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Synthesis and Spectroscopic Characterization of Novel Imidazole-Based Semicarbazone Schiff Base Ligands

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Abstract

Semicarbazone derivatives are important in organic synthesis and they are used in evaluating new product that possesses dif-ferent biological activities. Such as antibacterial, antifungal, antitumoral, antiviral, anticancer anticonvulsant, antidepressantand other biological activity. In this study, four novel: 4-methyl-5-imidazolecarboxaldehydesemicarbazone (C_1), 4-imidazole-carboxaldehydesemicabazone (C_2), 1-methyl-2-imidazolecarboxaldehydesemicarbazone (C_3) and 1-methyl-5-imidazolecar-boxaldehydesemicarbazone (C_4) were prepared by condensation reaction of Imidazolecarboxaldehydes with semicarbazidehydrochloride. The structures of the prepared compounds were characterized on the basis of their IR, UV-Vis, ESI Mas and ^{1}H ^{13}C-NMR data.

Keywords: Schiff Bases; Semicarbazone; Synthesis; Spectroscopic Analysis

Introduction

Heterocyclic Schiff base compounds have been an interesting field because of their various biological poperties. A number of heterocyclic derivatives containing nitrogen and sulphur atoms provide as a exclusive and multipurpose gallows for experimental drug design [1]. Benzothiazole is one of the most important heterocyclic compounds that have received overwhelming response owing to its diversified molecular design and remarkable optical, liquid and electronic properties [2]. Benzothiazole shows various biological activities such as antimicrobial [3-5], anticancer [6-7], anthelmintic [8], anti-diabetic [9] activities. The consequential compounds, Imidazole also reveal a number of significant biological activities such as antiparasitic, fungicidal, anithelemintic, antiinflammatory, antiprotozoal and herbicidal activity [10-14]. Hence, it was thought of interest to merge both of thiazole and imidazole moieties which may enhance the drug activity of compounds to some extent or they might possess some of the above-mentioned biological activities. Semicarbazones are versatile ligands that arise the interest of researchers, not only from a coordinative point of view, but also from a pharmacological one, in recent research some imidazole derivatives such as imidazole-2-carboxaldehyde semicarbazone and thiosemicarbazone, and their transition metal complexes have been synthesized [15]. From this point of view, the objective of the present work is to prepare new derivatives of semicarbazone Schiff base of imidazolecarboxaldehydes.

Materials and Methods

All chemicals and solvents of highest analytical grade were used as received form Sigma-Aldrich and Alfa-Aesar. The FT-IR of synthesized semicarbazones Schiff bases was recorded on Vertex-183387000 FT-IR spectrometer by using KBr disk in the range 400-4000 cm^{-1}. UV-Vis spectra in solid state was recorded on a Cary-4000. EL 05123055 UV-Vis spectrophotometer, Mass spectrum was carried out on Esquire LC-00084 electronic spray ionization (ESI) Mass spectrometer and ^1H and ^{13}C NMR spectra were recorded on Bruker AV-III 600 by using DMSO-d_6 as a solvent.

Synthesis of Imidazolecarboxaldehyde Semicarbazones (C_1-C_4)

Four new imidazolecarboxaldehyde semicarbazones Schiff bases were obtained by following a general procedure previously reported by Chatterjee [16]. Since the semicarbazide was in its hydrochloride form (0.11153 g, 0.001 mol), prior to its condensation with aldehydes separately, it was neutralised by addition of NaOH in a 1:1 ratio. Thus, an aqueous solution (20 mL) of the neutralised semicarbazide was slowly added to a warm (60°C) ethanolic solution (20 mL) of imidazolecarboxaldehydes (0.001 mol). The mixture was refluxed for 7 hrs and the resulting suspension was cooled at room temperature, and then allowed for slow solvent evaporation, the crystals obtained was filtrated, washed with water and ethanol, and finally dried under vacuum.

Synthesis of 4-Methyl-5-Imidazolecarboxaldehyde Semicarbazone (C_1)

White crystals,Yield: 80%, m.p 255°C. $C_6H_9N_5O$. FT-IR (KBr cm^{-1}), 3464, 3445, $\nu(NH_2)$; 3140, $\nu(NH)$; 1584, $\nu(C{=}N)$; 1690, $\nu(C{=}O)$; 1116. $\nu(N\text{-}N)$. NMR spectrum (600 MHz for ^1H and 151 MHz for ^{13}C, DMSO-d_6, ppm): ^1H NMR; δ=2.2 (3H, s, CH_3); 7.6 (H, CH ring), 6.4(2H, s, NH_2), 7.8(1H, s, =N-NH); 10.0 (1H, s, NH), 12.1 (1H, s, NH ring); ^{13}C NMR; 135.54(HC=N); 156.93(C=O). Ms (ESI.m/z): M^+ 168; Analysis for $C_6H_9N_5S$ (Mw 167.17). UV-Vis spectrum (λ_{max} nm): 298.

