



Synthesis of *N'*-(2,3-dihydroxybenzylidene)-4-methylbenzohydrazide: Spectroscopic Characterization and X-ray Structure Determination

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Abstract: p-Toluic hydrazide was used for synthesizing a new Schiff base *N'*-(2,3-dihydroxybenzylidene)-4-methylbenzohydrazide (I). The prepared compound was characterized by elemental analysis, infrared and ¹H and ¹³C NMR spectroscopy techniques, and the structure of compound (I) was determined by single-crystal X-ray diffraction study. The compound (C₁₅H₁₄N₂O₃) crystallises in the monoclinic space group *P*2₁/*n* with the following unit cell parameters: *a* = 11.7820 (6) Å, *b* = 8.5278 (4) Å, *c* = 13.6054 (7) Å, *β* = 109.346 (5)°, *V* = 1289.81 (12) Å³, *Z* = 4, *R*₁ = 0.071 and *wR*₂ = 0.197. The X-ray structure shows that the compound (I) adopts an *E* configuration with respect to the double bond C7=N1. The carbonyl oxygen atom O3 and the nitrogen atom N1 of the hydrazide moiety are in a *syn* conformation with respect to C8—N2 bond. The crystal packing of compound (I) is stabilized by intramolecular O(phenol)—H···N(carbohydrazide) which results in an *S*(6) ring motif and intermolecular O(phenol)—H···O(carbohydrazide) hydrogen bonds which form chains. The carbonohydrazide moiety C=N—N—C(O)—N=N=C fragment is almost coplanar with the two benzene rings with dihedral angles of their mean planes of 10.35° [C1—C6] and 7.99° [C9—C14], respectively. The dihedral angle between the mean planes of the phenyl rings is 2.46°.

Keywords: Schiff Base, 2,3-dihydroxybenzaldehyde, p-toluic Hydrazide, Single Crystal Structure

1. Introduction

The hydrazides are compounds possessing a very reactive arm with carbonyl compounds. Control of the hydrazide/carbonyl ratio allows to synthesize mono, di or tri condensation compounds. In these types of compounds, the part containing the azomethine group connected to a carbonyl group (—HC=N—NH—C(O)—) is responsible for certain pharmaceutical properties [1–4]. Hydrazide derivatives have been used as precursors for the synthesis of various heterocyclic compounds containing a nitrogen atom in the ring and/or in the free arms [5–7]. The reactivity as well as the

biological properties of compounds derived from hydrazide have been widely studied for the development of drugs or phytosanitary products [8–11]. They are known to possess a wide spectrum of biological activities such as antioxidant [12], analgesic [13], antifungal [14], antimicrobial [15], anti-spasmodic [16], anti-inflammatory [17], antituberculosis [18], and anticancer [19, 20]. These Schiff bases are also known for their physical properties. They are used in the field of fluorescence [21, 22], optics [23] and corrosion inhibition [24]. In the field of coordination chemistry, these Schiff bases are recognized as multitopic ligands for the targeted construction of original

metallo-organic architectures [25–27]. In this context, we report the synthesis and crystal structure of a derivative of p-toluic acid hydrazide and 2,3-dihydroxybenzaldehyde.

2. Experimental

2.1. Starting Materials and Instrumentations

p-toluic hydrazide and 2,3-dihydroxybenzaldehyde were purchased from Sigma-Aldrich and used as received without further purification. All solvents used were of reagent grade. Melting points were determined on a Büchi 570 melting-point apparatus and were uncorrected. Elemental analyses of C, H and N were recorded on a VxRio EL Instrument. Infrared spectra were obtained on a FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000–400 cm^{-1} region. The ^1H and ^{13}C NMR spectra were recorded in DMSO- d_6 on a BRUKER 300.13 MHz spectrometer at room temperature using TMS as an internal reference.

