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N'-(2-Hydroxy-5-chlorobenzylidene)-4-nitrobenzohydrazide methanol solvate

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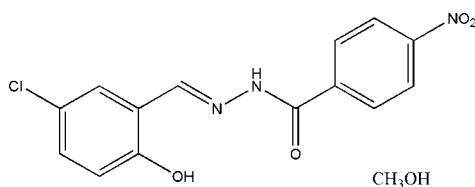
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.055; wR factor = 0.147; data-to-parameter ratio = 14.6.

The title compound, $\text{C}_{14}\text{H}_{10}\text{ClN}_3\text{O}_4 \cdot \text{CH}_4\text{O}$, was synthesized from the reaction of 5-chlorosalicylaldehyde with 4-nitrobenzohydrazide in methanol. The Schiff base molecule is nearly planar, with a dihedral angle of $9.1(3)^\circ$ between the two benzene rings. The methanol solvent molecules are linked to the Schiff base molecules by $\text{N}-\text{H} \cdots \text{O}$, $\text{O}-\text{H} \cdots \text{N}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds, forming chains running parallel to the a axis.

Related literature

For related structures, see: Brückner *et al.* (2000); Diao (2007); Diao, Huang *et al.* (2008); Diao, Shu *et al.* (2007); Diao, Zhen *et al.* (2008); Harrop *et al.* (2003); Huang *et al.* (2007); Li *et al.* (2007); Ma *et al.* (2008); Ren *et al.* (2002); Wang *et al.* (2008).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{10}\text{ClN}_3\text{O}_4 \cdot \text{CH}_4\text{O}$
 $M_r = 351.74$
 Monoclinic, $P2_1/n$
 $a = 6.628(1)$ Å
 $b = 18.980(3)$ Å
 $c = 12.521(2)$ Å
 $\beta = 91.29(3)^\circ$
 $V = 1574.7(4)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.27$ mm⁻¹
 $T = 298(2)$ K
 $0.20 \times 0.18 \times 0.17$ mm

Data collection

 Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.947$, $T_{\max} = 0.955$
 9258 measured reflections
 3259 independent reflections
 1776 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.147$
 $S = 1.01$
 3259 reflections
 223 parameters
 1 restraint
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.23$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

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

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O4}-\text{H4} \cdots \text{N3}$	0.82	2.04	2.745 (3)	144
$\text{O4}-\text{H4} \cdots \text{O5}^i$	0.82	2.47	2.930 (3)	116
$\text{O5}-\text{H5} \cdots \text{O3}^{ii}$	0.82	1.88	2.692 (3)	171
$\text{N2}-\text{H2A} \cdots \text{O5}$	0.899 (10)	2.016 (13)	2.888 (3)	163 (3)

 Symmetry codes: (i) $x + 1, y, z$; (ii) $x - 1, y, z$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2574).

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supplementary materials

Acta Cryst. (2008). E64, o781 [doi:10.1107/S160053680800843X]

N'-(2-Hydroxy-5-chlorobenzylidene)-4-nitrobenzohydrazide methanol solvate

L. Han, S.-S. Huang, Q.-B. Huang, X.-M. Zhou and Y.-P. Diao

Comment

Schiff base compounds have been found to have potential pharmacological and antitumor properties (Brückner *et al.*, 2000; Harrop *et al.*, 2003; Ren *et al.*, 2002). Recently, a few Schiff base compounds derived from the reaction of aldehydes with benzohydrazides have been reported (Diao 2007; Diao, Huang *et al.*, 2008; Diao, Shu *et al.*, 2007; Diao, Zhen *et al.*, 2008; Huang *et al.*, 2007; Li *et al.*, 2007; Ma *et al.*, 2008; Wang *et al.*, 2008). As a further study of such compounds, we report here the crystal structure of the title compound.

The asymmetric unit of the title compound consists of a Schiff base molecule and a lattice methanol molecule. The Schiff base molecule is nearly planar with the dihedral angle between the two benzene rings of 9.1 (3)°. The dihedral angle between the C1-C6 benzene ring and the O1/N1/O2 nitryl plane is 6.4 (3)°. The torsion angles C9—C8—N3—N2 and C4—C7—N2—N3 are 179.8 (2)° and -173.6 (2)°, respectively. The methanol solvent molecules are linked to the Schiff base molecules by N—H···O, O—H···N and O—H···O hydrogen bonds (Table 1), forming chains running along the *a* axis (Fig. 2).

Experimental

5-Chlorosalicylaldehyde (0.1 mmol, 15.7 mg) and 4-nitrobenzohydrazide (0.1 mmol, 18.1 mg) were dissolved in a methanol solution (20 ml). The mixture was stirred at reflux for 1 h and cooled to room temperature. After keeping the solution in air for a week, yellow block-like crystals were formed.

