

## Synthesis and Characterization of New Schiff Bases Ligands and Studies It's Complexes with Some Elemintes

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### Abstract

Preparation a new Schiff base ligand derived from reaction of terephthaldehyde with 2-aminobenzothiazol at (1:1) molar ratio and characterized by Uv-Vis, FT-IR, proton NMR and mass spectrometer and elemental analysis. The ligand mixed with Co (II), Ni (II), Cu (II), and Zn (II) chloride solutions at (2:1) molar ratio to form complexes. These complexes are characterized by elemental analysis, FT-IR, Uv-Vis, molar conductivity and magnetic sensitivity. This study concluded, the ligand acted as bidentate ligand, and all complexes have octahedral structures

**Keywords:** Terephthaldehyde, 2- aminobenzothiazol, Schiff base, Bidentate Ligand.

### Introduction

The coordination compounds have a great attention because of the Its importance in many different field such as analytical chemistry for the extraction of metals [1], spectral estimate of elements [2, 3], agriculture [4], industry [5] and other fields [6], so researchers have been developing the coordination chemistry by preparing new ligands in their atoms the ability to form stable chelating rings with elemental ions [7]. In the recent years bidentate Schiff bases constituted an important class of chelating organic ligands so researchers have prepared many of them.

There are many ways to prepare Schiff bases depending on raw materials, solvents, catalyst and conditions of reaction (pressure and temperature) [8, 10]. However, the direct method of condensation between aldehyde and ketone with primary amine is more common [11]. Schiff bases prepared from aromatic aldehydes and aromatic amines are more stable than Schiff's bases, due to the increased stability of resonance [12, 13].

Aromatic Schiff bases are not dissolved in acid, base or neutral solutions [14] The ligand content Nitrogen atom as donor atoms is more important in biosystem [15] J. Joseph and G. Boomadevi Janaki prepared bidentate Schiff bases ligand type N,N form 2-aminobenzothiazole and characterized by Uv-Vis, FT-IR, CHN, <sup>1</sup>HNMR and ESR [16]. In this paper we prepared bidentate Schiff bases ligand content Nitrogen donor atom by condensation terephthaldehyde with 2-aminobenzothiazol and there complexes with Co(II), Ni(II), Cu(II), and Zn(II).

### Experimental

All solution was prepared in doubly distilled deionized water.

### Materials

All the chemical used in this work of highest purity available and used without further purification. Tables (1) demonstrate the chemical substances and formula used in this research, purity and the companies supplied them.

**Table 1: Chemical substances used**

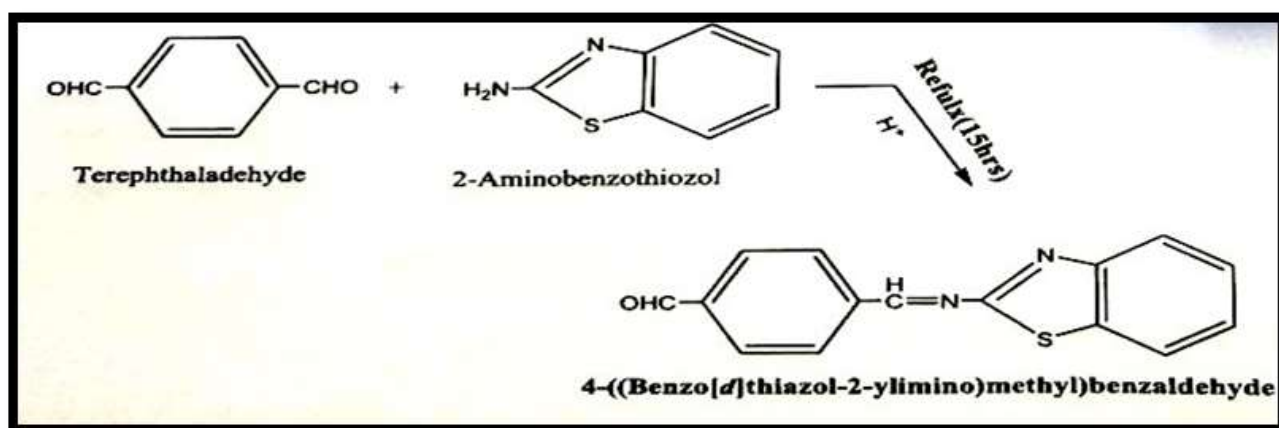
| No. | Substance                         | Formula                                        | Company        | % Purity |
|-----|-----------------------------------|------------------------------------------------|----------------|----------|
| 1.  | 2-Aminobenzothiazol               | C <sub>7</sub> H <sub>7</sub> N <sub>2</sub> S | Fluka          | 99.5     |
| 2.  | Terephthaldehyde                  | C <sub>8</sub> H <sub>6</sub> O <sub>2</sub>   | Fluka          | 99       |
| 3.  | Copper (II)Chloride dihydrate     | CuCl <sub>2</sub> .2H <sub>2</sub> O           | Riedel- deHaen | 99       |
| 4.  | Cobalte(II) Chloride hexa hydrate | CoCl <sub>2</sub> .6H <sub>2</sub> O           | B.D.H          | 99       |

