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4-Chloro-*N*′-[(*E*)-2-chlorobenzylidene]-benzohydrazide monohydrate

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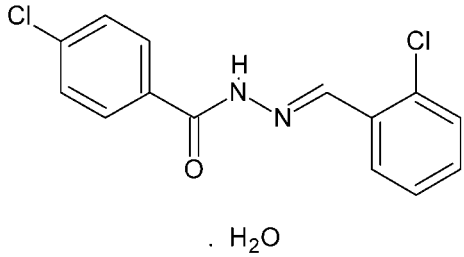
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.097; data-to-parameter ratio = 19.4.

The title compound, $C_{14}H_{10}Cl_2N_2O\cdot H_2O$, has a nearly planar extended conformation [C—N—N—C = −173.66 (15)°]. The dihedral angle between the aromatic rings is 4.6 (2)°. The water molecules alternate with benzohydrazide molecules in chains formed by O—H···O hydrogen bonds which run parallel to the *a* axis. These chains are linked to neighboring chains through N—H···O and C—H···O interactions, forming a layer parallel to (001).

Related literature

For the biological activity of hydrazone compounds, see: Koopaei *et al.* (2013); Almasirad *et al.* (2005, 2006). For a similar structure, see: Cao (2009).



Experimental

Crystal data

$C_{14}H_{10}Cl_2N_2O\cdot H_2O$ M_r = 311.16
Monoclinic, $P2_1/n$
a = 4.6160 (5) Å
b = 12.8664 (15) Å
c = 23.681 (3) Å
β = 92.6760 (17)°
V = 1404.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.46 mm^{-1}
T = 150 K
0.17 × 0.08 × 0.06 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.78, T_{max} = 0.97
24861 measured reflections
3511 independent reflections
2604 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.097
S = 1.02
3511 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.91	1.95	2.8510 (19)	168
O2—H2A···O1^{ii}	0.84	1.93	2.7632 (19)	172
O2—H2B···O1	0.84	1.95	2.7864 (19)	172
C2—H2···O2^{i}	0.95	2.43	3.286 (2)	150
C8—H8···O2^{i}	0.95	2.44	3.242 (2)	143

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: RZ5119).

References

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supporting information

Acta Cryst. (2014). E**70**, o612 [doi:10.1107/S1600536814008885]

4-Chloro-*N*′-[(*E*)-2-chlorobenzylidene]benzohydrazide monohydrate

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S1. Comment

Use of non-steroidal anti-inflammatory drugs (NSAIDs) in treatment of pain and inflammation is usually associated with undesirable side effect such as gastrointestinal toxins and ulceration. Recently arylhydrazone scaffold compounds have showed safer profiles of activity and enhanced efficacy in the battle of pain in inflammatory diseases (Koopaei *et al.*, 2013). They have been depicted as dual COX/5-LO inhibitors (Almasirad *et al.*, 2005; Almasirad *et al.*, 2006). In this context and as part of our on-going study in the synthesis of safe profiles of anti-inflammatory pro-drugs we report the synthesis and crystal structure of the title compound.

The title compound (I) in Fig. 1 is in the "*extended*" conformation with the ring C1–C6 making a dihedral angle of 15.3 (1)° with the mean plane of the C1/C7/N1/O1 unit while the ring C9–C14 makes a dihedral angle of 4.6 (2)° with the plane of the C8/C9/N2 unit. The bond lengths and angles of (I) are normal and comparable with those observed for a similar compound (Cao, 2009).

The lattice water molecules alternate with molecules of (I) in chains formed by O2—H2A(or B)···O1 hydrogen bonds (Table 1 and Fig. 2) which run parallel to the *a* axis. These chains are linked to neighboring chains through N1—H1···O2 and C8—H8···O2 interactions. In these, the mean plane of the benzohydrazide molecule is inclined approximately 48° to (110).

