N′-(2,4-Dichlorobenzylidene)-2-methyl-benzohydrazide

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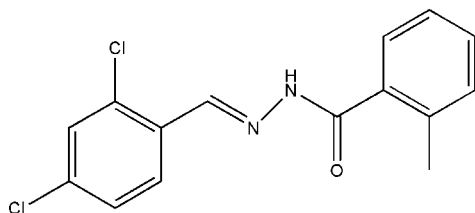
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In the title hydrazone compound, $C_{15}H_{12}Cl_2N_2O$, the dihedral angle between the two benzene rings is 12.2 (2)°. In the crystal, molecules are linked through intermolecular N—H···O hydrogen bonds, forming forming $C(4)$ chains propagating in [001].

Related literature

For general background to hydrazones, see: Rasras *et al.* (2010); Pyta *et al.* (2010); Angelusiu *et al.* (2010). For the crystal structures of related compounds, see: Fun *et al.* (2008); Singh & Singh (2010); Ahmad *et al.* (2010); Tang (2010). For reference bond-length data, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_{15}H_{12}Cl_2N_2O$	V = 1432.4 (4) Å^3
M_r = 307.17	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 7.563 (1) Å	μ = 0.45 mm^{-1}
b = 25.729 (2) Å	T = 298 K
c = 8.174 (2) Å	$0.15 \times 0.13 \times 0.10$ mm
β = 115.771 (2)°	

Data collection

Bruker SMART CCD area-detector diffractometer	7436 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3040 independent reflections
T_{min} = 0.936, T_{max} = 0.957	1529 reflections with $I > 2\sigma(I)$
	R_{int} = 0.089

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.097	
S = 0.85	
3040 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
185 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
1 restraint	

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)	2.03 (1)	2.892 (3)	159 (3)

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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: *SHELXL97*.

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supporting information

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S1. Comment

Hydrazone compounds have received much attention in biological and structural chemistry in the last few years (Rasras *et al.*, 2010; Pyta *et al.*, 2010; Angelusiu *et al.*, 2010; Fun *et al.*, 2008; Singh & Singh, 2010; Ahmad *et al.*, 2010). In the present paper, the author reports the crystal structure of the new title hydrazone compound (Fig. 1).

In the title molecule, the dihedral angle between the two benzene rings is 12.2 (2)°. The torsion angles C1—C7—N1—N2, C7—N1—N2—C8 and N1—N2—C8—C9 are 3.1 (2), 12.2 (2), and 3.0 (2)°, respectively. All the bond lengths have normal values (Allen *et al.*, 1987) and are comparable to those in the similar hydrazone compound reported recently (Tang, 2010).
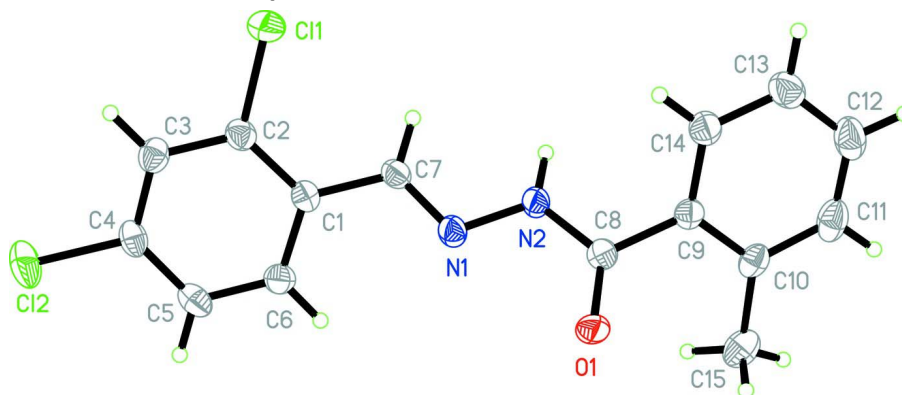
In the crystal structure of the title compound, molecules are linked through intermolecular N—H···O hydrogen bonds (Table 1), forming chains along the *c* axis (Fig. 2).