|     |                                  |                                      |         |      |
|-----|----------------------------------|--------------------------------------|---------|------|
| 5.  | Nickel(II) Chloride hexa hydrate | NiCl <sub>2</sub> .6H <sub>2</sub> O | Himedia | 98   |
| 6.  | Zinc(II)Chloride                 | ZnCl <sub>2</sub>                    | B.D.H   | 99   |
| 7.  | Toluene                          | C <sub>7</sub> H <sub>8</sub>        | B.D.H   | 99   |
| 8.  | Ethanol absolute                 | C <sub>2</sub> H <sub>5</sub> OH     | GCC     | 99.5 |
| 9.  | Glacial acetic acid              | CH <sub>3</sub> COOH                 | B.D.H   | 99.5 |
| 10. | Methanol                         | CH <sub>3</sub> OH                   | GCC     | 99.5 |

### Preparation of Ligand

Dissolved (4.0g, 0.029 mol) from terephthalaldehyde in (40 ml) absolute Ethanol and 4 drops Glacial acetic acid then added same volume of absolute ethanolic solution of

2-Aminobenzothiazol (4.47g, 0.029 mol) are mixed. The mixture is reflux up for 3 hours. follow-up interaction by TLC Observed the end of the interaction after 5 h the product was recrystallized from absolute ethanol, yield 78% and RF = 0.65.



Schema 1: Preparation of ligand

### Preparation of Complex

In the preparation of the complexes. Hot ethanolic solution of ligand (0.532g, 0.002 mol) and hot ethanolic solution of corresponding metal salts (0.001 mol) of (0.136g) Zn(II), (0.17g) Cu(II), (0.237g) Ni(II), (0.238g) Co(II) and are mixed together,

refluxed for (30 min-1h) coloured solid metal complexes were obtained. The products are filtered, washed with solution of ethanol and water by 1:1 ratio. Dried at room temperature, the products are recrystallized by using absolute ethanol. Some physical properties of the prepared Ligand and complexes listed in Table (2).

Table 2: Physical properties of the ligand and their complexes

| Compound         | Color        | M.P °C              | Yield % | M. Wt  |
|------------------|--------------|---------------------|---------|--------|
| L                | Yellow       | 129                 | 83      | 266    |
| ZnL <sub>2</sub> | Light yellow | 142                 | 71      | 740.41 |
| CuL <sub>2</sub> | Light yellow | 179                 | 68      | 774.47 |
| NiL <sub>2</sub> | Yellow       | 248                 | 52      | 769.69 |
| CoL <sub>2</sub> | Bluish green | 145 decompose point | 63      | 715.93 |

## Results and Discussion

### Solubility Test

A number of experiments were conducted to determine the solubility of prepared ligand and complexes.

The results were included in Table (3). Observe that are ligand and Complexes insoluble in water while their solubility ratio varies in organic solvents.

Table 3: Solubility of prepared ligand and complexes

| Compound         | Water | Methanol | Ethanol | Acetone | Cyclohexane | DMF | DMSO |
|------------------|-------|----------|---------|---------|-------------|-----|------|
| L                | -     | +        | +       | +       | ++          | ++  | ++   |
| ZnL <sub>2</sub> | -     | +        | +       | +       | ++          | ++  | ++   |
| CuL <sub>2</sub> | -     | +        | +       | +       | ++          | ++  | ++   |
| NiL <sub>2</sub> | -     | +        | +       | +       | ++          | ++  | ++   |
| CoL <sub>2</sub> | -     | +        | +       | +       | ++          | ++  | ++   |

(-) poor soluble (+) partial soluble (++) full soluble

### Infrared Spectra

Absorption bands for a number of active groups in free ligand were identified and their participation in the coordination

between ligand and metal ions. The change in the position, strength and shape of this band was measured after coordination and the results were included in Table 4.

