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Structural and spectroscopic characteristics of aroylhydrazones derived from nicotinic acid hydrazide

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Abstract

Aroylhydrazones derived from salicylaldehyde, *o*-vanillin and nicotinic acid hydrazide have been synthesized and characterized on the basis of NMR, IR and UV/Vis spectral data. The crystal and molecular structure of *N′*-salicylidene-3-pyridinecarbohydrazide has been determined by X-ray diffraction.

Keywords: *N′*-salicylidene-3-pyridinecarbohydrazide; *N′*-(2-hydroxy-3-methoxyphenylmethylidene)-3-pyridinecarbohydrazide; NMR; UV/Vis; IR spectroscopies; X-ray structure analysis

1. Introduction

The chemistry of aroylhydrazones has been intensively investigated in recent years. The reasons are manifold: the coordination ability of compounds from this group to chelate metal ions, particularly transition and lanthanide ions [1,2], their biological activity [1,3] and promising properties for analytical applications [1,4,5]. Aroylhydrazones containing pyridine ring have attracted special attention as analytical reagents, because of high sensitivity and selectivity [4,6–10]. As part of our study on analytical application of hydrazones and Schiff bases [11], we prepared and characterized two new aroylhydrazones from nicotinic acid hydrazide (Scheme 1 : *N′*-salicylidene-3-pyridinecarbohydrazide (compound **1**) and *N′*-(2-hydroxy-3-methoxyphenylmethylidene)-3-pyridinecarbohydrazide (compound **2**)). The characterization included UV/Vis, IR and NMR spectroscopic data, as well as X-ray structure analysis.

2. Experimental

2.1. Preparation of compounds *1* and *2*

Salicylaldehyde was obtained from Merck, *o*-vanillin and nicotinic acid hydrazide were purchased from Fluka. *N′*-salicylidene-3-pyridinecarbohydrazide (compound **1**) was prepared by mixing equimolar amounts of nicotinic acid hydrazide dissolved in absolute ethanol with salicylaldehyde. Triethylamine was added as a catalyst. The reaction mixture was refluxed for 1 h on a water bath, then evaporated to half of a volume, and finally, cooled to 5°C. The separated pale yellow needles of compound **1** were recrystallized from absolute ethanol (m.p. 191°C, elemental analysis

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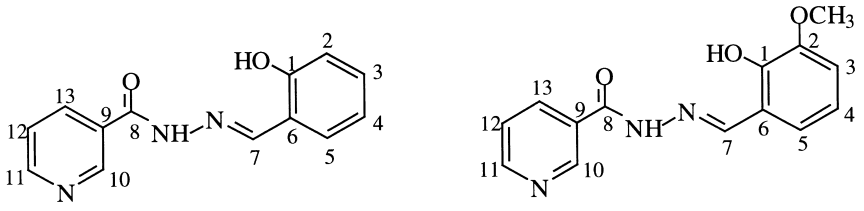


compound **1** compound **2**

Scheme 1.

for $C_{13}H_{11}N_3O_2 \cdot H_2O$, calculated: C 60.22, H 5.05, N 16.21%; found: C 59.46, H 5.01, N 15.89%).

The same procedure was used for preparation of *N′*-(2-hydroxy-3-methoxyphenylmethylidene)-3-pyridinecarbohydrazide (compound **2**) from *o*-vanillin and nicotinic acid hydrazide (m.p. 99–101°C, elemental analysis for $C_{14}H_{13}N_3O_3 \cdot H_2O$: calculated: C 58.13, H 5.23, N 14.52%; found C 57.70, H 5.15, N 14.25%).

2.2. Spectroscopy

UV/Vis spectra were obtained using Varian Cary 3 spectrometer with a 2 nm slit width. IR spectra of KBr discs were recorded with a Perkin Elmer 783 spectrometer at 2 cm^{-1} resolution. NMR spectra were recorded on a Varian XL gemini 300 spectrometer using tetramethylsilane as internal standard. The digital resolution was 0.01 ppm.