S2. Experimental

The compound was prepared by refluxing a mixture of 4-chlorobenzohydrazide (1 mmol, 171 mg)) with 2-chloro-benzaldehyde (1 mmol, 141 mg) in ethanol (30 mL) for 5h in the presence of a catalytic amount of glacial acetic acid. The mixture was cooled and the precipitate was filtered off, dried and recrystallized from ethanol to give pale brown crystals of poor quality. Slow evaporation of an aqueous ethanolic solution of the product afforded colorless block-like crystals of sufficient quality for x-ray diffraction. M. p. 452 454 K

S3. Refinement

H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while those attached to nitrogen and oxygen were placed in locations derived from a difference Fourier map and initially refined independently to ensure their initial positions were valid. In the final refinement, their coordinates adjusted to give N—H = 0.91 and O—H = 0.84 Å. All hydrogen atoms were then included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

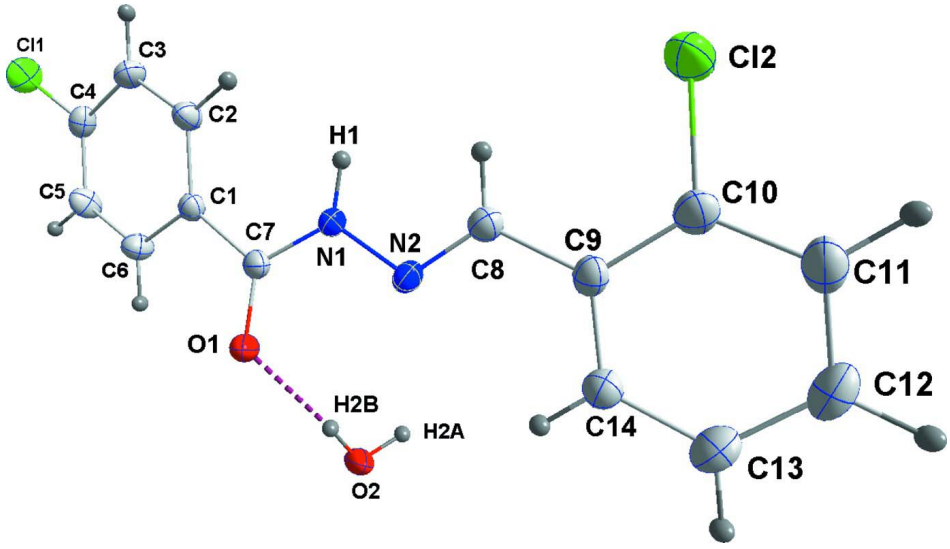

Figure 1

The asymmetric unit of the title compound showing one of the O—H···O interactions as a dotted line. Displacement ellipsoids are drawn at the 50% probability level.

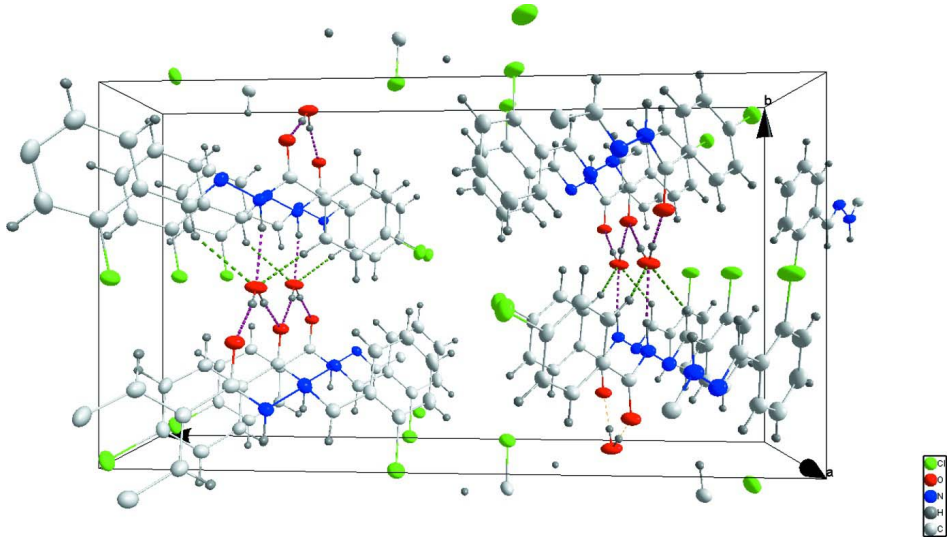

Figure 2

Packing of the title compound viewed down the *a* axis showing hydrogen interactions as dotted lines.

