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N′-(3-Chlorobenzylidene)-4-hydroxybenzohydrazide

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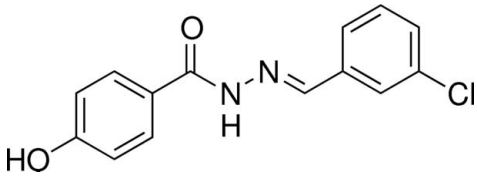
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.081; data-to-parameter ratio = 12.6.

The molecule of the title compound, $C_{14}H_{11}ClN_2O_2$ adopts an *E* conformation of the azomethine double bond and the dihedral angle between the benzene rings is 38.96 (13)°. In the crystal, molecules are linked by N—H···O and O—H···O (with the ketone O atom as acceptor) and C—H···O (with the hydroxy O atom as acceptor) hydrogen bonds, forming a three-dimensional network.

Related literature

For a related structure and background to the chemistry of the *N*-acylhydrazone unit, see: Taha *et al.* (2012). For a related structure, see: Hao (2009).



Experimental

Crystal data

$C_{14}H_{11}ClN_2O_2$
M_r = 274.70
Orthorhombic, $Pna2_1$
a = 9.0900 (8) Å
b = 9.9396 (9) Å
c = 13.8615 (12) Å
V = 1252.40 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 293 K
0.27 × 0.11 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.923, T_{max} = 0.970
6999 measured reflections
2274 independent reflections
1980 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.081
S = 1.02
2274 reflections
180 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 1060 Friedel pairs
Flack parameter: 0.12 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.84 (2)	2.20 (2)	3.026 (3)	169 (3)
O2—H2B···O1^{ii}	0.79 (3)	2.01 (3)	2.739 (3)	154 (3)
C4—H4A···O2^{iii}	0.93	2.55	3.216 (3)	128

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

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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6997).

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supporting information

Acta Cryst. (2012). E**68**, o3499 [doi:10.1107/S1600536812048325]

N′-(3-Chlorobenzylidene)-4-hydroxybenzohydrazide

Syed Muhammad Saad, Itrat Fatima, Shahnaz Perveen, Khalid M. Khan and Sammer Yousuf

S1. Comment

As part of our ongoing studies of the *N*-acylhydrazone moiety (Taha *et al.*, 2012), we now describe the structure of the title compound, which is similar to that of the previously published *N*′-(2-Chlorobenzylidene)-4-hydroxybenzohydrazide (Hao, 2009) with the difference that 2-chlorobenzne ring is replaced by 3-chlorophenyl ring (C1–C6). In the crystal, N2—H2A···O1, O2—H2B···O1,and C4—H4A···O2 hydrogen bonds link the moleucles into a three-dimensional-network (Table 2 and Fig. 2).

S2. Experimental

The title compound was synthesized by refluxing a mixture of 3-chlorobenzaldehyde (2 mmol, 0.23 ml), methanol (20 ml) and 3 drops of acetic acid. 4-hydroxybenzohydrazide (2 mmol, 0.304 g) was added into above mentioned mixture at ambient temperature and refluxed for 3 h with vigorous stirring. Progress of the reaction mixture was monitored by thin layer chromatography. After the completion of the reaction (TLC Analysis), the solvent of the reaction mixture was slowly evaporated at room temperature by keeping it in an open atmosphere in order to obtained colourless blocks (0.44 g, 80.3% yield).

S3. Refinement

H atoms on phenyl ring and methine carbon were positioned 0.93 Å (CH) and constrained to ride on their parent atoms with U_{iso}(H)= 1.2U_{eq}(CH). The H atoms on the nitrogen (N–H= 0.79 (3) Å) and oxygen (O–H= 0.89 (2) Å) atoms were located in difference fourier maps and refined isotropically.

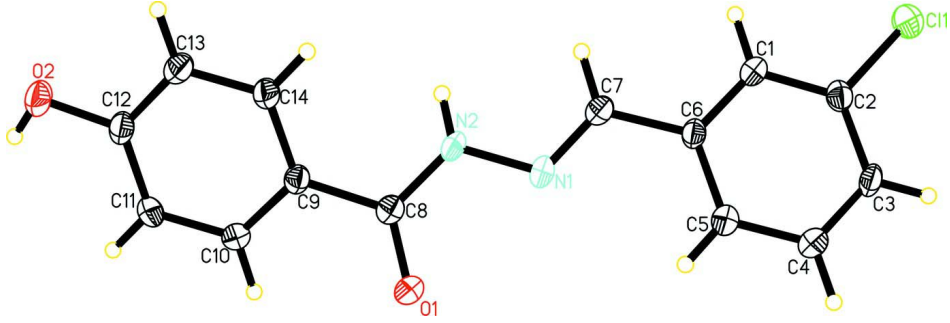

Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at 30% probability level.

