

Synthesis and Characterization of a Schiff Base and Its Metals (Zn (II) and Mo (VI)) Complexes Derived From 2-Hydroxy-4-Methoxybenzaldehyde

H.A. Usman^{1*}, A. B. Garba², M.A. Sani³, G.A. Yusuf⁴

¹Dept. of Chemistry, Faculty of Science, Federal University Dutse, Jigawa Nigeria

²Dept. of Chemistry, School of Science Education, Sa'adatu Rimi College of Education, Kumbotso, Kano, Nigeria

*Corresponding Author: hadiza4893@yahoo.com (H.U. Abdullahi), +2348053545062

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Abstract- In the present study, a new series of Zinc(II) and Mo (VI) complexes with two newly synthesized Schiff base ligands derived from 2-Hydroxy-4-methoxybenzaldehyde nicotinoylhydrazone monohydrate (H₂hmbn.H₂O) and 2-Hydroxy-4-methoxybenzaldehyde benzoylhydrazone (H₂hmhb) have been prepared. All the two free hydrazones (ligands) that were synthesized acts as a tridentate dianionic ONO donor towards the four complex centers. The conductance measurements in DMF of all the four metal complexes, suggest that they are non-electrolytes. Singlets found in the region 12-14 ppm of the free hydrazones indicates the present of iminol in solution. Also the NMR of zinc complexes was not as a result of its poor solubility. In the FTIR spectra of the free hydrazones, the bands due to carbonyl group were observed in the region 1630-1650 cm⁻¹ and these are absent in the spectra of the complexes suggesting enolization and deprotonation of ligands, whereas for the metal complexes, in compounds AA, AC and AD broad bands observed at 3409 and 3417 cm⁻¹ indicates the present of lattice water. In the thermal analysis, there is a weight loss due to lattice in compound AA, AC and AD.

Keywords: Schiff base, azomethine, complex, molybdenum, Zinc.

I. INTRODUCTION

Coordination chemistry is an important branch of inorganic chemistry. Since the appearance of water on earth, aqua complex ion of metal must have existed [1]. Schiff base are the compound containing azomethine group (-HC=N-) which are condensation products of ketones (or) aldehydes with primary amines [2-3]. Schiff bases of aliphatic aldehydes are relatively unstable and are readily polymerizable while those of aromatic aldehydes or ketones, having an effective conjugation system, are more stable [2]. They have often been used as chelating ligands in coordination chemistry [4-5], they are more effective as a chelating agent when they bear supporting and stabilizing group like -OH close to -HC=N- group [6]. The common Schiff base are crystalline solids, which are feebly basic but at least some form insoluble salts with strong acids. Schiff base are used as intermediates for the synthesis of amino acids or as ligands for Preparation of metal complexes having a series of different structures [3].

Schiff base ligands have significant importance in chemistry, especially in the development of Schiff base complexes, because Schiff base complexes are potentially capable of forming stable complexes with metal ions [3]. Schiff base

compounds and their metal complexes have been extensively investigated due to their wide range of applications including catalysts [3,7,8,9,10,11,12], medicine [9,13,14,15,], crystal engineering [16], anti-corrosion agent [11,17], medicinal chemistry [18,19], electrochemistry [18,20,21], environmental chemistry [20,21,22,23] bioinorganic, separation processes, and metallic deactivators [20,21,]. Schiff bases also have number of applications viz., preparative use, identification, detection and determination of aldehydes or ketones, purification of carbonyl or amino compounds, or protection of these groups during complex or sensitive reactions. They also form basic units in certain dyes [2].

II. MATERIALS AND METHODS

II.1 Chemicals and Solvents:

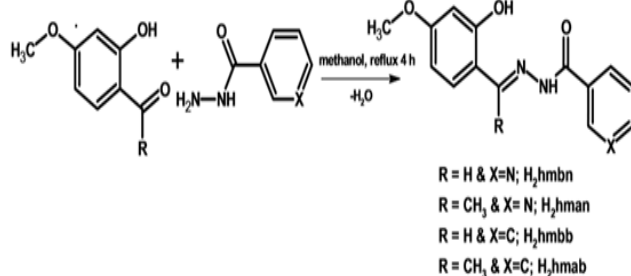
2-hydroxy-4-methoxybenzaldehyde (Aldrich), nicotinic acid hydrazide (Aldrich) and benzhydrazide (Aldrich), MoO₂(acac)₂ (Aldrich) 2,2'bipyridine (LOBA Chemie), 1,10-phenanthroline (LOBA Chemie) and Zn(CH₃COO)₂•2H₂O (S.D.Fine) were used without further purification. Solvents used were ethanol and methanol (Aldrich).