Synthesis of 4-Imidazolecarboxaldehyde Semicarbazone (C_2)

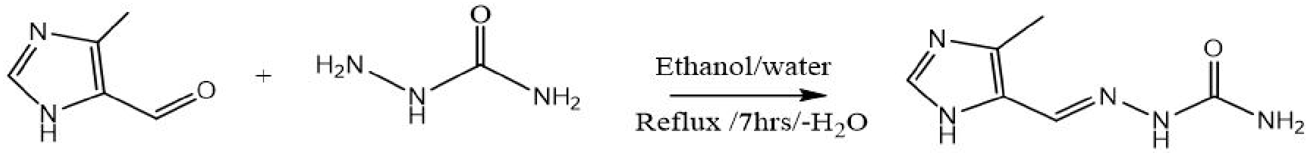
White crystals, Yield: 71%, m.p 220°C. $C_6H_9N_5O$. FT-IR (KBr cm^{-1}, 3382, 3323, $\nu(NH_2)$; 3217, $\nu(NH)$; 1592, $\nu(HC{=}N)$; 1655, $\nu(C{=}O)$; 1109. $\nu(N\text{-}N)$. NMR spectrum (600 MHz for ^1H and 151 MHz for ^{13}C, DMSO-d_6, ppm): ^1H NMR; δ=7.3-7.8 (2H, CH ring), 6.4(2H, s, NH_2), 7.8(1H, s, =N-NH); 10.2 (1H, s, NH), 12.4 (1H, s, NH ring); ^{13}C NMR; δ=136.89 (Cq ring); 128.70-129.45(2C-H ring), 129.79(HC=N); 157.03(C=O). Ms (ESI.m/z): M^+ 154.1; Analysis for $C_6H_9N_5O$ (Mw 153.14). UV-Vis spectrum (λ_{max} nm): 283.

Synthesis of 1-Methyl-2-Imidazolecarboxaldehyde Semicarbazone (C_3)

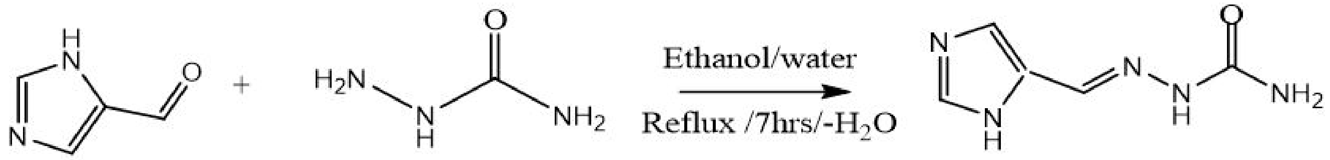
White crystals, Yield: 75%, m.p 248°C. $C_6H_9N_5O$. FT-IR (KBr cm^{-1}), 3294, 3256, $\nu(NH_2)$; 3093, $\nu(NH)$; 1541, $\nu(HC{=}N)$; 1640, $\nu(C{=}O)$; 1159. $\nu(N\text{-}N)$. NMR spectrum (600 MHz for ^1H and 151 MHz for ^{13}C, DMSO-d_6, ppm): ^1H NMR; $\delta=3.8$ (3H, s, CH_3); 6.9-7.3 (2H, CH ring), 6.4(2H, s, NH_2), 7.9(1H, s, =N-NH); 10.3 (1H, s, NH); ^{13}C NMR; $\delta=35.5$ (CH_3);141.40 (Cq ring); 124.76-128.32(2C-H ring), 132.75(HC=N); 156.43(C=O). Ms (ESI.m/z): M^+ 168.1; Analysis for $C_6H_9N_5O$ (Mw 167.17). UV-Vis spectrum (λ_{max} nm): 292.

Synthesis of 1-Methyl-5-Imidazolecarboxaldehyde Semicarbazone (C_4)

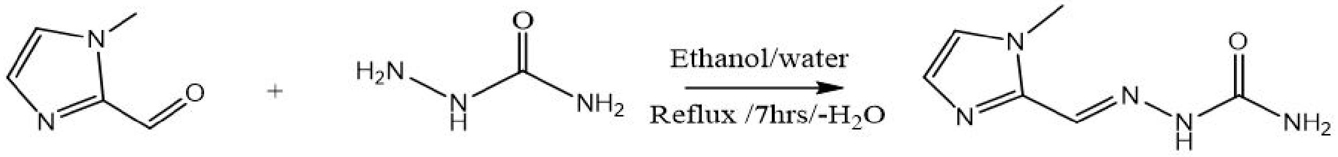
White crystals, Yield: 73%, m.p 240°C. $C_6H_9N_5O$. FT-IR (KBr cm^{-1}), 3471, 3309, $\nu(NH_2)$; 3147, $\nu(NH)$; 1579, $\nu(HC{=}N)$; 1691, $\nu(C{=}O)$; 1120, $\nu(N\text{-}N)$. NMR spectrum (600 MHz for ^1H and 151 MHz for ^{13}C, DMSO-d_6, ppm): ^1H NMR $\delta=3.9$ (3H, s, CH_3); 7.3-7.7 (2H, CH ring), 6.3(2H, s, NH_2), 7.9(1H, s, =N-NH); 10.2 (1H, s, NH); ^{13}C NMR; $\delta=33.87$ (CH_3);141.24 (Cq ring); 127.36-130.78(2C-H ring), 132.26(HC=N); 156.66(C=O). Ms (ESI.m/z): M^+ 168.1; Analysis for $C_6H_9N_5O$ (Mw 167.17). UV-Vis spectrum (λ_{max} nm): 288.



