Crystal structure of *N′*-(3-ethoxy-4-hydroxybenzylidene)-isonicotinohydrazide — methanol (1:1), $C_{15}H_{15}N_3O_3 \cdot CH_3OH$

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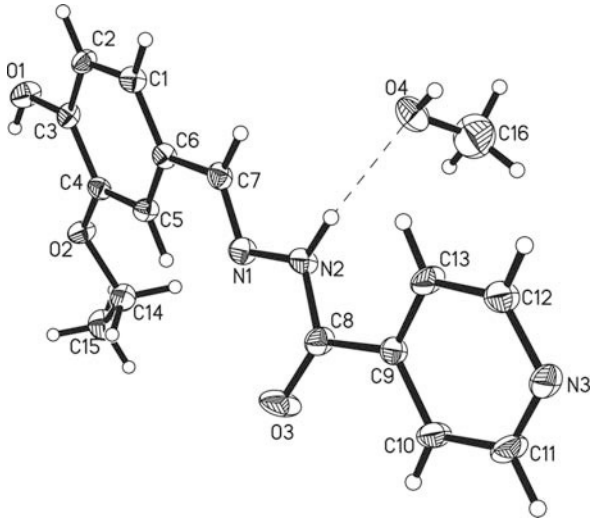

Abstract

$C_{16}H_{19}N_3O_4$, monoclinic, $P2_1$ (no. 4), a = 11.2785(8) Å, b = 6.4429(3) Å, c = 11.8901(8) Å, β = 109.068(8)°, V = 816.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.064, T = 293 K.

Source of material

Pyridine-4-carboxylic acid hydrazide (1 mmol, 0.137 g) was dissolved in anhydrous ethanol (10 ml), the mixture was stirred for several minutes at room temperature, 3-ethoxy-4-hydroxybenzaldehyde (1 mmol 0.166 g) in ethanol (5 ml) was added dropwise and the mixture was stirred at refluxing temperature for 4 h. The product was separated and recrystallised in methanol, colorless single crystals of the title compound were obtained after 1 d.

Experimental details

H1B bound to O1 was found in difference Fourier map and refined freely. The other hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.96 Å and U_{iso}(H) = 1.5 U_{eq}(C) for methyl group; d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C) for methylene and aromatic species; d(N—H) = 0.86 Å and U_{iso}(H) = 1.2 U_{eq}(N); d(O—H) = 0.82 Å U_{iso}(H) = 1.5 U_{eq}(O), respectively. The Flack parameter value x = 0.0(1) confirms the determined absolute configuration of the structure.

Discussion

The chemistry of Schiff bases has attracted a great deal of interest in recent years. These compounds play an important role in the development of various proteins and enzymes [1,2].

The title crystal structure consists of a Schiff base and a methanol molecules. The Schiff base molecule adopts an *E* geometry with an C6–C7–N1–N2 torsion angle of –177.0(2)°. The molecule is not planar, the dihedral angle between the pyridine ring and the benzene ring is 10.6(1)°, while the pyridine ring is coplanar with the C6/C7/N1/N2 plane with a dihedral angle of 5.3(3)°. Bond lengths and bond angles agree with the isonicotinohydrazide derevatives [3]. An intramolecular N–H···O hydrogen bond is observed in the crystal structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.19 × 0.21 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.94 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3209, 2617
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1481
$N(param)_{refined}$:	214
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6]

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

Atom	Site	x	y	z	U_{iso}
H(7A)	2*a*	0.4823	0.3804	0.3586	0.037
H(2A)	2*a*	0.7657	0.2342	0.7247	0.038
H(2B)	2*a*	0.3602	0.3962	0.1753	0.041
H(13A)	2*a*	0.2057	0.5714	0.0800	0.049
H(5A)	2*a*	0.6281	–0.1145	0.3654	0.034
H(14A)	2*a*	0.8119	–0.2974	0.3627	0.048
H(14B)	2*a*	0.7105	–0.4501	0.3804	0.048
H(1C)	2*a*	0.6112	0.3783	0.5661	0.037
H(11A)	2*a*	0.0052	0.2964	–0.2847	0.054
H(10A)	2*a*	0.1399	0.0886	–0.1420	0.047
H(12A)	2*a*	0.0619	0.7554	–0.0652	0.052
H(15A)	2*a*	0.8771	–0.6414	0.3574	0.078
H(15B)	2*a*	0.8705	–0.6779	0.4856	0.078
H(15C)	2*a*	0.9710	–0.5263	0.4666	0.078
H(4)	2*a*	0.4110	0.7768	0.1730	0.075
H(16A)	2*a*	0.5814	0.8039	0.1254	0.127
H(16B)	2*a*	0.4696	0.7001	0.0261	0.127
H(16C)	2*a*	0.5745	0.5616	0.1125	0.127
H(1B)	2*a*	0.902(3)	–0.213(4)	0.727(2)	0.03(1)