II.2 Physical Measurements

C, H, and N analyses were performed by Perkin-Elmer 2400 instrument. FT-IR spectra (4000-600 cm⁻¹) as KBr discs of the samples were recorded on a JASCO-460 plus spectrophotometer. Electronic spectra were obtained using a CECIL CE7400, 7000 SERIES, UV/VIS spectrophotometer. Magnetic susceptibility was measured with a Sherwood Scientific MSBMK1 sample magnetometer with Hg [Co(SCN)₄] as calibrant.

II.3 Syntheses of Ligands

All the ligands and their complexes were synthesized using a standard procedure as report [24,25]. The scheme for the synthesis of acylhydrazones is shown in scheme below.



Scheme 1- Schematic representation of the syntheses of acylhydrazones.

Benzhydrazide and Nicotinic acid hydrazide were taken in 1:1 ratio and reacted as shown in the **Scheme – 1**. 0.137g (1 mole) of Nicotinic acid hydrazide and 0.136g (1 mole) were separately was refluxed with 2-hydroxy-4-methoxybenzaldehyde (0.152 g, 1 mmol) in methanol in presence of a few drops of glacial acetic acid for 4 hours. The compound formed was filtered, washed and recrystallized using ethanol [26].

2-Hydroxy-4-methoxybenzaldehyde nicotinoylhydrazone monohydrate (H₂hmbn.H₂O): Yield 75%. Elemental Anal. Found (Calcd.) (%) C, 57.79 (58.13); H, 5.54 (5.23); N, 14.49 (14.53).

2-Hydroxy-4-methoxybenzaldehyde benzoylhydrazone (H₂hmbb): Yield 74%. Elemental Anal. Found (Calcd.) (%) C, 66.08 (66.66); H, 5.47 (5.22); N, 10.26 (10.36).

II.4 Syntheses of Zinc (II) and Molybdenum (VI) Complexes

II.4.1 [Zn(hmbn)]₂•H₂O (AA): To a methanolic solution of H₂hmbn•H₂O (0.289 g, 1 mmol), Zn(CH₃COO)₂•2H₂O (0.219 g, 1 mmol) in methanol was added and stirred for two hours. The yellow colored product formed was filtered, washed with methanol, followed by ether and dried over in P₄O₁₀ vacuo. Elemental Anal. Found (Calcd.) (%) C, 49.05 (48.93); H, 3.01 (3.52); N, 12.50 (12.23).

II.4.2 [Zn(Hhmbb)OAc] (AB): To a methanolic solution of H₂hmbb (0.270 g, 1 mmol), Zn(CH₃COO)₂•2H₂O (0.219 g, 1 mmol) in methanol was added. The yellow colored product

formed was filtered washed with methanol, followed by ether and dried over P₄O₁₀ in vacuo. Elemental Anal. Found (Calcd.) (%) C, 52.04 (51.86); H, 3.83 (4.10); N, 7.18 (7.11).

II.4.3 [MoO₂(hmbn)]₂•H₂O (AC) : To a methanolic solution of H₂hmbn.H₂O (0.289 g, 1 mmol), MoO₂(acac)₂ (0.326 g, 1 mmol) in methanol was added. The resulting orange color solution was refluxed for four hours. The orange colored product formed was filtered, washed with methanol, followed by ether and dried over P₄O₁₀ in vacuo. Elemental Anal. Found (Calcd.) (%) C, 41.50 (41.40); H, 2.29 (2.98); N, 10.57 (10.34)

II.4.4 [MoO₂(hmbb)]₂•H₂O (AD) : To a methanolic solution of H₂hmbb (0.270 g, 1 mmol), MoO₂(acac)₂ (0.326 g, 1 mmol) in methanol was added. The solution turns to orange color and it was refluxed for four hours. The orange colored product formed was filtered, washed with methanol, followed by ether and dried over P₄O₁₀ in vacuo. Elemental Anal. Found (Calcd.) (%) C, 44.87 (44.46); H, 3.64 (3.23); N, 6.57 (6.91).