2.3. X-ray structure analysis of *1*

X-ray data were collected at room temperature on an Enraf-Nonius CAD4 diffractometer using graphite-monochromated CuKα radiation (λ = 1.54184 Å). Data collection and unit cell determination were controlled using CAD-4 EXPRESS program [12] with MicroVAX II[e] computer. Crystallographic data and details of data collection and refinement are listed in Table 1. Lorentz and polarization effects were corrected using the HELENA data reduction program [13]. Standard reflections were monitored every 60 min indicating no significant change in intensities. An empirical psi-scan absorption correction was applied using the PLATON

Table 1
Crystal data and summary of experimental details and refinement of **1**

Empirical formula	$C_{13}H_{11}N_3O_2$, H_2O
Formula weight	259.26
Crystal system	Triclinic
Space group	$P\bar{1}$
a (Å)	7.3237(2)
b (Å)	8.7568(3)
c (Å)	10.1297(4)
α (°)	94.492(3)
β (°)	103.808(3)
γ (°)	95.962(4)
V (Å^3)	623.87(4)
Z	2
Density (calculated) (g cm^{-3})	1.380
μ(CuKα) (mm^{-1})	0.84
F(000)	272
Crystal size (mm)	0.1 × 0.1 × 0.3
Temperature (K)	293
No. of reflections for cell determination	23
Θ range for cell determination (°)	41.1 − 45.5
Θ range for data collection (°)	4.52 − 74.25
Scan type	$\omega/2\Theta$
hkl range	−9, 0; −10, 10; −12, 12
No. of measured reflections	2749
No. of symmetrical independent reflections	2541
No. of observed reflections ($I > 2\sigma(I)$)	1950
No. of parameters	225
R	0.0339
wR[a] (all reflections)	0.1093
S	1.084
Extinction coefficient	0.0145(17)
Max. and av. shift/error	0.001, 0.000
Min. and max. resd. dens. (*e* Å^{-3})	0.215, −0.159

[a] $w = 1/[\sigma^2(F_o^2) + (0.0656 \quad P)^2 + 0.0148 \quad P]$ where $P = (F_o^2 + 2F_c^2)/3$.

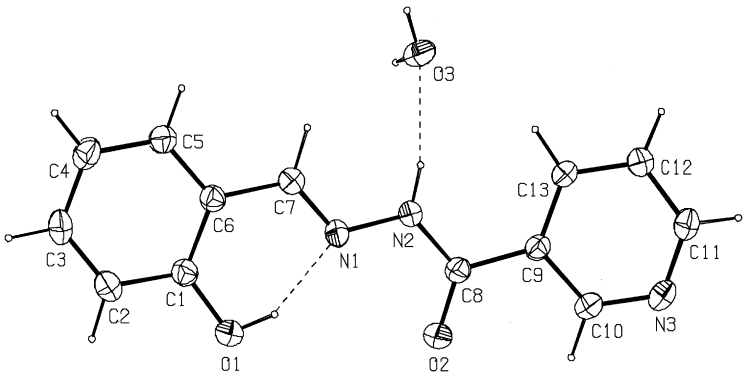


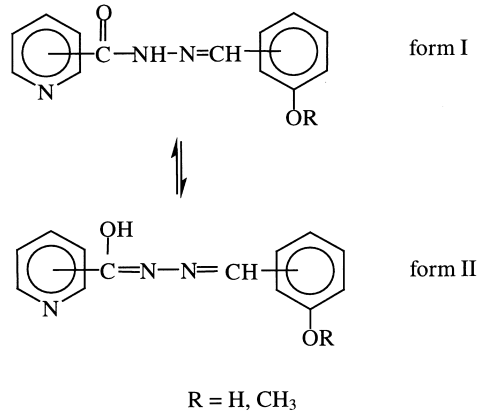
Fig. 1. ORTEPII [17] drawing of *N′*-salicylidene-3-pyridinecarbohydrazide monohydrate showing atom numbering. Intramolecular hydrogen bond O1–H1(O)···N1 and intermolecular hydrogen bond N2–H2(N)···O3 are shown. The thermal ellipsoids are at the 30% probability level.

program [14]. The structure was solved by direct methods using the SIR97 program [15], then refined with the SHELXL97 program [16]. Full-matrix least-squares refinement based on F^2 converged at $R = \Sigma||F_o| - |F_c|\,|/\Sigma|F_o| = 0.0339$ for 1950 reflections with $I > 2\sigma(I)$ and $wR = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2} = 0.1093$ for all 2541 symmetry independent reflections (Table 1). All hydrogen atoms were located from a difference Fourier map. Data reduction, structure solution and refinement were performed on a Silicon Graphics Indigo 2 workstation. For ORTEPII drawing [17] of the molecule (Fig. 1) and plots of crystal packing (Fig. 2) the PLATON program [14] was used. Scattering factors for atoms were those from International Tables for Crystallography, Vol C [18]. The atomic coordinates are given in Table 2.

