Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-3-Bromo-*N*′-(4-hydroxy-3-nitrobenzylidene)benzohydrazide

Guo-Biao Cao[a]* and Xiao-Ya Wang[b]

[a]Department of Chemistry, Ankang University, Ankang Shanxi 725000, People's Republic of China, and [b]Department of Biology, Ankang University, Ankang Shanxi 725000, People's Republic of China
Correspondence e-mail: guobiao_cao@126.com

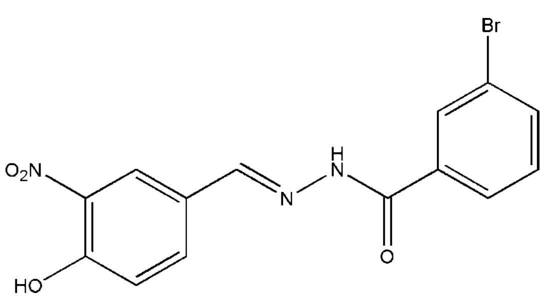
Received 23 June 2009; accepted 23 June 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.128; data-to-parameter ratio = 14.5.

The title compound, $C_{14}H_{10}BrN_3O_4$, was synthesized by the reaction of 4-hydroxy-3-nitrobenzaldehyde with an equimolar quantity of 3-bromobenzohydrazide in methanol. The molecule displays an E configuration about the C═N bond. The dihedral angle between the two benzene rings is 4.6 (2)°. The nitro group is almost coplanar with the attached benzene ring [dihedral angle = 4.7 (2)°]. In the crystal structure, molecules are linked into sheets parallel to (100) by intermolecular N—H···O, O—H···N and O—H···O hydrogen bonds.

Related literature

For the crystal structures of hydrazone compounds, see: Mohd Lair *et al.* (2009); Fun *et al.* (2008); Li & Ban (2009); Zhu *et al.* (2009); Yang (2007); You *et al.* (2008). For hydrazone compounds reported previously by our group, see: Qu *et al.* (2008); Yang *et al.* (2008); Cao & Lu (2009*a*,*b*).



Experimental

Crystal data

$C_{14}H_{10}BrN_3O_4$
M_r = 364.16
Monoclinic, $P2_1/c$
a = 12.323 (1) Å
b = 13.697 (1) Å
c = 8.430 (1) Å
β = 97.133 (2)°
V = 1411.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.93 mm^{-1}
T = 298 K
0.23 × 0.21 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.552, T_{max} = 0.592 (expected range = 0.519–0.556)
8326 measured reflections
2946 independent reflections
1834 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.128
S = 1.04
2946 reflections
203 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O4^{i}	0.90 (1)	2.06 (2)	2.914 (4)	159 (4)
O3—H3···N1^{ii}	0.82	2.56	2.999 (4)	115
O3—H3···O4^{ii}	0.82	2.30	2.992 (4)	142

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The Vital Foundation of Ankang University (Project No. 2008AKXY012) and the Special Scientific Research Foundation of the Education Office of Shanxi Province (Project No. 02 J K202) are gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2834).

References

Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cao, G.-B. & Lu, X.-H. (2009*a*). *Acta Cryst.* E**65**, o1587.
Cao, G.-B. & Lu, X.-H. (2009*b*). *Acta Cryst.* E**65**, o1600.
Fun, H.-K., Patil, P. S., Rao, J. N., Kalluraya, B. & Chantrapromma, S. (2008). *Acta Cryst.* E**64**, o1707.
Li, C.-M. & Ban, H.-Y. (2009). *Acta Cryst.* E**65**, o1466.
Mohd Lair, N., Mohd Ali, H. & Ng, S. W. (2009). *Acta Cryst.* E**65**, o189.
Qu, L.-Z., Yang, T., Cao, G.-B. & Wang, X.-Y. (2008). *Acta Cryst.* E**64**, o2061.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yang, D.-S. (2007). *J. Chem. Crystallogr.* **37**, 343–348.
Yang, T., Cao, G.-B., Xiang, J.-M. & Zhang, L.-H. (2008). *Acta Cryst.* E**64**, o1186.
You, Z.-L., Dai, W.-M., Xu, X.-Q. & Hu, Y.-Q. (2008). *Pol. J. Chem.* **82**, 2215–2219.
Zhu, C.-G., Wei, Y.-J. & Zhu, Q.-Y. (2009). *Acta Cryst.* E**65**, o85.

supplementary materials

Acta Cryst. (2009). E**65**, o1725 [doi:10.1107/S1600536809024131]

(*E*)-3-Bromo-*N*'-(4-hydroxy-3-nitrobenzylidene)benzohydrazide

G.-B. Cao and X.-Y. Wang

Comment

Study on the crystal structures of hydrazone derivatives is a hot topic in structural chemistry. In the last few years, the crystal structures of a large number of hydrazone compounds have been reported (Mohd Lair *et al.*, 2009; Fun *et al.*, 2008; Li & Ban, 2009; Zhu *et al.*, 2009; Yang, 2007; You *et al.*, 2008). As a continuation of our work in this area (Qu *et al.*, 2008; Yang *et al.*, 2008; Cao & Lu, 2009a,b), the title new hydrazone compound derived from the reaction of 4-hydroxy-3-nitrobenzaldehyde with an equimolar quantity of 3-bromobenzohydrazide is reported.

