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# Transition metal (II) Complexes with Schiff base of 1, 2, 4-Triazole-4-amine & 4-dimethylamino-benzaldehyde and their Antimicrobial Activity

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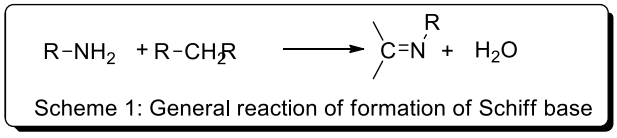
## ABSTRACT

This study summarizes the synthesis of new Schiff bases derivatives of 1, 2, 4-triazole-4-amine with 4-dimethylamino-benzaldehyde. The co-ordination compounds of transition metal ions like Cu (II) and Zn (II) with the Schiff bases were synthesized and characterized by UV-Visible Spectroscopy, Infra-Red Spectroscopy, NMR, Elemental Analysis and Magnetic Susceptibility Measurement. These co-ordination compounds show moderate biological activity against bacterial strains.

**Keywords:** 1, 2, 4- triazole-4-amine, Schiff base and Co-ordination compounds.

## I. INTRODUCTION

1,2,4-triazole heterocyclic compounds play a very vital role in our lives through their biological activities including anti-inflammatory, antiviral, antimicrobial, analgesic, antitubercular and anticancer properties[1-3]. Schiff bases are the condensation products (following Scheme 1)[1] of primary amines and carbonyl compounds, named after Hugo Schiff, who discovered them in 1864[4].People have synthesized some new 1, 2, 4-triazole[5-8] and 1, 3, 4-triazole[9]derivatives with their antimicrobial activity[10, 11].



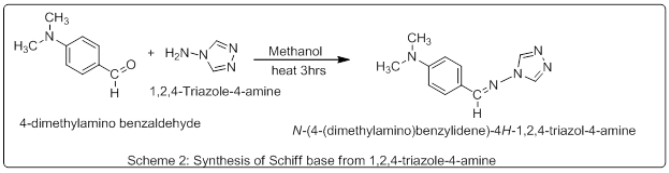
Scheme 1: General reaction of formation of Schiff base

The stability of metal complexes with Schiff bases and their applications as antifungal, antibacterial, anticancer, antiviral and herbicidal etc.[12] prompted us to synthesize new Schiff base of 1, 2, 4-triazole-4-amine with 4-dimethylamino-benzaldehyde and its metal complexes with Cu(II) and Zn(II), their characterization and investigation of their antimicrobial activity.

## II. EXPERIMENTAL

### 2.1 Preparation of ligand

4-dimethylamino benzaldehyde (2.38 mmole) and 1, 2, 4-triazole-4-amine (2.38 mmole) were mixed in 5 mL methanol and the mixture was refluxed for 3 hrs on water bath. The faint yellowish color product was obtained after the addition of 10 mL of ice water. The synthesized Schiff base was filtered and washed with ice water and dried at room temperature. It shows practical yield of 89.78% and M.P. is 202 $^0$C. Following reaction takes place (Scheme 2) and it was characterized by Spectrophotometer (Chemito UV-2100), Infra-Red Spectrophotometer (Bruker Optics, Model Alpha T), Bruker Advance II 400 NMR Spectrometer and Perkin-Elmer 2400 CHN Analyzer. The physical parameters and results of elemental analysis were shown in Table 1.



Scheme 2: Synthesis of Schiff base from 1,2,4-triazole-4-amine

**Table 1.** Characterization of ligand

| Sr. No. | Name & molecular formula of the ligand | Color | M.P. | % Yield | Elemental analysis Calculated (Found) % | | |
|---|---|---|---|---|---|---|---|
| | | | | | C | H | N |
| 1 | N-(1-(4-(dimethylamino)phenyl) ehtylidene)-4H-1,2,4,-triazol -4-amine | Faint Yellowish white | 202 $^0$C | 89.78 | 61.39 (61.10 ) | 6.04 ( 5.95) | 32.55 (32.25 ) |

### 2.3 Synthesis of metal complexes

The solid complexes were prepared by mixing of aqueous solution of Copper chloride (45 mg) and Zinc chloride (40 mg) with 5 mL of 2 % methanolic solution of ligand (100 mg) and refluxed for 2hrs. Blue and white

colored complexes were obtained for Copper (II) and Zinc (II) respectively (Figure 1).