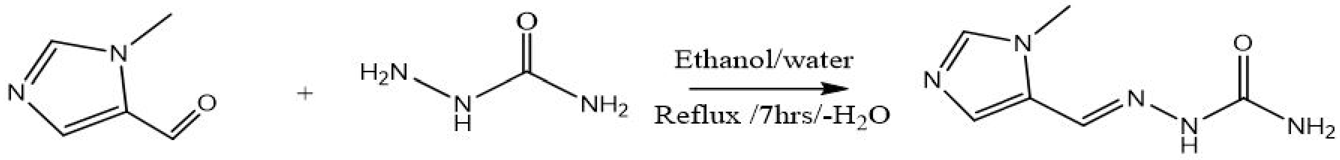
Scheme 1: Synthesis of 4-methyl-5-imidazolecarboxaldehyde semicarbazone (C_1)



Scheme 2: Synthesis of 4-imidazolecarboxaldehyde semicarbazone (C_2)



Scheme 3: Synthesis of 1-methyl-2-imidazolecarboxaldehyde semicarbazone (C_3)



Scheme 4: Synthesis of 1-methyl-5-imidazolecarboxaldehyde semicarbazone (C_4)

Results and Discussion

The synthesized imidazolecarboxaldehyde semicarbazones (C_1-C_4) were characterized by using spectroscopic methods (IR, UV-Vis, ESI Mass and ^{1}H, ^{13}C NMR).

UV-Vis Spectra of Prepared Imidazolecarboxaldehyde Semicarbazones

The electronic spectra of 4-methyl-5-imidazole carboxaldehyde semicarbazone (C_1), 4-imidazole carboxaldehyde semicabazone (C_2), 1-methyl-2-imidazolecarboxaldehydesemicarbazone (C_3) and 1-methyl-5-imidazolecarboxaldehyde semicarbazone (C_4). Showed that a strong absorption band at 298, 283, 292 and 288 nm respectively. These bands assigned to the π → π* transition of the azomethine group [17-18].

The infrared absorption bands become very useful for describing the prepared compounds. In the IR spectra, of (C_1) absorption peaks at 3464, 3445 cm^{-1} are assigned to ν(NH_2). The absorption peak at 3140 cm^{-1} is assigned to ν(N-H). The band at 1584 cm^{-1} which assigned to ν (nitrogen double bond stretching frequency, absorption band at 1690 cm^{-1} is attributed to ν (C=O) and peak at 1116 is assigned to ν(N-N). IR spectral data of (C_2) shows absorption peaks at 3382, 3323 cm^{-1} whish are assigned to ν(NH_2). The absorption peak at 3217 cm^{-1} is assigned to ν(N-H). The band at 1592 cm^{-1} which assigned to ν(C=N) carbon nitrogen double bond stretching frequency, absorption band at 1655 cm^{-1} is attributed to ν (C=O) and peak; at 1109 is assigned to ν(N-N).

IR spectral data of (C_3) shows absorption peaks at 3294, 3256 cm^{-1} whish are assigned to ν(NH_2). The absorption peak at 3093 cm^{-1} is assigned to ν(N-H). The band at 1541 cm^{-1} which assigned to ν(C=N) carbon nitrogen double bond stretching frequency, absorption band at 1640 cm^{-1} is attributed to ν(C=O) and peak; at1159 is assigned to ν(N-N).

IR spectral data of (C_4) Shows absorption peaks at 3471, 3309cm^{-1} whish is assigned to ν(NH_2). The absorption peak at 3141 cm^{-1} is assigned to ν(N-H). The band at 1579 cm^{-1} which assigned to ν(C=N) carbon nitrogen double bond stretching frequency, absorption band at 1691cm^{-1} is attributed to ν (C=O) and peak; at 1120 is assigned to ν(N-N).

Characterization of the Prepared Imidazolecarboxaldehyde Semicarbazones by ^{1}H And ^{13}C-NMR Spectroscopy

^{1}H and ^{13}C NMR spectra and chemical shift values of the prepared semicarebazones were record in DMSO-*d6* solvent. The ^{1}H NMR of (C_1-C_4) show signal at δ 7.8, 7.8, 7.9 and 7.9 ppm, respectively have been assigned to δ(HC=N) protons and signals at 6.4, 6.4, 6.4 and 6.3 ppm have been assigned to δ(NH_2) protons. The signals at δ 10.0, 10.2, 10.3 and 10.2ppm assignable to δ(NH) protons. Signals at 7.6-0.0, 7.3-7.8. 6.9-7.3 and 7.3-7.7 due to hydrogen protons of aromatic ring [19].

The ^{13}C NMR analysis of (C_1) the azomethine carbon (HC=N) was found at δ135.54ppm and signal at δ156.93 due to carbonyl group (C=O) [20]. The ^{13}C NMR analysis of (C_2) the azomethine carbon (HC=N) was found at δ129.79ppm. The aromatic carbon atoms in the ring appeared at δ 128.70, 129.45 and 136.89. Signal at δ157.03ppm due to carbonyl group (C=O) [19]. The ^{13}C NMR analysis of (C_3) the azomethine carbon (HC=N) was found at δ132.75ppm. The aromatic carbon atoms in the ring appeared at δ 124.76-128.32and 141.40ppm. Signal at δ156.43ppm due to carbonyl group (C=O). Methyl group carbon atom is shown at δ35.5 ppm [19]. The ^{13}C NMR analysis of (C_4) the azomethine carbon (HC=N) was found at δ132.26ppm. The aromatic carbon atoms in the ring appeared at δ 127.36-130.78 and 141.24. Signal at δ156.66ppm due to carbonyl group (C=O). Methyl group carbon atom is shown at δ35.5 ppm [19].

