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LIGAND BASED SYNTHESIS AND CHARACTERIZATION OF CU (II) AND CO (II) COMPLEXES WITH 4-N-5-METHYL ISOXAZOL-3-YL

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ABSTRACT

The new ligand of 4-[N-(5-methyl isoxazol-3-yl) benzene sulphonamide azo] - 5- nitro-4-phenyl imidazol is presented in this research paper. Three chelate compounds are as well arranged through this ligand reaction with Cu (II) and Co(II) metal ions. The preparing process was carried out later than setting up the optimal concentration circumstances of U.V- visible spectrum of the complex solutions that were investigated for various concentrations and pH. The complex structures have been realized in accordance with mole ratio process that have gotten from the spectroscopy investigation of the compound solutions. The acquired metal: ligand ratios are (1:2) for every complex ion. (UV-Vis) absorbing spectrum of ethanolic complex solution depicted bath-chromic shift, unlike the free ligand. The investigated infrared spectrum of the chelating complexes may be a sign of that an organization among the metal ions and the organized ligand happens effectively. The conductivity and the metal ions percentage of the complexes have been measured. Besides, the structural complexes formations of Cu (II) and Co(II). Ions are proposed to be as octahedral.

KEYWORDS: Ligand based synthesis, characterization of Cu (II) and Co (II), -N-5-METHYL ISOXAZOL-3-YL.

INTRODUCTION

Synthesized of ligand of 5-((2,4-dichlorophenyl)azo)-2,2-dimethyl-1,3-dioxane-4,6-dione (DPOD) were organized previously in the literature. Also, three chelate complexes were set up by making a reaction between this ligand and the metal ions Co (II), Ni(II) and Cd(II). The preparing procedure was achieved after fitting the finest **UV-Vis** spectroscopy concentration of these complex solutions for different pH and concentrations that comply with Lampert-

Beers rule. The complexes geometries are figured out in relation to mole ratio technique that acquired from the complex solutions spectroscopic study. The reported metal: ligand ratios are (1:2) for the entire complex ions. (UV-Vis) absorption spectrum of ethanolic compos solutions was shown a bathchromic shift unlike free ligand. The infrared spectrum of the chelating complexes were investigated with management of the metal ions as well as the organized ligand. The conductivity measurements and metal ions percentage of the complexes have been concluded as well as the geometrical complex structures of the of Co (II), Ni(II), Cd(II) ions have proposed to be octahedral[1] On the other hand, ligand based on 4,5-diphenyl imidazole was organized. The ligand coordination is by means of the metal ions of Co (II),Ni(II),Cu(II),Zn(II),Cd(II) in the most advantageous situations of PH and concentration. The outcomes were explained that the complex concentration meet the terms of Lambert- Beers rule. Accordingly, a mole ratio was computed for the mentioned ligand complexes, so the ratio of (metal: ligand) was (1:2). While the ligand and its metal compounds was discriminated by means of IR and **UV-Vis** measurements, the H1 NMR was illustrated a coordination with compound prescriptions and the magnetic sensitivity records of metal complex ions had been verified the octahedral structure.[2] The preparation of ligand and its metal complexes rooted in Mn(II), Co(II), Ni(II), and Cu(II), and Cd(NO3)2 chloride salts have been made. The geometrical figures had been verified using investigative, diverse spectral methods and thermo gravimetric analysis. Complexes have been shaped with stoichiometries 1:1 and 2:3 (M: L) with electrolytic feature, excluding Cd(II) compound which is non-electrolyte. Every one of complex have octahedral constitution, excluding Cu(II) compound that possesses 2D square structure. IR spectrum recognized that ligand match up metal ions throughout oxygen and nitrogen atoms of amino and carbonyl collections of hydrazide moiety in neutralized structure. Thermal decomposition method of compounds has been explained and the compounds antioxidant activity has been confirmed, where Cu(II) compound exhibits upper antioxidant action over other compounds. The modeling of Molecule and docking of the investigative compounds into hAChE has been adopted by using Molsoft ICM 3.4-8C software package and the these compounds enhanced the ligand activity.[3] The preparing and metal complexes classification for Cr(III), Mn(II), Fe(III), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) ions with heterocyclic azo dye in form of ligand 2-[2─- (1- Hydroxy - 4- Chloro phenyl) azo]- imidazole (HClPAI) have been organized by adizonium chloride salt solution of 2-amino -4- chloro phenol reaction with imidazole in alkaline ethanolic solution. Azo dye ligand and their metal compounds were identified by investigative data, Mass spectrum, 1H NMR, IR, XRD, SEM, Electronic spectral data, thermal analysis (TG-DSC-DTG), molar conductance and magnetic susceptibility. The fundamental investigation of the metal compounds substantiate the stoichiometry of [M(L)2] Cl type in the case of M = Cr(III), Fe(III), Co(II) and [M(L)2] in the case of M = Mn(II), Ni(II), Cu(II), Zn(II), Cd(II), Hg(II) and L = azo dye ligand. The measurements of molar conductance for prepared metal compounds demonstrated 1:1 electrolyte for Cr (III), Fe(III) and Co(III) ions and non-electrolyte for other metal compounds. The data illustrate that the azo dye ligand acts a tridentate and synchronizes to the metal ion through nitrogen atom of azo group that is the furthest imidazole molecule, nitrogen atom of azomethine group of heterocyclic imidazole ring and phenolic oxygen. Octahedral environment has proposed for every metal complex as stated in.[4] The preparation of (Co2+,Ni2+,Cu2+,Zn2+,Cd2+andHg2+) Complexes with the ligand Ethyl cyano (2methyl carboxylate phenyl azo acetate) (ECA) were made and identified by Atomic absorption spectroscopy, (UV-Visible), FTIR, Molar conductivity and magnetic moments measurements. These broad formulas were given for the [M(ECA)2]Cl2 complexes in the case of M = (Co2+, Ni2+, Cu2+, Zn2+, Cd2+ and Hg2+) in the case of octahedral structure.[5]

