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SYNTHESIS AND CHARACTERIZATION OF SCHIFF BASE METAL COMPLEXES DERIVED FROM NAPHTHO [2,1-B] FURAN-2-CARBOHYDRAZIDE AND INDOLINE-2,3-DIONE (ISATIN)

Chemistry

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ABSTRACT

New Schiff base metal complexes having general structural formulae ML2Cl2 where M= Co(II), Ni(II) and Cu(II), and MLCl2 where M=Zn(II), Cd(II) and Hg(II) were synthesized by the condensation of indoline-2,3-dione (Isatin) and naphtho[2,1-b]furan-2-carbohydrazide. Tentative structures for the synthesized compounds have been elucidated based on analytical, IR, 1HNMR, and Mass spectral studies. Further selected compounds were tested for their antimicrobial activity.

KEYWORDS

Schiff base, Ligand, Complexes, Spectroscopy.

INTRODUCTION

Benzofuran derivatives are found everywhere in nature, they mainly occur in Plantae having sort of industrial, agricultural, pharmaceutical, and biological importance.[1]–[4]. Numerous studies have shown that on complexation with metal ions these Schiff base derivatives show enhanced pharmacological properties. [5] Several studies show that on complexation with metals, Benzofuran derivatives show enhanced biological and catalytic activity. It was also reported that halide substitution on the aromatic ring greatly increased the antibacterial activity. The antimicrobial property of the compound either kills the microbe or prevent their multiplication by blocking active sites[6], [7] Isatin (1H-indol-2,3-dione) analogs have a variety of biological activity, for example, antimicrobial, anticonvulsant, analgesic, anti-inflammatory, anticancer, antitubercular, antiviral, and anti-HIV, because of the presence of the indole backbone[8].

Given the extensive use of Schiff bases derived from isatin and its metal complexes in the area of coordination chemistry, we thought it's worthwhile to prepare the napthofuran Schiff base and its metal complexes.

The current article describes the synthesis of Co(II), Ni(II), Cu(II), Zn(II), Cd(II), and Hg(II) complexes with Schiff base derived from the condensation of naphtho[2,1-b]furan-2-carbohydrazide and indoline-2,3-dione (Isatin) (Figure 1).

EXPERIMENTAL:

All the chemicals used are of AR grade with the highest purity available. Benzofuran-2-carboxamide was synthesized according to the literature procedure[9]. The metal and the chloride content were determined according to the Vogel's procedure[10].

PROCEDURE FOR THE SYNTHESIS OF SCHIFF BASE 3-(2-HYDROXY BENZYLID ENEAMINO)-5-BROMO BENZOFURAN-2-CARBOXAMIDE [L].

A mixture of naphtho[2,1-b]furan-2-carbohydrazide (0.1 mol) and indoline-2,3-dione (Isatin) (0.1 mol) in ethanol (30 mL) are refluxed on a water bath for about 8h in presence of few drops of glacial acetic acid with occasional shaking. The product which was separated as crystalline solids on cooling was collected and recrystallized from ethanol. $C_{21}H_{13}N_3O_3$ [L]: Molecular Weight = 355.10, mp = 228°C, Yield= 69%.

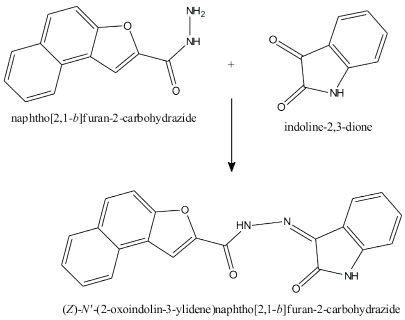

Figure 1: Synthesis of Ligand (L)

GENERAL PROCEDURE FOR PREPARATION OF METAL COMPLEXES OF Co(II), Ni(II), Cu(II), Zn(II), Cd(II), AND Hg(II) WITH THE SCHIFF BASE (L).

Metal chloride (0.01 mol) was mixed with Schiff base (0.01 mol) in an ethanolic medium 30 mL and the reaction mixture was refluxed for about 3hon water bath, then an aqueous solution of sodium acetate was added to the mixture to adjust the pH between 5 to 6 and further refluxed for about an hour. The complexes separated were filtered off and washed with distilled water, then with alcohol, and finally dried in vacuum over fused calcium chloride.

