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***N'*-(*E*)-1-(5-Bromo-2-hydroxyphenyl)-ethylidene]benzohydrazide**Chang-Zheng Zheng,^a Chang-You Ji,^{a*} Xiu-Li Chang^b and Li-qin Zhang^a^aCollege of Environmental and Chemical Engineering, Xi'an Polytechnic University, 710048 Xi'an, Shaanxi, People's Republic of China, and ^bDepartment of Materials Science and Chemical Engineering, Sichuan University of Science and Engineering, 643000 Zigong, Sichuan, People's Republic of China

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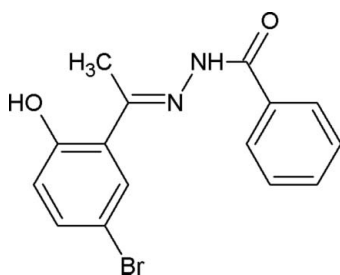
Received 23 November 2008; accepted 25 November 2008

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.068; wR factor = 0.188; data-to-parameter ratio = 15.5.

The $\text{C}=\text{N}$ double bond in the title compound, $\text{C}_{15}\text{H}_{13}\text{BrN}_2\text{O}_2$, is *trans E* configured and the dihedral angle between the aromatic ring planes is $22.3(1)^\circ$. The crystal structure is stabilized by intramolecular $\text{O}-\text{H}\cdots\text{O}$ and intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For aroylhydrazones and their biological activity, see: Singh *et al.* (1982); Salem (1998); Carcelli *et al.* (1995).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{13}\text{BrN}_2\text{O}_2$ $M_r = 333.18$

Monoclinic, $P2_1/c$
 $a = 7.3761(15)$ Å
 $b = 28.270(6)$ Å
 $c = 8.6089(13)$ Å
 $\beta = 116.928(12)^\circ$
 $V = 1600.5(5)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.57$ mm⁻¹
 $T = 298(2)$ K
 $0.12 \times 0.08 \times 0.06$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.748$, $T_{\max} = 0.861$

8028 measured reflections
 2830 independent reflections
 1490 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.188$
 $S = 1.01$
 2830 reflections

183 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.87$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.85	2.522 (6)	138
$\text{N2}-\text{H2}\cdots\text{O2}^i$	0.86	2.14	2.889 (6)	146

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

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2814).

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supplementary materials

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N'-[(*E*)-1-(5-Bromo-2-hydroxyphenyl)ethylidene]benzohydrazide

C.-Z. Zheng, C.-Y. Ji, X.-L. Chang and L. Zhang

Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions (Singh *et al.*, 1982; Salem, 1998) and their biological activity (Singh *et al.*, 1982; Carcelli *et al.*, 1995). As an extension of work on the structural characterization of aroylhydrazone derivatives, the title compound was synthesized and its crystal structure is reported here.

The title molecule displays a *trans* configured C=N double bond (Fig. 1). The crystal structure is stabilized by intramolecular O—H \cdots O and intermolecular N—H \cdots O hydrogen bonds (Table 1. and Fig. 2).

Experimental

Benzoylhydrazine (0.02 mol, 2.72 g) was dissolved in anhydrous ethanol (50 ml), and 1-(5-bromo-2-hydroxyphenyl)ethanone (0.02 mol, 4.30 g) was added. The reaction mixture was refluxed for 6 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 85%). The compound (2.0 mmol, 0.67 g) was dissolved in dimethylformamide (30 ml) and kept at room temperature for 30 d to obtain yellow single crystals suitable for X-ray diffraction.

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H(methyl) = 0.96 Å, C—H(aromatic) = 0.93 Å, O—H = 0.82 Å, and N—H = 0.86 Å and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}}, \text{O})$ and $1.2U_{\text{eq}}(\text{C}_{\text{aromatic}}, \text{N})$.

Figures

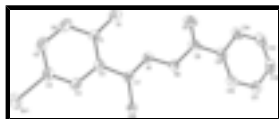


Fig. 1. The molecular structure the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

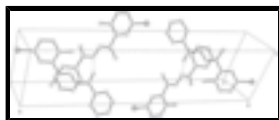


Fig. 2. The crystal packing of the title compound. Dashed lines show hydrogen bonds.

