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Synthesis and Characterization of Cu(II), Co(II), Ni(II), and Zn(II) Complexes of a Schiff Base Derived from 1-Amino-5-benzoyl-4-phenyl-1H-pyrimidine-2-one and 3-Hydroxysalicylaldehyde

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Synthesis and Characterization of Cu(II), Co(II), Ni(II), and Zn(II) Complexes of a Schiff Base Derived from 1-Amino-5-benzoyl-4-phenyl-1H-pyrimidine-2-one and 3-Hydroxysalicylaldehyde

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ABSTRACT

Cu(II), Co(II), Ni(II) and Zn(II) metal complexes of a new heterocyclic Schiff base derived from 1-amino-5-benzoyl-4-phenyl-1H-pyrimidine-2-one with 3-hydroxy-salicylaldehyde have been synthesized and characterized on the basis of elemental analyses, electronic, IR and ¹H NMR spectra, molar conductivity measurements, magnetic moment measurements and thermogravimetric analyses (TGA). It has been found that the Schiff base behaves as a neutral bidentate (NO) or tridentate (ONO) ligand forming chelates with 1:2 (metal:ligand) stoichiometry and

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octahedral geometry. The conductivity data for the Ni(II) complex is consistent with that expected for a weak electrolyte.

Key Words: N-Aminopyrimidine; 3-Hydroxysalicylaldehyde; Metal complexes.

INTRODUCTION

Metal complexes of Schiff bases have been described in a huge number of publications ranging from the purely synthetic to modern physico-chemically to biochemically relevant studies of these complexes. Schiff bases are compounds containing the azomethine group ($\text{RC}=\text{N}-$) and are usually formed by the condensation of a primary amine with an active carbonyl compound. Bidentate or tridentate Schiff bases derived from substituted salicylaldehyde derivatives and their transition metal complexes have been studied for many years.^[1–5] In recent years, metal complexes of Schiff bases have attracted considerable attention due to their remarkable antifungal, antibacterial and antitumor activity.^[6,7] Schiff base complexes derived from 4-hydroxysalicylaldehyde and amines have strong anticancer activity.^[8] Transition metal cation compounds with pyrimidine derivatives of nucleic acids (such as uracyl, cytosine and thymine) were also of special interest.^[9]

The present paper reports the synthesis of a new Schiff base ligand (Figure 1) containing a pyrimidine ring and its metal complexes. Spectral and magnetic studies have been used to characterize the structure of each of the complexes. ^1H NMR spectra were obtained to determine the structure of the ligand LH_2 .

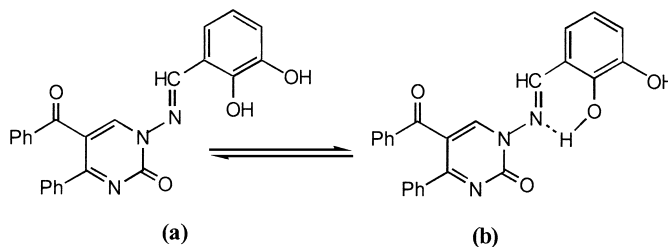


Figure 1. Structure of the ligand LH_2 .

EXPERIMENTAL

Materials and Methods

MeOH, EtOH, CHCl_3 , DMF, toluene, diethyl ether, *n*-heptane and 3-hydroxy-salicylaldehyde were obtained from Fluka or Aldrich. The metal salts $\text{Cu}(\text{OOCCH}_3)_2 \cdot \text{H}_2\text{O}$, $\text{Co}(\text{OOCCH}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Zn}(\text{OOCCH}_3)_2 \cdot 2\text{H}_2\text{O}$ and $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ were obtained from E. Merck. All solvents were dried and purified before use. 1-Amino-5-benzoyl-4-phenyl-1H-pyrimidine-2-one was prepared as described previously.^[10]

Elemental analyses (C, H, N) were performed using a Carlo Erba 1106 elemental analyser. IR spectra were obtained using KBr discs (4000–400) cm^{-1} on a Bio-Rad-Win-IR spectrophotometer. Electronic spectra in the 200–900 nm range were obtained in DMF on a Unicam UV2-100 UV/Visible spectrophotometer. Magnetic measurements were carried out by the Gouy method using $\text{Hg}[\text{Co}(\text{SCN})_4]$ as calibrant. Molar conductances of the Schiff base ligand and their transition metal complexes were determined in DMF at room temperature using a Jenway model 4070 conductivity meter. The ^1H NMR spectrum of the Schiff base was recorded with a Varian XL-200 NMR instrument. TGA measurements were carried out using a Shimadzu-50 thermal analyzer.

