

## Synthesis, Characterisation and Antibacterial Activity of Metal Complexes of N-(1-morpholinobenzyl)acetamide

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Solid complexes of a new Mannich base, N-(1-morpholinobenzyl) acetamide (MBA) with Cu(II), Ni(II) Co(II), and Zn(II) complexes have been synthesised and characterised by spectral studies, molar conductance, magnetic susceptibility and elemental analyses. The antibacterial activity of the complexes has also been studied.

**Key Words:** Synthesis, Cu(II), Ni(II) Co(II), Zn(II), Complexes, N-(1-morpholinobenzyl) acetamide, Antibacterial.

### INTRODUCTION

It is well known from the literature that the compounds containing amide moiety have a strong ability to form metal complexes and exhibit a wide range of biological activities<sup>1-4</sup>. We have synthesised a new Mannich base, N-(1-morpholinobenzyl) acetamide (MBA) which also contains amide moiety. In continuation of our earlier work<sup>5, 6</sup>, we herein report the synthesis of N-(1-morpholinobenzyl) acetamide and its complexation characteristics with Co(II), Ni(II), Cu(II) and Zn(II) salts. The antibacterial activity of these metal complexes has also been studied.

### EXPERIMENTAL

#### General procedure for preparation of N-(1-morpholinobenzyl) acetamide (MBA)

The MBA ligand has been prepared by the reaction of benzaldehyde with the mixture of acetamide and morpholine in 1 : 1 : 1 mole ratio.

#### Synthesis of metal complexes of MBA

MBA was dissolved in chloroform and mixed with an ethanolic solution of the respective metal salt in 1 : 1 mole ratio. The reaction mixture was gently warmed on a water-bath for 1/2 h. The resulting solid complex formed was washed with ethanol and dried.

## RESULTS AND DISCUSSION

All the coloured complexes are stable at room temperature. They are insoluble in water but soluble in  $\text{CHCl}_3$ , DMF and DMSO. The analytical data of the complexes along with some physical properties are summarised in Table-1. An examination of the elemental analysis of the metal complexes listed in Table-1 reveals the formation of 1 : 1 (metal : ligand) complexes. Molar conductance values of the complexes reveal their non-electrolytic nature<sup>7</sup>. The magnetic susceptibility measurement values show tetrahedral for cobalt and zinc, square-planar for nickel and octahedral for copper complex.

TABLE-1  
ANALYTICAL DATA OF THE METAL COMPLEXES

Complex (colour)	% Analysis, Found (Calcd.)				Yield (%)	$\mu_{\text{eff}}$ (B.M.)	$\lambda_{\text{m}}$ ( $\text{mho cm}^{-2} \text{ mol}^{-1}$ )
	Metal	C	H	N			
[CuCl <sub>2</sub> ·MBA·(H <sub>2</sub> O) <sub>2</sub> ] (green)	15.4 (15.6)	45.9 (38.6)	5.2 (5.4)	8.8 (6.9)	53	1.9	11.4
[CoCl <sub>2</sub> ·MBA](H <sub>2</sub> O) <sub>2</sub> (pink)	14.9 (15.3)	48.0 (40.8)	4.9 (5.2)	9.2 (7.3)	58	3.8	12.1
[NiCl <sub>2</sub> ·MBA](H <sub>2</sub> O) <sub>2</sub> (green)	14.3 (15.4)	47.9 (40.9)	5.1 (5.2)	9.4 (7.3)	52	0.0	10.6
[ZnCl <sub>2</sub> ·MBA](H <sub>2</sub> O) <sub>2</sub> (yellow)	15.7 (16.7)	47.4 (40.2)	4.8 (5.1)	9.1 (7.2)	56	Diamag.	11.2