Refinement

Atom H2A was located from a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.96 Å, O—H = 0.82 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{O and methyl C})$.

Figures

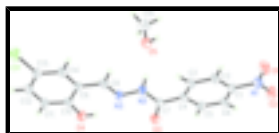


Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

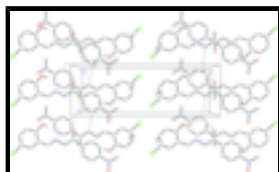


Fig. 2. Crystal packing of the compound viewed along the *c* axis. Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in the interactions have been omitted.

N'-(2-Hydroxy-5-chlorobenzylidene)-4-nitrobenzohydrazide methanol solvate

Crystal data

C₁₄H₁₀ClN₃O₄·CH₄O

M_r = 351.74

Monoclinic, *P*2₁/*n*

Hall symbol: -*P* 2yn

a = 6.628 (1) Å

b = 18.980 (3) Å

c = 12.521 (2) Å

β = 91.29 (3)°

V = 1574.7 (4) Å³

Z = 4

*F*₀₀₀ = 728

D_x = 1.484 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 1014 reflections

θ = 2.5–24.3°

μ = 0.27 mm⁻¹

T = 298 (2) K

Block, yellow

0.20 × 0.18 × 0.17 mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 298(2) K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2000)

*T*_{min} = 0.947, *T*_{max} = 0.955

9258 measured reflections

3259 independent reflections

1776 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.056

θ_{max} = 26.5°

θ_{min} = 2.0°

h = -8→7

k = -22→23

l = -15→15

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.055

wR(*F*²) = 0.147

S = 1.01

3259 reflections

223 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

$$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2]$$

where *P* = (*F*_o² + 2*F*_c²)/3

(Δ/σ)_{max} = 0.001

Δρ_{max} = 0.23 e Å⁻³

Δρ_{min} = -0.29 e Å⁻³

Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.68929 (13)	0.04557 (4)	0.92197 (7)	0.0738 (3)
N1	0.5353 (5)	0.73926 (13)	0.88443 (19)	0.0595 (7)
N2	0.8868 (3)	0.42344 (11)	0.86323 (19)	0.0495 (6)
N3	0.9879 (3)	0.35976 (11)	0.86989 (17)	0.0478 (6)
O1	0.3541 (4)	0.73510 (11)	0.89719 (19)	0.0807 (7)
O2	0.6246 (4)	0.79469 (11)	0.87488 (19)	0.0824 (7)
O3	1.1727 (3)	0.48808 (10)	0.86669 (16)	0.0623 (6)
O4	1.2997 (3)	0.26460 (10)	0.87161 (18)	0.0659 (6)
H4	1.2520	0.3044	0.8735	0.099*
O5	0.4676 (3)	0.40021 (11)	0.80587 (18)	0.0674 (6)
H5	0.3727	0.4232	0.8281	0.101*
C1	0.6514 (4)	0.67373 (13)	0.8806 (2)	0.0470 (7)
C2	0.5546 (4)	0.61164 (14)	0.9009 (2)	0.0548 (8)
H2	0.4187	0.6113	0.9177	0.066*
C3	0.6617 (4)	0.54978 (14)	0.8959 (2)	0.0518 (8)
H3	0.5977	0.5074	0.9108	0.062*
C4	0.8634 (4)	0.54947 (13)	0.8691 (2)	0.0426 (6)
C5	0.9558 (4)	0.61350 (14)	0.8499 (2)	0.0521 (7)
H5A	1.0915	0.6143	0.8327	0.063*
C6	0.8517 (5)	0.67595 (14)	0.8558 (2)	0.0549 (8)
H6	0.9156	0.7187	0.8432	0.066*
C7	0.9882 (4)	0.48478 (14)	0.8649 (2)	0.0443 (6)
C8	0.8723 (4)	0.30647 (14)	0.8789 (2)	0.0488 (7)
H8	0.7338	0.3140	0.8806	0.059*
C9	0.9467 (4)	0.23460 (13)	0.8868 (2)	0.0443 (7)
C10	1.1506 (4)	0.21677 (14)	0.8820 (2)	0.0499 (7)
C11	1.2063 (5)	0.14648 (15)	0.8879 (3)	0.0635 (9)
H11	1.3419	0.1344	0.8842	0.076*
C12	1.0660 (5)	0.09492 (15)	0.8992 (2)	0.0607 (8)
H12	1.1058	0.0480	0.9025	0.073*
C13	0.8651 (4)	0.11224 (14)	0.9058 (2)	0.0503 (7)
C14	0.8072 (4)	0.18097 (14)	0.8996 (2)	0.0497 (7)
H14	0.6711	0.1922	0.9042	0.060*

