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(E)-3-Bromo-N'-(2-chlorobenzylidene)-benzohydrazide

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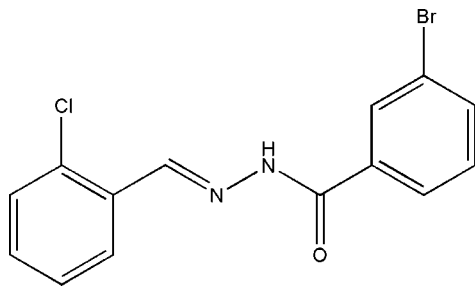
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.033; wR factor = 0.076; data-to-parameter ratio = 17.0.

The title compound, $\text{C}_{14}\text{H}_{10}\text{BrClN}_2\text{O}$, was synthesized by the reaction of 2-chlorobenzaldehyde with an equimolar quantity of 3-bromobenzohydrazide in methanol. The molecule displays an *E* configuration about the $\text{C}=\text{N}$ bond. The dihedral angle between the two benzene rings is $13.0(2)^\circ$. In the crystal structure, molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming chains propagating along the *c* axis.

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 (2009a,b).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{10}\text{BrClN}_2\text{O}$
 $M_r = 337.60$
 Monoclinic, $P2_1/c$
 $a = 13.140(1)$ Å

$b = 12.632(1)$ Å
 $c = 8.377(1)$ Å
 $\beta = 98.174(2)^\circ$
 $V = 1376.3(2)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.17$ mm⁻¹

$T = 298$ K
 $0.27 \times 0.25 \times 0.22$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.481$, $T_{\max} = 0.542$
 (expected range = 0.442–0.498)

8319 measured reflections
 2998 independent reflections
 2235 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.076$
 $S = 1.03$
 2998 reflections
 176 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.52$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.65$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O1}^i$	0.89 (1)	1.98 (1)	2.854 (2)	165 (3)

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

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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2833).

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supporting information

Acta Cryst. (2009). E65, o1705 [doi:10.1107/S160053680902412X]

(E)-3-Bromo-*N'*-(2-chlorobenzylidene)benzohydrazide

Lan-Zhu Qu and Guo-Biao Cao

S1. 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 2-chlorobenzaldehyde with an equimolar quantity of 3-bromobenzohydrazide is reported.

In the title compound (Fig. 1), the dihedral angle between the two benzene rings is 13.0 (2)°. The molecule displays an *E* configuration about the C=N bond. In the crystal structure, molecules are linked through intermolecular N—H···O hydrogen bonds (Table 1) to form chains running along the *c* axis (Fig. 2).

S2. Experimental

The title compound was prepared by refluxing equimolar quantities of 2-chlorobenzaldehyde with 3-bromobenzohydrazide in methanol. Colourless block-like crystals were formed by slow evaporation of the solution in air.

S3. 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, and with $U_{\text{iso}}(\text{H})$ set at $1.2U_{\text{eq}}(\text{C})$.

