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N′-(3,5-Dichloro-2-hydroxybenzylidene)-4-(dimethylamino)benzohydrazide methanol monosolvate

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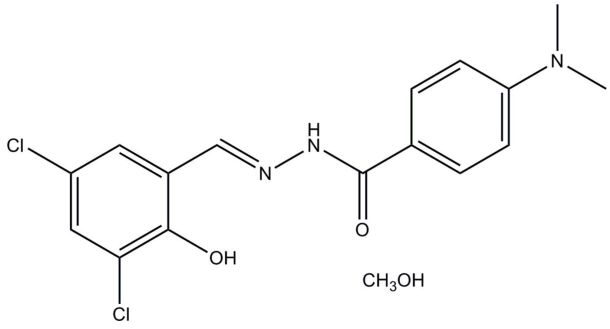
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.079; wR factor = 0.180; data-to-parameter ratio = 14.0.

The title compound, $C_{16}H_{15}Cl_2N_3O_2\cdot CH_3OH$, a Schiff base molecule, is prepared by the reaction of 3,5-dichlorosalicylaldehyde with 4-dimethylaminobenzohydrazide in methanol. The Schiff base molecule is approximately planar, with a mean deviation from the least-squares plane defined by the non-H atoms of 0.0452 (3) Å, and with a dihedral angle between the benzene rings of 4.2 (3)°. This planarity is assisted by the formation of an intramolecular O—H···N hydrogen bond. In the crystal, adjacent Schiff base molecules are linked by two methanol molecules through N—H···O and O—H···O hydrogen bonds, forming dimers.

Related literature

For the preparation of Schiff base compounds by the condensation reaction between aldehydes with organic primary amines, see: Miura *et al.* (2009); Zhao *et al.* (2010); Karadağ *et al.* (2011); Bingöl Alpaslan *et al.* (2010). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{15}Cl_2N_3O_2\cdot CH_4O$
M_r = 384.25
Monoclinic, $P2_1/n$
a = 7.6498 (15) Å
b = 14.338 (3) Å
c = 16.884 (2) Å
β = 103.076 (2)°
V = 1803.8 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 298 K
0.13 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.952, T_{max} = 0.963
8311 measured reflections
3238 independent reflections
1790 reflections with $I > 2\sigma(I)$
R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.180
S = 1.03
3238 reflections
231 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3^{i}	0.86	2.10	2.848 (5)	145
O1—H1···N1	0.82	1.91	2.600 (5)	141
O3—H3···O2	0.82	1.97	2.771 (5)	166

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QM2057).

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supplementary materials

Acta Cryst. (2012). E**68**, o1042 [doi:10.1107/S1600536812010148]

N′-(3,5-Dichloro-2-hydroxybenzylidene)-4-(dimethylamino)benzohydrazide methanol monosolvate

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Comment

The condensation reaction between aldehydes with organic primary amines readily forms Schiff bases containing the typical –C=N– groups (Miura *et al.*, 2009; Zhao *et al.*, 2010; Karadağ *et al.*, 2011; Bingöl Alpaslan *et al.*, 2010). In this paper, the new title compound (Fig. 1), was prepared by the reaction of 3,5-dichlorosalicylaldehyde with 4-dimethylaminobenzohydrazide in methanol.

The asymmetric unit comprises a Schiff base molecule and a methanol molecule. The Schiff base molecule is approximately planar, with mean deviation from the least squares plane defined by the non-hydrogen atoms of 0.0452 (3) Å, and with the dihedral angle between the two benzene rings of 4.2 (3)°. This planarity is assisted by the formation of an intramolecular O1—H1···N1 hydrogen bond (Table 1). All the bond lengths are within normal ranges (Allen *et al.*, 1987). In the crystal structure adjacent Schiff base molecules are linked by two methanol molecules through intermolecular N—H···O and O—H···O hydrogen bonds (Table 1) to form a dimer (Fig. 2).

