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(*E*)-4-Chloro-*N*′-(2-hydroxy-3-methoxybenzylidene)benzohydrazide monohydrate

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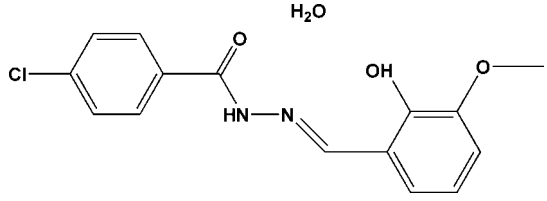
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.043; wR factor = 0.095; data-to-parameter ratio = 12.1.

In the title compound, $C_{15}H_{13}ClN_2O_3\cdot H_2O$, the molecule adopts a *trans* configuration with respect to the C=N double bond. The dihedral angle between the two rings is 15.03 (3)°. The structure is stabilized by intramolecular O—H···N and intermolecular N—H···O and O—H···O hydrogen bonds, forming an intricate three-dimensional network.

Related literature

For related compounds see: Yin & Chen (2006).



Experimental

Crystal data

$C_{15}H_{13}ClN_2O_3\cdot H_2O$
M_r = 322.74
Orthorhombic, $P2_12_12_1$
a = 4.8651 (16) Å
b = 12.9403 (17) Å
c = 22.884 (3) Å
V = 1440.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 298 (2) K
0.63 × 0.13 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.841, T_{max} = 0.972
7353 measured reflections
2522 independent reflections
1689 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.095
S = 0.97
2522 reflections
208 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 1003 Friedel pairs
Flack parameter: 0.08 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4^{i}	0.86	2.06	2.903 (3)	165
O2—H2···N2	0.82	1.99	2.694 (3)	144
O2—H2···O4	0.82	2.64	3.040 (4)	112
O4—H16···O1	0.84 (3)	1.914 (18)	2.725 (3)	161 (4)
O4—H17···O2^{ii}	0.85 (3)	2.36 (3)	3.057 (4)	140 (3)
O4—H17···O3^{ii}	0.85 (3)	2.35 (3)	3.076 (3)	144 (4)

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); *ORTEP-3 for Windows* (Farrugia, 1997); *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997*b*).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2215).

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supplementary materials

Acta Cryst. (2007). E**63**, o3548 [**doi:10.1107/S1600536807033168**]

(*E*)-4-Chloro-*N*'-(2-hydroxy-3-methoxybenzylidene)benzohydrazide monohydrate

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Comment

Recently, we have reported some organotin(IV) complexes with Schiff base of *o*-vanillin-2-thiophenoylhydrazone (Yin & Chen, 2006). As an extension of our work on the structural characterization of Schiff base compounds, the title compound, (I), is reported here (Scheme).

The molecule adopts a *trans* conformation with respect to the C8=N2 double bond length of 1.282 (4) Å (Fig. 1). The C1—N1 bond [1.350 (4) Å] and N1—N2 bond [1.380 (3) Å] are intermediate between a double bond and a single bond because of conjugation effects in the molecule. The two benzene rings make a dihedral angle of 15.0 (2) Å.

The occurrence of N—H···O and O—H···O hydrogen bonds (Table 1) results in the formation of an intricated three dimensionnal network (Fig. 2).

Experimental

Compound (I) was synthesized by the reaction of 4-chlorobenzohydrazide (5 mmol) with 2-hydroxy-3-methoxybenzaldehyde (5 mmol). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) and N—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(C or N). H atoms of water molecule were located in difference Fourier maps and included in the subsequent refinement using restraints (O—H= 0.85 (1)Å and H···H= 1.39 (2) Å) with U_{iso}(H) = 1.5U_{eq}(O).

Figures

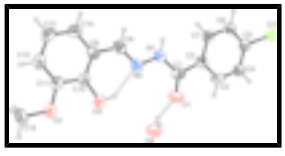

Fig. 1. Molecular structure of (I), with atom-labelling scheme. Ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii. H bonds are shown as dashed lines.

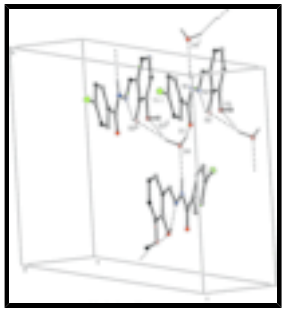

Fig. 2. Partial packing view showing the inticated N—H···O and O—H···O hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) 1 − *x*, 1/2 + *y*, 3/2 − *z*; (ii) *x* − 1, *y*, *z*].