2.2. Synthesis of

N'-(2,3-dihydroxybenzylidene)-4-methylbenzohydrazide (*H₃L*) (I)

p-toluic hydrazide (1.5 g, 10 mmol) and 2,3-dihydroxybenzaldehyde (1.38 g, 10 mmol) were mixed in ethanol and one drop of glacial acetic acid was added. The mixture was stirring under reflux for 120 minutes. On cooling the white precipitate which appears was recovered by filtration. The precipitate was washed with ethanol before being dried in air. Recrystallization of the compound in ethanol afforded white crystal suitable for X-ray analyze after two days. Yield 85%. M.p. > 260 °C. Anal. Calc. for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_3$: C, 66.66; H, 5.22; N, 10.36%. Found: C, 66.64; H, 5.20; N, 10.33%. FTIR (ν , cm^{-1}): 3287 (N—H); 1684 (C=O); 1611 (C=N); 1557–1481 $\text{C}_{\text{Ar}}=\text{C}_{\text{Ar}}$; 1266 (C—O)_{phenol}; 1070 (N—N). ^1H NMR (DMSO- d_6 , δ (ppm)): 2.5 (3H, s, CH₃), 6.8 (3H, mult, H—Ar), 7.4 (2H, d, H—Ar), 7.8 (2H, d, H—Ar), 8.6 (1H, s, H—C=N), 9.3 (1H, s, H—N), 11.3 (1H s, H—OPh), 12.2 (1H s, H—OPh). ^{13}C NMR (DMSO- d_6 , δ (ppm)): 163.10 (C=O); 149.35 (C=N); 146.60 ($\text{C}_{\text{Ar}}-\text{O}$), 146.08 ($\text{C}_{\text{Ar}}-\text{O}$), 142.5 (C_{Ar}), 130.54 (C_{Ar}), 129.55 (C_{Ar}), 128.13 (C_{Ar}), 120.64 (C_{Ar}), 119.60 (C_{Ar}), 119.22 (C_{Ar}), 117.84 (C_{Ar}), 21.46 (—CH₃).

2.3. Crystal Structure Determination

Crystals suitable for single-crystal X-ray diffraction, of the reported compound, was grown by slow evaporation of EtOH solution of the compound. Details of the crystal structure solution and refinement are given in Table 1. Diffraction data were collected using a Bruker APEX-II CCD diffractometer with graphite monochromatized MoK α radiation ($\lambda = 0.71073$ Å). All data were corrected for Lorentz and polarization effects. The structure was solved and refined using the Bruker SHELXTL Software Package [28]. All the structures were refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms [29]. H atoms of the NH group was located in the

Fourier difference maps and refined without restraints. Other H atoms were geometrically optimized and refined as riding on their carriers with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ (1.5 for CH₃ groups). Molecular graphics were generated using ORTEP-3 [30].

Table 1. Crystallographic data and refinement parameters for compound *H₃L* (I).

Chemical formula	$\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_3$
M_r	270.28
Crystal shape/color	Prismatic/Dark yellow
Crystal size (mm)	$0.13 \times 0.11 \times 0.06$
Crystal system, space group	Monoclinic, $P2_1/n$
a (Å)	11.7820 (6)
b (Å)	8.5278 (4)
c (Å)	13.6054 (7)
β (°)	109.346 (6)
V (Å ³)	1289.81 (12)
Z	4
D_{cal} (g cm ⁻³)	1.392
Temperature (K)	100
Radiation type	Mo K α
μ (mm ⁻¹)	0.10
Index ranges	$-14 \leq h \leq 14$; $-10 \leq k \leq 10$; $-16 \leq l \leq 16$
$F(000)$	568
q range (°)	2.851–34.063
No. of measured reflections	15026
No. of independent reflections	2449
No. of observed [$I > 2\sigma(I)$] reflections	2983
R_{int}	0.055
$R[F^2 > 2\sigma(F^2)]$	0.056
$wR(F^2)$	0.160
Goodness-of-fit (GOF)	1.07
No. of parameters	184
No. of restraints	0
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.36, -0.28

