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(*E*)-4-Bromo-*N*′-(2-nitrobenzylidene)-benzohydrazide

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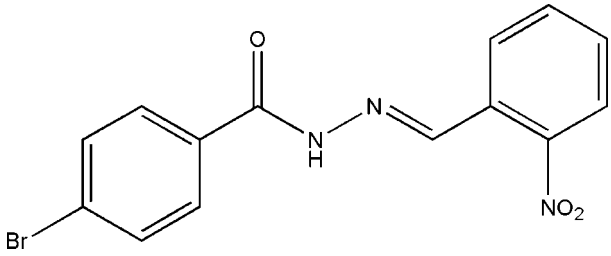
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.027; wR factor = 0.064; data-to-parameter ratio = 10.4.

The title compound, $C_{14}H_{10}BrN_3O_3$, was obtained by a condensation reaction between 2-nitrobenzaldehyde and 4-bromobenzohydrazide. The dihedral angle between the two benzene rings is 4.1 (2)°. The molecule displays an *E* configuration about the C=N bond. In the crystal, molecules are linked into a chain along [100] by intermolecular N—H···O hydrogen bonds.

Related literature

For the biological properties of Schiff base and hydrazone compounds, see: Kucukguzel *et al.* (2006); Khattab *et al.* (2005); Karthikeyan *et al.* (2006); Okabe *et al.* (1993). For bond-length data, see: Allen *et al.* (1987). For related structures, see: Shan *et al.* (2008); Fun *et al.* (2008); Ma *et al.* (2008); Diao *et al.* (2008*a*,*b*); Ejsmont *et al.* (2008).



Experimental

Crystal data

$C_{14}H_{10}BrN_3O_3$
M_r = 348.16
Triclinic, $P\overline{1}$
a = 4.8718 (17) Å
b = 6.842 (2) Å
c = 10.709 (4) Å
α = 98.014 (5)°
β = 93.258 (6)°
γ = 97.413 (5)°
V = 349.5 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 2.95 mm^{-1}
T = 298 (2) K
0.20 × 0.18 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.590, T_{max} = 0.634
2347 measured reflections
1998 independent reflections
1584 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.064
S = 0.95
1998 reflections
193 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 235 Friedel pairs
Flack parameter: 0.021 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.900 (11)	1.909 (19)	2.791 (3)	166 (6)

Symmetry code: (i) $x-1, y, z$.

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2760).

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supplementary materials

Acta Cryst. (2009). E**65**, o508 [**doi:10.1107/S1600536809002165**]

(*E*)-4-Bromo-*N*'-(2-nitrobenzylidene)benzohydrazide

M.-J. Zhang, L.-Z. Yin, D.-C. Wang, X.-M. Deng and J.-B. Liu

Comment

Hydrazones and Schiff bases have attracted much attention for their excellent biological properties, especially for their potential pharmacological and antitumor properties (Kucukguzel *et al.*, 2006; Khattab *et al.*, 2005; Karthikeyan *et al.*, 2006; Okabe *et al.*, 1993). Recently, a large number of hydrazone derivatives have been prepared and structurally characterized (Shan *et al.*, 2008; Fun *et al.*, 2008; Ma *et al.*, 2008; Diao *et al.*, 2008*a*,b; Ejsmont *et al.*, 2008). As part of the ongoing study, we report herein the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1. The dihedral angle between the two benzene rings is 4.1 (2)°. The molecule of the compound displays an *E* configuration about the C═N bond. The bond values are typical (Allen *et al.*, 1987). The molecules are linked into a chain along the [100] by intermolecular N—H···O hydrogen bonds.

Experimental

2-Nitrobenzaldehyde (1.0 mmol, 151.1 mg) was dissolved in methanol (50 ml) and then 4-bromobenzohydrazide (1.0 mmol, 215.0 mg) was added slowly into the solution, and the mixture was kept at reflux with continuous stirring for 1 h. After the solution had cooled to room temperature colourless crystals appeared. The crystals were filtered and washed with methanol for three times. Recrystallization from an absolute methanol yielded block-shaped single crystals of the title compound.

