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ISSN 2414-3146

1-Benzyl-5-bromoindoline-2,3-dione

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Received 31 March 2016
Accepted 4 April 2016

Edited by M. Bolte, Goethe-Universität Frankfurt, Germany

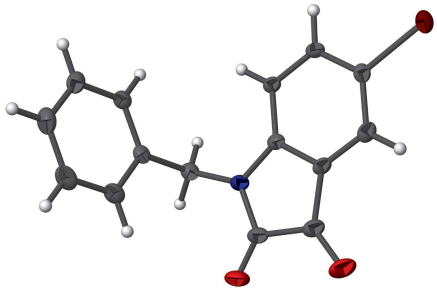
Keywords: crystal structure; indoline ring; hydrogen bonds; π–π interaction.

CCDC reference: 1472134

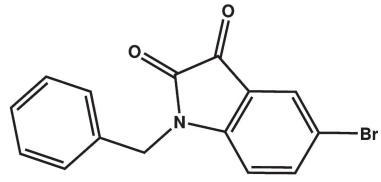
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{10}BrNO_2$, the indoline ring system, the two ketone O atoms and the Br atom lie in a common plane, with the largest deviation from the mean plane being 0.073 (1) Å for the Br atom. The fused-ring system is nearly perpendicular to the benzyl ring, as indicated by the dihedral angle between them of 74.58 (10)°. In the crystal, molecules are linked by weak C—H···O hydrogen bonds and by π–π interactions [inter-centroid distance = 3.625 (2) Å], forming a two-dimensional structure.

3D view



Chemical scheme



Structure description

Isatins and analogous compounds have been the focus of much research due to their anticancer, anti-oxygenic, anticonvulsant, antibacterial and sedative activities (Sridhar *et al.*, 2001*a*,*b*; Sarangapani *et al.*, 1994; Verma *et al.*, 2004; Pandeya *et al.*, 1999; Aboul-Fadl *et al.*, 2010). As a continuation of Qachchachi's research work devoted to the development of isatin (Qachchachi *et al.*, 2013, 2014*a*,*b*), we report in this paper the synthesis and crystal structure of 1-benzyl-5-bromoindoline-2,3-dione.

The title compound (Fig. 1) is built up from two fused five- and six-membered rings linked to two ketone atoms, a bromine atom and a benzyl group, as shown in Fig.1. The fused-ring system and the attached atoms lie in a common plane with a maximum deviation of 0.073 (1) Å for Br1. Moreover, the benzyl ring are nearly perpendicular to the indoline ring system, making a dihedral angle of 74.58 (10)°. The C10—C9—N1—C8 torsion angle is −77.3 (2) °.

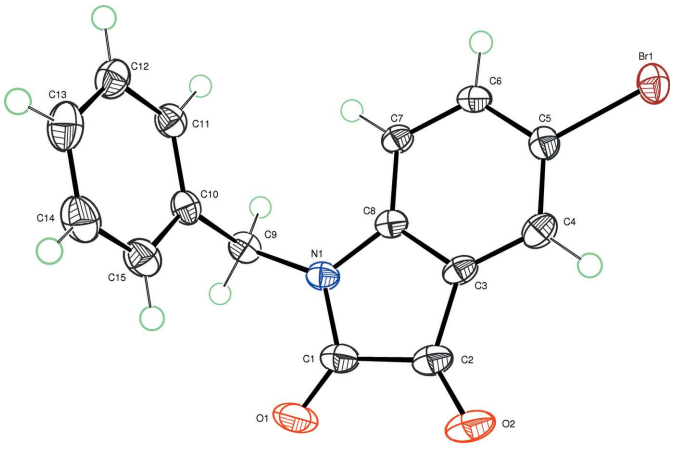


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

In the crystal, molecules are linked by weak C—H···O hydrogen bonds (Table 1) into chains running along the *b* axis. The chains are further connected by π–π interactions [inter-centroid distance = 3.625 (2) Å], forming layers in the *ab* plane (Fig. 2).

