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Synthesis of mono and *bis*-substituted asymmetrical compounds, (1-(pyridin-2-yl)ethylidene)carbonohydrazide and 1-(2'-hydroxybenzylidene)-5-(1'-pyridylethylidene)carbonohydrazone: Structural characterization and antioxidant activity study

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ABSTRACT

Carbonohydrazide was used for synthetizing a new dissymmetrical *bis*-substituted Schiff base 1-(2'-hydroxybenzylidene)-5-(1'-pyridylethylidene)carbonohydrazone (2). A mono substituted compound (1-(pyridin-2-yl)ethylidene)carbonohydrazide (1) was firstly prepared by condensation reaction of carbonohydrazide and 2-acetylpyridine in 1:1 ratio. Secondly, compound 2 was obtained by condensation reaction of compound 1 and salicylaldehyde in 1:1 ratio. The prepared compounds were characterized by elemental analysis, infrared and ^{1}H and ^{13}C NMR spectroscopy techniques, and the structure of compound 2 was determined by single-crystal X-ray diffraction study. The compound 2 ($C_{15}H_{15}N_5O_2$) crystallises in the monoclinic space group $P2_1/c$ with the following unit cell parameters: a = 8.3683(3) Å, b = 13.9986(4) Å, c = 12.1610(4) Å, β = 97.512(3)°, V = 1412.37(8) Å^3, Z = 4, T = 100(2) K, μ(MoKα) = 0.098 mm^{-1}, *Dcalc* = 1.398 g/cm^3, 6057 reflections measured (5.708° ≤ 2Θ ≤ 54.962°), 6057 unique (R_{sigma} = 0.0395) which were used in all calculations. The final R_1 was 0.0474 (I > 2σ(I)) and wR_2 was 0.1971 (all data). The oxygen atom O1 and the azomethine nitrogen atom N5 adopt *cis*-configuration relative to the C8-N4 bond, while O1 adopts *trans*-configuration with the azomethine nitrogen atom N2 relative to C8-N3 bond. The crystal packing of compound 2 is stabilized by intramolecular O(phenol)–H···N(carbohydrazide) and intermolecular N (carbohydrazide)–H···O (carbohydrazide) hydrogen bonds which form layers parallel to [010] axis. Additional C–H···O hydrogen bond consolidate the structure. The carbonohydrazide moiety C=N–N–C(O)–N–N=C fragment and the phenyl ring are almost coplanar; with an angle of 1.73(1)° between their means plans. The dihedral angle between the mean planes of the phenyl and the pyridine rings is 22.267(2)°.

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1. Introduction

Carbonohydrazide ($H_2NNHCONHNH_2$) is a compound with two identical moieties and very reactive with respect to carbonyl compounds. Controlling the ratio of carbonohydrazide/carbonyl allows to synthesize symmetrical or dissymmetrical compounds by condensation reaction. Carbohydrazide and its derivatives have been largely investigated since 1894 when their syntheses were first reported [1]. Carbohydrazide derivatives were used as precursors for the synthesis of various heterocyclic compounds containing nitrogen atom in the cycle and/or in the free arms [2-4]. The reactivity as well as the biological properties of these derivatives have been investigated in the past decades for the development of drugs [5,6] or industrial applications [7,8]. They are known to possess a broad spectrum of biological activities such as antioxidant [9], analgesic [10], antiplatelet [11], antifungal [2], antimicrobial [12], anticonvulsant [13], antidepressant [14], anti-inflammatory [15], anti-tubercular [16], anti-HIV [5], anti-diabetic [10,17], and anticancer activities [3,18]. While most of the older work on carbohydrazide Schiff bases focused on their applicability in classical fields, nowadays these systems are increasingly acknowledged as supreme multitopic ligands for the targeted construction of original metal-organic architectures such as grids [19,20]. As a part of our search for a suitable ligand for building grids of metal complexes, we have prepared 1-(2'-hydroxy benzylidene)-5-(1'-pyridylethylidene)carbonohydrazone (**2**), a

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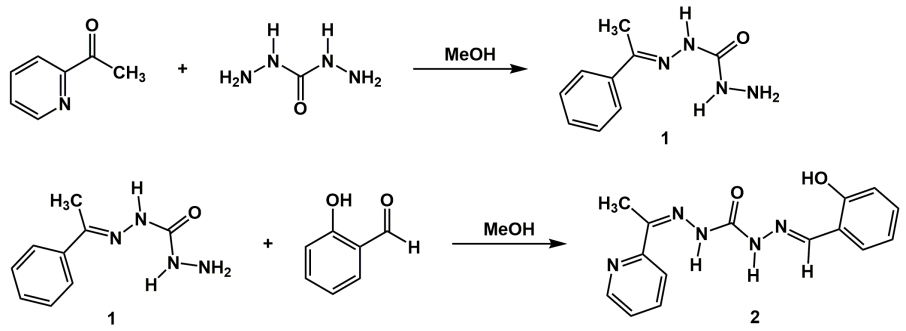

