Crystal structure of (*E*)-*N*′-(4-hydroxybenzylidene)benzohydrazide monohydrate, $C_{14}H_{12}N_2O_2 \cdot H_2O$

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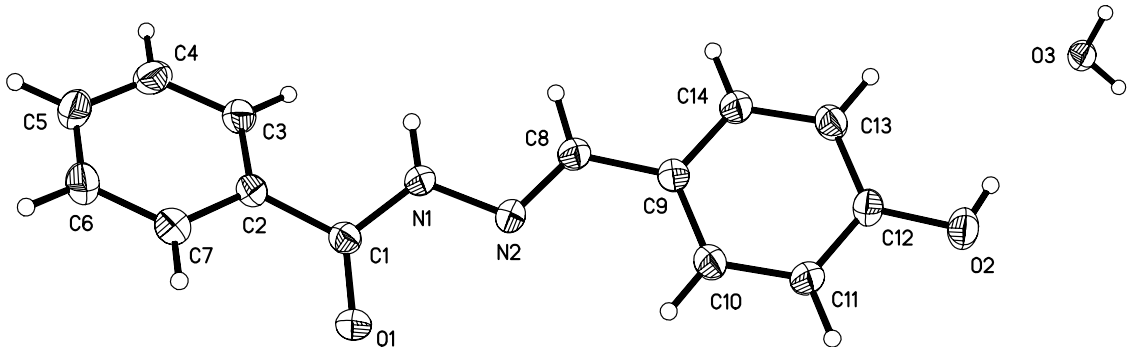

Abstract

$C_{14}H_{14}N_2O_3$, orthorhombic, *Pbca* (no. 61), a = 10.384(2) Å, b = 11.218(2) Å, c = 22.702(3) Å, V = 2644.4 Å^3, Z = 8, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.111, T = 298 K.

Source of material

All commercially available reagents were used as supplied. *p*-Hydroxylbenzaldehyde (10 mmol) was added to a solution of benzoic acid hydrazide (10 mmol) in 10 ml of ethanol. The mixture was continuously stirred for 3 h at refluxing temperature, evaporating some ethanol, then, upon cooling, the solid product was collected by filtration and dried in vacuo (yield 89 %). Clear block-shaped crystals of the title compound were obtained by evaporation from a methanol solution after two weeks.

Discussion

Arylhydrazone and its coordination chemistry are widely studied in the fields of biology, catalysis and also materials research and it is thus of high significance to develop new methods to prepare new arylhydrazones and its complexes and to investigate their properties and applications. As part of our ongoing study of complexes with arylhydrazone ligands [1], we synthesized (*E*)-*N*′-(4-hydroxybenzylidene)benzohydrazide.

The X-ray crystal structure analysis revealed the title compound as monohydrate. The location of the double bond between C8—N2 (1.277(3) Å) is as expected for this class of compound, and the geometrical parameters are normal. The *para*-hydroxybenzylidene and the hydrazone units of the molecule are planar with a dihedral angle of 11.7(3)° between the planes formed by N2 and C8 through C14 and that made up by O1, C1, N1 and N2. The second aromatic ring (C2 to C7), however, is significantly rotated out of the plane formed by the remainder of the molecule by 35.1(3)°. This rotation of the phenyl ring against the plane of the amide unit allows the molecule to form a strong N–H···O hydrogen bond with the interstitial water molecule without excessive steric interactions of the *ortho* hydrogen atoms of the aromatic ring, thus making up for the partial loss of delocalization energy along the π-system by the deviation form planarity. Several other O–H···O, O–H···N, and C–H···O hydrogen bonds form a closely knit hydrogen bonding network between the main molecule and the interstitial water molecule.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.37 × 0.38 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.93 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10208, 2328
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1506
$N(param)_{refined}$:	173
Program:	SHELXTL [2]

