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# Synthesis and Characterization of Some Metal Complexes Derived from Azo Ligand of 4,4' Methylenedianiline and Resorcinol

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**ABSTRACT**

The metal complexes of Azo dye; 4,4'-((methylene bis(3,l-phenylen)) bis (diazene-1,2-diyl)) bis(1,3-Benzenediol) were produced from 4,4`- methylenedianiline and Resorcinol were synthesized and characterized by using several analytical and spectral techniques. The ligand and its complexes were characterized by many chemo-physic methods like NMR, IR, UV-Vis, molar conductance measurement, analytical measurement, and melting point. The conditions of optimal reaction (for instance reagent concentration, pH etc) were studied and the analytical figures of merit such as limit of detection, linearity, sensitivity etc) were obtained.



## 1. Introduction

One of the main contributions to the development of coordination chemistry is the reaction of azo dyes with transition elements to form stable coordination complexes. Azo dyes are a class of broadly used as colorants which have huge applications in technology and many fields of sciences (Dakiky & Nemcova, 2000). Its role is evident in the textile and printing inks and paper, drugs industry in addition to food coloring (Vidya, et al., 2018; Gup, Giziroglu & Kırkan, 2007; Abdallah, 2012). Anticancer, Antioxidant, anti-Bacterial activities, biological actions for instance protein synthesis, nitrogen fixation and RNA, DNA inhibition its consider other important applications of Azo compounds (Karipcin & Kabalcilar, 2007; Ayoob, et al., 2020; Kadhium, Abdulmahdi & Ali, 2021). Particularly with most of the Azo compounds which serve as a ligands with their complexes where studies have proven to be effective as anti-inflammatory, antituberculosis, antifungal, herbicidal,and antidiabetic (Naseem, et al., 2021; Mallikarjuna & Keshavayya, 2020).

Diaminodiphenylmethane (MDA),4,4 ′ -Methylenedianiline has been shown to have risky biological effects such as eye and skin irritation, kidney and liver damage for human and animal. On the other hand, 4,4′ -Methylenedianiline has beneficial various uses, for instance cell strengthening of lubricating oils, polyurethane spume, for epoxy resins as curing agent, manufacture of azo dyes and Iron corrosion protection (AbouEl-Enein, 2008). Also,the complexation of azo ligands including in its structure MDA with several transition metals shows considerable biological activities against Eabanla vermiculate and *Monachaobstructa* (two land snail species) (Abouel-Enein, Emam, & Monir, 2018). Moreover, have been investigated and announced for many applications of compounds that incorporated with MDA moieties (Brunet, et al., 2018; Emam, 2017). Furthermore, Resorcinol-Azo moiety and its metal complexes have been studied before as they have shown biological significance (Abouel-Enein, Emam, & Monir, 2018; Saad, et al., 2019; Karmakar, et al., 2020; AbouEl-Enein, et al., 2015).

Azo was produced from the reaction between MDA and Resorcinol with its element complexes. Therefore, for improvement of the biological effective and decrease the MDA toxicity, our paper handle that production of aforementioned azo dye with some its complexes. The chemical structures were are studies by using the

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spectroscopic techniques like Infrared, Ultraviolet, 1H NMR , and Elemental analysis method.

## 2. Materials and Methods

### 2.1. Chemicals & Instruments

Entirely chemicals were purchased as reagent grade and were used without further purification, 4,4'-methylenedianiline (RIEDEL-DE HAEN AG SEELZE, Germany), Hydrochloric acid (BDH), Sodium nitrite (BDH), and ethanol were obtained from (GCC, England). NMR spectrum of ligand was recorded on a (VARIAN INOVA, 400 MHz NMR spectrometer, Dmso-$d_6$, TMS, 25 °C). measurement of the melting points are done by (SMP3/Stuart, UK). FT-IR spectrum was used to determine the spectral measurements (Shimadzu, made in Japan). KBr as reference is used for compare between the study measurements and reference values. The absorbance was confirmed with spectrophotometer (Shimadzu 1800 double beam).

