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

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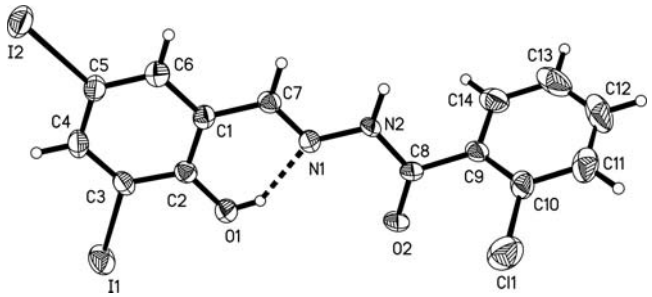

Abstract

$C_{14}H_9ClI_2N_2O_2$, monoclinic, $P12_1/c1$ (no. 14), a = 14.405(3) Å, b = 11.550(3) Å, c = 9.807(2) Å, β = 90.340(2)°, V = 1631.6 Å^3, Z = 4, $R_{gf}(F)$ = 0.042, $wR_{ref}(F^2)$ = 0.082, T = 298 K.

Source of material

3,5-Diiodo-2-hydroxybenzaldehyde (0.1 mmol, 37.4 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 title compound were formed by slow evaporation of the solvent over several days at room temperature.

Experimental details

H2 atom was located from a difference Fourier map and refined isotropically with 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 positions with d(C—H) = 0.93 Å, d(O—H) = 0.82 Å, and U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O). For the C9 - C14 benzene ring, the U_{22} values are larger than the U_{33} and U_{11}, which may be caused by the slight distortion of the C9 - C14 benzene ring.

Discussion

Schiff base compounds are a class of important materials used as 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 several Schiff base compounds bearing the hydrazone groups have been reported [7-10].

In the crystal structure of the title Schiff base compound, the dihedral angle between the two substituted benzene rings is 65.5(5)°. All the bond lengthes are within normal ranges and are comparable with those observed in the compounds cited above. The molecule adopts *E* configuration with respect to the C7=N1 bond. There exists an intramolecular O1–H1···N1 hydrogen bond. In the crystal structure, molecules are interlinked through intermolecular N–H···O hydrogen bonds, forming chains along [001].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.21 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	40.23 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8112, 3084
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2021
$N(param)_{refined}$:	194
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)	4*e*	0.6124	0.3605	0.7422	0.080
H(4)	4*e*	0.2842	0.4348	0.8830	0.065
H(6)	4*e*	0.4794	0.3248	1.1448	0.058
H(7)	4*e*	0.6404	0.2917	1.0712	0.050
H(11)	4*e*	1.0689	–0.0115	0.8135	0.093
H(12)	4*e*	1.1524	0.0960	0.9664	0.125
H(13)	4*e*	1.0931	0.2626	1.0564	0.122
H(14)	4*e*	0.9470	0.3246	0.9900	0.084
H(2)	4*e*	0.789(4)	0.252(5)	1.004(2)	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)	4*e*	0.37313(3)	0.47421(4)	0.60258(5)	0.0642(3)	0.0859(4)	0.0692(3)	0.0148(3)	–0.0123(2)	0.0194(3)
I(2)	4*e*	0.27345(3)	0.37447(4)	1.18621(5)	0.0494(3)	0.0853(4)	0.0761(3)	–0.0088(2)	0.0197(2)	–0.0115(3)
Cl(1)	4*e*	0.8983(2)	0.0082(2)	0.6939(2)	0.090(2)	0.099(2)	0.102(2)	–0.006(1)	0.009(1)	–0.034(1)
N(1)	4*e*	0.6825(3)	0.3041(4)	0.8868(4)	0.037(3)	0.050(3)	0.041(3)	0.002(2)	–0.001(2)	0.002(2)
N(2)	4*e*	0.7701(3)	0.2644(4)	0.9174(4)	0.036(3)	0.064(3)	0.026(3)	0.007(2)	–0.005(2)	0.007(2)
O(1)	4*e*	0.5628(3)	0.3885(4)	0.7174(4)	0.047(3)	0.068(3)	0.046(2)	0.010(2)	–0.001(2)	0.004(2)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(2)	4*e*	0.8059(3)	0.2550(5)	0.6952(4)	0.067(3)	0.162(5)	0.021(2)	0.032(3)	–0.004(2)	0.002(3)
C(1)	4*e*	0.5290(4)	0.3484(4)	0.9521(6)	0.040(3)	0.034(3)	0.049(4)	0.002(3)	0.001(3)	–0.005(3)
C(2)	4*e*	0.5017(4)	0.3837(4)	0.8204(6)	0.043(3)	0.039(3)	0.042(4)	–0.001(3)	–0.004(3)	–0.003(3)
C(3)	4*e*	0.4101(4)	0.4175(5)	0.7985(6)	0.041(4)	0.044(3)	0.054(4)	0.004(3)	–0.012(3)	–0.006(3)
C(4)	4*e*	0.3455(4)	0.4140(5)	0.9004(7)	0.034(3)	0.054(4)	0.073(5)	0.003(3)	–0.001(3)	–0.016(4)
C(5)	4*e*	0.3728(4)	0.3789(5)	1.0300(7)	0.038(4)	0.056(4)	0.055(4)	0.001(3)	0.003(3)	–0.008(3)
C(6)	4*e*	0.4623(4)	0.3472(5)	1.0571(6)	0.048(4)	0.045(4)	0.051(4)	–0.002(3)	0.000(3)	0.000(3)
C(7)	4*e*	0.6234(4)	0.3108(5)	0.9824(6)	0.043(3)	0.048(4)	0.036(3)	–0.003(3)	–0.001(3)	0.006(3)
C(8)	4*e*	0.8278(4)	0.2401(5)	0.8138(6)	0.047(4)	0.068(4)	0.027(4)	0.005(3)	0.001(3)	0.001(3)
C(9)	4*e*	0.9205(4)	0.1954(6)	0.8584(6)	0.038(4)	0.078(5)	0.029(3)	–0.002(3)	0.001(3)	0.007(3)
C(10)	4*e*	0.9576(4)	0.0950(6)	0.8063(6)	0.037(4)	0.079(5)	0.054(4)	0.002(3)	0.008(3)	0.008(4)
C(11)	4*e*	1.0444(5)	0.0569(7)	0.8480(8)	0.058(5)	0.099(6)	0.076(6)	0.016(4)	0.027(4)	0.028(5)
C(12)	4*e*	1.0936(6)	0.121(1)	0.940(1)	0.033(5)	0.19(1)	0.092(7)	0.006(6)	0.009(5)	0.060(8)
C(13)	4*e*	1.0588(6)	0.220(1)	0.9934(9)	0.049(6)	0.19(1)	0.067(6)	–0.036(6)	–0.017(4)	0.014(7)
C(14)	4*e*	0.9716(5)	0.2572(7)	0.9530(7)	0.056(5)	0.104(6)	0.050(4)	–0.018(4)	–0.002(4)	0.000(4)

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