Refinement

The amide H atom was located in a difference map and its positional parameters were refined. All other H atoms were placed in calculated positions (C-H = 0.93 Å) and refined using a riding model. The U_{iso}(H) values were set at 1.2U_{eq}(C,N).

Figures

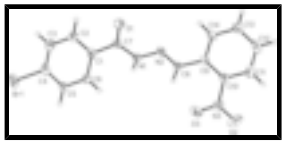

Fig. 1. The molecular structure of the title compound with 30% probability displacement ellipsoids for non-H atoms.

(*E*)-4-Bromo-*N*'-(2-nitrobenzylidene)benzohydrazide

Crystal data

$C_{14}H_{10}BrN_3O_3$	$Z = 1$
$M_r = 348.16$	$F_{000} = 174$
Triclinic, $P\overline{1}$	$D_x = 1.654$ Mg m^{-3}

Hall symbol: P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 4.8718 (17) Å	Cell parameters from 1163 reflections
b = 6.842 (2) Å	θ = 2.8–26.3º
c = 10.709 (4) Å	μ = 2.95 mm^{-1}
α = 98.014 (5)º	T = 298 K
β = 93.258 (6)º	Block, colourless
γ = 97.413 (5)º	0.20 × 0.18 × 0.17 mm
V = 349.5 (2) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1998 independent reflections
Radiation source: fine-focus sealed tube	1584 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.013
T = 298 K	θ_{max} = 30.7º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -6 \rightarrow 6$
T_{min} = 0.590, T_{max} = 0.634	$k = -8 \rightarrow 8$
2347 measured reflections	$l = -11 \rightarrow 15$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.027	$w = 1/[\sigma^2(F_o^2) + (0.0058P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.064	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.95	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
1998 reflections	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
193 parameters	Extinction correction: none
3 restraints	Absolute structure: Flack (1983), 235 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.021 (8)
Secondary atom site location: difference Fourier map	

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.28524 (6)	−0.18204 (5)	−0.03061 (4)	0.08281 (18)
O1	0.9938 (4)	0.7003 (4)	0.2644 (2)	0.0599 (6)
O2	0.1064 (8)	1.2188 (6)	0.7780 (3)	0.0860 (11)
O3	0.2286 (10)	0.9674 (5)	0.6631 (3)	0.1064 (15)
N1	0.5685 (5)	0.7559 (4)	0.3185 (2)	0.0399 (5)
H1	0.394 (9)	0.725 (7)	0.293 (4)	0.048*
N2	0.6709 (5)	0.9301 (4)	0.3980 (2)	0.0405 (5)
N3	0.2485 (7)	1.1447 (5)	0.6997 (3)	0.0584 (8)
C1	0.6174 (7)	0.4513 (5)	0.1848 (3)	0.0385 (7)
C2	0.7401 (7)	0.3754 (5)	0.0775 (3)	0.0506 (8)
H2	0.8910	0.4505	0.0495	0.061*
C3	0.6386 (8)	0.1890 (6)	0.0126 (3)	0.0564 (9)
H3	0.7180	0.1400	−0.0602	0.068*
C4	0.4217 (8)	0.0767 (5)	0.0555 (3)	0.0504 (8)
C5	0.2932 (7)	0.1486 (5)	0.1614 (3)	0.0447 (7)
H5	0.1429	0.0722	0.1889	0.054*
C6	0.3932 (6)	0.3357 (5)	0.2250 (3)	0.0420 (7)
H6	0.3090	0.3855	0.2962	0.050*
C7	0.7440 (6)	0.6473 (4)	0.2577 (3)	0.0404 (6)
C8	0.4917 (6)	1.0171 (4)	0.4562 (3)	0.0384 (6)
H8	0.3061	0.9609	0.4479	0.046*
C9	0.5831 (6)	1.2100 (4)	0.5376 (3)	0.0393 (6)
C10	0.4633 (7)	1.2766 (5)	0.6481 (3)	0.0433 (7)
C11	0.5462 (8)	1.4635 (6)	0.7158 (3)	0.0607 (9)
H11	0.4634	1.5029	0.7896	0.073*
C12	0.7486 (9)	1.5906 (6)	0.6752 (4)	0.0660 (10)
H12	0.8015	1.7178	0.7199	0.079*
C13	0.8755 (8)	1.5290 (5)	0.5668 (3)	0.0550 (9)
H13	1.0172	1.6139	0.5397	0.066*
C14	0.7919 (8)	1.3419 (6)	0.4990 (3)	0.0489 (9)
H14	0.8772	1.3031	0.4258	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1255 (4)	0.04369 (19)	0.0679 (2)	−0.00267 (18)	0.00284 (18)	−0.01585 (13)
O1	0.0231 (11)	0.0608 (15)	0.0872 (15)	−0.0003 (10)	0.0026 (10)	−0.0128 (12)
O2	0.082 (2)	0.113 (3)	0.074 (2)	0.030 (2)	0.0420 (17)	0.0233 (19)
O3	0.149 (4)	0.068 (3)	0.087 (2)	−0.047 (2)	0.049 (2)	−0.0006 (17)
N1	0.0237 (12)	0.0358 (13)	0.0545 (13)	−0.0004 (10)	−0.0015 (10)	−0.0072 (11)
N2	0.0343 (13)	0.0331 (12)	0.0494 (12)	−0.0022 (10)	−0.0040 (10)	−0.0015 (10)
N3	0.057 (2)	0.068 (2)	0.0476 (14)	−0.0014 (16)	0.0036 (14)	0.0092 (14)

