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5-Bromo-1-(6-bromohexyl)indoline-2,3-dione

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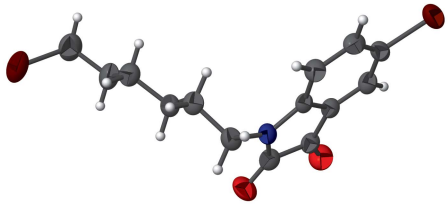
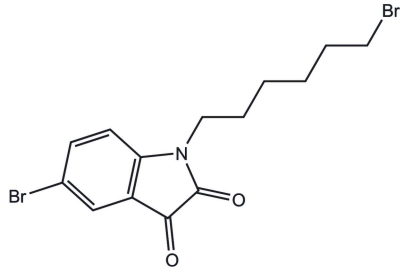
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{14}H_{15}Br_2NO_2$, the dihedral angle between the mean plane passing through the bromohexyl chain and the 5-bromoindoline ring system (r.m.s. deviation = 0.044Å) is 70.0 (3)°. In the crystal, molecules are connected by C—H···O hydrogen bonds, generating zigzag chains propagating along [010]. The packing is also influenced by inter-chain π–π interactions which form layers parallel to the *ab* plane [centroid–centroid distances = 3.765 (2) Å].

3D view



Chemical scheme



Structure description

Isatin derivatives have a wide range of biological properties and show pharmacological activity against bacteria and fungi (Pandeya *et al.*, 2005; Vine *et al.*, 2007). The most demanding prospect of research surrounding isatin derivatives has evolved in the context of their antifungal and antiviral activities (Aboul-Fadl *et al.*, 2010). The significance of isatin derivatives has even been extended to the design of novel anticancer drugs (Rodríuez-Argüelles *et al.*, 2004), and recently, a number of isatin-based compounds have been reported to be inhibitors of caspase-3 and caspase-7 (Chu *et al.*, 2007). As part of a continuing study on halogenated isatins (Kharbach *et al.*, 2016*a*, Kharbach *et al.*, 2016*b*), the structures of *N*-substituted derivatives of isatin have been reported using 1,3-dibromopropane (Qachchachi *et al.*, 2016). Herein, we report the crystal structure of 5-bromo-1-(6-bromohexyl)indoline-2,3-dione, obtained using 1,6-dibromohexane as an alkylating agent as part of our work to develop new 5-bromoisatin derivatives.

In the title compound (Fig. 1), the dihedral angle between the mean plane passing through the bromohexyl chain and the 5-bromo-indoline ring system (r.m.s. deviation: 0.044 Å is 70.0 (3)°. In the crystal, molecules are connected by C—H···O hydrogen bonds (Table 1), generating zigzag chains propagating in the [010] direction. The packing

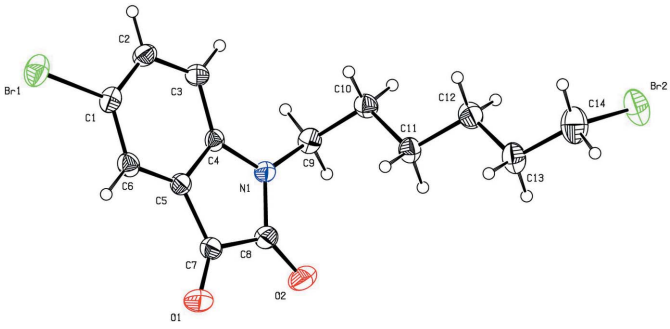


Figure 1
The molecular structure of the title molecule, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

(Fig. 2) is also influenced by inter-chain π–π interactions which form layers parallel to the *ab* plane [centroid-centroid distances = 3.765 (2) Å].

Synthesis and crystallization

To a solution of 5-bromoisatin (0.4 g, 1.76 mmol) and 1,6-dibromohexane (0,31 ml, 1.95 mmol) in DMF (25 ml), was added tetra-*n*-butylammonium bromide (0.1 g, 0.4 mmol) and potassium carbonate (0.6 g, 4.4 mmol). The reaction mixture was stirred for 48 h. After filtering, the solution was evaporated in reduced pressure. The title compound was obtained in 84% yield and recrystallized from ethanol solution to afford orange crystals (m.p. 355 K).