In MBA, the infrared bands observed at 3285, 1626 and 1120  $\text{cm}^{-1}$  have been assigned to  $\nu(\text{NH})$ , amide  $\nu(\text{C}=\text{O})$  and  $\nu(\text{C}-\text{N}-\text{C})$  of morpholine group respectively<sup>8</sup>. In the IR spectra of all the complexes, the  $\nu(\text{NH})$  band remained at the same position as in the free ligand, indicating that the secondary nitrogen is not coordinated. In all the complexes, the amide  $\nu(\text{C}=\text{O})$  and  $\nu(\text{C}-\text{N}-\text{C})$  of morpholine bands displayed substantial negative shifts with fairly low intensity indicating coordination through the oxygen of amide moiety and nitrogen of morpholine entity present in the ligand. The Cu(II) complex showed a hump at 3450–3000  $\text{cm}^{-1}$  for coordinated water molecules<sup>9</sup>. According to elemental analysis, the Co(II), Ni(II) and Zn(II) complexes contain water molecules. However, a band around 880  $\text{cm}^{-1}$ , characteristic of coordinated water, is not observed in the IR spectra of these complexes. It may thus be assumed that the water molecule is lattice held. Some new bands were found around 540–530, 450–440  $\text{cm}^{-1}$  of  $\nu(\text{M}-\text{O})$ ,  $\nu(\text{M}-\text{N})$  bonds respectively<sup>10, 11</sup> which further confirms that the ligands are bidentate in nature. In all the complexes, an additional medium band found at 325  $\text{cm}^{-1}$  is assigned to  $(\text{M}-\text{Cl})$  stretching vibration<sup>12, 13</sup>.

The <sup>1</sup>H-NMR spectra of MBA displayed the expected signals. MBA exhibits a multiplet signal at 7.2–7.5  $\delta$  (Ar—H), 6.6  $\delta$  (s, 1H, CH), 5.8  $\delta$  (d, 1H, NH), 2.4–2.6  $\delta$  (morpholine N—CH<sub>2</sub>), 3.5–3.7  $\delta$  (morpholine OCH<sub>2</sub>) and 2.3  $\delta$  (s,

CH<sub>3</sub>). In Zn(II) complex, the doublet of N—H proton shifted slightly downfield to 5.9–6.0  $\delta$  which reveals the coordination of carbonyl oxygen to Zn(II) ion. The signal due to morpholine N—CH<sub>2</sub> protons also shifted slightly downfield and appeared at 2.7  $\delta$  in the complex. This is an indication of the coordination of morpholine nitrogen. Thus, <sup>1</sup>H-NMR and IR results confirm the bidentate nature of MBA ligand.

The electronic absorption spectra of the ligand and its copper(II), cobalt(II), nickel(II) and zinc(II) complexes were recorded in DMSO solution. The green Cu(II) complex showed a broad band at 11961 cm<sup>-1</sup> which is assigned to <sup>2</sup>E<sub>g</sub> → <sup>2</sup>T<sub>2g</sub> transition indicating octahedral structure of the complex<sup>14</sup>. Co(II) complex exhibits a band at 15576 cm<sup>-1</sup> which is assigned to <sup>4</sup>A<sub>2</sub> → <sup>4</sup>T<sub>1</sub> for tetrahedral geometry<sup>15</sup>. Ni(II) complex showed a band at 14556 cm<sup>-1</sup> which is assigned to <sup>3</sup>E<sub>g</sub> → <sup>3</sup>A<sub>1g</sub> confirming a square-planar geometry for the complex.

The magnetic moment value for Cu(II) complex (1.91 B.M.) indicates the octahedral arrangement of the ligand around the central metal ion. The four coordinated Co(II) complex shows  $\mu_{\text{eff}}$  value of 3.8 B.M. which supports the tetrahedral geometry for the complex.

The ligand MBA and its copper(II), cobalt(II), nickel(II) and zinc(II) complexes were tested for antibacterial activity. The minimum inhibitory concentrations (MIC) values were determined at the end of an incubation period of 24 h at 37°C. The zones of inhibition against the growth of microorganisms are presented (Table-2). The ligand and its metal complexes are more active towards *Escherichia coli* than *Staphylococcus aureus*. The order of activity towards *Staphylococcus aureus* is Cu > Co > Ni > Zn and towards *Escherichia coli* is Cu > Zn > Co > Ni.

TABLE-2  
ANTIBACTERIAL ACTIVITY OF MBA AND ITS METAL COMPLEXES

No.	Compound	Inhibition zone (mm) at concentration (100 $\mu\text{g}/10 \mu\text{L}$ ) in DMSO	
		<i>S. aureus</i>	<i>E. coli</i>
1.	MBA	11	12
2.	[CuCl <sub>2</sub> ·MBA·(H <sub>2</sub> O) <sub>2</sub> ] (green)	19	21
3.	[CoCl <sub>2</sub> ·MBA](H <sub>2</sub> O) <sub>2</sub> (pink)	18	19
4.	[NiCl <sub>2</sub> ·MBA](H <sub>2</sub> O) <sub>2</sub> (green)	16	18
5.	[ZnCl <sub>2</sub> ·MBA](H <sub>2</sub> O) <sub>2</sub> (yellow)	15	20

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