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ISSN 2414-3146

5-Bromo-1-nonylindoline-2,3-dione

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Received 12 May 2016
Accepted 14 May 2016

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

Keywords: crystal structure; indoline ring; nonyle; hydrogen bonding.

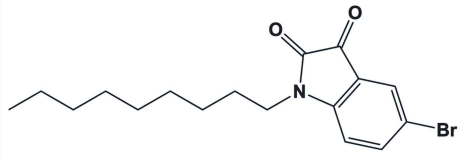
CCDC reference: 1479813

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

In the title compound, $C_{17}H_{22}BrNO_2$, the indoline ring system, the two ketone O atoms and the Br atom are nearly coplanar, with an r.m.s. deviation of 0.029 Å. The indoline ring system makes a dihedral angle of 70.64 (7)° with the mean plane through the nonyl chain, which has an extended conformation. In the crystal, molecules pack in a herringbone arrangement. They are linked by two strong and two weak C—H···O hydrogen bonds, forming slabs parallel to (010).

3D view

Chemical scheme



Structure description

1*H*-Indole-2,3-dione (isatin) is one of the most prevalent heterocyclic scaffolds found in natural products, pharmaceuticals and agrochemicals. Many indole derivatives are under development as drug candidates due to their biological properties, which include anti-viral, antitumor, antifungal, anti-angiogenic, anticonvulsant and antiparkinsonian activity (Sridhar, Muniyandy & Ramesh, 2001; Sridhar & Sreenivasulu, 2001; Sarangapani & Reddy, 1994; Varma *et al.*, 2004; Pandeya *et al.*, 1999; Aboul-Fadl *et al.*, 2010). Continuing our work on the synthesis of new 5-bromoisatins and the study of their applications (Qachchachi *et al.*, 2013, 2014; Kharbach *et al.*, 2016), we report herein on the synthesis and crystal structure of 5-bromo-1-nonylindoline-2,3-dione.

The molecular structure of the title compound is illustrated in Fig. 1. It is composed of an indoline-2,3-dione unit substituted by a Br atom and a nonyl alkyl chain. The indoline ring system and the two ketonic O atoms are virtually coplanar, with an r.m.s. deviation of 0.029 Å; the largest deviation is 0.059 (1) Å for atom C9. The nonyl chain has an extended conformation and its mean plane is nearly perpendicular to the indoline ring system, as indicated by the C10—C9—N1—C8 torsion angle of 89.85 (15)°.

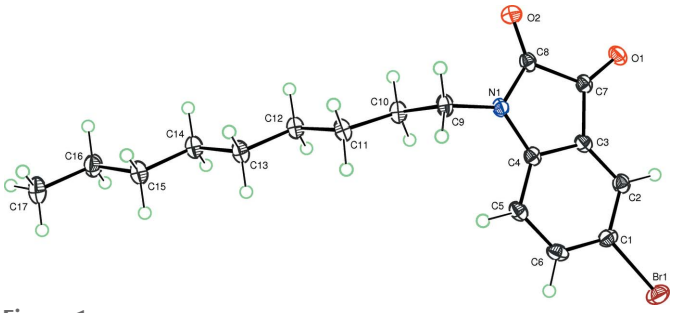


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

In the crystal, molecules pack in a herringbone arrangement. They are linked by two strong and two weak C—H···O hydrogen bonds, forming slabs parallel to the *ac* plane (Table 1 and Fig. 2).

Synthesis and crystallization

A mixture of 5-bromoisatin (0.4 g, 1.76 mmol) and 1-bromononane (0.37 ml, 1.93 mmol) in DMF (25 ml) in the presence of a catalytic amount of tetra-*n*-butylammonium bromide (0.1 g, 0.4 mmol) and potassium carbonate (0.6 g, 4.4 mmol) was stirred for 48 h. The reaction was monitored by thin-layer chromatography. On completion of the reaction, the mixture was filtered and the solvent removed under vacuum. The solid obtained was recrystallized from ethanol to afford the title compound as orange crystals (yield 78%; m.p. 338 K).

