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(*E*)-3-Bromo-*N*′-(2,4-dichlorobenzylidene)benzohydrazide

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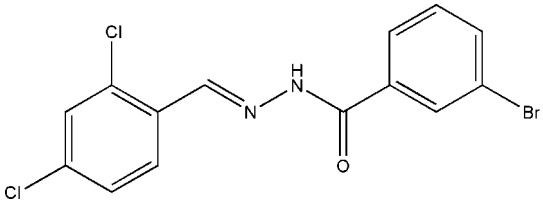
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.042; wR factor = 0.094; data-to-parameter ratio = 12.8.

The title compound, $C_{14}H_9BrCl_2N_2O$, was synthesized by the reaction of 2,4-dichlorobenzaldehyde 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 5.3 (2)°. In the crystal structure, molecules are linked through intermolecular N—H···O and C—H···O hydrogen bonds, forming chains running 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 (2009*a*,*b*); Qu & Cao (2009); Cao & Wang (2009); Cao (2009).



Experimental

Crystal data

$C_{14}H_9BrCl_2N_2O$
M_r = 372.04
Monoclinic, $P2_1/c$
a = 12.140 (2) Å
b = 14.356 (3) Å
c = 8.452 (2) Å
β = 96.019 (3)°
V = 1464.9 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.17 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.570, T_{max} = 0.600
(expected range = 0.538–0.566)
6615 measured reflections
2350 independent reflections
1561 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.094
S = 1.01
2350 reflections
184 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.64 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···O1^{i}	0.90 (1)	2.11 (3)	2.898 (4)	146 (4)
C7—H7···O1^{i}	0.93	2.32	3.134 (5)	146

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: CI2871).

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supporting information

Acta Cryst. (2009). E**65**, o2085 [doi:10.1107/S1600536809030220]

(*E*)-3-Bromo-*N*′-(2,4-dichlorobenzylidene)benzohydrazide

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, 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; Qu & Cao, 2009; Cao & Wang, 2009), the title new hydrazone compound derived from the reaction of 2,4-dichlorobenzaldehyde with an equimolar quantity of 3-bromobenzohydrazide is reported.

In the title compound (Fig. 1), the dihedral angle between the two benzene rings is 5.3 (2)°. The molecule displays an *E* configuration about the C═N bond. In the crystal structure, molecules are linked through intermolecular N—H···O and C—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 2,4-chlorobenzaldehyde with 3-bromo-benzohydrazide in methanol. Colourless block-shaped 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_{iso}(H) set at 1.2U_{eq}(C).

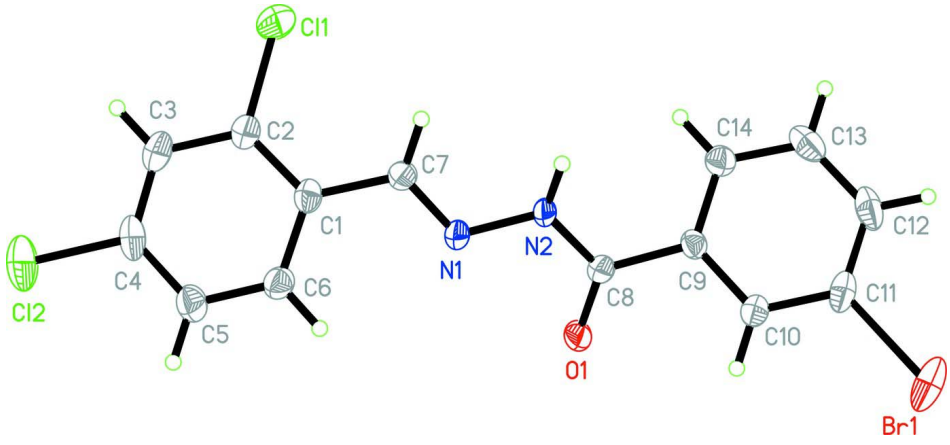

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

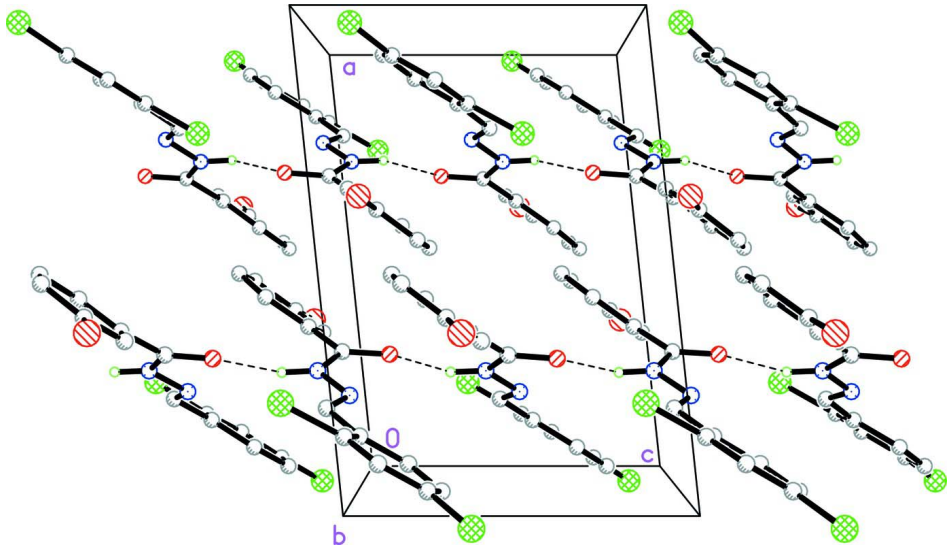

Figure 2

The molecular packing of the title compound, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines. C-bound H atoms have been omitted for clarity.