3. Result and Discussion

3.1. General Study

The compound obtained after the condensation reaction between p-toluic hydrazide and 2,3-dihydroxybenzaldehyde (Figure 1) is characterized by physico-chemical and spectroscopic analyses. Elemental analysis of the compound confirms the formation of the compound with the chemical formula $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_3$. The FTIR spectrum of the *H₃L* (I) ligand shows bands pointed at 3286 and 3140 cm^{-1} attributable respectively to the phenolic ν_{OH} and ν_{NH} vibrations of the hydrazide function [31]. The vibration of the $\nu_{\text{C=O}}$ carbonyl appears at 1634 cm^{-1} while the $\nu_{\text{C=N}}$ band is pointed at 1611 cm^{-1} [32]. Between 1557 and 1481 cm^{-1} we identify the bands corresponding to the $\nu_{\text{C=C}}$ vibrations of the aromatic ring. The FTIR spectrum also reveals a middle band at 1242 cm^{-1} attributed to the $\nu_{\text{C=N}}$ vibration and a strong band at 1266 cm^{-1} attributed to the phenolic $\nu_{\text{C-O}}$ bonding vibration. The band that appears at 1070 cm^{-1} is due to the $\nu_{\text{N-N}}$ vibration. The proton NMR spectrum confirms the proposed formula. Indeed, two characteristic phenolic proton signals are pointed at 12.2 and 11.3 ppm. A signal at 8.6 ppm attributed to the proton of the azomethine group confirms the condensation. At 9.3 ppm, a signal characteristic of the presence of HN— appears. The

signals that appear in the 6.8-7.8 ppm region are due to the protons of the aromatic nuclei. The protons of the CH₃-Ar group appear around 2.5 ppm as expected for protons of this type. The carbon NMR spectrum shows characteristic signals from the carbon atoms of the C=O and C=N groups at 163.10 and 149.35 ppm, respectively. The C_{ipso}-OH carbon atoms appear at 146.60 and 146.08 ppm, respectively.

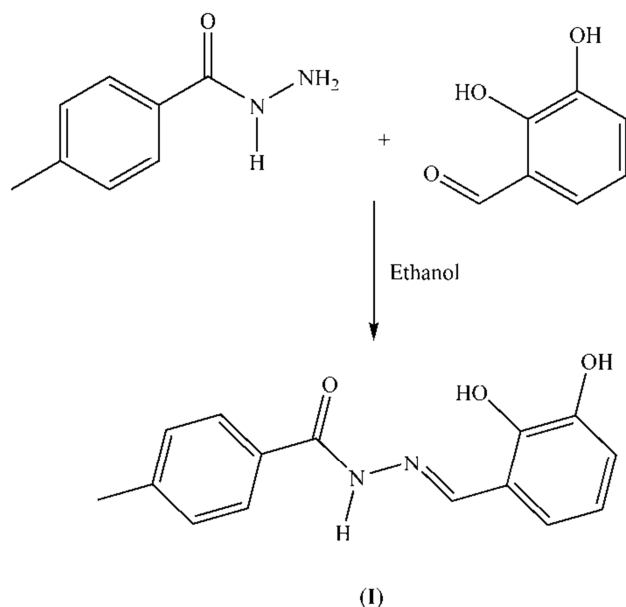


Figure 1. Synthetic scheme for the ligand (I).

3.2. X-ray Structure

The compound (I) crystallizes in Monoclinic, $P2_1/n$. The asymmetric unit contains one ligand molecule of the Schiff base ligand. The molecular structure of the title compound (I) with the atomic-labeling scheme is shown in Figure 2. Selected bond distances and torsion angle are listed in Table 2. The Schiff base adopts an *E* configuration with respect to the