Table 1: UV-Vis spectrum and physical properties of imidazolecarboxaldehyde semicarbazone (C_1-C_4)

S.No	Compounds	Color	Shape	Yield %	M.P°C	UV-Vis (λ_{max} nm)
1	C_1	White	Crystal	80	255	298
2	C_2	White	Crystal	71	220	283
3	C_3	White	Crystal	75	248	292
4	C_4	White	Crystal	73	240	288

Table 2: Infrared spectral data of imidazolecarboxaldehyde semicarbazone (C_1-C_4)

S.No	Compounds	$\nu(NH_2)$ cm^{-1}	$\nu(NH)$cm^{-1}	$\nu(HC=N)$ cm^{-1}	$\nu(C=O)$ cm^{-1}	$\nu(N-N)$ cm^{-1}
1	C_1	3464, 3445	3140	1584	1690	1116
2	C_2	3382, 3323	3217	1592	1655	1109
3	C_3	3294, 3256	3093	1541	1640	1159
4	C_4	3471, 3309	3147	1579	1691	1120

Table 3: ^{1}H NMR spectral data (ppm) of Imidazolecarboxaldehyde semicarbazone (C_1-C_4)

S.No	Compounds	$\delta(C-H)_{ring}$	$\delta(HC=N)$	$\delta(NH_2)$	$\delta(NH)$	$\delta(N-H)_{ring}$	$\delta(-CH_3)$
1	C_1	7.6-0.0	7.8	6.4	10.0	12.1	2.2
2	C_2	7.3-7.8	7.8	6.4	10.2	12.4	-
3	C_3	6.9-7.3	7.9	6.4	10.3	-	3.8
4	C_4	7.3-7.7	7.9	6.3	10.2	-	3.9

Table 4: ^{13}C-NMR spectral data (ppm) of Imidazolecarboxaldehyde semicarbazone (C_1-C_4)

S.No	Compounds	$\delta(HC=N)$	$\delta(C=O)$.	δ(2C-H) ring	δ(Cq ring);	$\delta(CH_3)$
1	C_1	135.54	156.93	-	-	-
2	C_2	129.79	157.03	128.70-129.45	136.89	-
3	C_3	132.75	156.43	124.76-128.32	141.40	35.5
4	C_4	132.26	156.66	127.36-130.78	141.24	33.87

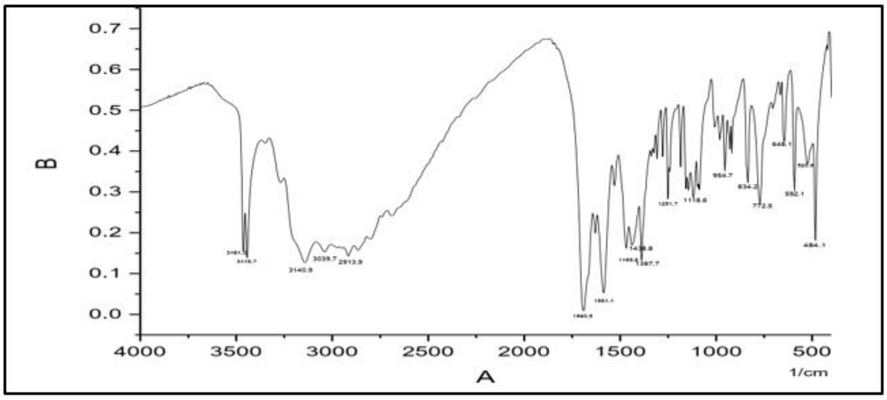


Figure 1: IR spectrum of 4-methyl-5-imidazolecarboxaldehyde semicarbazone (C_1)

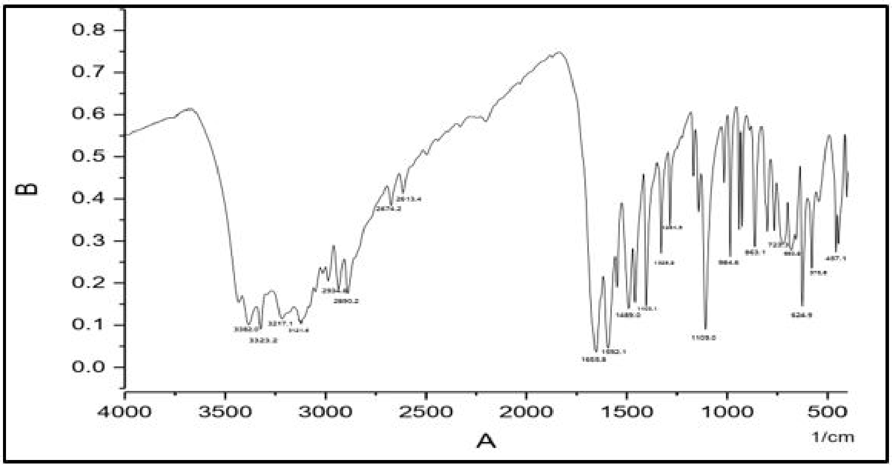


Figure 2: IR spectrum of 4-imidazolecarboxaldehyde semicabazone (C_2)

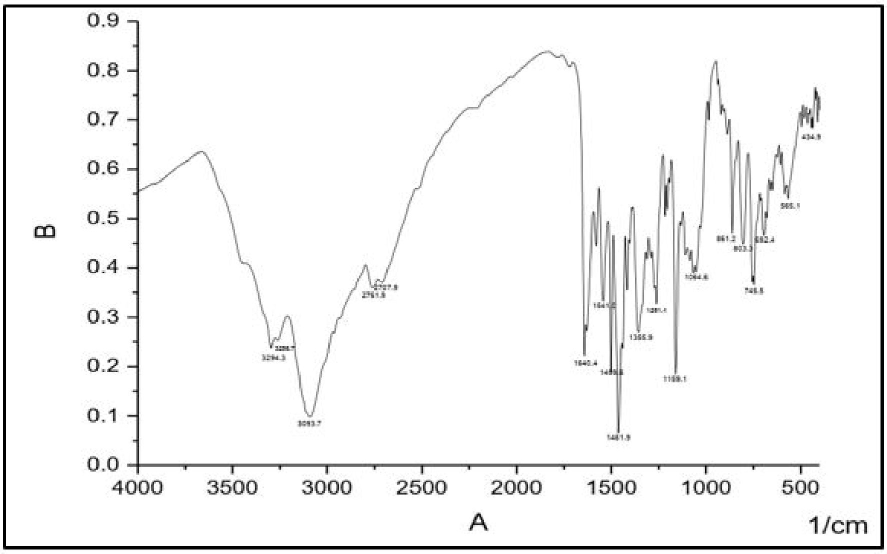


Figure 3: IR spectrum of 1-methyl-2-imidazolecarboxaldehyde semicarbazone (C_3)