(*E*)-3-Bromo-*N*′-(2,4-dichlorobenzylidene)benzohydrazide

Crystal data

$C_{14}H_9BrCl_2N_2O$
$M_r = 372.04$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 12.140$ (2) Å
$b = 14.356$ (3) Å
$c = 8.452$ (2) Å
$\beta = 96.019$ (3)°
$V = 1464.9$ (5) Å^3
$Z = 4$
$F(000) = 736$
$D_x = 1.687$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1215 reflections
$\theta = 2.2$–24.5°
$\mu = 3.17$ mm^{-1}
$T = 298$ K
Block, colourless
0.20 × 0.20 × 0.18 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)
$T_{min} = 0.570$, $T_{max} = 0.600$
6615 measured reflections
2350 independent reflections
1561 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 24.2°$, $\theta_{min} = 2.2°$
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 15$
$l = -4 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.094$
$S = 1.01$
2350 reflections
184 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
$w = 1/[\sigma^2(F_o^2) + (0.027P)^2 + 1.2965P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.76$ e Å^{-3}

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.63560 (5)	0.68952 (3)	0.11003 (7)	0.0796 (3)
Cl1	0.77292 (12)	−0.09489 (8)	0.12692 (16)	0.0724 (4)
Cl2	1.00210 (13)	−0.17858 (10)	−0.36215 (18)	0.0857 (5)
N1	0.7760 (3)	0.1874 (2)	−0.0336 (4)	0.0359 (8)
N2	0.7287 (3)	0.2522 (2)	0.0607 (4)	0.0371 (8)
O1	0.6990 (2)	0.34967 (18)	−0.1509 (3)	0.0505 (8)
C1	0.8471 (3)	0.0368 (3)	−0.0658 (5)	0.0376 (10)
C2	0.8405 (3)	−0.0571 (3)	−0.0332 (5)	0.0422 (10)
C3	0.8859 (4)	−0.1247 (3)	−0.1250 (6)	0.0494 (12)
H3	0.8792	−0.1877	−0.1025	0.059*
C4	0.9409 (4)	−0.0953 (3)	−0.2499 (6)	0.0511 (12)
C5	0.9513 (4)	−0.0029 (3)	−0.2835 (5)	0.0503 (12)
H5	0.9904	0.0157	−0.3670	0.060*
C6	0.9041 (3)	0.0622 (3)	−0.1940 (5)	0.0434 (11)
H6	0.9100	0.1249	−0.2192	0.052*
C7	0.7967 (3)	0.1081 (3)	0.0279 (5)	0.0380 (10)
H7	0.7803	0.0953	0.1307	0.046*
C8	0.6924 (3)	0.3329 (3)	−0.0095 (5)	0.0359 (10)
C9	0.6420 (3)	0.4015 (3)	0.0939 (4)	0.0339 (9)
C10	0.6578 (3)	0.4950 (3)	0.0618 (5)	0.0378 (10)
H10	0.7001	0.5125	−0.0188	0.045*
C11	0.6103 (4)	0.5617 (3)	0.1505 (5)	0.0471 (12)
C12	0.5449 (4)	0.5370 (3)	0.2661 (5)	0.0567 (13)
H12	0.5120	0.5826	0.3237	0.068*
C13	0.5282 (4)	0.4437 (4)	0.2965 (5)	0.0548 (13)
H13	0.4832	0.4266	0.3741	0.066*
C14	0.5781 (3)	0.3756 (3)	0.2123 (5)	0.0422 (11)
H14	0.5685	0.3129	0.2353	0.051*
H2	0.732 (4)	0.243 (3)	0.1660 (16)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0888 (4)	0.0329 (3)	0.1108 (5)	0.0064 (3)	−0.0197 (3)	−0.0116 (3)
Cl1	0.0879 (10)	0.0444 (7)	0.0906 (10)	−0.0053 (7)	0.0363 (8)	0.0120 (7)
Cl2	0.0982 (11)	0.0673 (9)	0.0926 (10)	0.0235 (8)	0.0146 (9)	−0.0379 (8)
N1	0.048 (2)	0.0266 (18)	0.0342 (19)	0.0030 (16)	0.0087 (16)	−0.0037 (16)
N2	0.059 (2)	0.0253 (18)	0.0288 (18)	0.0078 (16)	0.0119 (18)	−0.0020 (16)
O1	0.084 (2)	0.0390 (17)	0.0310 (18)	0.0149 (15)	0.0173 (16)	0.0037 (13)
C1	0.041 (2)	0.034 (2)	0.037 (2)	0.0047 (19)	−0.001 (2)	−0.0054 (19)
C2	0.042 (3)	0.035 (2)	0.049 (3)	0.003 (2)	0.003 (2)	−0.003 (2)
C3	0.050 (3)	0.032 (2)	0.064 (3)	0.004 (2)	−0.003 (3)	−0.007 (2)
C4	0.047 (3)	0.049 (3)	0.056 (3)	0.015 (2)	−0.003 (2)	−0.022 (2)
C5	0.058 (3)	0.048 (3)	0.047 (3)	0.005 (2)	0.011 (2)	−0.010 (2)
C6	0.056 (3)	0.035 (2)	0.039 (3)	0.000 (2)	0.005 (2)	−0.001 (2)
C7	0.050 (3)	0.033 (2)	0.031 (2)	0.001 (2)	0.006 (2)	−0.0001 (19)
C8	0.043 (3)	0.029 (2)	0.036 (3)	−0.0030 (18)	0.007 (2)	−0.0020 (19)
C9	0.040 (2)	0.034 (2)	0.028 (2)	0.0036 (19)	0.0027 (19)	−0.0033 (18)
C10	0.041 (2)	0.032 (2)	0.039 (2)	0.0013 (19)	0.001 (2)	−0.003 (2)
C11	0.055 (3)	0.032 (2)	0.050 (3)	0.011 (2)	−0.013 (2)	−0.009 (2)
C12	0.066 (3)	0.060 (3)	0.043 (3)	0.030 (3)	0.001 (3)	−0.013 (3)
C13	0.050 (3)	0.077 (4)	0.039 (3)	0.019 (3)	0.012 (2)	0.000 (3)
C14	0.047 (3)	0.042 (2)	0.038 (3)	0.005 (2)	0.006 (2)	0.002 (2)