III. RESULT AND DISCUSSION

III.1 Acylhydrazones ligands

The results indicate that the Schiff base and its corresponding Zn (II) and Mo (VI) complexes are colored. The yellow [12] and orange colors are for Zn (II) and Mo (VI) complexes respectively. The change in colour of the Schiff base to yellow and orange was due to complexation which resulted into the formation of coordination compounds [12]. All the synthesized acylhydrazones act as tridentate ligands during the synthesis of their metal complexes.

III.1.1 ¹H NMR spectral studies

The ¹H NMR spectrum of an organic compound provides effective information concerning the number of different types of hydrogen present in the molecule, the relative numbers of the different types of hydrogen, the electronic environment of the different types of hydrogen and also the number of hydrogen 'neighbor' a hydrogen has. Here the ¹H NMR spectra of all the four hydrazones were taken in DMSO.

III.1.1.1 H₂hmbn.H₂O: The ¹H NMR spectrum of the compound is shown in Fig. 1. The two sharp singlet in the very downfield region of the spectrum (ie. at 12.15 ppm and 11.49 ppm) each having a peak area corresponds to one are assigned to iminol OH and phenolic OH protons respectively. The high δ values of these are due to their attachment with highly electronegative elements. Upon deuterium exchange, the intensity of these peaks were considerably reduced confirmed the assignments. Methoxy protons give a sharp singlet at 3.78 ppm with an area integral

of three. The azomethine singlet was observed at 9.08 ppm and aromatic protons give peaks in the region of 6.5 – 9.5 ppm. Figure 1. ^1H NMR spectrum of $\text{H}_2\text{hmbn.H}_2\text{O}$.

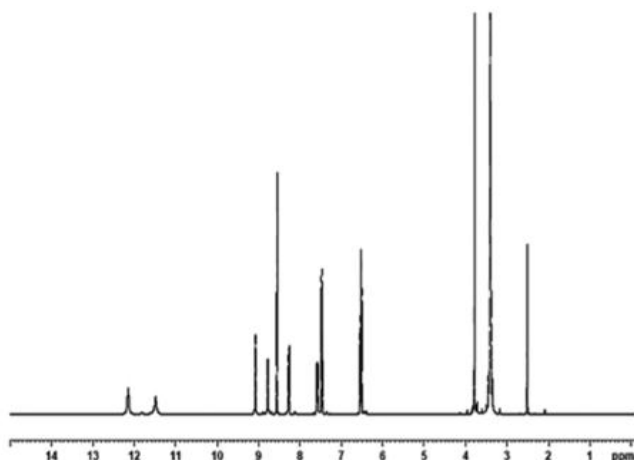


Figure 1: ^1H NMR Spectrum of $\text{H}_2\text{hmbn.H}_2\text{O}$

III.1.1.2 H_2hmbb : Here a singlet peak observed at 11.19 ppm is assigned to phenolic proton and another singlet observed at 13.63 ppm is assigned to proton attached to the iminol oxygen. Both of them have peak area corresponds to one. These assignments are confirmed by deuterium exchange studies. The methoxy protons gave a singlet peak at 3.81 ppm and azomethine proton singlet at 8.01 ppm. Aromatic proton multiplets were observed in between 6-9.5 ppm. Fig. 2 shows the ^1H NMR spectrum of H_2hmbb .