supplementary materials

C15	0.4413 (6)	0.38939 (17)	0.6953 (3)	0.0850 (11)
H15A	0.3767	0.4298	0.6636	0.127*
H15B	0.3587	0.3485	0.6830	0.127*
H15C	0.5704	0.3824	0.6637	0.127*
H2A	0.7515 (16)	0.4223 (17)	0.856 (2)	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0638 (6)	0.0528 (5)	0.1047 (7)	-0.0066 (4)	-0.0002 (5)	0.0189 (4)
N1	0.071 (2)	0.0480 (16)	0.0598 (16)	0.0150 (14)	0.0003 (14)	-0.0022 (12)
N2	0.0387 (13)	0.0391 (12)	0.0704 (16)	0.0081 (12)	-0.0020 (12)	0.0003 (11)
N3	0.0466 (15)	0.0393 (13)	0.0575 (15)	0.0082 (11)	-0.0014 (11)	0.0002 (11)
O1	0.0651 (17)	0.0686 (15)	0.1087 (19)	0.0264 (13)	0.0099 (14)	-0.0013 (12)
O2	0.099 (2)	0.0440 (13)	0.1044 (19)	0.0093 (13)	0.0000 (15)	0.0054 (12)
O3	0.0380 (13)	0.0549 (12)	0.0939 (16)	0.0046 (10)	0.0008 (11)	-0.0051 (11)
O4	0.0451 (13)	0.0489 (12)	0.1037 (17)	0.0004 (10)	0.0008 (12)	0.0042 (12)
O5	0.0445 (13)	0.0623 (14)	0.0952 (17)	0.0103 (10)	-0.0024 (11)	-0.0156 (11)
C1	0.053 (2)	0.0405 (15)	0.0476 (16)	0.0108 (13)	-0.0011 (14)	-0.0005 (12)
C2	0.0428 (17)	0.0491 (17)	0.073 (2)	0.0058 (14)	0.0056 (15)	-0.0036 (15)
C3	0.0441 (18)	0.0399 (15)	0.071 (2)	0.0018 (13)	0.0055 (15)	-0.0016 (13)
C4	0.0378 (16)	0.0421 (15)	0.0479 (16)	0.0023 (12)	-0.0013 (12)	-0.0007 (12)
C5	0.0388 (17)	0.0523 (17)	0.0653 (19)	-0.0020 (14)	0.0007 (14)	0.0024 (14)
C6	0.055 (2)	0.0425 (16)	0.067 (2)	-0.0003 (14)	-0.0032 (16)	0.0048 (14)
C7	0.0377 (17)	0.0446 (15)	0.0505 (17)	0.0030 (13)	-0.0018 (13)	-0.0017 (13)
C8	0.0412 (17)	0.0454 (16)	0.0599 (18)	0.0082 (13)	0.0017 (14)	0.0011 (13)
C9	0.0389 (16)	0.0425 (15)	0.0514 (17)	0.0040 (12)	-0.0009 (13)	0.0010 (12)
C10	0.0435 (18)	0.0461 (16)	0.0600 (18)	0.0012 (14)	-0.0002 (14)	0.0021 (14)
C11	0.0441 (18)	0.0485 (17)	0.098 (2)	0.0127 (15)	0.0008 (16)	0.0038 (17)
C12	0.058 (2)	0.0437 (17)	0.080 (2)	0.0052 (15)	-0.0042 (16)	0.0064 (15)
C13	0.0495 (18)	0.0420 (15)	0.0594 (18)	-0.0005 (13)	-0.0024 (14)	0.0065 (13)
C14	0.0379 (17)	0.0496 (16)	0.0616 (18)	0.0062 (13)	0.0014 (13)	0.0011 (14)
C15	0.092 (3)	0.076 (2)	0.087 (3)	0.000 (2)	0.006 (2)	-0.003 (2)

Geometric parameters (\AA , $^\circ$)

C11—C13	1.735 (3)	C4—C5	1.384 (3)
N1—O2	1.215 (3)	C4—C7	1.482 (4)
N1—O1	1.217 (3)	C5—C6	1.374 (4)
N1—C1	1.464 (3)	C5—H5A	0.93
N2—C7	1.344 (3)	C6—H6	0.93
N2—N3	1.384 (3)	C8—C9	1.453 (3)
N2—H2A	0.899 (10)	C8—H8	0.93
N3—C8	1.275 (3)	C9—C14	1.387 (4)
O3—C7	1.225 (3)	C9—C10	1.396 (4)
O4—C10	1.350 (3)	C10—C11	1.386 (4)
O4—H4	0.82	C11—C12	1.360 (4)
O5—C15	1.407 (4)	C11—H11	0.93
O5—H5	0.82	C12—C13	1.375 (4)

