

Synthesis, spectroscopic characterization, and crystal structures of Schiff bases derived from nicotinic hydrazide

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Abstract: Two Schiff bases were prepared from nicotinic hydrazide and *o*-vanillin (H_2L^1) (1) or salicylaldehyde (H_2L^2) (3) and were characterized by elemental analysis, 1H and ^{13}C NMR and IR spectroscopic studies. Compound were protonated in ethanol-nitric acid solution and gave compound $[(H_3L^1)(NO_3)]$ (2). The compound (2) crystallizes in the monoclinic space group $P2_1/c$ with unit cell dimensions $a = 7.2005$ (3) Å, $b = 8.8914$ (4) Å, $c = 22.6392$ (9) Å, $\beta = 96.753$ (2)°, $V = 1439.36$ (11) Å³, $Z = 4$, $R_1 = 0.059$ and $wR_2 = 0.037$. The compound (3) crystallizes in the monoclinic space group $P2_1/c$ with unit cell dimensions $a = 26.615$ (4) Å, $b = 11.3366$ (14) Å, $c = 8.1903$ (9) Å, $\beta = 98.814$ (3)°, $V = 2442.0$ (5) Å³, $Z = 8$, $R_1 = 0.085$ and $wR_2 = 0.141$. Both molecules present inter and intramolecular hydrogen bonds which consolidate the structures in three-dimensional networks.

Keywords: Nicotinic hydrazide, *o*-vanillin, 2-hydroxyacetophenone

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I. Introduction

Schiff bases are ligands with multiple coordination sites and are usually prepared from a keto precursor and an amine. Those obtained from nicotinic hydrazide have been widely reported in the literature by researchers interested in coordination chemistry [1-3]. The use of these ligands makes it possible to prepare coordination compounds with very diverse structures [4]. Nicotinic hydrazide derivatives have been used for the synthesis of products with important biological properties [5,6]. Nicotinic hydrazide has been used as a corrosion inhibiting agent [7] but also as an electrochemical sensor [8]. A wide variety of heterocyclic molecules with good medicinal properties are obtained from nicotinic precursors [9-11]. They are used as anticonvulsant [12,13], antituberculosis [6], anticancer [14] and antimicrobial [2] agents. Schiff bases which are synthesized from nicotinic hydrazide are used for the preparation of metal transition [15,16] and lanthanides [17-20] complexes. It is in this perspective that we have studied these types of ligands and reported their metal transition [21] and lanthanides [22] complexes. In the pursuit of our work in this field, we obtained the ligands (H_2L^1) and (H_2L^2) whose synthesis and spectroscopic characterization are the subject of this present study. We also report the X-ray diffraction structures of the two compounds.

II. Experimental section

2.1. Starting materials and Instrumentations

All chemicals and solvents were of analytical reagent grade and were used directly without further purification. *O*-vanillin, 2-hydroxyacetophenone, nicotinic hydrazide are obtained from Sigma-Aldrich. 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. NMR spectra were recorded in DMSO- d_6 solution with a Bruker 500 MHz spectrometer at room temperature using TMS as an internal reference.

2.2. Synthesis of the ligand N'-(2-hydroxy-3-methoxybenzylidene)nicotinohydrazide (H_2L^1) (1)

To a solution of *o*-vanillin (3.0 g, 19.7 mmol) in 20 mL of ethanol, was added dropwise a solution of nicotinic hydrazide (2.704 g, 19.7 mmol) in 20 mL of ethanol. The mixture was stirred under reflux for 2 hours. On cooling, the yellow precipitate was isolated by filtration and successively washed with 2 x 10 mL of ethanol before dried in air. Yield: 81.2%. M.P. 95 °C. Analytical for $C_{14}H_{13}N_3O_3$: Calc (found) %C = 61.99 (62.03); %H = 4.83 (4.80); %N = 15.49 (15.44). IRv (cm^{-1}): 3333 (NH); 3200 (OH); 1654 (C=O); 1603 (C=N); 1570; 1474; 1420 (C=C)_{arom}; 1300 (C-O phenolic); 1242 (C-O_{ether}); 1075 (N-N); 890; 833; 777; 721; 706 (CH_{Ar}). UV-vis