N'-[(*E*)-1-(5-Bromo-2-hydroxyphenyl)ethylidene]benzohydrazide

Crystal data

C₁₅H₁₃BrN₂O₂

$F_{000} = 672$

supplementary materials

$M_r = 333.18$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.3761$ (15) Å

$b = 28.270$ (6) Å

$c = 8.6089$ (13) Å

$\beta = 116.928$ (12)°

$V = 1600.5$ (5) Å³

$Z = 4$

$D_x = 1.383$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1189 reflections

$\theta = 2.9$ – 20.7 °

$\mu = 2.57$ mm⁻¹

$T = 298$ (2) K

Block, yellow

$0.12 \times 0.08 \times 0.06$ mm

Data collection

Siemens SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.748$, $T_{\max} = 0.861$

8028 measured reflections

2830 independent reflections

1490 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.062$

$\theta_{\text{max}} = 25.1$ °

$\theta_{\text{min}} = 3.0$ °

$h = -8 \rightarrow 8$

$k = -33 \rightarrow 33$

$l = -7 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.068$

$wR(F^2) = 0.188$

$S = 1.01$

2830 reflections

183 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.089P)^2 + 0.7896P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 0.87$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.37$ 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.81952 (14)	1.00196 (2)	0.69361 (11)	0.0935 (4)
O1	0.6647 (7)	0.79484 (14)	0.7321 (5)	0.0613 (11)
H1	0.6183	0.7820	0.6365	0.092*
O2	0.3297 (6)	0.70963 (13)	0.4419 (5)	0.0602 (11)
N1	0.4384 (6)	0.79657 (14)	0.4079 (5)	0.0391 (10)
N2	0.3180 (6)	0.76994 (15)	0.2660 (5)	0.0416 (10)
H2	0.2720	0.7810	0.1620	0.050*
C1	0.6935 (8)	0.84139 (19)	0.7143 (6)	0.0442 (13)
C2	0.6016 (8)	0.86503 (17)	0.5556 (6)	0.0390 (12)
C3	0.6426 (8)	0.91292 (19)	0.5540 (7)	0.0497 (14)
H3	0.5829	0.9294	0.4491	0.060*
C4	0.7677 (8)	0.9363 (2)	0.7018 (8)	0.0555 (15)
C5	0.8592 (10)	0.9132 (2)	0.8592 (8)	0.0655 (17)
H5	0.9461	0.9293	0.9598	0.079*
C6	0.8202 (9)	0.8661 (2)	0.8654 (8)	0.0658 (18)
H6	0.8789	0.8503	0.9716	0.079*
C7	0.4633 (7)	0.84066 (17)	0.3902 (6)	0.0370 (12)
C8	0.2737 (8)	0.72504 (18)	0.2962 (7)	0.0430 (13)
C9	0.1524 (8)	0.69622 (19)	0.1385 (7)	0.0460 (13)
C10	0.0099 (8)	0.7153 (2)	-0.0181 (7)	0.0509 (14)
H10	-0.0132	0.7477	-0.0273	0.061*
C11	-0.0978 (9)	0.6865 (2)	-0.1603 (8)	0.0581 (16)
H11	-0.1931	0.6994	-0.2649	0.070*
C12	-0.0621 (10)	0.6383 (2)	-0.1449 (9)	0.0641 (17)
H12	-0.1335	0.6186	-0.2400	0.077*
C13	0.0790 (11)	0.6191 (2)	0.0110 (9)	0.0618 (16)
H13	0.1027	0.5866	0.0200	0.074*
C14	0.1832 (9)	0.64743 (19)	0.1507 (7)	0.0496 (14)
H14	0.2760	0.6342	0.2557	0.060*
C15	0.3682 (9)	0.86765 (19)	0.2249 (6)	0.0535 (15)
H15A	0.4673	0.8733	0.1836	0.080*
H15B	0.3186	0.8973	0.2445	0.080*
H15C	0.2571	0.8498	0.1395	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1011 (7)	0.0462 (5)	0.0962 (7)	-0.0133 (4)	0.0123 (5)	-0.0172 (4)

