

STRUCTURE OF ORGANIC
COMPOUNDSCrystal Structure of *N'*-(Furan-2-ylmethylene)-4-hydroxybenzohydrazide¹N. Dilek^{a,*}, B. Güneş^b, C. Gokce^c, and R. Gup^c^a Department of Physics, Arts and Sciences Faculty, Aksaray University, 68100, Aksaray, Turkey^b Department of Physics Education, Gazi Education Faculty, Gazi University, 06500, Teknikokullar, Ankara, Turkey^c Department of Chemistry, Arts and Sciences Faculty, Muğla Sıtkı Koçman University, 48000, Kötekli, Muğla, Turkey

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Abstract—We report the molecular and crystal structures of the title compound. The structure of title compound in crystal was determined by the X-ray diffraction method. The molecule exists in a *trans*-configuration with respect to the C=N bond. The dihedral angle between two rings is 47.7(1)°. This compound crystallizes in the orthorhombic, *Pna*2₁ space group with unit cell parameter *a* = 9.6014(6) Å, *b* = 11.1849(8) Å, *c* = 10.3574(7) Å. The N–H···O and O–H···N interactions stabilize the molecules in the lattice. A weak C–H···π interactions are also present.

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INTRODUCTION

Schiff bases have attracted great and growing interest in chemistry and biology since 1864 when Hugo Schiff reported the condensation of primary amines with carbonyl compounds [1] due to their wide applications [2–5]. Many biologically important Schiff bases have been reported in the literature possessing, antimicrobial, antibacterial, antifungal, anti-inflammatory, anticonvulsant, antitumor, antihypertensive activity and anti HIV activities [6–10]. The importance of Schiff base complexes for bioinorganic chemistry, biomedical applications and supramolecular chemistry has been well-recognized. They have been widely studied because they have antibacterial [11],

antimicrobial [12], antitubercular [13], antifungal [14], anticancer and herbicidal applications [15]. They serve as models for biologically important species. Therefore, nowadays, the research field dealing with Schiff bases and their coordination chemistry has expanded enormously.

EXPERIMENTAL

Synthesis. This compound was synthesized as described in previously reported method [16]. The filtered was slowly evaporated in air to give brown ligand (Fig. 1). The crystals were isolated and washed with water. Yield 82%; M.p. 236°C; UV (*DMF*, nm) 232,

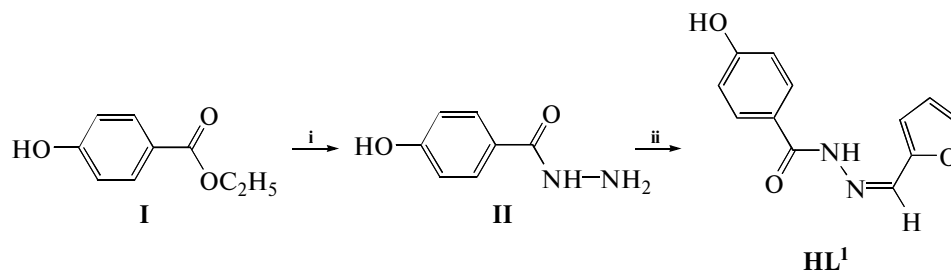


Fig. 1. Schematic diagram showing the synthesis of *N'*-(furan-2-ylmethylene)-4-hydroxybenzohydrazide. i, $\text{NH}_2\text{NH}_2 \cdot \text{H}_2\text{O}$, *EtOH*, reflux 4h. ii, furfural, *EtOH*, *AcOH*, reflux 4 h.

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Table 1. Crystallographic characteristics and the X-ray-data collection and structure-refinement parameters for C₁₂H₁₀N₂O₃

