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2-(3,5-Dibromo-2-hydroxybenzylideneamino)isoindole-1,3-dione

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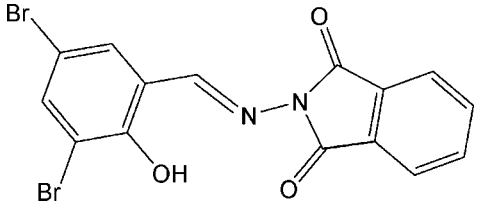
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.010 Å; R factor = 0.059; wR factor = 0.126; data-to-parameter ratio = 12.4.

In the title molecule, $C_{15}H_8Br_2N_2O_3$, the dihedral angle between the benzene ring and the isoindole ring system is 4.9 (1)°. An intramolecular O—H···N hydrogen bond stabilizes the molecular structure and intermolecular C—H···O hydrogen bonds link the molecules into a three-dimensional framework.

Related literature

For related literature, see: Belloni *et al.* (2005); Kahwa *et al.* (1986); Parashar *et al.* (1988); Santos *et al.* (2001); Tynan *et al.* (2005).



Experimental

Crystal data

$C_{15}H_8Br_2N_2O_3$
M_r = 424.05
Orthorhombic, *Pbca*
a = 16.8495 (9) Å
b = 7.2320 (3) Å
c = 23.3405 (11) Å
V = 2844.2 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 5.71 mm^{-1}
T = 113 (2) K
0.20 × 0.08 × 0.06 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.395, T_{max} = 0.726
20003 measured reflections
2513 independent reflections
2427 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.126
S = 1.20
2513 reflections
203 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.08 e Å^{-3}
$\Delta\rho_{min}$ = −1.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2	0.81 (3)	1.92 (6)	2.605 (7)	143 (8)
C3—H3···O3^{i}	0.95	2.33	3.264 (8)	169

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2298).

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supplementary materials

Acta Cryst. (2007). E**63**, o3002 [**doi:10.1107/S1600536807024804**]

2-(3,5-Dibromo-2-hydroxybenzylideneamino)isoindole-1,3-dione

Z.-L. Jing, R.-N. Li and J.-Q. Zhai

Comment

In order to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Belloni *et al.*, 2005; Tynan *et al.*, 2005; Parashar *et al.*, 1988),the synthesis of new and designed crystal structures has become a major strand of modern chemistry. Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Shiff bases functioning as ligands, we report the synthesis and structure of the title compound, (I).

In the molecular structure of (I) (Fig. 1), the expected geometric parameters are observed. The benzene ring system (C1—C6) is planar, with an r.m.s. deviation for the fitted atoms of 0.0056 (6) Å, as is the isoindole ring (C8—C15/N1), with an r.m.s. deviation of 0.0063 (5) Å. The dihedral angle between these two planes is 4.9 (1)°. An intramolecular O—H···N hydrogen bond stabilizes the molecular structure, and intermolecular C—H···O hydrogen bond link the molecules into a three-dimensional framework, as illustrated in Fig.2.

Experimental

An anhydrous ethanol solution (50 ml) of 3,5-dibromo-2-hydroxy- benzaldehyde (2.78 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 2-amino-isoindole-1,3-dione (1.62 g, 10 mmol), and the mixture was stirred at 350 K for 6 h under N_2, whereupon a red solution appeared. The solvent was removed and the residue was recrystallized from anhydrous ethanol. The product was isolated and then dried *in vacuo* to give pure compound (I) in 91% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Refinement

The hydroxyl H atom was found in a difference Fourier map and refined freely. C-bound H atoms were included in calculated positions, with C—H = 0.95 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

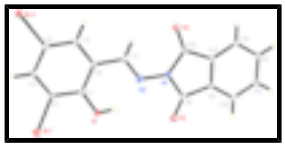

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

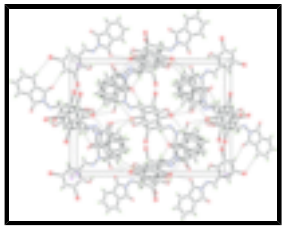

Fig. 2. The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are indicated by dashed lines.