Scheme 1. Synthesis procedure of the compounds.

structural analog of a series of carbohydrazide derivatives that have proved to be good ligands for developing original structures in metal-organic [21,22]. Herein, we report on the synthesized and the crystal structure of a dissymmetrical carbohydrazide derivative (**2**).

2. Experimental

2.1. Materials and physical methods

Salicylaldehyde, 2-pyridinecarboxaldehyde, carbohydrazide, and 1,1-diphenyl-2-picrylhydrazyl (DPPH) were of analytical reagent grade and were obtained from Sigma-Aldrich Company. All used solvents were of UV spectroscopic quality. The elemental analyses of C, H and N were recorded on a VxRio EL Instrument. FT-IR spectra were recorded in the region of 4000-400 cm^{-1} using a Perkin Elmer Spectrum Two FT-IR spectrometer. The UV-Visible spectra were recorded on a Perkin Elmer Lambda UV-Vis spectrophotometer. The ^{1}H and ^{13}C NMR spectra were recorded in DMSO-d_6 on a Bruker 500 MHz spectrometer at room temperature using TMS as an internal reference.

2.2. Synthesis

2.2.1. Synthesis of mono-substituted precursor (1-(pyridin-2-yl)ethylidene)carbonohydrazide (1)

The procedure is inspired by the method reported by Novak *et al.* [23], with some modification. Herein, 2-acetylpyridine was used instead of salicylaldehyde. To a mixture of 20 mL of methanol and 10 mL of distillated water was added carbohydrazide (3 g, 0.0333 mol) at room temperature. A solution of 2-acetylpyridine (4 g, 0.0330 mmol) dissolved in 20 mL of methanol was slowly dropwise over a period of one hour. The resulting mixture was heated under reflux for 4 h. The suspension was filtered, and the white precipitate obtained was washed with (2 × 10 mL) of hot methanol and dried under vacuum over P_2O_5 (Scheme 1).

(1-(Pyridin-2-yl)ethylidene)carbonohydrazide (**1**): Color: Dark. M.p.: 221.8-222.5 °C. Yield: 86.37 %. FT-IR (ATR, ν, cm^{-1}): 3306 (NH), 3086 (=C-H), 1671 (C=O), 1634 (C=N), 1578 (C_{Ar}=C_{Ar}), 1506 (C_{Ar}=C_{Ar}), 1466 (C_{Ar}=C_{Ar}), 1141. ^{1}H NMR (500 MHz, DMSO-d_6, δ, ppm): 2.36 (s, 3H, CH_3), 4.12 (s, 2H, NH_2), 7.32-8.51 (m, 4H, Py-H), 8.19 (s, 1H, N-H), 9.64 (s, 1H, N-H). ^{13}C NMR (125 MHz, DMSO-d_6, δ, ppm): 157.32 (C=O), 155.30 (Py), 148.37 (C=N), 145.45 (C_{Ar}), 136.43 (C_{Ar}), 123.47 (C_{Ar}), 120.13 (C_{Ar}), 11.03 (CH_3). Anal. calcd. for $C_8H_{11}N_5O$: C, 49.73; H, 5.74; N, 36.25. Found: C, 49.70; H, 5.72; N, 36.22%.

2.2.2. Synthesis of (1E,5E)-1-(2-hydroxybenzylidene)-5-(1-(pyridin-2-yl)ethylidene)carbonohydrazide (2)

The mono-substituted derivate prepared above (1 g, 0.0052 mol) was mixed with 20 mL of methanol, then salicylaldehyde (0.949 g, 0.00777 mol) in 20 mL of methanolic solution was added. The mixture was heated under reflux for 30 minutes. Few drops of glacial acetic acid were added and immediately the suspension disappears. After 4 h under reflux, yellow clear solution was obtained. On cooling, white precipitate appears and was isolated by filtration. The solid was washed with cold methanol (2 × 10 mL) and dried under vacuum over P_2O_5. The filtrate was left under slow evaporation at room temperature. Few days later, colorless crystals suitable for X-ray diffraction were collected (Scheme 1).