4-Chloro-*N*′-[(*E*)-2-chlorobenzylidene]benzohydrazide monohydrate

Crystal data

$C_{14}H_{10}Cl_2N_2O·H_2O$
$M_r = 311.16$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.6160$ (5) Å
$b = 12.8664$ (15) Å
$c = 23.681$ (3) Å
$\beta = 92.6760$ (17)°
$V = 1404.9$ (3) Å^3
$Z = 4$
$F(000) = 640$
$D_x = 1.471$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7842 reflections
$\theta = 2.3$–28.1°
$\mu = 0.46$ mm^{-1}

T = 150 K
Column, colourless

0.17 × 0.08 × 0.06 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3660 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.78, T_{max} = 0.97

24861 measured reflections
3511 independent reflections
2604 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 28.4°, θ_{min} = 1.8°
h = −6→6
k = −16→17
l = −31→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.097
S = 1.02
3511 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.6801P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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	−0.46090 (11)	0.93219 (4)	0.06216 (2)	0.0343 (2)
Cl2	1.10272 (13)	1.00504 (4)	0.41832 (2)	0.0434 (2)
O1	0.4046 (3)	0.64950 (9)	0.23672 (6)	0.0271 (4)
N1	0.5525 (3)	0.80924 (11)	0.26635 (6)	0.0213 (4)
N2	0.7522 (3)	0.76724 (11)	0.30567 (6)	0.0223 (4)
C1	0.1777 (3)	0.79601 (13)	0.19187 (7)	0.0195 (5)
C2	0.0963 (4)	0.90021 (13)	0.19483 (8)	0.0229 (5)
C3	−0.1015 (4)	0.94238 (14)	0.15527 (8)	0.0246 (5)
C4	−0.2175 (4)	0.87909 (14)	0.11261 (7)	0.0228 (5)
C5	−0.1440 (4)	0.77569 (14)	0.10916 (8)	0.0267 (6)
C6	0.0548 (4)	0.73438 (14)	0.14875 (8)	0.0248 (5)
C7	0.3874 (3)	0.74541 (13)	0.23339 (7)	0.0202 (5)
C8	0.8769 (4)	0.83478 (14)	0.33826 (7)	0.0235 (5)
C9	1.0970 (4)	0.80330 (14)	0.38149 (7)	0.0231 (5)