In the title compound (Fig. 1), the dihedral angle between the two benzene rings is 4.6 (2)°. The molecule displays an *E* configuration about the C═N bond. In the crystal structure, molecules are linked through intermolecular N—H···O, O—H···N, and O—H···O hydrogen bonds (Table 1) to form layers parallel to the (100) (Fig. 2).

Experimental

The title compound was prepared by refluxing equimolar quantities of 4-hydroxy-3-nitrobenzaldehyde with 3-bromobenzohydrazide in methanol. Colourless block-like crystals were formed by slow evaporation of the solution in air.

Refinement

Atom H2 was located in a difference Fourier map and refined isotropically, with the N-H distance restrained to 0.90 (1) Å. The other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C-H distances of 0.93 Å, O-H distance of 0.82 Å, and with U_{iso}(H) set at 1.2U_{eq}(C) and 1.5U_{eq}(O). A rotating group model was used for the OH group.

Figures

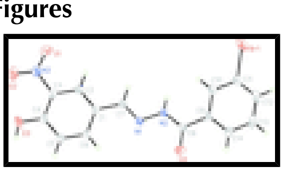


Fig. 1. The molecular structure of the title compound, showing 30% displacement ellipsoids.

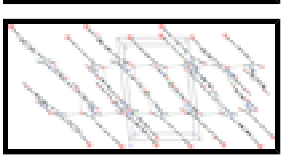


Fig. 2. The molecular packing of the title compound, viewed along the *b* axis. H atoms not involved in hydrogen bonding (dashed lines) have been omitted.

(*E*)-3-Bromo-*N*'-(4-hydroxy-3-nitrobenzylidene)benzohydrazide

Crystal data

$C_{14}H_{10}BrN_3O_4$	F_{000} = 728
M_r = 364.16	D_x = 1.713 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1828 reflections
a = 12.323 (1) Å	θ = 2.7–24.5º
b = 13.697 (1) Å	μ = 2.93 mm^{-1}
c = 8.430 (1) Å	*T* = 298 K
β = 97.133 (2)º	Block, colourless
V = 1411.9 (2) Å^3	0.23 × 0.21 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2946 independent reflections
Radiation source: fine-focus sealed tube	1834 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
T = 298 K	θ_{max} = 26.6º
ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	*h* = −14→15
T_{min} = 0.552, T_{max} = 0.592	*k* = −16→17
8326 measured reflections	*l* = −10→10

