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

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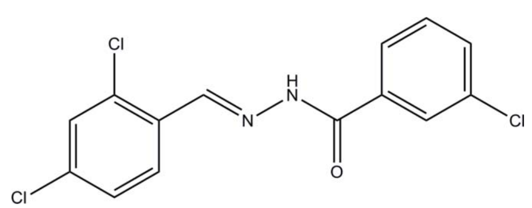
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.129; data-to-parameter ratio = 16.7.

The title compound, $C_{14}H_9Cl_3N_2O$, adopts an *E* configuration about the methylidene unit and the two aromatic rings form a dihedral angle of 6.6 (2)°. In the crystal, molecules are linked *via* N—H···O hydrogen bonds, forming *C*(4) chains propagating in [001]. C—H···O interactions reinforce the chains.

Related literature

For background to hydrazones, see: El-Asmy *et al.* (2010); El-Sherif (2009); Singh *et al.* (2009); El-Tabl *et al.* (2007). For structures of hydrazone compounds, see: Qiao *et al.* (2010); Hussain *et al.* (2010); Han & Zhao (2010); Ahmad *et al.* (2010).



Experimental

Crystal data

$C_{14}H_9Cl_3N_2O$
M_r = 327.58
Monoclinic, $P2_1/c$
a = 12.372 (3) Å
b = 14.071 (2) Å
c = 8.332 (2) Å
β = 97.582 (3)°
V = 1437.9 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.63 mm^{-1}
T = 298 K
0.30 × 0.27 × 0.27 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.833, T_{max} = 0.848
7125 measured reflections
3081 independent reflections
1776 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.129
S = 1.02
3081 reflections
184 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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)	2.08 (2)	2.900 (3)	152 (3)
C7—H7···O1^{i}	0.93	2.35	3.155 (3)	145

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5766).

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supporting information

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

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S1. Comment

Significant attention has been attracted on the hydrazones for their biological properties, coordinative capability, and applications in analytical chemistry (El-Asmy *et al.*, 2010; El-Sherif, 2009; Singh *et al.*, 2009; El-Tabl *et al.*, 2007). Recently, a number of hydrazones have been prepared and investigated for their structures (Qiao *et al.*, 2010; Hussain *et al.*, 2010; Han & Zhao, 2010; Ahmad *et al.*, 2010). As a continuation of hydrazones, the author reports herein the title new compound.

The molecule of the title compound, Fig. 1, adopts an *E* configuration about the methylidene unit. The two aromatic rings form a dihedral angle of 6.6 (2)°. In the crystal, the molecules are linked *via* intermolecular N—H···O and C—H···O hydrogen bonds (Table 1), to form chains at the *c*-axis direction (Fig. 2).

S2. Experimental

3-Chlorobenzohydrazide (1 mmol, 0.170 g) was dissolved in methanol (50 ml), then 2,4-dichlorobenzaldehyde (1 mmol, 0.174 g) was added into the solution. The reaction mixture was heated under reflux for 1 h and cooled to room temperature. Colourless needle-shaped crystals were formed by slow evaporation of the solvent for a week.

S3. Refinement

The amino H atom was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. Other H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

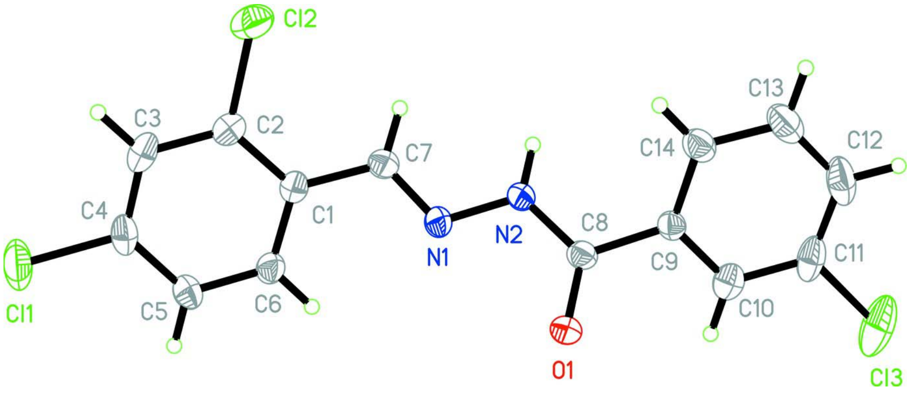

Figure 1

The molecular structure of the title compound with 30% probability displacement ellipsoids.

