Crystal structure of 2′-(2-hydroxybenzylidene)-2-hydroxybenzoylhydrazide, $C_{14}H_{12}N_2O_3$

F.-Y. Pan*,[I] and J.-G. Yang[II]

[I] Taizhou University, Department of Chemistry, Taizhou, Zhejiang, 317000 P. R. China
[II] Taizhou University, Office of Assets Administration, Taizhou, Zhejiang, 317000 P. R. China

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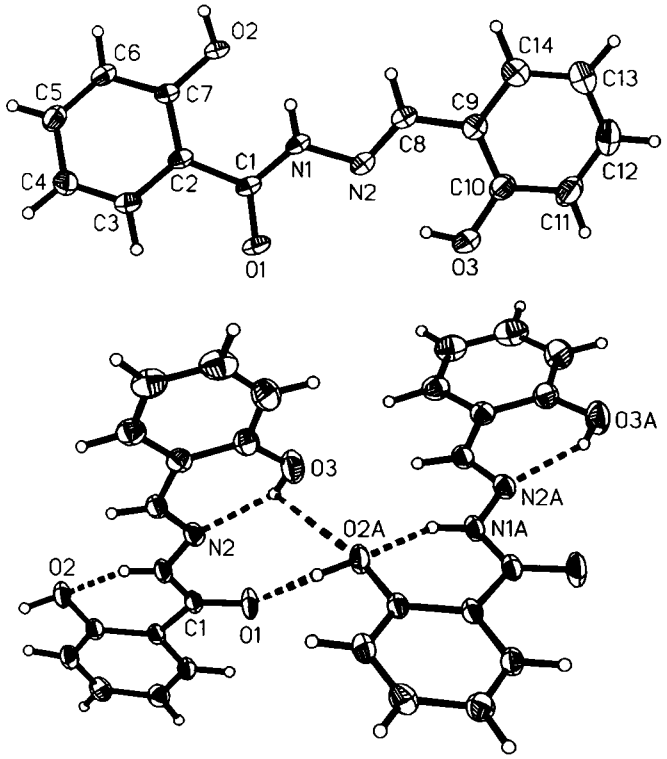

Abstract

$C_{14}H_{12}N_2O_3$, monoclinic, $P12_1/c1$ (no. 14), $a = 18.838(3)$ Å, $b = 5.2381(7)$ Å, $c = 12.709(2)$ Å, $\beta = 107.021(3)°$, $V = 1199.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.040$, $wR_{ref}(F^2) = 0.115$, $T = 293$ K.

Source of material

2-Hydroxybenzoylhydrazine (20.0 mmol, 3.04 g) was dissolved in anhydrous ethanol (50 ml) and 2-hydroxybenzaldehyde (20.0 mmol, 2.44 g) was added. The mixture was refluxed for 1 h and the precipitate formed was collected by filtration, and washed with ethanol. The product was recrystallized from ethanol and dried under reduced pressure to give the title compound. The compound (2.0 mmol, 0.51 g) was dissolved in DMF (25 ml) and kept at room temperature for 45 d to obtain yellow block-shaped crystals, which were collected and washed with distilled water.

Discussion

Some benzoylhydrazone compounds possess bacteriostatic activity. This type of compound has wide application in tuberculosis treatment and also exhibits fungicidal activity [1]. Some hydrazonecarbonyl compounds also show bioactivity [2]. In order to explore more effective antibacterial medicines, we have synthesized the title compound, 2′-(2-hydroxybenzylidene)-2-hydroxybenzoylhydrazide. In the literature, there are structures of two coordination compounds with this compound as ligand [3,4]. The title molecule (figure, top) is non-planar; the dihedral angle between the two aromatic rings is 16.85(7)°. As a result of the conjugation, the C=O distance (1.232(2) Å) is longer than the normal value of 1.20 Å, and the C1—N1 bond distance (1.330(2) Å) is longer than the C=N double-bond distance and shorter than the C—N single-bond distance [5]. The values of C7–C2–C1, C3–C2–C1, C8–C9–C14 and C8–C9–C10 angles close to 120° and C1—C2 distance (1.492(2) Å), C8—C9 distance (1.450(2) Å) confirm sp^2 hybridization of carbon atoms [6]. Intramolecular N1–H1···O2 and O3–H7···N2 hydrogen bonds which form a six-membered ring is observed (figure, bottom). In addition, intermolecular O2–H2···O1[i] and O3–H7···O2[ii] (symmetry codes (i) $x,½–y,z–½$, (ii) $x,½–y,z+½$) hydrogen bonds occur, linking the hydroxyl H with the keto group to a hydroxyl O of an adjacent molecule which forms a seven-membered ring (figure, bottom).