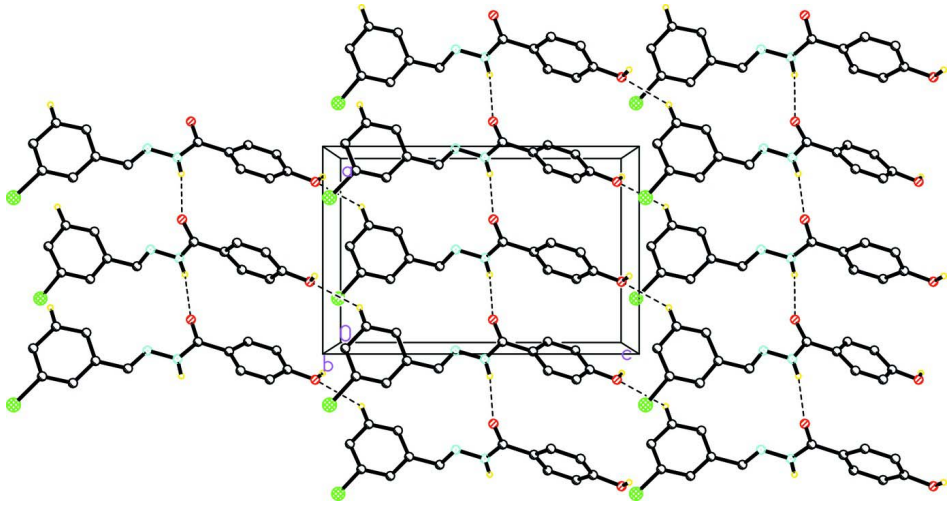

Figure 2

The crystal packing of the title compound I. Only hydrogen atoms involved in hydrogen bonding are shown.

N′-(3-Chlorobenzylidene)-4-hydroxybenzohydrazide

Crystal data

$C_{14}H_{11}ClN_2O_2$
$M_r = 274.70$
Orthorhombic, $Pna2_1$
$a = 9.0900$ (8) Å
$b = 9.9396$ (9) Å
$c = 13.8615$ (12) Å
$V = 1252.40$ (19) Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.457$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1415 reflections
$\theta = 2.5$–23.8°
$\mu = 0.30$ mm^{-1}
$T = 293$ K
Block, colorles
0.27 × 0.11 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.923$, $T_{max} = 0.970$
6999 measured reflections
2274 independent reflections
1980 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.5°$
$h = -11 \rightarrow 11$
$k = -10 \rightarrow 12$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.081$
$S = 1.02$
2274 reflections
180 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
$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983), 1060 Friedel pairs
Absolute structure parameter: 0.12 (9)