(Solution, DMF, nm): 273; 337; 372. NMR ¹H (DMSO-d₆, δ(ppm)): 12.21 (s, 1H, OH); 10.78 (s, 1H, NH); 9.09 (s, 1H, HC=N); [6.82 – 8.78] (m, 7H, H_{Ar}); 3.82 (s, 3H, -OCH₃). NMR ¹³C (DMSO-d₆, δ(ppm)): 161.33 (C=O); 152.38 (C=N); 148.56 (C-O_{phenolic}); 148.24 (C-O_{ether}); [147.88; 147.04; 135.40; 128.57; 120.60; 120.39; 119.03; 113.75] (C_{arom}.); 55.74 (OCH₃).

2.3. Synthesis of (H₃L¹)(NO₃) (2)

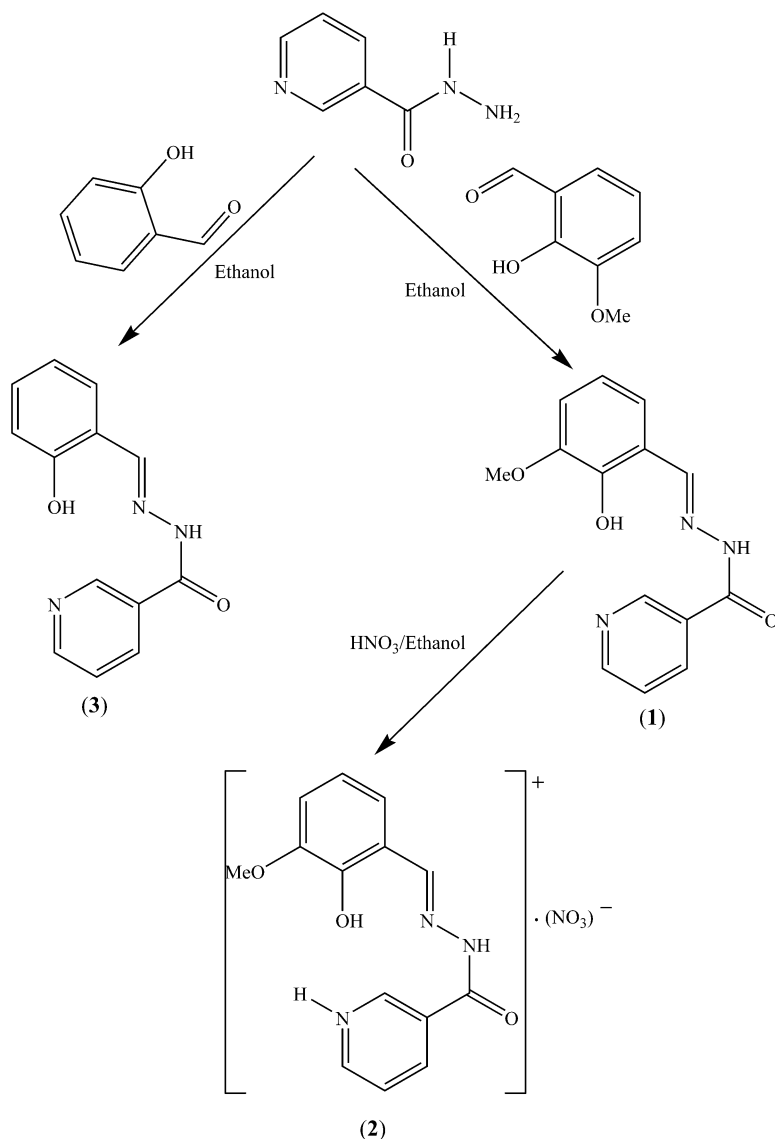
Compound H₂L¹ was dissolved in a mixture of ethanol and nitric acid. After one hour of stirring, the mixture was evaporated to dryness. The yellow solid obtained is then recrystallized in DMSO solution which was left at room temperature. Two weeks later, yellow crystals suitable for X-ray diffraction were collected.