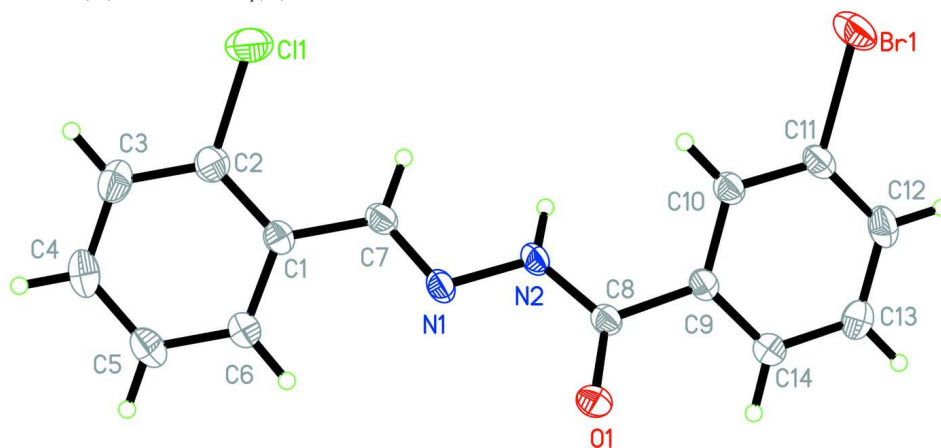
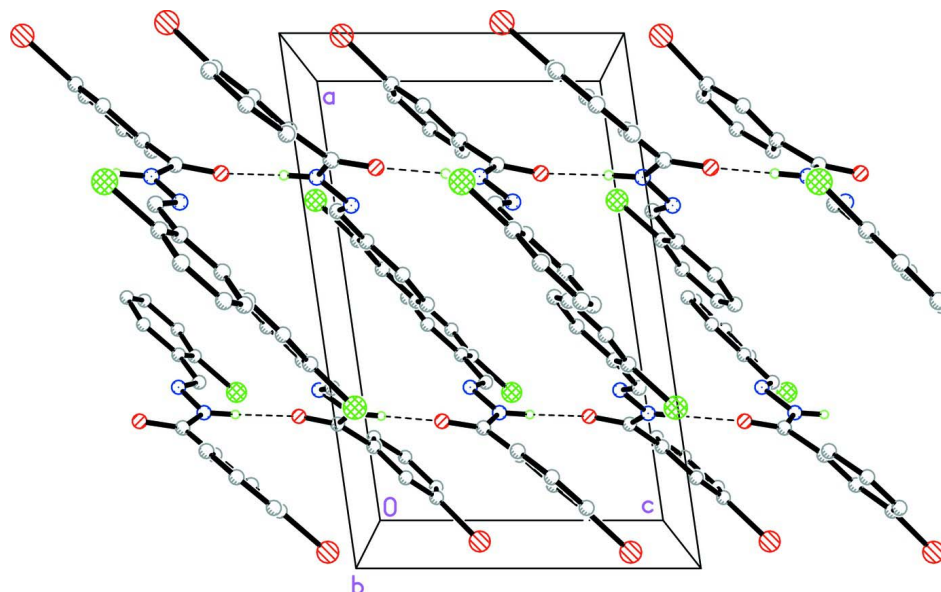


Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

**Figure 2**

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

(*E*)-3-Bromo-*N'*-(2-chlorobenzylidene)benzohydrazide

Crystal data

$C_{14}H_{10}BrClN_2O$

$M_r = 337.60$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 13.140$ (1) Å

$b = 12.632$ (1) Å

$c = 8.377$ (1) Å

$\beta = 98.174$ (2)°

$V = 1376.3$ (2) Å³

$Z = 4$

$F(000) = 672$

$D_x = 1.629$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2981 reflections

$\theta = 2.2$ – 27.5 °

$\mu = 3.17$ mm⁻¹

$T = 298$ K

Block, colourless

$0.27 \times 0.25 \times 0.22$ mm

Data collection

Bruker SMART CCD area-detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.481$, $T_{\max} = 0.542$

8319 measured reflections

2998 independent reflections

2235 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 27.0$ °, $\theta_{\min} = 1.6$ °

$h = -16 \rightarrow 15$

$k = -16 \rightarrow 13$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.076$