(1E, 5E)-1-(2-Hydroxybenzylidene)-5-(1-(pyridin-2-yl)ethylidene)carbonohydrazide (**2**): Color: Colorless. Yield: 78.25%. M.p.: 189.8-190.4 °C. FT-IR (ATR, ν, cm^{-1}): 3432 (OH), 3192 (NH), 3090 (=CH), 1694 (C=O), 1619 (C=N), 1584 (C_{Ar}=C_{Ar}), 1536 (C_{Ar}=C_{Ar}), 1488 (C_{Ar}=C_{Ar}), 1462 (C_{Ar}=C_{Ar}), 1270 (C-O), 1146. ^{1}H NMR (500 MHz, DMSO-d_6, δ, ppm): 2.37 (s, 3H, CH_3), 6.90-8.62 (m, 8H, HPh + HPy), 8.52 (s, 1H, N=C–H), 11.34 (s, 1H, N-H), 10.99 (s, 1H, N-H), 10.25 (s, 1H, O-$H_{phenolic}$). ^{13}C NMR (125 MHz, DMSO-d_6, δ, ppm): 157.07 (C=O), 154.71 (PhCOH), 152.19 (Py), 148.37 (C=N), 147.47 (C_{Ar}), 136.71 (C_{Ar}), 116.29-136.13 (C_{Ar}), 11.05 (CH_3). Anal. calcd. for $C_{15}H_{15}N_5O_2$: C, 60.60, H, 5.09, N, 23.56. Found: C, 60.58, H, 5.10, N, 23.53%.

2.3. Free radical scavenging antioxidant assay

Antioxidant capacities of compounds **1** and **2** are measured according to Akhtar *et al.* [24] method with modifications. The methanol solution of 3.8 mL DPPH• (40 mg/L) was added to test compounds (200 µL) at different concentrations. The mixture was shaken vigorously and incubated in dark for 30 min at room temperature. After the incubation time, the absorbance of the solution was measured at 517 nm by using UV-vis spectrophotometer Perkin two. The DPPH• radical scavenger effect was calculated using the Equation (1):

$$\text{Scavenging activity (\% control)} = \frac{A_{control} - A_{sample}}{A_{control}} \times 100 \qquad (1)$$

where $A_{control}$ is the absorbance of the control reaction and A_{sample} is the absorbance of the test compound. The tests were carried out in triplicate. Trolox was used as positive control.

2.4. Crystal structure determination

Crystals suitable for X-ray single crystal diffraction of the reported compound were grown by slow evaporation of MeOH solution of compound **2**. Details of the X-rays crystal structure solution and refinement are given in Table 1. Diffraction data were collected using an ENRAF NONIUS Kappa CCD diffractometer with graphite monochromatized MoKα radiation (λ = 0.71073 Å). All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL [25].

Table 1. Crystal data and structure refinement for compound **2**.

Parameters	2
Empirical formula	$C_{15}H_{15}N_5O_2$
Formula weight	297.32
Temperature (K)	100(2)
Crystal system	Monoclinic
Space group	$P2_1/c$
a (Å)	8.3683(3)
b (Å)	13.9986(4)
c (Å)	12.1610(4)
α (°)	90
β (°)	97.512(3)
γ (°)	90
Volume (Å^3)	1412.37(8)
Z	4
ρ_{calc} (g/cm^3)	1.398
μ (mm^{-1})	0.098
F(000)	624.0
Crystal size (mm^3)	0.175 × 0.06 × 0.025
Radiation	MoKα (λ = 0.71075 Å)
2Θ range for data collection (°)	5.708 to 54.962
Index ranges	$-10 \le h \le 10, -18 \le k \le 18, -14 \le l \le 15$
Reflections collected	6057
Independent reflections	6057 [R_{sigma} = 0.0395]
Data/restraints/parameters	6057/0/210
Goodness-of-fit on F^2	1.008
Final R indexes [I≥2σ (I)]	R_1 = 0.0474, wR_2 = 0.1475
Final R indexes [all data]	R_1 = 0.0698, wR_2 = 0.1971
Largest diff. peak/hole (e Å^{-3})	0.28/-0.23

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 [26]. The hydrogen atoms of water molecules and NH groups were located in the Fourier difference maps and refined. Others H atoms (CH and CH_3 groups) were geometrically optimized and refined as riding model by AFIX instructions. Molecular graphics were generated using ORTEP-3 [27]

