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

Yassine Kharbach,[a]* Amal Haoudi,[a] Frédéric Capet,[b] Ahmed Mazzah[c] and Lahcen El Ammari[d]

[a]Laboratoire de Chimie Organique Appliquée-Chimie Appliquée, Faculté des Sciences et Techniques, Université Sidi Mohamed Ben Abdallah, Fès, Morocco, [b]Unité de Catalyse et de Chimie du Solide (UCCS), UMR 8181, Ecole Nationale Supérieure de Chimie de Lille, Université Lille 1, 59650 Villeneuve d'Ascq Cedex, France, [c]USR 3290 Miniaturisation pour l'analyse, la synthèse et la protéomique, 59655 Villeneuve d'Ascq Cedex, Université Lille1, France, and [d]Laboratoire de Chimie du Solide Appliquée, Faculty of Sciences, Mohammed V University in Rabat, Avenue Ibn Battouta, BP 1014, Rabat, Morocco. *Correspondence e-mail: kharbachy26@gmail.com

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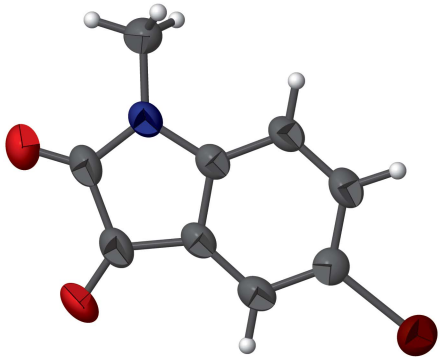
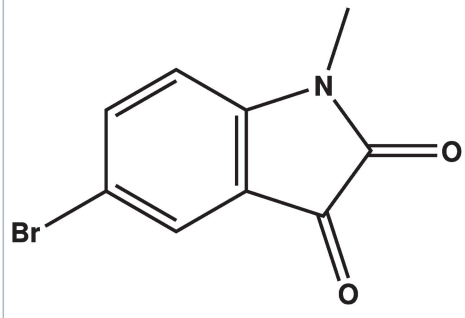
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_9H_6BrNO_2$, the indoline ring system, the two ketone O atoms and the Br atom are nearly coplanar, with the largest deviation from the mean plane being −0.1025 (4) Å. In the crystal, molecules are linked by two weak C—H···O hydrogen bonds and π–π interactions [inter-centroid distance = 3.510 (2) Å], forming a three-dimensional structure.

3D view



Chemical scheme



Structure description

Isatin derivatives have a wide range of biological properties. They display moderate antimicrobial effects in a wide variety of preclinical antimicrobial models. Isatin also exhibits other biological activities, such as anticonvulsant, cytotoxic, antifungal *etc*. Isatin and its analogs are versatile substrates, which can be used for the synthesis of numerous heterocyclic compounds (Sridhar *et al.*, 2001; Sridhar & Sreenivasulu, 2001; Sarangapani & Reddy, 1994; Varma *et al.*, 2004; Pandeya *et al.*, 1999; Aboul-Fadl *et al.*, 2010). In our work, we are interested in developing a new 5-bromoisatin and continuing the research work of Qachchachi to explore other applications (Qachchachi *et al.*, 2013, 2014; Kharbach *et al.*, 2016). The present paper reports the synthesis and crystal structure of 5-bromo-1-methylindoline-2,3-dione (see Scheme).

The title compound is built up from two fused five- and six-membered rings linked to two ketone O atoms, a Br atom and a methyl group, as shown in Fig. 1. Besides the methyl H atoms, all the atoms of the structure are almost coplanar, with a maximum deviation of −0.1025 (4) Å for the Br1 atom.

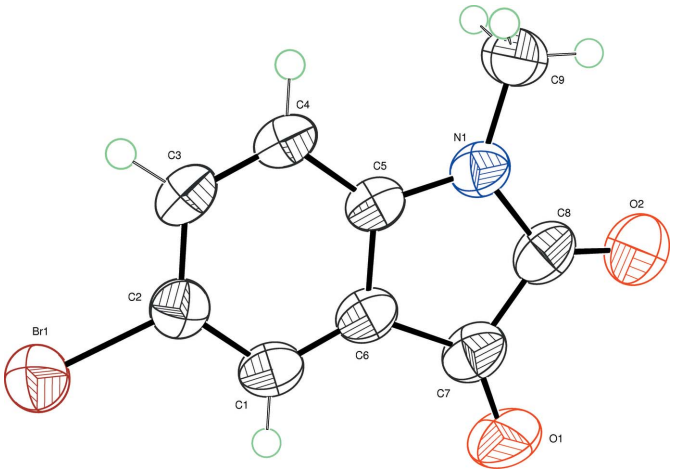


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

In the crystal, molecules are linked by two weak C—H···O hydrogen bonds (Table 1) and π–π interactions [inter-centroid distance = 3.510 (2) Å], forming a three-dimensional network as shown in Fig. 2.