Crystallographic data have been deposited with the *Cambridge Crystallographic Data Centre*, supplementary publication No. CCDC-143724. Copies of the data may be obtained free of charge on application to CSD, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +441223/336033, e-mail: deposit@ccdc.cam.ac.uk).

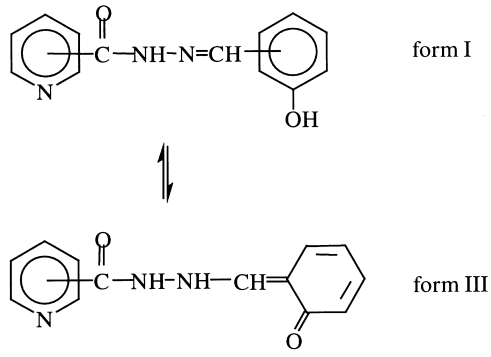
3. Results and discussion

It was reported [19–21] that hydrazones derived from pyridinecarbohydrazide and salicylaldehyde or 2-methoxybenzaldehyde can exist as tautomeric enolimines, according to following equilibrium:



R = H, CH_3

When R = H, the tautomeric equilibrium can also involve aldehyde moiety:



The tautomeric interconversion to forms II and III was not observed in compounds **1** and **2** which are present in form I in crystalline state and in solution.

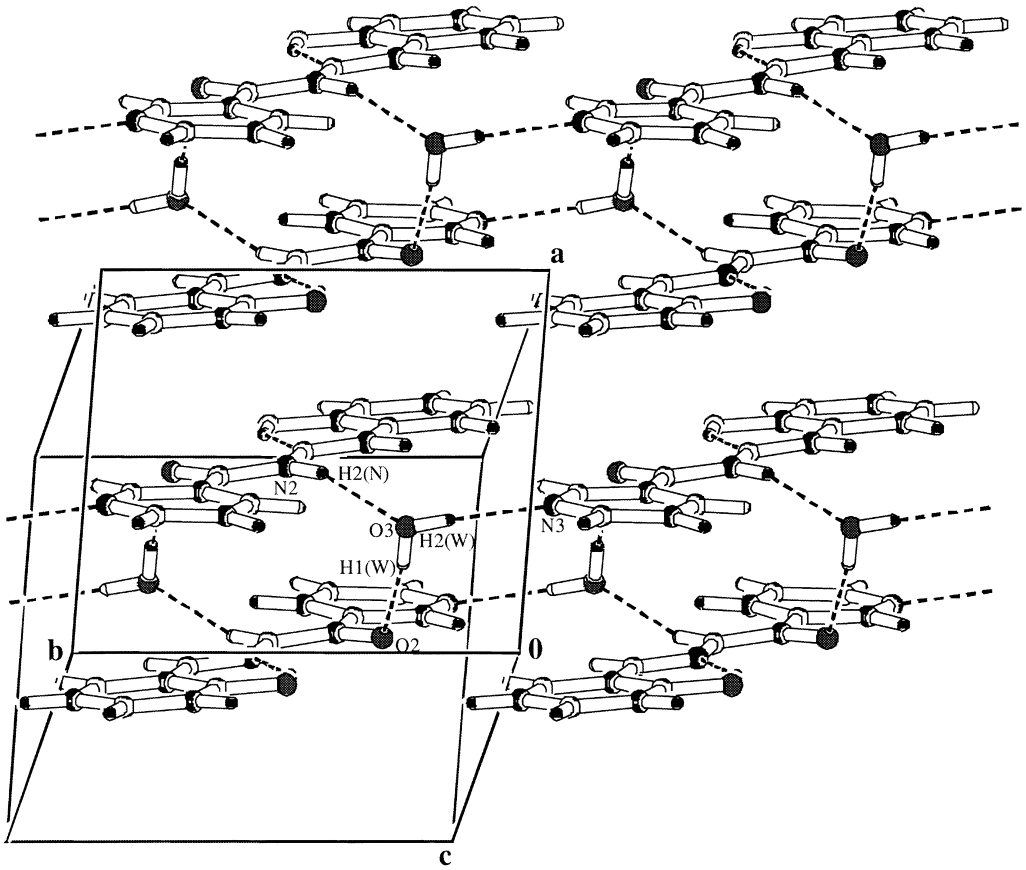


Fig. 2. Packing of molecules of N'-salicylidene-3-pyridinecarbohydrazide monohydrate. Three intermolecular hydrogen bonds of a ladder pattern are shown. The molecules are connected by $\pi\cdots\pi$ interactions in direction of *a*.