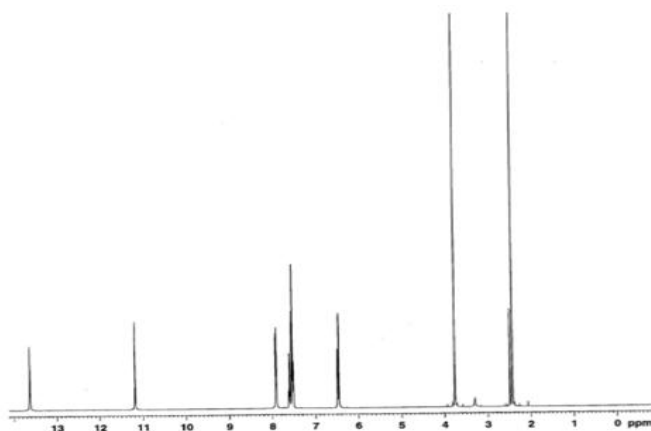


Figure 2: ^1H NMR Spectrum of H_2hmbb

III.1.2 Infrared spectral studies

The characteristic bands of different functional groups provide valuable information regarding the structure. Table 1 below lists the main vibrational bands of the hydrazones. In $\text{H}_2\text{hmbn.H}_2\text{O}$ a band is observed at 3382 cm^{-1} which is due to the presence of lattice water in the molecule. In the other ligand there is no bands in this region due to the absence of water molecule. In all the two hydrazones, a

band observed around 3200 cm^{-1} is assigned to phenolic O—H and N—H groups gave bands around 3050 cm^{-1} . A strong band is observed in between $1630\text{--}1650\text{ cm}^{-1}$ which indicates the presence of carbonyl group in these compounds. The azomethine bands are observed around 1600 cm^{-1} which is in tandem with the findings of [27], and slightly below the finding of [12,28], which was within the range of $1615.90\text{--}1614.80\text{ cm}^{-1}$. The C—O stretching is observed around 1275 cm^{-1} . The N—N stretching vibrations are found in between $1110\text{ and }1130\text{ cm}^{-1}$. Figs. 3-4 show the infrared spectra of the two hydrazones.

Table 1: Selected IR frequencies (cm^{-1}) of the Acylhydrazones.

Ligand	$\nu(\text{O-H})$	$\nu(\text{N-H})$	$\nu(\text{C=O})$	$\nu(\text{C=N})$	$\nu(\text{N-N})$	$\nu(\text{C-O})$
$\text{H}_2\text{hmbn.H}_2\text{O}$	3371, 3208	3074	1643	1604	1110	1284
H_2hmbb	3224	3038	1630	1600	1130	1286