Preparation of the Schiff Base (LH_2)

The Schiff base ligand was prepared by the condensation of the 1-amino-5-benzoyl-4-phenyl-1H-pyrimidine-2-one (0.291 g, 1.00 mmole) with 3-hydroxysalicylaldehyde (0.138 g, 1.00 mmole) in ethanol (40 mL) by boiling the mixture under reflux for 2 h. The precipitated ligand was filtered, recrystallized from EtOH and dried in a vacuum desiccator.

Preparation of the Schiff Base Complexes

The ligand (0.82 g, 2.0 mmole) was dissolved in 30 mL of chloroform, and a solution of 1.0 mmole of the metal salt [$\text{Cu}(\text{OOCCH}_3)_2 \cdot \text{H}_2\text{O}$ (0.20 g), $\text{Co}(\text{OOCCH}_3)_2 \cdot 4\text{H}_2\text{O}$ (0.25 g), $\text{Zn}(\text{OOCCH}_3)_2 \cdot 2\text{H}_2\text{O}$ (0.22 g) and $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (0.24 g)] in 15 mL methanol was added dropwise with continuous stirring. The mixture was stirred further for 1.5–2.5 h. at 80 °C. The precipitated solid was then filtered off, washed with diethyl ether, followed by cold ethanol and dried in a vacuum desiccator.



Table 1. Analytical and physical data of the ligand and complexes.

Compounds (FW)	Yield %	Color	M.p (°C)	Elemental analyses found (calcd.)%			Λ_M (S cm ² mol ⁻¹)	μ_{eff} [B.M]
				C	H	N		
LH ₂ -C ₂₄ H ₁₇ N ₃ O ₄ (411.00)	80	Yellow	218	70.0 (70.0)	4.3 (4.1)	10.1 (10.2)	1.4	—
[Cu(LH) ₂].2H ₂ O- C ₄₈ H ₃₆ CuN ₆ O ₁₀ (919.54)	65	Brown	210	62.3 (62.6)	3.8 (3.9)	9.0 (9.1)	4.6	2.20
[Co(LH) ₂].H ₂ O- C ₄₈ H ₃₄ CoN ₆ O ₉ (896.93)	68	Dark- brown	293	63.1 (64.2)	3.5 (3.7)	9.1 (9.3)	1.5	3.84
[Ni(LH ₂)Cl ₂].2H ₂ O- C ₄₈ H ₃₈ Cl ₂ N ₆ NiO ₁₀ (987.71)	60	Orange- red	225	58.3 (58.3)	3.7 (3.8)	8.6 (8.5)	17.0	3.40
[Zn(LH ₂) ₂].H ₂ O- C ₄₈ H ₃₄ ZnN ₆ O ₉ (903.37)	63	Red	250	63.6 (63.7)	3.5 (3.7)	9.1 (9.3)	1.2	Dia

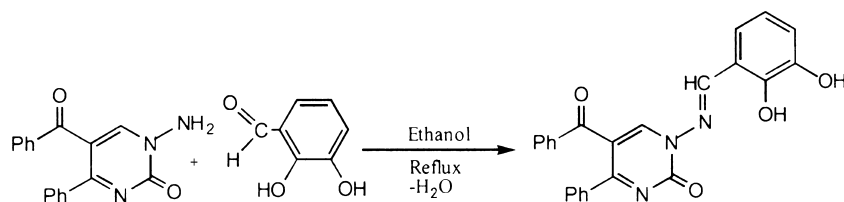


Figure 2. Synthesis scheme for the preparation of the ligand LH_2 .