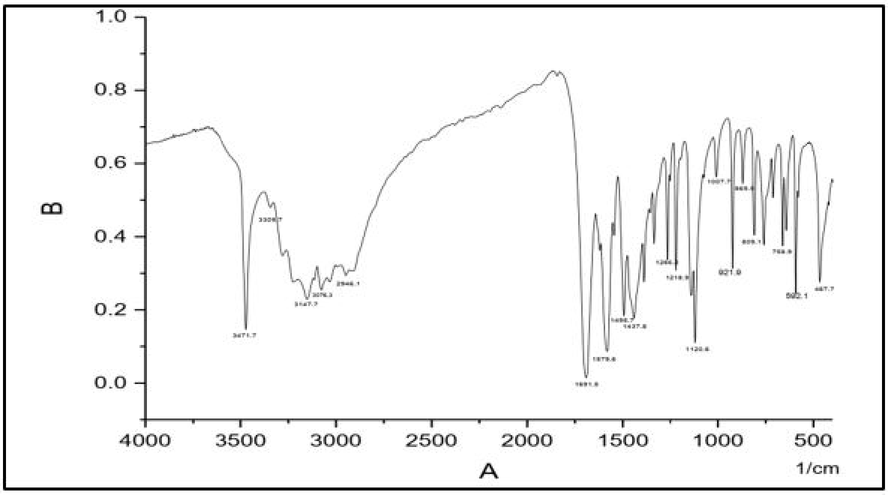


Figure 4: IR spectrum of 1-methyl-5-imidazolecarboxaldehyde semicarbazone (C_4)

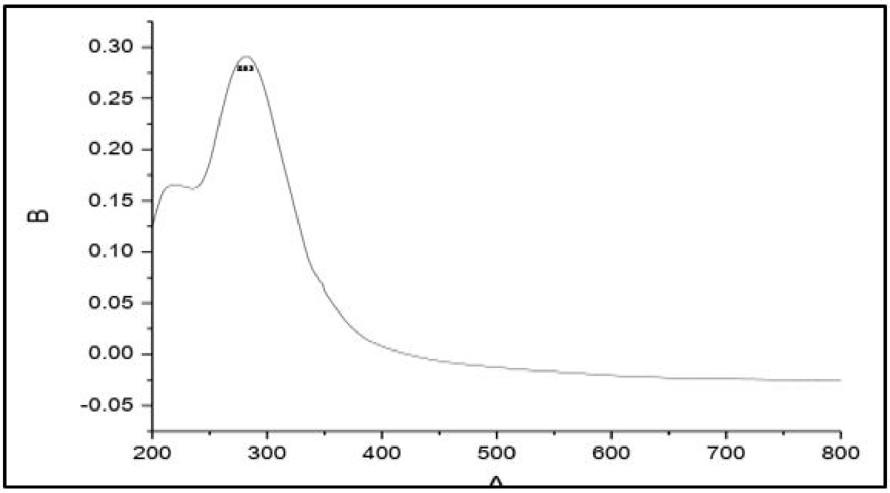


Figure 5: UV-Vis spectrum of 4-methyl-5-imidazolecarboxaldehyde semicarbazone (C_1)

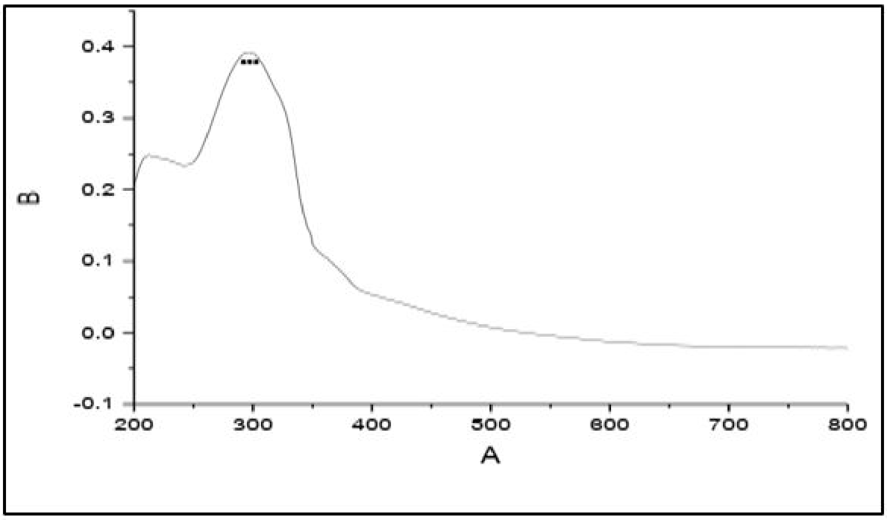


Figure 6: UV-Vis spectrum of 4-imidazolecarboxaldehyde semicabazone (C_2)