*3.1. Molecular and crystal structure of **1***

The solid state structure of compound **1** determined by X-ray diffraction is shown in Fig. 1. Selected bond lengths and angles are listed in Table 3. The crystal packing of the molecule is shown in Fig. 2. The hydrogen bond geometry is given in Table 4.

The bond lengths are in accordance with the structure of tautomeric form I. The bonds C8–O2 of 1.222(1) Å and C7–N1 of 1.276(2) Å have a double bond character, whereas bonds C8–N2 of 1.354(1) Å, C7–C6 of 1.455(1) Å and C1–O1 of 1.354(1) Å are typical for a single bond. Bond lengths observed in the *N*-salicylidene fragment are in agreement with the values found in analogues compounds [22–28]. The weights of the possible canonical structures of *N*-salicylidene fragments using the HOSE model in relation to observed changes in geometry of X-ray determined structures were discussed in the literature [29]. Our approach is based on calculation of difference Fourier densities using refined parameters of compound **1** with removed hydrogens on hydroxyl oxygen (O1), then on amino nitrogen (N2). In both cases, the calculations reveal the position of highest peak in accordance with the tautomeric form I and with no peak at all (among 20 highest) in the vicinity of imino nitrogen (N1) or carbonyl oxygen (O2) (required for tautomeric forms II or III). Thus, the experimental evidences are in favour of the tautomeric form I in the solid state. The Cambridge Structural Database (CSD) search [30] on a hydrazone moiety in tautomeric form I (–CO–NH–N=CH–) revealed 65 fragments in 57 structures and the geometries observed are in agreement with data for

Table 2
Final atomic coordinates and equivalent isotropic displacement parameters of the non-hydrogen atoms of **1**

Atom	*x*	*y*	*z*	U_{eq} (Å^2)[a]
O1	0.0169(2)	0.4935(1)	0.20368(9)	0.0563(3)
O2	0.2717(2)	0.2537(1)	0.47452(9)	0.0572(3)
N1	0.2121(1)	0.5450(1)	0.45832(9)	0.0405(3)
N2	0.3081(1)	0.4963(1)	0.5784(1)	0.0407(3)
N3	0.5284(2)	0.0738(1)	0.8236(1)	0.0492(3)
C1	0.0092(2)	0.6477(1)	0.2125(1)	0.0402(3)
C2	−0.0799(2)	0.7096(2)	0.0947(1)	0.0473(4)
C3	−0.0924(2)	0.8652(2)	0.0978(1)	0.0490(4)
C4	−0.0169(2)	0.9629(2)	0.2169(1)	0.0515(4)
C5	0.0714(2)	0.9029(1)	0.3337(1)	0.0462(4)
C6	0.0870(2)	0.7450(1)	0.3342(1)	0.0375(3)
C7	0.1856(2)	0.6868(1)	0.4587(1)	0.0415(3)
C8	0.3337(2)	0.3453(1)	0.5769(1)	0.0386(3)
C9	0.4379(2)	0.2949(1)	0.7085(1)	0.0361(3)
C10	0.4450(2)	0.1372(1)	0.7129(1)	0.0450(4)
C11	0.6109(2)	0.1691(1)	0.9354(1)	0.0461(4)
C12	0.6159(2)	0.3275(1)	0.9410(1)	0.0468(4)
C13	0.5284(2)	0.3913(1)	0.8260(1)	0.0420(3)
O3	0.5494(2)	0.7556(1)	0.7491(1)	0.0595(3)

[a] $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \boldsymbol{a}_i \cdot \boldsymbol{a}_j$.