Experimental

3,5-Dichlorosalicylaldehyde (1.0 mmol, 0.190 g) and 4-dimethylaminobenzohydrazide (1.0 mmol, 0.179 g) were refluxed for 30 min in 30 ml me thanol, and cooled to room temperature to give colorless solid, which was isoloated by filtration. Single crystals of the title compound were formed by recrystallization of the solid product in methanol.

Refinement

Hydrogen atoms were positioned geometrically and refined using the riding-model approximation, with C—H = 0.93–0.96 Å, O—H = 0.82 Å, N—H = 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(C,*N*) or U_{iso}(H) = 1.5U_{eq}(O).

Computing details

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

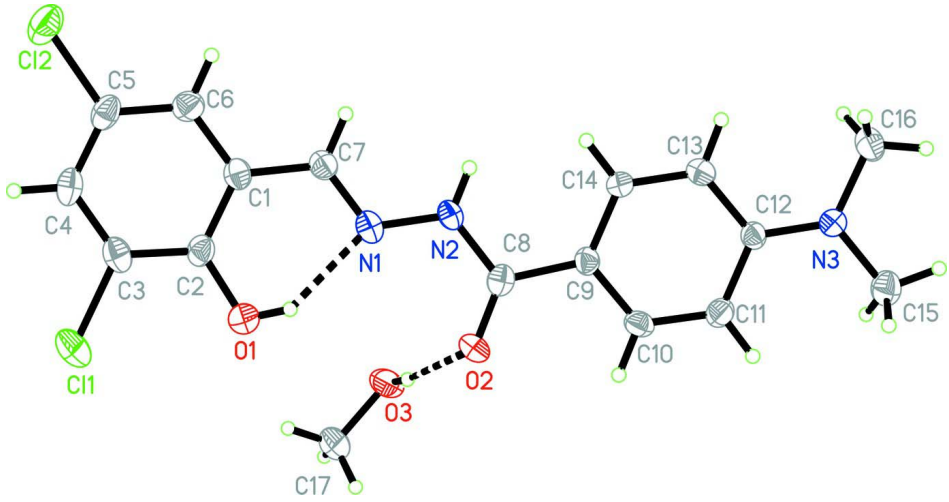

Figure 1

The molecular structure of the title compounds with atom labels and the 30% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

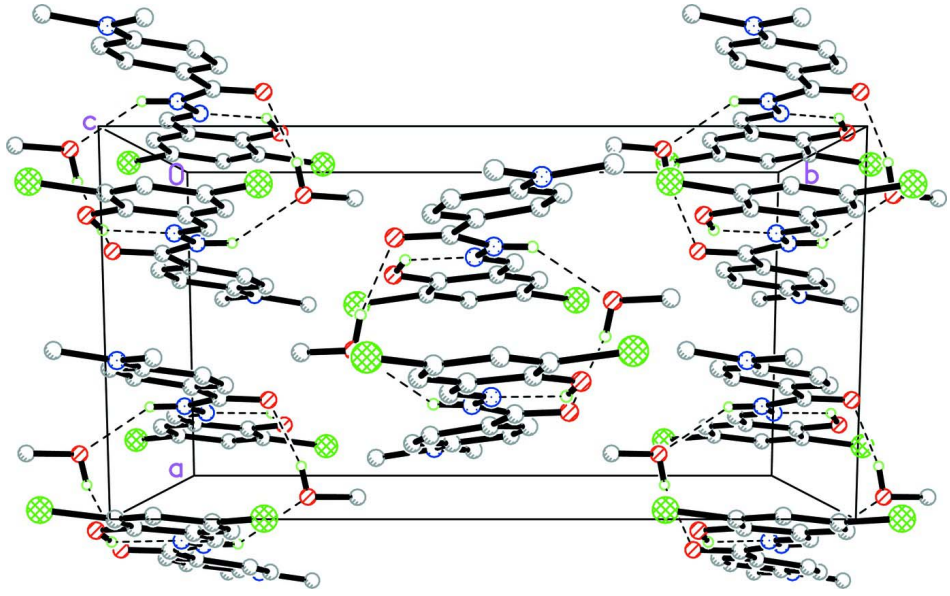

Figure 2

The molecular packing of the title compound. Hydrogen bonds are shown as dashed lines.