S2. Experimental

2,4-Dichlorobenzaldehyde (0.1 mmol, 19.1 mg) and 2-methylbenzohydrazide (0.1 mmol, 15.0 mg) were dissolved in methanol (20 ml). The mixture was stirred at reflux for 10 min to give a clear colourless solution. Colourless block-shaped crystals of the compound were formed by slow evaporation of the solvent over several days.

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 [U_{iso}(H) = 0.08 Å^2]. Other H atoms were constrained to ideal geometries and refined as riding, with Csp^2—H = 0.93 Å and C(methyl)—H = 0.96 Å; U_{iso}(H) = kU_{eq}(C), where k = 1.5 for methyl H and 1.2 for all other H atoms.

Figure 1

The molecular structure of the compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radius.
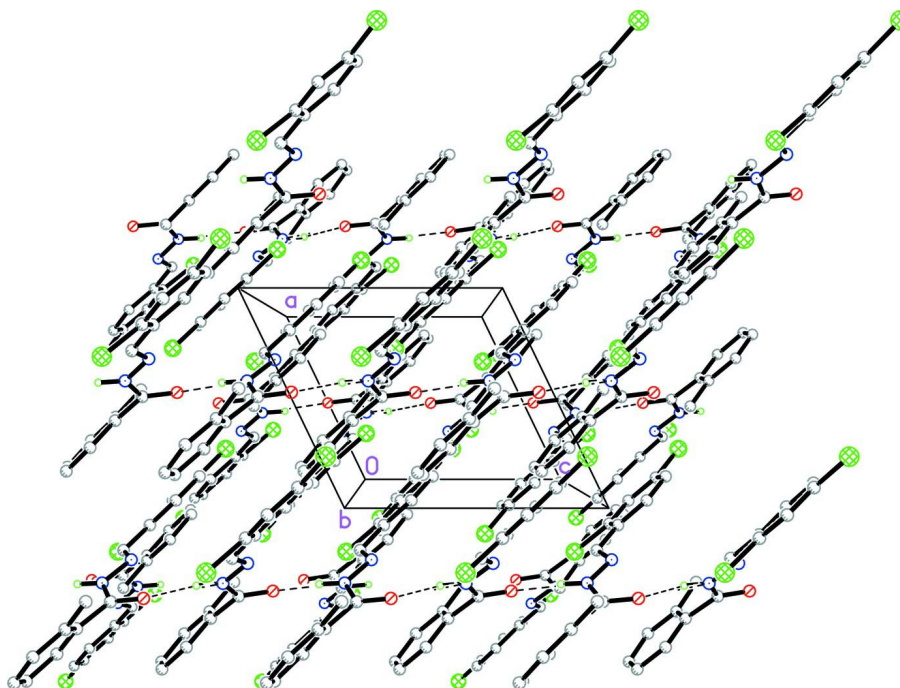
Figure 2

Molecular packing of the title compound, with hydrogen bonds shown as dashed lines.

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Crystal data

$C_{15}H_{12}Cl_2N_2O$	$F(000) = 632$
$M_r = 307.17$	$D_x = 1.424$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.563$ (1) Å	Cell parameters from 1160 reflections
$b = 25.729$ (2) Å	$\theta = 2.7$–24.3°
$c = 8.174$ (2) Å	$\mu = 0.45$ mm^{-1}
$\beta = 115.771$ (2)°	$T = 298$ K
$V = 1432.4$ (4) Å^3	Block, colourless
$Z = 4$	$0.15 \times 0.13 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	7436 measured reflections
	3040 independent reflections
Radiation source: fine-focus sealed tube	1529 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.089$
ω scans	$\theta_{max} = 27.0°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan	$h = -9 \rightarrow 6$
(*SADABS*; Sheldrick, 1996)	$k = -32 \rightarrow 28$
$T_{min} = 0.936$, $T_{max} = 0.957$	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$S = 0.85$
Least-squares matrix: full	3040 reflections
$R[F^2 > 2\sigma(F^2)] = 0.047$	185 parameters
$wR(F^2) = 0.097$	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