### 2.2. Reagents & Solutions

For diluting the reagent and samples distilled water were used. Stock solutions of Cd(II), Pb (II), Cu(II) and Ni(II) ions (0.001M) were prepared by dissolving of $CuCl_2.6H_2O$ (BDH) (0.0242 g), $NiCl_2.6H_2O$(BDH) (0.0237 g), Pb $(NO_3)_2$ (BDH) (0.0331 g) and $CdCl_2$ (Merck) (0.0183 g) in distilled water, respectively. Appropriate dilutions of the stock standard solutions were freshly prepared for each metal ion. A solution of acetic acid and sodium acetate were used to prepare acetate buffer solution (0.1 mol $L^{-1}$).

### 2.3. Azo Ligand Synthesis

4,4'-methylenedianiline (0.198 g, 1 mmol) was added to water (10 mL) which formed from of HCl 1.5 mL and diazotized under 5 °C with $NaNO_2$ (1 mmol, 0.069 g,) in low temperature water (10 ml). The producing diazonium salt was mixed with Resorcinol (2mmol, 0.22 g) then adding 15 mL of ethanol and NaOH (10 mL,10%) then kept at ice-bath (0 – 5) °C, the red color would be formed then left the product without move to be stable for (60) minutes and regulate the pH to be 6, keep the solid precipitate 24 hrs. Submitted filtration of the colored solids, then make washing many times by using distilled water, then drying to produce concentrated ligand and have red color then purified by recrystallization, yield 84% and melting point was (286-288) (AL-Adilee, Abass & Taher, 2016). Synthesis of a ligand is given in Figure1.

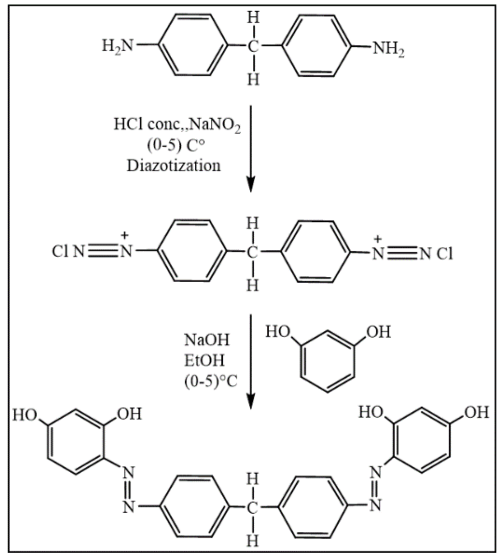


**Fig. 1.** Synthesis of diazo ligand

### 2.4. Azo-Metal Complexes Preparation

Preparation of the complexes was done by dissolving (10 mmol, 3.06 g) of reagent in the ethanol 50 mL then adding of the dropwise with shaking to (Cu(II),Ni(II), Pb(II), Cd(II)) (5 mmol) in buffer solution (20 mL). The products were shaken at 25 ˚C for 15 minutes, then left for 24 hrs. Filtration of the mixture was washed with ethanol with high temperature and distilled water then dried by using the oven at (60) °C (Islam, 2015). Figure 2 shows proposed chemical structure of complexes.