Refinement

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

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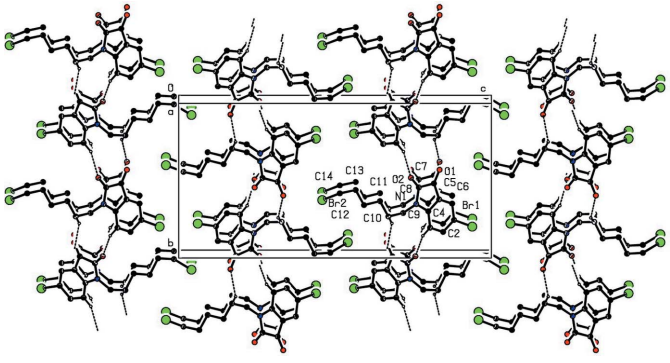

Figure 2
The crystal structure of the title compound, viewed along the *c* axis, showing zigzag chains parallel to the *b* axis linked by C— H···O hydrogen bonds (dashed lines).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.93	2.58	3.406 (5)	148
C10—H10*A*···O1^{ii}	0.97	2.53	3.364 (5)	143

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{15}Br_2NO_2$
M_r	389.09
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	299
a, *b*, *c* (Å)	4.6344 (2), 12.6284 (6), 25.3537 (12)
V (Å^3)	1483.83 (12)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	5.46
Crystal size (mm)	0.22 × 0.11 × 0.05
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.576, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28990, 3663, 2699
R_{int}	0.039
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.072, 1.02
No. of reflections	3663
No. of parameters	172
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.52
Absolute structure	Flack *x* determined using 932 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.006 (4)

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

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full crystallographic data

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5-Bromo-1-(6-bromohexyl)indoline-2,3-dione

Crystal data

$C_{14}H_{15}Br_2NO_2$
M_r = 389.09
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 4.6344 (2) Å
b = 12.6284 (6) Å
c = 25.3537 (12) Å
V = 1483.83 (12) Å^3
Z = 4
F(000) = 768
D_x = 1.742 Mg m^{-3}
Melting point: 355 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8821 reflections
θ = 2.9–23.7°
μ = 5.46 mm^{-1}
T = 299 K
Platelet, orange
0.22 × 0.11 × 0.05 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.576, T_{max} = 0.746
28990 measured reflections
3663 independent reflections
2699 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 28.3°, θ_{min} = 1.6°
h = −6→6
k = −16→15
l = −33→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.072
S = 1.02
3663 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0272P)^2 + 0.7737P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}
Absolute structure: Flack x determined using 932 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.006 (4)

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	0.97543 (11)	0.74683 (4)	0.93893 (2)	0.06086 (16)
Br2	0.48372 (15)	0.60074 (4)	0.45917 (2)	0.07524 (19)
C1	0.7295 (9)	0.7167 (3)	0.88077 (16)	0.0400 (10)
C2	0.6607 (10)	0.7974 (3)	0.84574 (17)	0.0420 (10)
H2	0.7425	0.8641	0.8502	0.050*
C3	0.4716 (11)	0.7800 (3)	0.80417 (15)	0.0403 (9)
H3	0.4225	0.8342	0.7810	0.048*
C4	0.3591 (8)	0.6796 (3)	0.79838 (14)	0.0317 (9)
C5	0.4348 (8)	0.5988 (3)	0.83316 (14)	0.0342 (9)
C6	0.6199 (9)	0.6160 (3)	0.87463 (16)	0.0399 (10)
H6	0.6693	0.5616	0.8977	0.048*
C7	0.2780 (10)	0.5037 (3)	0.81645 (16)	0.0391 (9)
C8	0.0976 (10)	0.5385 (3)	0.76811 (17)	0.0429 (11)
C9	0.0365 (10)	0.7071 (3)	0.71786 (15)	0.0414 (9)
H9A	−0.0100	0.7767	0.7317	0.050*
H9B	−0.1422	0.6743	0.7065	0.050*
C10	0.2323 (9)	0.7199 (3)	0.67068 (16)	0.0407 (10)
H10A	0.4080	0.7550	0.6819	0.049*
H10B	0.1379	0.7654	0.6452	0.049*
C11	0.3121 (10)	0.6165 (3)	0.64382 (16)	0.0416 (10)
H11A	0.1374	0.5769	0.6364	0.050*
H11B	0.4292	0.5745	0.6677	0.050*
C12	0.4772 (11)	0.6332 (3)	0.59266 (16)	0.0496 (10)
H12A	0.3580	0.6738	0.5686	0.060*
H12B	0.6490	0.6745	0.6000	0.060*
C13	0.5652 (11)	0.5312 (4)	0.56595 (17)	0.0553 (12)
H13A	0.6842	0.4908	0.5902	0.066*
H13B	0.3929	0.4899	0.5590	0.066*
C14	0.7274 (12)	0.5448 (5)	0.5153 (2)	0.0712 (15)
H14A	0.8053	0.4769	0.5044	0.085*
H14B	0.8881	0.5927	0.5210	0.085*
N1	0.1629 (7)	0.6432 (2)	0.76016 (13)	0.0353 (7)
O1	0.2758 (8)	0.4155 (2)	0.83436 (13)	0.0592 (9)
O2	−0.0693 (8)	0.4850 (2)	0.74288 (13)	0.0603 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0551 (3)	0.0779 (3)	0.0496 (3)	−0.0053 (3)	−0.0101 (3)	−0.0151 (2)