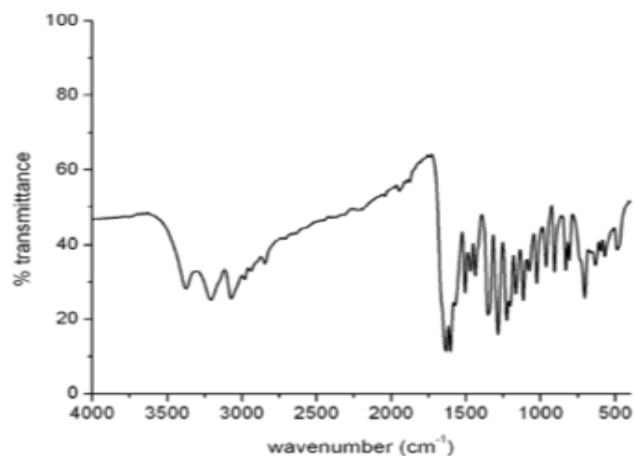


Figure 3: IR Spectrum of $\text{H}_2\text{hmbn.H}_2\text{O}$

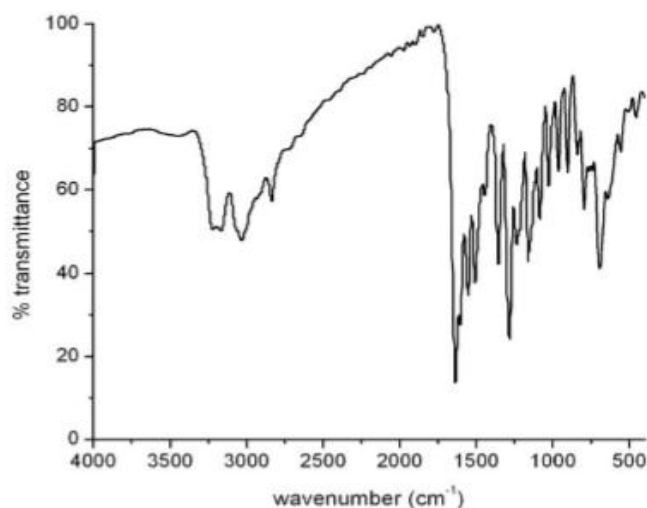


Figure 4: IR Spectrum of H_2hmbb

III.2 Zinc (II) Complexes and Molybdenum (VI) Complexes

III.2.1 Molar Conductivity

The experimental and calculated analytical data of all the four Zn (II) and Mo (VI) complexes are in close agreement. Magnetic susceptibility studies indicate diamagnetic nature of these complexes and it indicates the d^{10} outer electronic configuration of zinc, it is also an evidence for molybdenum is in +6 oxidation state. The molar conductivity measured for all the four complexes in 10^{-3} M DMF solution was found to be below $15 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$ which is much less than the value of $65\text{-}90 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$ obtained for a 1:1 electrolyte in the same solvent [29]. So the conductance measurements in DMF suggest that they are non-electrolytes. The table 2 below present the molar conductivity measurement.

Table 2: Molar conductivity measurements

AA	[Zn(hmbn)] ₂ .H ₂ O	11
AB	[Zn(hmbb)OAc]	12
AC	[MoO ₂ (hmbn)] ₂ .H ₂ O	03
AD	[MoO ₂ (hmbb)] ₂ .H ₂ O	05

#molar conductivity (in mho $\text{cm}^2 \text{ mol}^{-1}$) taken in 10^{-3} DMF

III.2.2 Infrared spectra

The IR spectra of the complexes, in comparison with those of the free ligands, display certain changes, which give an idea about the type of coordination and their structure. Significant IR spectral bands of the complexes are listed in Table 3 and 4. Free hydrazones show strong absorptions in the $1630\text{-}1650 \text{ cm}^{-1}$ region which are assigned to carbonyl group, and bands due to azomethine group were observed around 1600 cm^{-1} . In compound 1, broad bands were observed at 3409 and 3417 cm^{-1} due to lattice water. Coordination of hydrazones to the zinc ion through the azomethine nitrogen atom is expected to reduce the electron density in the azomethine link and thus, lower the $\nu(\text{C}=\text{N})$ absorption frequency. Hence this band undergoes a shift to lower wavenumber [29]. In complexes 1 and 2, a lower shift is observed in $\nu(\text{C}=\text{N})$ absorption frequency indicates coordination of azomethine nitrogen to zinc. Coordination of azomethine nitrogen is also evident from the increase in N–N stretching frequency in all the complexes. In complexes AA and AB, bands correspond to carbonyl groups are absent suggesting the enolization of ligands during complexation. For all the complexes, phenolic C–O stretching occurs at lower wavenumber when compared to that of the ligands indicating the deprotonation and coordination of phenolic OH. Appearance of new bands in the regions of $525\text{-}560$ and $440\text{-}460 \text{ cm}^{-1}$ are assignable to $\nu(\text{Zn}-\text{O})$ and $\nu(\text{Zn}-\text{N})$ respectively. The IR spectra of the complexes are shown in the Figs. 5-6.

Table 3: Selected IR bands (cm^{-1}) with speculative assignments of Zn(II) complexes (newly formed)

Compound	$\nu(\text{C}=\text{O})$	$\nu(\text{C}=\text{N})^a$	$\nu(\text{N}-\text{N})$	$\nu(\text{C}-\text{O})$	$\nu(\text{Zn}-\text{O})$	$\nu(\text{Zn}-\text{N})$
[Zn(hmbn)] ₂ .H ₂ O (AA)	1597	1527	1167	1244	527	454
[Zn(hmbb)OAc] (AB)	1600	1540	1124	1247	560	460

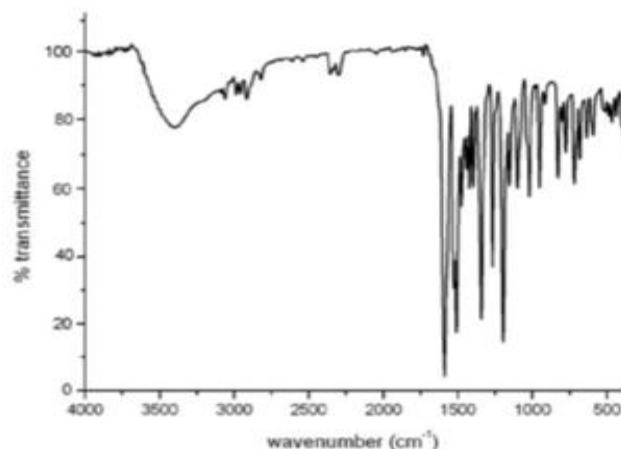


Figure 5: IR-Spectrum of [Zn(hmbn)]₂.H₂O (AA)