2-(3,5-Dibromo-2-hydroxybenzylideneamino)isoindole-1,3-dione

Crystal data

$C_{15}H_8Br_2N_2O_3$	F_{000} = 1648
M_r = 424.05	D_x = 1.981 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71070 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 4946 reflections
a = 16.8495 (9) Å	θ = 2.6–25.0º
b = 7.2320 (3) Å	μ = 5.71 mm^{-1}
c = 23.3405 (11) Å	*T* = 113 (2) K
V = 2844.2 (2) Å^3	Prism, red
Z = 8	0.20 × 0.08 × 0.06 mm

Data collection

Rigaku Saturn diffractometer	2513 independent reflections
Radiation source: rotating anode	2427 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.067
T = 113(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (Jacobson, 1998)	*h* = −20→19
T_{min} = 0.395, T_{max} = 0.726	*k* = −8→8
20003 measured reflections	*l* = −27→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.059	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.126	$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2 + 33.0268P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.20	$(\Delta/\sigma)_{max}$ = 0.001
2513 reflections	$\Delta\rho_{max}$ = 1.08 e Å^{-3}
203 parameters	$\Delta\rho_{min}$ = −1.36 e Å^{-3}

1 restraint
Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br2	0.51772 (4)	0.89425 (9)	0.62815 (3)	0.0188 (2)
Br1	0.28824 (5)	0.65005 (13)	0.46789 (3)	0.0352 (3)
O1	0.4297 (3)	0.6161 (7)	0.39257 (19)	0.0189 (10)
H1	0.468 (3)	0.585 (11)	0.374 (3)	0.028*
N2	0.5809 (3)	0.6040 (8)	0.3697 (2)	0.0176 (12)
O2	0.5523 (3)	0.4722 (7)	0.26039 (19)	0.0202 (11)
C11	0.7913 (4)	0.3648 (9)	0.1776 (3)	0.0163 (14)
H11	0.8038	0.3194	0.1405	0.020*
C10	0.7122 (4)	0.3830 (9)	0.1933 (3)	0.0171 (14)
H10	0.6704	0.3505	0.1678	0.021*
C6	0.5326 (4)	0.7088 (9)	0.4591 (3)	0.0157 (14)
C5	0.5513 (4)	0.7713 (9)	0.5143 (3)	0.0165 (14)
H5	0.6052	0.7896	0.5249	0.020*
N1	0.6408 (3)	0.5580 (7)	0.3326 (2)	0.0119 (11)
O3	0.7580 (3)	0.6294 (7)	0.38100 (19)	0.0251 (12)
C9	0.6966 (4)	0.4512 (8)	0.2483 (3)	0.0128 (13)
C1	0.4524 (4)	0.6784 (9)	0.4442 (3)	0.0161 (14)
C7	0.5968 (4)	0.6680 (9)	0.4197 (3)	0.0172 (14)
H7	0.6504	0.6884	0.4309	0.021*
C14	0.7583 (4)	0.4983 (9)	0.2851 (3)	0.0138 (13)
C4	0.4917 (4)	0.8061 (9)	0.5535 (3)	0.0163 (14)
C3	0.4128 (4)	0.7761 (9)	0.5402 (3)	0.0159 (14)
H3	0.3723	0.7989	0.5676	0.019*
C2	0.3943 (4)	0.7120 (10)	0.4860 (3)	0.0175 (14)
C8	0.6195 (4)	0.4884 (9)	0.2770 (3)	0.0152 (14)
C12	0.8533 (4)	0.4117 (9)	0.2151 (3)	0.0183 (14)
H12	0.9068	0.3980	0.2030	0.022*
C15	0.7245 (4)	0.5694 (9)	0.3394 (3)	0.0155 (14)
C13	0.8370 (4)	0.4780 (9)	0.2699 (3)	0.0171 (14)
H13	0.8785	0.5083	0.2958	0.020*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br2	0.0230 (4)	0.0225 (4)	0.0107 (3)	0.0054 (3)	−0.0009 (3)	−0.0016 (3)
Br1	0.0221 (4)	0.0516 (5)	0.0319 (5)	−0.0006 (4)	−0.0008 (3)	−0.0080 (4)
O1	0.020 (3)	0.024 (3)	0.012 (2)	0.001 (2)	−0.0026 (19)	−0.005 (2)
N2	0.023 (3)	0.018 (3)	0.012 (3)	0.005 (3)	0.001 (2)	−0.003 (2)
O2	0.014 (3)	0.031 (3)	0.015 (2)	−0.003 (2)	0.000 (2)	−0.002 (2)
C11	0.018 (3)	0.018 (3)	0.013 (3)	0.001 (3)	0.004 (3)	−0.002 (3)
C10	0.026 (4)	0.015 (3)	0.010 (3)	0.000 (3)	−0.001 (3)	0.004 (3)
C6	0.013 (3)	0.018 (3)	0.016 (3)	0.000 (3)	0.001 (3)	0.002 (3)
C5	0.015 (3)	0.017 (3)	0.018 (3)	0.002 (3)	0.000 (3)	0.002 (3)
N1	0.011 (3)	0.014 (3)	0.010 (3)	0.001 (2)	0.000 (2)	−0.002 (2)
O3	0.016 (2)	0.042 (3)	0.017 (2)	0.001 (2)	−0.003 (2)	−0.013 (2)
C9	0.010 (3)	0.012 (3)	0.016 (3)	0.000 (3)	0.001 (3)	0.000 (3)
C1	0.022 (4)	0.017 (3)	0.009 (3)	−0.001 (3)	−0.002 (3)	0.000 (3)
C7	0.021 (4)	0.014 (3)	0.017 (3)	0.002 (3)	0.000 (3)	0.000 (3)
C14	0.014 (3)	0.017 (3)	0.010 (3)	0.000 (3)	0.000 (3)	0.004 (3)
C4	0.019 (4)	0.015 (3)	0.015 (3)	0.003 (3)	−0.003 (3)	0.004 (3)
C3	0.017 (3)	0.016 (3)	0.015 (3)	0.003 (3)	0.008 (3)	0.000 (3)
C2	0.011 (3)	0.024 (4)	0.018 (3)	0.000 (3)	−0.002 (3)	0.000 (3)
C8	0.019 (4)	0.012 (3)	0.014 (3)	0.002 (3)	−0.001 (3)	0.003 (3)
C12	0.019 (4)	0.016 (3)	0.020 (3)	0.001 (3)	0.004 (3)	0.002 (3)
C15	0.016 (3)	0.014 (3)	0.017 (3)	−0.003 (3)	0.000 (3)	0.000 (3)
C13	0.016 (3)	0.019 (3)	0.016 (3)	−0.001 (3)	−0.002 (3)	−0.003 (3)