### 2.4 Analysis of synthesized metal complexes

The characterization of metal complexes were interpreted by UV Spectrophotometer (Chemito UV-2100), Infra-Red Spectrophotometer (Bruker Optics, Model Alpha T) and elemental analysis by Perkin-Elmer 2400 CHN Analyzer. The physical parameters and result of elemental analysis were shown in Table 2.

**Table 2.** Characterization of metal complexes

| Sr. No. | Name & molecular formula of the complex | Color | M.P. | % Yield | Elemental analysis Calculated (Found) % | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | C | H | N | M |
| 1 | $(C_{11}H_{13}N_5)_2.CuCl_2$ | Blue | 96 $^0$C | 30 % | 46.76 (46.65) | 4.60 (4.52) | 24.79 (24.69) | 11.25 (11.15) |
| 2 | $(C_{11}H_{13}N_5)_2.ZnCl_2$ | White | 72 $^0$C | 34.42% | 46.61 (46.59) | 4.59 (4.55) | 24.71 (24.68) | 11.54 (11.45) |

### 2.5 Antimicrobial activity

The antimicrobial activity of the metal complexes was evaluated with the help of ATCC cultures including gram positive (S.aureus) and gram negative (E.coli & P. aeruginosa) using Gentamicin as standard and antifungal activity was tested against Candida sp. using Nystatin as standard and adopting standard protocols[13]. Saturated solutions of complexes in DMSO were used for the antimicrobial studies.

## III. RESULT AND DISCUSSION

The prepared complexes were found to be solids, insoluble in water but soluble in DMSO.

### 3.1 Electronic spectra

The characteristic peaks in electronic spectra of solutions of Schiff base in methanol and its complexes in DMSO are summarized in Table 3.The UV-Visible spectrum of Schiff base showed shoulder peaks and maximum absorbance at 336 nm (Figure 1). These shoulder peaks are observed due to $\pi$-$\pi$* transition.

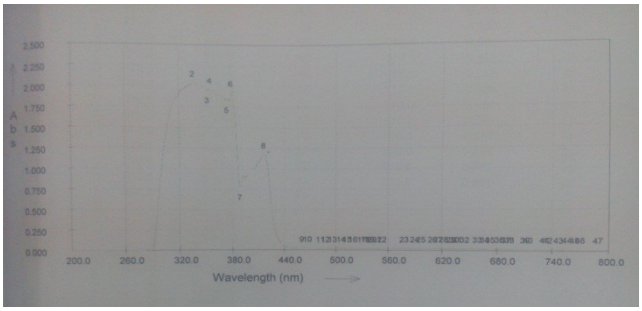



**Figure 1.** UV Spectra of Synthesized Ligand

The UV-Visible spectrum of Cu (II) complex assigned to d-d transition. The spectrum of Cu (II) complex shows band at 338 nm which is attributed to the electronic transition $^2E_g \rightarrow {}^2T_{2g}$ (Figure 2).

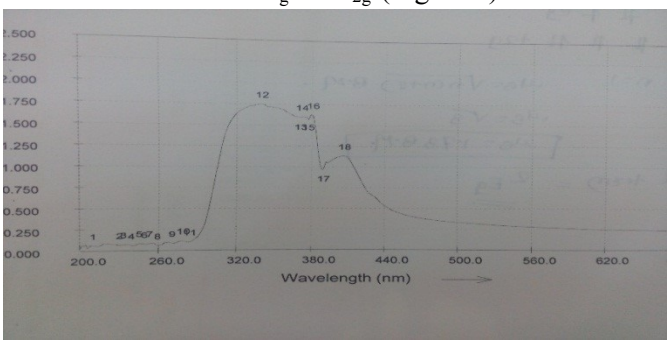



**Figure 2.** UV Spectra $CuL_2$ Complex

The magnetic susceptibility measurements provide data to characterize the structure of complex. The magnetic

moment for Cu (II) complex was approximately 1.73 BM. The Zn (II) complex was diamagnetic as expected for $d^{10}$ ions so that no d-d transition can be expected in the Zn (II) complex (Figure 3). Ligand had no magnetic moment[14].

