Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

Crystal structure of 2-(5-bromo-2-hydroxybenzylidene)-2,3-dihydro-1*H*-indene-1,3-dione

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Received 9 April 2015; accepted 15 April 2015

Edited by M. Weil, Vienna University of Technology, Austria

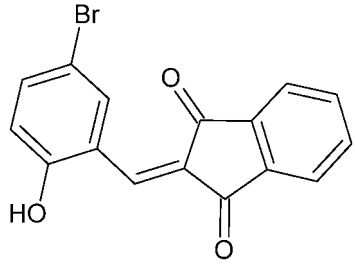
The title molecule, $C_{16}H_9BrO_3$, deviates slightly from planarity. The benzene ring makes a dihedral angle of 1.02 (9)° with the plane defined by the five-membered ring of the indandione moiety. The latter exhibits a minute twist indicated by the dihedral angle of 0.47 (9)° between the planes of the five- and six-membered rings. An intramolecular C—H···O hydrogen bond between the attached benzene ring with one of the indandione carbonyl O atoms stabilizes the molecular conformation. In the crystal, the molecules form dimers across centres of inversion *via* pairwise O—H···O hydrogen bonds. The dimers form stacks running parallel to [010] and interact through π–π interactions between the five-membered ring of one molecule and the six-membered rings of the indandione moiety of an adjacent molecule [centroid-to-centroid distance = 3.5454 (10) Å].

Keywords: crystal structure; 3-substituted indan-1,3-diones; hydrogen bonding; π–π interactions.

CCDC reference: 1059869

1. Related literature

Indan-1,3-dione and its analogues are synthons for building highly interesting compounds with a wide range of applications in both pharmaceutical and industrial chemistry (Kuhn & Rae, 1971; Junek & Sterk, 1968; Kunz & Polansky, 1969; Aldersley *et al.*, 1983). For chemical reactions and bio-activities of 3-substituted indan-1,3-diones, see: Hochrainer & Wessely (1966); Zargar & Khan (2015).



2. Experimental

2.1. Crystal data

$C_{16}H_9BrO_3$
M_r = 329.14
Monoclinic, $P2_1/c$
a = 13.8820 (4) Å
b = 3.8695 (1) Å
c = 24.0068 (5) Å
β = 102.483 (1)°
V = 1259.07 (6) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 4.50 mm^{-1}
T = 150 K
0.22 × 0.07 × 0.04 mm

2.2. Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Absorption correction: numerical (*SADABS*; Bruker, 2014)
T_{min} = 0.67, T_{max} = 0.84
8943 measured reflections
2510 independent reflections
2386 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.056
S = 1.09
2510 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O3	0.95	2.15	2.994 (2)	148
O1—H1···O2^{i}	0.84	1.83	2.6641 (16)	173

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

The support of NSF–MRI grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5145).

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supporting information

Acta Cryst. (2015). E**71**, o324–o325 [doi:10.1107/S2056989015007434]

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S1. Experimental

A mixture of 1 mmol (146 mg) of 1*H*-indene-1,3(2*H*)-dione and 1 mmol (201 mg) of 5-bromo-2-hydroxybenzaldehyde in 30 ml ethanol was refluxed for 30 min. The resulting solid product was collected under vacuum and re-crystallized from ethanol to afford yellow needles suitable for X-ray diffraction in 83% yield.

S2. Refinement

H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while that attached to oxygen was placed in a location derived from a difference map and its coordinates adjusted to give a distance O—H = 0.84 Å. All H atoms were included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

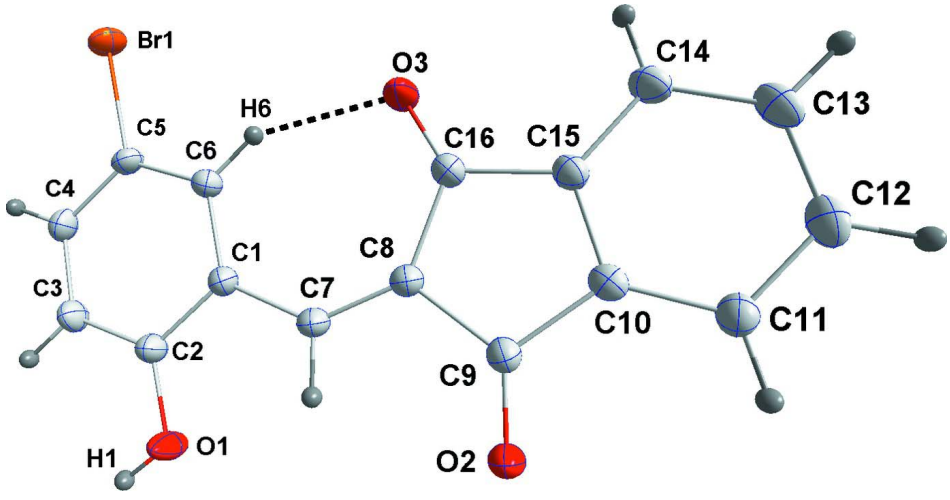

Figure 1

The title molecule with labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular C—H···O interaction is shown as a dotted line.