(*E*)-4-chloro-*N*'-(2-hydroxy-3-methoxybenzylidene)benzohydrazide monohydrate

Crystal data

$C_{15}H_{13}ClN_2O_3 \cdot H_2O$	$F_{000} = 672$
$M_r = 322.74$	$D_x = 1.488$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 1656 reflections
$a = 4.8651$ (16) Å	$\theta = 3.1$–21.9º
$b = 12.9403$ (17) Å	$\mu = 0.29$ mm^{-1}
$c = 22.884$ (3) Å	$T = 298$ (2) K
$V = 1440.7$ (5) Å^3	Block, colourless
$Z = 4$	0.63 × 0.13 × 0.10 mm

Data collection

CCD area-detector diffractometer	2522 independent reflections
Radiation source: fine-focus sealed tube	1689 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.065$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.8$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -5 \rightarrow 5$
$T_{min} = 0.841$, $T_{max} = 0.972$	$k = -15 \rightarrow 11$
7353 measured reflections	$l = -27 \rightarrow 25$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.095$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.97$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
2522 reflections	$\Delta\rho_{min} = -0.21$ e Å^{-3}
208 parameters	Extinction correction: none
3 restraints	Absolute structure: Flack (1983), 1003 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.08 (10)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	−0.44005 (19)	0.94535 (6)	0.99650 (3)	0.0487 (3)
N1	0.5212 (6)	0.89997 (19)	0.78551 (10)	0.0398 (8)
H1	0.4895	0.9640	0.7930	0.048*
N2	0.7123 (6)	0.8731 (2)	0.74363 (11)	0.0393 (7)
O1	0.4210 (6)	0.73389 (17)	0.80590 (10)	0.0599 (8)
O2	1.0116 (6)	0.75187 (16)	0.67217 (9)	0.0472 (6)
H2	0.9081	0.7627	0.6998	0.071*
O3	1.3785 (5)	0.73687 (17)	0.59184 (10)	0.0511 (7)
O4	0.5138 (6)	0.62346 (17)	0.70642 (9)	0.0569 (8)
H16	0.522 (9)	0.662 (2)	0.7362 (9)	0.085*
H17	0.404 (7)	0.646 (3)	0.6806 (11)	0.085*
C1	0.3841 (8)	0.8254 (3)	0.81471 (14)	0.0408 (9)
C2	0.1830 (7)	0.8619 (2)	0.85941 (13)	0.0334 (8)
C3	0.0707 (8)	0.7873 (2)	0.89629 (13)	0.0445 (9)
H3	0.1256	0.7188	0.8923	0.053*
C4	−0.1173 (8)	0.8124 (3)	0.93798 (14)	0.0439 (10)
H4	−0.1894	0.7616	0.9623	0.053*
C5	−0.1999 (7)	0.9138 (3)	0.94387 (12)	0.0358 (9)
C6	−0.0920 (8)	0.9884 (2)	0.90805 (13)	0.0380 (9)
H6	−0.1482	1.0567	0.9121	0.046*
C7	0.0972 (7)	0.9631 (2)	0.86649 (13)	0.0389 (9)
H7	0.1695	1.0145	0.8426	0.047*
C8	0.8421 (8)	0.9500 (3)	0.72158 (13)	0.0411 (9)
H8	0.8010	1.0163	0.7346	0.049*
C9	1.0492 (7)	0.9369 (2)	0.67743 (12)	0.0350 (8)
C10	1.1250 (7)	0.8417 (2)	0.65468 (12)	0.0341 (9)
C11	1.3243 (7)	0.8360 (3)	0.61088 (13)	0.0379 (9)
C12	1.4518 (8)	0.9226 (3)	0.59104 (14)	0.0441 (9)
H12	1.5860	0.9177	0.5622	0.053*
C13	1.3812 (9)	1.0186 (3)	0.61392 (15)	0.0531 (11)
H13	1.4685	1.0780	0.6006	0.064*
C14	1.1836 (8)	1.0251 (3)	0.65588 (15)	0.0481 (10)
H14	1.1363	1.0897	0.6707	0.058*