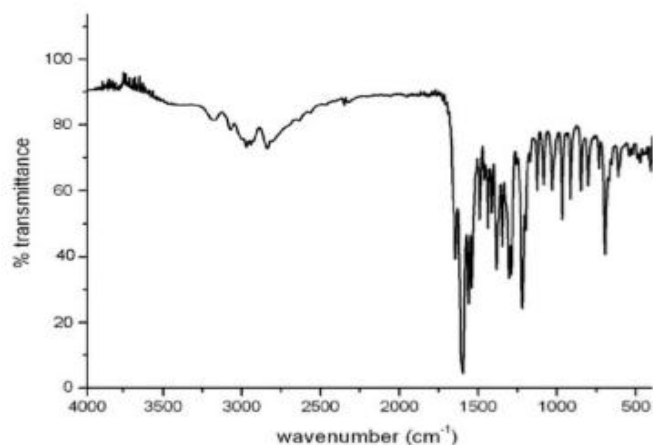


Figure 6: IR-Spectrum of [Zn(hmbb)OAc] (AB)

Table 4: Selected IR bands (cm^{-1}) with speculative assignments of Mo(VI) complexes (newly form)

Compound	$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{N})^2$	$\nu(\text{N}-\text{N})$	$\nu(\text{C}-\text{O})$	$\nu(\text{Mo}-\text{O})$	$\nu(\text{Mo}-\text{O}-\text{OMo})$
[MoO ₂ (hmbn)] ₂ .H ₂ O (AC)	1589	1540	1129	1225	903,937	833
[MoO ₂ (hmbb)] ₂ .H ₂ O (AD)	1596	1535	1138	1230	910,930	850

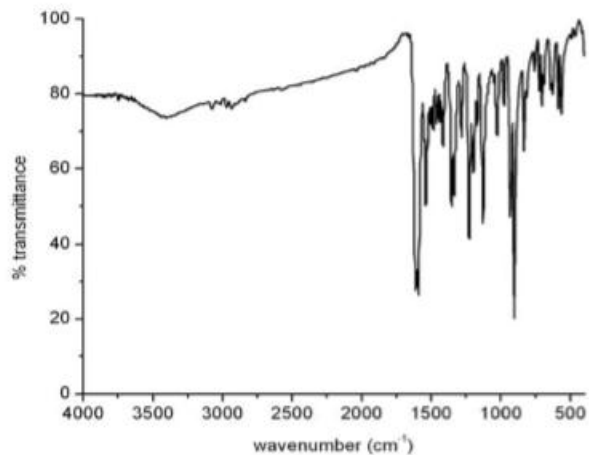


Figure 7: IR-Spectrum of $[\text{MoO}_2(\text{hmbn})]_2 \cdot \text{H}_2\text{O}$ (AC)

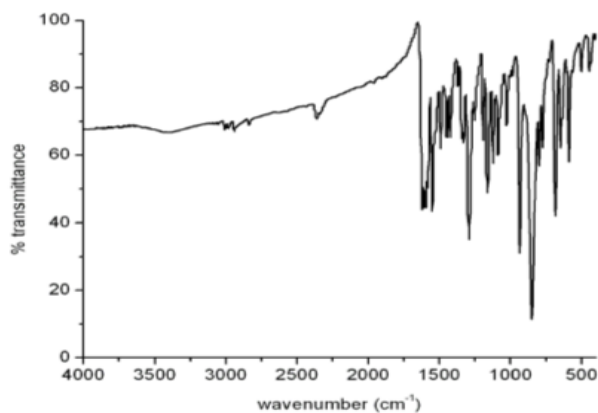


Figure 8: IR-Spectrum of $[\text{MoO}_2(\text{hmbb})]_2 \cdot \text{H}_2\text{O}$ (AD)