2.4. Synthesis of the ligand N'-(1-(2-hydroxyphenyl)ethylidene)nicotinohydrazide (H₂L²) (3)

To a suspension of nicotinic hydrazide (3 g, 21.8 mmol) in 40 mL of methanol, was added a 2-hydroxyacetophenone (2.98 g, 21.8 mmol). The mixture was heated under reflux for 24 hours. The resulting solution was cooled at room temperature. The yellow precipitate which appeared was recovered by filtration and washed with methanol before dried in air. Yield: 94% and the M.P. 188°C. Analytical for C₁₄H₁₃N₃O₂: Calc (found) %C = 65.87 (65.85); %H = 5.13 (5.15); %N = 16.46 (16.43). IR ν(cm⁻¹): 3215 (O-H); 3008 (N-H); 1646 (C=O); 1610 (C=N). NMR ¹H (DMSO-d₆, δ(ppm)): 13.26 (s, 1H, NH) ; 11.53 (s, 1H, OH) ; 9.09-9.08 (1H, H_{arom}) ; 8.79-8.78 (m, 1H, H_{arom}) ; 8.29-8.27 (m, 1H, H_{arom}) ; 7.66-7.57 (m, 2H, H_{arom}) ; 7.33-7.30 (m, 1H, H_{arom}) ; 6.93-6.89 (m, 2H, H_{arom}) ; 2.49 (3H, CH₃). NMR ¹³C (DMSO-d₆, δ(ppm)): 162.03 ; 157.79 ; 157.64 ; 151.48 ; 148.06 ; 135.04 ; 130.51 ; 127.93 ; 127.72 ; 122.58; 118.35; 117.64; 116.41.

2.5. Crystal structure determination

Crystals suitable for single-crystal X-ray diffraction, of the reported compounds (2) and (3), were grown by slow evaporation of EtOH solution of the complexes. Details of the crystal structure solution and refinement are given in Table 1. Diffraction data were collected using an Bruker APEX-II CCD diffractometer with graphite monochromatized MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL [23]. The structures were solved by direct methods which revealed the position of all non-hydrogen atoms. All the structures were refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms [24]. The hydrogen atoms of water molecules and NH groups were located in the Fourier difference maps and refined. Others H atoms (CH and CH₃ groups) were geometrically optimized and refined as riding model by AFIX instructions. Molecular graphics were generated using ORTEP [25].



Scheme 1. The process of preparation of the compounds.

Table-1. Crystallographic data and refinement parameter for the compounds (2) and (3).

Chemical formula	C ₁₄ H ₁₄ N ₄ O ₆	C ₁₄ H ₁₃ N ₃ O ₂
<i>M_r</i>	334.29	255.27
Crystal shape/color	Prismatic, yellow	Prismatic, yellow
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Crystal size (mm)	0.20 × 0.15 × 0.15	0.14 × 0.12 × 0.12
<i>a</i> (Å)	7.2005 (3)	26.615 (4)
<i>b</i> (Å)	8.8914 (4)	11.3366 (14)
<i>c</i> (Å)	22.6392 (9)	8.1903 (9)
β (°)	96.753 (2)	98.814 (3)
<i>V</i> (Å ³)	1439.36 (11)	2442.0 (5)
<i>Z</i>	4	8
<i>D</i> _{calc} (g.cm ⁻³)	1.543	1.389
λ (MoKα) (Å)	0.71073	0.71073

T (K)	173(2)	173(2)
μ (mm^{-1})	0.12	0.02
Index ranges	$-9 \leq h \leq 9, -11 \leq k \leq 11, -29 \leq l \leq 29$	$-35 \leq h \leq 34, -14 \leq k \leq 14, -7 \leq l \leq 10$
$F(000)$	696	268
θ range ($^\circ$)	1.8-28.1	2.48-27.57
No. of measured reflections	74119	17689
No. of independent reflections	3311	5829
No. of observed [$I > 2\sigma(I)$] reflections	2652	3140
R_{int}	0.059	0.085
$R[F^2 > 2\sigma(F^2)]$	0.037	0.141
$wR(F^2)$	0.107	0.384
Goodness-of-fit (Gof) on F^2	1.05	1.18
No. of parameters	219	348
No. of restraints	0	0
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ ($\text{e } \text{\AA}^{-3}$)	0.29, -0.24	0.88, -0.45