C10	1.2181 (4)	0.87532 (15)	0.41988 (8)	0.0277 (6)
C11	1.4287 (4)	0.84754 (16)	0.46082 (8)	0.0337 (6)
C12	1.5197 (4)	0.74555 (17)	0.46427 (8)	0.0338 (6)
C13	1.4015 (4)	0.67216 (16)	0.42740 (8)	0.0315 (6)
C14	1.1943 (4)	0.70056 (15)	0.38619 (8)	0.0266 (6)
O2	0.9062 (3)	0.53028 (10)	0.23079 (6)	0.0339 (4)
H1	0.54250	0.87980	0.26400	0.0260*
H2	0.17730	0.94280	0.22430	0.0280*
H3	−0.15640	1.01340	0.15740	0.0300*
H5	−0.22840	0.73310	0.08000	0.0320*
H6	0.10770	0.66320	0.14640	0.0300*
H8	0.82620	0.90610	0.33440	0.0280*
H11	1.50920	0.89820	0.48620	0.0400*
H12	1.66410	0.72570	0.49210	0.0410*
H13	1.46280	0.60180	0.43030	0.0380*
H14	1.11690	0.64950	0.36070	0.0320*
H2A	1.04760	0.57140	0.23300	0.0410*
H2B	0.76260	0.56910	0.23510	0.0410*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0347 (3)	0.0399 (3)	0.0273 (3)	0.0029 (2)	−0.0098 (2)	0.0061 (2)
Cl2	0.0618 (4)	0.0240 (3)	0.0424 (3)	−0.0030 (2)	−0.0175 (3)	−0.0013 (2)
O1	0.0226 (6)	0.0179 (6)	0.0401 (8)	0.0003 (5)	−0.0062 (5)	0.0002 (5)
N1	0.0193 (7)	0.0184 (7)	0.0258 (8)	0.0001 (5)	−0.0042 (6)	0.0022 (6)
N2	0.0193 (7)	0.0230 (8)	0.0242 (8)	0.0020 (6)	−0.0027 (6)	0.0039 (6)
C1	0.0179 (8)	0.0201 (8)	0.0206 (9)	−0.0016 (6)	0.0013 (6)	0.0017 (7)
C2	0.0244 (9)	0.0201 (8)	0.0240 (9)	−0.0011 (7)	−0.0024 (7)	−0.0016 (7)
C3	0.0249 (9)	0.0217 (9)	0.0272 (10)	0.0027 (7)	0.0000 (7)	0.0015 (7)
C4	0.0202 (8)	0.0295 (9)	0.0186 (9)	−0.0006 (7)	0.0001 (7)	0.0041 (7)
C5	0.0326 (10)	0.0268 (10)	0.0203 (9)	−0.0043 (8)	−0.0035 (7)	−0.0030 (7)
C6	0.0288 (9)	0.0208 (9)	0.0247 (9)	0.0001 (7)	0.0009 (7)	−0.0020 (7)
C7	0.0166 (8)	0.0192 (8)	0.0251 (9)	−0.0010 (6)	0.0028 (6)	0.0003 (7)
C8	0.0233 (9)	0.0228 (9)	0.0243 (9)	0.0000 (7)	−0.0004 (7)	0.0018 (7)
C9	0.0202 (8)	0.0265 (9)	0.0223 (9)	−0.0036 (7)	−0.0009 (7)	0.0027 (7)
C10	0.0308 (10)	0.0265 (10)	0.0256 (10)	−0.0037 (7)	−0.0018 (8)	0.0043 (8)
C11	0.0366 (11)	0.0383 (11)	0.0252 (10)	−0.0077 (9)	−0.0082 (8)	0.0010 (9)
C12	0.0294 (10)	0.0461 (12)	0.0252 (10)	0.0031 (9)	−0.0055 (8)	0.0089 (9)
C13	0.0305 (10)	0.0340 (11)	0.0298 (11)	0.0069 (8)	−0.0003 (8)	0.0058 (8)
C14	0.0263 (9)	0.0272 (10)	0.0261 (10)	−0.0004 (7)	−0.0011 (7)	0.0014 (8)
O2	0.0232 (6)	0.0178 (6)	0.0601 (10)	−0.0009 (5)	−0.0037 (6)	0.0041 (6)

Geometric parameters (Å, º)

Cl1—C4	1.7410 (18)	C8—C9	1.465 (2)
Cl2—C10	1.752 (2)	C9—C10	1.397 (3)
O1—C7	1.239 (2)	C9—C14	1.399 (3)

O2—H2B	0.8400	C10—C11	1.387 (3)
O2—H2A	0.8400	C11—C12	1.379 (3)
N1—C7	1.345 (2)	C12—C13	1.381 (3)
N1—N2	1.389 (2)	C13—C14	1.383 (3)
N2—C8	1.281 (2)	C2—H2	0.9500
N1—H1	0.9100	C3—H3	0.9500
C1—C6	1.393 (2)	C5—H5	0.9500
C1—C2	1.395 (2)	C6—H6	0.9500
C1—C7	1.496 (2)	C8—H8	0.9500
C2—C3	1.388 (3)	C11—H11	0.9500
C3—C4	1.386 (3)	C12—H12	0.9500
C4—C5	1.376 (3)	C13—H13	0.9500
C5—C6	1.387 (3)	C14—H14	0.9500