Synthesis and crystallization

A mixture of 5-bromoisatin (0.4 g, 1.76 mmol) and iodomethane (0.12 ml, 0.84 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 title compound was obtained in 69% yield (m.p. 446 K). The solid obtained was recrystallized from ethanol to afford the title compound as orange crystals suitable for the X-ray diffraction.

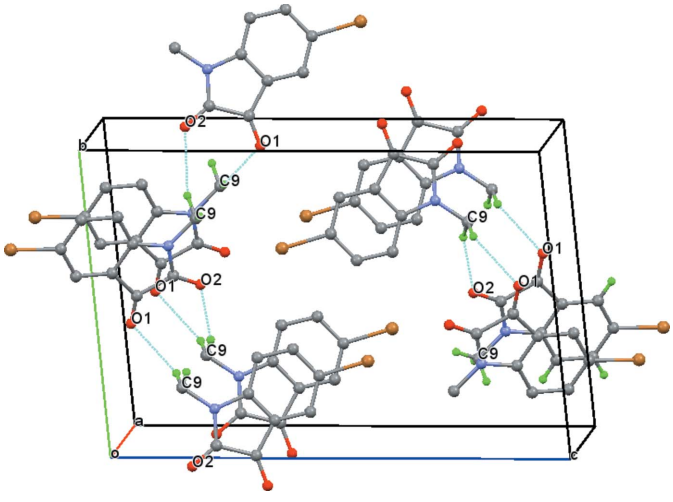


Figure 2
Molecules of the title compound linked by C—H···O hydrogen bonds and π–π interactions, forming a three-dimensionnal network.

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O2^{i}	0.96	2.56	3.454 (5)	155
C9—H9C···O1^{ii}	0.96	2.61	3.403 (5)	141

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_6BrNO_2$
M_r	240.06
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	4.0634 (1), 11.9235 (3), 18.0978 (5)
β (°)	96.170 (2)
V (Å^3)	871.76 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	4.68
Crystal size (mm)	0.57 × 0.22 × 0.03
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.452, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9696, 2022, 1606
R_{int}	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.102, 1.07
No. of reflections	2022
No. of parameters	119
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.34

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

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The reflections 011 and 002 were affected by the beam-stop and were removed during the refinement.