III. Result and discussion

3.1. General study

The mass spectrum of H_2L^1 shows a base peak located at $m/z = 272.10$ corresponding to the molar mass of the molecular ion (M+1) of the product. This result makes it possible to determine the molecular formula ($\text{C}_{14}\text{H}_{13}\text{N}_3\text{O}_3$) of the ligand. The infrared spectrum of the H_2L^1 ligand shows a thin band pointed at 3543 cm^{-1} attributed to the vibration of the N-H bond [26]. The two bands pointed at 3200 cm^{-1} and 3068 cm^{-1} are respectively attributed to the valence vibrations of the hydroxyl group (OH) and the C-H bonds of the aromatic ring [27]. The valence vibration of the carbonyl (C=O) is pointed at 1654 cm^{-1} and that of the imine function at 1603 cm^{-1} [28]. Between 1570 and 1420 cm^{-1} we identify the vibrations of the C=N and C=C bonds of the two aromatic nuclei. A strong band at 1366 cm^{-1} is attributed to the vibration of the C-N bond. The presence of this band and that of $\nu(\text{C}=\text{O})$ indicates that the ligand is in its amide form. The bands pointed at 1300 and 1242 cm^{-1} are respectively attributed to the vibrations of the phenolic (C-O) bond and that of the ether. The vibration of the N-N bond is located at 1075 cm^{-1} . Towards the low frequencies, we locate the vibrations of deformation of the aromatic C-H bonds between 890 and 706 cm^{-1} .

Nuclear Magnetic Resonance spectral analyzes of the ^1H proton and the ^{13}C carbon are carried out using solutions of dimethylsulfoxide. The ^1H NMR spectrum reveals a signal in the form of a singlet at 12.21 ppm and integrating a proton attributed to the hydroxyl proton OH. Two other singlets at 10.78 and 9.09 are identified, each integrating a proton which are attributed respectively to NH, to the proton of the imine function (HC=N). The seven protons of the two aromatic nuclei are identified as multiplets in the range ($6.82 - 8.78$) ppm. And finally, the singlet signal located at 3.82 ppm and integrating three protons is attributed to those of methoxy (OCH_3). The ^{13}C NMR spectrum reveals a signal at 161.33 ppm attributed to the carbon atom of the carbonyl function (C=O). The carbon signal of the imine function (C=N) is identified at 152.38 ppm. As for the aromatic carbon atoms, they have their chemical shifts between 148.52 and 113.75 . The signal at 55.74 ppm is attributed to the carbon atom of the methoxy group (OCH_3).

The infrared spectrum of the H_2L^2 ligand shows characteristic absorption bands at 3215 and 3008 cm^{-1} which are attributed respectively to the O-H and N-H groups. The bands pointed at 1646 and 1610 cm^{-1} are attributed respectively to the C=O and C=N functions, [28] while the vibrations of the C=C bonds of the aromatics are identified between 1584 and 1498 cm^{-1} . The vibration of the C-O_{phenolic} function is identified between 1279 and 1242 cm^{-1} . The vibration of the N-N bond of the hydrazone is peaked at 1020 cm^{-1} . In addition, we note the presence of deformation vibrations of the C-H_{aromatic} bonds between 833 and 730 cm^{-1} . The nuclear magnetic resonance of the ^1H and ^{13}C nuclei of the organic ligand is recorded in DMSO- d_6 . The broad signals at 13.26 and 11.53 ppm are attributed to NH and OH from the hydrazone and the phenol group, respectively. Aromatic protons have their chemical shifts between 9.09 and 6.89 ppm. The chemical shift peaked at 2.49 ppm is attributed to the methyl group (CH_3). For ^{13}C NMR, shifts at 162.03 ppm are noted for the carbonyl function (C=O). The carbon atom of the imine function (C=N) is pointed at 157.79 ppm. The chemical shift at 157.64 ppm is attributed to the ipso carbon of phenol (C-OH). The chemical shifts between 151.48 and

148.06 ppm are attributed to the carbon atoms of the pyridine ring. The signals of the carbon atoms of the benzene ring are between 116.41-151.48 ppm. Note the signal from the methyl group (CH₃-) at 13.26 ppm.