Synthesis and crystallization

To a solution of 5-bromoisatin (0.4 g, 1.76 mmol) dissolved in DMF (25 ml) was added potassium carbonate (0.6 g, 4.4 mmol), benzyl chloride (0.22 ml, 1.76 mmol) and a catalytic amount of tetra-*n*-butylammonium bromide (0.1 g, 0.4 mmol). The mixture was stirred for 48 h. After filtering, the reaction was monitored by thin layer chromatography. The title compound was obtained in 72% yield, m.p. = 427 K. The red crystals obtained were analysed by X-ray diffraction.

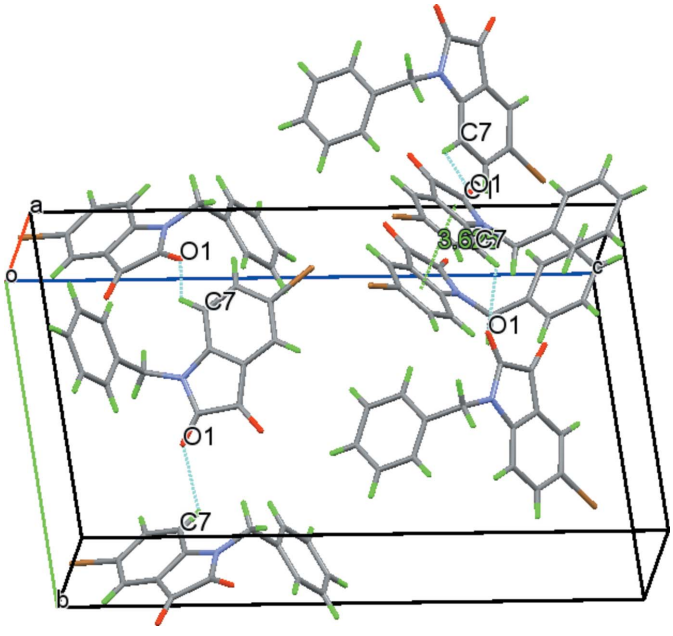


Figure 2
Molecules linked by C—H···O hydrogen bonds and π–π interactions, forming a two-dimensional network.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1^{i}	0.95	2.46	3.056 (2)	121

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

Refinement

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

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{10}BrNO_2$
M_r	316.15
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, *b*, *c* (Å)	4.5205 (1), 13.4538 (3), 21.0436 (5)
V (Å^3)	1279.83 (5)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	3.21
Crystal size (mm)	0.36 × 0.32 × 0.21
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.649, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	40933, 4484, 4045
R_{int}	0.038
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.755
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.026, 0.058, 1.06
No. of reflections	4484
No. of parameters	172
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.54, −0.31
Absolute structure	Flack *x* determined using 1538 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.111 (2)

Computer programs: *APEX2* and *SAINT-Plus* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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full crystallographic data

IUCrData (2016). **1**, x160559 [doi:10.1107/S2414314616005599]

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1-Benzyl-5-bromoindoline-2,3-dione

