Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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]

[a]College of Environmental and Chemical Engineering, Xi'an Polytechnic University, 710048 Xi'an, Shaanxi, People's Republic of China, and [b]Department of Materials Science and Chemical Engineering, Sichuan University of Science and Engineering , 643000 Zigong, Sichuan, People's Republic of China
Correspondence e-mail: jichangyou789456@126.com

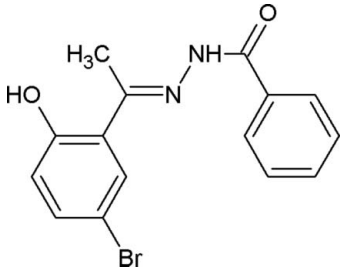
Received 23 November 2008; accepted 25 November 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.068; wR factor = 0.188; data-to-parameter ratio = 15.5.

The C=N double bond in the title compound, $C_{15}H_{13}BrN_2O_2$, is *trans E* configured and the dihedral angle between the aromatic ring planes is 22.3 (1)°. The crystal structure is stabilized by intramolecular O—H···O and intermolecular N—H···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

$C_{15}H_{13}BrN_2O_2$ M_r = 333.18
Monoclinic, $P2_1/c$
a = 7.3761 (15) Å
b = 28.270 (6) Å
c = 8.6089 (13) Å
β = 116.928 (12)°
V = 1600.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.57 mm^{-1}
T = 298 (2) K
0.12 × 0.08 × 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_{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
$\Delta\rho_{max}$ = 0.87 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.85	2.522 (6)	138
N2—H2···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*.

This project was supported by the Postgraduate Foundation of Xi'an Polytechnic University (grant No. Y05–2–09)

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

References

Carcelli, M., Mazza, P., Pelizzi, G. & Zani, F. (1995). *J. Inorg. Biochem.* **57**, 43–62.
Salem, A. A. (1998). *Microchem. J.* **60**, 51–66.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Singh, R. B., Jain, P. & Singh, R. P. (1982). *Talanta*, **29**, 77–84.

supporting information

Acta Cryst. (2008). E**64**, o2487 [doi:10.1107/S1600536808039755]

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

Chang-Zheng Zheng, Chang-You Ji, Xiu-Li Chang and Li-qin Zhang

S1. 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···O and intermolecular N—H···O hydrogen bonds (Table 1. and Fig. 2).

S2. 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.

S3. 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_{iso}(H) =1.5U_{eq}(C_{methyl},*O*) and 1.2U_{eq}($C_{aromatic}$,*N*).

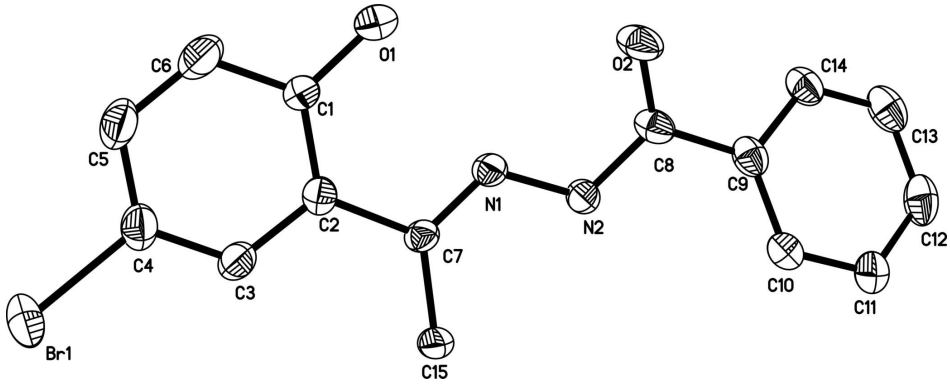


Figure 1

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

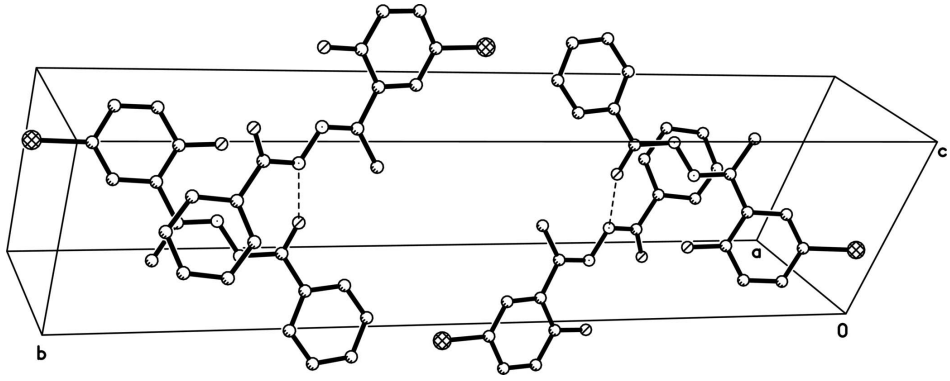


Figure 2

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

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

Crystal data

$C_{15}H_{13}BrN_2O_2$
$M_r = 333.18$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.3761\ (15)$ Å
$b = 28.270\ (6)$ Å
$c = 8.6089\ (13)$ Å
$\beta = 116.928\ (12)°$
$V = 1600.5\ (5)$ Å^3
$Z = 4$
$F(000) = 672$
$D_x = 1.383$ 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}
$T = 298$ K
Block, yellow
0.12 × 0.08 × 0.06 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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 $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 25.1°$, $\theta_{min} = 3.0°$
$h = -8\rightarrow8$
$k = -33\rightarrow33$
$l = -7\rightarrow10$

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
0 restraints
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.87$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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)
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, º)

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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.85	2.522 (6)	138

N2—H2···O2^{i}	0.86	2.14	2.889 (6)	146

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