C7=N1. The carbonyl oxygen atom O3 and the nitrogen atom N1 of the hydrazide moiety are in a *syn* conformation with respect to C8—N2 [O3—C8—N2—N1 = 2.1 (3)°]. The carbonohydrazide C7=N1—N2—C8=O3 moiety is almost coplanar with the two benzene rings with dihedral angles of their mean planes of 10.35° [C1—C6] and 7.99° [C9—C14]. The two benzene rings are almost coplanar with dihedral angle of 2.46°. The C8=O3 and C7=N1 bond length of 1.240 (2) Å and 1.283 (3) Å, respectively, which are double-character are indicative of non-enolization of the compound (I) which exist only in its amid form. The C8—N2 [1.348 (3) Å] and the N1—N2 [1.379 (2) Å] distances indicate that these correspond to single bonds and are comparable to those observed for 1-(1-Phenylethylidene)carbonohydrazide [33]. The intramolecular hydrogen bonds O1(phenol)—H···N1(azomethine) forms an *S*(6) ring motif. In the crystal, each independent molecule (I) donates hydrogen bonds to the carbonyl oxygen atom of one ligand molecule, forming a chain [O2(phenol)—H···O3ⁱ(carbonyl); *i* = -*x*+3/2, *y*+1/2, -*z*+3/2] (Figure 3, Table 3).

Table 2. Selected bond lengths (Å) and torsion angles (deg) for the compound (I).

Atom-Atom		Atom-Atom-Atom-Atom	
O3—C8	1.240 (2)	C14—C9—C8—O3	9.6 (3)
O1—C1	1.352 (2)	C8—N2—N1—C7	170.1 (2)
O2—C2	1.369 (2)	N1—C7—C6—C1	-2.5 (3)
N2—N1	1.379 (2)	C10—C9—C8—O3	172.1 (2)
N2—C8	1.348 (3)	O3—C8—N2—N1	2.1 (3)
N1—C7	1.283 (3)	N2—N1—C7—C6	-179.4 (2)

Table 3. Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.84	1.90	2.630 (2)	144.6
O2—H2···O3 ⁱ	0.84	1.83	2.6647 (19)	171.5
N2—H2A···O2 ⁱⁱ	0.88	2.19	2.914 (2)	139.6

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

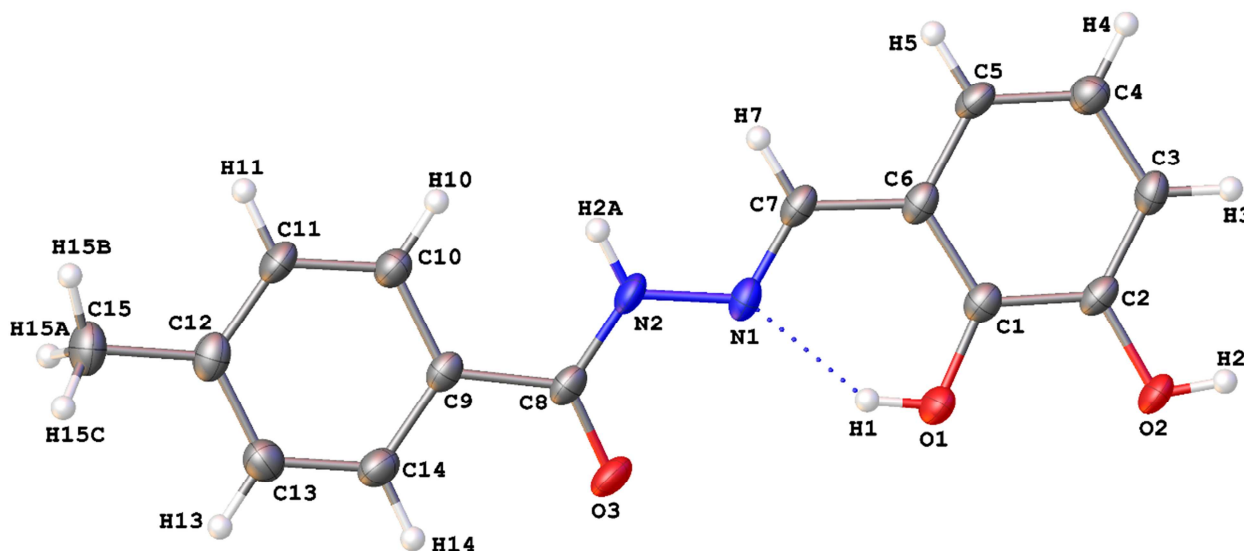


Figure 2. ORTEP plot (30% probability ellipsoids) showing the structure of (I).

