Crystal structure of 2-methoxy-*N′*-(3,5-dichloro-2-hydroxybenzylidene)benzohydrazide, $C_{15}H_{12}Cl_2N_2O_3$

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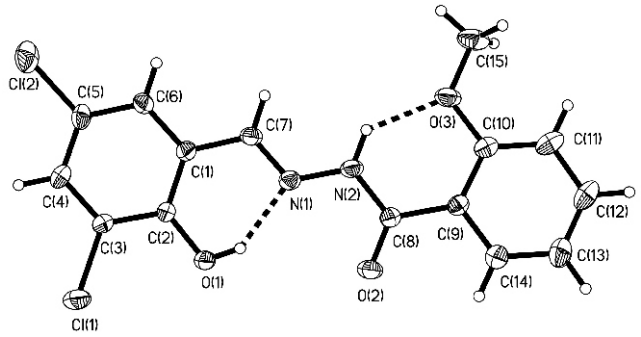

Abstract

$C_{15}H_{12}Cl_2N_2O_3$, monoclinic, *C*1*c*1 (no. 9), a = 10.845(2) Å, b = 12.773(2) Å, c = 10.853(2) Å, β = 96.688(2)°, V = 1493.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.081, T = 298 K.

Source of material

3,5-Dichlorosalicylaldehyde (0.19 g, 1 mmol) and 2-methoxybenzohydrazide (0.17 g, 1 mmol) were stirred at room temperature in MeOH (25 mL) for 30 min. Colorless block-shaped crystals were formed by slow evaporation of the solution in air for a week.

Experimental details

The amino H atom was located from a difference Fourier map and refined isotropically, with d(N—H) restrained to 0.90(1) Å. The remaining hydrogen atoms were constrained to ideal positions, with d(C—H) = 0.93-0.96 Å, d(O—H) = 0.82 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O1 and C15). The absolute structure of the compound is evidenced by the Flack parameter of –0.03(6) [1]. The displacement parameters of Cl atoms are slightly larger than the other atoms, which might be caused by terminal effects. The disorder of the molecule is very little, which is common for the data collection at ambient conditions.

Discussion

Hydrazone derivatives have been widely investigated for their biological activity [2-5]. Recently, the crystal structures of a number of hydrazone derivatives have been reported [6-9].

The molecule of the compound is approximately coplanar with mean deviation from the plane defined by the non-hydrogen atoms of 0.060(2) Å. The dihedral angle between the two substituted benzene rings is 5.0(3)°. The two intramolecular hydrogen bonds O1–H1···N1 and N2–H2···O3 may contribute to the planarity of the molecule. All the bond values in the compound are similar to those reported in the hydrazone derivatives [10-12]. The crystal structure is stabilized by intermolecular N–H···O hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.20 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.48 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	56.62°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6426, 3218
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2573
$N(param)_{refined}$:	205
Programs:	SHELXS-97, SHELXL-97, SHELXTL [13]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.0175	0.5427	0.2808	0.065
H(7)	4*a*	0.1048	0.5763	0.5856	0.044
H(6)	4*a*	0.0498	0.7632	0.5983	0.045
H(4)	4*a*	–0.1048	0.9200	0.3005	0.051
H(11)	4*a*	0.3218	0.0980	0.7044	0.060
H(14)	4*a*	0.1434	0.1445	0.3029	0.056
H(12)	4*a*	0.3061	–0.0403	0.5678	0.070
H(13)	4*a*	0.2145	–0.0178	0.3670	0.068
H(15A)	4*a*	0.3861	0.2567	0.7845	0.094
H(15B)	4*a*	0.2588	0.2300	0.8323	0.094
H(15C)	4*a*	0.3031	0.3467	0.8284	0.094
H(2)	4*a*	0.146(2)	0.398(2)	0.568(1)	0.047(8)