supplementary materials

O1	0.073 (3)	0.058 (3)	0.040 (2)	-0.003 (2)	0.014 (2)	0.0092 (19)
O2	0.103 (3)	0.040 (2)	0.041 (2)	0.000 (2)	0.037 (2)	0.0038 (17)
N1	0.051 (3)	0.036 (3)	0.029 (2)	0.002 (2)	0.0173 (19)	-0.0012 (19)
N2	0.054 (3)	0.037 (3)	0.032 (2)	-0.006 (2)	0.019 (2)	-0.0022 (19)
C1	0.043 (3)	0.045 (3)	0.037 (3)	-0.003 (2)	0.011 (2)	0.004 (2)
C2	0.034 (3)	0.039 (3)	0.044 (3)	0.002 (2)	0.017 (2)	-0.005 (2)
C3	0.047 (3)	0.049 (3)	0.040 (3)	0.008 (3)	0.009 (3)	-0.005 (3)
C4	0.047 (3)	0.048 (4)	0.062 (4)	0.001 (3)	0.015 (3)	-0.014 (3)
C5	0.064 (4)	0.067 (4)	0.051 (4)	-0.005 (3)	0.013 (3)	-0.020 (3)
C6	0.062 (4)	0.079 (5)	0.040 (3)	0.002 (3)	0.008 (3)	-0.003 (3)
C7	0.044 (3)	0.037 (3)	0.028 (3)	0.004 (2)	0.014 (2)	-0.001 (2)
C8	0.064 (4)	0.032 (3)	0.041 (3)	0.006 (3)	0.031 (3)	0.000 (2)
C9	0.057 (3)	0.043 (3)	0.049 (3)	-0.009 (3)	0.033 (3)	-0.004 (3)
C10	0.057 (3)	0.040 (3)	0.053 (4)	-0.006 (3)	0.022 (3)	0.004 (3)
C11	0.058 (4)	0.052 (4)	0.055 (4)	-0.013 (3)	0.017 (3)	-0.006 (3)
C12	0.070 (4)	0.062 (4)	0.064 (4)	-0.024 (3)	0.034 (4)	-0.027 (3)
C13	0.082 (4)	0.041 (3)	0.079 (5)	-0.009 (3)	0.050 (4)	-0.011 (3)
C14	0.066 (4)	0.036 (3)	0.053 (3)	-0.004 (3)	0.033 (3)	-0.003 (3)
C15	0.071 (4)	0.036 (3)	0.034 (3)	0.001 (3)	0.006 (3)	0.002 (2)

Geometric parameters (Å, °)

Br1—C4	1.902 (6)	C6—H6	0.9300
O1—C1	1.353 (6)	C7—C15	1.482 (7)
O1—H1	0.8200	C8—C9	1.485 (7)
O2—C8	1.210 (6)	C9—C10	1.389 (7)
N1—C7	1.279 (6)	C9—C14	1.394 (7)
N1—N2	1.366 (5)	C10—C11	1.382 (7)
N2—C8	1.365 (6)	C10—H10	0.9300
N2—H2	0.8600	C11—C12	1.384 (8)
C1—C2	1.391 (7)	C11—H11	0.9300
C1—C6	1.397 (8)	C12—C13	1.383 (9)
C2—C3	1.389 (7)	C12—H12	0.9300
C2—C7	1.492 (7)	C13—C14	1.358 (8)
C3—C4	1.358 (7)	C13—H13	0.9300
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.376 (8)	C15—H15A	0.9600
C5—C6	1.367 (9)	C15—H15B	0.9600
C5—H5	0.9300	C15—H15C	0.9600
C1—O1—H1	109.5	O2—C8—C9	122.1 (5)
C7—N1—N2	120.1 (4)	N2—C8—C9	115.7 (4)
C8—N2—N1	117.2 (4)	C10—C9—C14	118.9 (5)
C8—N2—H2	121.4	C10—C9—C8	123.5 (5)
N1—N2—H2	121.4	C14—C9—C8	117.6 (5)
O1—C1—C2	123.2 (4)	C11—C10—C9	120.7 (6)
O1—C1—C6	117.0 (5)	C11—C10—H10	119.6
C2—C1—C6	119.8 (5)	C9—C10—H10	119.6
C3—C2—C1	117.8 (5)	C10—C11—C12	119.1 (6)
C3—C2—C7	119.9 (4)	C10—C11—H11	120.4