Refinement

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

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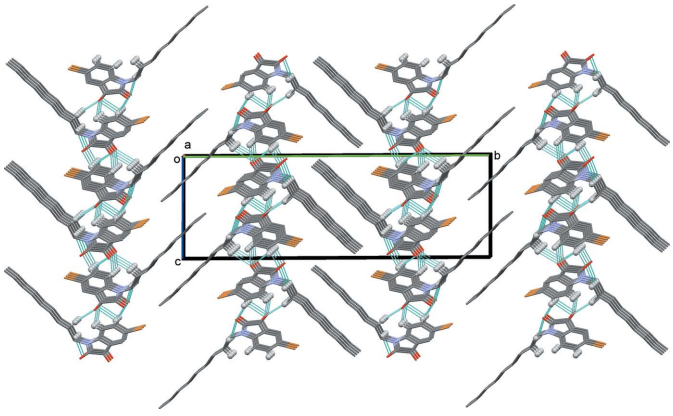

Figure 2
A view along the *a* axis of the crystal packing of the title compound. The C—H···O hydrogen bonds are shown as dashed lines (see Table 1) and, for clarity, only the H atoms (grey balls) involved in these interactions have been included.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.95	2.37	3.3028 (17)	166
C10—H10*B*···O1^{ii}	0.99	2.50	3.4809 (17)	169
C5—H5···O1^{i}	0.95	2.61	3.2431 (17)	124
C9—H9*B*···O2^{iii}	0.99	2.66	3.2544 (17)	120

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{22}BrNO_2$
M_r	352.26
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, *b*, *c* (Å)	4.8428 (2), 31.7181 (14), 10.7171 (5)
β (°)	101.206 (2)
V (Å^3)	1614.81 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.55
Crystal size (mm)	0.20 × 0.17 × 0.07
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.658, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	33463, 4532, 4029
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.694
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.026, 0.061, 1.10
No. of reflections	4532
No. of parameters	191
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.44, −0.43

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

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full crystallographic data

IUCrData (2016). **1**, x160791 [doi:10.1107/S2414314616007914]

