Crystal structure of 2'-(4-dimethylaminobenzylidene)-2-hydroxybenzoylhydrazide, $(\text{CH}_3)_2\text{N}(\text{C}_6\text{H}_4)(\text{CH})\text{N}(\text{NH})(\text{CO})(\text{C}_6\text{H}_4\text{OH})$

J.-G. Yang^I and F.-Y. Pan^{*,II}^I Taizhou University, Office of Assets Administration, Taizhou, Zhejiang, 317000 P. R. China^{II} Taizhou University, Department of Chemistry, Taizhou, Zhejiang, 317000 P. R. China

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Abstract

$\text{C}_{16}\text{H}_{17}\text{N}_3\text{O}_2$, orthorhombic, $Pna2_1$ (no. 33), $a = 20.196(4)$ Å, $b = 6.215(1)$ Å, $c = 11.243(2)$ Å, $V = 1411.3$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.048$, $wR_{\text{ref}}(F^2) = 0.113$, $T = 293$ K.

Source of material

2-Hydroxybenzoylhydrazine (0.02 mol, 3.04 g) was dissolved in anhydrous ethanol (50 ml) and 4-dimethylaminobenzaldehyde (0.02 mol, 2.98 g) was added to it. The mixture was refluxed for 2 h, 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, 0.56 g) was dissolved in DMF (30 ml) and kept at room temperature for 30 d to obtain yellow block crystals, which were collected and washed with distilled water.

Experimental details

H and H2 atoms were located from difference maps, and their parameters were refined. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms.

Discussion

Some benzoylhydrazone compounds possess bacteriostatic activity. This type of compound has wide application in tuberculo-

sis treatment and also exhibits fungicidal activity [1]. Some hydrazonecarbonyl compounds also show bioactivity [2]. The title compound was synthesized in the course of our studies [3,4] to explore more effective antibacterial medicines.

In the crystal structure, the molecule (figure, top) is non-planar; the dihedral angle between the two aromatic rings is $16.65(1)^\circ$. As a result of conjugation, the C=O distance ($1.229(3)$ Å) is longer than the normal value of 1.20 Å, and the C1—N1 bond distance ($1.337(3)$ Å) is longer than the C=N double-bond distance and shorter than the C—N single-bond distance [5]. The two methyl groups are slightly twisted away from the plane of the attached benzene ring ($\angle \text{C16—N3—C12—C13} = -1.7(3)^\circ$ and $\angle \text{C15—N3—C12—C11} = 11.2(4)^\circ$). An intramolecular N1—H1...O2 hydrogen bond which forms a six-membered ring is observed between the NH group and the hydroxy O atom (figure, bottom). The O2—H2...N2ⁱ and O2—H2...O1ⁱ (symmetry code (i): $\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}+z$) hydrogen bonds form a five-membered ring.

Table 1. Data collection and handling.

Crystal:	yellow block, size $0.11 \times 0.12 \times 0.51$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.90 cm^{-1}
Diffraction, scan mode:	Bruker SMART APEX CCD, ϕ/ω
$2\theta_{\text{max}}$:	53.98°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	7792, 2974
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2404
$N(\text{param})_{\text{refined}}$:	201
Programs:	SHELXS-97 [6], SHELXL-97 [7]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(3)	4a	0.1603	−0.1472	0.1681	0.056
H(4)	4a	0.1050	−0.4482	0.2305	0.067
H(5)	4a	0.1058	−0.5400	0.4302	0.068
H(6)	4a	0.1609	−0.3278	0.5645	0.058
H(8)	4a	0.3104	0.5351	0.4636	0.049
H(10)	4a	0.3727	0.7259	0.1838	0.052
H(11)	4a	0.4421	1.0116	0.1579	0.053
H(13)	4a	0.4354	1.1345	0.5087	0.054
H(14)	4a	0.3662	0.8474	0.5335	0.054
H(15A)	4a	0.5298	1.2565	0.1549	0.092
H(15B)	4a	0.5275	1.4935	0.2024	0.092
H(15C)	4a	0.4629	1.3844	0.1537	0.092
H(16A)	4a	0.4691	1.4906	0.4422	0.083
H(16B)	4a	0.5400	1.5176	0.3874	0.083
H(16C)	4a	0.5254	1.3273	0.4751	0.083
H(1)	4a	0.263(1)	0.244(4)	0.420(2)	0.043(7)
H(2)	4a	0.226(2)	−0.046(5)	0.597(3)	0.09(1)