3.2. Description of the crystal structure of compound 2

The molecular structure of the compounds [(H₃L)¹·(NO₃)] with the atomic labelling is shown in Figure 1. The compound crystallizes in the monoclinic system with P2₁/c group. The asymmetric unit contains one protonated H₂L cation H₃L⁺ and one NO₃⁻ anion. The cation adopts an *E* configuration with respect to the C7=N1 bond. The benzene ring and the pyridine ring are almost coplanar with dihedral angle of 5.75° between their mean planes. The carbohydrazide moiety O2=C9—N2—N1=C7 is almost coplanar with the C1/C6 aromatic ring dihedral angle of 2.80° and C11/N-C14 pyridine ring with dihedral angle of 8.83°. The C9=O2 bond length value of 1.218(2) Å which is compatible with double-bond character shows that the compound remains in its amid form. This form is consolidated by the bond length values of 1.357(2) Å and 1.375(2) Å for C9—N2 and N1—N2 respectively which has single-bond character and the bond length value of 1.282(2) Å for C7=N1 which has double-bond character (Table 2) [29]. The O2 and N1 atoms are in a *syn* conformation with respect to the C9=N2 bond with a torsion angle of -0.9(2)° [O2—C9—N2—N1].

The molecule forms intramolecular hydrogen bonds between the N atom of the hydrazine moiety and the hydroxy H atom producing *S*(6) ring (O1—H1···N1, Table 3). Other intramolecular hydrogen bonds are formed in the structure. The protonated nitrogen atom of the pyridine ring form hydrogen with oxygen atoms of the nitrate counter anion (N3—H3···O4 and N3—H3···O5, Table 1). Hydrogen bond producing *S*(5) ring is also formed between the carbonyl O atoms as acceptor and H—C atom of the pyridine ring (C14—H14···O2) (Table 3). The three-dimensional structure is consolidated by intermolecular bond : N2—H2···O6, C7—H7···O6 and C11—H11···O4 (Figure 2).

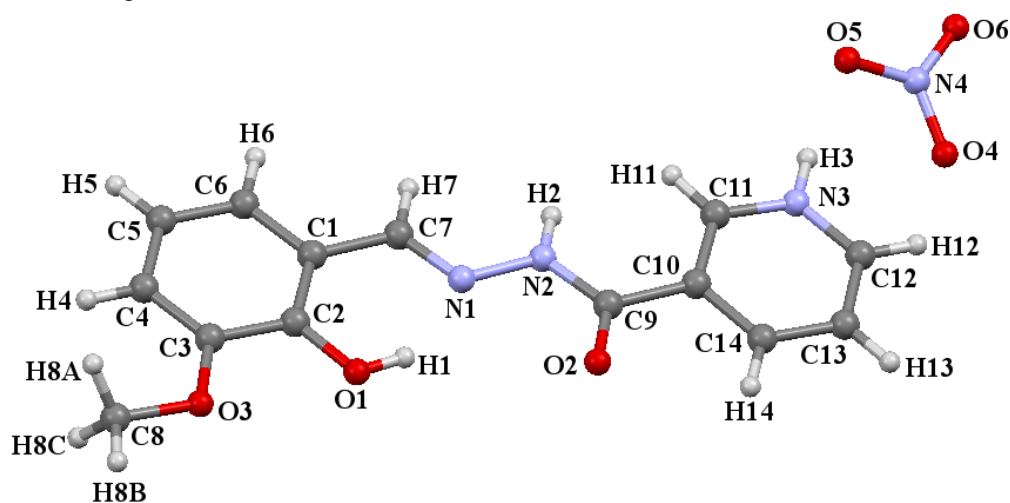


Fig 1. Crystal structure of the of (2).