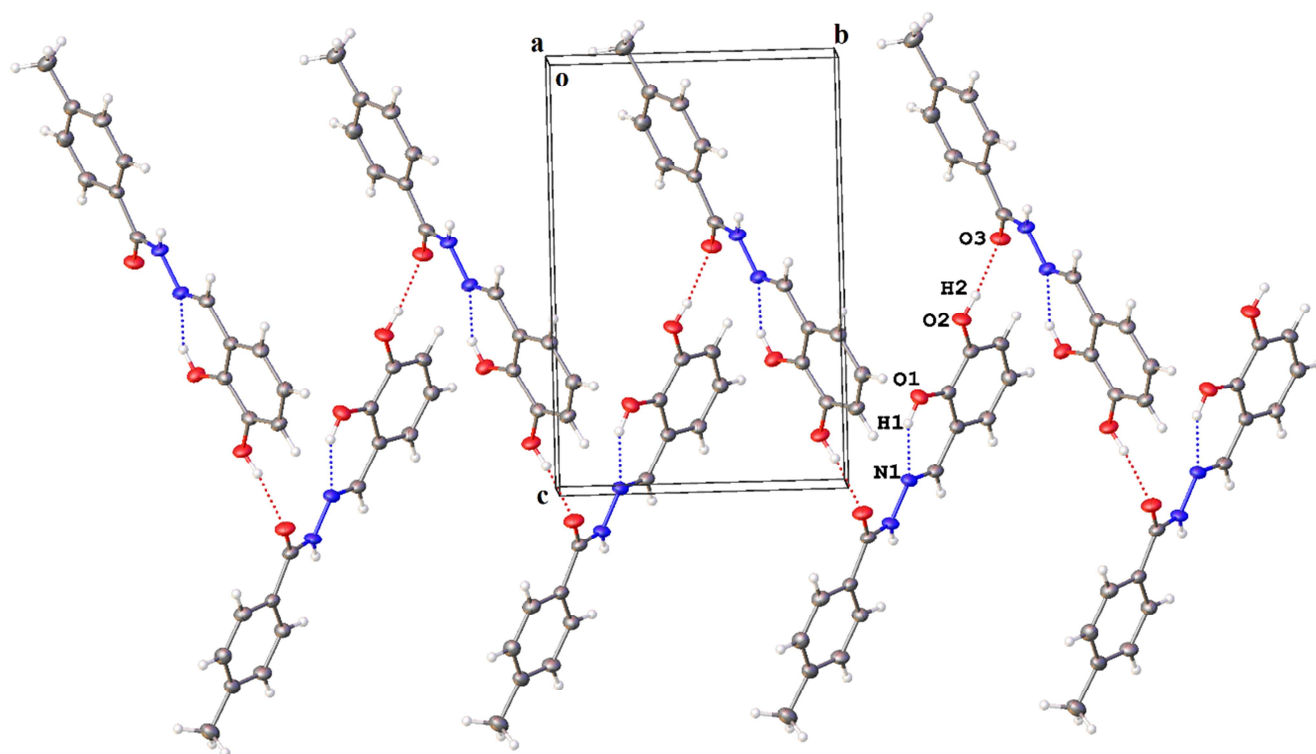


Figure 3. Chain of the ligand formed by hydrogen bonding.

4. Conclusion

The hydrazone derivative *N'*-(2,3-dihydroxybenzylidene)-4-methylbenzohydrazide (I) was prepared from *p*-toluic acid hydrazide and 2,3-dihydroxybenzaldehyde. The structure of (I) was confirmed by elemental analysis and spectroscopic techniques (FT-IR, ^1H and ^{13}C NMR). The molecular structure of (I) was determined by X-ray diffraction technique. The X-ray structure reveals that the carbohydrazide moiety and the two benzene ring are almost coplanar. This compound present several donor sites such as nitrogen and oxygen atoms. It can be used as Schiff base ligand for the synthesis of coordination compounds.

Supporting Information

CCDC-2253533 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>.

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