**Table4: IR data of free ligand and complexes**

| Compound         | $\nu(\text{O-H})$<br>hydrated<br>water | $\nu(\text{C-H})$ Ar | $\nu(\text{C=O})$ as | $\nu(\text{C=N})$ | Water | (H <sub>2</sub> O)<br>Coor. | $\nu(\text{M-O})$<br>$\nu(\text{M-N})$ |
|------------------|----------------------------------------|----------------------|----------------------|-------------------|-------|-----------------------------|----------------------------------------|
| L                | -----                                  | 3055w                | 1695m                | 1593st            | 1247w | -----                       | -----                                  |
| ZnL <sub>2</sub> | 3281b<br>3219b                         | 3124w                | 1693st               | 1639st<br>1570s   | 1255m | 903w<br>814m                | 603m<br>474m                           |
| CuL <sub>2</sub> | 3394b<br>3269b                         | 3107w                | 1691 <sup>st</sup>   | 1645st<br>1577s   | 1251m | 922w<br>813m                | 520m<br>420m                           |
| NiL <sub>2</sub> | 3375b                                  | 3053w                | 1691s                | 1649st<br>1581m   | 1255m | 922w<br>883m                | 633m<br>488m                           |
| CoL <sub>2</sub> | 3392b<br>3388b                         | 3037w                | 1693s                | 1649st<br>1583m   | 1257m | 916w<br>813m                | 530m<br>432m                           |

St: Strong, s: sharp, m: Medium, w: weak, coor: coordination

From I.R spectra of the complexes, observed change in the intensity and shape of the frequency of the aliphatic and aromatic  $\nu(\text{C=N})$  peak (Cannot be separated due to interference of band). It was also observed that there was a change in the  $\nu(\text{O-H})$  Frequency of the water molecule which indicates its involvement in the formation of the complexes. The spectra of complexes recorded a new band in the range of 603-

420cm<sup>-1</sup> for coordination with metal, the coordination occurred through the nitrogen atom of azomethene and oxygen atom of (OH) group of Water molecule. It was also observed that there was no change in the absorption packages of the two groups (C=O) and (C-S) indicating that they did not participate in the coordination [17, 19]. Fig. 1 and 2 show spectra of free ligand and copper complex.



**Fig 1: FTIR Spectra of free Ligand**



**Fig2: FTIR Spectra of Cu (II) Complex**

### Micro-Elemental Analysis

The ratios of elements (C.H.N.S) were calculated for prepared both ligand and complexes. Also the ratio of elements in the complexes was calculated by the atomic

absorption technique. The results were listed in Table 5. When comparing the practical values with the theoretical values, the added ratios of metal and ligand support the correctness of the proposed formulas for the complexes.

**Table 5: Shows the results of the elements analysis of ligand and the complexes**

| Sy.              | Chemical Formula                                                                                                                       | M.Wt   | C Found% (Cal. %) | H Found% (Cal. %) | N Found% (Cal. %) | S Found% (Cal. %) | Metal Found% (Cal. %) |
|------------------|----------------------------------------------------------------------------------------------------------------------------------------|--------|-------------------|-------------------|-------------------|-------------------|-----------------------|
| L                | C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O                                                                                       | 266    | 76.45 (67.72)     | 3.60 (3.75)       | 9.32 (10.52)      | 11.91 (12.05)     | -----                 |
| ZnL <sub>2</sub> | [Zn(C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub> .2H <sub>2</sub> O | 740.41 | 48.86 (48.66)     | 3.56 (3.78)       | 7.50 (7.56)       | 8.50 (8.66)       | 7.55 (8.83)           |
| CuL <sub>2</sub> | [Cu(C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub> .4H <sub>2</sub> O | 774.47 | 46.36 (46.52)     | 4.08 (4.13)       | 7.01 (7.23)       | 8.23 (8.27)       | 7.67 (8.20)           |
| NiL <sub>2</sub> | [Ni(C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub> .4H <sub>2</sub> O | 769.69 | 46.58 (46.81)     | 4.01 (4.15)       | 7.23 (7.27)       | 7.89 (8.33)       | 7.01 (7.62)           |
| CoL <sub>2</sub> | [Co(C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub> .H <sub>2</sub> O  | 715.93 | 50.28 (50.32)     | 3.51 (3.63)       | 7.60 (7.82)       | 8.51 (8.95)       | 8.05 (8.32)           |

### Molar Conductivity Measurements

The solutions of all (Co, Ni, Cu, Zn) complexes in DMSO have a Molecular

Conductivity of (68.7-73.2) S cm<sup>2</sup> mol<sup>-1</sup>, this indicating that all of these complexes solutions have a 1: 2 ratio of M:L (20,21). The data listed in Table 6.