$w = 1/[\sigma^2(F_o^2) + (0.0246P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.30785 (12)	0.05472 (3)	0.66289 (10)	0.0762 (3)
Cl2	−0.23133 (11)	0.02731 (3)	−0.01806 (10)	0.0694 (3)
H2	0.424 (4)	0.2371 (10)	0.803 (2)	0.080*
N1	0.3080 (3)	0.21618 (9)	0.5418 (2)	0.0400 (5)
N2	0.4152 (3)	0.24626 (9)	0.6931 (3)	0.0412 (6)
O1	0.4719 (3)	0.30767 (7)	0.5227 (2)	0.0506 (5)
C1	0.1513 (3)	0.13548 (10)	0.4312 (3)	0.0354 (6)
C2	0.1547 (3)	0.08202 (11)	0.4558 (3)	0.0411 (7)
C3	0.0400 (4)	0.04854 (11)	0.3178 (3)	0.0455 (7)
H3	0.0474	0.0128	0.3363	0.055*
C4	−0.0840 (3)	0.06916 (12)	0.1541 (3)	0.0422 (7)
C5	−0.0965 (4)	0.12162 (12)	0.1234 (3)	0.0473 (7)
H5	−0.1832	0.1350	0.0114	0.057*
C6	0.0214 (4)	0.15428 (11)	0.2613 (3)	0.0438 (7)
H6	0.0142	0.1899	0.2403	0.053*
C7	0.2715 (3)	0.17057 (11)	0.5775 (3)	0.0392 (7)
H7	0.3215	0.1595	0.6976	0.047*
C8	0.4929 (4)	0.29150 (10)	0.6711 (3)	0.0375 (6)
C9	0.6141 (4)	0.31927 (10)	0.8446 (3)	0.0367 (6)
C10	0.5972 (4)	0.37312 (11)	0.8577 (3)	0.0419 (7)
C11	0.7197 (5)	0.39632 (12)	1.0223 (4)	0.0587 (8)
H11	0.7096	0.4320	1.0353	0.070*
C12	0.8536 (5)	0.36888 (15)	1.1652 (4)	0.0649 (9)
H12	0.9344	0.3859	1.2720	0.078*
C13	0.8691 (4)	0.31614 (14)	1.1512 (4)	0.0601 (9)
H13	0.9602	0.2972	1.2482	0.072*
C14	0.7481 (4)	0.29148 (11)	0.9917 (3)	0.0472 (7)
H14	0.7564	0.2556	0.9827	0.057*
C15	0.4557 (4)	0.40581 (11)	0.7048 (4)	0.0604 (9)

H15A	0.4380	0.4386	0.7516	0.091*
H15B	0.3317	0.3882	0.6475	0.091*
H15C	0.5068	0.4115	0.6174	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0772 (6)	0.0571 (6)	0.0563 (5)	−0.0056 (4)	−0.0065 (4)	0.0130 (4)
Cl2	0.0518 (5)	0.0768 (6)	0.0616 (5)	−0.0099 (4)	0.0080 (4)	−0.0297 (4)
N1	0.0466 (14)	0.0428 (15)	0.0341 (12)	−0.0087 (12)	0.0208 (10)	−0.0055 (11)
N2	0.0559 (14)	0.0402 (15)	0.0316 (12)	−0.0128 (12)	0.0229 (12)	−0.0057 (11)
O1	0.0797 (14)	0.0437 (13)	0.0342 (10)	−0.0083 (10)	0.0301 (10)	0.0013 (8)
C1	0.0309 (14)	0.0418 (18)	0.0359 (15)	−0.0042 (13)	0.0169 (12)	−0.0042 (12)
C2	0.0340 (15)	0.0457 (19)	0.0390 (15)	−0.0025 (13)	0.0116 (12)	−0.0008 (13)
C3	0.0380 (16)	0.0406 (19)	0.0550 (17)	−0.0034 (13)	0.0175 (14)	−0.0038 (14)
C4	0.0309 (15)	0.051 (2)	0.0421 (16)	−0.0033 (13)	0.0134 (13)	−0.0131 (14)
C5	0.0403 (16)	0.062 (2)	0.0339 (15)	0.0094 (15)	0.0113 (13)	−0.0041 (14)
C6	0.0496 (17)	0.0418 (19)	0.0391 (16)	0.0049 (14)	0.0185 (14)	0.0018 (13)
C7	0.0411 (16)	0.0446 (19)	0.0303 (14)	−0.0041 (14)	0.0139 (12)	−0.0009 (13)
C8	0.0429 (16)	0.0371 (18)	0.0369 (15)	0.0020 (13)	0.0213 (13)	−0.0031 (13)
C9	0.0420 (16)	0.0399 (19)	0.0355 (15)	−0.0059 (13)	0.0236 (13)	−0.0013 (12)
C10	0.0492 (17)	0.0383 (19)	0.0520 (17)	−0.0061 (14)	0.0348 (15)	−0.0031 (14)
C11	0.076 (2)	0.050 (2)	0.066 (2)	−0.0191 (18)	0.046 (2)	−0.0198 (18)
C12	0.074 (2)	0.079 (3)	0.0475 (19)	−0.032 (2)	0.0317 (19)	−0.0183 (19)
C13	0.056 (2)	0.077 (3)	0.0438 (18)	−0.0152 (18)	0.0183 (16)	0.0042 (17)
C14	0.0507 (17)	0.0504 (19)	0.0431 (16)	−0.0092 (15)	0.0228 (14)	−0.0014 (15)
C15	0.065 (2)	0.043 (2)	0.079 (2)	0.0056 (16)	0.0376 (19)	0.0034 (17)