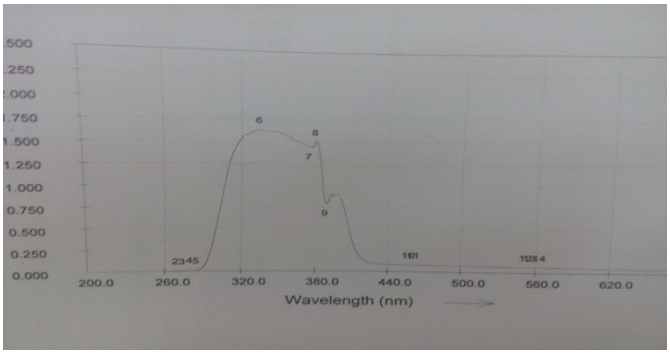



**Figure 3.** UV Spectra $ZnL_2$ Complex

**Table 3.** Important Peaks from UV Spectra of free Ligand and its complexes

| Product | $\lambda_{max}$ (nm) | Abs | Wavenumber $cm^{-1}$ | Transition | BM |
|---|---|---|---|---|---|
| L | 336 | 2.037 | 29,761 | π- π*, | - |
| $CuL_2Cl_2$ | 338 | 1.748 | 29,585 | π- π*, $^2E_g \rightarrow {}^2T_{2g}$ | 1.73 |
| $ZnL_2Cl_2$ | 334 | 1.636 | 29,940 | π- π* | - |

### 3.1 FTIR spectra

The functional groups information of prepared ligand and complexes were confirmed by Infrared Spectroscopy (Figure 2-4). The wavenumber ($cm^{-1}$) of characteristic bands in the IR spectra of ligand and complexes were reported in Table 4.