**Table 6: Molecular conductivity  $\Lambda_m$  values for complex solutions in (DMSO) at a concentration of (1x10<sup>-3</sup>M) at Rome temperature**

| Sy.              | Complexes                                                                                                                              | $\Lambda_m$ (S.mol <sup>-1</sup> .cm <sup>2</sup> ) In DMSO |
|------------------|----------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------|
| ZnL <sub>2</sub> | [Zn(C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub> .2H <sub>2</sub> O | 68.7                                                        |
| CuL <sub>2</sub> | [Cu(C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub> .4H <sub>2</sub> O | 72.6                                                        |
| NiL <sub>2</sub> | [Ni(C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub> .4H <sub>2</sub> O | 71.5                                                        |
| CoL <sub>2</sub> | [Co(C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub> .H <sub>2</sub> O  | 73.2                                                        |

### Magnetic Susceptibility Measurements

The values of magnetic sensitivity of the prepared complexes were calculated at 298 k. The magnetic values of the organic atoms, inorganic roots and metal ions were determined using Pascal constants [22].

The values of magnetic momentum ( $\mu_{\text{eff}}$ ) calculated according to the following equation.

$$\mu_{\text{eff}} = 2.828 \sqrt{XAT} \quad \text{B.M}$$

The magnetic momentum values of the Co (II), Ni (II) and Cu (II) ion complexes are (4.82, 3.08, 1.78) BM, respectively, consistent with the literature [23, 24]. A complex of the Zn (II) ion complex is their diamagnetic properties, which are evidence that they are single electrons [25]. The results of the magnetic measurements are consistent with the literature and also indicate the validity of the proposed formulas for the complexes.

### Electronic Spectra

The electronic spectra of ligand and its solid

complexes were recorded using a DMSO solvent.

### Ligand Spectra

The spectrum of the free ligand showed three absorption peaks, the first at wavelengths (342 nm), which were due to the electronic transfer of type (n  $\longrightarrow$   $\pi^*$ ) to aliphatic azomethane group, heterocyclic ring and carbonyl group, the second at (290 nm) which due to the ( $\pi \longrightarrow \pi^*$ ) for benzene ring [26, 27] and the third at (270 nm) which due to the (n  $\longrightarrow$   $\sigma^*$ ) for Sulphur atom in the heterocyclic ring [28]. As shown in Figure 3.

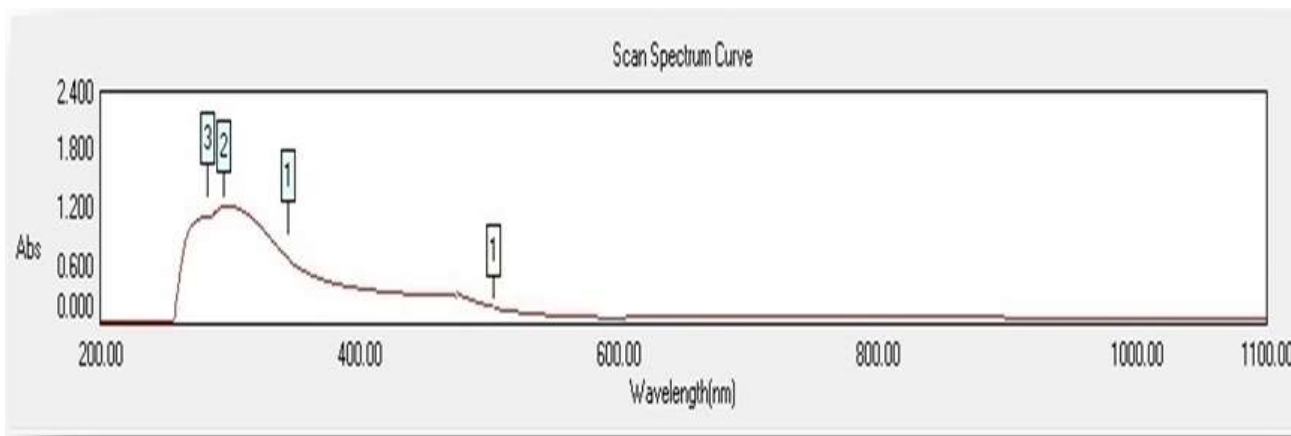


Fig 3: Uv-Vis spectrum of the free ligand

### Complexes Spectra

#### Cobalt Complex

The cobalt complex showed four peaks, two peaks at the wavelength (660 and 470 nm). These peaks are allowed due to the electronic

transfer. The appearance of this movement at that site supports the octahedral shape of this complex accordance with the literature [29]. The peak at the wavelength (362 nm) returns to the complex Charge transfer (CT) in the complex. Peak at (296 nm) due to the transfer of the charge in the ligand [30]. As shown in Figure 4.

