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

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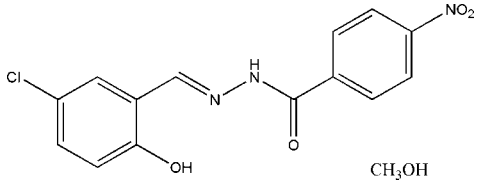
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.055; wR factor = 0.147; data-to-parameter ratio = 14.6.

The title compound, $C_{14}H_{10}ClN_3O_4\cdot CH_4O$, 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)° between the two benzene rings. The methanol solvent molecules are linked to the Schiff base molecules by N—H···O, O—H···N and O—H···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

$C_{14}H_{10}ClN_3O_4\cdot CH_4O$
M_r = 351.74
Monoclinic, $P2_1/n$
a = 6.628 (1) Å
b = 18.980 (3) Å
c = 12.521 (2) Å
β = 91.29 (3)°
V = 1574.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 298 (2) K
0.20 × 0.18 × 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_{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—H···A	D—H	H···A	D···A	D—H···A
O4—H4···N3	0.82	2.04	2.745 (3)	144
O4—H4···O5^{i}	0.82	2.47	2.930 (3)	116
O5—H5···O3^{ii}	0.82	1.88	2.692 (3)	171
N2—H2*A*···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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supporting information

Acta Cryst. (2008). E**64**, o781 [doi:10.1107/S160053680800843X]

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

Ling Han, Shan-Shan Huang, Qing-Bai Huang, Xue-Mei Zhou and Yun-Peng Diao

S1. 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).

S2. 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.

S3. 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_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O and methyl C).

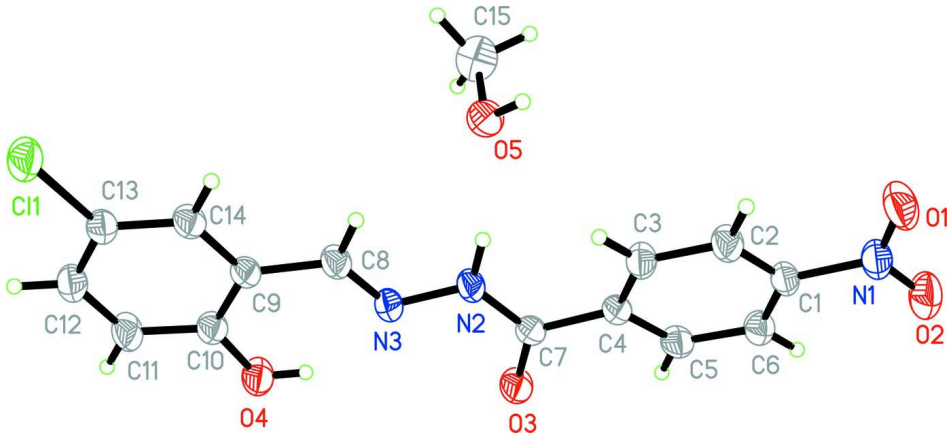

Figure 1

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

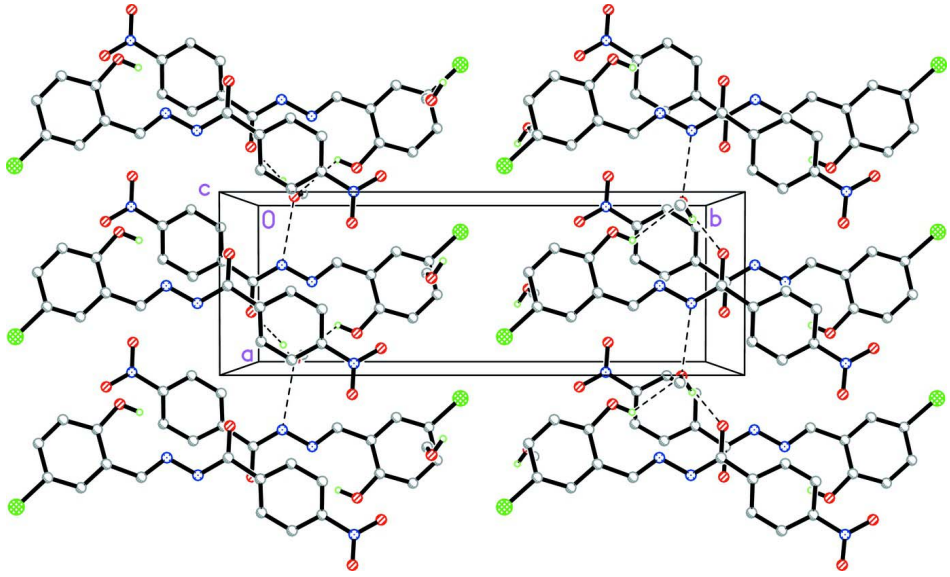

Figure 2

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

$C_{14}H_{10}ClN_3O_4·CH_4O$
$M_r = 351.74$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.628$ (1) Å
$b = 18.980$ (3) Å
$c = 12.521$ (2) Å
$\beta = 91.29$ (3)°
$V = 1574.7$ (4) Å^3
$Z = 4$
$F(000) = 728$
$D_x = 1.484$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1014 reflections
$\theta = 2.5$–24.3°
$\mu = 0.27$ mm^{-1}
$T = 298$ K
Block, yellow
0.20 × 0.18 × 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*; Bruker, 2000)
T_{min} = 0.947, T_{max} = 0.955
9258 measured reflections
3259 independent reflections
1776 reflections with $I > 2\sigma(I)$
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
$R[F^2 > 2\sigma(F^2)]$ = 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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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	0.68929 (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*
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 (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	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 (Å, º)

Cl1—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—Cl1	121.1 (2)
C5—C4—C3	118.1 (2)	C12—C13—Cl1	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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···N3	0.82	2.04	2.745 (3)	144
O4—H4···O5^{i}	0.82	2.47	2.930 (3)	116
O5—H5···O3^{ii}	0.82	1.88	2.692 (3)	171
N2—H2*A*···O5	0.90 (1)	2.02 (1)	2.888 (3)	163 (3)

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