Crystal structure of *N′*-(3-hydroxybenzylidene)-4-dimethylamino benzohydrazide, $C_{16}H_{17}N_3O_2$

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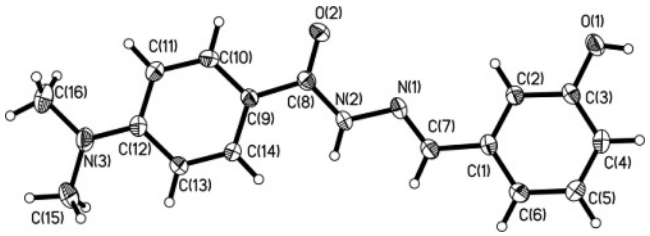

Abstract

$C_{16}H_{17}N_3O_2$, monoclinic, $P2_1/n$ (no. 14), a = 10.825(3) Å, b = 8.916(2) Å, c = 15.310(2) Å, β = 106.291(2)°, V = 1418.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.0473, $wR_{ref}(F^2)$ = 0.1344, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.20×0.20×0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.90 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD area-detector, ω
$2\theta_{max}$:	53°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6903, 2866
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1836
$N(param)_{refined}$:	196
Programs:	SADABS [13], SMART, SAINT [14] SHELX [15]

Source of material

The compound was prepared by the condensation reaction of 3-hydroxybenzaldehyde (1 mmol, 0.122 g) and 4-dimethylaminobenzohydrazide (1 mmol, 0.179 g) in anhydrous methanol (30 ml) at ambient temperature. Colourless block-shaped single crystals suitable for X-ray structural determination were obtained by slow evaporation of the solution for a week.

Experimental details

H(2) was located from a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90(1) Å. The remaining H atoms were positioned geometrically and constrained to ride on their parent atoms, with C–H distances of 0.93-0.96 Å, O–H distance of 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq} (O1 and C_{methyl}).

Discussion

Considerable attention has been focused on hydrazones and their medicinal applications [1-5]. The study of the crystal structures of such compounds is of particular interest [6-8]. As a continuation of our work on such compounds, we report herein the crystal structure of the title hydrazone derivative. The molecular structure of the title compound is shown in the figure. The dihedral angle between the C(1)–C(6) and C(9)–C(14) benzene rings is 5.9(3)°. The dihedral angle between the C(15)–N(3)–C(16) plane and the C(9)–C(14) benzene ring is 5.7(3)°. All bond lengths are within normal values [9], and comparable to those observed in similar compounds [10-12]. The crystal structure of the compound is stabilized by intermolecular N–H···O and O–H···O hydrogen bonds.

