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(*E*)-*N*′-(3,5-Dibromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide

Zheng-Chen Bai[a]* and Zuo-Liang Jing[b]

[a]College of Biotechnology and Food Science, Tianjin University of Commerce, Tianjin 300134, People's Republic of China, and [b]College of Sciences, Tianjin University of Science and Technology, Tianjin 300457, People's Republic of China
Correspondence e-mail: jzl74@tust.edu.cn

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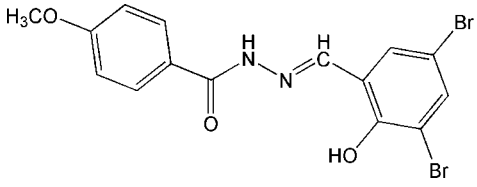
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.040; wR factor = 0.130; data-to-parameter ratio = 15.4.

In the crystal structure of the title compound, $C_{15}H_{12}Br_2N_2O_3$, the dihedral angle between the two ring planes is 11.40 (5)°. An intramolecular O—H···N hydrogen bond stabilizes the molecular structure. The molecules are linked *via* weak intermolecular N—H···O hydrogen bonding, forming an extended supramolecular arrangement.

Related literature

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



Experimental

Crystal data

$C_{15}H_{12}Br_2N_2O_3$
M_r = 428.07
Monoclinic, $P2_1/c$
a = 18.701 (4) Å
b = 8.8269 (17) Å
c = 9.6106 (18) Å
β = 97.571 (3)°
V = 1572.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.17 mm^{-1}
T = 294 (2) K
0.20 × 0.16 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.425, T_{max} = 0.747
8720 measured reflections
3199 independent reflections
2125 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.130
S = 1.05
3199 reflections
208 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.81 (3)	1.91 (4)	2.612 (5)	145 (5)
N2—H2···O2^{i}	0.81 (3)	2.10 (3)	2.898 (5)	169 (4)

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2376).

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supplementary materials

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(*E*)-*N*'-(3,5-Dibromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide

Z.-C. Bai and Z.-L. Jing

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 the 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 Schiff bases functioning as ligands, we report the synthesis and structure of the title compound, (I).

In the structure of the title molecule (I) (Fig. 1), the geometric parameters are normal. The first benzene ring system (C1—C6) is planar, with an r.m.s. deviation for fitted atoms of 0.0071 (3) Å and the r.m.s. deviation for the other benzene group (C9—C14) is 0.0046 (2) Å. The dihedral angle between the two planes is 168.60 (5)°.

An intramolecular O—H···N hydrogen bond stabilizes the molecular structure. The molecules are linked *via* weak intermolecular N—H···O hydrogen bond, forming an extented supramolecular arrangement, as illustrated in Fig. 2 and Table. 1.

Experimental

An anhydrous ethanol solution (50 ml) of 4-methoxybenzohydrazide(1.66 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 3,5-dibromo-2-hydroxybenzaldehyde (2.77 g, 10 mmol), and the mixture was stirred at 350 K for 6 h under N_2, whereupon a red precipitate appeared. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give pure compound (I) in 85% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Refinement

The N-bound H atom was located in a difference Fourier map and refined freely. C-bound H atoms were included in calculated positions, with C—H = 0.93–0.96 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms.

Figures

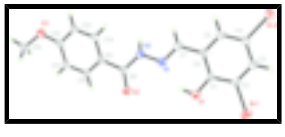

Fig. 1. The structure of the title molecule (I). Displacement ellipsoids are drawn at the 30% probability level.

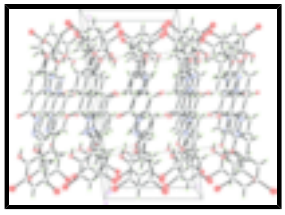

Fig. 2. The crystal packing of (I). Hydrogen bonds are indicated by dashed lines.