RESULTS AND DISCUSSION

The newly synthesized Schiff base ligand and its complexes are very stable at room temperature in the solid state. The ligand is soluble in common organic solvents. However, its metal complexes are generally soluble in DMF and DMSO. The colors, yields, melting points, elemental analyses and molar conductances of all the compounds are presented in Table 1. The analytical data are in good agreement with the proposed stoichiometry of the complexes. Condensation of 3-hydroxysalicylaldehyde with 1-amino-5-benzyl-4-phenyl-1H-pyrimidine-2-one readily gives the corresponding imine, which is easily identified by its IR, ^1H NMR spectra (Figure 2).

Tridentate and bidentate complexes were obtained from reaction of a 1:2 molar ratio of metal ions and LH_2 ligand. The ligand LH_2 , on reaction with $\text{Co}(\text{OOCCH}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Cu}(\text{OOCCH}_3)_2 \cdot \text{H}_2\text{O}$, $\text{Zn}(\text{OOCCH}_3)_2 \cdot 2\text{H}_2\text{O}$, and $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ yields complexes corresponding to the general formulas $[\text{Co}(\text{LH}_2)] \cdot \text{H}_2\text{O}$, $[\text{Cu}(\text{LH}_2)] \cdot 2\text{H}_2\text{O}$, $[\text{Zn}(\text{LH}_2)] \cdot \text{H}_2\text{O}$, and $[\text{Ni}(\text{LH}_2)_2\text{Cl}_2] \cdot 2\text{H}_2\text{O}$ respectively. The analytical data for all of the complexes are presented in Tables 1 and 2. The complexes were prepared by the general reaction shown below.

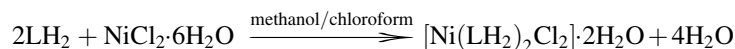
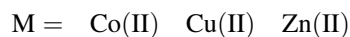
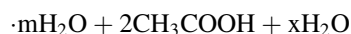
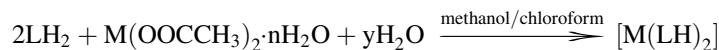


Table 2. Characteristic IR (cm^{-1}) and electronic absorption spectral data of the ligand and complexes.

Compounds	C=O	C=N	C-O	M-N	M-O	$\lambda_{\text{max}}(\text{nm})$ ($\epsilon_{\text{max}}, \text{M}^{-1} \text{cm}^{-1}$)
LH ₂	1691–1646 m,s	1622 s	1273 s	–	–	385 (4850), 338 (24740)
[Cu(LH) ₂].2H ₂ O	1653 m	1612 m	1266 m	495 w	428 w	743 (15), 590 (187), 497 (360), 350 (9800), 298 (23200)
[Co(LH) ₂].H ₂ O	1658 s	1620 m	1260 m	485 w	420 w	760 (7), 523 (480), 357(11500), 287 (18600), 261(21200)
[Ni(LH ₂) ₂ Cl ₂].2H ₂ O	1682–1650 m	1610 m	1273 m	485 w	423 w	673 (28), 517(120), 308 (17000)
[Zn(LH) ₂].H ₂ O	1660 m	1616 m	1270 m	476 w	439 w	393 (8500), 359 (12700), 285 (21000), 260 (28200)

s = strong, m = medium, w = weak.

Infrared Spectral Study

Table 2 presents the most important IR spectral bands of the ligand LH₂ in all the metal complexes. The IR spectra of the Schiff base ligand LH₂, showed a broad band at 3616 cm⁻¹ which may be due to $\nu(\text{OH})$. The free $\nu(\text{OH})$ is generally observed^[11–13] between 3500–3650 cm⁻¹. The observed low frequency of this band is due to intramolecular hydrogen bonding between H of OH and the azomethine nitrogen suggesting the presence of structure **b** (see Figure 1) at least in the solid state.