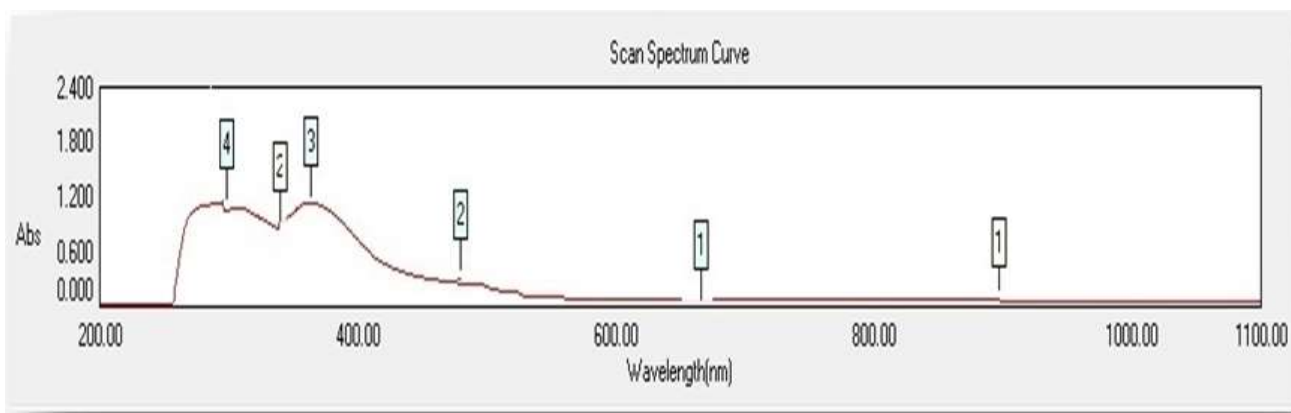


Fig 4: Uv-Vis spectrum of the Co (II) complex

#### Nickel Complex

Observed three peaks in the nickel complex spectrum at wavelengths (352, 290 and 270 nm) that electronic transitions of type ( ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(p) = u_3$ ) were blocked to occur under the peak of the observed charge

transfer at wavelength (352 nm). This is in line with the studies of the octahedral symmetry complexes [31, 32] as they belong to the Charge transfer (CT) in the complex and have red shifted due to consistency. Two other peaks due to the transmission of the charge in the ligand. As shown in Figure 5.

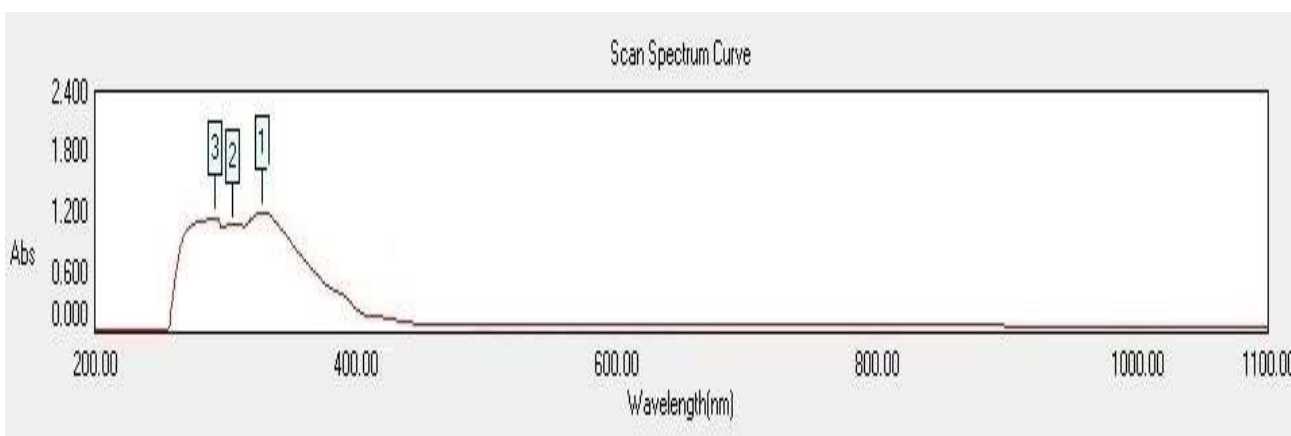


Fig 5: Uv-Vis spectrum of the Ni (II) complex



## Copper Complex

The Uv-visible spectrum of the copper complex (II) showed peak absorption at (578 nm) due to electronic transfer ( ${}^2B_{1g} \rightarrow {}^2E_g$ ). The peak of the charge transfer cannot be

distinguished as it overlapped with the top of the electronic transition. This was accompanied by the appearance of two other peaks at wavelengths (309 and 296 nm) due to the transfer of the internal charge of the ligand. As shown in Figure 6.

