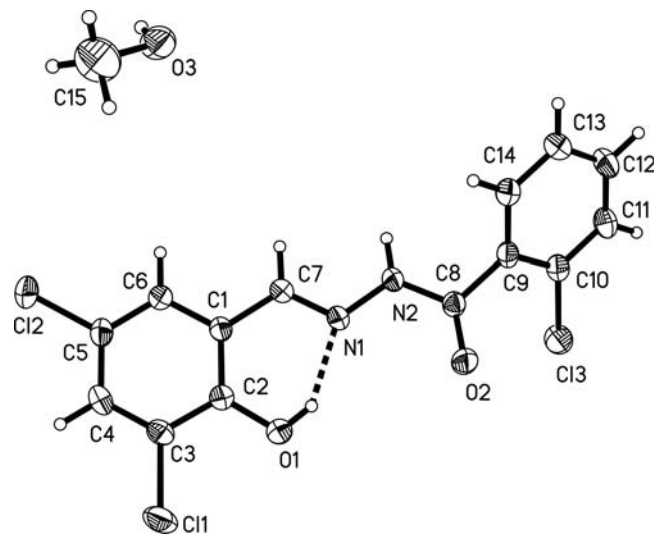


Crystal structure of 2-chloro-*N'*-(2-hydroxy-3,5-dichlorobenzylidene)-benzohydrazide — methanol (1:1), C₁₄H₉Cl₃N₂O₂ · CH₃OH

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Abstract

C₁₅H₁₃Cl₃N₂O₃, monoclinic, *P*2₁/*n* (no. 14), *a* = 11.172(3) Å, *b* = 9.713(3) Å, *c* = 15.671(4) Å, β = 96.516(3)°, *V* = 1689.5 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.035, *wR*_{ref}(*F*²) = 0.100, *T* = 298 K.

Source of material

3,5-Dichloro-2-hydroxybenzaldehyde (0.1 mmol, 19.0 mg) and 2-chlorobenzohydrazide (0.1 mmol, 17.0 mg) were refluxed in a 30 ml methanol for 30 min to give a clear colorless solution. Colorless block-shaped single crystals of the compound were formed by slow evaporation of the solvent over several days at room temperature.

Experimental details

H2 was located from a difference Fourier map and refined isotropically with *d*(N—H) restrained to 0.90(1) Å and *U*_{iso}(H) = 0.08 Å². Other H atoms were constrained to ideal geometries, with *d*(C—H) = 0.93–0.96 Å, *d*(O—H) = 0.82 Å, and *U*_{iso}(H) = 1.2 *U*_{eq}(C) and 1.5 *U*_{eq}(O1, O3, C15).

Discussion

Schiff base compounds are a class of important materials used in pharmaceutical and medicinal fields [1–3]. Schiff bases have also been used as versatile ligands in coordination chemistry [4–6]. Recently, the crystal structures of a number of Schiff base compounds bearing the hydrazone groups have been reported [7–10].

The independent unit of the title crystal structure consists of a Schiff base molecule and a methanol molecule of crystallization. The dihedral angle between the two substituted benzene rings is 49.0(2)°. All the bond lengths are within normal values and are comparable with those observed in the known compounds [7–10]. The Schiff base molecule adopts *E* configuration around the C7=N1 bond. An intramolecular O1–H1...N1 hydrogen bond forms in the Schiff base molecule. In the crystal structure, the Schiff base molecules are linked by methanol molecules through intermolecular N–H...O and O–H...O hydrogen bonds, forming chains along [010].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.12 × 0.15 × 0.17 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	5.57 cm ^{−1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
2θ _{max} :	54°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	10319, 3661
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2545
<i>N</i> (<i>param</i>) _{refined} :	214
Programs:	SHELXS-97, SHELXL-97 [11]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	4e	0.816(2)	0.126(2)	0.160(2)	0.080
H(1)	4e	0.5129	−0.0566	0.1372	0.080
H(3)	4e	0.6269	0.8426	0.3208	0.101
H(4)	4e	0.1620	0.1906	−0.0210	0.058
H(6)	4e	0.4889	0.3607	0.0244	0.055
H(7)	4e	0.6592	0.2348	0.0968	0.051
H(11)	4e	1.2130	−0.1746	0.2617	0.068
H(12)	4e	1.2697	0.0225	0.3370	0.075
H(13)	4e	1.1284	0.1861	0.3614	0.072
H(14)	4e	0.9298	0.1540	0.3088	0.061
H(15A)	4e	0.4968	0.9027	0.4229	0.175
H(15B)	4e	0.4549	0.7531	0.3966	0.175
H(15C)	4e	0.4508	0.8693	0.3269	0.175