$S = 1.03$

2998 reflections

176 parameters

1 restraint

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

Hydrogen site location: inferred from
neighbouring sites
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0266P)^2 + 0.7889P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.52 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.65 \text{ e } \text{\AA}^{-3}$
Extinction correction: *SHELXTL* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0155 (8)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	1.03493 (2)	0.63367 (3)	0.15480 (4)	0.06855 (15)
Cl1	0.71461 (6)	1.15851 (6)	0.46918 (11)	0.0757 (3)
N1	0.69113 (14)	0.83453 (15)	0.6106 (2)	0.0358 (4)
N2	0.74414 (15)	0.76627 (15)	0.5225 (2)	0.0378 (4)
O1	0.75662 (14)	0.64019 (12)	0.71573 (18)	0.0477 (4)
C1	0.62315 (16)	1.00731 (17)	0.6291 (3)	0.0342 (5)
C2	0.62851 (18)	1.11440 (19)	0.5940 (3)	0.0433 (6)
C3	0.5678 (2)	1.1884 (2)	0.6563 (3)	0.0542 (7)
H3	0.5732	1.2596	0.6310	0.065*
C4	0.4996 (2)	1.1563 (2)	0.7553 (3)	0.0572 (7)
H4	0.4581	1.2057	0.7970	0.069*
C5	0.4924 (2)	1.0510 (2)	0.7933 (3)	0.0523 (7)
H5	0.4459	1.0293	0.8607	0.063*
C6	0.55369 (18)	0.9775 (2)	0.7319 (3)	0.0429 (6)
H6	0.5487	0.9066	0.7596	0.052*
C7	0.68317 (17)	0.92838 (18)	0.5560 (3)	0.0367 (5)
H7	0.7159	0.9469	0.4686	0.044*
C8	0.77228 (16)	0.67075 (17)	0.5825 (2)	0.0336 (5)
C9	0.82605 (15)	0.60205 (17)	0.4763 (2)	0.0319 (5)
C10	0.89265 (16)	0.64395 (18)	0.3775 (3)	0.0359 (5)
H10	0.9036	0.7166	0.3742	0.043*
C11	0.94190 (17)	0.5767 (2)	0.2851 (3)	0.0395 (5)
C12	0.9267 (2)	0.4693 (2)	0.2864 (3)	0.0487 (6)
H12	0.9603	0.4249	0.2225	0.058*
C13	0.8606 (2)	0.4287 (2)	0.3844 (3)	0.0498 (6)
H13	0.8490	0.3561	0.3859	0.060*
C14	0.81123 (18)	0.49435 (18)	0.4804 (3)	0.0411 (5)
H14	0.7679	0.4658	0.5479	0.049*
H2	0.749 (2)	0.784 (2)	0.4206 (17)	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0647 (2)	0.0867 (3)	0.0630 (2)	0.00759 (16)	0.03940 (15)	0.00891 (15)
Cl1	0.0682 (5)	0.0610 (5)	0.1056 (6)	0.0046 (4)	0.0393 (4)	0.0307 (4)
N1	0.0373 (10)	0.0395 (11)	0.0326 (9)	0.0065 (8)	0.0121 (8)	-0.0033 (8)
N2	0.0477 (11)	0.0400 (11)	0.0292 (9)	0.0105 (9)	0.0168 (8)	0.0004 (8)
O1	0.0696 (12)	0.0445 (10)	0.0339 (8)	0.0059 (8)	0.0236 (8)	0.0038 (7)
C1	0.0307 (11)	0.0393 (12)	0.0322 (11)	0.0033 (9)	0.0031 (9)	-0.0017 (9)
C2	0.0375 (12)	0.0439 (14)	0.0487 (14)	0.0022 (10)	0.0063 (10)	0.0019 (10)
C3	0.0551 (15)	0.0383 (14)	0.0686 (18)	0.0081 (12)	0.0063 (14)	-0.0028 (12)
C4	0.0551 (16)	0.0562 (17)	0.0609 (17)	0.0170 (13)	0.0102 (14)	-0.0140 (13)
C5	0.0474 (14)	0.0642 (18)	0.0484 (15)	0.0073 (13)	0.0175 (12)	-0.0042 (13)
C6	0.0429 (13)	0.0435 (14)	0.0443 (13)	0.0035 (11)	0.0127 (11)	-0.0016 (11)
C7	0.0383 (12)	0.0423 (14)	0.0314 (11)	0.0033 (10)	0.0114 (9)	0.0015 (10)
C8	0.0352 (11)	0.0369 (12)	0.0300 (11)	0.0002 (9)	0.0096 (9)	-0.0022 (9)
C9	0.0297 (11)	0.0382 (12)	0.0280 (10)	0.0051 (9)	0.0049 (8)	0.0002 (9)
C10	0.0369 (12)	0.0392 (12)	0.0329 (11)	0.0027 (10)	0.0088 (9)	-0.0006 (9)
C11	0.0341 (11)	0.0526 (15)	0.0336 (11)	0.0064 (10)	0.0112 (9)	0.0018 (10)
C12	0.0508 (14)	0.0518 (16)	0.0450 (14)	0.0149 (12)	0.0119 (11)	-0.0089 (12)
C13	0.0573 (15)	0.0353 (13)	0.0576 (15)	0.0054 (12)	0.0114 (12)	-0.0040 (11)
C14	0.0410 (13)	0.0413 (14)	0.0427 (13)	0.0021 (10)	0.0120 (10)	0.0025 (10)