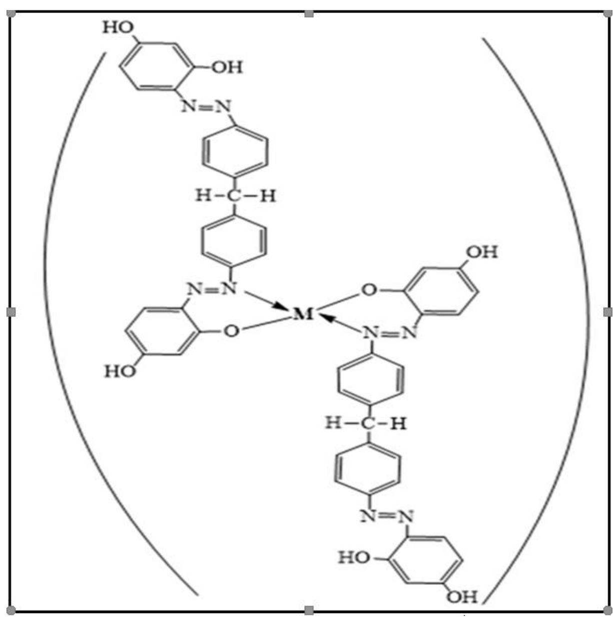

**Fig. 2.** Proposed ligand structures with its complexes

## 3. Results and Discussion

The ligand and its complexes have stability to air and colored. The compounds can dissolve in ethanol, acetone, and chloroform. The high melting degree of the complexes showed high thermal stability of the complexes as compared with the ligand. Table 1 show the analytic results and physical characteristics.

**Table 1**
C.H.N analytic data and physical properties of ligand as well its complexes

| NO. | compound | color | melting point ºC | Yield % | Molecular formula | % Found(calcd) | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | M | N | H | C |
| 1 | C25H20N4O4 (Ligand) | Red | 288-286 | 84% | C25H20N4O4 (440.46) | ___ | 12.72(12.78) | 4.11(4.6) | 68.11(68.46) |
| 2 | [Ni(L)2] | green | 303-301 | 70% | C50H38N8O8Ni -937.28 | 6(6.25) | 11.9(11.5) | 4(4.8) | 63.99(64) |
| 3 | [Cu(L)2] | yellowish orange | 329-331 | 82% | C50H38N8O8Cu -942.14 | 6.72(6.2) | 11.89(11.35) | 4.06(4) | 63.67(63.48) |
| 4 | [Pb(L)2] | light orange | 298-296 | 74% | C50H38N8O8Pb -1085.79 | 19(19.08) | 10(10.32) | 3.52(3) | 55.25(55) |
| 5 | [Cd(L)2] | reddish brown | 325-323 | 79% | C50H38N8O8Cd -991.01 | 11(11.34) | 11(11.3) | 3.5(3.86) | 60.3(60.54) |

3.1. Conductance Measurements

Conductance of the metal complexes was ($10^{-3}$ M in DMF) at (11-25) $Ohm^{-1}$ $cm^2$ $mol^{-1}$. That mean that the values of the complexes haven't Conducting (Ali, Wani, & Saleem, 2013).

3.2. NMR Spectra of Ligand

Spectrum of $^1H$ NMR of ligand in DMSO-$d_6$ was shown in Figure (3), which exhibits many signals at δ(6.19-7.76 ppm) due to protons of aromatic rings (Reddy, et al., 2011), singlet signal at 3.98 ppm attributed to (2H, $CH_2$ ), also a broad peak at δ 12.66 ppm due to Aromatic hydroxyl groups (Pavia, 2014; Khudair, Khawla & Seewan, 2021).