Geometric parameters (Å, °)

Br2—C4	1.906 (7)	C5—H5	0.9500
Br1—C2	1.891 (6)	N1—C15	1.423 (8)
O1—C1	1.343 (8)	N1—C8	1.438 (8)
O1—H1	0.81 (3)	O3—C15	1.202 (8)
N2—C7	1.284 (8)	C9—C14	1.391 (9)
N2—N1	1.370 (7)	C9—C8	1.487 (9)
O2—C8	1.203 (8)	C1—C2	1.402 (9)
C11—C10	1.389 (9)	C7—H7	0.9500
C11—C12	1.404 (9)	C14—C13	1.381 (9)
C11—H11	0.9500	C14—C15	1.482 (9)
C10—C9	1.400 (9)	C4—C3	1.383 (9)
C10—H10	0.9500	C3—C2	1.382 (9)
C6—C5	1.402 (9)	C3—H3	0.9500
C6—C1	1.412 (9)	C12—C13	1.392 (9)
C6—C7	1.451 (9)	C12—H12	0.9500
C5—C4	1.382 (9)	C13—H13	0.9500
C1—O1—H1	111 (6)	C6—C7—H7	120.2
C7—N2—N1	120.5 (6)	C13—C14—C9	122.1 (6)
C10—C11—C12	121.8 (6)	C13—C14—C15	128.8 (6)