Geometric parameters (\AA , $^\circ$)

Br1—C11	1.892 (2)	C5—C6	1.376 (3)
Cl1—C2	1.738 (2)	C5—H5	0.93
N1—C7	1.270 (3)	C6—H6	0.93
N1—N2	1.386 (2)	C7—H7	0.93
N2—C8	1.339 (3)	C8—C9	1.491 (3)
N2—H2	0.893 (10)	C9—C14	1.376 (3)
O1—C8	1.226 (2)	C9—C10	1.392 (3)
C1—C2	1.388 (3)	C10—C11	1.372 (3)
C1—C6	1.393 (3)	C10—H10	0.93
C1—C7	1.459 (3)	C11—C12	1.372 (4)
C2—C3	1.378 (3)	C12—C13	1.376 (4)
C3—C4	1.366 (4)	C12—H12	0.93
C3—H3	0.93	C13—C14	1.379 (3)
C4—C5	1.374 (4)	C13—H13	0.93
C4—H4	0.93	C14—H14	0.93
C7—N1—N2	114.24 (17)	N1—C7—H7	119.7
C8—N2—N1	119.58 (17)	C1—C7—H7	119.7
C8—N2—H2	122 (2)	O1—C8—N2	123.53 (19)
N1—N2—H2	118 (2)	O1—C8—C9	121.02 (19)
C2—C1—C6	116.9 (2)	N2—C8—C9	115.44 (18)
C2—C1—C7	122.0 (2)	C14—C9—C10	119.6 (2)
C6—C1—C7	121.1 (2)	C14—C9—C8	118.69 (19)

C3—C2—C1	122.1 (2)	C10—C9—C8	121.7 (2)
C3—C2—C11	118.1 (2)	C11—C10—C9	119.2 (2)
C1—C2—C11	119.79 (18)	C11—C10—H10	120.4
C4—C3—C2	119.6 (2)	C9—C10—H10	120.4
C4—C3—H3	120.2	C10—C11—C12	121.8 (2)
C2—C3—H3	120.2	C10—C11—Br1	119.05 (18)
C3—C4—C5	120.1 (2)	C12—C11—Br1	119.17 (17)
C3—C4—H4	120.0	C11—C12—C13	118.6 (2)
C5—C4—H4	120.0	C11—C12—H12	120.7
C4—C5—C6	120.2 (2)	C13—C12—H12	120.7
C4—C5—H5	119.9	C12—C13—C14	120.8 (2)
C6—C5—H5	119.9	C12—C13—H13	119.6
C5—C6—C1	121.3 (2)	C14—C13—H13	119.6
C5—C6—H6	119.4	C9—C14—C13	120.0 (2)
C1—C6—H6	119.4	C9—C14—H14	120.0
N1—C7—C1	120.54 (19)	C13—C14—H14	120.0

Hydrogen-bond geometry (Å, °)

<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>
N2—H2 \cdots O1 ⁱ	0.89 (1)	1.98 (1)	2.854 (2)	165 (3)

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