The intense band at 1273 cm⁻¹ present in the IR spectrum of the Schiff base ligand may be assigned to the phenolic (C–O) stretching mode, according to previous assignments. The pyrimidine ring shows characteristic stretching absorption bands at 3060 cm⁻¹. The phenyl group shows C–H stretching at 3030 cm⁻¹ and C=C stretching at 1540 cm⁻¹. The bands at 1590 cm⁻¹ can be very safely assigned to $\nu(\text{C}=\text{N})$ (pyrimidine).^[10] Strong bands at 1691–1646 and 1622 cm⁻¹ in the IR spectra of the free ligand assigned to $\nu(\text{C}=\text{O})$ ^[15] and $\nu(\text{C}=\text{N})$ ^[11,15,16] are shifted by ± 10 –30 cm⁻¹ in the spectra of complexes, indicating coordination through the azomethine nitrogen, phenolic oxygen and carbonyl oxygen of the Schiff base (Figure 2). In the spectra of a metal complex such as that of Ni(II), the band at 1273 cm⁻¹ for the phenolic group did not shift, suggesting that this oxygen atom of the phenolic group is not coordinated to the metal ion.^[15,16] In the spectra of the Cu(II), Ni(II), Co(II) and Zn(II) complexes, the bands observed in the 495–476 and 420–439 cm⁻¹ region may be due to $\nu(\text{M}-\text{N})$ and $\nu(\text{M}-\text{O})$, respectively,^[13] suggesting that they are bidentate complexes with the oxygen atom of the carbonyl group and the nitrogen

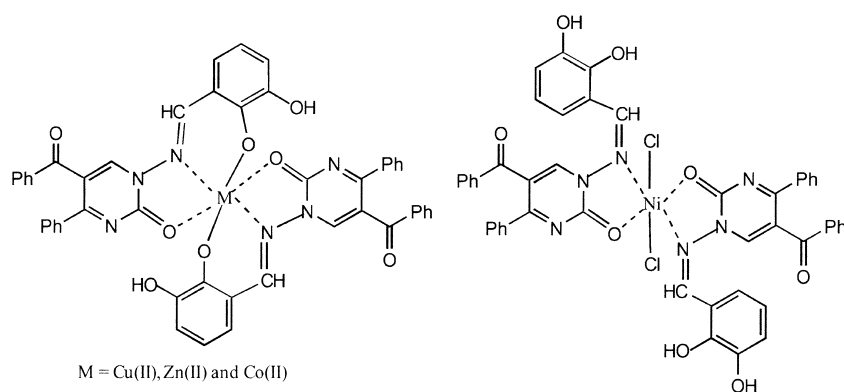


Figure 3. Suggested structures of the octahedral complexes of the ligand LH₂.



atom of the azomethine group coordinated to the metal ion (Figure 3). Broad bands of the Cu(II), Ni(II), Co(II) and Zn(II) complexes in the 3240–3350 cm^{-1} region are assigned to the $\nu(\text{OH})$ vibration of water molecules.^[13]

Proton Nuclear Magnetic Resonance Spectra

Deuterated chloroform was used as a solvent to measure the ^1H NMR spectra of the ligand and its Zn(II) complex but not those of Cu(II), Co(II) and Ni(II), because of their paramagnetic behaviour. The sharp singlet observed at about δ 10.43 and 12.55 ppm is assigned to the phenolic protons of the ligand. The singlets at δ 9.56 ppm and 8.33 ppm, respectively, are assigned to the azomethine proton and the pyrimidine ring (C–H) proton in the spectrum of the ligand. In the spectrum of the Schiff base, the phenyl multiplet was observed between at δ 6.89–7.71 ppm. The ^1H NMR spectra of Zn(II) complex shows approximately the same peaks as those of the free ligand with the exception that the peak due to the (2-OH) phenolic group resonance is absent. This is considered to be additional evidence for the deprotonation of the phenolic OH group.

Electronic Spectra and Magnetic Measurements

The electronic absorption data of the compounds, which were measured in DMF, are given in Table 2. Because the complexes are insoluble in all of the other polar organic solvents, the spectra were recorded in DMF as solvent. In the spectrum of the ligand, the bands in the 390–348 nm range are assigned to the $n \rightarrow \pi^*$ transitions of the azomethine group. During the formation of the complexes, these bands are shifted to lower wavelength, suggesting that the nitrogen atom of the azomethine group is coordinated to the metal ion. The values in the 300–250 nm range are attributed to the $\pi \rightarrow \pi^*$ transition of the aromatic and pyrimidine rings. In the spectra of the complexes, these bands are shifted slightly to lower wave length.

The electronic spectrum of the Zn(II) complex has bands in the 280–250 nm range and these bands may be due to the $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ transitions of the benzene and pyrimidine rings as well as the azomethine group. In the spectra of the other complexes, the less intense and broad bands in the 400–260 nm range result from the overlap of the low-energy $\pi \rightarrow \pi^*$ transitions which are mainly localized within the imine chromophore and the LMCT (ligand to metal charge-transfer bands) transition from the lone pairs of the phenolate oxygen donor to the M(II) ions.