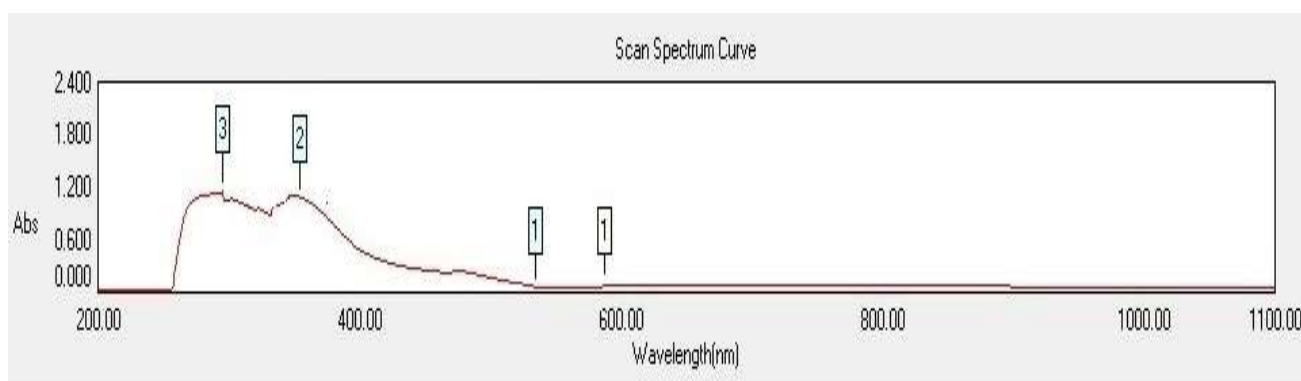


Fig 6: Uv-Vis spectrum of the Cu (II) complex

## Zinc Complex

The zinc ion complex is characterized by the appearance of the peak transmission of the charge at the wavelength (379 nm),

accompanied by two other peaks at the (308 and 296nm) due to the transmission of the ligand charge. As shown in Figure 7.

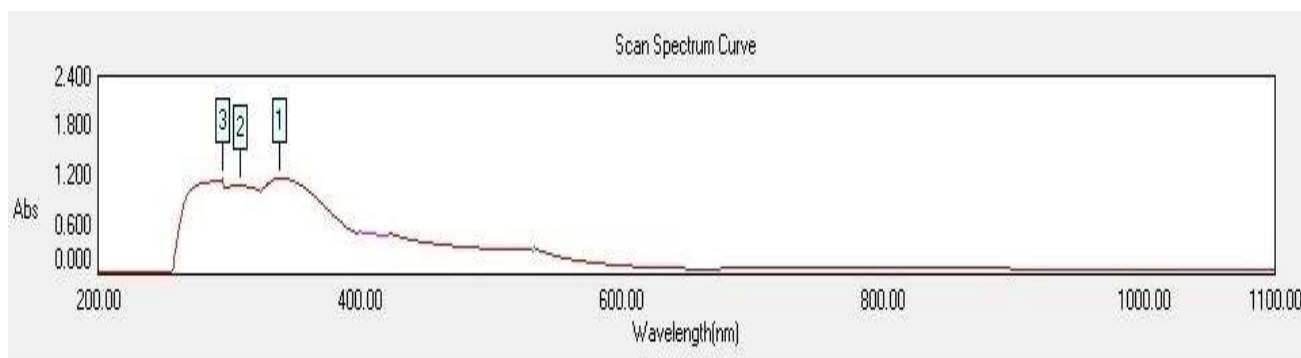


Fig 7: Uv-Vis spectrum of the Zn (II) complex

All the results obtained for these spectra of

ligand and complexes were included in Table 7.

Table 7: Electronic Spectra and Magnetic Measurements of ligand and their complexities in (DMSO) solvent at laboratory temperature