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	0.75369 (7)	0.98855 (8)	0.02100 (8)	0.0540 (2)
O1	1.15017 (17)	0.62769 (17)	0.54009 (13)	0.0374 (4)
O2	0.8459 (2)	0.6183 (2)	0.95100 (14)	0.0518 (6)
H2B	0.874 (4)	0.549 (3)	0.971 (2)	0.059 (11)*
N1	1.0036 (2)	0.7862 (2)	0.41778 (15)	0.0339 (5)
N2	0.9609 (2)	0.7697 (2)	0.51292 (16)	0.0340 (5)
H2A	0.878 (3)	0.799 (2)	0.529 (2)	0.032 (7)*
C1	0.8540 (3)	0.9246 (3)	0.19819 (17)	0.0353 (6)
H1B	0.7633	0.9546	0.2206	0.042*
C2	0.8869 (3)	0.9307 (3)	0.10113 (19)	0.0359 (6)
C3	1.0208 (3)	0.8895 (3)	0.06536 (19)	0.0391 (7)
H3A	1.0407	0.8931	−0.0004	0.047*
C4	1.1252 (3)	0.8426 (3)	0.1302 (2)	0.0437 (7)
H4A	1.2175	0.8169	0.1078	0.052*
C5	1.0943 (3)	0.8336 (3)	0.2265 (2)	0.0405 (7)
H5A	1.1654	0.8008	0.2686	0.049*
C6	0.9579 (3)	0.8729 (2)	0.26244 (17)	0.0317 (6)
C7	0.9198 (3)	0.8545 (2)	0.36386 (18)	0.0354 (6)
H7A	0.8344	0.8927	0.3887	0.043*
C8	1.0415 (3)	0.6900 (2)	0.57049 (19)	0.0307 (6)
C9	0.9925 (2)	0.6764 (2)	0.67131 (18)	0.0301 (6)
C10	1.0357 (3)	0.5631 (3)	0.72305 (19)	0.0349 (6)
H10A	1.0971	0.5003	0.6938	0.042*
C11	0.9897 (3)	0.5416 (3)	0.81628 (18)	0.0357 (6)
H11A	1.0196	0.4651	0.8496	0.043*
C12	0.8986 (3)	0.6348 (3)	0.85996 (18)	0.0364 (6)
C13	0.8576 (3)	0.7497 (3)	0.81142 (19)	0.0446 (7)
H13A	0.7991	0.8137	0.8419	0.053*
C14	0.9029 (3)	0.7698 (3)	0.71824 (18)	0.0400 (6)
H14A	0.8735	0.8471	0.6857	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0529 (4)	0.0712 (5)	0.0378 (4)	0.0143 (4)	−0.0044 (4)	0.0075 (4)
O1	0.0349 (9)	0.0442 (10)	0.0330 (11)	0.0057 (8)	0.0062 (8)	−0.0020 (8)

O2	0.0712 (14)	0.0554 (15)	0.0289 (12)	0.0118 (11)	0.0139 (10)	0.0100 (11)
N1	0.0393 (12)	0.0384 (12)	0.0240 (12)	−0.0034 (10)	0.0052 (9)	0.0007 (9)
N2	0.0333 (11)	0.0452 (13)	0.0237 (12)	0.0008 (10)	0.0092 (11)	0.0022 (11)
C1	0.0346 (13)	0.0401 (15)	0.0311 (16)	0.0029 (12)	0.0072 (11)	0.0000 (11)
C2	0.0440 (15)	0.0345 (15)	0.0292 (15)	0.0000 (11)	−0.0006 (12)	0.0019 (11)
C3	0.0500 (16)	0.0418 (16)	0.0255 (15)	0.0020 (13)	0.0134 (12)	0.0053 (12)
C4	0.0408 (15)	0.0499 (18)	0.0403 (19)	0.0084 (13)	0.0128 (13)	0.0071 (14)
C5	0.0395 (14)	0.0500 (17)	0.0321 (16)	0.0065 (13)	0.0040 (12)	0.0058 (13)
C6	0.0386 (14)	0.0308 (14)	0.0257 (15)	−0.0007 (11)	0.0042 (11)	0.0033 (12)
C7	0.0364 (14)	0.0384 (16)	0.0315 (16)	0.0025 (12)	0.0065 (12)	−0.0004 (13)
C8	0.0299 (12)	0.0322 (14)	0.0299 (15)	−0.0051 (11)	−0.0001 (10)	−0.0018 (11)
C9	0.0295 (13)	0.0356 (15)	0.0252 (15)	−0.0020 (11)	−0.0003 (10)	−0.0020 (11)
C10	0.0355 (13)	0.0359 (15)	0.0332 (16)	0.0057 (12)	0.0009 (12)	−0.0021 (12)
C11	0.0401 (14)	0.0357 (16)	0.0313 (16)	0.0039 (12)	−0.0037 (12)	0.0073 (12)
C12	0.0380 (14)	0.0457 (17)	0.0253 (15)	−0.0034 (12)	0.0009 (12)	0.0010 (13)
C13	0.0578 (17)	0.0432 (16)	0.0327 (16)	0.0138 (13)	0.0094 (13)	−0.0026 (14)
C14	0.0520 (16)	0.0368 (15)	0.0311 (16)	0.0105 (13)	0.0046 (12)	0.0051 (12)

Geometric parameters (Å, º)

