Crystal structure of [3,4]dioxole-*N′*-(3,5-dichloro-2-hydroxybenzylidene)-benzohydrazide — methanol (1:1), $C_{15}H_{10}Cl_2N_2O_4 \cdot CH_3OH$

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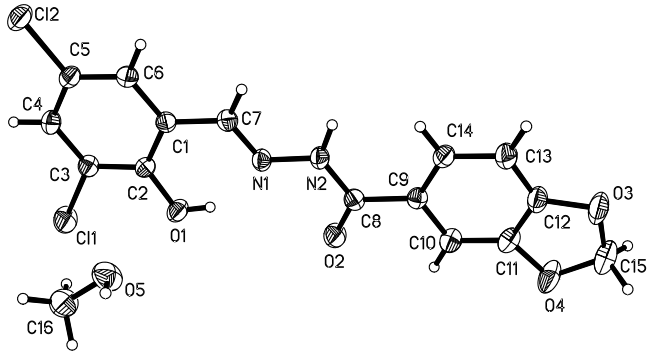

Abstract

$C_{16}H_{14}Cl_2N_2O_5$, monoclinic, $P12_1/c1$ (no. 14), a = 8.041(1) Å, b = 13.744(2) Å, c = 15.530(2) Å, β = 100.201(2)°, V = 1689.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.113, T = 298 K.

Source of material

[3,4]Dioxolebenzohydrazide (18.2 mg, 0.1 mmol) was dissolved in methanol (10 ml), then 3,5-dichloro-2-hydroxybenzaldehyde (19.2 mg, 0.1 mmol) was added slowly into the solution, and the mixture was stirred at room temperature for 3 h. The resulting solution was left in air for a few days, yielding colorless block-shaped crystals.

Experimental details

H2 was located in a difference Fourier map and refined with N—H distance restrained to 0.90(1) Å and fixed U_{iso}. The other H atoms were placed in calculated positions with d(C—H) = 0.93 - 0.97 Å, d(O—H) = 0.82 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O).

Discussion

Hyrazone compounds have shown interesting structures and biological properties. Recently, a great deal of hydrazone derivatives and their metal complexes have been reported [1-5]. As part of the ongoing investigation of the crystal structure of such compounds, the crystal structure of a new hydrazone compound is reported in this paper.

The molecular structure of the title compound consists of a [3,4]dioxolebenzohydrazide and a 3,5-dichloro-2-hydroxybenzylidene groups that are located on opposite sides of the C=N bond showing an *E* configuration. The dihedral angle between the two substituted benzene rings C1-C6 and C9-C14 is 4.5(2)°, indicating the molecule is nearly coplanar. The crystal structure involves an intramolecular O1–H1···N1 hydrogen bond. The methanol molecules are linked to the hydrazone molecules through intermolecular N2–H2···O5 and O5–H5···O2 hydrogen bonds. The bond lengths and angles are in good agreement with the corresponding values observed in the similar structures [6-8]. The torsion angles of C7–N1–N2–C8 and N1–N2–C8–C9 are 0.4(2) and 3.0(2)°, respectively.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.23 × 0.25 × 0.27 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.15 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	53.7°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9443, 3590
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2508
$N(param)_{refined}$:	232
Programs:	SHELXS-97 [9], SHELXL-97 [10], SHELXTL [11]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.6595	0.3650	0.4833	0.080
H(5)	4*e*	0.0273	0.3071	0.5692	0.111
H(4)	4*e*	0.2447	0.3398	0.2031	0.054
H(6)	4*e*	0.4635	0.5915	0.2891	0.052
H(7)	4*e*	0.6686	0.6015	0.4253	0.050
H(10)	4*e*	1.1424	0.4490	0.7717	0.057
H(13)	4*e*	1.1794	0.7863	0.7416	0.075
H(14)	4*e*	1.0031	0.6946	0.6369	0.061
H(15A)	4*e*	1.5577	0.6216	0.9424	0.090
H(15B)	4*e*	1.4125	0.6377	0.9968	0.090
H(16A)	4*e*	–0.0002	0.1500	0.5671	0.109
H(16B)	4*e*	0.0718	0.1519	0.4797	0.109
H(16C)	4*e*	–0.1053	0.1979	0.4832	0.109
H(2)	4*e*	0.868(3)	0.6084(9)	0.541(2)	0.080