| Complex                                              | Absorption band(nm) | Absorption band( $cm^{-1}$ ) | Assignment                                | $\mu_{eff}$ (B.M) | Proposed Structure |
|------------------------------------------------------|---------------------|------------------------------|-------------------------------------------|-------------------|--------------------|
| $C_{15}H_{10}N_2OS$                                  | 342                 | 33037.03                     | $n \rightarrow \pi^*$                     | -----             | -----              |
|                                                      | 290                 | 34483.75                     | $\pi \rightarrow \pi^*$                   |                   |                    |
|                                                      | 270                 | 29239.76                     |                                           |                   |                    |
| $[Co (C_{15}H_{10}N_2OS)_2(H_2O)_2]Cl_2 \cdot H_2O$  | 660                 | 15151.51                     | ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(p)$ | 4.82              | Oh                 |
|                                                      | 470                 | 21276.59                     | ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ |                   |                    |
|                                                      | 362                 | 27624.30                     | C.T                                       |                   |                    |
|                                                      | 294                 | 34013.60                     | Intraligand                               |                   |                    |
| $[Ni (C_{15}H_{10}N_2OS)_2(H_2O)_2]Cl_2 \cdot 4H_2O$ | 352                 | 29385.22                     | C.T                                       | 3.08              | Oh                 |
|                                                      | 270                 | 32467.53                     | Intraligand                               |                   |                    |
|                                                      | 260                 | 37037.03                     |                                           |                   |                    |
| $[Cu (C_{15}H_{10}N_2OS)_2(H_2O)_2]Cl_2 \cdot 4H_2O$ | 578                 | 17301.03                     | ${}^2B_{1g} \rightarrow {}^2E_g$          | 1.78              | Oh                 |
|                                                      | 394                 | 25380.71                     | C.T                                       |                   |                    |
|                                                      | 280                 | 35714.28                     | Intraligand                               |                   |                    |
| $[Zn (C_{15}H_{10}N_2OS)_2(H_2O)_2]Cl_2 \cdot 2H_2O$ | 379                 | 26385.22                     | C.T                                       | Dia               | Oh                 |
|                                                      | 308                 | 32467.53                     | Intraligand                               |                   |                    |
|                                                      | 296                 | 33783.78                     |                                           |                   |                    |

### Nuclear Magnetic Resonance Spectrum

The ligand spectrum was recorded in a solvent DMSO-d<sub>6</sub>, multiple signals of ligand were observed at 7.3-7.8ppm. The aromatic protons of the benzene ring could be associated with a different signal at 7.9-8.3

ppm due to benzothiazole,benzene protons and other signals at the site 9.3ppm was attributed to protons of the azomethin group [33] with a final reference at 10.2 ppm dating to the proton aldehyde group [34]. As shown in Figure (8).

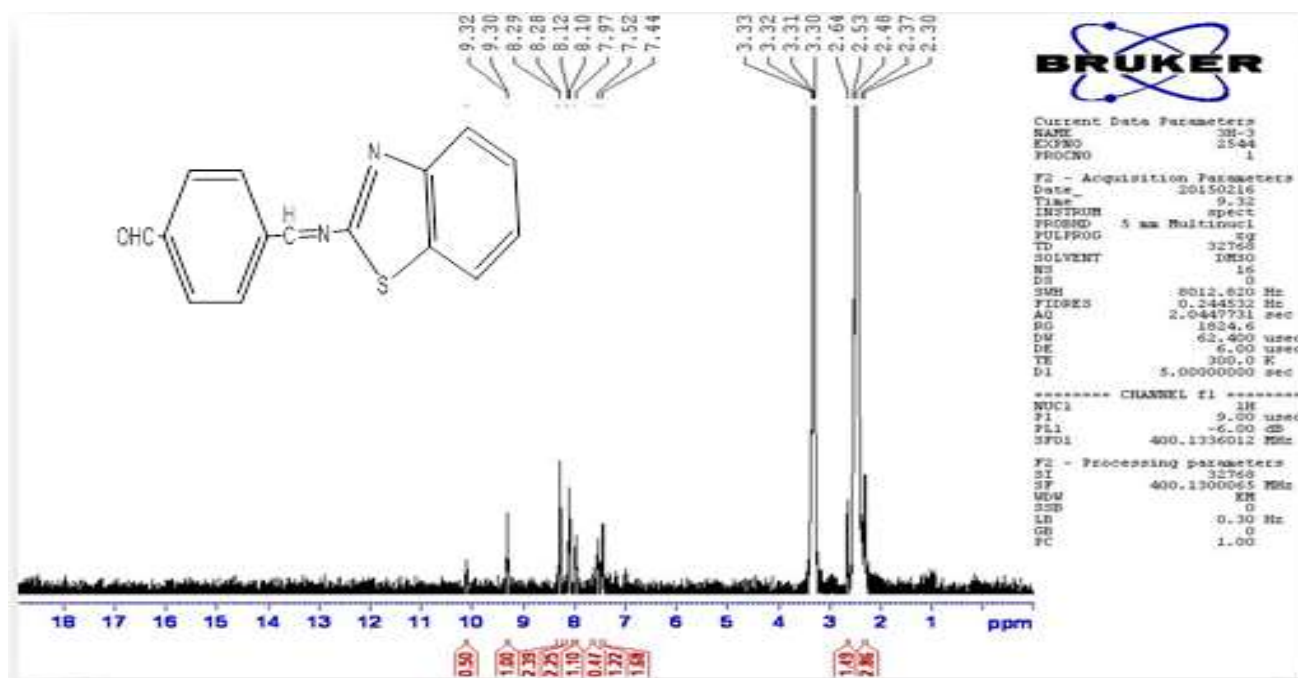


Fig 8: HNMR Spectrum of ligand

### Mass Spectrum

For confirming the structural formula of the ligand, the mass spectrometry was recorded as shown in Figure 9. The molecular structure of the ligand was shown to be good for the appearance of the mother molecular ion peaks at 266 (m/e) and very high relative

abundance. This is in line with the molecular mass of organic ligand Prepared masculinity previously in Table (2).The ligand has taken several fragmentation paths, most notably azomethane, with the formation of positive molecular ions and other positively charged parts.