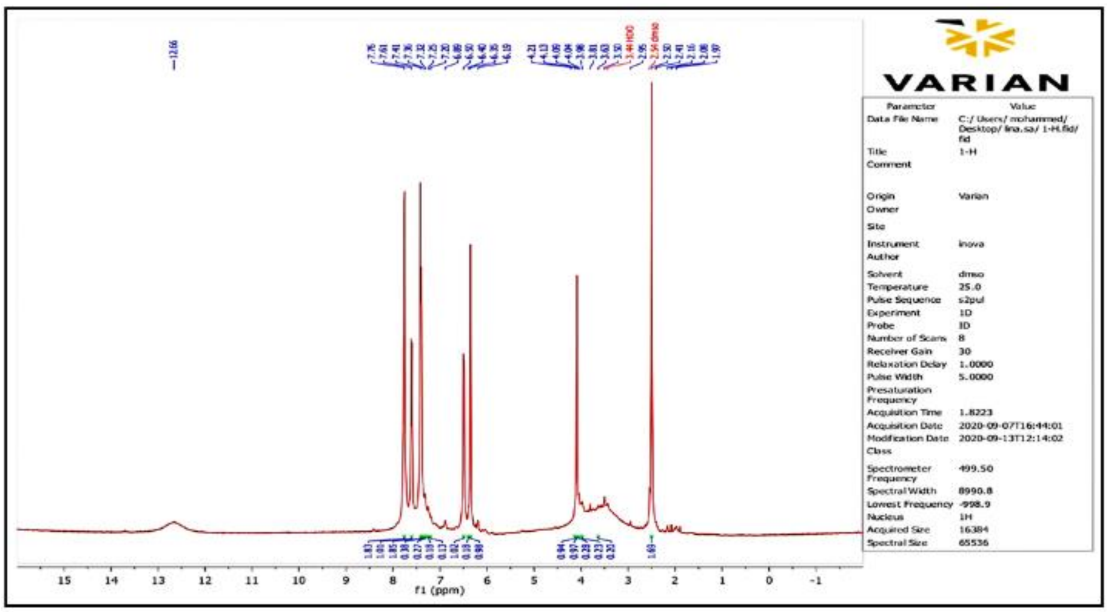


**Fig. 3.** $^1H$ NMR spectra of ligand

In the $^{13}C$ NMR spectrum, Figure (4) shows signal at (103-157) ppm allocate to a (Aromatic-Carbon) atoms, A peak at δ 46.0 ppm which is due to carbon atom in the methylene carbon($CH_2$) (Reddy, et al., 2011; Anitha, et al., 2013).

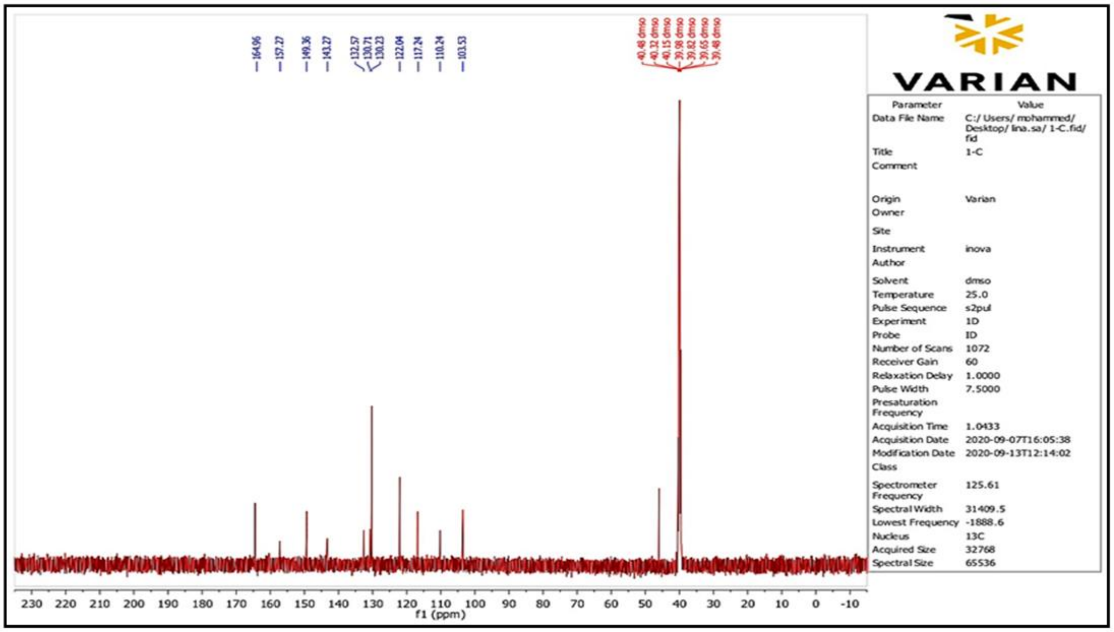


**Fig. 4.** $^{13}C$ NMR spectra of ligand

3.3. IR Spectra and Mode of Bonding

The infrared Studies of ligand and its complexes were recorded at 4000-400 $cm^{-1}$, Spectrum of the ligand. Figure 5 shows a band at 1427 $cm^{-1}$ that meaning to presence of azo group and the spectral bands at 3402, 1242 and 1380 $cm^{-1}$ caused by ν(H-O), ν(N-C), ν(C-O), respectively, A bands between(1620-1500)$cm^{-1}$ assignable to aromatic C═C,3024$cm^{-1}$ for aromatic C-H and 2923$cm^{-1}$ aliphatic C-H.