C1—C2—C7	122.3 (5)	C12—C11—H11	120.4
C4—C3—C2	121.6 (5)	C13—C12—C11	120.4 (5)
C4—C3—H3	119.2	C13—C12—H12	119.8
C2—C3—H3	119.2	C11—C12—H12	119.8
C3—C4—C5	120.9 (6)	C14—C13—C12	120.2 (6)
C3—C4—Br1	120.2 (5)	C14—C13—H13	119.9
C5—C4—Br1	118.9 (4)	C12—C13—H13	119.9
C6—C5—C4	118.9 (6)	C13—C14—C9	120.7 (6)
C6—C5—H5	120.6	C13—C14—H14	119.7
C4—C5—H5	120.6	C9—C14—H14	119.7
C5—C6—C1	121.0 (6)	C7—C15—H15A	109.5
C5—C6—H6	119.5	C7—C15—H15B	109.5
C1—C6—H6	119.5	H15A—C15—H15B	109.5
N1—C7—C15	125.8 (4)	C7—C15—H15C	109.5
N1—C7—C2	114.2 (4)	H15A—C15—H15C	109.5
C15—C7—C2	120.0 (5)	H15B—C15—H15C	109.5
O2—C8—N2	122.2 (5)		
C7—N1—N2—C8	-170.7 (5)	C1—C2—C7—N1	-0.3 (7)
O1—C1—C2—C3	179.8 (5)	C3—C2—C7—C15	-0.6 (7)
C6—C1—C2—C3	0.8 (8)	C1—C2—C7—C15	179.6 (5)
O1—C1—C2—C7	-0.4 (8)	N1—N2—C8—O2	3.3 (7)
C6—C1—C2—C7	-179.4 (5)	N1—N2—C8—C9	-176.6 (4)
C1—C2—C3—C4	-0.2 (8)	O2—C8—C9—C10	149.0 (5)
C7—C2—C3—C4	-180.0 (5)	N2—C8—C9—C10	-31.1 (7)
C2—C3—C4—C5	0.2 (9)	O2—C8—C9—C14	-30.3 (8)
C2—C3—C4—Br1	-180.0 (4)	N2—C8—C9—C14	149.6 (5)
C3—C4—C5—C6	-0.8 (9)	C14—C9—C10—C11	-0.8 (8)
Br1—C4—C5—C6	179.3 (5)	C8—C9—C10—C11	179.9 (5)
C4—C5—C6—C1	1.5 (10)	C9—C10—C11—C12	0.0 (8)
O1—C1—C6—C5	179.4 (6)	C10—C11—C12—C13	0.3 (9)
C2—C1—C6—C5	-1.5 (9)	C11—C12—C13—C14	0.3 (9)
N2—N1—C7—C15	1.2 (8)	C12—C13—C14—C9	-1.2 (9)
N2—N1—C7—C2	-178.9 (4)	C10—C9—C14—C13	1.4 (8)
C3—C2—C7—N1	179.5 (5)	C8—C9—C14—C13	-179.3 (5)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.82	1.85	2.522 (6)	138
N2—H2 \cdots O2 ⁱ	0.86	2.14	2.889 (6)	146

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

Fig. 1

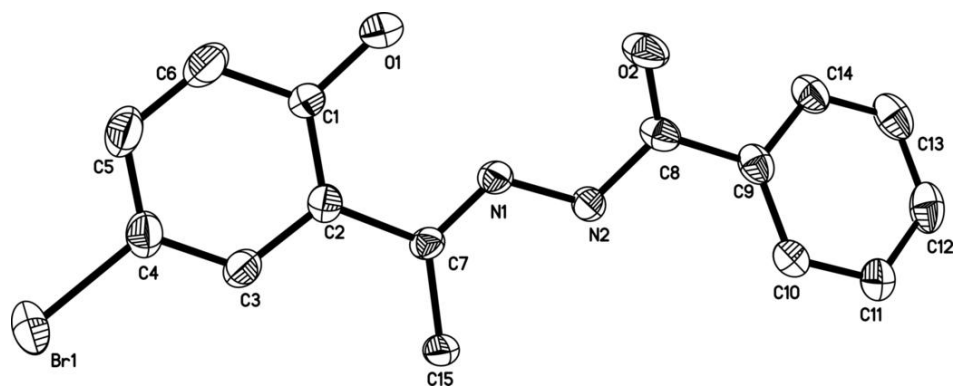


Fig. 2