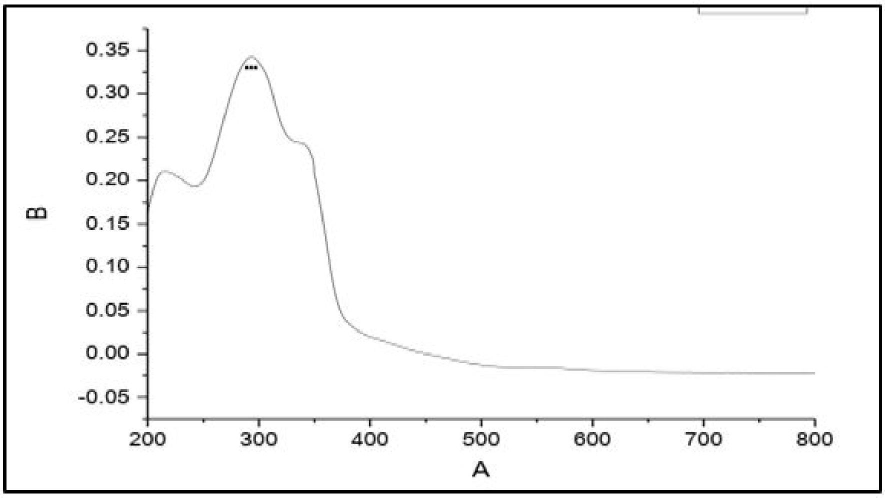


Figure 7: UV-Vis spectrum of 1-methyl-2-im idazolecarboxaldehyde semicarbazone (C_3)

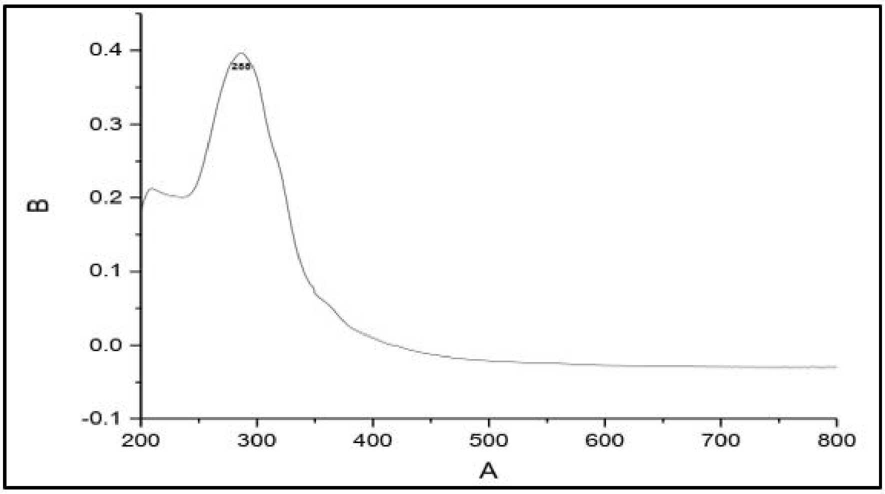


Figure 8: UV-Vis spectrum of 1-methyl-5-imidazolecarboxaldehyde semicarbazone (C_4)

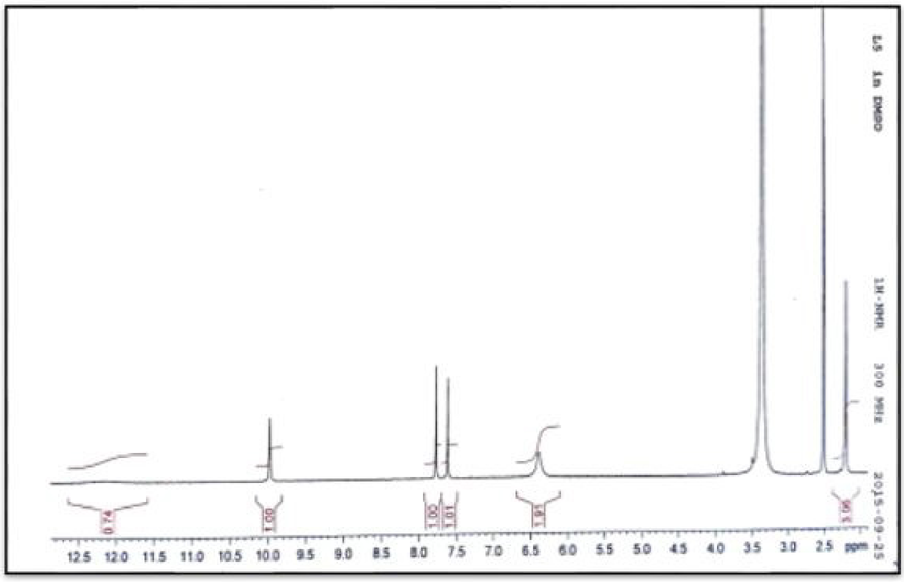


Figure 9: ^{1}H NMR spectrum of 4-methyl-5-imidazolecarboxaldehyde semicarbazone (C_1)