Infrared spectrum of the ligand was compared with the metal complexes spectrum for testing the linking between ligand and complexes (Table 2). The infrared of the ligand was at 3402 $cm^{-1}$ that is missing from the spectra of the metal complexes Figure 6 and Figure 7 that means deprotonating of phenolic{-OH} and linking of the atoms with (oxygen) of hydroxyl group. The band showed 1427 $cm^{-1}$ to –N=N- group in the ligand is alters to ~1389 $cm^{-1}$ in the complexes that indicates attachment between azo nitrogen with the ions (Adnan, 2020; Tapabashi, Taha & El-Subeyhi, 2021). M-N and M-O bonds vibration frequencies was at 655-624 $cm^{-1}$ and 540-439 $cm^{-1}$ respectively and that another indication to the bonding between the ligand and its metal ions by azo nitrogen atoms and phenolic oxygen (Dinçalp, et al., 2007). The FTIR spectra of the ligand and its complexes are computationally generated and compared with the experimental IR spectra. The computationally generated data closely match with the experimental data.

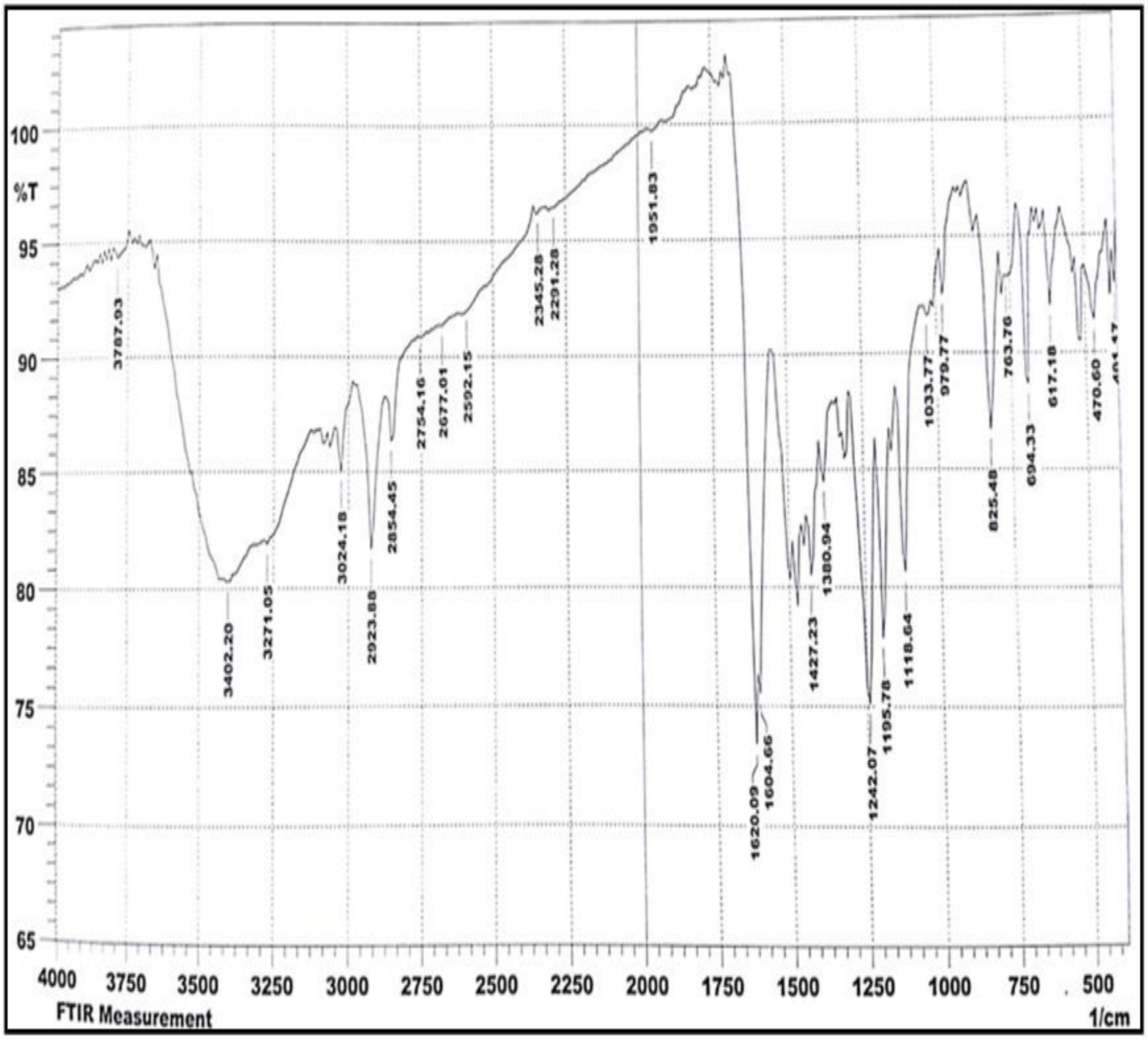


**Fig. 5.** FTIR spectra of the ligand

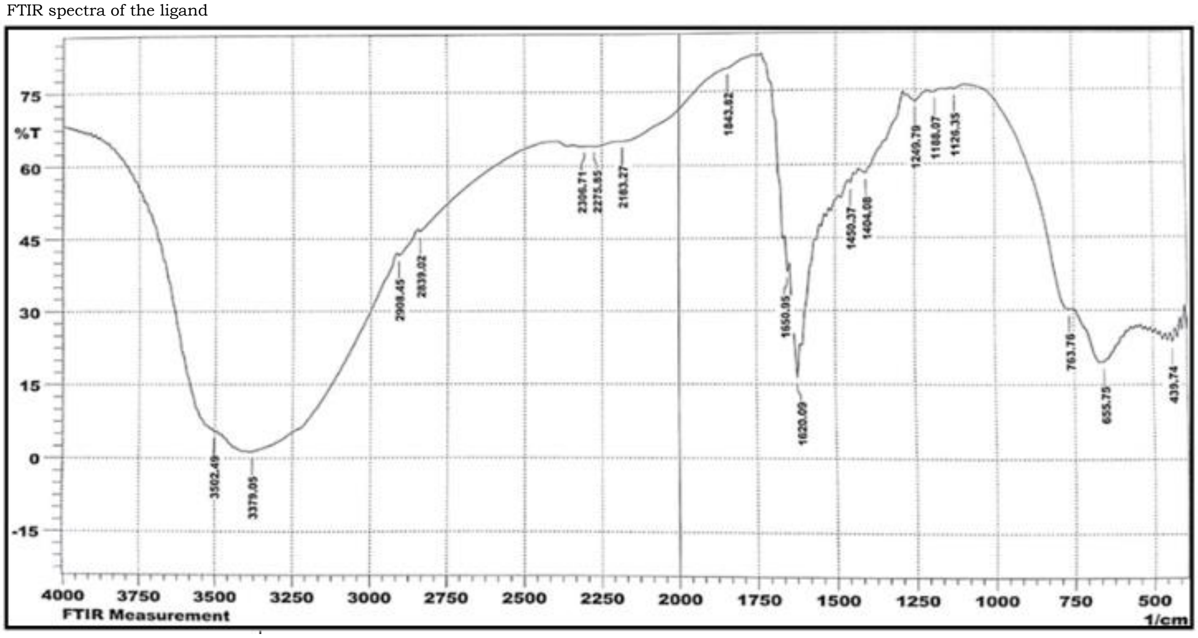

**Fig. 6.** FTIR – spectrum for Ni-Lcomplex

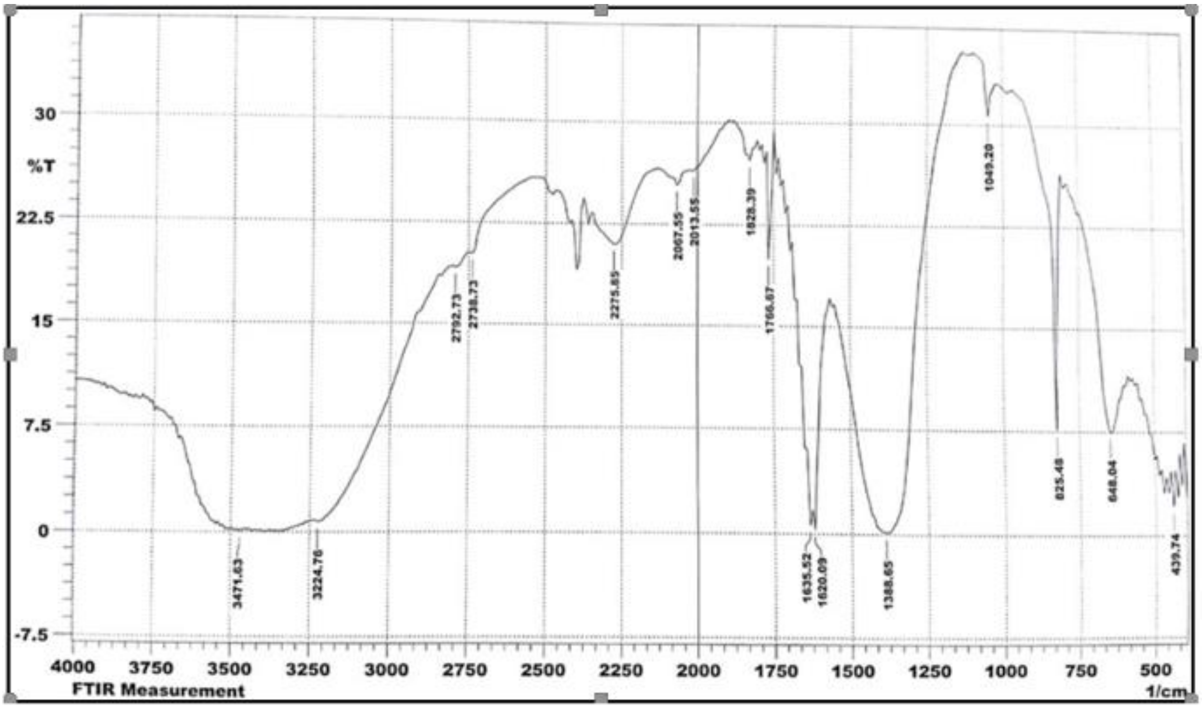


**Fig. 7.** FTIR – spectrum for Cu-L complex

**Table 2**
Investigating compounds FTIR data

| M-O | M-N | N=N | OH | Compound |
|---|---|---|---|---|
| --- | --- | 1427 | 3402 | Ligand |
| 439 | 648 | 1388 | --- | [Ni(L)2] |
| 439 | 655 | 1404 | --- | [Cu(L)2] |
| 540 | 624 | 1380 | --- | [Pb(L)2] |
| 542 | 620 | 1380.9 | --- | [Cd(L)2] |

## 3.4. Characteristics and Absorption Spectra of the Complexes

The electronic spectra of ligand under investigation were executed in an absolute ethanol. Ultraviolet visible of the ligand and its chelate ($10^{-4}$ M) were showed at (200 to 1000) nm at (25) °C by utilize similar solvent. In the UV-visible region, a free ligand showed that broad bands was 390 and 271 nm. First band was allocated to the intra ligand electronic transition n→π∗. The next broad band was assigned at electronic transition π→π∗ at (271) nm. These transition bands (n→π∗ and π→π∗) were changed to 339–395and 261–285 nm, consequence (Liu, et al., 2013; Irving & Williams, 1953).

