Crystal structure of (*Z*)-3-(2-hydroxyphenylamino)-1-(pyridin-3-yl)but-2-en-1-one, $C_{15}H_{14}N_2O_2$

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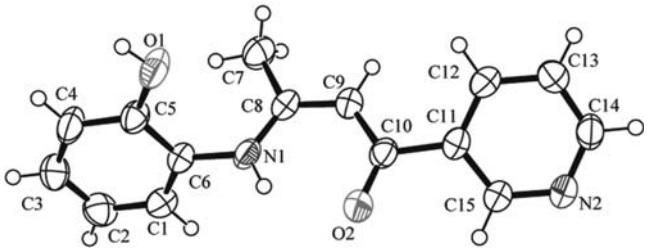

Abstract

$C_{15}H_{14}N_2O_2$, monoclinic, $P2_1/n$ (no. 14), a = 12.252(2) Å, b = 7.719(1) Å, c = 14.500(3) Å, β = 112.731(7)°, V = 1264.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.148, T = 293 K.

Source of material

Solution of 1-(pyridin-3-yl)butane-1,3-dione (0.02 mol) in ethanol (10 ml) was added to a stirred ethanol solution (10 ml) of 2-aminophenol (0.02 mol) in a three-necked flask. The reaction mixture was refluxed for 5 h, then the solution was cooled to room temperature. The yellow precipitated residue was removed from the solution by filtration and then washed with ethanol 3 times. Single crystals suitable for X-ray diffraction were obtained from an ethanol/chloroform mixture (1:1, *v*:*v*) by slow evaporation at room temperature.

Experimental details

Hydrogen atoms bound to nitrogen and oxygen atoms were found in the difference Fourier maps and refined freely. The other hydrogen atoms bound to carbon atoms were positioned geometrically with d(C—H) = 0.93 Å, U_{iso}(H) = 1.2 U_{eq}(C) for Csp^2 and d(C—H) = 0.96 Å, U_{iso}(H) = 1.5 U_{eq}(C) for Csp^3.

Discussion

Pyridinone derivatives are of interest due to their biological and pharmacological activity. Recently, some of these compounds were studied as potential hypnotic drugs [1]. Aminophenols in their turn shows the ability to control the packing arrangement often producing the assembly of supramolecular ladders linked via hydrogen bonding and π-π stacking interaction [2,3].

The title crystal structure is built up of the $C_{15}H_{14}N_2O_2$ molecules with a bezene and a pyridine rings. The bond lengths and bond angles are in the normal ranges. The planes of pyridine and benzene rings form a dihedral angle of 31.0(1)°.