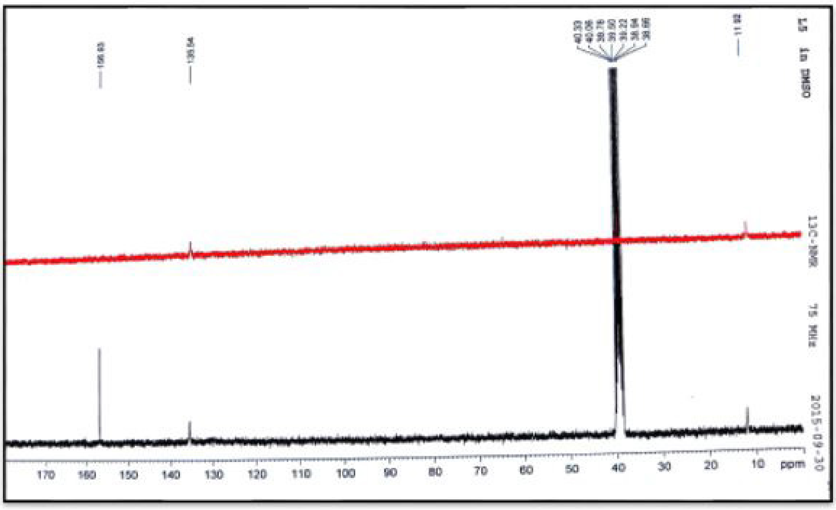


Figure 10: ^{13}C NMR spectrum of 4-methyl-5-imidazolecarboxaldehyde semicarbazone (C_1)

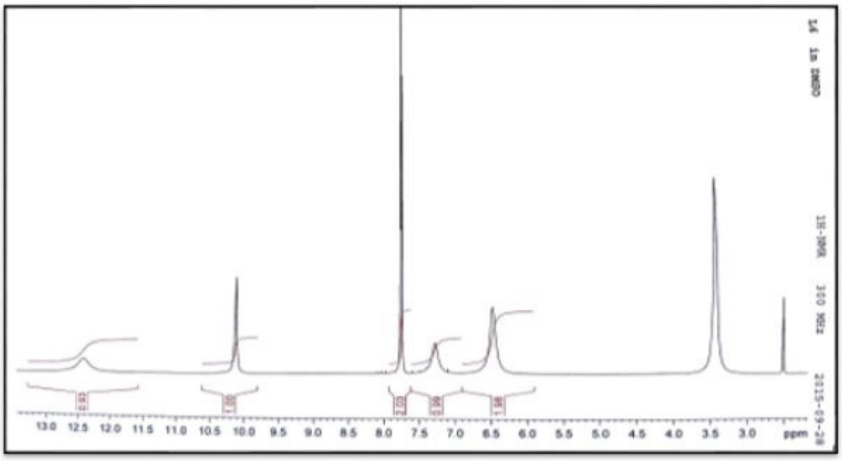


Figure 11: ^{1}H NMR spectrum of 4-imidazolecarboxaldehyde semicabazone (C_2)

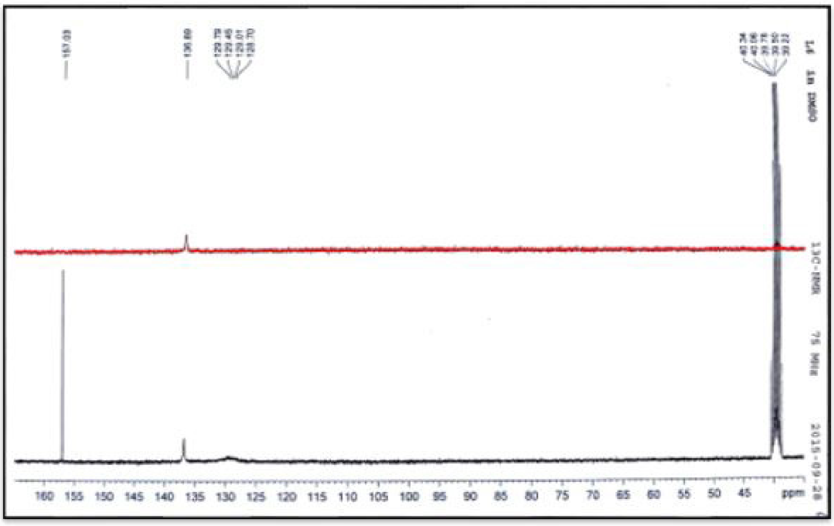


Figure 12: ^{13}C NMR spectrum of 4-imidazolecarboxaldehyde semicabazone (C_2)

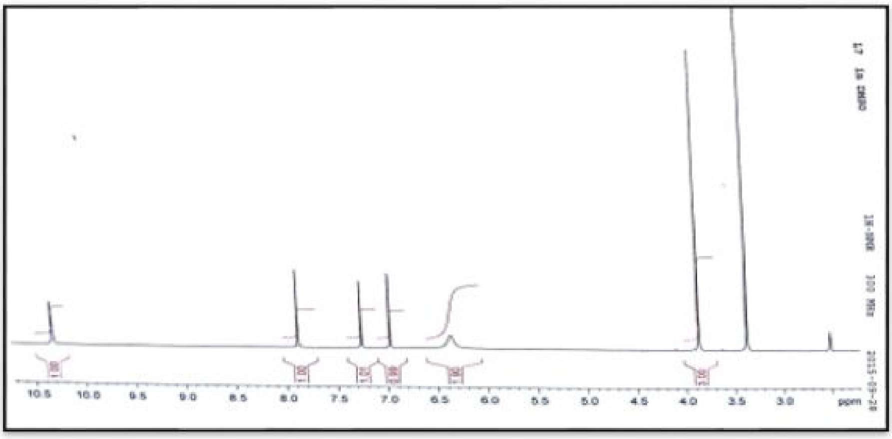


Figure 13: ^{1}H NMR spectrum of 1-methyl-2-imidazolecarboxaldehyde semicarbazone (C_3)