C15	1.6082 (8)	0.7249 (3)	0.55397 (15)	0.0567 (11)
H15A	1.7717	0.7478	0.5736	0.085*
H15B	1.5805	0.7654	0.5193	0.085*
H15C	1.6273	0.6534	0.5435	0.085*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0443 (5)	0.0541 (6)	0.0476 (5)	−0.0028 (5)	0.0090 (5)	−0.0020 (4)
N1	0.042 (2)	0.0359 (17)	0.0417 (15)	0.0058 (15)	0.0079 (15)	−0.0073 (13)
N2	0.0376 (19)	0.0410 (19)	0.0393 (16)	0.0035 (16)	0.0042 (15)	−0.0046 (14)
O1	0.087 (2)	0.0306 (14)	0.0618 (15)	0.0113 (15)	0.0204 (17)	0.0000 (12)
O2	0.0457 (17)	0.0405 (14)	0.0555 (15)	−0.0067 (13)	0.0194 (13)	0.0013 (11)
O3	0.0486 (18)	0.0449 (15)	0.0597 (15)	−0.0042 (13)	0.0200 (14)	−0.0067 (12)
O4	0.074 (2)	0.0400 (15)	0.0571 (16)	0.0051 (16)	0.0115 (16)	−0.0022 (12)
C1	0.048 (2)	0.037 (2)	0.0376 (19)	0.0046 (19)	−0.004 (2)	0.0051 (17)
C2	0.033 (2)	0.034 (2)	0.0331 (17)	0.0000 (17)	−0.0048 (17)	−0.0034 (15)
C3	0.055 (3)	0.0281 (19)	0.051 (2)	0.001 (2)	−0.001 (2)	0.0007 (16)
C4	0.048 (3)	0.036 (2)	0.048 (2)	0.0012 (19)	0.013 (2)	0.0072 (16)
C5	0.032 (2)	0.042 (2)	0.0329 (18)	−0.0028 (18)	−0.0028 (17)	0.0009 (16)
C6	0.036 (2)	0.0316 (19)	0.0466 (19)	0.0033 (18)	0.001 (2)	−0.0030 (17)
C7	0.046 (2)	0.033 (2)	0.0378 (18)	−0.0042 (19)	0.0059 (19)	0.0051 (15)
C8	0.037 (2)	0.043 (2)	0.0434 (19)	0.005 (2)	−0.0043 (18)	−0.0045 (17)
C9	0.0310 (19)	0.038 (2)	0.0362 (17)	0.0080 (19)	−0.0043 (18)	0.0007 (16)
C10	0.032 (2)	0.035 (2)	0.0347 (19)	−0.0038 (18)	−0.0011 (17)	0.0034 (15)
C11	0.038 (2)	0.041 (2)	0.0351 (18)	−0.0034 (19)	−0.0005 (18)	−0.0002 (16)
C12	0.038 (2)	0.052 (2)	0.0419 (18)	−0.004 (2)	0.0014 (19)	0.0037 (18)
C13	0.052 (3)	0.042 (2)	0.065 (2)	−0.009 (2)	0.006 (2)	0.0122 (19)
C14	0.047 (3)	0.039 (2)	0.059 (2)	0.0032 (19)	−0.006 (2)	−0.0006 (17)
C15	0.043 (3)	0.069 (3)	0.058 (2)	0.003 (2)	0.015 (2)	−0.016 (2)

Geometric parameters (Å, °)

Cl1—C5	1.727 (3)	C5—C6	1.371 (4)
N1—C1	1.350 (4)	C6—C7	1.363 (4)
N1—N2	1.380 (3)	C6—H6	0.9300
N1—H1	0.8600	C7—H7	0.9300
N2—C8	1.282 (4)	C8—C9	1.437 (5)
O1—C1	1.215 (4)	C8—H8	0.9300
O2—C10	1.348 (3)	C9—C10	1.387 (4)
O2—H2	0.8200	C9—C14	1.405 (5)
O3—C11	1.380 (4)	C10—C11	1.397 (4)
O3—C15	1.422 (4)	C11—C12	1.359 (4)
O4—H16	0.84 (3)	C12—C13	1.391 (5)
O4—H17	0.85 (3)	C12—H12	0.9300
C1—C2	1.492 (4)	C13—C14	1.361 (5)
C2—C7	1.384 (4)	C13—H13	0.9300
C2—C3	1.394 (4)	C14—H14	0.9300
C3—C4	1.361 (5)	C15—H15A	0.9600