In the title molecule, an intramolecular N1–H1A···O2 hydrogen bond links the aniline NH group to the carbonyl oxygen atom, the distance of the hydrogen bond is d(N1–H1A···O2) = 2.740(2) Å and ∠N1–H1A···O2 = 139(2)°. The intermolecular hydrogen bond, which involves phenol OH-group and the pyridine N atom (d(O1–H1B···N2) = 2.771(3) Å and ∠O1–H1B···N2 = 171(3)°), at the same time, there is also another intermolecular hydrogen bond between the NH-group and the carbonyl oxygen of adjacent molecules (d(N1–H1A···O2) = 3.169(3) Å and ∠N1–H1A···O2 = 130(2)°). These hydrogen bonds interlink the molecules into a 2D net structure.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.21 × 0.22 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.9 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12951, 2227
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1489
$N(param)_{refined}$:	180
Programs:	SHELXS-97, SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.5874	0.2684	1.2293	0.056
H(2)	4*e*	0.5975	0.3146	1.3893	0.065
H(3)	4*e*	0.4262	0.3642	1.4155	0.066
H(4)	4*e*	0.2453	0.3567	1.2831	0.065
H(7A)	4*e*	0.3203	0.0049	1.1061	0.083
H(7B)	4*e*	0.3555	–0.1248	1.0390	0.083
H(7C)	4*e*	0.2303	–0.0372	0.9975	0.083
H(9)	4*e*	0.3217	0.0219	0.8649	0.052
H(12)	4*e*	0.2437	0.0787	0.6988	0.054
H(13)	4*e*	0.2495	0.0159	0.5445	0.057
H(14)	4*e*	0.4147	0.0854	0.5154	0.057
H(15)	4*e*	0.5559	0.3151	0.7735	0.051
H(1A)	4*e*	0.443(2)	0.337(3)	1.033(2)	0.059(8)
H(1B)	4*e*	0.135(3)	0.294(4)	1.118(2)	0.10(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(1)	4*e*	0.5179(2)	0.2859(3)	1.2397(2)	0.040(1)	0.051(1)	0.051(2)	–0.002(1)	0.021(1)	0.002(1)
C(2)	4*e*	0.5246(2)	0.3144(3)	1.3358(2)	0.053(2)	0.061(2)	0.041(1)	–0.002(1)	0.010(1)	0.003(1)
C(3)	4*e*	0.4225(3)	0.3425(3)	1.3513(2)	0.066(2)	0.066(2)	0.036(1)	–0.003(1)	0.024(1)	0.000(1)
C(4)	4*e*	0.3141(2)	0.3385(4)	1.2718(2)	0.053(2)	0.078(2)	0.042(1)	0.003(1)	0.030(1)	0.000(1)
C(5)	4*e*	0.3066(2)	0.3077(3)	1.1747(2)	0.041(1)	0.059(1)	0.034(1)	0.004(1)	0.017(1)	0.007(1)
C(6)	4*e*	0.4112(2)	0.2824(3)	1.1587(2)	0.041(1)	0.043(1)	0.038(1)	0.001(1)	0.021(1)	0.004(1)
C(7)	4*e*	0.3124(2)	–0.0209(3)	1.0390(2)	0.066(2)	0.053(2)	0.055(2)	–0.008(1)	0.032(1)	0.006(1)
C(8)	4*e*	0.3610(2)	0.1264(3)	0.9992(2)	0.038(1)	0.044(1)	0.044(1)	0.002(1)	0.023(1)	0.004(1)
C(9)	4*e*	0.3586(2)	0.1174(3)	0.9034(2)	0.049(2)	0.048(1)	0.040(1)	–0.005(1)	0.024(1)	–0.004(1)
C(10)	4*e*	0.4080(2)	0.2418(3)	0.8590(2)	0.038(1)	0.045(1)	0.042(1)	0.001(1)	0.022(1)	–0.001(1)
C(11)	4*e*	0.4015(2)	0.2031(3)	0.7552(2)	0.042(1)	0.039(1)	0.040(1)	0.005(1)	0.022(1)	0.0028(9)
C(12)	4*e*	0.3084(2)	0.1123(3)	0.6845(2)	0.040(1)	0.051(1)	0.050(1)	–0.003(1)	0.025(1)	–0.001(1)
C(13)	4*e*	0.3125(2)	0.0724(3)	0.5934(2)	0.045(2)	0.054(1)	0.041(1)	–0.004(1)	0.015(1)	–0.006(1)
C(14)	4*e*	0.4110(2)	0.1174(3)	0.5759(2)	0.057(2)	0.054(1)	0.039(1)	0.000(1)	0.026(1)	–0.004(1)
N(2)	4*e*	0.5022(2)	0.2052(3)	0.6417(1)	0.049(1)	0.057(1)	0.042(1)	–0.003(1)	0.027(1)	–0.0009(9)
C(15)	4*e*	0.4948(2)	0.2494(3)	0.7285(2)	0.045(1)	0.047(1)	0.042(1)	–0.005(1)	0.024(1)	–0.002(1)
N(1)	4*e*	0.4086(2)	0.2614(3)	1.0598(1)	0.043(1)	0.048(1)	0.042(1)	–0.0022(9)	0.027(1)	0.0026(9)
O(1)	4*e*	0.2012(2)	0.3013(3)	1.0951(1)	0.039(1)	0.103(2)	0.040(1)	0.0122(9)	0.0205(9)	0.0077(9)
O(2)	4*e*	0.4602(2)	0.3759(2)	0.9024(1)	0.064(1)	0.053(1)	0.048(1)	–0.0141(8)	0.0324(9)	–0.0085(8)

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