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

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	4a	0.22234(9)	0.1855(3)	0.1685(1)	0.076(1)	0.060(1)	0.0280(9)	−0.0094(9)	−0.0059(8)	0.0112(8)
O(2)	4a	0.22459(9)	0.0219(3)	0.5294(1)	0.067(1)	0.058(1)	0.030(1)	−0.0164(9)	−0.0080(8)	0.0152(8)
N(1)	4a	0.2625(1)	0.2687(3)	0.3480(2)	0.053(1)	0.042(1)	0.026(1)	−0.005(1)	−0.0003(9)	0.0105(8)
N(2)	4a	0.29758(9)	0.4432(3)	0.3037(2)	0.045(1)	0.039(1)	0.035(1)	−0.0015(9)	0.0010(8)	0.0103(8)
N(3)	4a	0.4885(1)	1.2820(4)	0.3142(2)	0.061(1)	0.054(1)	0.044(1)	−0.016(1)	−0.001(1)	0.000(1)
C(1)	4a	0.2271(1)	0.1437(4)	0.2750(2)	0.040(1)	0.041(1)	0.032(1)	0.005(1)	−0.0004(9)	0.008(1)
C(2)	4a	0.1931(1)	−0.0470(4)	0.3274(2)	0.035(1)	0.039(1)	0.034(1)	0.0059(9)	−0.0002(9)	0.006(1)
C(3)	4a	0.1601(1)	−0.1814(4)	0.2486(2)	0.050(1)	0.053(2)	0.038(1)	0.001(1)	−0.000(1)	−0.001(1)
C(4)	4a	0.1273(1)	−0.3627(5)	0.2853(2)	0.060(2)	0.053(2)	0.055(2)	−0.007(1)	−0.006(1)	−0.004(1)
C(5)	4a	0.1277(1)	−0.4169(5)	0.4044(3)	0.054(2)	0.048(2)	0.068(2)	−0.012(1)	0.001(1)	0.009(1)
C(6)	4a	0.1604(1)	−0.2893(4)	0.4845(2)	0.048(2)	0.054(2)	0.045(1)	−0.004(1)	−0.001(1)	0.017(1)
C(7)	4a	0.1930(1)	−0.1035(4)	0.4486(2)	0.039(1)	0.042(1)	0.037(1)	0.001(1)	−0.001(1)	0.006(1)
C(8)	4a	0.3208(1)	0.5667(4)	0.3849(2)	0.045(1)	0.044(1)	0.035(1)	0.001(1)	−0.000(1)	0.009(1)
C(9)	4a	0.3618(1)	0.7514(4)	0.3619(2)	0.039(1)	0.041(1)	0.035(1)	0.002(1)	−0.002(1)	0.0060(9)
C(10)	4a	0.3854(1)	0.8071(4)	0.2495(2)	0.049(1)	0.045(1)	0.037(1)	−0.004(1)	−0.001(1)	−0.002(1)
C(11)	4a	0.4269(1)	0.9797(4)	0.2340(2)	0.052(1)	0.050(1)	0.032(1)	−0.004(1)	0.001(1)	0.006(1)
C(12)	4a	0.4473(1)	1.1101(4)	0.3300(2)	0.040(1)	0.042(1)	0.037(1)	0.002(1)	−0.0013(9)	0.003(1)
C(13)	4a	0.4231(1)	1.0534(4)	0.4428(2)	0.050(1)	0.052(2)	0.034(1)	−0.007(1)	−0.002(1)	−0.001(1)
C(14)	4a	0.3816(1)	0.8800(4)	0.4577(2)	0.049(1)	0.054(2)	0.033(1)	−0.002(1)	0.003(1)	0.005(1)
C(15)	4a	0.5034(1)	1.3607(5)	0.1965(2)	0.075(2)	0.059(2)	0.051(2)	−0.019(2)	0.007(1)	0.008(1)
C(16)	4a	0.5073(1)	1.4155(4)	0.4129(3)	0.057(2)	0.050(2)	0.058(2)	−0.007(1)	−0.006(1)	−0.006(1)

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