3. Results and discussion

3.1. Synthesis

The compound (1-(pyridin-2-yl)ethylidene)carbonohydrazide (**1**) was prepared by a condensation reaction of 2-acetylpyridine and carbonohydrazide in methanol. The isolated product was used for the synthesis of the compound (1*E*,5*E*)-1-(2-hydroxybenzylidene)-5-(1-(pyridin-2-yl)ethylidene)carbono hydrazide (**2**). Salicylaldehyde and compound **1** were mixed in methanol under reflux (Scheme 1). The compounds yielded are soluble in polar organic solvents such as DMSO or DMF. The elemental analyses results are in accordance with the chemical formulae obtained from spectroscopic studies. Both infrared spectra of compounds **1** and **2** exhibit broad bands in the range 3310-3185 cm^{-1} which are attributed to N-H stretching [28]. The vibration of the imine functions appears in the range 1634-1619 cm^{-1} while the band due to C=O group is in the range 1694-1671 cm^{-1} as reported for similar Schiff base [29]. Bands due to the aromatic ring are pointed in the region 1584-1462 cm^{-1}. Additional broad band is pointed in the spectrum of compound **2** at 3432 cm^{-1} and attributed to the vibration of the O-H of the phenol function [30].

The ^{1}H NMR spectra of the compounds **1** and **2** were recorded in DMSO-d_6. The ^{1}H NMR spectra of the compound **1** reveal a singlet at δ 2.36 ppm attributed to the CH_3 group, a singlet at δ 4.12 ppm assigned to -NH_2, a multiplet in the range δ 7.32-8.51 ppm which is representative of the aromatic protons. The signals at δ 8.19 and 9.64 ppm attributed to -NH are indicative of the dissymmetry of the monosubstituted compound. These observations are in accordance with the ^{13}C spectrum of compound **1**. The signal at δ 148.37 ppm attributed to the C=N is indicative of the successful of the condensation reaction. Additional signals are pointed for the aromatic carbon atoms (δ 145.45-120.13 ppm), for methyl group (δ 11.03 ppm) and for C=O (δ 157.32 ppm). Upon condensation of compound **1** with salicylaldehyde, the ^{1}H and ^{13}C NMR spectra of the yielded compound **2** show a new signal at δ 10.50 ppm appears a broad singlet which is due to Ar-OH. This signal is supported by the new signal at δ 157.71 ppm attributed to C_{ipso} of the phenyl ring. The signals of -NH and CH_3 are slightly shifted comparatively to the spectrum of compound **1**.

3.2. Structure description of compound 2

The molecular structure of the compound **2** with atomic labelling scheme is shown in Figure 1. The asymmetric unit of compound **2** consists of one molecule of the dissymmetrical Schiff base ligand. Crystal structure reveals that the organic molecule adopts the keto form, as showed by bond length of 1.227(3) Å for C8-O1 which is double bond character [31]. Additionally, C6-N2 and C9-N5 have double bond character as shown by the distances values of 1.282(3) and 1.284(3) Å, respectively (Table 2). The values of 1.372(3) and 1.373(3) Å for C8-N3 and C8-N4 are indicative of single bond character [32,33]. Oxygen atom O1 and the azomethine nitrogen atom N5 adopt *cis*-configuration relative to the C8-N4 bond, while atom O1 and azomethine nitrogen atom N2 adopt *trans*-configuration relative to C8-N3 bond. The torsion angles C6–N2–N6–C8 (178.8(2)°) and C8–N4–N5–C9 (-179.4(2)°) show that the central part of the molecule is almost linear. The molecule adopts an *E, E* configuration with respect to the C6-N2 and C9-N5 bonds. The carbonohydrazide moiety C=N–N–C(O)–N–N=C fragment and the phenyl ring are almost coplanar with an angle value of 1.73(1)° between their means plans, but the carbonohydrazide and the pyridine ring are not coplanar with dihedral angle value of 21.38(8)°. The dihedral angle value between the mean planes of the phenyl and the pyridine rings is 22.27(1) °.

The crystal packing of compound **2** is stabilized by intramolecular O (phenol)–H···N (carbohydrazide) and intermolecular N (carbohydrazide)–H···O (Carbohydrazide) hydrogen bonds which form layers parallel to *b* axis. Additional C–H···O hydrogen bonds consolidate the structure. In the crystal, intramolecular and intermolecular hydrogen bonds are simultaneously present.

Table 2. Selected bond lengths, bond angles and torsion angles for the compound **2**.

