Crystal structure of (*Z*)-*N*′-[amino(pyridin-2-yl)-methylene]-2-hydroxybenzohydrazide, $C_{13}H_{12}N_4O_2$

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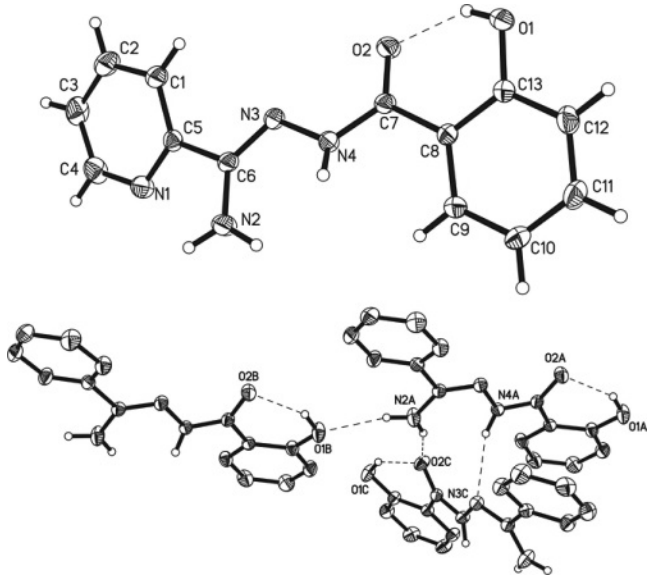
Received June 27, 2012, accepted September 26, 2012, available online November 09, 2012, CCDC no. 1267/3873



Abstract

$C_{13}H_{12}N_4O_2$, monoclinic, *Cc* (no. 9), a = 8.749(4) Å, b = 17.707(8) Å, c = 8.005(4) Å, β = 102.751(5)°, V = 1209.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.0270, $wR_{ref}(F^2)$ = 0.0701, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow prisms, size 0.15×0.18×0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.99 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4253, 1983
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1898
$N(param)_{refined}$:	189
Programs:	SHELX [5]

Source of material

Equimolar amounts of methylester-2-pyridinecarboximidic acid and 2-hydroxybenzohydrazide were dissolved in ethanol and refluxed for 8h. Yellow crystals were precipitated, filtered, washed with ethanol and dried in air (yield 86%). Yellow prism-shaped crystals suitable for X-ray analysis of this compound were obtained from acetonitrile by slow evaporation for a few days.

Experimental details

The H atoms were calculated geometrically and refined as riding, with C–H = 0.93 Å (except for H1B, H2B, H2C, H4B) and with U_{iso}(H) = 1.2U_{eq} (parent atom).

Discussion

N′-[amino(pyridin-2-yl)methylene]-2-hydroxybenzohydrazide is an important intermediate medicament [1] and ligand. Its copper(II) and zinc(II) complexes were prepared and structurally determined by single crystal X-ray analysis [2-4], however, to our knowledge, there are no structure reports about the title compound. The X-ray structure analysis reveals that the title molecule shows the *Z*-configuration for the *N*′-[amino(pyridin-2-yl)methylene] and 2-hydroxybenzohydrazide moieties. The dihedral angle between the pyridine ring and the phenol ring is 8.39(9)°. All of the aromatic C–C and C–N bond lengths are in the expected ranges of 1.334(2)-1.399(2) Å. The bond lengths in the bridging groups C5–C6, N3–C6, N3–N4, N4–C7, C7–C8, N2–C6, C7–O2 are 1.498(2), 1.298(2), 1.399(2), 1.337(2), 1.488(2), 1.337(2), and 1.225(2)Å respectively. The torsion angles in the bridges C1–C5–C6–N3, C5–C6–N3–N4, C6–N3–N4–C7, C8–C7–N3–N4, and N4–C7–C8–C13 are -24.9(3)°, 179.86(13)°, 170.84(13)°, 174.72(13)°, 121.3(2)°, and 159.64(14)°, respectively. There is an intramolecular hydrogen bond O1–H1B···O2 (length 2.645 Å, angle 144.24°), which is different from the one in the copper(II) complex [3]. In addition, there are three intremolecular hydrogen bonds, N2–H2B···O2 (length 2.960 Å, angle 157.96°), N4–H4B···N3 (length 3.101 Å, angle 158.59°), and N2–H2C···O1 (length 3.052 Å, angle 166.12°). The former two hydrogen bonds link two adjacent molecules to each other forming dimers, and the last one links another dimer into chains along the *b*-axis (Fig. bottom).