System, sp. gr., <i>Z</i>	Orthorhombic, <i>Pna</i> 2 ₁ , 4
<i>a</i> , <i>b</i> , <i>c</i> , Å	9.6014(6), 11.1849(8), 10.3574(7)
<i>V</i> , Å ³	1112.29(13)
<i>D_x</i> , g cm ⁻³	1.375
Radiation, λ, Å	MoK _α , 0.71073
μ, mm ⁻¹	0.101
<i>T</i> , K	296(2)
Sample size, mm	0.634 × 0.334 × 0.178
Scan mode	φ- and ω-
Absorption correction, <i>T_{min}</i> , <i>T_{max}</i>	Multi-scan, 0.9391, 0.9820
θ _{max} , deg	28.31
<i>h</i> , <i>k</i> , <i>l</i> ranges	-12 ≤ <i>h</i> ≤ 12, -14 ≤ <i>k</i> ≤ 14, -13 ≤ <i>l</i> ≤ 13
Number of reflections: measured/unique (<i>N</i> ₁), <i>R_{int}</i> /with <i>I</i> > 2σ(<i>I</i>) (<i>N</i> ₂)	19571/1461 0.0222/1375
Refinement method	full-matrix least square in <i>F</i> ²
Number of refined parameters	155
<i>R</i> ₁ / <i>wR</i> ₂ relative to <i>N</i> ₁	0.0338/0.0913
<i>R</i> ₁ / <i>wR</i> ₂ relative to <i>N</i> ₂	0.0366/0.0942
<i>S</i>	1.121
Δρ _{max} /Δρ _{min} , e/Å ³	0.273/-0.161
Programs	APEX2 SAINT [17], SADABS [18], SHELXS97 [19], SHELXL97 [19], ORTEP-3 [20], PLATON [21], WinGX [22]

317; IR (KBr) (ν, cm⁻¹) 3246 (OH), 1634 (C=O), 1605 (C=N), 1234 (C-O); ¹H NMR (*DMSO d*₆) δ 6.94 (d, 2H, *Ar*-He), 7.89 (d, 2H, *Ar*-Hd), 7.84 (s, 1H, Ha), 6.91 (d, 1H, Hc), 6.35 (q, 1H, Hb), 8.42 (s, 1H, HC=N), 10.24 (s, 1H, OH), 11.75 (s, 1H, NH); ¹³C NMR (*DMSO d*₆, ppm) 145.6 (C1), 112.8 (C2), 124.4 (C3), 150.3 (C4), 161.5 (C5), 163.7 (C6), 130.4 (C7), 113.7 (C8), 115.8 (C9) and 137.6 (C10). Analysis (%Calculated/found) for C₁₂H₁₀N₂O₃: C: 62.60/62.27, H: 4.40/4.53, N:12.17/11.89.

X-ray diffraction study. Crystallographic data were recorded on a Bruker Smart Breeze CCD diffractometer. Crystallographic characteristics and the X-ray data collection and structure-refinement parameters are given in Table 1. The structure was solved by direct method and refined in anisotropic approximation for non-hydrogen atoms with crystallographic packet programs [17–22]. The H atoms were positioned geometrically, with C–H = 0.93, N–H = 0.86 and O–H = 0.82 Å, and constrained to ride on their parent atoms, with *U*_{iso}(H) = 1.2*U*_{eq}(C,N,O).

The selected bond distance, bond angles, and torsion angles are given in Table 2.

Crystallographic data for the structural analysis have been deposited with Cambridge crystallographic Data Centre, CCDC reference number 975952. Copies of this information can be obtained free of charge from The Director, CCDC, 12 Union road, Cambridge, CB12 1EZ, UK. (Fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Description of the crystal structure. The crystal structure is shown in Fig. 2 with atom-numbering

Table 2. Selected bond lengths (Å), bond angles (°) and torsion angles (°)

Bond lengths					
O1–C1	1.357(2)	O2–C7	1.231(2)	O3–C9	1.365(2)
O3–C12	1.365(3)	N2–C8	1.280(2)	N1–C7	1.354(2)
N1–N2	1.385(2)				
Bond angles					
O1–C1–C2	118.8(2)	N1–C7–C4	117.5(2)	C11–C12–O3	110.9(2)
O1–C1–C6	121.8(2)	C7–N1–N2	117.8(1)	C9–O3–C12	105.7(2)
O2–C7–N1	120.9(2)	C8–N2–N1	114.9(2)	O3–C9–C8	119.4(2)
O2–C7–C4	121.6(2)	N2–C8–C9	121.5(2)		
Torsion angles					
C5–C4–C7–O2	8.2(3)	N2–C8–C9–C10	-164.8(3)	O2–C7–N1–N2	3.8(3)
C3–C4–C7–N1	7.6(3)	N2–C8–C9–O3	8.5(3)	C7–N1–N2–C8	-153.1(2)