Table 3
Selected bond lengths (Å) and angles (°) of **1**

O2–C8	1.222(1)	N2–N1–C7	117.98(9)
O1–C1	1.354(1)	N1–N2–C8	117.38(9)
N1–C7	1.276(2)	C10–N3–C11	117.0(1)
N1–N2	1.375(1)	O1–C1–C6	122.53(9)
N3–C11	1.329(2)	O1–C1–C2	117.7(1)
N2–C8	1.354(1)	C1–C6–C7	121.9(1)
N3–C10	1.334(2)	N1–C7–C6	120.27(9)
C6–C7	1.455(1)	O2–C8–C9	121.4(1)
C8–C9	1.494(1)	N2–C8–C9	116.41(9)
		O2–C8–N2	122.2(1)
		C8–C9–C10	116.92(9)
		C8–C9–C13	125.76(9)

compound **1** (Table 3). However, the search of CSD on the crystal structures containing hydrazone in the tautomeric form II revealed no hits.

The molecule of **1** is close to be planar. The three relevant dihedral angles which show a slight deviation from planarity are: between phenyl and pyridine rings, 4.61(6)°, between the central hydrazone moiety and each aromatic ring, 3.54(7)° and 7.49(7)°, respectively. The intramolecular hydrogen bond between the hydroxyl and imino group O1–H1···N1 (Table 4) stabilizes such a conformation. The CSD search [30] on unsubstituted salicylideneamino moieties in enolimino form (tautomeric form I) revealed 165 fragments in 123 structures. All fragments are nearly planar with O–H···N intramolecular hydrogen bonds.

In crystal packing the water molecules connect the molecules of **1** via N2–H2(N)···O3 and O3–H1(W)···O2 into a centrosymmetric dimer classified as $\mathbf{R}_4^4(\mathbf{12})$ [31]. These dimers are connected through O3–H2(W)···N3 into an infinite ladder pattern running along *b* defined by descriptor $\mathbf{C}_2^2(\mathbf{8})$ (Fig. 2, Table 4). Thus, the water molecule acts as a double donor (O–H···O and O–H···N types) and an acceptor (N–H···O). The infinite chains are subjected to a π··π interactions acting between the aromatic rings in the direction of *a*.

3.2. Spectroscopic characterization of *1* and *2*

3.2.1. IR spectra

The characteristic IR bands (KBr) are shown in Table 5. The broad band with the maximum at about 3200 cm^{-1} in the spectra of both compounds is due to stretching vibrations of hydrogen bonded NH [32]. An additional stretching vibration band at 3360 cm^{-1} in the spectrum of compound **2** indicates the presence of the free NH group [32]. It can be concluded that hydrogen bond association of compound **2** is

Table 4
Hydrogen-bond geometry of **1**

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
O1–H1(O)···N1 (intra)	0.88(2)	1.81(2)	2.612(1)	150(2)
O3–H1(W)···O2$^{(i)}$	0.86(2)	2.03(2)	2.875 (2)	170(2)
N2–H2(N)···O3	0.88(2)	2.07(2)	2.892 (1)	156(2)
O3–H2(W)···N3$^{(ii)}$	0.82(2)	2.05(2)	2.859 (1)	167(2)

Symmetry codes:(*i*) 1−*x*,1−*y*,1−*z*; (*ii*) *x*, 1 + *y*, *z*

Table 5
Characteristic IR bands (KBr) of compounds **1** and **2**

	ν/cm^{-1}			
	C=N	C=O	C–O	NH
1	1600 1570	1655	1265	3200
2	1605 1590 1570	1655	1265 1250	3360 3210

considerably lower compared with compound **1**. Both compounds exhibit stretching vibration frequencies of imino bond formed by condensation of aldehyde and hydrazide in the range 1605–1570 cm^{-1}. Intensive band originating from stretching vibrations of hydrogen bonded C=O group of hydrazide moiety is at 1655 cm^{-1} in the spectra of both compounds [33]. The C–O stretching vibration frequencies of hydroxy and methoxy group substituted on benzene ring are at 1265 and 1250 cm^{-1}. The bands at about 1530 cm^{-1} which should appear by formation of II (ν(N=C–O)) or III (ν(NHCH=CC=O)) [19,20,34,35] are missing in both spectra. Accordingly, both compounds take in crystalline state the tautomeric form I. The band at about 3500 cm^{-1}, present in both spectra, reveals the presence of crystalline water.

