Crystal structure of (*E*)-*N*′-(4-(diethylamino)-2-hydroxybenzylidene)nicotinohydrazide — water (1:1), $C_{17}H_{20}N_4O_2 \cdot H_2O$

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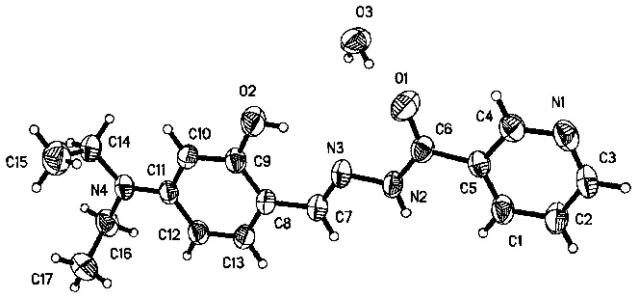

Abstract

$C_{17}H_{22}N_4O_3$, triclinic, $P\bar{1}$ (no. 2), a = 6.6858(3) Å, b = 7.9713(3) Å, c = 16.7515(6) Å, α = 86.044(2)°, β = 78.555(2)°, γ = 74.218(2)°, V = 841.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.045, $wR_{ref}(F^2)$ = 0.139, T = 296 K.

Source of material

4-(Diethylamino)-2-hydroxybenzaldehyde (1.0 mmol, 193 mg) and nicotinohydrazide (1.0 mmol, 137 mg) were mixed in 50 ml 95% ethanol. The mixture was stirred at ambient temperature for 2 h and filtered. Pale yellow block-like crystals of the title compound were formed by slowl evaporation of the filtrate for a week.

Experimental details

Hydrogen atoms potentially involved in hydrogen-bonding interactions were located from difference Fourier maps, and their positional and isotropic displacement parameters were refined. Other H atoms were included in the refinement at calculated positions [d(C—H$_{aromatic}$) = 0.93 Å] and treated as riding with U_{iso}(H) = 1.5U_{eq}(C).

Discussion

Acylhydrazones possessing an azomethine –NHN=CH– proton constitute an important class of compounds for new drug development. Hydrazone derivatives have been found to possess potential tuberculostatic activity [1,2]. Acylhydrazone compounds are obtained by the reaction of aromatic and heterocyclic hydrazides with aldehydes or ketones and have revealed very versatile behaviour in metal coordination [3].

The asymmetric unit of the title crystal structure comprises the hydrazide molecule and a water molecule of crystallization. The molecule is in a *trans*-configuration with respect to the C=N double bond. The bond distances and angles agree with the corresponding bond distances and angles reported in closely related compounds, i.e., *N*′-(2-methoxybenzylidene)nicotinohydrazide and 4-nitro-*N*′-[(*E*)-3-pyridylmethylidene]benzohydrazide [4,5].

The dihedral angle between the pyridine ring and the benzene ring is 1.80(6)°, indicated that these two rings are almost parallel. The crystal packing is consolidated by intermolecular O–H···N and N–H···O hydrogen bonds. The 3D supramolecular architecture of the title structure is further supported by the π–π stacking.

Table 1. Data collection and handling.

Crystal:	pale yellow block, size 0.18 × 0.21 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.91 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, Gemini, φ/ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13684, 3669
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3004
$N(param)_{refined}$:	225
Programs:	SHELXS-97, SHELXL-97, SHELXTL [6]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	2*i*	0.6977	0.1858	0.1531	0.050
H(10A)	2*i*	0.2408	–0.4119	0.3572	0.051
H(7A)	2*i*	0.7922	–0.0796	0.2018	0.051
H(13A)	2*i*	0.9174	–0.3755	0.2481	0.054
H(12A)	2*i*	0.8695	–0.6311	0.3065	0.054
H(4A)	2*i*	0.1980	0.6446	0.1278	0.059
H(1B)	2*i*	0.8080	0.3868	0.0920	0.058
H(16A)	2*i*	0.7968	–0.8666	0.3237	0.064
H(16B)	2*i*	0.6455	–0.9647	0.3790	0.064
H(3B)	2*i*	0.6161	0.8827	0.0231	0.062
H(2B)	2*i*	0.8848	0.6391	0.0321	0.064
H(14A)	2*i*	0.3062	–0.8444	0.4100	0.065
H(14B)	2*i*	0.2045	–0.6531	0.3832	0.065
H(17A)	2*i*	0.9238	–0.9640	0.4419	0.106
H(17B)	2*i*	0.7113	–0.8590	0.4947	0.106
H(17C)	2*i*	0.8618	–0.7596	0.4395	0.106
H(15A)	2*i*	0.1153	–0.6828	0.5228	0.114
H(15B)	2*i*	0.2546	–0.5504	0.5026	0.114
H(15C)	2*i*	0.3551	–0.7422	0.5294	0.114
H(2)	2*i*	0.2683	–0.0353	0.2699	0.094
H(3A)	2*i*	0.108(4)	0.175(3)	0.145(1)	0.098(7)
H(3C)	2*i*	0.131(4)	0.017(3)	0.110(1)	0.095(6)