Geometric parameters (Å, º)

Cl1—C2	1.731 (2)	C7—H7	0.9300
Cl2—C4	1.737 (2)	C8—C9	1.494 (3)
N1—C7	1.268 (3)	C9—C14	1.387 (3)
N1—N2	1.383 (3)	C9—C10	1.400 (3)
N2—C8	1.351 (3)	C10—C11	1.393 (4)
N2—H2	0.901 (10)	C10—C15	1.501 (4)
O1—C8	1.226 (3)	C11—C12	1.364 (4)
C1—C2	1.389 (3)	C11—H11	0.9300
C1—C6	1.393 (3)	C12—C13	1.371 (4)
C1—C7	1.458 (3)	C12—H12	0.9300
C2—C3	1.384 (3)	C13—C14	1.379 (3)
C3—C4	1.364 (3)	C13—H13	0.9300
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.368 (3)	C15—H15A	0.9600
C5—C6	1.377 (3)	C15—H15B	0.9600
C5—H5	0.9300	C15—H15C	0.9600
C6—H6	0.9300		

C7—N1—N2	114.4 (2)	O1—C8—C9	122.6 (2)
C8—N2—N1	118.9 (2)	N2—C8—C9	114.1 (2)
C8—N2—H2	120.6 (18)	C14—C9—C10	120.0 (2)
N1—N2—H2	120.3 (18)	C14—C9—C8	119.5 (2)
C2—C1—C6	116.7 (2)	C10—C9—C8	120.5 (2)
C2—C1—C7	121.9 (2)	C11—C10—C9	116.9 (3)
C6—C1—C7	121.4 (3)	C11—C10—C15	119.9 (3)
C3—C2—C1	122.2 (2)	C9—C10—C15	123.2 (2)
C3—C2—Cl1	117.4 (2)	C12—C11—C10	122.7 (3)
C1—C2—Cl1	120.39 (19)	C12—C11—H11	118.7
C4—C3—C2	118.5 (3)	C10—C11—H11	118.7
C4—C3—H3	120.7	C11—C12—C13	120.0 (3)
C2—C3—H3	120.7	C11—C12—H12	120.0
C3—C4—C5	121.8 (2)	C13—C12—H12	120.0
C3—C4—Cl2	118.6 (2)	C12—C13—C14	119.2 (3)
C5—C4—Cl2	119.6 (2)	C12—C13—H13	120.4
C4—C5—C6	118.9 (2)	C14—C13—H13	120.4
C4—C5—H5	120.5	C13—C14—C9	121.1 (3)
C6—C5—H5	120.5	C13—C14—H14	119.4
C5—C6—C1	121.9 (3)	C9—C14—H14	119.4
C5—C6—H6	119.0	C10—C15—H15A	109.5
C1—C6—H6	119.0	C10—C15—H15B	109.5
N1—C7—C1	120.3 (2)	H15A—C15—H15B	109.5
N1—C7—H7	119.9	C10—C15—H15C	109.5
C1—C7—H7	119.9	H15A—C15—H15C	109.5
O1—C8—N2	123.2 (2)	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^i	0.90 (1)	2.03 (1)	2.892 (3)	159 (3)

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