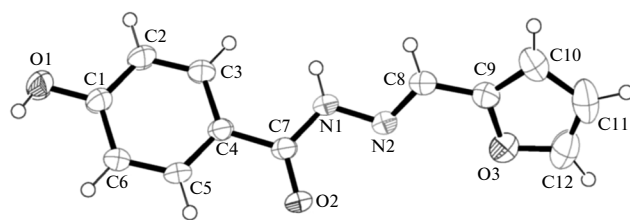


Fig. 2. The asymmetric unit of the title compound, represented with displacement ellipsoids are drawn at 50% probability level and showing the labeling scheme.

scheme. The bond lengths and angles are in the normal ranges in the molecule. The molecule exists in a *trans* configuration with respect to the C8=N2 [1.280(2) Å] bond and the torsion angle C9–C8–N2–N1 = 177.9(2)°. The all bond lengths and angles in the molecule are within expected ranges, and similar to the other studies [23–28]. The ring A (C1–C6) and B (C9–C12, O3) are each essentially planar. The dihedral angle between two rings is 47.7(1)°, indicating the

Schiff base molecule is twisted. The C8 atom lie above 0.134 (4) from the B plane.

As can be seen from the packing diagram (Fig. 3), inter-molecular N–H···O, O–H···N and C–H···O hydrogen bonds (Table 3) link the molecules and these hydrogen bonds may be effective in the stabilization of the crystal structure. In this interaction, there are the O1, N1, C5, C6, C8 atoms of molecule as donor and

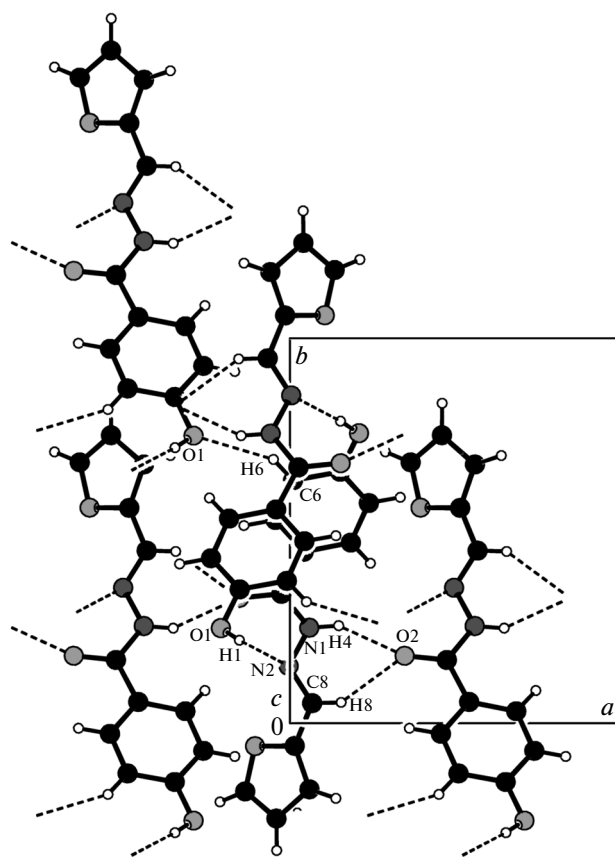


Fig. 3. View of the crystal packing of the title compound, along the c axis. Hydrogen bonds are indicated by dashed lines.

Table 3. Hydrogen bond geometry (Å, °)

<i>D</i> –H... <i>A</i>	Distances			<i>D</i> –H... <i>A</i> angle
	<i>D</i> ... <i>A</i>	<i>D</i> –H	H... <i>A</i>	
O1–H1...N2 ⁱ	2.970(2)	0.82	2.15	175
N1–H4...O2 ⁱⁱ	2.917(2)	0.86	2.14	151
C6–H6...O1 ⁱⁱⁱ	3.202(2)	0.93	2.42	142
C8–H8...O2 ⁱⁱ	3.157(2)	0.93	2.35	145

Symmetry codes: (i) $-x, 1 - y, 1/2 + z$; (ii) $1/2 + x, 1/2 - y, z$; (iii) $-1/2 + x, 3/2 - y, z$.

the N2, O2 and O1 atoms the other molecules as acceptor.

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