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Crystal data

$C_{17}H_{22}BrNO_2$
M_r = 352.26
Monoclinic, $P2_1/c$
a = 4.8428 (2) Å
b = 31.7181 (14) Å
c = 10.7171 (5) Å
β = 101.206 (2)°
V = 1614.81 (12) Å^3
Z = 4
$F(000)$ = 728
D_x = 1.449 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4532 reflections
θ = 1.3–29.6°
μ = 2.55 mm^{-1}
T = 100 K
Plate, orange
0.20 × 0.17 × 0.07 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.658, T_{max} = 0.746
33463 measured reflections
4532 independent reflections
4029 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 29.6°, θ_{min} = 1.3°
h = −6→5
k = −44→44
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.061
S = 1.10
4532 reflections
191 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0246P)^2 + 0.8548P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 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	0.46011 (3)	0.37424 (2)	0.85103 (2)	0.02240 (5)
C1	0.5594 (3)	0.31889 (4)	0.81017 (12)	0.0151 (3)
C2	0.7367 (3)	0.31325 (4)	0.72398 (13)	0.0144 (2)
H2	0.8106	0.3365	0.6853	0.017*
C3	0.8006 (3)	0.27209 (4)	0.69712 (12)	0.0130 (2)
C4	0.6918 (3)	0.23768 (4)	0.75382 (12)	0.0126 (2)
C5	0.5180 (3)	0.24367 (4)	0.84074 (12)	0.0146 (3)
H5	0.4456	0.2204	0.8801	0.017*
C6	0.4529 (3)	0.28509 (5)	0.86841 (12)	0.0157 (3)
H6	0.3344	0.2902	0.9278	0.019*
C7	0.9820 (3)	0.25508 (4)	0.61420 (12)	0.0125 (2)
C8	0.9634 (3)	0.20624 (4)	0.62788 (12)	0.0136 (2)
C9	0.6994 (3)	0.15753 (4)	0.74596 (13)	0.0151 (3)
H9A	0.6938	0.1381	0.6732	0.018*
H9B	0.5066	0.1593	0.7637	0.018*
C10	0.8951 (3)	0.13925 (4)	0.86226 (13)	0.0160 (3)
H10B	0.9539	0.1619	0.9252	0.019*
H10A	1.0661	0.1279	0.8366	0.019*
C11	0.7510 (3)	0.10416 (4)	0.92355 (13)	0.0163 (3)
H11B	0.5986	0.1166	0.9613	0.020*
H11A	0.6637	0.0841	0.8567	0.020*
C12	0.9512 (3)	0.08012 (4)	1.02647 (13)	0.0164 (3)
H12A	1.0914	0.0650	0.9871	0.020*
H12B	1.0541	0.1005	1.0885	0.020*
C13	0.8004 (3)	0.04848 (5)	1.09660 (13)	0.0178 (3)
H13B	0.6669	0.0639	1.1392	0.021*
H13A	0.6892	0.0292	1.0336	0.021*
C14	0.9960 (3)	0.02245 (4)	1.19543 (13)	0.0178 (3)
H14B	1.1101	0.0417	1.2577	0.021*
H14A	1.1266	0.0064	1.1528	0.021*
C15	0.8397 (3)	−0.00833 (5)	1.26640 (14)	0.0186 (3)
H15B	0.7163	0.0079	1.3124	0.022*
H15A	0.7176	−0.0265	1.2035	0.022*
C16	1.0312 (3)	−0.03633 (5)	1.36108 (14)	0.0200 (3)
H16A	1.1499	−0.0184	1.4257	0.024*
H16B	1.1576	−0.0523	1.3159	0.024*
C17	0.8684 (4)	−0.06729 (5)	1.42776 (16)	0.0252 (3)
H17B	0.7418	−0.0517	1.4722	0.038*
H17C	1.0007	−0.0839	1.4893	0.038*
H17A	0.7579	−0.0861	1.3647	0.038*
N1	0.7852 (2)	0.19929 (4)	0.71043 (11)	0.0134 (2)
O1	1.1213 (2)	0.27258 (3)	0.54746 (9)	0.01603 (19)
O2	1.0843 (2)	0.18008 (3)	0.57614 (10)	0.0181 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02663 (9)	0.01885 (8)	0.02210 (8)	0.00614 (6)	0.00564 (6)	−0.00365 (5)
C1	0.0154 (6)	0.0157 (6)	0.0134 (6)	0.0036 (5)	0.0008 (5)	−0.0017 (5)
C2	0.0131 (6)	0.0156 (6)	0.0141 (6)	0.0004 (5)	0.0018 (5)	0.0022 (5)
C3	0.0107 (6)	0.0165 (6)	0.0119 (5)	0.0007 (5)	0.0023 (5)	0.0020 (5)
C4	0.0103 (6)	0.0153 (6)	0.0112 (5)	0.0008 (5)	−0.0003 (4)	0.0018 (4)
C5	0.0114 (6)	0.0198 (6)	0.0124 (6)	−0.0004 (5)	0.0020 (5)	0.0032 (5)
C6	0.0124 (6)	0.0239 (7)	0.0110 (6)	0.0021 (5)	0.0022 (5)	−0.0008 (5)
C7	0.0104 (6)	0.0154 (6)	0.0110 (5)	0.0011 (5)	−0.0001 (4)	0.0023 (4)
C8	0.0125 (6)	0.0158 (6)	0.0121 (6)	0.0004 (5)	0.0011 (5)	0.0029 (5)
C9	0.0153 (6)	0.0134 (6)	0.0161 (6)	−0.0028 (5)	0.0021 (5)	0.0029 (5)
C10	0.0157 (6)	0.0150 (6)	0.0163 (6)	−0.0017 (5)	0.0008 (5)	0.0031 (5)
C11	0.0159 (6)	0.0176 (6)	0.0149 (6)	−0.0025 (5)	0.0018 (5)	0.0038 (5)
C12	0.0171 (7)	0.0157 (6)	0.0159 (6)	−0.0018 (5)	0.0015 (5)	0.0028 (5)
C13	0.0181 (7)	0.0182 (6)	0.0170 (6)	−0.0009 (5)	0.0029 (5)	0.0053 (5)
C14	0.0186 (7)	0.0172 (6)	0.0169 (6)	−0.0012 (5)	0.0019 (5)	0.0039 (5)
C15	0.0198 (7)	0.0178 (6)	0.0177 (6)	−0.0018 (5)	0.0023 (5)	0.0046 (5)
C16	0.0213 (7)	0.0171 (7)	0.0214 (7)	0.0016 (5)	0.0036 (6)	0.0050 (5)
C17	0.0296 (8)	0.0197 (7)	0.0260 (8)	−0.0007 (6)	0.0049 (6)	0.0089 (6)
N1	0.0137 (5)	0.0131 (5)	0.0139 (5)	0.0005 (4)	0.0042 (4)	0.0028 (4)
O1	0.0152 (5)	0.0199 (5)	0.0140 (4)	0.0007 (4)	0.0053 (4)	0.0039 (4)
O2	0.0194 (5)	0.0179 (5)	0.0180 (5)	0.0034 (4)	0.0062 (4)	0.0000 (4)