C10—C11—H11	119.1	C9—C14—C15	109.1 (6)
C12—C11—H11	119.1	C5—C4—C3	121.4 (6)
C11—C10—C9	117.0 (6)	C5—C4—Br2	119.9 (5)
C11—C10—H10	121.5	C3—C4—Br2	118.7 (5)
C9—C10—H10	121.5	C2—C3—C4	118.4 (6)
C5—C6—C1	119.5 (6)	C2—C3—H3	120.8
C5—C6—C7	118.6 (6)	C4—C3—H3	120.8
C1—C6—C7	121.8 (6)	C3—C2—C1	122.5 (6)
C4—C5—C6	120.2 (6)	C3—C2—Br1	119.8 (5)
C4—C5—H5	119.9	C1—C2—Br1	117.6 (5)
C6—C5—H5	119.9	O2—C8—N1	124.1 (6)
N2—N1—C15	130.3 (5)	O2—C8—C9	131.3 (6)
N2—N1—C8	118.1 (5)	N1—C8—C9	104.6 (5)
C15—N1—C8	111.6 (5)	C13—C12—C11	120.6 (6)
C14—C9—C10	120.9 (6)	C13—C12—H12	119.7
C14—C9—C8	109.2 (6)	C11—C12—H12	119.7
C10—C9—C8	129.9 (6)	O3—C15—N1	125.1 (6)
O1—C1—C2	118.9 (6)	O3—C15—C14	129.5 (6)
O1—C1—C6	123.0 (6)	N1—C15—C14	105.4 (5)
C2—C1—C6	118.0 (6)	C14—C13—C12	117.6 (6)
N2—C7—C6	119.5 (6)	C14—C13—H13	121.2
N2—C7—H7	120.2	C12—C13—H13	121.2
C12—C11—C10—C9	0.3 (10)	O1—C1—C2—C3	−180.0 (6)
C1—C6—C5—C4	−1.3 (10)	C6—C1—C2—C3	1.1 (10)
C7—C6—C5—C4	−178.0 (6)	O1—C1—C2—Br1	4.3 (9)
C7—N2—N1—C15	−0.4 (10)	C6—C1—C2—Br1	−174.6 (5)
C7—N2—N1—C8	−179.5 (6)	N2—N1—C8—O2	1.9 (9)
C11—C10—C9—C14	0.0 (9)	C15—N1—C8—O2	−177.4 (6)
C11—C10—C9—C8	179.1 (6)	N2—N1—C8—C9	−179.6 (5)
C5—C6—C1—O1	−179.0 (6)	C15—N1—C8—C9	1.1 (7)
C7—C6—C1—O1	−2.3 (10)	C14—C9—C8—O2	177.9 (7)
C5—C6—C1—C2	−0.1 (10)	C10—C9—C8—O2	−1.3 (12)
C7—C6—C1—C2	176.5 (6)	C14—C9—C8—N1	−0.4 (7)
N1—N2—C7—C6	−177.6 (6)	C10—C9—C8—N1	−179.6 (6)
C5—C6—C7—N2	177.4 (6)	C10—C11—C12—C13	0.2 (10)
C1—C6—C7—N2	0.7 (10)	N2—N1—C15—O3	−2.1 (11)
C10—C9—C14—C13	−0.8 (10)	C8—N1—C15—O3	177.1 (6)
C8—C9—C14—C13	179.9 (6)	N2—N1—C15—C14	179.4 (6)
C10—C9—C14—C15	178.9 (6)	C8—N1—C15—C14	−1.4 (7)
C8—C9—C14—C15	−0.4 (7)	C13—C14—C15—O3	2.4 (12)
C6—C5—C4—C3	1.8 (10)	C9—C14—C15—O3	−177.3 (7)
C6—C5—C4—Br2	−179.1 (5)	C13—C14—C15—N1	−179.2 (7)
C5—C4—C3—C2	−0.8 (10)	C9—C14—C15—N1	1.1 (7)
Br2—C4—C3—C2	−179.9 (5)	C9—C14—C13—C12	1.3 (10)
C4—C3—C2—C1	−0.6 (10)	C15—C14—C13—C12	−178.3 (6)
C4—C3—C2—Br1	174.9 (5)	C11—C12—C13—C14	−1.0 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2	0.81 (3)	1.92 (6)	2.605 (7)	143 (8)
C3—H3···O3^{i}	0.95	2.33	3.264 (8)	169

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

Fig. 1

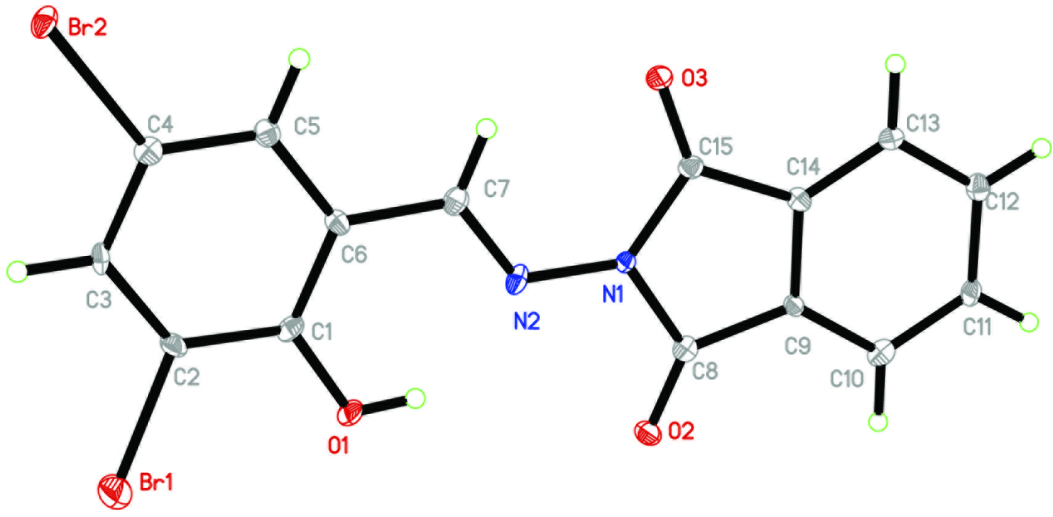
Br2
C4
C5
C7
O3
C15
C14
C13
C12
C6
C3
N2
N1
C1
C9
C11
C2
C8
C10
O1
O2
Br1

Fig. 2