Cl1—C2	1.741 (3)	C5—C6	1.392 (3)
O1—C8	1.240 (3)	C5—H5A	0.9300
O2—C12	1.359 (3)	C6—C7	1.460 (3)
O2—H2B	0.79 (3)	C7—H7A	0.9300
N1—C7	1.265 (3)	C8—C9	1.473 (3)
N1—N2	1.384 (3)	C9—C10	1.392 (3)
N2—C8	1.342 (3)	C9—C14	1.396 (3)
N2—H2A	0.84 (2)	C10—C11	1.375 (3)
C1—C2	1.380 (3)	C10—H10A	0.9300
C1—C6	1.396 (3)	C11—C12	1.382 (4)
C1—H1B	0.9300	C11—H11A	0.9300
C2—C3	1.376 (3)	C12—C13	1.377 (4)
C3—C4	1.388 (4)	C13—C14	1.370 (4)
C3—H3A	0.9300	C13—H13A	0.9300
C4—C5	1.368 (4)	C14—H14A	0.9300
C4—H4A	0.9300		
C12—O2—H2B	109 (2)	N1—C7—H7A	120.2
C7—N1—N2	117.2 (2)	C6—C7—H7A	120.2
C8—N2—N1	118.9 (2)	O1—C8—N2	121.9 (2)
C8—N2—H2A	122.6 (19)	O1—C8—C9	121.2 (2)
N1—N2—H2A	117.6 (19)	N2—C8—C9	117.0 (2)
C2—C1—C6	119.4 (2)	C10—C9—C14	117.6 (2)
C2—C1—H1B	120.3	C10—C9—C8	118.6 (2)
C6—C1—H1B	120.3	C14—C9—C8	123.9 (2)
C3—C2—C1	122.0 (2)	C11—C10—C9	121.6 (2)
C3—C2—Cl1	118.9 (2)	C11—C10—H10A	119.2
C1—C2—Cl1	119.1 (2)	C9—C10—H10A	119.2

C2—C3—C4	118.1 (2)	C10—C11—C12	119.3 (2)
C2—C3—H3A	120.9	C10—C11—H11A	120.3
C4—C3—H3A	120.9	C12—C11—H11A	120.3
C5—C4—C3	120.9 (2)	O2—C12—C13	117.2 (2)
C5—C4—H4A	119.6	O2—C12—C11	122.5 (2)
C3—C4—H4A	119.6	C13—C12—C11	120.3 (2)
C4—C5—C6	120.9 (3)	C14—C13—C12	120.0 (2)
C4—C5—H5A	119.5	C14—C13—H13A	120.0
C6—C5—H5A	119.5	C12—C13—H13A	120.0
C5—C6—C1	118.5 (2)	C13—C14—C9	121.1 (2)
C5—C6—C7	121.4 (2)	C13—C14—H14A	119.4
C1—C6—C7	120.0 (2)	C9—C14—H14A	119.4
N1—C7—C6	119.6 (2)		

C7—N1—N2—C8	175.3 (2)	N1—N2—C8—C9	179.5 (2)
C6—C1—C2—C3	−1.2 (4)	O1—C8—C9—C10	−21.1 (3)
C6—C1—C2—Cl1	177.28 (19)	N2—C8—C9—C10	157.1 (2)
C1—C2—C3—C4	−1.0 (4)	O1—C8—C9—C14	159.9 (2)
Cl1—C2—C3—C4	−179.4 (2)	N2—C8—C9—C14	−21.9 (3)
C2—C3—C4—C5	2.0 (4)	C14—C9—C10—C11	1.4 (4)
C3—C4—C5—C6	−0.8 (4)	C8—C9—C10—C11	−177.6 (2)
C4—C5—C6—C1	−1.4 (4)	C9—C10—C11—C12	−0.1 (4)
C4—C5—C6—C7	175.4 (3)	C10—C11—C12—O2	178.3 (2)
C2—C1—C6—C5	2.3 (4)	C10—C11—C12—C13	−1.7 (4)
C2—C1—C6—C7	−174.4 (2)	O2—C12—C13—C14	−177.8 (3)
N2—N1—C7—C6	−178.8 (2)	C11—C12—C13—C14	2.3 (4)
C5—C6—C7—N1	−9.2 (4)	C12—C13—C14—C9	−1.0 (4)
C1—C6—C7—N1	167.4 (2)	C10—C9—C14—C13	−0.8 (4)
N1—N2—C8—O1	−2.3 (3)	C8—C9—C14—C13	178.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.84 (2)	2.20 (2)	3.026 (3)	169 (3)
O2—H2*B*···O1^{ii}	0.79 (3)	2.01 (3)	2.739 (3)	154 (3)
C4—H4*A*···O2^{iii}	0.93	2.55	3.216 (3)	128

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