Geometric parameters (Å, º)

Br1—C1	1.8936 (13)	C10—H10A	0.9900
C1—C6	1.389 (2)	C11—C12	1.5238 (19)
C1—C2	1.3898 (19)	C11—H11B	0.9900
C2—C3	1.3847 (18)	C11—H11A	0.9900
C2—H2	0.9500	C12—C13	1.5229 (19)
C3—C4	1.4009 (18)	C12—H12A	0.9900
C3—C7	1.4684 (18)	C12—H12B	0.9900
C4—C5	1.3845 (19)	C13—C14	1.5204 (19)
C4—N1	1.4092 (17)	C13—H13B	0.9900
C5—C6	1.396 (2)	C13—H13A	0.9900
C5—H5	0.9500	C14—C15	1.526 (2)
C6—H6	0.9500	C14—H14B	0.9900
C7—O1	1.2093 (16)	C14—H14A	0.9900
C7—C8	1.5602 (19)	C15—C16	1.5209 (19)
C8—O2	1.2102 (17)	C15—H15B	0.9900
C8—N1	1.3687 (17)	C15—H15A	0.9900
C9—N1	1.4606 (17)	C16—C17	1.522 (2)
C9—C10	1.5261 (18)	C16—H16A	0.9900
C9—H9A	0.9900	C16—H16B	0.9900
C9—H9B	0.9900	C17—H17B	0.9800
C10—C11	1.5281 (19)	C17—H17C	0.9800

