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Crystal structure of 3-chloro-*N′*-(2-hydroxy-3,5-diiodobenzylidene)-benzohydrazide methanol solvate, $C_{14}H_9ClI_2N_2O_2 \cdot CH_3OH$

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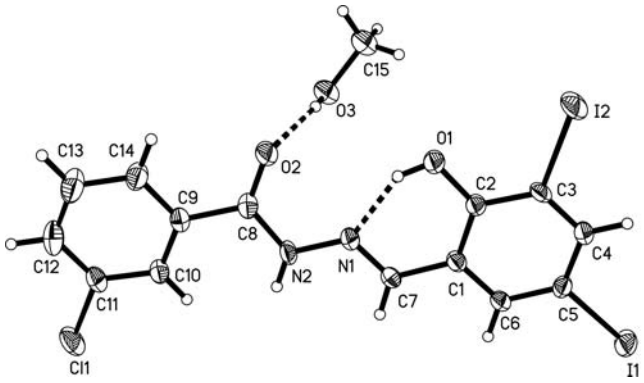

Abstract

$C_{15}H_{13}ClI_2N_2O_3$, triclinic, $P\bar{1}$ (no. 2), a = 9.057(2) Å, b = 9.478(2) Å, c = 11.343(2) Å, α = 95.600(2)°, β = 109.723(2)°, γ = 98.840(2)°, V = 894.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.080, T = 298 K.

Source of material

2-Hydroxy-3,5-diiodobenzaldehyde (0.1 mmol, 37.4 mg) and 3-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 the d(N—H) restrained to 0.90(1) Å, and U_{iso}(H) restrained to 0.08 Å^2. 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) or 1.5 U_{eq}(O,C15).

Discussion

In recent years, Schiff base compounds have been received considerable attention has for their pharmaceutical and medicinal applications [1-3], coordination chemistry [4-6], and structural chemistry [7-10].

The title crystal structure consists of Schiff base molecules and methanol molecules of crystallization. The dihedral angle between the two substituted benzene rings is 4.8(4)°. All the bond lengths are within normal values and are comparable with those observed in the compounds cited above. The Schiff base molecule adopts *E* configuration in respect to the C7=N1 bond. There is an intramolecular O1–H1···N1 hydrogen bond in the Schiff base molecule. In the crystal structure, the strong hydrogen bonding interactions among atoms O2, O3 and N2: N2–H2···O3 [d(N2–H2···O3) = 2.911(5) Å, ∠N2–H2···O3 = 159(5)°] and O3–H3···O2 [d(O3–H3···O2) = 2.780(5) Å, ∠O3–H3···O2 = 163(5)°], link the adjacent two Schiff base molecules to form a dimer.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.30 × 0.30 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	36.82 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6937, 3777
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2509
$N(param)_{refined}$:	214
Programs:	SHELXS-97, SHELXL-97 [11]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.8014	0.7597	–0.1191	0.076
H(3)	2*i*	0.6999	0.4518	0.8195	0.095
H(4)	2*i*	1.0503	1.1190	–0.3266	0.049
H(6)	2*i*	1.3088	0.9641	–0.0243	0.047
H(7)	2*i*	1.1815	0.7902	0.0781	0.046
H(10)	2*i*	1.0505	0.4708	0.2862	0.050
H(12)	2*i*	0.7750	0.2153	0.4241	0.077
H(13)	2*i*	0.5491	0.2478	0.2672	0.085
H(14)	2*i*	0.5729	0.3903	0.1183	0.064
H(15A)	2*i*	0.4977	0.3979	0.6375	0.112
H(15B)	2*i*	0.6239	0.3751	0.5747	0.112
H(15C)	2*i*	0.6261	0.5300	0.6388	0.112
H(2)	2*i*	1.052(4)	0.614(6)	0.162(4)	0.080