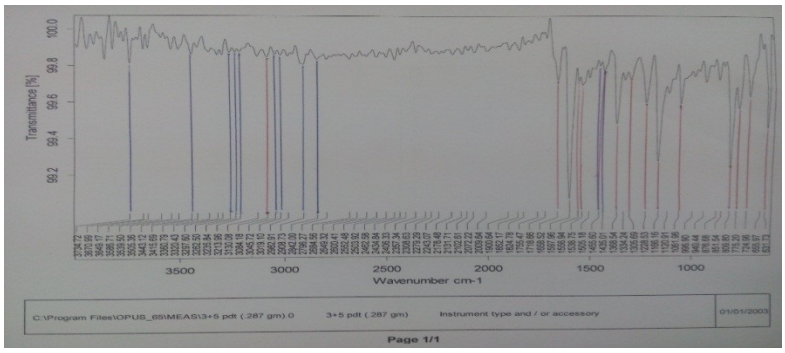



**Figure 2.** FTIR Spectra of Synthesized Ligand

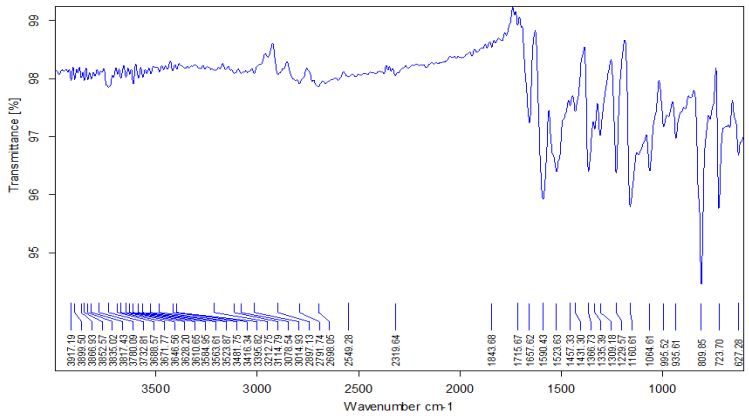



**Figure 3.** FTIR of $CuL_2$ Complex

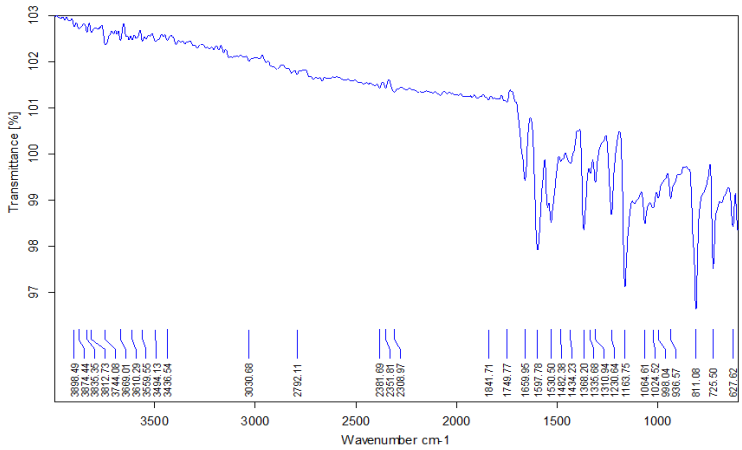



**Figure 4.** FTIR of $ZnL_2$ Complex

**Table 4.** Important Peaks from IR spectra of free ligand and its complexes

| Product | Azomethine νC=N | Triazole νN-N | νM-N |
|---|---|---|---|
| L | 1597.96 | 1305.69 | - |
| $CuL_2Cl_2$ | 1590.43 | 1309.18 | 627.78 |
| $ZnL_2Cl_2$ | 1597.78 | 1310.43 | 627.62 |

### 3.2 Nuclear Magnetic Resonance

The $^1H$ NMR spectra of ligand was recorded in DMSO. The $^1H$ NMR spectrum show sharp signals at δ 8.00 for one proton which could be attributed to the CH=N groups, aromatic proton in the range of δ 6.72-7.69 and two triazole proton shows a sharp signal at δ 8.76-8.87 (Figure 5).

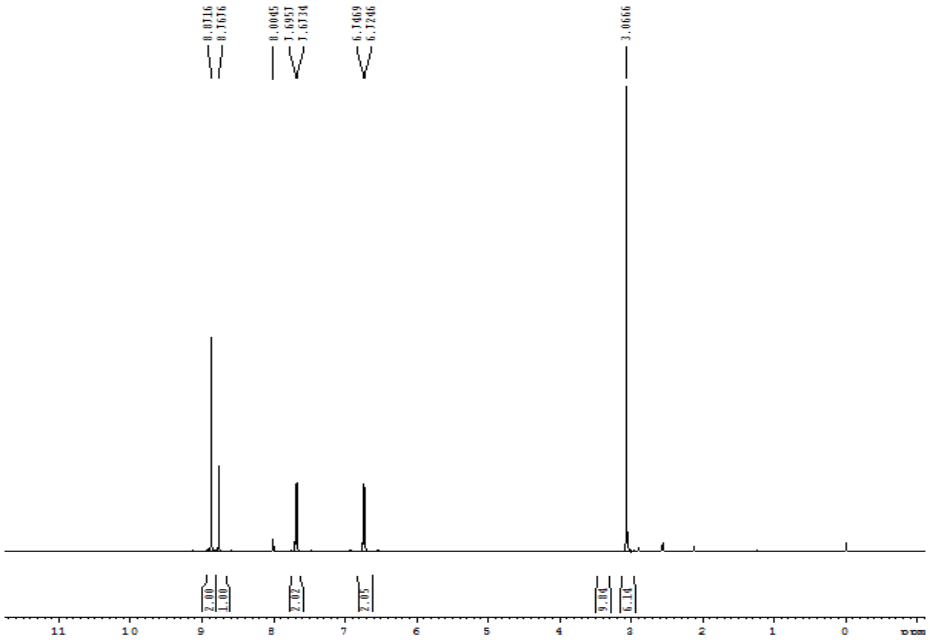


**Figure 5.** NMR of Synthesized Ligand

### 3.3 Antimicrobial activity

The synthesized metal complexes exhibited moderate biological activity against a Gram positive, two Gram negative as shown in Table 5.

**Table 5.** Effect of complexes on antimicrobial bacteria (Note- (-) indicates no activity.)

| Compound | E.coli ATCC 25922 | P.aeruginosa ATCC 27853 | S.aureus ATCC 25923 | Candida sp. |
|---|---|---|---|---|
| $CuL_2Cl_2$ | 08mm | 08 mm | 07 mm | (-) |
| $ZnL_2Cl_2$ | 08 mm | 09 mm | 07 mm | (-) |
| Gentamicin(Standard) | 22 mm | 27 mm | 31 mm | (-) |
| Nystatin (Standard) | (-) | (-) | (-) | 22 mm |

## IV. CONCLUSION

The complexes of Cu (II) and Zn (II) were synthesized by reaction of the synthesized ligand with the respective metal salts in 1:2 (M: L) ratio. The synthesized ligand and its complexes were characterized by UV-Visible, IR spectroscopic technique, NMR, magnetic susceptibility measurement, elemental analysis and their antimicrobial activity. The elemental and other spectral studies confirm the binding of Schiff base and metal ions and show the octahedral geometry of Cu (II) and Zn (II) complexes.

## V. ACKNOWLEDGMENT

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