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.128	$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.7063P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
2946 reflections	$\Delta\rho_{max}$ = 0.66 e Å^{-3}
203 parameters	$\Delta\rho_{min}$ = −0.76 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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	1.07300 (4)	0.14793 (4)	0.14031 (6)	0.0779 (2)
N1	0.6951 (2)	0.3333 (2)	0.5669 (3)	0.0418 (7)
N2	0.7511 (2)	0.2683 (2)	0.4801 (3)	0.0402 (7)
N3	0.6518 (3)	0.7702 (2)	0.5895 (4)	0.0525 (8)
O1	0.7225 (3)	0.7781 (2)	0.5036 (4)	0.0735 (9)
O2	0.6110 (4)	0.8411 (2)	0.6463 (5)	0.0963 (13)
O3	0.4820 (2)	0.7296 (2)	0.7858 (4)	0.0660 (8)
H3	0.4390	0.7082	0.8440	0.099*
O4	0.7360 (3)	0.14917 (19)	0.6595 (3)	0.0731 (10)
C1	0.6432 (3)	0.4996 (2)	0.5965 (4)	0.0356 (8)
C2	0.6664 (3)	0.5944 (2)	0.5594 (4)	0.0366 (8)
H2A	0.7163	0.6068	0.4878	0.044*
C3	0.6159 (3)	0.6722 (3)	0.6279 (4)	0.0389 (8)
C4	0.5363 (3)	0.6554 (3)	0.7283 (4)	0.0444 (9)
C5	0.5151 (3)	0.5582 (3)	0.7673 (4)	0.0471 (9)
H5	0.4645	0.5452	0.8378	0.057*
C6	0.5678 (3)	0.4816 (3)	0.7031 (4)	0.0431 (9)
H6	0.5530	0.4177	0.7311	0.052*
C7	0.6986 (3)	0.4217 (2)	0.5201 (4)	0.0390 (8)
H7	0.7374	0.4368	0.4354	0.047*
C8	0.7693 (3)	0.1783 (3)	0.5364 (4)	0.0415 (9)
C9	0.8344 (3)	0.1142 (2)	0.4401 (4)	0.0373 (8)
C10	0.9097 (3)	0.1540 (3)	0.3480 (4)	0.0412 (9)
H10	0.9198	0.2212	0.3441	0.049*
C11	0.9692 (3)	0.0920 (3)	0.2625 (4)	0.0475 (9)
C12	0.9556 (3)	−0.0073 (3)	0.2673 (5)	0.0562 (11)
H12	0.9957	−0.0482	0.2084	0.067*
C13	0.8825 (3)	−0.0454 (3)	0.3592 (5)	0.0541 (11)
H13	0.8734	−0.1127	0.3633	0.065*
C14	0.8215 (3)	0.0146 (3)	0.4469 (4)	0.0442 (9)
H14	0.7723	−0.0123	0.5098	0.053*
H2	0.762 (4)	0.284 (3)	0.380 (2)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0654 (4)	0.0956 (4)	0.0811 (4)	0.0124 (3)	0.0426 (3)	−0.0031 (3)
N1	0.0517 (19)	0.0410 (18)	0.0368 (16)	0.0099 (14)	0.0213 (15)	0.0001 (13)
N2	0.0514 (18)	0.0363 (16)	0.0370 (17)	0.0089 (14)	0.0211 (15)	−0.0005 (13)
N3	0.063 (2)	0.0412 (19)	0.055 (2)	0.0061 (17)	0.0153 (18)	−0.0007 (15)
O1	0.079 (2)	0.0502 (18)	0.098 (2)	−0.0069 (15)	0.036 (2)	0.0067 (15)
O2	0.142 (3)	0.0425 (19)	0.117 (3)	0.0176 (19)	0.066 (3)	−0.0030 (17)
O3	0.0595 (19)	0.0610 (18)	0.084 (2)	0.0089 (14)	0.0343 (16)	−0.0240 (16)
O4	0.136 (3)	0.0419 (16)	0.0512 (18)	0.0135 (16)	0.0520 (19)	0.0077 (12)
C1	0.0356 (19)	0.0390 (19)	0.0329 (19)	0.0037 (15)	0.0067 (16)	−0.0029 (14)
C2	0.038 (2)	0.040 (2)	0.0325 (18)	0.0033 (16)	0.0098 (16)	0.0017 (15)
C3	0.040 (2)	0.039 (2)	0.039 (2)	0.0015 (15)	0.0091 (16)	−0.0020 (15)
C4	0.040 (2)	0.048 (2)	0.046 (2)	0.0051 (17)	0.0109 (18)	−0.0127 (17)
C5	0.046 (2)	0.053 (2)	0.046 (2)	−0.0032 (18)	0.0214 (18)	−0.0066 (17)
C6	0.047 (2)	0.041 (2)	0.044 (2)	−0.0028 (17)	0.0170 (18)	−0.0011 (16)
C7	0.044 (2)	0.042 (2)	0.0338 (19)	0.0043 (16)	0.0142 (16)	0.0030 (15)
C8	0.054 (2)	0.038 (2)	0.036 (2)	0.0040 (17)	0.0182 (18)	−0.0010 (15)
C9	0.043 (2)	0.0367 (19)	0.0326 (19)	0.0069 (15)	0.0054 (16)	−0.0014 (14)
C10	0.042 (2)	0.043 (2)	0.039 (2)	0.0085 (16)	0.0062 (17)	−0.0027 (16)
C11	0.044 (2)	0.055 (2)	0.045 (2)	0.0103 (18)	0.0102 (19)	−0.0068 (18)
C12	0.053 (3)	0.056 (3)	0.060 (3)	0.019 (2)	0.008 (2)	−0.021 (2)
C13	0.059 (3)	0.036 (2)	0.065 (3)	0.0088 (19)	−0.002 (2)	−0.0138 (18)
C14	0.046 (2)	0.042 (2)	0.044 (2)	0.0005 (17)	0.0044 (18)	−0.0029 (17)

Geometric parameters (Å, °)