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*a*	–0.2910	0.4841	0.0630	0.050
H(2A)	4*a*	–0.4521	0.4234	–0.1693	0.065
H(3A)	4*a*	–0.4771	0.2936	–0.1658	0.063
H(4A)	4*a*	–0.3402	0.2274	0.0640	0.059
H(9A)	4*a*	0.2514	0.5020	0.7551	0.045
H(10A)	4*a*	0.4235	0.5408	1.0007	0.055
H(11A)	4*a*	0.5061	0.6646	1.0302	0.058
H(12A)	4*a*	0.4159	0.7504	0.8149	0.052
H(2B)	4*a*	–0.044(2)	0.395(1)	0.594(2)	0.043(5)
H(2C)	4*a*	–0.153(2)	0.3370(7)	0.485(3)	0.048(5)
H(4B)	4*a*	0.018(2)	0.5036(9)	0.582(2)	0.037(5)
H(1B)	4*a*	0.192(3)	0.708(1)	0.434(2)	0.088(9)

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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}
O(1)	4*a*	0.2359(2)	0.73032(7)	0.5285(2)	0.0595(8)	0.0308(6)	0.0433(7)	–0.0081(5)	–0.0070(6)	0.0071(5)
O(2)	4*a*	0.1108(2)	0.61493(7)	0.3365(1)	0.0563(8)	0.0383(7)	0.0258(6)	–0.0104(5)	–0.0019(5)	0.0051(5)
C(1)	4*a*	–0.3022(2)	0.4319(1)	0.0612(2)	0.0427(9)	0.0399(9)	0.0360(9)	–0.0041(8)	–0.0028(7)	0.0016(8)
C(2)	4*a*	–0.3980(3)	0.3956(1)	–0.0763(2)	0.056(1)	0.062(1)	0.036(1)	–0.0047(9)	–0.0109(8)	0.0016(9)
C(3)	4*a*	–0.4127(2)	0.3188(1)	–0.0748(2)	0.056(1)	0.062(1)	0.035(1)	–0.018(1)	0.0004(8)	–0.0150(9)
C(4)	4*a*	–0.3305(2)	0.2797(1)	0.0637(3)	0.063(1)	0.043(1)	0.042(1)	–0.0161(9)	0.0079(8)	–0.0121(9)
C(5)	4*a*	–0.2240(2)	0.38774(9)	0.1954(2)	0.0324(8)	0.0356(9)	0.0274(8)	–0.0062(7)	0.0045(6)	–0.0023(7)
C(6)	4*a*	–0.1211(2)	0.42206(9)	0.3514(2)	0.0343(8)	0.0316(8)	0.0240(8)	–0.0014(6)	0.0006(7)	–0.0005(6)
C(7)	4*a*	0.1175(2)	0.57948(9)	0.4722(2)	0.0343(8)	0.0277(8)	0.0254(9)	0.0000(6)	0.0022(7)	–0.0006(6)
C(8)	4*a*	0.2265(2)	0.60315(9)	0.6338(2)	0.0299(8)	0.0311(9)	0.0252(8)	–0.0019(6)	0.0029(6)	–0.0003(6)
C(9)	4*a*	0.2837(2)	0.5521(1)	0.7663(2)	0.0417(9)	0.0356(9)	0.0324(9)	–0.0062(7)	0.0016(7)	0.0055(7)
C(10)	4*a*	0.3870(2)	0.5753(1)	0.9133(2)	0.047(1)	0.054(1)	0.031(1)	–0.0059(8)	–0.0042(8)	0.0107(8)
C(11)	4*a*	0.4364(2)	0.6492(1)	0.9308(2)	0.048(1)	0.059(1)	0.0321(9)	–0.0128(9)	–0.0045(7)	–0.0025(8)
C(12)	4*a*	0.3831(2)	0.7004(1)	0.8019(2)	0.048(1)	0.041(1)	0.038(1)	–0.0128(8)	0.0026(8)	–0.0063(7)
C(13)	4*a*	0.2808(2)	0.67764(9)	0.6529(2)	0.0347(8)	0.0315(8)	0.0325(9)	–0.0031(7)	0.0028(7)	0.0014(7)
N(2)	4*a*	–0.1035(2)	0.38074(9)	0.4942(2)	0.066(1)	0.0377(8)	0.0303(8)	–0.0173(7)	–0.0090(7)	0.0044(6)
N(3)	4*a*	–0.0620(1)	0.48787(7)	0.3323(2)	0.0381(7)	0.0329(7)	0.0232(7)	–0.0055(6)	–0.0014(6)	–0.0021(5)
N(4)	4*a*	0.0334(1)	0.51723(7)	0.4817(2)	0.0380(7)	0.0318(7)	0.0200(7)	–0.0077(5)	0.0010(5)	–0.0013(5)
N(1)	4*a*	–0.2372(2)	0.31272(8)	0.1984(2)	0.0532(8)	0.0348(7)	0.0330(8)	–0.0077(7)	0.0017(6)	–0.0039(6)

Acknowledgments. We are grateful to a Project Funded by the Priority Academic Program Development of Jiangsu Higher Education Institutions, the National Natural Science Foundation of China (20802061), and Natural Science Foundation of Jiangsu Education Deparatment (02KJB 150007) for financial support.

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