N′-(3,5-Dichloro-2-hydroxybenzylidene)-4-(dimethylamino)benzohydrazide methanol monosolvate

Crystal data

$C_{16}H_{15}Cl_2N_3O_2·CH_4O$
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b = 14.338 (3) Å
c = 16.884 (2) Å
β = 103.076 (2)°
V = 1803.8 (5) Å^3
Z = 4
F(000) = 800
D_x = 1.415 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2546 reflections
θ = 2.5–28.3°

μ = 0.38 mm^{-1}
T = 298 K

Block, colorless
0.13 × 0.12 × 0.10 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, 2001)
T_{min} = 0.952, T_{max} = 0.963

8311 measured reflections
3238 independent reflections
1790 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 25.5°, θ_{min} = 2.5°
h = −9→9
k = −16→17
l = −13→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.180
S = 1.03
3238 reflections
231 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 0.8315P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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	0.4325 (2)	0.29209 (10)	0.15198 (9)	0.0674 (5)
Cl2	0.4175 (2)	0.65692 (11)	0.07222 (9)	0.0719 (5)
N1	0.2957 (5)	0.4874 (3)	0.4015 (2)	0.0450 (10)
N2	0.2686 (5)	0.5167 (3)	0.4749 (2)	0.0431 (10)
H2	0.2702	0.5751	0.4864	0.052*
N3	0.1064 (6)	0.5808 (3)	0.8296 (2)	0.0490 (11)
O1	0.3448 (5)	0.3581 (2)	0.3007 (2)	0.0564 (10)
H1	0.3013	0.3780	0.3376	0.085*
O2	0.2453 (5)	0.3677 (2)	0.5161 (2)	0.0530 (10)
O3	0.5727 (5)	0.3035 (2)	0.4941 (2)	0.0608 (10)
H3	0.4685	0.3145	0.4960	0.091*
C1	0.3425 (6)	0.5229 (3)	0.2711 (3)	0.0394 (12)
C2	0.3621 (6)	0.4282 (3)	0.2505 (3)	0.0420 (12)
C3	0.4009 (6)	0.4078 (4)	0.1756 (3)	0.0483 (13)