Crystal data

$C_{15}H_{10}BrNO_2$
M_r = 316.15
Orthorhombic, $P2_12_12_1$
a = 4.5205 (1) Å
b = 13.4538 (3) Å
c = 21.0436 (5) Å
V = 1279.83 (5) Å^3
Z = 4
$F(000)$ = 632
D_x = 1.641 Mg m^{-3}
Melting point: 427 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4483 reflections
θ = 1.8–32.4°
μ = 3.21 mm^{-1}
T = 100 K
Block, red
0.36 × 0.32 × 0.21 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.649, T_{max} = 0.746
40933 measured reflections
4484 independent reflections
4045 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 32.4°, θ_{min} = 1.8°
h = −6→6
k = −19→19
l = −31→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.058
S = 1.06
4484 reflections
172 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0292P)^2 + 0.1626P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack x determined using 1538 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.111 (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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.4855 (5)	0.52077 (16)	0.26022 (11)	0.0228 (5)
C2	−0.2947 (6)	0.53887 (15)	0.32114 (11)	0.0240 (5)
C3	−0.1280 (5)	0.44646 (15)	0.33040 (10)	0.0188 (4)
C4	0.0679 (6)	0.41672 (15)	0.37752 (10)	0.0215 (4)
H4	0.1217	0.4599	0.4113	0.026*
C5	0.1815 (5)	0.32134 (16)	0.37318 (9)	0.0188 (4)
C6	0.1060 (5)	0.25767 (15)	0.32387 (10)	0.0193 (4)
H6	0.1891	0.1928	0.3224	0.023*
C7	−0.0907 (5)	0.28774 (14)	0.27636 (9)	0.0179 (4)
H7	−0.1428	0.2447	0.2424	0.022*
C8	−0.2063 (5)	0.38247 (14)	0.28070 (10)	0.0167 (4)
C9	−0.5340 (5)	0.38185 (15)	0.18194 (10)	0.0203 (4)
H9A	−0.7186	0.4165	0.1698	0.024*
H9B	−0.5837	0.3115	0.1906	0.024*
C10	−0.3180 (5)	0.38661 (16)	0.12731 (10)	0.0191 (4)
C11	−0.1799 (5)	0.30103 (15)	0.10502 (10)	0.0204 (4)
H11	−0.2225	0.2387	0.1241	0.024*
C12	0.0198 (5)	0.30618 (17)	0.05502 (10)	0.0243 (4)
H12	0.1159	0.2477	0.0405	0.029*
C13	0.0785 (6)	0.39639 (19)	0.02647 (11)	0.0301 (5)
H13	0.2132	0.3997	−0.0081	0.036*
C14	−0.0587 (7)	0.48221 (18)	0.04813 (11)	0.0314 (5)
H14	−0.0176	0.5442	0.0284	0.038*
C15	−0.2550 (6)	0.47753 (18)	0.09839 (11)	0.0255 (5)
H15	−0.3475	0.5365	0.1133	0.031*
Br1	0.44066 (5)	0.27424 (2)	0.43746 (2)	0.02327 (6)
N1	−0.4148 (4)	0.42721 (12)	0.23980 (8)	0.0189 (3)
O1	−0.6580 (5)	0.57789 (13)	0.23682 (9)	0.0341 (4)
O2	−0.2970 (5)	0.61533 (13)	0.35125 (9)	0.0359 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0233 (11)	0.0170 (9)	0.0281 (11)	0.0025 (8)	0.0062 (9)	0.0024 (8)
C2	0.0264 (11)	0.0164 (10)	0.0291 (11)	−0.0002 (9)	0.0055 (10)	−0.0018 (8)
C3	0.0215 (11)	0.0137 (8)	0.0211 (9)	−0.0023 (7)	0.0044 (8)	−0.0027 (7)
C4	0.0232 (10)	0.0203 (9)	0.0211 (9)	−0.0044 (9)	0.0029 (10)	−0.0037 (7)
C5	0.0165 (9)	0.0227 (10)	0.0172 (9)	−0.0007 (9)	0.0012 (8)	0.0017 (7)
C6	0.0195 (10)	0.0161 (9)	0.0223 (9)	0.0030 (7)	0.0026 (8)	−0.0003 (7)
C7	0.0198 (10)	0.0143 (8)	0.0197 (8)	−0.0010 (8)	0.0009 (8)	−0.0017 (6)
C8	0.0159 (9)	0.0150 (9)	0.0193 (9)	−0.0011 (8)	0.0037 (8)	0.0005 (7)
C9	0.0167 (9)	0.0206 (9)	0.0235 (9)	−0.0015 (8)	−0.0009 (9)	0.0012 (7)
C10	0.0159 (9)	0.0221 (10)	0.0193 (9)	−0.0017 (8)	−0.0032 (8)	0.0023 (8)
C11	0.0191 (9)	0.0207 (10)	0.0214 (9)	−0.0018 (8)	−0.0034 (9)	0.0007 (7)
C12	0.0225 (10)	0.0291 (10)	0.0214 (10)	−0.0021 (8)	−0.0004 (9)	−0.0028 (8)