C1	0.0303 (16)	0.0361 (15)	0.0469 (15)	0.0039 (13)	−0.0061 (13)	0.0027 (12)
C2	0.0450 (19)	0.050 (2)	0.0551 (17)	0.0058 (15)	0.0104 (14)	0.0015 (15)
C3	0.067 (2)	0.057 (2)	0.0417 (15)	0.0094 (18)	0.0084 (15)	−0.0075 (14)
C4	0.062 (2)	0.0360 (16)	0.0488 (16)	0.0072 (15)	−0.0087 (15)	−0.0032 (13)
C5	0.0430 (18)	0.0365 (16)	0.0515 (16)	−0.0015 (13)	0.0030 (14)	0.0024 (13)
C6	0.0379 (17)	0.0418 (17)	0.0438 (15)	0.0048 (13)	0.0031 (13)	−0.0008 (13)
C7	0.0318 (16)	0.0389 (16)	0.0484 (14)	0.0018 (12)	−0.0009 (12)	0.0038 (12)
C8	0.0322 (15)	0.0356 (15)	0.0435 (14)	−0.0016 (13)	0.0004 (12)	−0.0006 (12)
C9	0.0385 (16)	0.0325 (15)	0.0444 (14)	−0.0001 (12)	−0.0029 (12)	0.0039 (12)
C10	0.0401 (17)	0.0418 (16)	0.0464 (14)	0.0022 (13)	0.0033 (13)	0.0042 (12)
C11	0.070 (2)	0.053 (2)	0.0542 (17)	0.0114 (19)	0.0013 (17)	−0.0095 (15)
C12	0.079 (3)	0.0351 (17)	0.075 (2)	−0.0046 (18)	−0.004 (2)	−0.0064 (16)
C13	0.058 (2)	0.0404 (19)	0.0614 (19)	−0.0124 (15)	0.0002 (17)	0.0091 (15)
C14	0.0495 (18)	0.041 (2)	0.051 (2)	−0.0091 (15)	0.0001 (17)	0.0075 (18)

Geometric parameters (Å, °)