Br1—C11	1.900 (4)	C4—C5	1.404 (5)
N1—C7	1.276 (4)	C5—C6	1.380 (5)
N1—N2	1.389 (4)	C5—H5	0.93
N2—C8	1.329 (4)	C6—H6	0.93
N2—H2	0.898 (10)	C7—H7	0.93
N3—O1	1.206 (4)	C8—C9	1.495 (5)
N3—O2	1.219 (4)	C9—C14	1.376 (5)
N3—C3	1.462 (5)	C9—C10	1.392 (5)
O3—C4	1.341 (4)	C10—C11	1.381 (5)
O3—H3	0.82	C10—H10	0.93
O4—C8	1.229 (4)	C11—C12	1.373 (6)
C1—C2	1.374 (5)	C12—C13	1.362 (6)
C1—C6	1.393 (5)	C12—H12	0.93
C1—C7	1.459 (5)	C13—C14	1.388 (5)
C2—C3	1.394 (5)	C13—H13	0.93
C2—H2A	0.93	C14—H14	0.93
C3—C4	1.392 (5)		
C7—N1—N2	114.0 (3)	C1—C6—H6	119.9
C8—N2—N1	118.6 (3)	N1—C7—C1	121.5 (3)

C8—N2—H2	121 (3)	N1—C7—H7	119.3
N1—N2—H2	119 (3)	C1—C7—H7	119.3
O1—N3—O2	121.9 (4)	O4—C8—N2	122.9 (3)
O1—N3—C3	118.5 (3)	O4—C8—C9	121.7 (3)
O2—N3—C3	119.6 (3)	N2—C8—C9	115.4 (3)
C4—O3—H3	109.5	C14—C9—C10	120.0 (3)
C2—C1—C6	119.2 (3)	C14—C9—C8	119.2 (3)
C2—C1—C7	118.0 (3)	C10—C9—C8	120.9 (3)
C6—C1—C7	122.8 (3)	C11—C10—C9	119.0 (3)
C1—C2—C3	120.8 (3)	C11—C10—H10	120.5
C1—C2—H2A	119.6	C9—C10—H10	120.5
C3—C2—H2A	119.6	C12—C11—C10	121.3 (4)
C4—C3—C2	120.7 (3)	C12—C11—Br1	120.5 (3)
C4—C3—N3	122.8 (3)	C10—C11—Br1	118.2 (3)
C2—C3—N3	116.6 (3)	C13—C12—C11	119.2 (4)
O3—C4—C3	121.1 (3)	C13—C12—H12	120.4
O3—C4—C5	121.2 (3)	C11—C12—H12	120.4
C3—C4—C5	117.7 (3)	C12—C13—C14	121.0 (4)
C6—C5—C4	121.3 (3)	C12—C13—H13	119.5
C6—C5—H5	119.4	C14—C13—H13	119.5
C4—C5—H5	119.4	C9—C14—C13	119.5 (4)
C5—C6—C1	120.2 (3)	C9—C14—H14	120.2
C5—C6—H6	119.9	C13—C14—H14	120.2

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4^{i}	0.90 (1)	2.06 (2)	2.914 (4)	159 (4)
O3—H3···N1^{ii}	0.82	2.56	2.999 (4)	115
O3—H3···O4^{ii}	0.82	2.30	2.992 (4)	142

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

Fig. 1

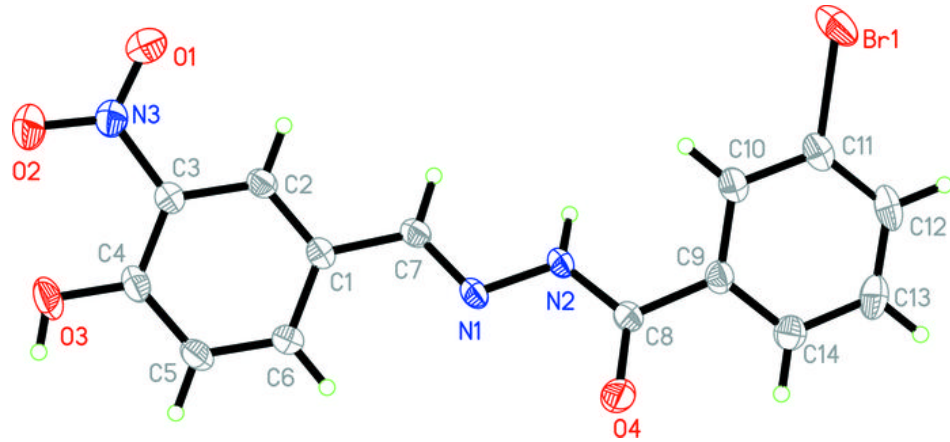

O1
N3
O2
C3
C2
C7
C4
C1
N1
N2
O3
C5
C6
Br1
C10
C11
C12
C9
C13
C8
C14
O4

Fig. 2