3.2.2. NMR spectra

The ^{1}H NMR spectra of compounds **1** and **2** in dimethylsulphoxide are shown in Table 6. The spectra of both compounds exhibit broad signals at 10.80–11.16 ppm due to the –NH proton. Strong deshielding of this proton can be explained by hydrogen bond formation. The shift is more pronounced for compound **1**, indicating the stronger hydrogen bond association, as revealed by IR spectral data. The signals of hydroxy group substituted on benzene ring appears at 12.24–12.27 ppm at 20°C, i. e. in the range characteristic for intramolecular bonded OH···N=C. Upfield shift of these signals caused by increase in the temperature (Table 7) is typical for intramolecular hydrogen bond. There is no evidence in the spectra for intramolecular proton transfer and formation of III, which would cause splitting of the methine proton signal. The signal of the Py–C–OH proton is missing in the spectra of both compounds and accordingly the presence of II in dimethylsulphoxide can be also ruled out.

Table 6
^{1}H NMR spectral data for **1** and **2** dissolved in dimethylsulphoxide at 20°C

Compound **1**		Compound **2**	
Proton	δ/ppm	Proton	δ/ppm
H (O)	12.27	H (O)	12.24
H (C2)	6.95	H (OCH_3)	3.83
H (C3)	7.33	H (C3)	7.06
H (C4)	6.94	H (C4)	6.88
H (C5)	7.59	H (C5)	7.20
H (C7)	8.66	H (C7)	8.68
H (C10)	9.10	H (C10)	9.09
H (C11)	8.79	H (C11)	8.79
H (C12)	7.60	H (C12)	7.60
H (C13)	8.29	H (C13)	8.29
H (N)	11.16	H (N)	10.80

Table 7
Dependence of chemical shifts of hydroxy protons on the temperature in dimethylsulphoxide

δ (OH)/ppm	t/°C				
	30	50	70	90	110
Compound **1**	12.25	12.18	12.14	12.12	12.07
Compound **2**	12.23	12.16	12.07	12.03	12.04

The data obtained by ^{1}H NMR and ^{13}C NMR are in accord. The spectrum of compound **1** (Table 8) contains only signals of form I. The typical signals

Table 8
^{13}C NMR spectral data for **1** in dimethylsulphoxide at 20°C

C atom	δ/ppm
1	157.6
2	116.6
3	123.8
4	119.5
5	129.5
6	118.8
7	131.8
8	161.6
9	128.8
10	152.6
11	148.8
12	148.7
13	135.6

Table 9
UV spectral data of compounds **1** and **2**

	λ/nm ($\epsilon \times 10^{-4}$/cm^{-1} mol^{-1} dm^3)					
	Chloroform	Dimethylsulphoxide	Dimethylformamide	Dioxane	Dioxane/water 1/1	Methanol
1	331 sh	332 (1.49)	330 (1.44)	329 (1.27)	329 (1.36)	330 (1.40)
	298 (1.55)	300 (1.49)	299 (1.67)	298 (1.55)	298 (1.73)	298 (1.74)
	288 (1.72)	289 (1.49)	288 (1.68)	287 (1.68)	288 (1.78)	288 (1.83)
2	347 sh	345 sh	340 sh	340 sh	344 sh	342 sh
	301 (2.02)	305 (2.01)	302 (2.09)	300 (1.97)	302 (2.24)	300 (2.18)

of form III (C1, ~180 ppm) and form II (C8, 100 ppm) are missing [36]. The signals of C1 and C8 appear at 157.8 and 161.6 ppm, respectively, confirming the presence of form I.

3.2.3. UV spectra

Table 9 illustrates UV/Vis absorption spectra of compounds **1** and **2**. No solvent effects on spectra were observed. Therefore, no shifts of tautomeric equilibria can be expected.

4. Concluding remarks

N′-salicylidene-3-pyridinecarbohydrazide and *N′*-(2-hydroxy-3-methoxyphenylmethylidene)-3-pyridinecarbohydrazide do not show the tendency to tautomeric ketoamino–enolimino interconversion. In the solid state and solution both compounds appear in form I. The preference for form I can be attributed to the π–p conjugation which takes place between the π-electrons of the –C=N– bond and the lone electron pair of the nitrogen of the –C=N–N– group. The stability of form I makes these compounds attractive for analytical application.

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