Negative Chemical Ionization (NCI) Mode

Target LB @ 150C

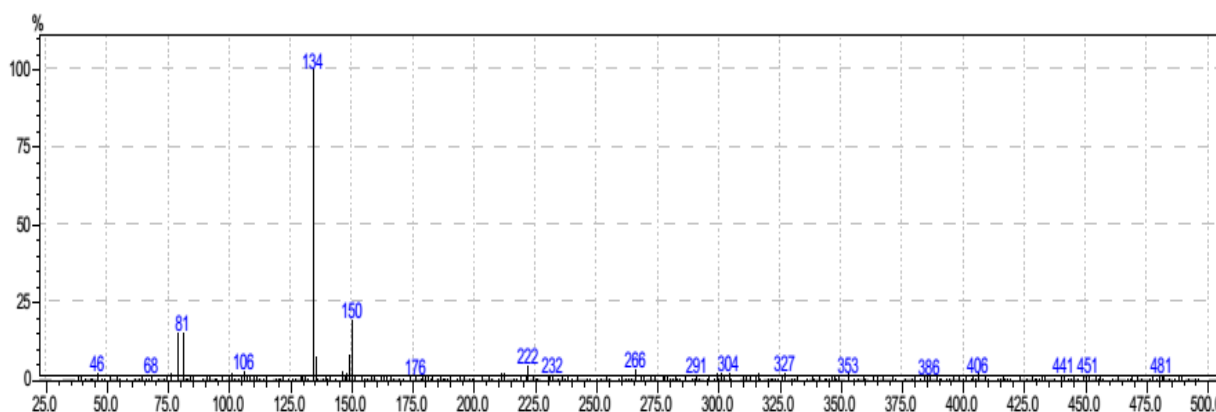
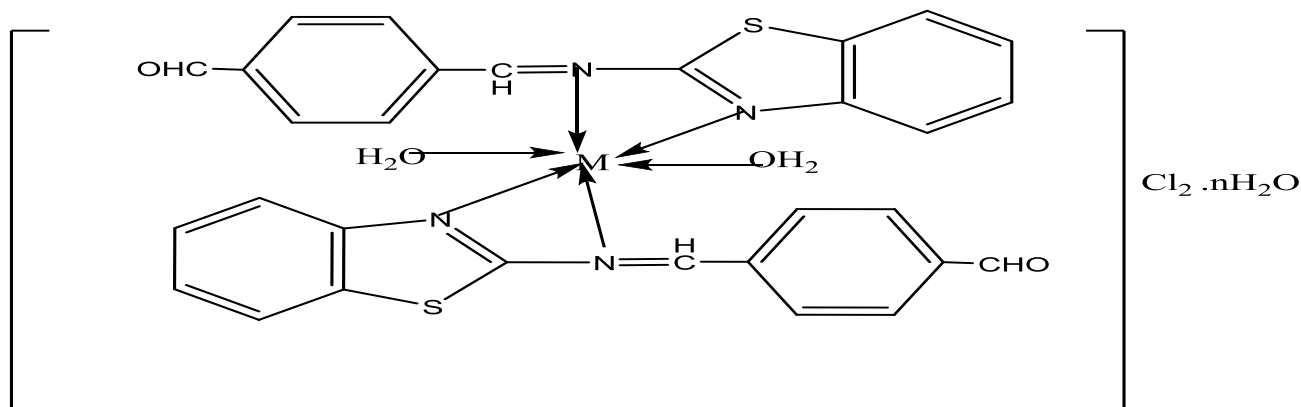


Fig9: Mass spectrum of ligand

### Proposed Molecular Structures of Ligand Complexes

Ligand was associated with metal ions by the nitrogen atom of the azomethane group and

the nitrogen atom of a heterocyclic ring. The ligand is thus a bidentate, so formation four member chelating rings with metals ion. Figure (10) illustrates the proposed structure of the complexes.



M= Zn(II) n = 2 , Cu (II) n = 4 , Ni(II) n =4 , Co(II) n= 1

Fig 10: Proposed structure of the complexes

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