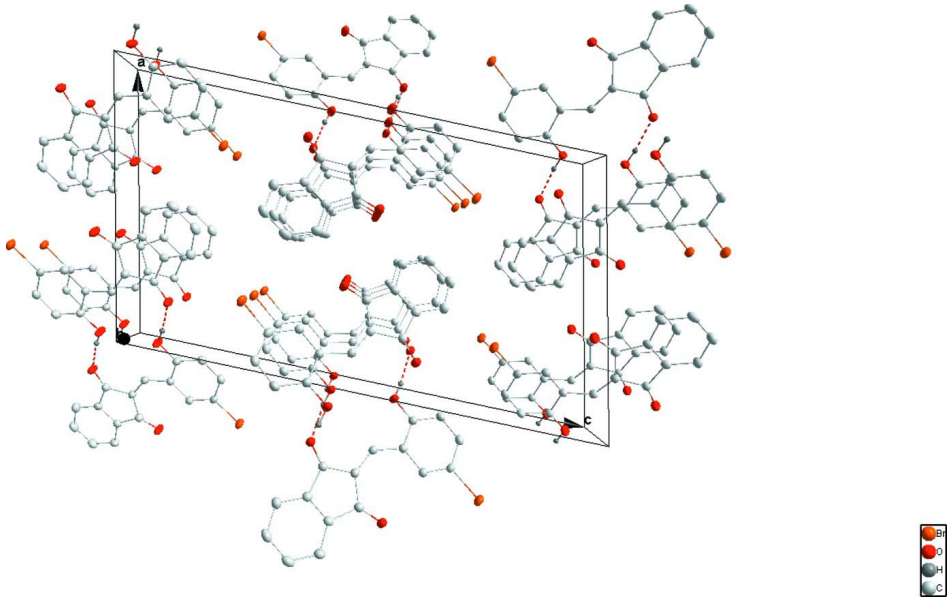

Figure 2

Crystal packing of the title compound viewed down [010]. Intermolecular O—H···O hydrogen bonds are shown as dotted lines.

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Crystal data

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$\beta = 102.483$ (1)°
$V = 1259.07$ (6) Å^3
$Z = 4$
$F(000) = 656$
$D_x = 1.736$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 7789 reflections
$\theta = 3.3$–74.5°
$\mu = 4.50$ mm^{-1}
$T = 150$ K
Needle, yellow
0.22 × 0.07 × 0.04 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: numerical (*SADABS*; Bruker, 2014)
$T_{min} = 0.67$, $T_{max} = 0.84$
8943 measured reflections
2510 independent reflections
2386 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 74.5°$, $\theta_{min} = 3.3°$
$h = -16\rightarrow17$
$k = -4\rightarrow4$
$l = -29\rightarrow29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.056$
$S = 1.09$
2510 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 0.6412P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while that attached to oxygen was placed in a location derived from a difference map and its coordinates adjusted to give O—H = 0.84 Å. All were included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.74761 (2)	0.32460 (5)	0.72665 (2)	0.02597 (8)
O1	1.00458 (9)	−0.0525 (4)	0.56417 (5)	0.0324 (3)
H1	1.0561	−0.1618	0.5789	0.039*
O2	0.82877 (9)	0.4018 (4)	0.39794 (5)	0.0275 (3)
O3	0.63542 (9)	0.6391 (4)	0.53435 (5)	0.0331 (3)
C1	0.85867 (12)	0.2074 (4)	0.57969 (6)	0.0188 (3)
C2	0.94845 (11)	0.0308 (4)	0.60120 (6)	0.0216 (3)
C3	0.97616 (11)	−0.0550 (4)	0.65901 (6)	0.0227 (3)
H3	1.0370	−0.1702	0.6731	0.027*
C4	0.91553 (12)	0.0270 (4)	0.69553 (6)	0.0215 (3)
H4	0.9339	−0.0334	0.7348	0.026*
C5	0.82703 (12)	0.1991 (4)	0.67450 (6)	0.0195 (3)
C6	0.79773 (12)	0.2902 (4)	0.61786 (6)	0.0195 (3)
H6	0.7370	0.4079	0.6046	0.023*
C7	0.83772 (12)	0.2944 (4)	0.51956 (6)	0.0196 (3)
H7	0.8883	0.2221	0.5011	0.024*
C8	0.76223 (11)	0.4559 (4)	0.48379 (6)	0.0193 (3)
C9	0.76467 (11)	0.4982 (4)	0.42218 (6)	0.0195 (3)
C10	0.67288 (11)	0.6780 (4)	0.39402 (7)	0.0191 (3)
C11	0.64124 (13)	0.7720 (4)	0.33723 (7)	0.0235 (3)
H11	0.6801	0.7253	0.3100	0.028*
C12	0.55046 (13)	0.9372 (4)	0.32177 (7)	0.0262 (3)
H12	0.5268	1.0061	0.2833	0.031*
C13	0.49331 (12)	1.0039 (5)	0.36193 (7)	0.0269 (3)
H13	0.4315	1.1172	0.3502	0.032*
C14	0.52519 (12)	0.9075 (5)	0.41869 (7)	0.0243 (3)
H14	0.4862	0.9515	0.4459	0.029*
C15	0.61626 (12)	0.7441 (4)	0.43405 (7)	0.0202 (3)

