)
C4—C5—C6—C7	177.2 (3)	C7—C8—N1—C1	2.1 (3)
C2—C1—C6—C5	1.6 (4)	O2—C8—N1—C9	2.6 (5)

N1—C1—C6—C5	−178.6 (2)	C7—C8—N1—C9	−176.7 (2)
C2—C1—C6—C7	−177.5 (2)	C2—C1—N1—C8	177.0 (3)
N1—C1—C6—C7	2.3 (3)	C6—C1—N1—C8	−2.8 (3)
C5—C6—C7—O1	−0.5 (5)	C2—C1—N1—C9	−4.3 (4)
C1—C6—C7—O1	178.5 (3)	C6—C1—N1—C9	175.9 (2)
C5—C6—C7—C8	−179.9 (3)	C10—C9—N1—C8	−88.0 (3)
C1—C6—C7—C8	−0.9 (3)	C10—C9—N1—C1	93.4 (3)
O1—C7—C8—O2	0.6 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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