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(*E*)-2-Chloro-*N*′-(4-hydroxybenzylidene)-benzohydrazide

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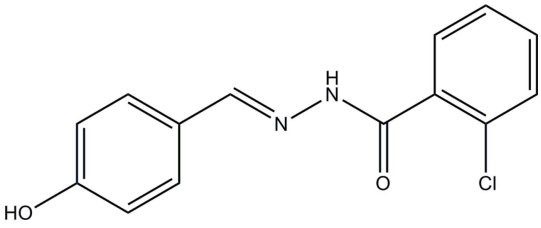
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.058; wR factor = 0.167; data-to-parameter ratio = 13.7.

The title hydrazone molecule, $C_{14}H_{11}ClN_2O_2$, has a *trans* conformation with respect to the methylidene unit. The dihedral angle between the two benzene rings is 37.6 (3)°. In the crystal, the presence of O—H···O, O—H···N and N—H···O hydrogen bonds leads to the formation of a three-dimensional network. The title compound crystallized in the chiral orthorhombic space group $P2_12_12_1$ and was refined as an inversion twin [Flack parameter = −0.20 (18)].

Related literature

For the syntheses and crystal structures of hydrazone compounds, see: Hashemian *et al.* (2011); Lei (2011); Shalash *et al.* (2010). For the crystal structures of similar compounds, reported recently by the author, see: Li (2011*a*,*b*).



Experimental

Crystal data

$C_{14}H_{11}ClN_2O_2$
M_r = 274.70
Orthorhombic, $P2_12_12_1$
a = 7.627 (3) Å
b = 11.859 (2) Å
c = 14.297 (2) Å
V = 1293.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 298 K
0.18 × 0.17 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.949, T_{max} = 0.952
6966 measured reflections
2408 independent reflections
1717 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.167
S = 1.07
2408 reflections
176 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Absolute structure: Flack (1983), 999 Friedel pairs
Flack parameter: −0.20 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.82	1.99	2.751 (4)	155
O2—H2···N2^{i}	0.82	2.48	3.012 (4)	124
N1—H1···O2^{ii}	0.90 (1)	2.12 (2)	2.987 (4)	164 (5)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); 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*.

The author is grateful to the Zibo Vocational Institute for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2376).

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supplementary materials

Acta Cryst. (2012). E**68**, o709 [doi:10.1107/S1600536812005661]

(*E*)-2-Chloro-*N*′-(4-hydroxybenzylidene)benzohydrazide

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Comment

In recent years, hydrazone compounds have attracted much attention due to their syntheses and crystal structures (Hashemian *et al.*, 2011; Lei, 2011; Shalash *et al.*, 2010). As a continuation of our work on such compounds (Li, 2011*a*,*b*), the author reports herein on the crystal structure of the new title hydrazone compound.

The molecule of the title compound (Fig. 1) exists in a *trans* conformation with respect to the methylidene unit. The dihedral angle between the (C1–C6) and (C9–C1) benzene rings is 37.6 (3)°.

In the crystal, O–H···O, O–H···N, and N–H···O hydrogen bonds leads to the formation of a three-dimensional network (Table 1, Fig. 2).

Experimental

A mixture of 4-hydroxybenzaldehyde (0.122 g, 1 mmol) and 2-chlorobenzohydrazide (0.171 g, 1 mmol) in 30 ml of methanol containing few drops of acetic acid was refluxed for about 1 h. On cooling to room temperature, a solid precipitate was formed. The solid was filtered and then recrystallized from methanol. Colourless crystals, suitable for X-ray diffraction analysis, were obtained by slow evaporation of a solution of the title compound in methanol.

Refinement

H atom H1 was located in a difference Fourier map and was freely refined. The remaining H-atoms were positioned geometrically and refined using a riding model: O–H = 0.82 Å, C–H = 0.93 Å, with U_{iso}(H) = 1.5U_{eq}(O) and = 1.2U_{eq}(C).

Computing details

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT* (Bruker, 1998); 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* (Sheldrick, 2008).