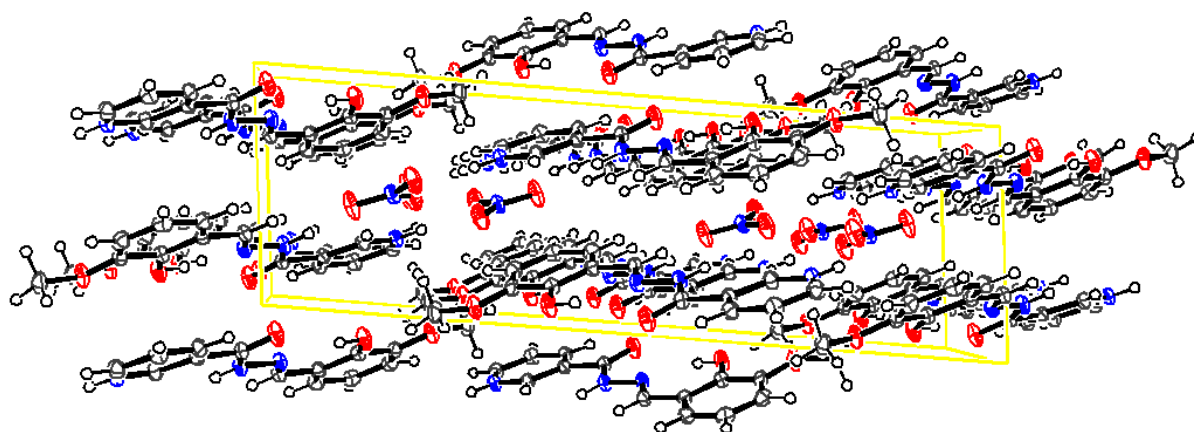


Fig 2. Packing diagram of (2) viewed along the *ab* plane.

Table-2. Selected bond distances [Å] and angles [deg] for the compounds (2) and (3).

(2)		(3)	
N3—C12	1.3372 (18)	C7—N1	1.310 (8)
C9—O2	1.2178 (15)	N1—N2	1.374 (7)
C9—N2	1.3566 (17)	N2—C9	1.338 (8)
C2—O1	1.3510 (16)	C9—O2	1.249 (8)
C3—O3	1.3654 (16)	C21—N4	1.296 (8)
O3—C8	1.4323 (16)	N4—N5	1.382 (7)
N3—C11	1.3415(17)	N5—C23	1.343 (8)
N1—N2	1.3755(15)	C23—O4	1.251 (8)
C12—N3—C11	122.88(11)	N1—C7—C8	123.7 (5)
C11—C10—C9	124.30(12)	C7—N1—N2	117.5 (5)
O2—C9—N2	122.96 (12)	C9—N2—N1	119.9 (5)
O2—C9—C10	120.35 (12)	O2—C9—N2	123.4 (6)
N2—C9—C10	116.68 (11)	N4—C21—C22	123.9 (5)
C9—N2—N1	116.71 (10)	C21—N4—N5	116.5 (5)
C7—N1—N2	118.12 (11)	C23—N5—N4	119.4 (5)
N1—C7—C1	119.64 (11)	O4—C23—N5	124.4 (6)

Table-3. Hydrogen-bond geometry (Å, °) of compounds (2) and (3).