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}
Cl(1)	4*a*	–0.11992(8)	0.77316(5)	0.10855(7)	0.0863(5)	0.0512(4)	0.0429(4)	0.0045(4)	–0.0099(4)	0.0126(3)
Cl(2)	4*a*	–0.04615(8)	0.96474(5)	0.55175(8)	0.0771(5)	0.0383(3)	0.0770(5)	–0.0039(4)	0.0223(4)	–0.0189(4)
N(2)	4*a*	0.1288(2)	0.4048(2)	0.4856(2)	0.058(2)	0.035(1)	0.029(1)	0.011(1)	0.000(1)	0.0045(9)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(2)	4*a*	–0.0123(2)	0.6772(2)	0.3153(2)	0.037(1)	0.030(1)	0.033(1)	–0.005(1)	0.002(1)	0.002(1)
O(3)	4*a*	0.2417(2)	0.2899(2)	0.6647(2)	0.061(1)	0.054(1)	0.031(1)	0.0136(9)	–0.0040(9)	0.0033(9)
O(1)	4*a*	–0.0062(2)	0.5943(1)	0.2396(1)	0.065(1)	0.0328(9)	0.0293(9)	0.0013(9)	–0.0010(9)	0.0004(7)
C(8)	4*a*	0.1351(2)	0.3219(2)	0.4087(2)	0.037(1)	0.035(1)	0.033(1)	–0.001(1)	0.004(1)	0.002(1)
O(2)	4*a*	0.1027(2)	0.3306(1)	0.2975(2)	0.073(1)	0.049(1)	0.031(1)	0.010(1)	–0.006(1)	0.0015(9)
C(9)	4*a*	0.1832(2)	0.2207(2)	0.4645(2)	0.034(1)	0.034(1)	0.036(1)	0.003(1)	0.008(1)	0.004(1)
N(1)	4*a*	0.0853(2)	0.4974(1)	0.4337(2)	0.043(1)	0.034(1)	0.033(1)	0.0066(9)	0.0009(9)	0.0027(9)
C(1)	4*a*	0.0298(2)	0.6747(2)	0.4421(2)	0.036(1)	0.033(1)	0.033(1)	–0.002(1)	0.006(1)	0.001(1)
C(7)	4*a*	0.0788(2)	0.5785(2)	0.5009(2)	0.042(2)	0.040(1)	0.027(1)	0.002(1)	0.002(1)	0.001(1)
C(6)	4*a*	0.0210(2)	0.7643(2)	0.5142(2)	0.043(2)	0.039(1)	0.032(2)	–0.004(1)	0.007(1)	–0.005(1)
C(4)	4*a*	–0.0717(2)	0.8584(2)	0.3360(3)	0.049(2)	0.029(1)	0.050(2)	–0.002(1)	0.009(1)	0.007(1)
C(3)	4*a*	–0.0634(2)	0.7703(2)	0.2646(2)	0.045(2)	0.038(1)	0.034(1)	–0.005(1)	0.001(1)	0.009(1)
C(10)	4*a*	0.2366(2)	0.2051(2)	0.5868(2)	0.039(2)	0.042(1)	0.039(2)	0.006(1)	0.009(1)	0.007(1)
C(5)	4*a*	–0.0304(2)	0.8541(2)	0.4607(2)	0.047(2)	0.030(1)	0.051(2)	–0.005(1)	0.013(1)	–0.007(1)
C(11)	4*a*	0.2840(3)	0.1075(2)	0.6238(3)	0.044(2)	0.052(2)	0.056(2)	0.010(1)	0.011(1)	0.021(1)
C(14)	4*a*	0.1779(3)	0.1354(2)	0.3848(3)	0.050(2)	0.043(1)	0.046(2)	0.001(1)	0.005(1)	0.000(1)
C(12)	4*a*	0.2753(3)	0.0250(2)	0.5419(3)	0.058(2)	0.039(2)	0.080(2)	0.012(1)	0.018(2)	0.015(2)
C(13)	4*a*	0.2214(3)	0.0382(2)	0.4220(3)	0.064(2)	0.039(2)	0.070(2)	0.003(1)	0.017(2)	–0.003(2)
C(15)	4*a*	0.3023(3)	0.2800(3)	0.7874(3)	0.064(2)	0.083(2)	0.038(2)	0.019(2)	–0.006(2)	0.006(2)

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