Br1—C4	1.897 (3)	C4—C5	1.387 (5)
O1—C7	1.219 (4)	C5—C6	1.377 (4)
O2—N3	1.216 (5)	C5—H5	0.93
O3—N3	1.211 (5)	C6—H6	0.93
N1—C7	1.341 (4)	C8—C9	1.478 (4)
N1—N2	1.382 (3)	C8—H8	0.93
N1—H1	0.87 (4)	C9—C14	1.391 (5)
N2—C8	1.264 (4)	C9—C10	1.395 (4)
N3—C10	1.474 (5)	C10—C11	1.378 (4)
C1—C6	1.389 (5)	C11—C12	1.360 (6)
C1—C2	1.393 (5)	C11—H11	0.93
C1—C7	1.493 (4)	C12—C13	1.386 (6)
C2—C3	1.380 (5)	C12—H12	0.93
C2—H2	0.93	C13—C14	1.380 (5)
C3—C4	1.364 (5)	C13—H13	0.93
C3—H3	0.93	C14—H14	0.93
C7—N1—N2	119.9 (2)	O1—C7—N1	122.7 (3)
C7—N1—H1	116 (3)	O1—C7—C1	121.2 (3)
N2—N1—H1	123 (3)	N1—C7—C1	116.0 (2)
C8—N2—N1	115.5 (2)	N2—C8—C9	118.6 (3)
O3—N3—O2	123.8 (4)	N2—C8—H8	120.7
O3—N3—C10	117.7 (3)	C9—C8—H8	120.7
O2—N3—C10	118.5 (4)	C14—C9—C10	116.6 (3)
C6—C1—C2	118.7 (3)	C14—C9—C8	118.6 (3)
C6—C1—C7	122.3 (3)	C10—C9—C8	124.7 (3)
C2—C1—C7	118.8 (3)	C11—C10—C9	121.9 (3)
C3—C2—C1	120.2 (3)	C11—C10—N3	117.4 (3)
C3—C2—H2	119.9	C9—C10—N3	120.7 (3)
C1—C2—H2	119.9	C12—C11—C10	120.3 (3)
C4—C3—C2	119.9 (3)	C12—C11—H11	119.8
C4—C3—H3	120.1	C10—C11—H11	119.8
C2—C3—H3	120.1	C11—C12—C13	119.5 (3)

C3—C4—C5	121.3 (3)	C11—C12—H12	120.3
C3—C4—Br1	120.4 (3)	C13—C12—H12	120.3
C5—C4—Br1	118.3 (3)	C14—C13—C12	120.1 (3)
C6—C5—C4	118.6 (3)	C14—C13—H13	119.9
C6—C5—H5	120.7	C12—C13—H13	119.9
C4—C5—H5	120.7	C13—C14—C9	121.5 (3)
C5—C6—C1	121.3 (3)	C13—C14—H14	119.2
C5—C6—H6	119.4	C9—C14—H14	119.2
C1—C6—H6	119.4		
C7—N1—N2—C8	−176.5 (3)	N2—C8—C9—C14	36.5 (4)
C6—C1—C2—C3	0.3 (5)	N2—C8—C9—C10	−147.7 (3)
C7—C1—C2—C3	175.9 (3)	C14—C9—C10—C11	0.1 (4)
C1—C2—C3—C4	−1.6 (5)	C8—C9—C10—C11	−175.8 (3)
C2—C3—C4—C5	2.2 (5)	C14—C9—C10—N3	−177.4 (3)
C2—C3—C4—Br1	−178.6 (3)	C8—C9—C10—N3	6.6 (4)
C3—C4—C5—C6	−1.4 (5)	O3—N3—C10—C11	−160.0 (4)
Br1—C4—C5—C6	179.4 (2)	O2—N3—C10—C11	18.4 (5)
C4—C5—C6—C1	0.1 (4)	O3—N3—C10—C9	17.7 (5)
C2—C1—C6—C5	0.4 (4)	O2—N3—C10—C9	−163.9 (3)
C7—C1—C6—C5	−175.0 (3)	C9—C10—C11—C12	0.6 (5)
N2—N1—C7—O1	−3.6 (4)	N3—C10—C11—C12	178.3 (4)
N2—N1—C7—C1	173.8 (2)	C10—C11—C12—C13	−1.5 (6)
C6—C1—C7—O1	143.2 (3)	C11—C12—C13—C14	1.6 (6)
C2—C1—C7—O1	−32.3 (4)	C12—C13—C14—C9	−0.8 (6)
C6—C1—C7—N1	−34.3 (4)	C10—C9—C14—C13	0.0 (5)
C2—C1—C7—N1	150.3 (3)	C8—C9—C14—C13	176.1 (3)
N1—N2—C8—C9	−176.7 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.900 (11)	1.909 (19)	2.791 (3)	166 (6)

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1