III.2.3 ^1H NMR spectral studies

In the case of diamagnetic Mo (VI) complexes the coordination of acylhydrazones to the metal centre is substantiated by ^1H NMR spectral studies of the ligands and complexes, in DMSO solution. Singlets found in the region 12-14 ppm due to iminol protons in free hydrazones were not found in the spectra of complexes indicate the coordination of iminol oxygen to metal center. Similarly singlets due to OH proton were found in-between 11 ppm and 12 ppm in free hydrazones were also absent in the spectra of complexes indicate the coordination of phenolic oxygen to metal centre. This leads to the conclusion that the hydrazones are coordinated in all the complexes in a dianionic form suggesting the ONO coordination to molybdenum. The singlets with an area integral of three in the range of 3-3.8 ppm indicate the presence of three methoxy hydrogens in free acylhydrazones were slightly shifted upon complexation. Multiplets for aromatic protons were found in the region 6-8 ppm in ligands do not show much shift in complexes. The ^1H NMR spectra of the complexes are shown in the Figs 9-10.

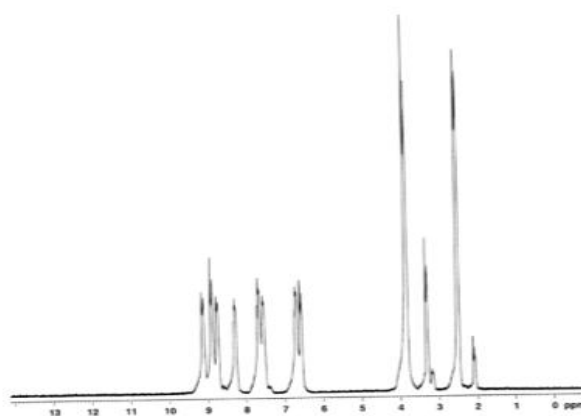


Figure 9: ^1H NMR Spectra of $[\text{MoO}_2(\text{hmbn})]_2 \cdot \text{H}_2\text{O}$ (AC)

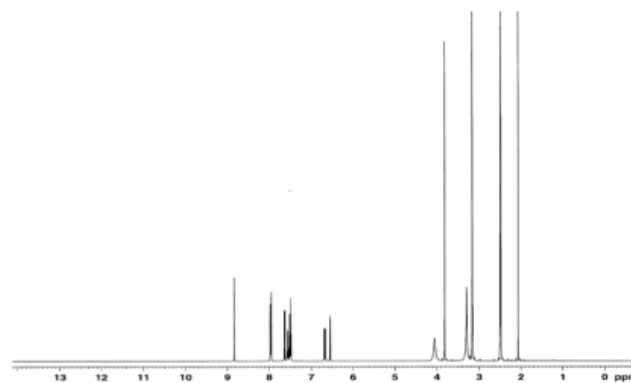


Figure 10: ^1H NMR Spectra of $[\text{MoO}_2(\text{hmbb})]_2 \cdot \text{H}_2\text{O}$ (AD)

III.2.4 Thermal analyses

In complex AA, there is a weight loss below 120 °C indicating the presence of lattice water. In complex AB, no weight loss observed in between 50-250 °C indicating absence of water molecules in this complex. Above 400 °C the complexes begin to decompose and the decomposition was not seen to be completed even at 1000 °C. TG – DTG plots of complexes AA and AB are shown in Figs. 11 and 12.

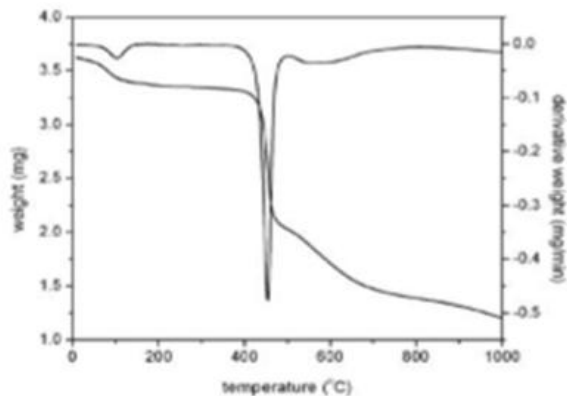


Figure 11: TG-DTG plot of $[\text{Zn}(\text{hmbn})]_2 \cdot \text{H}_2\text{O}$ (AA)