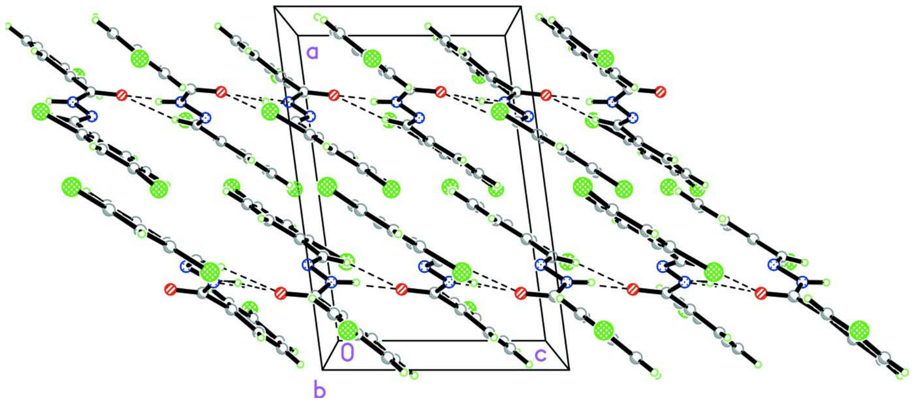

Figure 2

The packing of the title compound. Hydrogen bonding is shown in dashed lines.

3-Chloro-*N'*-(2,4-dichlorobenzylidene)benzohydrazide

Crystal data

$C_{14}H_9Cl_3N_2O$
$M_r = 327.58$
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$b = 14.071$ (2) Å
$c = 8.332$ (2) Å
$\beta = 97.582$ (3)°
$V = 1437.9$ (5) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.513$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1072 reflections
$\theta = 2.3$–25.1°
$\mu = 0.63$ mm^{-1}
$T = 298$ K
Cut from needle, colourless
0.30 × 0.27 × 0.27 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.833$, $T_{max} = 0.848$
7125 measured reflections
3081 independent reflections
1776 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.2°$
$h = -15 \rightarrow 11$
$k = -17 \rightarrow 17$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.129$
$S = 1.02$
3081 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
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.3462P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ 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}
Cl1	0.50321 (9)	1.17766 (6)	0.13090 (13)	0.0852 (4)
Cl2	0.27379 (8)	1.10398 (6)	0.61537 (13)	0.0791 (3)
Cl3	0.12211 (9)	0.31908 (6)	0.63795 (16)	0.1013 (5)
N1	0.27659 (18)	0.81292 (16)	0.4682 (3)	0.0403 (6)
N2	0.22918 (19)	0.74940 (16)	0.5631 (3)	0.0408 (6)
O1	0.20262 (17)	0.64606 (14)	0.3554 (2)	0.0544 (6)
C1	0.3473 (2)	0.96532 (19)	0.4329 (3)	0.0384 (7)
C2	0.3406 (2)	1.0619 (2)	0.4604 (4)	0.0456 (8)
C3	0.3859 (2)	1.1283 (2)	0.3661 (4)	0.0528 (8)
H3	0.3788	1.1930	0.3845	0.063*
C4	0.4413 (2)	1.0962 (2)	0.2456 (4)	0.0503 (8)
C5	0.4516 (2)	1.0012 (2)	0.2160 (4)	0.0503 (8)
H5	0.4903	0.9805	0.1342	0.060*
C6	0.4041 (2)	0.9370 (2)	0.3086 (3)	0.0435 (7)
H6	0.4102	0.8724	0.2874	0.052*
C7	0.2964 (2)	0.8950 (2)	0.5281 (4)	0.0418 (7)
H7	0.2790	0.9101	0.6302	0.050*
C8	0.1933 (2)	0.66637 (19)	0.4955 (4)	0.0396 (7)
C9	0.1403 (2)	0.59954 (19)	0.5991 (3)	0.0361 (6)
C10	0.1539 (2)	0.5032 (2)	0.5755 (4)	0.0475 (8)
H10	0.1974	0.4820	0.4999	0.057*
C11	0.1030 (3)	0.4393 (2)	0.6640 (4)	0.0582 (9)
C12	0.0359 (3)	0.4696 (3)	0.7725 (4)	0.0723 (11)
H12	0.0004	0.4258	0.8307	0.087*
C13	0.0219 (3)	0.5651 (3)	0.7939 (4)	0.0663 (10)
H13	−0.0235	0.5860	0.8671	0.080*
C14	0.0743 (2)	0.6307 (2)	0.7086 (3)	0.0490 (8)
H14	0.0650	0.6954	0.7250	0.059*
H2	0.227 (2)	0.763 (2)	0.6680 (15)	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1072 (8)	0.0612 (6)	0.0882 (8)	−0.0214 (5)	0.0174 (6)	0.0292 (5)
Cl2	0.0891 (7)	0.0575 (6)	0.0975 (8)	0.0031 (5)	0.0376 (6)	−0.0209 (5)
Cl3	0.1025 (8)	0.0464 (5)	0.1416 (11)	−0.0168 (5)	−0.0342 (7)	0.0293 (6)