The Cu(II) complex exhibits a tetragonally distorted octahedral geometry. It shows intense absorption bands in the 298 and 367 nm ranges

which can be assigned to the $\pi \rightarrow \pi^*$ transition of the azomethine group and charge-transfer bands, respectively. The electronic spectrum of the complex exhibits an absorption band in the range 743 nm, attributed to ${}^2E_g \rightarrow {}^2T_{2g}$ transition suggesting a distorted octahedral geometry. The other bands in the range 590 and 497 nm can be assigned to the transitions: ${}^2B_{1g} \rightarrow {}^2B_{2g}$ and ${}^2B_{1g} \rightarrow {}^2E_g$ in order of increasing energy. But generally, such chelates exhibit a broad, structureless band with or without a shoulder between 715–520 nm depending upon the strength of the field of the ligand. The obtained value of the effective magnetic moment (2.2 B.M.) is typical for distorted octahedral Cu(II) chelates.

$[Ni(LH_2)_2Cl_2] \cdot 2H_2O$ shows d-d transitions at 517 and 673 nm corresponding to ${}^3A_{2g} \rightarrow {}^3T_{1g}(F)$ and ${}^3A_{2g} \rightarrow {}^3T_{2g}(F)$, respectively, which suggests octahedral geometry. The magnetic moment values for the Ni(II) complex of the Schiff base ligand at room temperature was found to be 3.40 B.M. value is in the normal range (2.8–3.8 BM).^[19]

The magnetic moment values for the $[Co(LH)_2]$ complex is near the spin-only value for high-spin octahedral complexes. The electronic spectrum shows two bands at 760 and 523 nm attributed to ${}^1T_{1g} \rightarrow {}^4A_{2g}(v_2)$ and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)(v_3)$ transitions, respectively, in an octahedral geometry around the Co(II) ion.^[19] The band observed at 261–357 nm is assigned to the $\pi \rightarrow \pi^*$ transition of the azomethine group and charge-transfer bands. The Zn(II) complex is diamagnetic as expected for its d^{10} configuration.

The metal-to-ligand ratio of all the complexes was found to be 1:2. The Zn(II) and Cu(II) complexes have, respectively, one and two additional molecules of water of crystallization. The metal-to-ligand ratio of the Ni(II) complex was found to be 1:2, in addition to two chloride ligands and two additional molecules of water of crystallization.

Molar conductance value of the Ni(II) complex is $17.00 \Omega^{-1} \text{ cm}^2 \text{ mole}^{-1}$. The Ni(II) complex, which contains coordinated chloride ions, is non-conducting or shows an increasing degree of conductivity with time which would be due to their replacements by DMF solvent molecules.^[2,9] The other complexes were non-electrolytes.

Single crystals of the complexes could not be isolated from any solutions, thus no definitive structure can be described. However, the analytical, spectroscopic and magnetic data enable us to suggest possible structures as shown in Figure 3.

Thermal Studies

The thermal stability of the complexes was investigated using thermal gravimetric analysis. The TGA curves were obtained at a heating rate of $10^\circ\text{C}/\text{min}$. in N_2 atmosphere over the temperature range at $30\text{--}850.0^\circ\text{C}$.



The Cu(II) complex was stable up to 32.0 °C and its decomposition started at this temperature and was completed at 551.0°C. The Cu(II) complex decomposed and produced CuO as residue [calculated (found) %: 8.65 (9.10)] in three steps in the temperature ranges 32.0–220.0, 220.0–449.0 and 449.0–551.0 °C. In the decomposition process of the Cu(II) complex, the mass losses corresponded to H₂O, Ph-C=O- and the other organic moieties leaving in the first, second and third stages of the decomposition, respectively. The three stages of the decomposition of the Cu(II) complex were irreversible.

The Ni(II) complex was stable up to 38.0 °C and its decomposition started at this temperature and was completed at 672.0 °C. The Ni complex decomposed and produced NiO as residue [found (calculated) %: 9.16 (7.56)] in four steps in the temperature ranges 38.0–148.0, 148.0–280.0, 280.0–592.0 and 592.0–672.0 °C. In the decomposition process of the Ni(II) complex, the mass losses corresponded to H₂O, Cl, Ph and the other organic moieties leaving in the first, second, third and fourth stages of the decomposition, respectively. The four stages of decomposition of Ni(II) complex were irreversible.