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full crystallographic data

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Crystal data

$C_9H_6BrNO_2$
M_r = 240.06
Monoclinic, $P2_1/n$
a = 4.0634 (1) Å
b = 11.9235 (3) Å
c = 18.0978 (5) Å
β = 96.170 (2)°
V = 871.76 (4) Å^3
Z = 4
$F(000)$ = 472
D_x = 1.829 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2022 reflections
θ = 3.4–27.9°
μ = 4.68 mm^{-1}
T = 296 K
Sheet, orange
0.57 × 0.22 × 0.03 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.452, T_{max} = 0.746
9696 measured reflections
2022 independent reflections
1606 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.9°, θ_{min} = 3.4°
h = −5→5
k = −15→15
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.102
S = 1.07
2022 reflections
119 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.9585P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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}
C1	0.8560 (9)	0.3090 (2)	0.90400 (19)	0.0403 (7)
C2	0.8118 (8)	0.2178 (3)	0.85539 (18)	0.0370 (7)
C3	0.9557 (8)	0.1155 (3)	0.87379 (19)	0.0421 (7)
H3	0.9308	0.0550	0.8413	0.050*
C4	1.1383 (8)	0.1065 (3)	0.9425 (2)	0.0436 (8)
H4	1.2377	0.0385	0.9565	0.052*
C5	1.1765 (8)	0.1971 (3)	0.99091 (19)	0.0409 (7)
C6	1.0369 (9)	0.3001 (3)	0.9723 (2)	0.0433 (8)
H6	1.0640	0.3608	1.0047	0.052*
C7	0.6624 (10)	0.4026 (3)	0.8673 (2)	0.0494 (8)
C8	0.5016 (10)	0.3540 (3)	0.7928 (2)	0.0484 (8)
C9	0.5091 (11)	0.1663 (3)	0.7309 (2)	0.0526 (9)
H9A	0.3644	0.2029	0.6929	0.079*
H9B	0.3959	0.1041	0.7504	0.079*
H9C	0.7023	0.1397	0.7101	0.079*
N1	0.6078 (7)	0.2458 (2)	0.79063 (16)	0.0424 (6)
O1	0.6250 (8)	0.4971 (2)	0.88782 (17)	0.0689 (8)
O2	0.3142 (8)	0.4020 (2)	0.74695 (17)	0.0685 (8)
Br1	1.41762 (10)	0.17426 (3)	1.08562 (2)	0.05405 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0440 (18)	0.0291 (15)	0.0493 (19)	−0.0038 (13)	0.0112 (15)	0.0013 (13)
C2	0.0377 (16)	0.0318 (15)	0.0431 (17)	−0.0029 (13)	0.0120 (14)	0.0021 (13)
C3	0.0481 (19)	0.0330 (16)	0.0465 (18)	0.0046 (14)	0.0117 (15)	−0.0030 (14)
C4	0.0449 (18)	0.0351 (17)	0.0526 (19)	0.0081 (13)	0.0134 (15)	0.0032 (15)
C5	0.0361 (17)	0.0450 (18)	0.0426 (17)	0.0005 (13)	0.0091 (14)	0.0010 (14)
C6	0.0465 (19)	0.0341 (16)	0.0504 (19)	−0.0047 (14)	0.0108 (16)	−0.0035 (14)
C7	0.061 (2)	0.0324 (17)	0.056 (2)	0.0008 (15)	0.0117 (18)	0.0074 (15)
C8	0.056 (2)	0.0343 (17)	0.056 (2)	0.0037 (15)	0.0116 (18)	0.0101 (15)
C9	0.065 (2)	0.045 (2)	0.0461 (19)	−0.0019 (17)	0.0011 (18)	−0.0021 (16)
N1	0.0478 (16)	0.0323 (14)	0.0473 (16)	−0.0017 (11)	0.0060 (13)	0.0020 (12)
O1	0.103 (2)	0.0291 (13)	0.0731 (18)	0.0102 (13)	0.0036 (16)	−0.0032 (13)
O2	0.084 (2)	0.0502 (16)	0.0679 (18)	0.0132 (15)	−0.0051 (16)	0.0111 (14)
Br1	0.0514 (3)	0.0586 (3)	0.0511 (2)	0.00589 (17)	0.00106 (17)	−0.00134 (17)

Geometric parameters (Å, º)

C1—C6	1.373 (5)	C5—Br1	1.900 (4)
C1—C2	1.398 (5)	C6—H6	0.9300
C1—C7	1.480 (5)	C7—O1	1.202 (4)
C2—C3	1.378 (5)	C7—C8	1.546 (6)
C2—N1	1.401 (5)	C8—O2	1.208 (5)
C3—C4	1.381 (5)	C8—N1	1.363 (4)

C3—H3	0.9300	C9—N1	1.461 (5)
C4—C5	1.389 (5)	C9—H9A	0.9600
C4—H4	0.9300	C9—H9B	0.9600
C5—C6	1.379 (5)	C9—H9C	0.9600
C6—C1—C2	121.8 (3)	C5—C6—H6	121.5
C6—C1—C7	132.0 (3)	O1—C7—C1	130.3 (4)
C2—C1—C7	106.1 (3)	O1—C7—C8	124.4 (3)
C3—C2—C1	120.9 (3)	C1—C7—C8	105.3 (3)
C3—C2—N1	127.6 (3)	O2—C8—N1	127.4 (4)
C1—C2—N1	111.5 (3)	O2—C8—C7	126.7 (3)
C2—C3—C4	117.3 (3)	N1—C8—C7	105.9 (3)
C2—C3—H3	121.3	N1—C9—H9A	109.5
C4—C3—H3	121.3	N1—C9—H9B	109.5
C3—C4—C5	121.4 (3)	H9A—C9—H9B	109.5
C3—C4—H4	119.3	N1—C9—H9C	109.5
C5—C4—H4	119.3	H9A—C9—H9C	109.5
C6—C5—C4	121.6 (3)	H9B—C9—H9C	109.5
C6—C5—Br1	120.4 (3)	C8—N1—C2	111.2 (3)
C4—C5—Br1	118.0 (3)	C8—N1—C9	124.9 (3)
C1—C6—C5	117.0 (3)	C2—N1—C9	123.8 (3)
C1—C6—H6	121.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*B*···O2^{i}	0.96	2.56	3.454 (5)	155
C9—H9*C*···O1^{ii}	0.96	2.61	3.403 (5)	141

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