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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}
C(7)	2*a*	0.5042(2)	0.2431(4)	0.3514(2)	0.031(2)	0.027(2)	0.032(2)	0.004(2)	0.006(2)	0.006(2)
N(1)	2*a*	0.4468(2)	0.1439(3)	0.2557(2)	0.032(1)	0.028(1)	0.032(1)	0.006(1)	–0.001(1)	0.005(1)
C(6)	2*a*	0.6032(2)	0.1471(4)	0.4495(2)	0.019(2)	0.029(2)	0.028(2)	0.003(2)	–0.001(1)	0.006(1)
C(8)	2*a*	0.2759(2)	0.1672(5)	0.0783(2)	0.035(2)	0.034(2)	0.029(2)	0.007(2)	–0.001(2)	0.002(2)
C(2)	2*a*	0.7386(2)	0.1645(4)	0.6522(2)	0.035(2)	0.034(2)	0.023(2)	0.004(2)	0.005(1)	–0.001(2)
N(2)	2*a*	0.3598(2)	0.2631(3)	0.1696(2)	0.036(2)	0.018(1)	0.034(2)	0.010(1)	–0.007(1)	0.006(1)
C(4)	2*a*	0.7503(3)	–0.1270(4)	0.5317(2)	0.030(2)	0.020(2)	0.033(2)	0.004(2)	0.009(2)	0.002(1)
O(1)	2*a*	0.8844(2)	–0.0981(3)	0.7360(2)	0.048(1)	0.039(2)	0.030(1)	0.018(1)	–0.004(1)	0.002(1)
C(13)	2*a*	0.1648(3)	0.5093(4)	0.0070(2)	0.045(2)	0.035(2)	0.026(2)	0.012(2)	–0.011(2)	–0.001(1)
C(5)	2*a*	0.6563(2)	–0.0436(4)	0.4374(2)	0.022(2)	0.030(2)	0.028(2)	0.002(2)	–0.000(1)	0.000(2)
N(3)	2*a*	0.0211(2)	0.5474(4)	–0.1904(2)	0.040(2)	0.040(2)	0.031(2)	0.008(1)	–0.007(1)	0.006(1)
C(9)	2*a*	0.1888(3)	0.3058(4)	–0.0128(2)	0.029(2)	0.025(2)	0.029(2)	0.004(1)	0.002(2)	0.004(1)
C(14)	2*a*	0.7958(3)	–0.4002(4)	0.4156(2)	0.044(2)	0.038(2)	0.039(2)	0.007(2)	0.016(2)	0.002(1)
O(2)	2*a*	0.8119(2)	–0.3097(3)	0.5296(2)	0.041(1)	0.030(1)	0.030(1)	0.012(1)	0.0019(9)	–0.0027(9)
C(1)	2*a*	0.6455(2)	0.2505(4)	0.5575(2)	0.026(2)	0.028(2)	0.034(2)	0.008(1)	0.005(2)	0.003(1)
O(3)	2*a*	0.2696(2)	–0.0212(3)	0.0684(2)	0.074(2)	0.020(1)	0.062(2)	0.003(1)	–0.021(1)	0.000(1)
C(3)	2*a*	0.7914(2)	–0.0224(4)	0.6408(2)	0.024(2)	0.031(2)	0.025(2)	0.009(2)	0.003(1)	0.006(2)
C(11)	2*a*	0.0452(3)	0.3523(5)	–0.2097(3)	0.043(2)	0.046(2)	0.029(2)	0.001(2)	–0.011(2)	–0.006(2)
C(10)	2*a*	0.1270(3)	0.2256(4)	–0.1242(2)	0.042(2)	0.031(2)	0.034(2)	0.000(2)	–0.002(2)	–0.007(2)
C(12)	2*a*	0.0796(3)	0.6202(4)	–0.0820(3)	0.049(2)	0.029(2)	0.040(2)	0.008(2)	–0.001(2)	0.001(2)
C(15)	2*a*	0.8868(3)	–0.5775(4)	0.4329(3)	0.060(2)	0.040(2)	0.063(2)	0.015(2)	0.029(2)	0.001(2)
O(4)	2*a*	0.4582(2)	0.6766(3)	0.1838(2)	0.054(2)	0.032(1)	0.063(2)	0.009(1)	0.017(1)	0.008(1)
C(16)	2*a*	0.5261(4)	0.6863(7)	0.1060(3)	0.098(3)	0.084(3)	0.084(3)	0.006(3)	0.048(3)	–0.001(2)

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