Br2	0.0904 (4)	0.0907 (4)	0.0447 (3)	0.0124 (4)	0.0123 (3)	0.0084 (2)
C1	0.035 (2)	0.047 (2)	0.038 (2)	0.0017 (18)	−0.0012 (19)	−0.0089 (19)
C2	0.047 (2)	0.035 (2)	0.045 (3)	−0.0051 (19)	0.006 (2)	−0.0083 (19)
C3	0.050 (2)	0.0298 (18)	0.041 (2)	0.001 (2)	0.000 (2)	0.0024 (14)
C4	0.035 (2)	0.0316 (19)	0.028 (2)	0.0048 (16)	0.0040 (17)	−0.0029 (15)
C5	0.039 (2)	0.0302 (17)	0.0330 (19)	0.0030 (17)	0.0041 (17)	−0.0016 (15)
C6	0.047 (2)	0.037 (2)	0.035 (2)	0.0071 (18)	0.0012 (18)	0.0037 (18)
C7	0.047 (2)	0.034 (2)	0.036 (2)	−0.0003 (18)	0.005 (2)	0.0000 (17)
C8	0.051 (3)	0.036 (2)	0.042 (2)	−0.002 (2)	0.006 (2)	−0.0039 (19)
C9	0.042 (2)	0.0414 (19)	0.041 (2)	0.007 (2)	−0.004 (2)	−0.0021 (16)
C10	0.047 (2)	0.036 (2)	0.040 (2)	0.0064 (17)	−0.002 (2)	0.0052 (17)
C11	0.045 (2)	0.044 (2)	0.036 (2)	0.005 (2)	−0.0016 (19)	0.0003 (18)
C12	0.054 (3)	0.052 (2)	0.043 (2)	0.000 (3)	0.001 (3)	0.0028 (17)
C13	0.064 (3)	0.061 (3)	0.040 (2)	0.013 (2)	0.004 (2)	0.000 (2)
C14	0.063 (3)	0.094 (4)	0.056 (3)	0.015 (3)	0.008 (3)	−0.009 (3)
N1	0.0437 (19)	0.0297 (16)	0.0325 (18)	−0.0013 (14)	−0.0024 (15)	0.0004 (14)
O1	0.083 (3)	0.0332 (16)	0.062 (2)	−0.0068 (16)	−0.0018 (19)	0.0053 (14)
O2	0.071 (3)	0.0488 (18)	0.061 (2)	−0.0179 (18)	−0.0154 (19)	−0.0060 (15)

Geometric parameters (Å, º)

Br1—C1	1.902 (4)	C9—C10	1.510 (6)
Br2—C14	1.949 (5)	C9—H9A	0.9700
C1—C6	1.379 (6)	C9—H9B	0.9700
C1—C2	1.389 (6)	C10—C11	1.518 (5)
C2—C3	1.388 (6)	C10—H10A	0.9700
C2—H2	0.9300	C10—H10B	0.9700
C3—C4	1.379 (5)	C11—C12	1.521 (6)
C3—H3	0.9300	C11—H11A	0.9700
C4—C5	1.393 (5)	C11—H11B	0.9700
C4—N1	1.406 (5)	C12—C13	1.511 (6)
C5—C6	1.374 (6)	C12—H12A	0.9700
C5—C7	1.466 (6)	C12—H12B	0.9700
C6—H6	0.9300	C13—C14	1.498 (7)
C7—O1	1.203 (5)	C13—H13A	0.9700
C7—C8	1.547 (6)	C13—H13B	0.9700
C8—O2	1.210 (5)	C14—H14A	0.9700
C8—N1	1.371 (5)	C14—H14B	0.9700
C9—N1	1.465 (5)		
C6—C1—C2	121.3 (4)	C11—C10—H10A	108.7
C6—C1—Br1	119.5 (3)	C9—C10—H10B	108.7
C2—C1—Br1	119.1 (3)	C11—C10—H10B	108.7
C3—C2—C1	120.9 (4)	H10A—C10—H10B	107.6
C3—C2—H2	119.5	C10—C11—C12	112.7 (3)
C1—C2—H2	119.5	C10—C11—H11A	109.1
C4—C3—C2	117.7 (3)	C12—C11—H11A	109.1
C4—C3—H3	121.1	C10—C11—H11B	109.1