C4	0.4178 (6)	0.4767 (4)	0.1208 (3)	0.0463 (13)
H4	0.4441	0.4616	0.0712	0.056*
C5	0.3950 (6)	0.5682 (4)	0.1411 (3)	0.0481 (13)
C6	0.3577 (6)	0.5909 (4)	0.2145 (3)	0.0474 (13)
H6	0.3422	0.6532	0.2266	0.057*
C7	0.3097 (6)	0.5495 (3)	0.3482 (3)	0.0407 (12)
H7	0.2984	0.6123	0.3599	0.049*
C8	0.2386 (6)	0.4510 (4)	0.5302 (3)	0.0388 (12)
C9	0.1976 (6)	0.4892 (3)	0.6054 (3)	0.0363 (11)
C10	0.1902 (6)	0.4268 (3)	0.6673 (3)	0.0408 (12)
H10	0.2087	0.3637	0.6592	0.049*
C11	0.1562 (6)	0.4551 (3)	0.7403 (3)	0.0436 (12)
H11	0.1497	0.4109	0.7799	0.052*
C12	0.1311 (6)	0.5505 (3)	0.7556 (3)	0.0358 (11)
C13	0.1359 (6)	0.6129 (3)	0.6922 (3)	0.0455 (12)
H13	0.1173	0.6761	0.6996	0.055*
C14	0.1672 (6)	0.5830 (3)	0.6193 (3)	0.0427 (12)
H14	0.1681	0.6263	0.5784	0.051*
C15	0.0961 (8)	0.5155 (4)	0.8937 (3)	0.0577 (15)
H15A	0.0012	0.4716	0.8743	0.087*
H15B	0.0723	0.5487	0.9395	0.087*
H15C	0.2080	0.4827	0.9098	0.087*
C16	0.0752 (7)	0.6783 (4)	0.8461 (3)	0.0591 (15)
H16A	0.1787	0.7145	0.8425	0.089*
H16B	0.0538	0.6841	0.8997	0.089*
H16C	−0.0274	0.7007	0.8069	0.089*
C17	0.5794 (7)	0.2197 (4)	0.4506 (3)	0.0619 (16)
H17A	0.7002	0.1964	0.4626	0.093*
H17B	0.5411	0.2315	0.3934	0.093*
H17C	0.5016	0.1743	0.4665	0.093*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0718 (10)	0.0608 (9)	0.0729 (10)	0.0044 (7)	0.0235 (8)	−0.0241 (8)
Cl2	0.0840 (11)	0.0797 (11)	0.0536 (9)	−0.0156 (9)	0.0192 (8)	0.0149 (8)
N1	0.047 (3)	0.048 (3)	0.040 (3)	0.000 (2)	0.011 (2)	−0.009 (2)
N2	0.057 (3)	0.039 (2)	0.033 (2)	−0.002 (2)	0.010 (2)	−0.0098 (19)
N3	0.071 (3)	0.037 (2)	0.046 (3)	−0.001 (2)	0.026 (2)	0.000 (2)
O1	0.078 (3)	0.045 (2)	0.048 (2)	0.0047 (19)	0.018 (2)	0.0026 (18)
O2	0.074 (2)	0.035 (2)	0.055 (2)	0.0019 (17)	0.0234 (19)	−0.0095 (17)
O3	0.069 (2)	0.041 (2)	0.077 (3)	−0.0039 (19)	0.025 (2)	−0.0124 (19)
C1	0.035 (3)	0.046 (3)	0.034 (3)	−0.002 (2)	0.001 (2)	−0.005 (2)
C2	0.043 (3)	0.049 (3)	0.031 (3)	−0.002 (2)	0.002 (2)	0.001 (2)
C3	0.042 (3)	0.058 (3)	0.047 (3)	−0.002 (2)	0.014 (2)	−0.013 (3)
C4	0.037 (3)	0.070 (4)	0.032 (3)	−0.002 (2)	0.007 (2)	0.000 (3)
C5	0.044 (3)	0.062 (4)	0.036 (3)	−0.016 (3)	0.004 (2)	0.006 (3)
C6	0.050 (3)	0.044 (3)	0.046 (3)	−0.008 (2)	0.005 (2)	0.000 (3)
C7	0.042 (3)	0.040 (3)	0.039 (3)	0.002 (2)	0.007 (2)	−0.004 (2)
C8	0.029 (3)	0.050 (3)	0.037 (3)	−0.003 (2)	0.008 (2)	0.000 (3)

C9	0.036 (3)	0.038 (3)	0.038 (3)	−0.002 (2)	0.015 (2)	−0.003 (2)
C10	0.040 (3)	0.032 (3)	0.053 (3)	0.002 (2)	0.015 (2)	−0.002 (2)
C11	0.047 (3)	0.043 (3)	0.043 (3)	0.005 (2)	0.016 (2)	0.008 (2)
C12	0.036 (3)	0.036 (3)	0.037 (3)	−0.005 (2)	0.013 (2)	−0.005 (2)
C13	0.055 (3)	0.033 (3)	0.052 (3)	0.000 (2)	0.020 (3)	−0.003 (3)
C14	0.058 (3)	0.032 (3)	0.043 (3)	0.004 (2)	0.021 (2)	0.005 (2)
C15	0.070 (4)	0.063 (4)	0.042 (3)	0.003 (3)	0.015 (3)	−0.001 (3)
C16	0.068 (4)	0.054 (3)	0.062 (4)	0.001 (3)	0.030 (3)	−0.011 (3)
C17	0.074 (4)	0.053 (4)	0.063 (4)	0.009 (3)	0.024 (3)	−0.009 (3)

Geometric parameters (Å, º)