C16	0.66754 (12)	0.6133 (4)	0.49114 (7)	0.0216 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03096 (11)	0.02856 (12)	0.02164 (10)	0.00308 (7)	0.01281 (7)	−0.00170 (6)
O1	0.0248 (6)	0.0525 (8)	0.0217 (5)	0.0200 (6)	0.0089 (4)	0.0075 (6)
O2	0.0239 (6)	0.0394 (7)	0.0207 (5)	0.0109 (5)	0.0084 (4)	0.0042 (5)
O3	0.0278 (6)	0.0513 (8)	0.0223 (6)	0.0172 (6)	0.0102 (5)	0.0053 (5)
C1	0.0181 (7)	0.0208 (8)	0.0175 (7)	0.0013 (6)	0.0038 (6)	−0.0007 (6)
C2	0.0193 (7)	0.0260 (8)	0.0200 (7)	0.0031 (7)	0.0057 (6)	0.0000 (6)
C3	0.0193 (7)	0.0266 (8)	0.0208 (7)	0.0037 (7)	0.0012 (6)	0.0023 (6)
C4	0.0236 (7)	0.0225 (8)	0.0172 (6)	−0.0010 (7)	0.0019 (6)	0.0004 (6)
C5	0.0206 (7)	0.0203 (8)	0.0193 (7)	−0.0011 (6)	0.0079 (6)	−0.0030 (6)
C6	0.0186 (7)	0.0201 (8)	0.0199 (7)	0.0023 (6)	0.0042 (6)	−0.0011 (6)
C7	0.0193 (7)	0.0217 (8)	0.0189 (7)	0.0021 (6)	0.0062 (6)	−0.0013 (6)
C8	0.0190 (7)	0.0208 (7)	0.0184 (7)	0.0014 (6)	0.0049 (5)	−0.0006 (6)
C9	0.0190 (7)	0.0201 (7)	0.0189 (7)	0.0011 (6)	0.0032 (5)	0.0006 (6)
C10	0.0181 (7)	0.0173 (7)	0.0215 (7)	−0.0004 (6)	0.0033 (6)	−0.0010 (5)
C11	0.0262 (8)	0.0230 (8)	0.0207 (7)	0.0017 (7)	0.0039 (6)	0.0016 (6)
C12	0.0299 (8)	0.0222 (8)	0.0224 (7)	0.0013 (7)	−0.0033 (6)	0.0022 (6)
C13	0.0218 (8)	0.0230 (8)	0.0317 (8)	0.0043 (7)	−0.0032 (6)	−0.0005 (7)
C14	0.0191 (7)	0.0259 (8)	0.0271 (8)	0.0039 (7)	0.0031 (6)	−0.0019 (7)
C15	0.0181 (7)	0.0203 (7)	0.0211 (7)	0.0010 (6)	0.0019 (6)	−0.0010 (6)
C16	0.0196 (7)	0.0238 (8)	0.0211 (7)	0.0040 (6)	0.0037 (6)	0.0005 (6)

Geometric parameters (Å, º)

Br1—C5	1.9014 (15)	C7—H7	0.9500
O1—C2	1.3418 (19)	C8—C16	1.493 (2)
O1—H1	0.8400	C8—C9	1.4956 (19)
O2—C9	1.2221 (19)	C9—C10	1.481 (2)
O3—C16	1.218 (2)	C10—C11	1.387 (2)
C1—C6	1.412 (2)	C10—C15	1.390 (2)
C1—C2	1.417 (2)	C11—C12	1.390 (2)
C1—C7	1.449 (2)	C11—H11	0.9500
C2—C3	1.398 (2)	C12—C13	1.399 (3)
C3—C4	1.377 (2)	C12—H12	0.9500
C3—H3	0.9500	C13—C14	1.390 (2)
C4—C5	1.393 (2)	C13—H13	0.9500
C4—H4	0.9500	C14—C15	1.390 (2)
C5—C6	1.378 (2)	C14—H14	0.9500
C6—H6	0.9500	C15—C16	1.490 (2)
C7—C8	1.355 (2)		
C2—O1—H1	113.9	O2—C9—C10	124.70 (14)
C6—C1—C2	118.43 (14)	O2—C9—C8	127.73 (14)
C6—C1—C7	125.05 (14)	C10—C9—C8	107.57 (13)