C2—C3—H3	121.1	C12—C11—H11B	109.1
C3—C4—C5	120.7 (4)	H11A—C11—H11B	107.8
C3—C4—N1	128.2 (3)	C13—C12—C11	113.6 (3)
C5—C4—N1	111.1 (3)	C13—C12—H12A	108.9
C6—C5—C4	121.8 (3)	C11—C12—H12A	108.9
C6—C5—C7	131.2 (4)	C13—C12—H12B	108.9
C4—C5—C7	107.0 (3)	C11—C12—H12B	108.9
C5—C6—C1	117.5 (4)	H12A—C12—H12B	107.7
C5—C6—H6	121.3	C14—C13—C12	114.9 (4)
C1—C6—H6	121.3	C14—C13—H13A	108.5
O1—C7—C5	130.8 (4)	C12—C13—H13A	108.5
O1—C7—C8	123.9 (4)	C14—C13—H13B	108.5
C5—C7—C8	105.3 (3)	C12—C13—H13B	108.5
O2—C8—N1	127.0 (4)	H13A—C13—H13B	107.5
O2—C8—C7	127.3 (4)	C13—C14—Br2	112.2 (3)
N1—C8—C7	105.7 (3)	C13—C14—H14A	109.2
N1—C9—C10	113.5 (4)	Br2—C14—H14A	109.2
N1—C9—H9A	108.9	C13—C14—H14B	109.2
C10—C9—H9A	108.9	Br2—C14—H14B	109.2
N1—C9—H9B	108.9	H14A—C14—H14B	107.9
C10—C9—H9B	108.9	C8—N1—C4	110.9 (3)
H9A—C9—H9B	107.7	C8—N1—C9	123.4 (3)
C9—C10—C11	114.2 (3)	C4—N1—C9	125.6 (3)
C9—C10—H10A	108.7		

C6—C1—C2—C3	2.0 (7)	C5—C7—C8—O2	−178.0 (4)
Br1—C1—C2—C3	−177.0 (3)	O1—C7—C8—N1	−179.4 (4)
C1—C2—C3—C4	−1.2 (6)	C5—C7—C8—N1	1.2 (4)
C2—C3—C4—C5	−0.2 (6)	N1—C9—C10—C11	60.9 (5)
C2—C3—C4—N1	179.2 (4)	C9—C10—C11—C12	172.7 (4)
C3—C4—C5—C6	0.8 (6)	C10—C11—C12—C13	178.6 (4)
N1—C4—C5—C6	−178.7 (4)	C11—C12—C13—C14	179.8 (4)
C3—C4—C5—C7	179.6 (4)	C12—C13—C14—Br2	−69.3 (5)
N1—C4—C5—C7	0.1 (4)	O2—C8—N1—C4	178.0 (4)
C4—C5—C6—C1	0.0 (6)	C7—C8—N1—C4	−1.2 (4)
C7—C5—C6—C1	−178.4 (4)	O2—C8—N1—C9	−0.7 (7)
C2—C1—C6—C5	−1.4 (6)	C7—C8—N1—C9	−179.9 (4)
Br1—C1—C6—C5	177.7 (3)	C3—C4—N1—C8	−178.7 (4)
C6—C5—C7—O1	−1.6 (8)	C5—C4—N1—C8	0.8 (5)
C4—C5—C7—O1	179.9 (5)	C3—C4—N1—C9	−0.1 (6)
C6—C5—C7—C8	177.9 (4)	C5—C4—N1—C9	179.4 (4)
C4—C5—C7—C8	−0.7 (4)	C10—C9—N1—C8	−100.0 (5)
O1—C7—C8—O2	1.5 (7)	C10—C9—N1—C4	81.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.93	2.58	3.406 (5)	148

C10—H10*A*···O1^{ii}	0.97	2.53	3.364 (5)	143

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+3/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2.