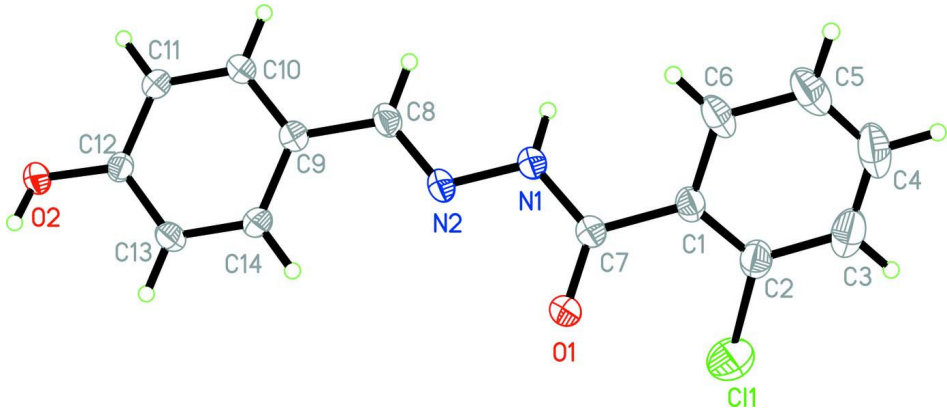

Figure 1

The molecular structure of the title compound, showing the atom labelling scheme. The displacement ellipsoids are drawn at the 30% probability level.

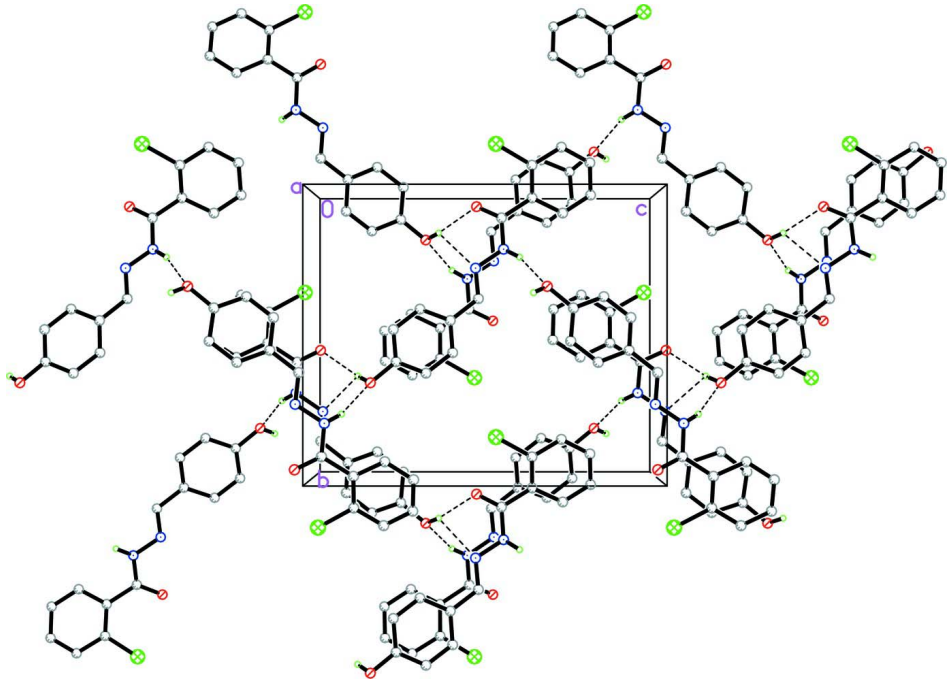

Figure 2

A view along the *a* axis of the crystal packing of the title compound. The various hydrogen bonds are indicated by dashed lines (see Table 1 for details; C-bound H-atoms have been omitted for clarity)..

(*E*)-2-Chloro-*N*′-(4-hydroxybenzylidene)benzohydrazide

Crystal data

$C_{14}H_{11}ClN_2O_2$
$M_r = 274.70$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.627$ (3) Å
$b = 11.859$ (2) Å
$c = 14.297$ (2) Å
$V = 1293.2$ (5) Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.411$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1608 reflections
$\theta = 2.2$–$24.3°$

μ = 0.29 mm^{-1}
T = 298 K

Block, colourless
0.18 × 0.17 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.949, T_{max} = 0.952