Geometric parameters (Å, º)

Br1—C11	1.897 (4)	C5—C6	1.365 (5)
Cl1—C2	1.741 (4)	C5—H5	0.93
Cl2—C4	1.740 (4)	C6—H6	0.93
N1—C7	1.265 (4)	C7—H7	0.93
N1—N2	1.388 (4)	C8—C9	1.489 (5)
N2—C8	1.354 (5)	C9—C14	1.380 (5)
N2—H2	0.897 (10)	C9—C10	1.386 (5)
O1—C8	1.230 (4)	C10—C11	1.380 (5)
C1—C2	1.380 (5)	C10—H10	0.93
C1—C6	1.394 (5)	C11—C12	1.369 (6)
C1—C7	1.467 (5)	C12—C13	1.382 (6)
C2—C3	1.391 (6)	C12—H12	0.93
C3—C4	1.374 (6)	C13—C14	1.386 (6)
C3—H3	0.93	C13—H13	0.93
C4—C5	1.365 (6)	C14—H14	0.93
C7—N1—N2	116.3 (3)	N1—C7—H7	120.6
C8—N2—N1	117.2 (3)	C1—C7—H7	120.6
C8—N2—H2	123 (3)	O1—C8—N2	122.8 (3)
N1—N2—H2	119 (3)	O1—C8—C9	120.9 (3)
C2—C1—C6	117.1 (4)	N2—C8—C9	116.3 (3)
C2—C1—C7	122.5 (4)	C14—C9—C10	120.2 (3)

C6—C1—C7	120.4 (4)	C14—C9—C8	123.0 (4)
C1—C2—C3	122.3 (4)	C10—C9—C8	116.8 (4)
C1—C2—Cl1	120.1 (3)	C11—C10—C9	119.5 (4)
C3—C2—Cl1	117.6 (3)	C11—C10—H10	120.3
C4—C3—C2	117.9 (4)	C9—C10—H10	120.3
C4—C3—H3	121.1	C12—C11—C10	121.0 (4)
C2—C3—H3	121.1	C12—C11—Br1	119.7 (3)
C5—C4—C3	121.4 (4)	C10—C11—Br1	119.3 (4)
C5—C4—Cl2	120.0 (4)	C11—C12—C13	119.4 (4)
C3—C4—Cl2	118.5 (4)	C11—C12—H12	120.3
C4—C5—C6	119.8 (4)	C13—C12—H12	120.3
C4—C5—H5	120.1	C12—C13—C14	120.6 (4)
C6—C5—H5	120.1	C12—C13—H13	119.7
C5—C6—C1	121.5 (4)	C14—C13—H13	119.7
C5—C6—H6	119.2	C9—C14—C13	119.4 (4)
C1—C6—H6	119.2	C9—C14—H14	120.3
N1—C7—C1	118.8 (4)	C13—C14—H14	120.3

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.90 (1)	2.11 (3)	2.898 (4)	146 (4)
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Symmetry code: (i) x, $-y+1/2$, $z+1/2$.