The spectrum Figure 8 displays intense absorption peaks at (242 nm) and (391 nm) which were allocated to π→π* and n→π∗. The colors produced by reacting metal ions with azo ligands are very significant for electronic spectral studies. Due to the presence of sharp and high absorption peak which belong to the metal complex, In the visible region of solution of complex spectra with respect to ligand this peak was shifted and this may be due to good designated for coordination and formation of the complex.

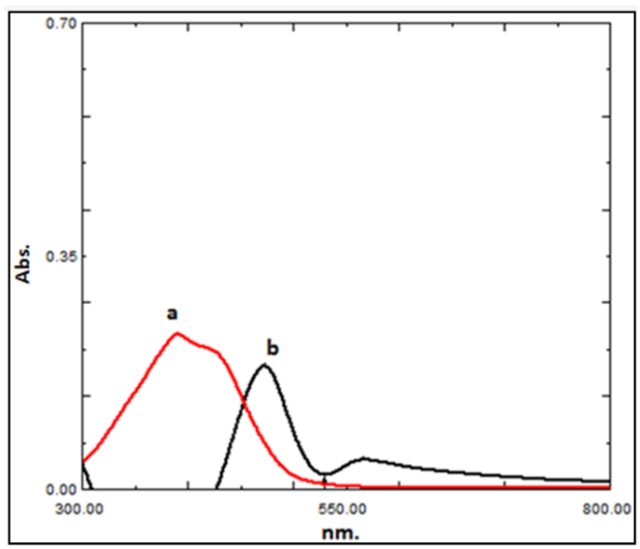


**Fig. 8.** Absorption spectrum of (a) Reagent NPAI = 5 x$10^{-4}$ M (b) Cu(II)- NPAI complex, Cu (II) = 1 x$10^{-5}$ M , pH = 7.5

## 3.5. Method Validation

The curves at the normal conditions were built by plotting the absorbance signal toward the each analytic concentrations. The solutions were put in the optical cell (10-mm) of the metal ion spectrophotometrically at the particular absorption maxima opposite the blank that formed at same conditions as shown in Table 3.

**Table 3**
Method validation of the spectrophotometric determination of complexes

| Parameter | Ni(II) | Cu (II) | Pb (II) | Cd(II) |
|---|---|---|---|---|
| λ max (nm) | 538 | 473 | 480 | 488 |
| Regression equation | A=0.0082C-0.0122 | A=0.0281 C- 0.0015 | A=0.0288C+0.0009 | A= 0.0061C + 0.0086 |
| Correlationcoefficient(r) | 0.9991 | 0.999 | 0.999 | 0.9995 |
| pH | 7 | 7.5 | 5-Apr | 4.5-5.5 |
| oncentrationrange(μgmL-1) | 25-Jan | 0.5-13 | 0.2-12.5 | 0.8-30 |
| LimitofDetection(μgmL-1) | 0.8 | 0.45 | 0.11 | 0.77 |
| LimitofQuantitation(μgmL-1) | 2.926 | 1.53 | 0.37 | 2.59 |
| Sandell'ssensitivity(μg.cm-2) | 0.0582 | 0.0262 | 0.022 | 0.0227 |
| Molarabsorptivity(L.mol-1.cm-1) | 1123 | 7640 | 4898.3 | 1182.2 |

## 4. Conclusions

Synthesis of Azo; 4,4'-((methylene bis(3,l-phenylen)) bis (diazene-2,l-diyl)) bis (benzene-1,3-diol) was done by using several methods from many metal salts to provide the metal complexes. Ligand and complexes structures were prepared depending on $^{1}$H-,$^{13}$C-NMR for ligand , elemental analysis, UV-Vis, FTIR, and electronic absorption. Coordinating of the azo ligand was done by N and (oxygen) of OH groups and (N=N) with metal ions resulting in complexes formation.

### Competing Interests

The authors have declared that no competing interests exist.

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