PHYSICAL MEASUREMENTS:

The IR spectra of a Schiff base and its metal complexes were recorded in KBr pellets. Molar conductivity measurement was recorded on an Elico CM-180 conductivity bridge in DMF (10^{-3} M) solution using a dip-type conductivity cell fitted with a platinum electrode and the magnetic susceptibility measurement was made at room temperature on Gouy balance using Hg[Co(NCS)4] as the calibrant.

RESULTS AND DISCUSSION:

The physical appearance and analytical results show that complexes are of the type ML_2Cl_2 where M=Co/Ni/Cu) and MLCl (Where M=Zn/Cd/Hg). The low molar conductance values (10.08 to 17.75 ohm-1 cm^2 mol^{-1}) indicate the non-electrolytic nature of the complexes. All the complexes are light-colored, stable, and non-hygroscopic in nature having high melting points (>300 °C).

Table 1: IR Data of Ligand and its metal complexes

Ligand/ Complex	ν(C=O)	ν(C=N)	ν(M-O)	ν(M-N)	ν(M-Cl)
[L]	1670	1622	----	----	----
Co(II)	1655	1686	553	457	390
Ni(II)	1658	1590	551	451	361
Co(II)	1661	1590	550	455	350
Zi(II)	1623	1590	551	420	372
Cd(II)	1656	1592	554	449	365
Hg(II)	1654	1589	551	456	355

IR STUDIES: The Infrared spectra of the ligand [L] and its metal complexes have been depicted in and I.R. assignments are listed in the above *Table.* The band observed in the ligand at 3234 cm^{-1} is attributed to ν(-NH) stretching frequency. This band unaltered in all the complexes indicating non-involvement of the nitrogen of (-NH-) in bonding with metal ion[11]. The strong band observed at 1670 cm^{-1} is assigned to ν(C=O) stretch of amide group undergo negative shift by 15 – 30 cm^{-1} in the complexes indicating the involvement of the carbonyl group in coordination[12]. The Schiff base shows a strong to medium intensity band at 1622 cm^{-1} due to ν(C=N) stretching vibrations, this band undergoes negative shifts in the spectra of the complexes showing the involvement of the azomethine group in the coordination with the metal ions[13], [14]. The band at 1192 cm^{-1} is assigned to ν(C-O-C) stretch of the furan ring in the free ligand. This band is unchanged on complexation indicating non-involvement of the furan ring oxygen in the bonding–[15]. In the far IR spectra of the complexes, the bands in the region 554 -550 cm^{-1}, 457 - 420 cm^{-1}, and 350-390 cm^{-1} assigned to M-O, M-N, and MCl stretching vibrations in all the complexes[16]. All the above value indicates that the ligand acts as the bidentate, with azomethine nitrogen and carbonyl oxygen atom as a donor site.

Mass spectral studies:

The LC-MS spectra of the Schiff base (L) (Figure-2) have shown a molecular ion peak at m/z 355.1, which is one mass unit more than that of the molecular weight of the Schiff base suggesting the proposed structure.

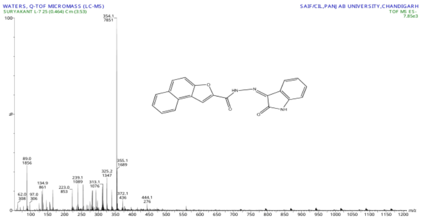

Figure 2: LCMS of Ligand L

Magnetic Properties:

Magnetic susceptibility measurements at the room temperature exhibit paramagnetism for Co(II), Ni(II), and Cu(II) complexes. The cobalt complex exhibits a magnetic moment of 3.08 B.M. suggesting octahedral geometry. The Ni(II) complex shows the magnetic moment value of 2.54 B.M. slightly higher than spin only value (2.83 B.M), indicating an octahedral environment. The observed magnetic moment values for the Cu(II) complex are 1.82 suggesting a distorted octahedral geometry[17].

CONCLUSION:

The synthesized Schiff base acts as a neutral bidentate ligand. The metal ion is coordinated through the amide oxygen and nitrogen of the azomethine group. The bonding of the metal to the ligand is confirmed by the analytical data, spectral and magnetic studies. Based on the above results we proposed the probable structures for all the complexes as octahedral to Co(II), Ni(II), and Cu(II) complexes and tetrahedral to Zn(II), Cd(II), and Hg(II) complexes.

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