C1—C2	1.368 (4)	C12—H12	0.93
C1—C6	1.371 (4)	C13—C14	1.362 (3)
C2—C3	1.374 (3)	C14—H14	0.93
C2—H2	0.93	C15—H15A	0.96
C3—C4	1.386 (4)	C15—H15B	0.96
C3—H3	0.93	C15—H15C	0.96
O2—N1—O1	123.6 (3)	N2—C7—C4	116.0 (2)
O2—N1—C1	118.3 (3)	N3—C8—C9	123.2 (3)
O1—N1—C1	118.0 (3)	N3—C8—H8	118.4
C7—N2—N3	121.0 (2)	C9—C8—H8	118.4
C7—N2—H2A	121 (2)	C14—C9—C10	118.4 (2)
N3—N2—H2A	118 (2)	C14—C9—C8	118.1 (2)
C8—N3—N2	114.0 (2)	C10—C9—C8	123.5 (3)
C10—O4—H4	109.5	O4—C10—C11	117.2 (3)
C15—O5—H5	109.5	O4—C10—C9	123.6 (2)
C2—C1—C6	121.9 (3)	C11—C10—C9	119.2 (3)
C2—C1—N1	118.5 (3)	C12—C11—C10	121.1 (3)
C6—C1—N1	119.6 (3)	C12—C11—H11	119.4
C1—C2—C3	118.9 (3)	C10—C11—H11	119.4
C1—C2—H2	120.6	C11—C12—C13	120.0 (3)
C3—C2—H2	120.6	C11—C12—H12	120.0
C2—C3—C4	121.1 (3)	C13—C12—H12	120.0
C2—C3—H3	119.4	C14—C13—C12	119.8 (3)
C4—C3—H3	119.4	C14—C13—C11	121.1 (2)
C5—C4—C3	118.1 (2)	C12—C13—C11	119.1 (2)
C5—C4—C7	118.2 (2)	C13—C14—C9	121.5 (3)
C3—C4—C7	123.7 (2)	C13—C14—H14	119.3
C6—C5—C4	121.6 (3)	C9—C14—H14	119.3
C6—C5—H5A	119.2	O5—C15—H15A	109.5
C4—C5—H5A	119.2	O5—C15—H15B	109.5
C1—C6—C5	118.4 (3)	H15A—C15—H15B	109.5
C1—C6—H6	120.8	O5—C15—H15C	109.5
C5—C6—H6	120.8	H15A—C15—H15C	109.5
O3—C7—N2	122.9 (2)	H15B—C15—H15C	109.5
O3—C7—C4	121.0 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O4—H4 \cdots N3	0.82	2.04	2.745 (3)	144
O4—H4 \cdots O5 ⁱ	0.82	2.47	2.930 (3)	116
O5—H5 \cdots O3 ⁱⁱ	0.82	1.88	2.692 (3)	171
N2—H2A \cdots O5	0.899 (10)	2.016 (13)	2.888 (3)	163 (3)

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$.

Fig. 1

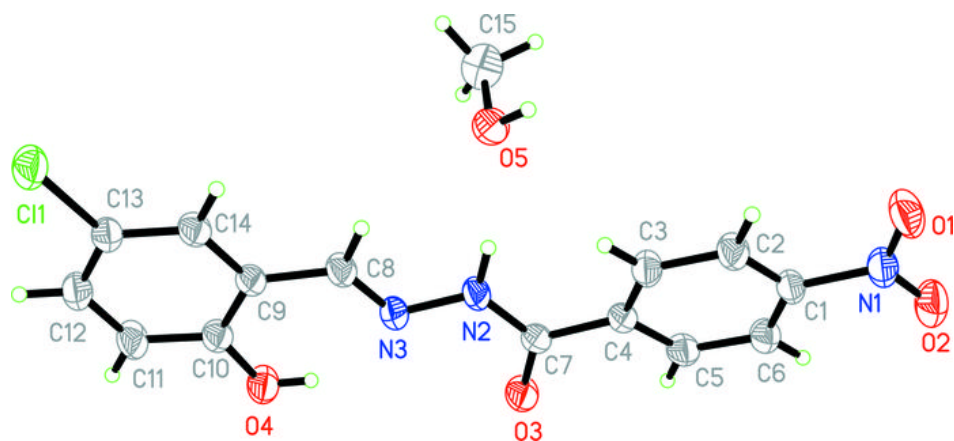


Fig. 2