C2—C1—C7	116.51 (14)	C11—C10—C15	121.66 (15)
O1—C2—C3	121.74 (14)	C11—C10—C9	129.07 (15)
O1—C2—C1	117.73 (13)	C15—C10—C9	109.27 (13)
C3—C2—C1	120.53 (14)	C10—C11—C12	117.32 (15)
C4—C3—C2	120.16 (14)	C10—C11—H11	121.3
C4—C3—H3	119.9	C12—C11—H11	121.3
C2—C3—H3	119.9	C11—C12—C13	121.16 (15)
C3—C4—C5	119.47 (14)	C11—C12—H12	119.4
C3—C4—H4	120.3	C13—C12—H12	119.4
C5—C4—H4	120.3	C14—C13—C12	121.23 (15)
C6—C5—C4	121.94 (14)	C14—C13—H13	119.4
C6—C5—Br1	119.65 (12)	C12—C13—H13	119.4
C4—C5—Br1	118.37 (11)	C13—C14—C15	117.44 (15)
C5—C6—C1	119.46 (14)	C13—C14—H14	121.3
C5—C6—H6	120.3	C15—C14—H14	121.3
C1—C6—H6	120.3	C14—C15—C10	121.19 (15)
C8—C7—C1	134.39 (15)	C14—C15—C16	128.67 (15)
C8—C7—H7	112.8	C10—C15—C16	110.15 (14)
C1—C7—H7	112.8	O3—C16—C15	124.43 (15)
C7—C8—C16	133.85 (14)	O3—C16—C8	128.87 (14)
C7—C8—C9	119.83 (14)	C15—C16—C8	106.70 (13)
C16—C8—C9	106.32 (13)		

C6—C1—C2—O1	178.65 (15)	C8—C9—C10—C11	−179.35 (16)
C7—C1—C2—O1	−2.4 (2)	O2—C9—C10—C15	179.00 (16)
C6—C1—C2—C3	−0.7 (2)	C8—C9—C10—C15	−0.22 (18)
C7—C1—C2—C3	178.19 (15)	C15—C10—C11—C12	0.3 (2)
O1—C2—C3—C4	−178.38 (17)	C9—C10—C11—C12	179.34 (16)
C1—C2—C3—C4	1.0 (3)	C10—C11—C12—C13	−0.3 (3)
C2—C3—C4—C5	−0.7 (3)	C11—C12—C13—C14	0.0 (3)
C3—C4—C5—C6	0.1 (3)	C12—C13—C14—C15	0.4 (3)
C3—C4—C5—Br1	−177.54 (13)	C13—C14—C15—C10	−0.4 (3)
C4—C5—C6—C1	0.1 (2)	C13—C14—C15—C16	−179.70 (17)
Br1—C5—C6—C1	177.74 (12)	C11—C10—C15—C14	0.1 (3)
C2—C1—C6—C5	0.2 (2)	C9—C10—C15—C14	−179.15 (15)
C7—C1—C6—C5	−178.61 (15)	C11—C10—C15—C16	179.48 (15)
C6—C1—C7—C8	−1.5 (3)	C9—C10—C15—C16	0.27 (19)
C2—C1—C7—C8	179.65 (18)	C14—C15—C16—O3	−1.4 (3)
C1—C7—C8—C16	−0.2 (3)	C10—C15—C16—O3	179.24 (17)
C1—C7—C8—C9	−179.21 (17)	C14—C15—C16—C8	179.14 (17)
C7—C8—C9—O2	0.1 (3)	C10—C15—C16—C8	−0.22 (19)
C16—C8—C9—O2	−179.12 (17)	C7—C8—C16—O3	1.5 (3)
C7—C8—C9—C10	179.33 (15)	C9—C8—C16—O3	−179.35 (18)
C16—C8—C9—C10	0.08 (17)	C7—C8—C16—C15	−179.02 (18)
O2—C9—C10—C11	−0.1 (3)	C9—C8—C16—C15	0.08 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O3	0.95	2.15	2.994 (2)	148
O1—H1···O2^{i}	0.84	1.83	2.6641 (16)	173

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