C13	0.0294 (12)	0.0394 (13)	0.0216 (10)	−0.0062 (12)	0.0026 (11)	0.0036 (9)
C14	0.0376 (12)	0.0279 (11)	0.0287 (11)	−0.0056 (12)	0.0018 (11)	0.0101 (9)
C15	0.0272 (12)	0.0223 (10)	0.0269 (11)	−0.0017 (9)	−0.0018 (9)	0.0041 (9)
Br1	0.02127 (10)	0.03034 (11)	0.01820 (9)	−0.00156 (9)	−0.00057 (9)	0.00275 (8)
N1	0.0196 (9)	0.0146 (7)	0.0224 (8)	0.0003 (7)	0.0014 (8)	0.0011 (6)
O1	0.0394 (10)	0.0225 (8)	0.0403 (10)	0.0128 (8)	0.0027 (9)	0.0045 (7)
O2	0.0460 (12)	0.0182 (8)	0.0434 (11)	0.0034 (8)	0.0027 (10)	−0.0095 (7)

Geometric parameters (Å, º)

C1—O1	1.201 (3)	C9—N1	1.465 (3)
C1—N1	1.368 (3)	C9—C10	1.510 (3)
C1—C2	1.564 (3)	C9—H9A	0.9900
C2—O2	1.208 (3)	C9—H9B	0.9900
C2—C3	1.467 (3)	C10—C11	1.391 (3)
C3—C4	1.388 (3)	C10—C15	1.396 (3)
C3—C8	1.400 (3)	C11—C12	1.388 (3)
C4—C5	1.385 (3)	C11—H11	0.9500
C4—H4	0.9500	C12—C13	1.380 (3)
C5—C6	1.388 (3)	C12—H12	0.9500
C5—Br1	1.898 (2)	C13—C14	1.388 (4)
C6—C7	1.398 (3)	C13—H13	0.9500
C6—H6	0.9500	C14—C15	1.382 (3)
C7—C8	1.381 (3)	C14—H14	0.9500
C7—H7	0.9500	C15—H15	0.9500
C8—N1	1.411 (3)		
O1—C1—N1	127.7 (2)	C10—C9—H9A	109.2
O1—C1—C2	126.5 (2)	N1—C9—H9B	109.2
N1—C1—C2	105.78 (18)	C10—C9—H9B	109.2
O2—C2—C3	131.0 (2)	H9A—C9—H9B	107.9
O2—C2—C1	123.9 (2)	C11—C10—C15	119.1 (2)
C3—C2—C1	105.08 (18)	C11—C10—C9	120.78 (19)
C4—C3—C8	121.17 (19)	C15—C10—C9	120.1 (2)
C4—C3—C2	131.8 (2)	C12—C11—C10	120.4 (2)
C8—C3—C2	107.0 (2)	C12—C11—H11	119.8
C5—C4—C3	117.15 (19)	C10—C11—H11	119.8
C5—C4—H4	121.4	C13—C12—C11	119.9 (2)
C3—C4—H4	121.4	C13—C12—H12	120.0
C4—C5—C6	122.0 (2)	C11—C12—H12	120.0
C4—C5—Br1	119.41 (16)	C12—C13—C14	120.2 (2)
C6—C5—Br1	118.58 (16)	C12—C13—H13	119.9
C5—C6—C7	120.82 (19)	C14—C13—H13	119.9
C5—C6—H6	119.6	C15—C14—C13	120.0 (2)
C7—C6—H6	119.6	C15—C14—H14	120.0
C8—C7—C6	117.44 (19)	C13—C14—H14	120.0
C8—C7—H7	121.3	C14—C15—C10	120.3 (2)
C6—C7—H7	121.3	C14—C15—H15	119.8

C7—C8—C3	121.4 (2)	C10—C15—H15	119.8
C7—C8—N1	127.32 (19)	C1—N1—C8	110.91 (18)
C3—C8—N1	111.23 (18)	C1—N1—C9	123.96 (19)
N1—C9—C10	112.18 (18)	C8—N1—C9	125.09 (17)
N1—C9—H9A	109.2		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1^{i}	0.95	2.46	3.056 (2)	121

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