N1	0.0504 (14)	0.0348 (13)	0.0360 (15)	−0.0021 (11)	0.0066 (11)	0.0053 (11)
N2	0.0556 (15)	0.0390 (13)	0.0290 (13)	−0.0088 (11)	0.0094 (12)	−0.0006 (12)
O1	0.0815 (15)	0.0482 (12)	0.0366 (13)	−0.0143 (11)	0.0201 (11)	−0.0062 (10)
C1	0.0382 (15)	0.0357 (16)	0.0393 (17)	−0.0020 (13)	−0.0017 (13)	0.0028 (13)
C2	0.0452 (17)	0.0405 (17)	0.050 (2)	0.0006 (13)	0.0034 (14)	−0.0049 (15)
C3	0.0559 (19)	0.0327 (16)	0.066 (2)	−0.0040 (14)	−0.0073 (17)	0.0029 (16)
C4	0.0522 (18)	0.0457 (18)	0.051 (2)	−0.0096 (15)	−0.0022 (16)	0.0176 (16)
C5	0.0604 (19)	0.049 (2)	0.042 (2)	−0.0016 (15)	0.0087 (15)	0.0100 (15)
C6	0.0555 (18)	0.0354 (15)	0.0388 (18)	−0.0012 (13)	0.0025 (14)	0.0029 (14)
C7	0.0488 (17)	0.0427 (17)	0.0336 (17)	−0.0007 (14)	0.0050 (13)	−0.0005 (14)
C8	0.0461 (17)	0.0397 (17)	0.0336 (18)	0.0025 (13)	0.0080 (13)	0.0014 (14)
C9	0.0393 (15)	0.0412 (16)	0.0267 (16)	−0.0059 (13)	0.0007 (12)	0.0022 (13)
C10	0.0417 (16)	0.0453 (18)	0.052 (2)	−0.0023 (13)	−0.0060 (14)	0.0035 (15)
C11	0.060 (2)	0.0458 (19)	0.061 (2)	−0.0173 (16)	−0.0191 (18)	0.0191 (17)
C12	0.074 (2)	0.090 (3)	0.049 (2)	−0.041 (2)	−0.0053 (19)	0.025 (2)
C13	0.058 (2)	0.099 (3)	0.043 (2)	−0.024 (2)	0.0126 (16)	−0.001 (2)
C14	0.0478 (17)	0.062 (2)	0.0370 (18)	−0.0102 (15)	0.0069 (14)	−0.0009 (16)

Geometric parameters (Å, º)

Cl1—C4	1.733 (3)	C5—C6	1.370 (4)
Cl2—C2	1.728 (3)	C5—H5	0.9300
Cl3—C11	1.726 (3)	C6—H6	0.9300
N1—C7	1.269 (3)	C7—H7	0.9300
N1—N2	1.375 (3)	C8—C9	1.487 (4)
N2—C8	1.347 (3)	C9—C14	1.374 (4)
N2—H2	0.896 (10)	C9—C10	1.383 (4)
O1—C8	1.222 (3)	C10—C11	1.368 (4)
C1—C2	1.382 (4)	C10—H10	0.9300
C1—C6	1.384 (4)	C11—C12	1.373 (5)
C1—C7	1.462 (4)	C12—C13	1.371 (5)
C2—C3	1.386 (4)	C12—H12	0.9300
C3—C4	1.365 (4)	C13—C14	1.377 (4)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.369 (4)	C14—H14	0.9300
C7—N1—N2	116.1 (2)	N1—C7—H7	120.7
C8—N2—N1	117.7 (2)	C1—C7—H7	120.7
C8—N2—H2	122.6 (19)	O1—C8—N2	122.4 (3)
N1—N2—H2	119.5 (19)	O1—C8—C9	120.8 (3)
C2—C1—C6	117.1 (3)	N2—C8—C9	116.8 (3)
C2—C1—C7	122.4 (3)	C14—C9—C10	120.1 (3)
C6—C1—C7	120.5 (3)	C14—C9—C8	122.0 (3)
C1—C2—C3	122.1 (3)	C10—C9—C8	117.8 (3)
C1—C2—Cl2	120.4 (2)	C11—C10—C9	119.6 (3)
C3—C2—Cl2	117.5 (2)	C11—C10—H10	120.2
C4—C3—C2	118.3 (3)	C9—C10—H10	120.2
C4—C3—H3	120.8	C10—C11—C12	120.8 (3)

C2—C3—H3	120.8	C10—C11—Cl3	119.7 (3)
C3—C4—C5	121.5 (3)	C12—C11—Cl3	119.5 (3)
C3—C4—Cl1	119.2 (2)	C13—C12—C11	119.2 (3)
C5—C4—Cl1	119.2 (3)	C13—C12—H12	120.4
C4—C5—C6	119.1 (3)	C11—C12—H12	120.4
C4—C5—H5	120.4	C12—C13—C14	120.9 (3)
C6—C5—H5	120.4	C12—C13—H13	119.5
C5—C6—C1	121.9 (3)	C14—C13—H13	119.5
C5—C6—H6	119.0	C9—C14—C13	119.3 (3)
C1—C6—H6	119.0	C9—C14—H14	120.3
N1—C7—C1	118.5 (3)	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)	2.08 (2)	2.900 (3)	152 (3)
C7—H7···O1^{i}	0.93	2.35	3.155 (3)	145

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