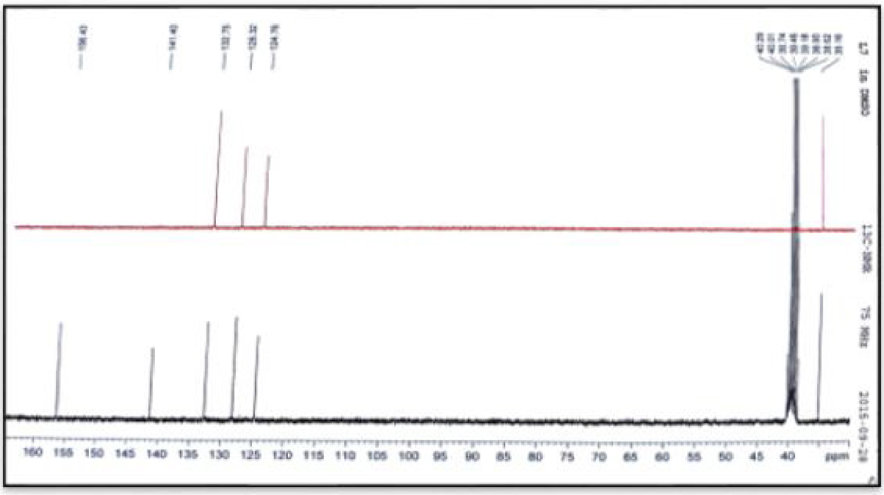


Figure 14: ^{13}C NMR spectrum of 1-methyl-2-imidazolecarboxaldehyde semicarbazone (C_3)

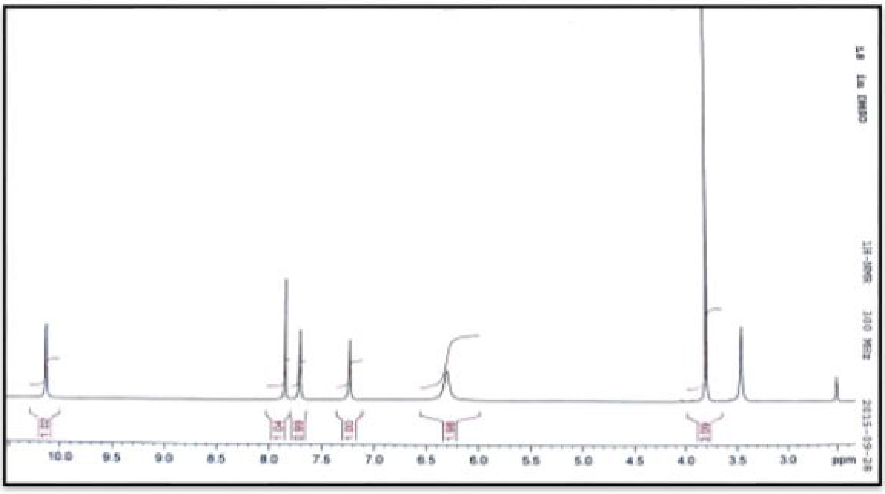


Figure 15: ^{1}H NMR spectrum of 1-methyl-5-imidazolecarboxaldehyde semicarbazone (C_4)

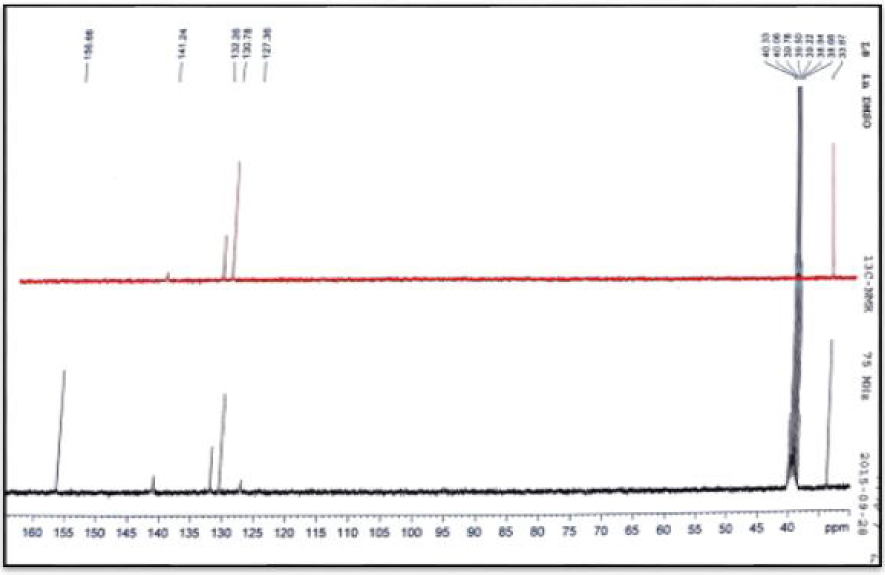


Figure 16: ^{1}H NMR spectrum of 1-methyl-5-imidazolecarboxaldehyde semicarbazone (C_4)

Conclusion

In this study, condensation reaction was adopted for preparing four new imidazolecarboxaldehyde semicarbazone Schiff bases namely; 4-methyl-5-imidazolecarboxaldehyde semicarbazone (C_1), 4-imidazolecarboxaldehyde semicabazone (C_2),1-methyl-2-imidazolecarboxaldehyde semicarbazone (C_3) and 1-methyl-5-imidazolecarboxaldehyde semicarbazone (C_4). These compounds were characterized by using spectroscopic methods (IR, UV-Vis, ESI Mas and ^{1}H, ^{13}C NMR,). The Prepared ligands can be use of future studies, such as metal complexation and biological activities.

Conflicts of Interest

The authors declare that there is no conflict of interests.

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