6966 measured reflections
2408 independent reflections
1717 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 25.5°, θ_{min} = 2.2°
h = −9→8
k = −14→9
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.167
S = 1.07
2408 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
$w = 1/[\sigma^2(F_o^2) + (0.0863P)^2 + 0.1245P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Absolute structure: Flack (1983), **999 Friedel pairs**
Flack parameter: −0.20 (18)

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	0.7790 (2)	1.15240 (11)	0.03121 (11)	0.0961 (6)
N1	0.8701 (5)	0.7962 (3)	0.0532 (2)	0.0446 (9)
N2	0.8920 (5)	0.7314 (3)	−0.0268 (2)	0.0442 (8)
O1	0.9755 (4)	0.9473 (2)	−0.02416 (18)	0.0479 (8)
O2	0.8755 (4)	0.3362 (2)	−0.32680 (17)	0.0439 (7)
H2	0.9466	0.3582	−0.3657	0.066*
C1	0.8856 (5)	0.9734 (3)	0.1347 (3)	0.0405 (10)
C2	0.8308 (6)	1.0829 (4)	0.1335 (3)	0.0521 (12)
C3	0.8123 (6)	1.1435 (5)	0.2166 (4)	0.0698 (15)
H3	0.7742	1.2180	0.2156	0.084*
C4	0.8506 (8)	1.0924 (6)	0.2987 (4)	0.0780 (18)
H4	0.8395	1.1328	0.3541	0.094*
C5	0.9046 (7)	0.9840 (6)	0.3022 (3)	0.0729 (16)

H5	0.9287	0.9510	0.3598	0.087*
C6	0.9240 (6)	0.9228 (5)	0.2230 (3)	0.0551 (12)
H6	0.9621	0.8484	0.2260	0.066*
C7	0.9150 (5)	0.9061 (3)	0.0472 (3)	0.0368 (9)
C8	0.8585 (5)	0.6256 (3)	−0.0187 (3)	0.0408 (9)
H8	0.8278	0.5966	0.0395	0.049*
C9	0.8678 (5)	0.5508 (3)	−0.0988 (2)	0.0375 (9)
C10	0.8151 (6)	0.4403 (3)	−0.0916 (3)	0.0430 (10)
H10	0.7773	0.4128	−0.0341	0.052*
C11	0.8172 (6)	0.3700 (3)	−0.1673 (3)	0.0433 (10)
H11	0.7812	0.2955	−0.1607	0.052*
C12	0.8724 (5)	0.4086 (3)	−0.2536 (2)	0.0335 (8)
C13	0.9259 (5)	0.5197 (3)	−0.2627 (3)	0.0387 (9)
H13	0.9637	0.5465	−0.3204	0.046*
C14	0.9231 (5)	0.5905 (3)	−0.1863 (3)	0.0413 (10)
H14	0.9581	0.6652	−0.1929	0.050*
H1	0.802 (6)	0.767 (4)	0.098 (3)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1370 (15)	0.0600 (8)	0.0912 (11)	0.0223 (9)	0.0056 (10)	0.0157 (8)
N1	0.062 (2)	0.0378 (19)	0.0339 (18)	−0.0073 (18)	0.0079 (17)	−0.0053 (14)
N2	0.062 (2)	0.0404 (19)	0.0304 (17)	−0.0049 (17)	0.0069 (18)	−0.0085 (15)
O1	0.069 (2)	0.0401 (15)	0.0349 (14)	−0.0095 (14)	0.0108 (14)	−0.0016 (13)
O2	0.063 (2)	0.0377 (15)	0.0310 (14)	−0.0025 (15)	0.0058 (13)	−0.0083 (12)
C1	0.040 (2)	0.043 (2)	0.038 (2)	−0.007 (2)	0.0070 (18)	−0.0085 (18)
C2	0.054 (3)	0.047 (3)	0.055 (3)	−0.010 (2)	0.011 (2)	−0.012 (2)
C3	0.061 (3)	0.057 (3)	0.091 (4)	−0.013 (3)	0.025 (3)	−0.027 (3)
C4	0.074 (4)	0.094 (5)	0.066 (4)	−0.027 (4)	0.015 (3)	−0.041 (3)
C5	0.078 (4)	0.102 (5)	0.039 (3)	−0.013 (4)	−0.003 (2)	−0.013 (3)
C6	0.057 (3)	0.076 (3)	0.032 (2)	−0.010 (2)	0.0049 (19)	−0.014 (2)
C7	0.039 (2)	0.038 (2)	0.033 (2)	−0.0001 (18)	0.0004 (17)	−0.0018 (17)
C8	0.048 (2)	0.044 (2)	0.0309 (19)	−0.002 (2)	−0.0013 (19)	−0.0076 (17)
C9	0.042 (2)	0.037 (2)	0.0332 (19)	0.001 (2)	0.0016 (17)	−0.0013 (17)
C10	0.061 (3)	0.038 (2)	0.0300 (19)	0.002 (2)	0.0041 (18)	0.0006 (17)
C11	0.060 (3)	0.030 (2)	0.040 (2)	0.0002 (19)	0.006 (2)	0.0002 (16)
C12	0.038 (2)	0.0296 (19)	0.0330 (19)	0.0046 (17)	−0.0029 (16)	−0.0027 (15)
C13	0.044 (2)	0.044 (2)	0.0284 (19)	0.0010 (18)	0.0038 (16)	0.0011 (17)
C14	0.054 (3)	0.030 (2)	0.040 (2)	−0.0063 (19)	0.0028 (19)	−0.0012 (17)