(*E*)-*N*'-(3,5-Dibromo-2-hydroxybenzylidene)-4-methoxybenzohydrazide

Crystal data

$C_{15}H_{12}Br_2N_2O_3$	F_{000} = 840
M_r = 428.07	D_x = 1.808 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2240 reflections
a = 18.701 (4) Å	θ = 2.6–24.4º
b = 8.8269 (17) Å	μ = 5.17 mm^{-1}
c = 9.6106 (18) Å	T = 294 (2) K
β = 97.571 (3)º	Block, red
V = 1572.6 (5) Å^3	0.20 × 0.16 × 0.06 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3199 independent reflections
Radiation source: fine-focus sealed tube	2125 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.050
T = 294(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 1.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −23→23
T_{min} = 0.425, T_{max} = 0.747	k = −10→10
8720 measured reflections	l = −10→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.130	$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
3199 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}

208 parameters
2 restraints
Primary atom site location: structure-invariant direct methods

$\Delta\rho_{min} = -0.71$ e Å^{-3}
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}
Br1	0.07397 (3)	0.32116 (6)	0.13724 (6)	0.0652 (2)
Br2	0.08446 (3)	0.69466 (6)	0.61765 (6)	0.0599 (2)
N1	0.35157 (18)	0.3267 (4)	0.4140 (4)	0.0409 (9)
N2	0.42226 (19)	0.2971 (4)	0.4625 (4)	0.0402 (9)
H2	0.433 (2)	0.306 (4)	0.547 (3)	0.038 (12)*
O1	0.23327 (18)	0.2858 (4)	0.2409 (3)	0.0502 (8)
H1	0.2757 (16)	0.273 (5)	0.270 (5)	0.060 (16)*
O2	0.43775 (15)	0.1678 (3)	0.2649 (3)	0.0485 (8)
O3	0.75077 (17)	0.1069 (4)	0.6158 (3)	0.0589 (9)
C1	0.2017 (2)	0.3777 (5)	0.3273 (4)	0.0367 (9)
C2	0.1287 (2)	0.4105 (4)	0.2970 (4)	0.0392 (10)
C3	0.0937 (2)	0.5058 (5)	0.3800 (4)	0.0423 (10)
H3	0.0451	0.5283	0.3561	0.051*
C4	0.1324 (2)	0.5673 (4)	0.4994 (4)	0.0399 (10)
C5	0.2038 (2)	0.5347 (4)	0.5354 (4)	0.0413 (10)
H5	0.2287	0.5756	0.6169	0.050*
C6	0.2399 (2)	0.4404 (4)	0.4505 (4)	0.0366 (10)
C7	0.3156 (2)	0.4061 (5)	0.4935 (4)	0.0421 (11)
H7	0.3382	0.4417	0.5792	0.051*
C8	0.4624 (2)	0.2125 (5)	0.3822 (4)	0.0368 (9)
C9	0.5380 (2)	0.1835 (4)	0.4464 (4)	0.0346 (9)
C10	0.5767 (2)	0.0680 (4)	0.3907 (4)	0.0382 (10)
H10	0.5542	0.0100	0.3168	0.046*
C11	0.6475 (2)	0.0386 (5)	0.4434 (4)	0.0419 (10)
H11	0.6725	−0.0383	0.4043	0.050*
C12	0.6816 (2)	0.1230 (5)	0.5542 (4)	0.0429 (10)
C13	0.6424 (2)	0.2362 (6)	0.6113 (5)	0.0520 (12)
H13	0.6645	0.2924	0.6870	0.062*
C14	0.5722 (2)	0.2668 (5)	0.5587 (5)	0.0488 (11)