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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*e*	0.36993(9)	0.20619(4)	0.33429(4)	0.0827(5)	0.0390(3)	0.0719(4)	–0.0080(3)	–0.0085(3)	–0.0038(3)
Cl(2)	4*e*	0.23227(8)	0.53164(5)	0.14271(4)	0.0673(4)	0.0655(4)	0.0510(4)	0.0049(3)	–0.0109(3)	0.0107(3)
O(1)	4*e*	0.5965(2)	0.3284(1)	0.4510(1)	0.066(1)	0.0435(8)	0.0436(9)	–0.0019(7)	–0.0087(7)	0.0047(7)
O(2)	4*e*	0.9374(2)	0.4130(1)	0.6370(1)	0.073(1)	0.0395(9)	0.056(1)	–0.0053(8)	–0.0011(8)	–0.0028(7)
O(3)	4*e*	1.3757(2)	0.7086(1)	0.8840(1)	0.092(1)	0.077(1)	0.053(1)	–0.013(1)	–0.020(1)	–0.015(1)
O(4)	4*e*	1.3541(2)	0.5422(1)	0.9009(1)	0.091(1)	0.074(1)	0.056(1)	0.013(1)	–0.028(1)	–0.0069(9)
O(5)	4*e*	0.0839(2)	0.2767(1)	0.5393(1)	0.088(1)	0.060(1)	0.077(1)	–0.021(1)	0.022(1)	–0.0132(9)
N(1)	4*e*	0.7453(2)	0.4923(1)	0.4985(1)	0.0410(9)	0.0458(9)	0.0374(9)	–0.0066(8)	0.0047(7)	–0.0071(8)
N(2)	4*e*	0.8538(2)	0.5470(1)	0.5573(1)	0.046(1)	0.0399(9)	0.040(1)	–0.0062(8)	–0.0008(8)	–0.0044(8)
C(1)	4*e*	0.5408(2)	0.4789(1)	0.3697(1)	0.038(1)	0.041(1)	0.037(1)	–0.0016(9)	0.0077(9)	–0.0018(9)
C(2)	4*e*	0.5174(2)	0.3782(1)	0.3812(1)	0.041(1)	0.039(1)	0.034(1)	0.0022(8)	0.0060(9)	–0.0005(8)
C(3)	4*e*	0.4038(3)	0.3289(1)	0.3182(1)	0.046(1)	0.036(1)	0.043(1)	0.0004(9)	0.0051(9)	–0.0040(9)
C(4)	4*e*	0.3180(3)	0.3746(2)	0.2451(1)	0.043(1)	0.050(1)	0.042(1)	–0.0010(9)	0.0022(9)	–0.008(1)
C(5)	4*e*	0.3420(3)	0.4731(2)	0.2349(1)	0.041(1)	0.050(1)	0.038(1)	0.0049(9)	0.0034(9)	0.0034(9)
C(6)	4*e*	0.4505(3)	0.5249(2)	0.2965(1)	0.045(1)	0.040(1)	0.044(1)	–0.0015(9)	0.0062(9)	0.0022(9)
C(7)	4*e*	0.6577(2)	0.5348(2)	0.4328(1)	0.045(1)	0.040(1)	0.041(1)	–0.0043(9)	0.0064(9)	–0.0026(9)
C(8)	4*e*	0.9462(2)	0.5009(2)	0.6275(1)	0.041(1)	0.042(1)	0.042(1)	–0.0018(9)	0.0102(9)	–0.0048(9)
C(9)	4*e*	1.0562(2)	0.5634(2)	0.6925(1)	0.038(1)	0.042(1)	0.037(1)	0.0011(9)	0.0081(9)	–0.0035(9)
C(10)	4*e*	1.1496(3)	0.5160(2)	0.7648(1)	0.052(1)	0.042(1)	0.047(1)	0.008(1)	0.002(1)	–0.002(1)
C(11)	4*e*	1.2515(3)	0.5715(2)	0.8247(1)	0.049(1)	0.057(1)	0.039(1)	0.011(1)	0.0011(9)	–0.004(1)
C(12)	4*e*	1.2642(3)	0.6703(2)	0.8158(1)	0.054(1)	0.060(2)	0.042(1)	–0.007(1)	0.002(1)	–0.013(1)
C(13)	4*e*	1.1722(3)	0.7191(2)	0.7469(2)	0.081(2)	0.049(1)	0.051(1)	–0.015(1)	–0.005(1)	–0.003(1)
C(14)	4*e*	1.0674(3)	0.6637(2)	0.6848(1)	0.062(1)	0.046(1)	0.041(1)	–0.005(1)	–0.004(1)	0.004(1)
C(15)	4*e*	1.4364(4)	0.6279(2)	0.9384(2)	0.071(2)	0.092(2)	0.053(2)	0.011(2)	–0.011(1)	–0.022(2)
C(16)	4*e*	0.0065(4)	0.1871(2)	0.5155(2)	0.089(2)	0.053(2)	0.073(2)	–0.014(1)	0.006(2)	0.001(1)

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