Cl1—C3	1.736 (5)	C6—H6	0.9300
Cl2—C5	1.758 (5)	C7—H7	0.9300
N1—C7	1.288 (6)	C8—C9	1.480 (6)
N1—N2	1.367 (5)	C9—C10	1.387 (6)
N2—C8	1.381 (6)	C9—C14	1.394 (6)
N2—H2	0.8600	C10—C11	1.378 (6)
N3—C12	1.374 (6)	C10—H10	0.9300
N3—C15	1.447 (6)	C11—C12	1.414 (6)
N3—C16	1.456 (6)	C11—H11	0.9300
O1—C2	1.342 (5)	C12—C13	1.402 (6)
O1—H1	0.8200	C13—C14	1.374 (6)
O2—C8	1.222 (5)	C13—H13	0.9300
O3—C17	1.415 (6)	C14—H14	0.9300
O3—H3	0.8200	C15—H15A	0.9600
C1—C6	1.388 (6)	C15—H15B	0.9600
C1—C2	1.417 (6)	C15—H15C	0.9600
C1—C7	1.433 (6)	C16—H16A	0.9600
C2—C3	1.393 (6)	C16—H16B	0.9600
C3—C4	1.379 (7)	C16—H16C	0.9600
C4—C5	1.378 (7)	C17—H17A	0.9600
C4—H4	0.9300	C17—H17B	0.9600
C5—C6	1.372 (7)	C17—H17C	0.9600
C7—N1—N2	118.3 (4)	C14—C9—C8	125.3 (4)
N1—N2—C8	119.1 (4)	C11—C10—C9	122.2 (4)
N1—N2—H2	120.5	C11—C10—H10	118.9
C8—N2—H2	120.5	C9—C10—H10	118.9
C12—N3—C15	121.1 (4)	C10—C11—C12	120.6 (4)
C12—N3—C16	122.6 (4)	C10—C11—H11	119.7
C15—N3—C16	116.1 (4)	C12—C11—H11	119.7
C2—O1—H1	109.5	N3—C12—C13	121.7 (4)
C17—O3—H3	109.5	N3—C12—C11	121.6 (4)
C6—C1—C2	118.3 (4)	C13—C12—C11	116.7 (4)
C6—C1—C7	119.8 (5)	C14—C13—C12	121.6 (4)
C2—C1—C7	121.9 (5)	C14—C13—H13	119.2
O1—C2—C3	119.2 (5)	C12—C13—H13	119.2
O1—C2—C1	122.1 (4)	C13—C14—C9	121.5 (5)
C3—C2—C1	118.7 (5)	C13—C14—H14	119.3

C4—C3—C2	122.0 (5)	C9—C14—H14	119.3
C4—C3—Cl1	119.4 (4)	N3—C15—H15A	109.5
C2—C3—Cl1	118.6 (4)	N3—C15—H15B	109.5
C5—C4—C3	118.6 (5)	H15A—C15—H15B	109.5
C5—C4—H4	120.7	N3—C15—H15C	109.5
C3—C4—H4	120.7	H15A—C15—H15C	109.5
C6—C5—C4	120.9 (5)	H15B—C15—H15C	109.5
C6—C5—Cl2	119.9 (4)	N3—C16—H16A	109.5
C4—C5—Cl2	119.2 (4)	N3—C16—H16B	109.5
C5—C6—C1	121.5 (5)	H16A—C16—H16B	109.5
C5—C6—H6	119.3	N3—C16—H16C	109.5
C1—C6—H6	119.3	H16A—C16—H16C	109.5
N1—C7—C1	120.7 (4)	H16B—C16—H16C	109.5
N1—C7—H7	119.7	O3—C17—H17A	109.5
C1—C7—H7	119.7	O3—C17—H17B	109.5
O2—C8—N2	120.9 (4)	H17A—C17—H17B	109.5
O2—C8—C9	123.7 (4)	O3—C17—H17C	109.5
N2—C8—C9	115.4 (4)	H17A—C17—H17C	109.5
C10—C9—C14	117.3 (4)	H17B—C17—H17C	109.5
C10—C9—C8	117.4 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3^{i}	0.86	2.10	2.848 (5)	145
O1—H1···N1	0.82	1.91	2.600 (5)	141
O3—H3···O2	0.82	1.97	2.771 (5)	166

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.