H14	0.5474	0.3436	0.5982	0.059*
C15	0.7983 (3)	0.0133 (6)	0.5473 (5)	0.0610 (13)
H15A	0.7832	−0.0905	0.5501	0.091*
H15B	0.8466	0.0230	0.5945	0.091*
H15C	0.7969	0.0449	0.4513	0.091*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0543 (3)	0.0859 (4)	0.0525 (3)	−0.0181 (3)	−0.0040 (2)	−0.0166 (3)
Br2	0.0501 (3)	0.0648 (4)	0.0654 (4)	0.0094 (2)	0.0096 (3)	−0.0154 (2)
N1	0.0360 (19)	0.050 (2)	0.0366 (19)	0.0031 (16)	0.0034 (16)	0.0062 (16)
N2	0.037 (2)	0.053 (2)	0.0295 (19)	0.0076 (17)	−0.0023 (16)	0.0011 (17)
O1	0.049 (2)	0.060 (2)	0.0400 (18)	0.0066 (17)	−0.0004 (16)	−0.0089 (15)
O2	0.0429 (17)	0.073 (2)	0.0280 (16)	−0.0033 (15)	−0.0012 (13)	−0.0036 (14)
O3	0.0439 (19)	0.081 (2)	0.049 (2)	0.0173 (17)	−0.0041 (16)	−0.0098 (18)
C1	0.041 (2)	0.041 (2)	0.029 (2)	−0.0017 (19)	0.0061 (19)	0.0015 (18)
C2	0.038 (2)	0.045 (3)	0.032 (2)	−0.0066 (19)	−0.0043 (19)	0.0015 (18)
C3	0.034 (2)	0.044 (2)	0.048 (3)	−0.005 (2)	0.002 (2)	0.004 (2)
C4	0.039 (2)	0.039 (2)	0.041 (2)	0.0013 (19)	0.005 (2)	0.0008 (18)
C5	0.045 (3)	0.042 (3)	0.035 (2)	−0.004 (2)	0.000 (2)	0.0009 (18)
C6	0.038 (2)	0.040 (2)	0.032 (2)	0.0012 (18)	0.0011 (18)	0.0059 (17)
C7	0.044 (3)	0.049 (3)	0.031 (2)	0.004 (2)	−0.002 (2)	0.0052 (19)
C8	0.039 (2)	0.043 (2)	0.028 (2)	−0.0027 (19)	0.0053 (18)	0.0043 (18)
C9	0.035 (2)	0.042 (2)	0.027 (2)	−0.0031 (18)	0.0043 (17)	0.0023 (17)
C10	0.044 (2)	0.038 (2)	0.032 (2)	0.0026 (19)	0.0035 (19)	0.0018 (18)
C11	0.056 (3)	0.039 (2)	0.032 (2)	0.010 (2)	0.008 (2)	−0.0004 (18)
C12	0.040 (2)	0.050 (3)	0.038 (2)	0.005 (2)	0.007 (2)	0.002 (2)
C13	0.045 (3)	0.064 (3)	0.045 (3)	0.006 (2)	−0.004 (2)	−0.024 (2)
C14	0.045 (3)	0.054 (3)	0.048 (3)	0.002 (2)	0.008 (2)	−0.018 (2)
C15	0.047 (3)	0.075 (3)	0.062 (3)	0.016 (3)	0.012 (3)	0.004 (3)

Geometric parameters (Å, °)

Br1—C2	1.901 (4)	C5—H5	0.9300
Br2—C4	1.905 (4)	C6—C7	1.452 (6)
N1—C7	1.291 (5)	C7—H7	0.9300
N1—N2	1.368 (5)	C8—C9	1.488 (5)
N2—C8	1.368 (5)	C9—C14	1.391 (6)
N2—H2	0.81 (3)	C9—C10	1.396 (5)
O1—C1	1.351 (5)	C10—C11	1.380 (5)
O1—H1	0.81 (3)	C10—H10	0.9300
O2—C8	1.226 (5)	C11—C12	1.385 (6)
O3—C12	1.358 (5)	C11—H11	0.9300
O3—C15	1.436 (6)	C12—C13	1.393 (6)
C1—C2	1.388 (5)	C13—C14	1.370 (6)
C1—C6	1.413 (6)	C13—H13	0.9300
C2—C3	1.382 (6)	C14—H14	0.9300
C3—C4	1.384 (6)	C15—H15A	0.9600