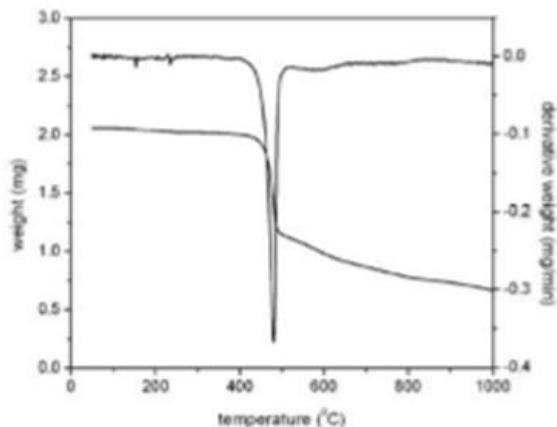


Figure 12: TG-DTG plot of $[Zn(hmbb)OAc]$ (AB)

In molybdenum, the complexes AC and AD showed loss of weight corresponds to one water molecule in the temperature range 80-110 °C indicate the presence of lattice water in these complexes. All the complexes decompose over 250 °C. TG-DTG curves of the complexes are shown in Figs. 13-14.

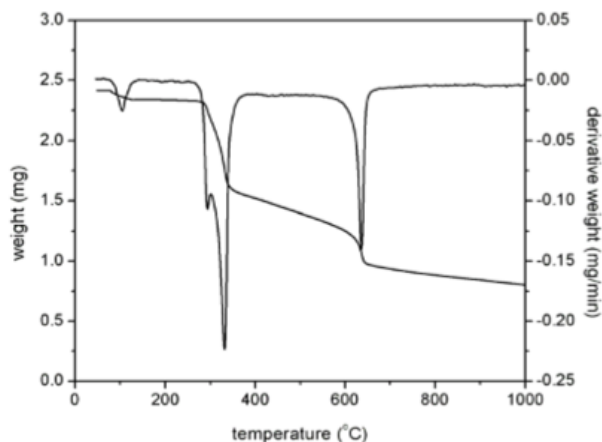


Figure 13: TG-DTG plot of $[MoO_2(hmbn)]_2 \cdot H_2O$ (AC)

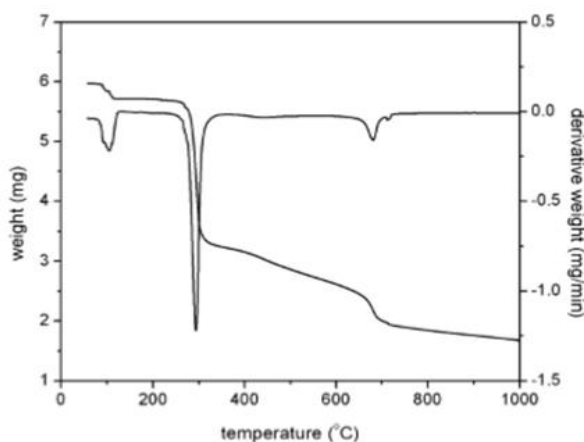


Figure 14: TG-DTG plot of $[MoO_2(hmbb)]_2 \cdot H_2O$ (AD)

IV. CONCLUSION

Acyhydrazones are known to be a class of versatile ligands, capable of generating various molecular architectures and coordination polyhedra and the study of the synthesis, structures and properties of transition metal complexes is a key line of research in modern coordination chemistry. The four new complexes of zinc and molybdenum prepared using different acylhydrazones which were derived from 2-hydroxy-4-methoxybenzaldehyde, nicotinic acid hydrazide and benzhyrazide, were characterized. All the two free ligands that were synthesized acts as a tridentate dianionic ONO donor towards the four complex centers. The conductance measurements in DMF of all the four metal complexes, suggest that they are non-electrolytes. Singlets found in the region 12-14 ppm of the free hydrazones indicates the present of iminol in solution. Also the NMR of zinc complexes was not as a result of its poor solubility. In the FTIR spectra of the free hydrazones, the bands due to carbonyl group are observed in the region 1630-1650 cm^{-1} and these are absent in the spectra of the complexes suggesting enolization and deprotonation of ligands, whereas for the metal complexes, in compounds AA, AC and AD broad bands observed at 3409 and 3417 cm^{-1} indicates the present of lattice water. In the thermal analysis, there is a weight loss due to lattice in compound AA, AC and AD.

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