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

Atom	Site	x	y	z	U_{iso}
H(1)	8*c*	0.2591	0.6526	0.0676	0.050
H(2)	8*c*	0.4726	1.0547	0.3580	0.085
H(3A)	8*c*	0.0750	–0.0257	0.0553	0.054
H(3B)	8*c*	0.0263	0.0876	0.0482	0.054
H(3)	8*c*	0.1613	0.4978	0.0284	0.054
H(4)	8*c*	0.1546	0.3508	–0.0426	0.069
H(5)	8*c*	0.1354	0.4014	–0.1406	0.079
H(6)	8*c*	0.1206	0.5981	–0.1676	0.080
H(7)	8*c*	0.1193	0.7457	–0.0969	0.063
H(8)	8*c*	0.3437	0.7035	0.1484	0.048
H(10)	8*c*	0.2450	1.0057	0.1666	0.059
H(11)	8*c*	0.3061	1.1310	0.2412	0.062
H(13)	8*c*	0.4911	0.8632	0.3208	0.053
H(14)	8*c*	0.4337	0.7385	0.2448	0.052

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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(1)	8*c*	0.2162(2)	0.7179(2)	0.06427(7)	0.051(1)	0.033(1)	0.041(1)	0.0084(8)	–0.0102(9)	–0.0049(9)
N(2)	8*c*	0.2266(2)	0.8045(2)	0.10730(8)	0.049(1)	0.037(1)	0.040(1)	0.0034(9)	–0.0063(9)	–0.0077(9)
O(1)	8*c*	0.0748(2)	0.8262(1)	0.01120(7)	0.060(1)	0.0402(9)	0.051(1)	0.0130(8)	–0.0140(8)	–0.0020(8)
O(2)	8*c*	0.4365(2)	1.0894(1)	0.33084(7)	0.078(1)	0.046(1)	0.047(1)	–0.0023(9)	–0.0188(9)	–0.0082(8)
O(3)	8*c*	0.0694(1)	0.0428(1)	0.07112(6)	0.057(1)	0.0388(9)	0.0400(9)	0.0044(7)	–0.0050(7)	–0.0031(7)
C(1)	8*c*	0.1397(2)	0.7348(2)	0.01726(9)	0.040(1)	0.037(1)	0.037(1)	0.001(1)	–0.004(1)	0.002(1)
C(2)	8*c*	0.1410(2)	0.6370(2)	–0.02675(9)	0.036(1)	0.042(1)	0.038(1)	0.001(1)	–0.006(1)	–0.003(1)
C(3)	8*c*	0.1518(2)	0.5185(2)	–0.0110(1)	0.045(1)	0.042(1)	0.048(1)	0.001(1)	–0.007(1)	–0.004(1)
C(4)	8*c*	0.1487(2)	0.4306(2)	–0.0535(1)	0.051(2)	0.045(2)	0.077(2)	0.009(1)	–0.012(1)	–0.017(1)
C(5)	8*c*	0.1369(2)	0.4607(3)	–0.1119(1)	0.058(2)	0.076(2)	0.064(2)	0.015(1)	–0.015(1)	–0.034(2)
C(6)	8*c*	0.1274(2)	0.5779(3)	–0.1280(1)	0.069(2)	0.089(2)	0.042(2)	0.020(2)	–0.011(1)	–0.015(2)
C(7)	8*c*	0.1279(2)	0.6663(2)	–0.0858(1)	0.058(2)	0.054(2)	0.045(2)	0.010(1)	–0.008(1)	–0.002(1)
C(8)	8*c*	0.3042(2)	0.7779(2)	0.14894(9)	0.046(1)	0.035(1)	0.040(1)	0.003(1)	–0.003(1)	–0.000(1)
C(9)	8*c*	0.3334(2)	0.8584(2)	0.19699(9)	0.038(1)	0.038(1)	0.037(1)	–0.001(1)	–0.002(1)	–0.001(1)
C(10)	8*c*	0.2955(2)	0.9766(2)	0.1972(1)	0.059(2)	0.043(2)	0.044(1)	0.004(1)	–0.016(1)	–0.001(1)
C(11)	8*c*	0.3312(2)	1.0514(2)	0.2421(1)	0.068(2)	0.034(1)	0.054(2)	0.009(1)	–0.017(1)	–0.004(1)
C(12)	8*c*	0.4044(2)	1.0097(2)	0.28856(9)	0.046(1)	0.040(1)	0.039(1)	–0.006(1)	–0.004(1)	–0.005(1)
C(13)	8*c*	0.4422(2)	0.8922(2)	0.28957(9)	0.049(1)	0.044(1)	0.040(1)	–0.000(1)	–0.009(1)	0.002(1)
C(14)	8*c*	0.4073(2)	0.8178(2)	0.2441(1)	0.051(1)	0.036(1)	0.043(1)	0.004(1)	–0.007(1)	–0.002(1)

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