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I(1)	2*i*	1.41007(4)	1.17530(5)	–0.18482(4)	0.0396(2)	0.0744(3)	0.0757(3)	–0.0018(2)	0.0148(2)	0.0365(2)
I(2)	2*i*	0.69752(4)	0.99101(5)	–0.39404(4)	0.0387(2)	0.1037(4)	0.0777(3)	0.0163(2)	0.0008(2)	0.0545(3)
Cl(1)	2*i*	1.1021(2)	0.3146(2)	0.4895(1)	0.090(1)	0.067(1)	0.0456(8)	0.0254(9)	0.0148(8)	0.0269(7)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	0.9566(5)	0.7058(4)	0.0115(3)	0.041(2)	0.039(2)	0.037(2)	0.008(2)	0.011(2)	0.016(2)
N(2)	2*i*	0.9593(5)	0.6214(4)	0.1032(4)	0.040(2)	0.046(3)	0.038(2)	0.012(2)	0.014(2)	0.025(2)
O(1)	2*i*	0.7836(4)	0.8048(4)	–0.1788(3)	0.033(2)	0.062(3)	0.054(2)	0.003(2)	0.009(2)	0.029(2)
O(2)	2*i*	0.7002(4)	0.5242(4)	–0.0101(3)	0.038(2)	0.075(3)	0.050(2)	0.007(2)	0.011(2)	0.029(2)
O(3)	2*i*	0.7121(5)	0.4032(4)	0.7608(3)	0.055(2)	0.078(3)	0.051(2)	0.012(2)	0.008(2)	0.022(2)
C(1)	2*i*	1.0709(5)	0.8807(5)	–0.0833(4)	0.035(3)	0.039(3)	0.032(2)	0.010(2)	0.010(2)	0.011(2)
C(2)	2*i*	0.9215(5)	0.8855(5)	–0.1746(4)	0.033(3)	0.037(3)	0.036(2)	0.008(2)	0.012(2)	0.011(2)
C(3)	2*i*	0.9182(5)	0.9771(5)	–0.2629(4)	0.030(3)	0.051(3)	0.030(2)	0.010(2)	0.002(2)	0.015(2)
C(4)	2*i*	1.0556(6)	1.0590(5)	–0.2657(4)	0.042(3)	0.041(3)	0.039(3)	0.008(2)	0.012(2)	0.017(2)
C(5)	2*i*	1.2020(5)	1.0516(5)	–0.1771(4)	0.035(3)	0.037(3)	0.044(3)	0.003(2)	0.016(2)	0.013(2)
C(6)	2*i*	1.2102(6)	0.9657(5)	–0.0853(4)	0.033(3)	0.045(3)	0.034(2)	0.008(2)	0.006(2)	0.013(2)
C(7)	2*i*	1.0835(6)	0.7884(5)	0.0150(4)	0.037(3)	0.040(3)	0.035(3)	0.010(2)	0.005(2)	0.014(2)
C(8)	2*i*	0.8186(6)	0.5314(5)	0.0855(5)	0.041(3)	0.043(3)	0.046(3)	0.015(2)	0.022(2)	0.018(2)
C(9)	2*i*	0.8131(6)	0.4435(5)	0.1867(4)	0.049(3)	0.042(3)	0.037(3)	0.010(2)	0.020(2)	0.012(2)
C(10)	2*i*	0.9495(6)	0.4256(5)	0.2816(4)	0.046(3)	0.044(3)	0.039(3)	0.011(2)	0.018(2)	0.016(2)
C(11)	2*i*	0.9322(7)	0.3388(5)	0.3697(4)	0.062(4)	0.043(3)	0.036(3)	0.018(3)	0.019(3)	0.015(2)
C(12)	2*i*	0.7843(8)	0.2731(6)	0.3643(6)	0.088(5)	0.054(4)	0.073(4)	0.023(4)	0.048(4)	0.034(3)
C(13)	2*i*	0.6498(8)	0.2922(7)	0.2708(6)	0.064(4)	0.078(5)	0.092(5)	0.016(4)	0.046(4)	0.040(4)
C(14)	2*i*	0.6642(6)	0.3774(6)	0.1819(5)	0.045(3)	0.060(4)	0.064(3)	0.011(3)	0.025(3)	0.027(3)
C(15)	2*i*	0.6063(7)	0.4286(7)	0.6434(5)	0.061(4)	0.099(5)	0.055(4)	0.008(4)	0.008(3)	0.029(4)

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