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cl(1)	4e	0.18898(5)	−0.07151(7)	0.05047(4)	0.0536(4)	0.0854(5)	0.0746(4)	−0.0277(3)	−0.0002(3)	0.0141(3)
Cl(2)	4e	0.26246(6)	0.44658(6)	−0.05126(4)	0.0634(4)	0.0599(4)	0.0790(4)	0.0156(3)	−0.0136(3)	0.0135(3)
Cl(3)	4e	0.99708(6)	−0.27720(6)	0.18236(4)	0.0618(4)	0.0590(4)	0.0714(4)	0.0081(3)	0.0013(3)	−0.0132(3)
N(1)	4e	0.6504(1)	0.0546(2)	0.1461(1)	0.0324(8)	0.0426(9)	0.0480(9)	0.0015(7)	−0.0001(7)	0.0000(7)
N(2)	4e	0.7715(1)	0.0546(2)	0.1741(1)	0.0320(8)	0.044(1)	0.055(1)	−0.0004(7)	−0.0007(7)	0.0087(8)
O(1)	4e	0.4403(1)	−0.0607(2)	0.1221(1)	0.0479(8)	0.0478(8)	0.0634(9)	−0.0053(7)	−0.0014(7)	0.0132(7)
O(2)	4e	0.7600(1)	−0.1503(2)	0.2407(1)	0.0451(8)	0.0529(9)	0.082(1)	−0.0051(7)	0.0049(8)	0.0224(8)
O(3)	4e	0.6102(1)	0.8015(2)	0.3636(1)	0.058(1)	0.060(1)	0.087(1)	0.0038(8)	0.0201(9)	−0.0023(9)
C(1)	4e	0.4815(2)	0.1674(2)	0.0720(1)	0.037(1)	0.041(1)	0.038(1)	0.0030(8)	0.0016(8)	−0.0019(8)
C(2)	4e	0.4038(2)	0.0562(2)	0.0810(1)	0.041(1)	0.043(1)	0.037(1)	0.0005(8)	0.0054(8)	0.0003(8)
C(3)	4e	0.2843(2)	0.0675(2)	0.0445(1)	0.040(1)	0.057(1)	0.041(1)	−0.0069(9)	0.0061(9)	−0.0012(9)
C(4)	4e	0.2419(2)	0.1851(2)	0.0029(1)	0.035(1)	0.067(1)	0.041(1)	0.005(1)	0.0003(9)	−0.002(1)
C(5)	4e	0.3186(2)	0.2948(2)	−0.0030(1)	0.046(1)	0.049(1)	0.044(1)	0.0096(9)	−0.0012(9)	0.0015(9)
C(6)	4e	0.4376(2)	0.2866(2)	0.0301(1)	0.045(1)	0.041(1)	0.050(1)	0.0007(9)	−0.0006(9)	−0.0003(9)
C(7)	4e	0.6086(2)	0.1612(2)	0.1053(1)	0.038(1)	0.043(1)	0.047(1)	−0.0017(8)	0.0010(9)	−0.0001(9)
C(8)	4e	0.8184(2)	−0.0508(2)	0.2226(1)	0.037(1)	0.044(1)	0.044(1)	0.0032(9)	0.0056(8)	0.0048(9)
C(9)	4e	0.9493(2)	−0.0312(2)	0.2539(1)	0.037(1)	0.046(1)	0.041(1)	0.0032(8)	0.0032(8)	0.0084(9)
C(10)	4e	1.0364(2)	−0.1290(2)	0.2408(1)	0.042(1)	0.050(1)	0.043(1)	0.0037(9)	0.0027(9)	0.0042(9)
C(11)	4e	1.1556(2)	−0.1088(3)	0.2714(2)	0.040(1)	0.068(2)	0.061(1)	0.012(1)	0.002(1)	0.002(1)
C(12)	4e	1.1892(2)	0.0089(3)	0.3162(2)	0.040(1)	0.075(2)	0.070(2)	−0.001(1)	−0.008(1)	0.003(1)
C(13)	4e	1.1051(2)	0.1068(3)	0.3306(2)	0.054(1)	0.057(1)	0.067(2)	−0.004(1)	−0.008(1)	−0.003(1)
C(14)	4e	0.9863(2)	0.0870(2)	0.2993(1)	0.047(1)	0.047(1)	0.057(1)	0.005(1)	0.001(1)	0.001(1)
C(15)	4e	0.4944(3)	0.8341(4)	0.3786(3)	0.061(2)	0.115(3)	0.180(4)	0.015(2)	0.044(2)	−0.013(3)

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