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.18 × 0.37 × 0.52 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.02 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6550, 2602
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2018
$N(param)_{refined}$:	221
Programs:	SHELXS-97 [7], SHELXL-97 [8]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.2738(8)	0.115(3)	0.275(1)	0.051(4)
H(2)	4e	0.302(1)	0.294(4)	0.099(2)	0.080(6)
H(3)	4e	0.4299(8)	0.631(3)	0.483(1)	0.051(4)
H(4)	4e	0.4906(9)	0.924(3)	0.397(1)	0.056(4)
H(5)	4e	0.4529(8)	0.960(3)	0.209(1)	0.059(4)
H(6)	4e	0.3621(9)	0.686(3)	0.099(2)	0.065(5)
H(7)	4e	0.210(1)	−0.142(4)	0.517(2)	0.093(7)
H(8)	4e	0.2020(7)	−0.209(2)	0.248(1)	0.039(4)
H(11)	4e	0.0939(9)	−0.580(4)	0.553(2)	0.074(5)
H(12)	4e	0.029(1)	−0.873(4)	0.417(2)	0.086(6)
H(13)	4e	0.040(1)	−0.861(4)	0.244(2)	0.082(6)
H(14)	4e	0.1230(8)	−0.556(3)	0.195(1)	0.054(4)

* Correspondence author (e-mail: panfy@tzc.edu.cn)