D—H...A	D—H	H...A	D...A	D—H...A
(2)				
O1—H1...N1	0.84	1.87	2.6035(15)	145
N2—H2...O6 ⁱ	0.88	2.08	2.9075(16)	157
N3—H3...O4 ⁱⁱ	0.88	2.54	3.0788(16)	120
N3—H3...O5 ⁱⁱ	0.88	1.91	2.7836(16)	171
C7—H7...O6 ⁱ	0.95	2.41	3.2210(18)	143
C11—H11...O4 ⁱ	0.95	2.26	3.0552(18)	141
C12—H12...O4 ⁱⁱ	0.95	2.40	3.0193(18)	123
C14—H14...O2 ⁱⁱⁱ	0.95	2.54	3.1756(17)	125
Symmetry codes : i = x, 1/2-y, -1/2+z ; ii = 1-x, -y, -z ; iii = 2-x, -1-y, -z.				
(3)				
D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N1	0.84	1.80	2.529(7)	144
N2—H2...O2 ⁱ	0.88	2.37	2.987(7)	127
O3—H3A...N4	0.84	1.79	2.527(7)	146
N5—H5A...O4 ⁱⁱ	0.88	2.29	2.958(7)	132
C6—H6...O1 ⁱⁱⁱ	0.95	2.48	3.347(9)	152
C20—H20...O3 ^{iv}	0.95	2.49	3.363(9)	152
Symmetry codes : i = x, 3/2-y, 1/2+z ; ii = x, 1/2-y, 1/2+z ; iii = -x, -1/2+y, 3/2-z ; iv = 1-x, -1/2+y, 1/2-z.				

3.3. Description of the crystal structure of compound 3

The molecular structure of the compounds H_2L^2 with the atomic labelling is shown in Figure 3. The asymmetric unit contains two neutral molecules of H_2L . The compound crystallizes in the monoclinic system with $P2_1/c$ group. The pyridine rings of the two molecules are not coplanar with dihedral angle of 51.85° and the two phenyl rings are more staggered with dihedral angle of 75.39° . In each molecule the carbonyl oxygen atom and the azomethine nitrogen atom are in *syn* conformation with respect of the corresponding C—N bond [O2 and N1 are in *syn* conformation with respect of C9—N2 and O4 and N4 are in *syn* conformation with respect of C23—N5] with torsion angles values of $-1.2(9)^\circ$ [O2—C9—N2—N1] and $2.6(9)^\circ$ [O4—C23—N5—N4]. The hydrazone moieties are almost planar, with a maximum deviation from least-squares planes of $0.0251(4)$ Å for N1 [O2/C9/N2/N1/C7, rms 0.0145 Å] and -0.0260 Å for N4 [O4/C23/N5/N4/C21, rms 0.0201 Å]. The dihedral angle between the hydrazone moieties and their corresponding phenyl rings are $4.381(5)^\circ$ [O2/C9/N2/N1/C7 and C1-C6] and $5.078(5)^\circ$ [O4/C23/N5/N4/C21 and C15-C20]. The O2—C9 [1.249 (8) Å] and O4—C23 [1.251 (8) Å] distances indicate that these correspond to double bonds and both molecules remain in their amide

forms as observed in the literature [30]. Additionally, the C7—N1 [1.310 (8) Å] and C23—N5 [1.343 (8) Å] bond lengths are in the normal range observed for a single C—N bond [31] (Table 2). The molecules form intramolecular hydrogen bonds between the N atom of the hydrazine moiety and the hydroxy H atom producing *S*(6) rings (O1—H1···N1 and O3—H3A···N4) (Table 3). The three-dimensional structure is consolidated by intermolecular bond : N2—H2···O2, N5—H5A···O4, C6—H6···O1 and C20—H20···O3 (Figure 4).