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.7277	0.8992	0.0860	0.074
H(2A)	4*e*	0.7920	0.7415	0.2896	0.044
H(4)	4*e*	0.8933	1.0613	0.1391	0.055
H(5)	4*e*	1.0611	1.1297	0.2618	0.057
H(6)	4*e*	1.0935	1.0094	0.3995	0.053
H(7)	4*e*	1.0418	0.8198	0.4934	0.048
H(10)	4*e*	0.7633	0.3448	0.6708	0.052
H(11)	4*e*	0.8277	0.2596	0.8165	0.055
H(13)	4*e*	1.1329	0.5337	0.8657	0.050
H(14)	4*e*	1.0659	0.6174	0.7198	0.047
H(15A)	4*e*	1.2221	0.3504	0.9861	0.085
H(15B)	4*e*	1.1592	0.3439	1.0666	0.085
H(15C)	4*e*	1.1499	0.4909	1.0090	0.085
H(16A)	4*e*	0.8818	0.2453	0.9773	0.100
H(16B)	4*e*	1.0137	0.1824	1.0371	0.100
H(16C)	4*e*	0.9571	0.1266	0.9367	0.100
H(2)	4*e*	1.009(2)	0.671(3)	0.590(1)	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}
N(1)	4*e*	0.8957(2)	0.6922(2)	0.4652(1)	0.046(1)	0.047(1)	0.0248(9)	0.0029(9)	0.0052(8)	0.0034(7)
N(2)	4*e*	0.9326(2)	0.6399(2)	0.5536(1)	0.044(1)	0.049(1)	0.0264(9)	–0.0057(9)	0.0013(8)	0.0056(8)
N(3)	4*e*	1.0323(2)	0.3305(2)	0.9452(1)	0.053(1)	0.055(1)	0.040(1)	0.0033(9)	0.0114(9)	0.0165(9)
O(1)	4*e*	0.7292(1)	0.8479(2)	0.13070(9)	0.0473(9)	0.067(1)	0.0299(8)	–0.0009(8)	0.0046(7)	0.0069(7)
O(2)	4*e*	0.7689(1)	0.4739(2)	0.52760(9)	0.047(1)	0.062(1)	0.0322(8)	–0.0137(7)	0.0027(7)	–0.0051(7)
C(1)	4*e*	0.9449(2)	0.8638(2)	0.3591(1)	0.038(1)	0.038(1)	0.032(1)	0.0067(9)	0.0091(9)	0.0037(8)
C(2)	4*e*	0.8459(2)	0.8202(2)	0.2845(1)	0.035(1)	0.040(1)	0.034(1)	0.0029(9)	0.0097(9)	0.0029(9)
C(3)	4*e*	0.8274(2)	0.8940(2)	0.2025(1)	0.034(1)	0.045(1)	0.030(1)	0.0081(9)	0.0074(9)	0.0013(9)
C(4)	4*e*	0.9070(2)	1.0109(2)	0.1942(1)	0.053(1)	0.046(1)	0.039(1)	0.006(1)	0.014(1)	0.012(1)
C(5)	4*e*	1.0064(2)	1.0523(2)	0.2676(2)	0.051(1)	0.043(1)	0.050(1)	–0.006(1)	0.013(1)	0.007(1)
C(6)	4*e*	1.0260(2)	0.9802(2)	0.3500(1)	0.042(1)	0.046(1)	0.040(1)	–0.002(1)	0.004(1)	–0.000(1)
C(7)	4*e*	0.9691(2)	0.7917(2)	0.4476(1)	0.038(1)	0.046(1)	0.033(1)	0.002(1)	0.0057(9)	0.0016(9)
C(8)	4*e*	0.8638(2)	0.5322(2)	0.5811(1)	0.038(1)	0.039(1)	0.031(1)	0.0023(9)	0.0095(9)	–0.0041(8)
C(9)	4*e*	0.9077(2)	0.4894(2)	0.6774(1)	0.034(1)	0.036(1)	0.028(1)	–0.0001(8)	0.0075(9)	–0.0019(8)
C(10)	4*e*	0.8387(2)	0.3826(2)	0.7100(1)	0.040(1)	0.049(1)	0.038(1)	–0.012(1)	0.007(1)	–0.005(1)
C(11)	4*e*	0.8773(2)	0.3308(2)	0.7974(1)	0.050(1)	0.047(1)	0.043(1)	–0.011(1)	0.017(1)	0.004(1)
C(12)	4*e*	0.9906(2)	0.3836(2)	0.8587(1)	0.039(1)	0.040(1)	0.033(1)	0.0061(9)	0.0128(9)	0.0046(9)
C(13)	4*e*	1.0585(2)	0.4937(2)	0.8265(1)	0.038(1)	0.051(1)	0.032(1)	–0.007(1)	0.0042(9)	0.0022(9)
C(14)	4*e*	1.0180(2)	0.5442(2)	0.7388(1)	0.042(1)	0.042(1)	0.033(1)	–0.0085(9)	0.010(1)	0.0039(9)
C(15)	4*e*	1.1508(2)	0.3833(3)	1.0069(1)	0.063(2)	0.062(2)	0.038(1)	0.012(1)	0.004(1)	0.008(1)
C(16)	4*e*	0.9657(3)	0.2112(3)	0.9767(2)	0.085(2)	0.062(2)	0.060(2)	0.003(1)	0.031(2)	0.025(1)

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