C10—H10B	0.9900	C17—H17A	0.9800
C6—C1—C2	122.04 (13)	H11B—C11—H11A	107.7
C6—C1—Br1	118.59 (10)	C13—C12—C11	112.84 (12)
C2—C1—Br1	119.37 (10)	C13—C12—H12A	109.0
C3—C2—C1	116.84 (12)	C11—C12—H12A	109.0
C3—C2—H2	121.6	C13—C12—H12B	109.0
C1—C2—H2	121.6	C11—C12—H12B	109.0
C2—C3—C4	121.75 (12)	H12A—C12—H12B	107.8
C2—C3—C7	131.03 (12)	C14—C13—C12	114.08 (12)
C4—C3—C7	107.21 (11)	C14—C13—H13B	108.7
C5—C4—C3	120.93 (13)	C12—C13—H13B	108.7
C5—C4—N1	128.07 (12)	C14—C13—H13A	108.7
C3—C4—N1	110.99 (11)	C12—C13—H13A	108.7
C4—C5—C6	117.64 (12)	H13B—C13—H13A	107.6
C4—C5—H5	121.2	C13—C14—C15	113.10 (12)
C6—C5—H5	121.2	C13—C14—H14B	109.0
C1—C6—C5	120.79 (12)	C15—C14—H14B	109.0
C1—C6—H6	119.6	C13—C14—H14A	109.0
C5—C6—H6	119.6	C15—C14—H14A	109.0
O1—C7—C3	131.11 (13)	H14B—C14—H14A	107.8
O1—C7—C8	124.07 (12)	C16—C15—C14	114.14 (12)
C3—C7—C8	104.82 (11)	C16—C15—H15B	108.7
O2—C8—N1	127.40 (13)	C14—C15—H15B	108.7
O2—C8—C7	126.58 (12)	C16—C15—H15A	108.7
N1—C8—C7	106.02 (11)	C14—C15—H15A	108.7
N1—C9—C10	113.23 (11)	H15B—C15—H15A	107.6
N1—C9—H9A	108.9	C15—C16—C17	112.69 (13)
C10—C9—H9A	108.9	C15—C16—H16A	109.1
N1—C9—H9B	108.9	C17—C16—H16A	109.1
C10—C9—H9B	108.9	C15—C16—H16B	109.1
H9A—C9—H9B	107.7	C17—C16—H16B	109.1
C9—C10—C11	111.40 (11)	H16A—C16—H16B	107.8
C9—C10—H10B	109.3	C16—C17—H17B	109.5
C11—C10—H10B	109.3	C16—C17—H17C	109.5
C9—C10—H10A	109.3	H17B—C17—H17C	109.5
C11—C10—H10A	109.3	C16—C17—H17A	109.5
H10B—C10—H10A	108.0	H17B—C17—H17A	109.5
C12—C11—C10	113.34 (11)	H17C—C17—H17A	109.5
C12—C11—H11B	108.9	C8—N1—C4	110.93 (11)
C10—C11—H11B	108.9	C8—N1—C9	124.18 (11)
C12—C11—H11A	108.9	C4—N1—C9	124.88 (11)
C10—C11—H11A	108.9		
C6—C1—C2—C3	0.8 (2)	O1—C7—C8—N1	−179.64 (12)
Br1—C1—C2—C3	−179.59 (10)	C3—C7—C8—N1	0.39 (13)
C1—C2—C3—C4	0.07 (19)	N1—C9—C10—C11	160.28 (12)
C1—C2—C3—C7	−178.64 (13)	C9—C10—C11—C12	170.64 (12)

C2—C3—C4—C5	−0.9 (2)	C10—C11—C12—C13	173.80 (12)
C7—C3—C4—C5	178.12 (12)	C11—C12—C13—C14	177.11 (12)
C2—C3—C4—N1	179.48 (12)	C12—C13—C14—C15	178.74 (12)
C7—C3—C4—N1	−1.54 (14)	C13—C14—C15—C16	177.04 (12)
C3—C4—C5—C6	0.75 (19)	C14—C15—C16—C17	−178.65 (13)
N1—C4—C5—C6	−179.65 (12)	O2—C8—N1—C4	178.27 (13)
C2—C1—C6—C5	−0.9 (2)	C7—C8—N1—C4	−1.34 (14)
Br1—C1—C6—C5	179.48 (10)	O2—C8—N1—C9	−3.0 (2)
C4—C5—C6—C1	0.10 (19)	C7—C8—N1—C9	177.35 (11)
C2—C3—C7—O1	−0.4 (2)	C5—C4—N1—C8	−177.75 (13)
C4—C3—C7—O1	−179.28 (14)	C3—C4—N1—C8	1.88 (15)
C2—C3—C7—C8	179.53 (13)	C5—C4—N1—C9	3.6 (2)
C4—C3—C7—C8	0.68 (13)	C3—C4—N1—C9	−176.80 (12)
O1—C7—C8—O2	0.7 (2)	C10—C9—N1—C8	89.85 (15)
C3—C7—C8—O2	−179.23 (13)	C10—C9—N1—C4	−91.64 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.95	2.37	3.3028 (17)	166
C10—H10*B*···O1^{ii}	0.99	2.50	3.4809 (17)	169
C5—H5···O1^{i}	0.95	2.61	3.2431 (17)	124
C9—H9*B*···O2^{iii}	0.99	2.66	3.2544 (17)	120

Symmetry codes: (i) $x-1, -y+1/2, z+1/2$; (ii) $x, -y+1/2, z+1/2$; (iii) $x-1, y, z$.