Atom-Atom	Bond lengths (Å)	Atom-Atom	Bond lengths (Å)
C8-O1	1.227 (3)	N2-N3	1.384 (3)
C8-N3	1.372 (3)	N4-N5	1.354 (3)
C8-N4	1.373 (3)	C6-N2	1.282 (3)
C5-C6	1.488 (3)	C9-N5	1.284 (3)
C6-C7	1.508 (3)	C9-C10	1.451 (3)
Atom-Atom-Atom	**(°)**	**Atom-Atom-Atom**	**Bond angles (°)**
O1-C8-N4	122.9 (2)	N5-C9-C10	119.5 (2)
O1-C8-N3	121.9 (2)	N2-C6-C5	115.6 (2)
N3-C8-N4	115.22 (19)	C6-N2-N3	116.51 (18)
N2-N3- C8	118.15 (18)	C9-N5-N4	119.14 (19)
N5-N4-C8	117.26 (19)	N2-C6-C7	124.6 (2)
Atom-Atom-Atom-Atom	**(°)**	**Atom-Atom-Atom-Atom**	**Torsion angles (°)**
O1-C8-N3-N2	168.7 (2)	O1-C8-N4-N5	1.6 (3)
C8-N4-N5-C9	-179.4 (2)	C6-N2-N3-C8	178.8 (2)
C10-C9-N5-N4	178.73 (18)	C5-C6-N2-N3	175.54 (18)
N4-C8-N3-N2	-12.9 (3)	N3-C8-N4-N5	-176.78 (19)

Table 3. Hydrogen-bond geometry for compound **2**.

D-H···*A*	*D*-H (Å)	H···*A* (Å)	*D*···*A* (Å)	∠*D*-H···*A* (°)
N3-H3A···O1^{i}	0.95(3)	1.91(3)	2.836(3)	166(2)
C7-H7C···O1^{i}	0.98	2.68	3.397(3)	130.6
O2-H2A···N5	0.84	1.87	2.600(2)	145.0
N4-H4A···O2^{ii}	0.95(4)	2.49(4)	3.148(3)	127(3)

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

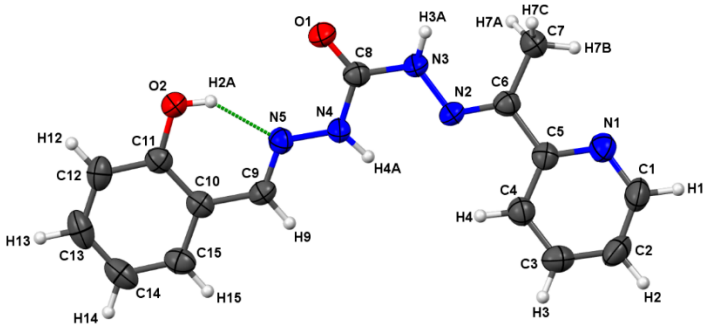

Figure 1. The crystal structure of the compound **2**. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small sphere.

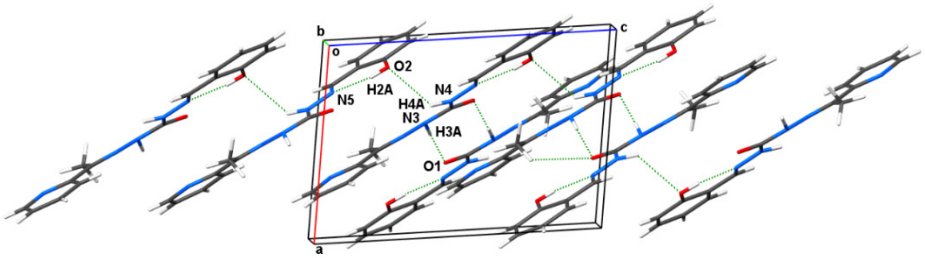

Figure 2. Layers of the title compound **2** viewed along the *b* axis.

The intramolecular hydrogen bond O2$_{(phenol)}$–H···N5$_{(azomethine)}$ forms a six-membered ring. Intermolecular hydrogen bonds, N3$_{(hydrazinyl)}$–H3A···O1^{i}$_{(carbonyl)}$ (*i*: 1-*x*, 2-*y*, 1-*z*) and N4$_{(hydrazinyl)}$–H4A···O2^{ii}$_{(phenolic)}$ (*ii*: *x*, -*y*+3/2, *z*-1/2) lead to the formation of layers parallel to *b* axis (Figure 2, Table 3). Additional C–H···O1^{i}$_{(carbonyl)}$ (*i*: 1-*x*, 2-*y*, 1-*z*) and C–H···O2$_{(phenol)}$ connect the layers and consolidate the structure into a three-dimensional network (Figure 3).