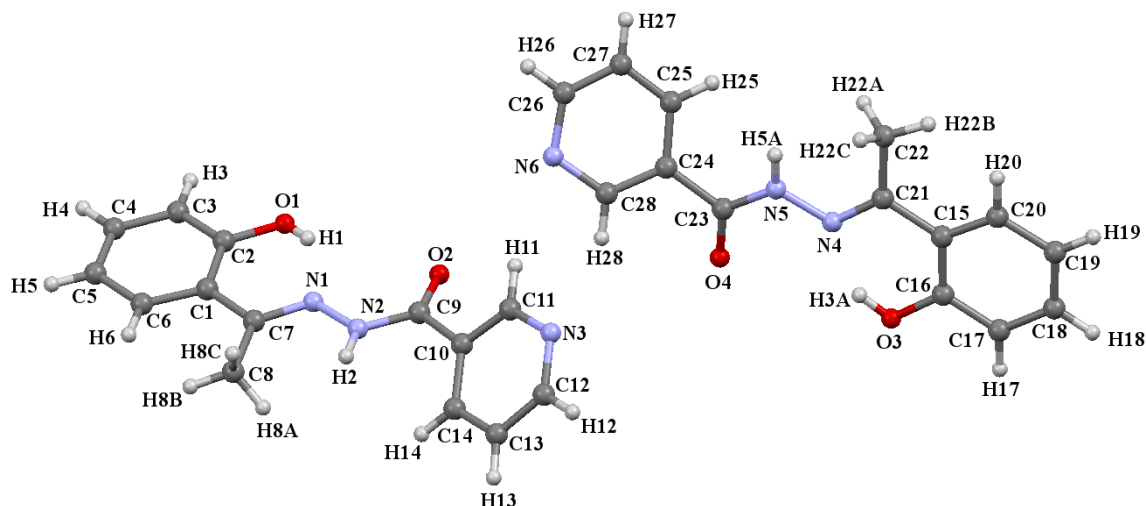


Fig 3. Crystal structure of the of (3).

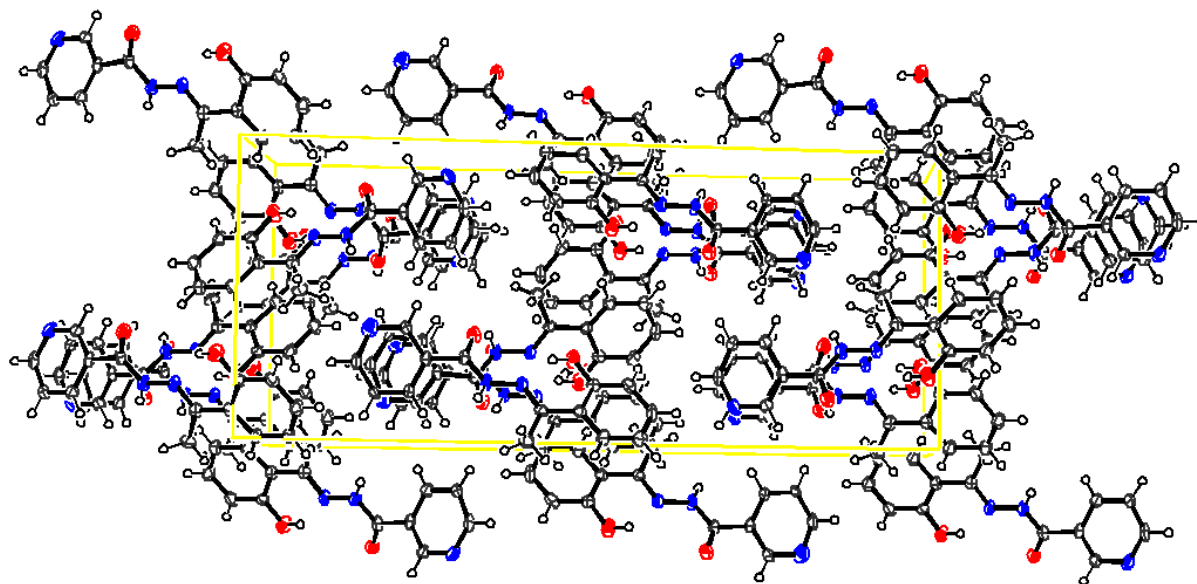


Fig 4. Packing diagram of (3) viewed along the *ab* plane.

IV. Conclusion

The nicotinic hydrazide derivatives *N'*-(2-hydroxy-3-methoxybenzylidene)nicotinothiazide (H_2L^1) (1) and *N'*-(1-(2-hydroxyphenyl)ethylidene)nicotinothiazide (H_2L^2) (3) were prepared by condensation reaction. The solution of in nitric acid-ethanol gave the protonated compound [$(H_3L^1) \cdot (NO_3)$] (2). The structures of the two derivatives (1) and (3) were confirmed by elemental analysis and spectroscopic techniques (FT-IR, 1H and ^{13}C NMR). The molecular structure of the two molecules (2) and (3) are determined by X-ray diffraction technique.

V. Supporting information

CCDC-2143055 and 2143054 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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