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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(3)	2*i*	0.5028(2)	0.0517(1)	0.20958(6)	0.0532(6)	0.0324(5)	0.0447(6)	–0.0169(4)	–0.0041(4)	0.0048(4)
N(2)	2*i*	0.5657(2)	0.1916(1)	0.16949(6)	0.0457(6)	0.0317(5)	0.0478(6)	–0.0143(4)	–0.0044(4)	0.0081(4)
C(11)	2*i*	0.5478(2)	–0.5535(1)	0.33761(7)	0.0476(6)	0.0359(6)	0.0348(6)	–0.0146(5)	–0.0104(5)	0.0078(4)
C(8)	2*i*	0.6116(2)	–0.2366(1)	0.26243(7)	0.0458(6)	0.0341(6)	0.0373(6)	–0.0139(5)	–0.0043(5)	0.0042(4)
C(10)	2*i*	0.3772(2)	–0.4062(2)	0.33386(7)	0.0389(6)	0.0422(6)	0.0450(7)	–0.0127(5)	–0.0043(5)	0.0095(5)
C(9)	2*i*	0.4078(2)	–0.2530(1)	0.29630(7)	0.0419(6)	0.0340(5)	0.0407(6)	–0.0066(4)	–0.0038(5)	0.0045(4)
C(5)	2*i*	0.4918(2)	0.4880(1)	0.11675(7)	0.0445(6)	0.0302(5)	0.0356(6)	–0.0072(4)	–0.0060(5)	0.0002(4)
C(7)	2*i*	0.6527(2)	–0.0825(2)	0.22102(7)	0.0489(7)	0.0367(6)	0.0418(6)	–0.0161(5)	–0.0045(5)	0.0048(5)
C(6)	2*i*	0.4162(2)	0.3372(1)	0.15654(7)	0.0429(6)	0.0328(6)	0.0457(6)	–0.0107(5)	–0.0021(5)	–0.0013(5)
C(13)	2*i*	0.7804(2)	–0.3832(2)	0.26885(8)	0.0388(6)	0.0449(6)	0.0490(7)	–0.0124(5)	–0.0040(5)	0.0091(5)
O(1)	2*i*	0.2264(2)	0.3479(1)	0.17572(7)	0.0438(5)	0.0428(5)	0.0987(8)	–0.0116(4)	0.0028(5)	0.0017(5)
N(4)	2*i*	0.5173(2)	–0.7091(1)	0.37063(7)	0.0545(6)	0.0378(5)	0.0505(6)	–0.0171(5)	–0.0141(5)	0.0147(4)
N(1)	2*i*	0.3805(2)	0.7889(1)	0.07499(8)	0.0573(7)	0.0349(5)	0.0662(7)	–0.0026(5)	–0.0142(6)	0.0092(5)
C(12)	2*i*	0.7526(2)	–0.5369(2)	0.30433(8)	0.0426(6)	0.0389(6)	0.0484(7)	–0.0065(5)	–0.0075(5)	0.0104(5)
C(4)	2*i*	0.3390(2)	0.6429(2)	0.10854(8)	0.0443(7)	0.0379(6)	0.0598(8)	–0.0051(5)	–0.0097(6)	0.0045(5)
C(1)	2*i*	0.6998(2)	0.4871(2)	0.08774(8)	0.0459(7)	0.0312(6)	0.0578(8)	–0.0029(5)	–0.0007(6)	0.0083(5)
C(16)	2*i*	0.6958(2)	–0.8605(2)	0.37448(9)	0.0671(9)	0.0358(6)	0.0592(8)	–0.0138(6)	–0.0186(7)	0.0106(5)
C(3)	2*i*	0.5831(2)	0.7826(2)	0.04723(8)	0.0653(8)	0.0330(6)	0.0520(7)	–0.0110(5)	–0.0075(6)	0.0110(5)
C(2)	2*i*	0.7455(2)	0.6371(2)	0.05229(9)	0.0505(7)	0.0397(6)	0.0615(8)	–0.0104(5)	0.0032(6)	0.0097(6)
C(14)	2*i*	0.3106(2)	–0.7242(2)	0.41221(9)	0.0605(8)	0.0470(7)	0.0614(8)	–0.0271(6)	–0.0139(6)	0.0189(6)
C(17)	2*i*	0.8084(3)	–0.8608(2)	0.4439(1)	0.071(1)	0.072(1)	0.072(1)	–0.0171(8)	–0.0301(8)	0.0203(8)
C(15)	2*i*	0.2538(3)	–0.6700(2)	0.4996(1)	0.072(1)	0.080(1)	0.066(1)	–0.0159(9)	–0.0011(8)	0.0079(8)
O(2)	2*i*	0.2330(2)	–0.1195(1)	0.29316(7)	0.0454(5)	0.0433(5)	0.0827(7)	0.0004(4)	0.0030(5)	0.0196(5)
O(3)	2*i*	0.0348(2)	0.1090(2)	0.12856(9)	0.0433(6)	0.0436(5)	0.121(1)	–0.0083(4)	–0.0166(6)	0.0091(6)

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