A search into the Cambridge Structural Database (Version 5.41, update November 2019; Groom *et al.*, 2016) of two fragments for the title compound gave several hits. The majority of these are symmetrically *bis*-substituted compounds. By using carbohydrazide, salicylaldehyde, and acetylpyridine as fragments, refcodes representing symmetrical and dissymmetrical compounds, were collected. Combining carbonohydrazide and salicyaldehyde, symmetrical disubstituted 1,5-*bis*(salicylidene)carbohydrazide compounds were obtained: SAGXOP [34] and SAGXOP01 [35]. However, when carbonohydrazide was combined with acetylpyridine in 1:2 ratio, disubstituted symmetrical *bis*(methyl-2-pyridylketone) carbonohydrazone compound was obtained: TIRYIC [36]. Dissymmetrical carbonohydrazone compounds are quite rare. However, it has been reported in CSD two dissymetrical carbonohydrazone Schiff bases: AROLUP [37] and MILZOZ [38].

3.3. Antioxidant activity

The method of scavenging the DPPH• radical is largely used to evaluate the antioxidant activity of organic or inorganic compounds [39,40]. The antioxidant activities of the two compounds **1** and **2** have been substantially investigated. Figure 4 shows the plots of DPPH• free radical scavenging activity (%) for Trolox, compounds **1** and **2**. The DPPH• is a stable free radical and becomes a stable molecule when it accepts an electron or hydrogen radical.

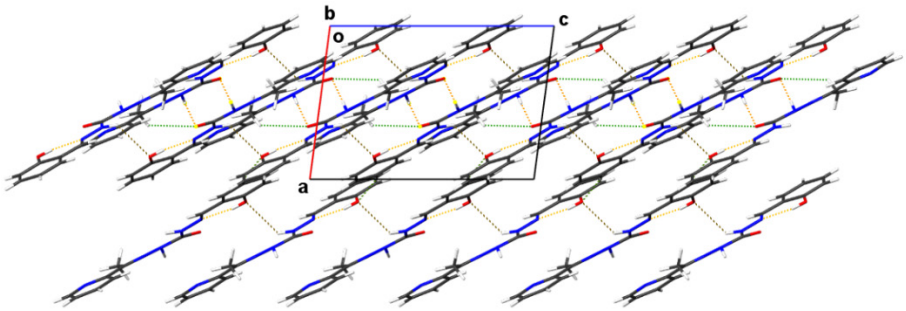

Figure 3. Crystal packing of the title compound **2** viewed along the *b* axis.

The antioxidant molecules scavenge the DPPH• radical by hydrogen donating ability. For compounds **1** and **2**, it is observed that the scavenging activity increases with increasing the concentration in the range tested (50-500 mmol/L). Compound **1** has scavenging activity between 7.21±0.42 and 32.86±0.01% within the investigated concentration range due to the NH groups which can react with DPPH• radical by the typical H-abstraction reaction to form a stable radical. Radical scavenging activity of compound **2** (5.62±0.12 - 29.96 ±0.12 %) is slightly lower than that observed for compound **1** (Figure 4). Comparatively to the scavenging activity of Trolox (7.28±0.69 - 70.36±0.34%), the values observed for compound **1** are higher than those of Trolox for low concentration (50- 200 mM) while those for compound **2** are comparable to those of Trolox. When increasing (300 to 500 mM) the concentration, the scavenging activity of Trolox increases rapidly while those of compounds **1** and **2** increase very slightly and do not exceed 33% for compound **1** and 30% for compound **2**.

4. Conclusion

The disubstituted carbonohydrazide derivative namely, (1*E*, 5*E*)-1-(2-hydroxybenzylidene)-5-(1-(pyridin-2-yl)ethylidene) carbonohydrazide (**2**) was successfully synthesized from the mono substituted carbohydrazide derivative (1-(pyridin-2-yl)ethylidene)carbonohydrazide (**1**). The structures of the compounds were confirmed by elemental analysis and spectroscopic techniques (FT-IR, ^{1}H and ^{13}C NMR). The molecular structure of the newly (1*E*, 5*E*)-1-(2-hydroxybenzylidene)-5-(1-(pyridin-2-yl)ethylidene)carbonohydrazide was also determined using X-ray crystallography technique. Compounds **1** and **2** showed moderate antioxidant activity of about 30-33 %.

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Supporting information

CCDC-2018491 contains 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 e-mailing 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.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.
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