Geometric parameters (Å, º)

Cl1—C2	1.724 (5)	C5—C6	1.353 (6)
N1—C7	1.350 (5)	C5—H5	0.9300
N1—N2	1.388 (4)	C6—H6	0.9300
N1—H1	0.896 (10)	C8—C9	1.450 (5)
N2—C8	1.286 (5)	C8—H8	0.9300
O1—C7	1.222 (5)	C9—C10	1.375 (5)
O2—C12	1.354 (4)	C9—C14	1.402 (5)

O2—H2	0.8200	C10—C11	1.366 (5)
C1—C2	1.364 (6)	C10—H10	0.9300
C1—C6	1.429 (6)	C11—C12	1.382 (5)
C1—C7	1.500 (5)	C11—H11	0.9300
C2—C3	1.396 (7)	C12—C13	1.384 (5)
C3—C4	1.353 (8)	C13—C14	1.377 (5)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.350 (9)	C14—H14	0.9300
C4—H4	0.9300		

C7—N1—N2	116.9 (3)	O1—C7—C1	122.7 (3)
C7—N1—H1	125 (3)	N1—C7—C1	115.1 (3)
N2—N1—H1	116 (3)	N2—C8—C9	121.1 (4)
C8—N2—N1	116.2 (3)	N2—C8—H8	119.5
C12—O2—H2	109.5	C9—C8—H8	119.5
C2—C1—C6	118.3 (4)	C10—C9—C14	118.3 (3)
C2—C1—C7	122.8 (4)	C10—C9—C8	120.7 (3)
C6—C1—C7	118.8 (4)	C14—C9—C8	120.9 (3)
C1—C2—C3	120.7 (5)	C11—C10—C9	121.3 (4)
C1—C2—Cl1	122.4 (3)	C11—C10—H10	119.3
C3—C2—Cl1	116.9 (4)	C9—C10—H10	119.3
C4—C3—C2	119.1 (5)	C10—C11—C12	120.5 (3)
C4—C3—H3	120.5	C10—C11—H11	119.7
C2—C3—H3	120.5	C12—C11—H11	119.7
C5—C4—C3	121.6 (5)	O2—C12—C11	119.0 (3)
C5—C4—H4	119.2	O2—C12—C13	121.8 (3)
C3—C4—H4	119.2	C11—C12—C13	119.3 (3)
C4—C5—C6	120.9 (5)	C14—C13—C12	120.1 (3)
C4—C5—H5	119.6	C14—C13—H13	120.0
C6—C5—H5	119.6	C12—C13—H13	120.0
C5—C6—C1	119.5 (5)	C13—C14—C9	120.5 (4)
C5—C6—H6	120.3	C13—C14—H14	119.7
C1—C6—H6	120.3	C9—C14—H14	119.7
O1—C7—N1	122.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.82	1.99	2.751 (4)	155
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N1—H1···O2^{ii}	0.90 (1)	2.12 (2)	2.987 (4)	164 (5)

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