H2A—O2—H2B	103.00	Cl2—C10—C11	117.56 (15)
N2—N1—C7	119.47 (14)	C10—C11—C12	119.22 (18)
N1—N2—C8	113.93 (14)	C11—C12—C13	120.19 (18)
N2—N1—H1	117.00	C12—C13—C14	120.34 (19)
C7—N1—H1	123.00	C9—C14—C13	120.97 (18)
C2—C1—C7	123.55 (15)	C3—C2—H2	120.00
C2—C1—C6	118.86 (15)	C1—C2—H2	120.00
C6—C1—C7	117.58 (15)	C2—C3—H3	121.00
C1—C2—C3	120.81 (16)	C4—C3—H3	121.00
C2—C3—C4	118.82 (16)	C6—C5—H5	120.00
Cl1—C4—C3	119.00 (14)	C4—C5—H5	120.00
C3—C4—C5	121.56 (17)	C1—C6—H6	120.00
Cl1—C4—C5	119.44 (14)	C5—C6—H6	120.00
C4—C5—C6	119.18 (17)	C9—C8—H8	120.00
C1—C6—C5	120.77 (16)	N2—C8—H8	120.00
N1—C7—C1	116.57 (14)	C10—C11—H11	120.00
O1—C7—C1	120.85 (15)	C12—C11—H11	120.00
O1—C7—N1	122.58 (15)	C13—C12—H12	120.00
N2—C8—C9	120.72 (16)	C11—C12—H12	120.00
C10—C9—C14	117.26 (16)	C12—C13—H13	120.00
C8—C9—C14	121.76 (16)	C14—C13—H13	120.00
C8—C9—C10	120.99 (16)	C9—C14—H14	120.00
Cl2—C10—C9	120.42 (14)	C13—C14—H14	120.00
C9—C10—C11	122.01 (18)		

C7—N1—N2—C8	−173.66 (15)	C3—C4—C5—C6	−1.2 (3)
N2—N1—C7—O1	0.3 (2)	C4—C5—C6—C1	0.5 (3)
N2—N1—C7—C1	−179.81 (13)	N2—C8—C9—C10	−175.19 (17)
N1—N2—C8—C9	−178.99 (15)	N2—C8—C9—C14	4.7 (3)
C6—C1—C2—C3	−0.6 (3)	C8—C9—C10—Cl2	1.6 (2)
C7—C1—C2—C3	−179.41 (16)	C8—C9—C10—C11	−179.38 (17)
C2—C1—C6—C5	0.4 (3)	C14—C9—C10—Cl2	−178.37 (14)
C7—C1—C6—C5	179.20 (16)	C14—C9—C10—C11	0.7 (3)
C2—C1—C7—O1	163.87 (16)	C8—C9—C14—C13	−179.76 (17)

C2—C1—C7—N1	−16.0 (2)	C10—C9—C14—C13	0.2 (3)
C6—C1—C7—O1	−14.9 (2)	Cl2—C10—C11—C12	178.38 (15)
C6—C1—C7—N1	165.23 (15)	C9—C10—C11—C12	−0.7 (3)
C1—C2—C3—C4	0.0 (3)	C10—C11—C12—C13	−0.1 (3)
C2—C3—C4—Cl1	−178.89 (14)	C11—C12—C13—C14	1.0 (3)
C2—C3—C4—C5	0.9 (3)	C12—C13—C14—C9	−1.0 (3)
Cl1—C4—C5—C6	178.61 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.91	1.95	2.8510 (19)	168
O2—H2*A*···O1^{ii}	0.84	1.93	2.7632 (19)	172
O2—H2*B*···O1	0.84	1.95	2.7864 (19)	172
C2—H2···O2^{i}	0.95	2.43	3.286 (2)	150
C8—H8···O2^{i}	0.95	2.44	3.242 (2)	143

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