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*e*	0.34147(6)	0.3170(2)	0.50184(8)	0.0823(8)	0.0673(7)	0.0248(5)	–0.0143(5)	0.0199(5)	–0.0007(5)
O(2)	4*e*	0.29254(5)	0.3315(2)	0.16065(8)	0.0590(6)	0.0563(6)	0.0249(5)	–0.0104(4)	0.0166(4)	–0.0042(4)
O(3)	4*e*	0.18258(7)	–0.2424(3)	0.54126(9)	0.0870(9)	0.0878(9)	0.0410(6)	–0.0283(7)	0.0255(6)	0.0002(6)
N(1)	4*e*	0.28195(6)	0.1213(2)	0.34250(9)	0.0481(6)	0.0484(6)	0.0266(6)	–0.0018(5)	0.0176(5)	0.0031(5)
N(2)	4*e*	0.24292(6)	–0.0435(2)	0.38960(8)	0.0449(6)	0.0437(6)	0.0343(6)	0.0031(5)	0.0171(5)	0.0067(5)
C(1)	4*e*	0.32792(7)	0.2961(2)	0.4014(1)	0.0443(7)	0.0425(7)	0.0260(6)	0.0081(6)	0.0151(5)	0.0031(5)
C(2)	4*e*	0.36345(7)	0.4736(2)	0.34003(9)	0.0398(7)	0.0367(6)	0.0278(6)	0.0078(5)	0.0143(5)	0.0010(5)
C(3)	4*e*	0.41715(7)	0.6393(3)	0.4022(1)	0.0463(8)	0.0483(8)	0.0299(7)	0.0048(6)	0.0123(6)	–0.0033(6)
C(4)	4*e*	0.45121(8)	0.8170(3)	0.3541(1)	0.0492(8)	0.0482(8)	0.0470(8)	–0.0059(6)	0.0175(7)	–0.0082(6)
C(5)	4*e*	0.43043(9)	0.8354(3)	0.2405(1)	0.0587(9)	0.0459(8)	0.0493(8)	–0.0044(7)	0.0259(7)	0.0041(7)
C(6)	4*e*	0.37805(8)	0.6749(3)	0.1770(1)	0.0555(9)	0.0512(8)	0.0319(7)	0.0014(6)	0.0185(6)	0.0057(6)
C(7)	4*e*	0.34485(7)	0.4896(2)	0.2249(1)	0.0410(7)	0.0395(7)	0.0281(6)	0.0036(5)	0.0134(5)	–0.0015(5)
C(8)	4*e*	0.20289(7)	–0.2036(3)	0.3224(1)	0.0432(7)	0.0472(8)	0.0340(7)	0.0055(6)	0.0151(6)	0.0035(6)
C(9)	4*e*	0.15531(7)	–0.3883(2)	0.3535(1)	0.0402(7)	0.0412(7)	0.0447(8)	0.0069(6)	0.0128(6)	0.0063(6)
C(10)	4*e*	0.14667(8)	–0.4008(3)	0.4586(1)	0.0528(8)	0.0551(8)	0.0447(8)	–0.0016(7)	0.0154(7)	0.0080(7)
C(11)	4*e*	0.0990(1)	–0.5821(4)	0.4807(2)	0.073(1)	0.077(1)	0.059(1)	–0.010(1)	0.0250(9)	0.0190(9)
C(12)	4*e*	0.0612(1)	–0.7476(3)	0.4002(2)	0.059(1)	0.057(1)	0.091(2)	–0.0098(8)	0.021(1)	0.017(1)
C(13)	4*e*	0.06894(9)	–0.7379(3)	0.2966(2)	0.056(1)	0.0505(9)	0.080(1)	–0.0042(8)	0.0147(9)	–0.0039(8)
C(14)	4*e*	0.11568(8)	–0.5592(3)	0.2734(1)	0.0488(8)	0.0503(8)	0.058(1)	0.0033(7)	0.0134(7)	–0.0023(7)

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References

1. Edwards, E. I.; Epton, R.; Marr, G.: Organometallic derivatives of penicillins and cephalosporins a new class of semi-synthetic antibiotics. J. Organomet. Chem. **85** (1975) C23-C25.
2. Fan, Z. J.; Zhong, B.; Wang, S. H.; Li, Z. M.: Synthesis and Fungicidal Activity of Derivatives of 1,3-Dimethyl-5-methylthio-4-phenylhydrazonocarboxyl Pyrazole. Chinese J. Appl. Chem. **4** (2003) 365-367.
3. Banße, W.; Fliegner, J.; Sawusch, S.; Schilde, U.; Uhlemann, E.: Dioxomolybdän(VI)-Komplexe mit dreizähnigen diaziden Liganden. Z. Naturforsch. **53b** (1998) 689-693.
4. Rao, S. N.; Munshi, K. N.; Rao, N. N.; Bhadbhade, M. M.; Suresh, E.: Synthesis, spectral and X-ray structural characterization of [*cis*-MoO_2(*L*)(solv)] (*L* = salicylidene salicyloyl hydrazine) and its use as catalytic oxidant. Poly hedron **18** (1999) 2491-2497.
5. John, A. D.: Lang's Handbook of Chemistry. McGraw-Hill, New York 1998.
6. Liu, S. L.; Dai, J. F.; Chen, Y.; Cao, G. B; Liu, H. W: Synthesis and Crystal Structure of 3-(2-Hydroxy-phenyl)-5-phenyl-6-ethoxycarbonyl-cyclohex-2-enone. Chin. J. Org. Chem. **12** (2004) 1583-1586.
7. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct methods for larger structures. Acta Crystallogr. A**46** (1990) 467-473.
8. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.