C3—H3	0.9300	C15—H15B	0.9600
C4—C5	1.365 (6)	C15—H15C	0.9600
C5—C6	1.400 (6)		
C7—N1—N2	117.2 (4)	O2—C8—N2	121.8 (4)
N1—N2—C8	119.3 (4)	O2—C8—C9	123.2 (4)
N1—N2—H2	114 (3)	N2—C8—C9	115.0 (3)
C8—N2—H2	123 (3)	C14—C9—C10	118.4 (4)
C1—O1—H1	111 (4)	C14—C9—C8	123.0 (4)
C12—O3—C15	118.2 (4)	C10—C9—C8	118.6 (4)
O1—C1—C2	119.6 (4)	C11—C10—C9	121.2 (4)
O1—C1—C6	122.4 (4)	C11—C10—H10	119.4
C2—C1—C6	118.0 (4)	C9—C10—H10	119.4
C3—C2—C1	122.2 (4)	C10—C11—C12	120.2 (4)
C3—C2—Br1	118.3 (3)	C10—C11—H11	119.9
C1—C2—Br1	119.5 (3)	C12—C11—H11	119.9
C2—C3—C4	118.8 (4)	O3—C12—C11	126.4 (4)
C2—C3—H3	120.6	O3—C12—C13	115.0 (4)
C4—C3—H3	120.6	C11—C12—C13	118.5 (4)
C5—C4—C3	121.0 (4)	C14—C13—C12	121.5 (4)
C5—C4—Br2	119.6 (3)	C14—C13—H13	119.2
C3—C4—Br2	119.4 (3)	C12—C13—H13	119.2
C4—C5—C6	120.5 (4)	C13—C14—C9	120.2 (4)
C4—C5—H5	119.7	C13—C14—H14	119.9
C6—C5—H5	119.7	C9—C14—H14	119.9
C5—C6—C1	119.4 (4)	O3—C15—H15A	109.5
C5—C6—C7	119.0 (4)	O3—C15—H15B	109.5
C1—C6—C7	121.6 (4)	H15A—C15—H15B	109.5
N1—C7—C6	120.6 (4)	O3—C15—H15C	109.5
N1—C7—H7	119.7	H15A—C15—H15C	109.5
C6—C7—H7	119.7	H15B—C15—H15C	109.5
C7—N1—N2—C8	179.6 (4)	C1—C6—C7—N1	5.6 (6)
O1—C1—C2—C3	−178.8 (4)	N1—N2—C8—O2	3.3 (6)
C6—C1—C2—C3	2.5 (6)	N1—N2—C8—C9	−178.4 (3)
O1—C1—C2—Br1	1.6 (5)	O2—C8—C9—C14	161.3 (4)
C6—C1—C2—Br1	−177.1 (3)	N2—C8—C9—C14	−16.9 (6)
C1—C2—C3—C4	−1.8 (6)	O2—C8—C9—C10	−18.2 (6)
Br1—C2—C3—C4	177.7 (3)	N2—C8—C9—C10	163.6 (4)
C2—C3—C4—C5	−0.1 (6)	C14—C9—C10—C11	−1.3 (6)
C2—C3—C4—Br2	−178.4 (3)	C8—C9—C10—C11	178.3 (4)
C3—C4—C5—C6	1.2 (6)	C9—C10—C11—C12	0.6 (6)
Br2—C4—C5—C6	179.5 (3)	C15—O3—C12—C11	11.8 (7)
C4—C5—C6—C1	−0.5 (6)	C15—O3—C12—C13	−168.5 (4)
C4—C5—C6—C7	−178.9 (4)	C10—C11—C12—O3	−179.7 (4)
O1—C1—C6—C5	−180.0 (4)	C10—C11—C12—C13	0.7 (7)
C2—C1—C6—C5	−1.3 (6)	O3—C12—C13—C14	179.1 (4)
O1—C1—C6—C7	−1.6 (6)	C11—C12—C13—C14	−1.2 (7)
C2—C1—C6—C7	177.1 (4)	C12—C13—C14—C9	0.5 (8)
N2—N1—C7—C6	−179.0 (3)	C10—C9—C14—C13	0.7 (7)

C5—C6—C7—N1	−176.0 (4)	C8—C9—C14—C13	−178.8 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.81 (3)	1.91 (4)	2.612 (5)	145 (5)
N2—H2···O2^{i}	0.81 (3)	2.10 (3)	2.898 (5)	169 (4)

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

Fig. 1

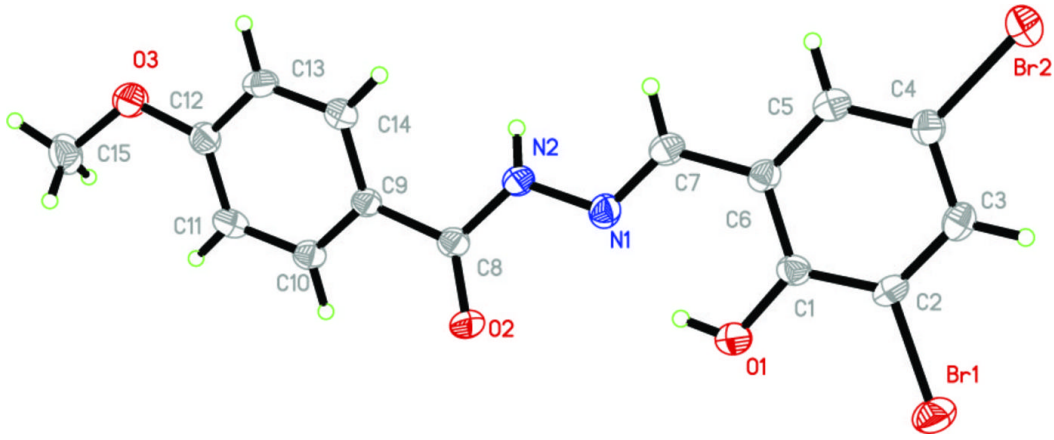
O3
C12
C13
C14
C15
C9
C11
C10
C8
O2
N2
N1
C7
C5
C4
Br2
C6
C3
C1
C2
O1
Br1

Fig. 2