C3—H3	0.9300	C15—H15B	0.9600
C4—C5	1.379 (5)	C15—H15C	0.9600
C4—H4	0.9300		
C1—N1—N2	119.8 (3)	N2—C8—C9	122.1 (3)
C1—N1—H1	120.1	N2—C8—H8	119.0
N2—N1—H1	120.1	C9—C8—H8	119.0
C8—N2—N1	114.2 (3)	C10—C9—C14	117.8 (3)
C10—O2—H2	109.5	C10—C9—C8	123.7 (3)
C11—O3—C15	116.3 (3)	C14—C9—C8	118.5 (3)
H16—O4—H17	113 (2)	O2—C10—C9	123.1 (3)
O1—C1—N1	122.8 (3)	O2—C10—C11	116.8 (3)
O1—C1—C2	121.3 (3)	C9—C10—C11	120.1 (3)
N1—C1—C2	115.9 (3)	C12—C11—O3	125.0 (3)
C7—C2—C3	117.8 (3)	C12—C11—C10	120.8 (3)
C7—C2—C1	125.3 (3)	O3—C11—C10	114.1 (3)
C3—C2—C1	116.9 (3)	C11—C12—C13	119.9 (3)
C4—C3—C2	121.5 (3)	C11—C12—H12	120.1
C4—C3—H3	119.2	C13—C12—H12	120.1
C2—C3—H3	119.2	C14—C13—C12	119.7 (4)
C3—C4—C5	119.4 (3)	C14—C13—H13	120.2
C3—C4—H4	120.3	C12—C13—H13	120.2
C5—C4—H4	120.3	C13—C14—C9	121.7 (3)
C6—C5—C4	120.0 (3)	C13—C14—H14	119.1
C6—C5—Cl1	120.6 (3)	C9—C14—H14	119.1
C4—C5—Cl1	119.4 (3)	O3—C15—H15A	109.5
C7—C6—C5	120.4 (3)	O3—C15—H15B	109.5
C7—C6—H6	119.8	H15A—C15—H15B	109.5
C5—C6—H6	119.8	O3—C15—H15C	109.5
C6—C7—C2	120.8 (3)	H15A—C15—H15C	109.5
C6—C7—H7	119.6	H15B—C15—H15C	109.5
C2—C7—H7	119.6		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4^{i}	0.86	2.06	2.903 (3)	165
O2—H2···N2	0.82	1.99	2.694 (3)	144
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O4—H17···O2^{ii}	0.85 (3)	2.36 (3)	3.057 (4)	140 (3)
O4—H17···O3^{ii}	0.85 (3)	2.35 (3)	3.076 (3)	144 (4)

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

Fig. 1

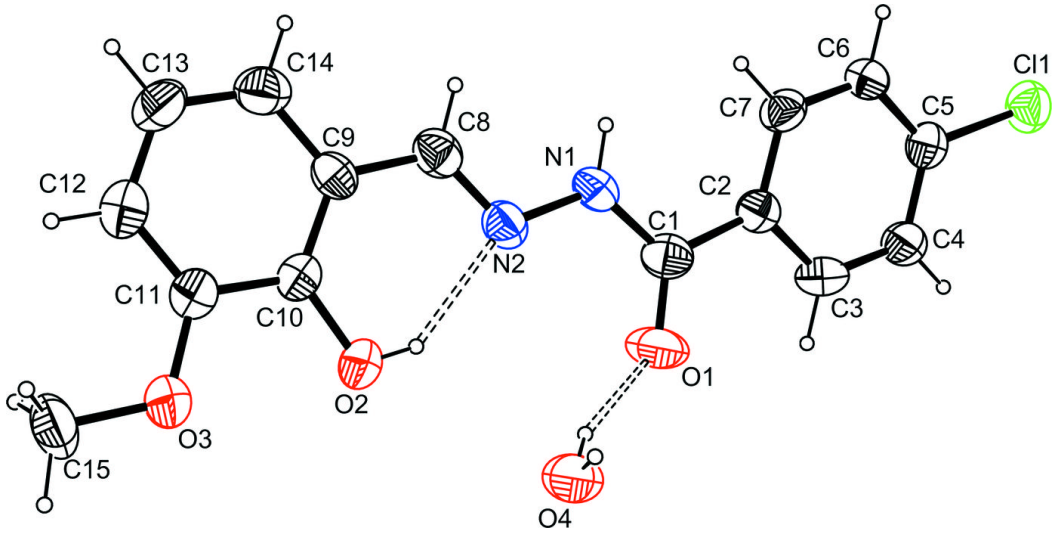
C13
C14
C9
C8
N1
C6
Cl1
C7
C5
C12
C2
C1
C4
N2
C11
C10
C3
O1
O2
O3
C15
O4

Fig. 2