The Co(II) complex was stable up to 68.0 °C and its decomposition started at this temperature and was completed at 570.0 °C. The Co(II) complex decomposed and produced CoO as residue [found (calculated) %: 8.68 (8.52)] in three steps in the temperature ranges 68.0–270.0, 270.0–443.0 and 443.0–570.0 °C. In the decomposition process of the Co(II) complex, the mass losses corresponded to H₂O, Ph-C=O- and other organic moieties leaving in the first and second stages of the decomposition, respectively. The two stages of the decomposition of the Co(II) complex were irreversible.

The Zn(II) complex was stable up to 30.0 °C and its decomposition started at this temperature and was completed at 590.0 °C. The Zn(II) complex decomposed and produced ZnO as residue [found (calculated) %: 10.02 (9.01)] in three steps in the temperature ranges 30.0–260.0, 260.0–478.0 and 478.0–590.0 °C. In the decomposition process of the Zn(II) complex, the mass losses corresponded to H₂O, Ph-C=O- and the other organic moieties leaving in the first, second and third stages of the decomposition, respectively. The three stages of decomposition of the Zn(II) complex were irreversible.

The Co(II) complex is thermally stable up to 68.0 °C. In the TGA curve of the Zn(II) complex, 1.99% weight loss was observed at 260.0 °C. In the TGA curve of the Cu(II) complex, 3.92% weight loss was observed at 220.0 °C. In the TGA curve of the Ni(II) complex, 3.64% weight loss was observed at 148.0 °C. These observations show that the complexes contain 1, 2 and 1 mols of water of crystallization per complex molecule,

Table 3. TGA data of the complexes [weight loss %, found (calculated)].

Compounds	First step, (°C)	Second step, (°C)	Third step, (°C)	Fourth step, (°C)	Residue, found (calcd.) %
[Cu(LH) ₂].2H ₂ O	32.0–220.0 4.57 (3.92)	220.0–449.0 22.41 (22.54)	449.0–551.0 – 64.57 (64.24)	–	9.10 (8.65) CuO 90.90 (91.35)*
[Ni(LH) ₂ Cl ₂].2O.2H ₂ O .2H	38.0–148.0 3.98 (3.64)	148.0–280.0 7.34 (7.19)	280.0–592.0 29.86 (31.18)	592.0–672.0 49.66 (50.43)	9.16 (7.56) NiO 90.84 (92.44)*
[Co(LH) ₂].H ₂ O	68.0–270.0 1.76 (2.00)	270.0–443.0 24.06 (23.40)	443.0–570.0 65.50 (66.08)	–	8.68 (8.52) CoO 91.32 (91.48)*
[Zn(LH) ₂].H ₂ O	30.0–260.0 1.88 (1.99)	260.2–478.0 23.20 (23.25)	478.0–590.0 64.90 (65.75)	–	10.02 (9.01) ZnO 89.98 (90.99)*

*Total values.

respectively. The IR spectra of the complexes include a broad band in the region $3245\text{--}3360\text{ cm}^{-1}$ due to the $\nu(\text{O--H})$ frequency of water of crystallization.^[14–18,20] This water content was also identified by the elemental analyses. This decomposition process of the Cu(II), Ni(II), Co(II) and Zn(II) complexes was also confirmed by TGA data. As can be seen in Table 3, the most stable complex is the Co(II) complex. The weight losses for the Cu(II), Ni(II) Co(II) and Zn(II) complexes were found to be approximately the same, when expressed as the percentages calculated stoichiometrically from their chemical formulas given in Tables 1 and 3. All of these complexes undergo complete decomposition to the corresponding metal oxides, CuO, NiO, CoO or ZnO^[14–16,21–36] (Table 3).

CONDUCTIVITY

The complexes are non-electrolytes as shown by their molar conductivity (Λ_M) in DMF, which are in the range^[2,8,14–36] $1.5\text{--}17.0\text{ }\Omega^{-1}\text{ cm}^2\text{ mol}^{-1}$.

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