MATERIALS AND PHYSICAL MEASUREMENTS

Every used chemical was of maximum clarity (BDH or Fluka).Absorption spectrum had been evaluated by UV-Vis 1700 spectrophotometer for complexes solution in aqueous ethanol by means of 1cm quartz cell at room temperature. IR spectrum had been determined with FT-IR-8000 Shimadzu, in (4000-400) cm-1 range by means of KBr disc. Measured Auto. Electrical conductivity using digital conductivity meter under Alpha – 800 and solute concentration of 10-3M in ethanol at room temperature. pH measurements had been conducted by (PH–meter), 720, WTW 82362.

Synthesis and characterization of azo ligand

Followed the method proposed by (Gusev)[5] and et.al in the preparation of this type of ligand by dissolving (2.5g,0.01mole)of 4-amino-N-(5-methylisoxazol -3-yl) benezensulfoamide in a mixture in a solution of 4mL concentration HCl and 25mL distilled water. After cooling this solution to 0 °C, 0.7g of sodium nitrite dissolved in 10mL distilled water was added with maintaining the temperature at 0 °C. This combination has been left inside for 15min to complete diazotization reaction. at latter, the diazonium solution has been drop by drop appended to a solution of (1.89g, 0.01 mole) 5- nitro-4-phenyl imidazol and 10% of sodium hydroxide dissolved in 150mL ethanol with keeping temperature at 0 °C. After completing addition, the contents were left for two hours, then added 150mL of cooled distilled water, and the pH of the solution maintained at 6 by HClleaving the precepitate for 24 hours and then washed several times with distilled water, then dried has been calculated the percentage of output and the degree of melting point.

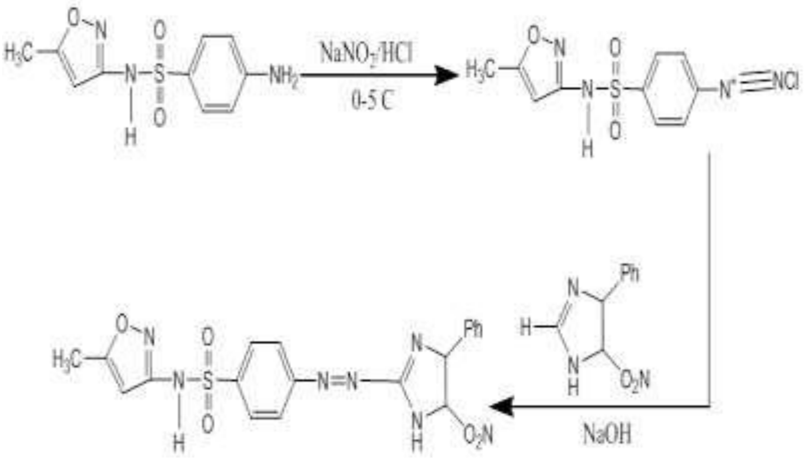

Fig. 1: 4-[N-(5-methyl isoxazol-3-yl) benzene sulfonamide azo] - 5- nitro-4-phenyl.

Imidazole

Complexes Synthesis

The chelate complexes have been structured at finest liquefied pH values of (0.137) of ligand (SMAS) in 10 ml ethanol and subsequently metal chloride 0.01 mol, MCl2= Cu (II) and Co(II). Under dissolving conditions in 10 ml, purified water drop has been supplemented through strong excitation to the ligand solution. The reaction mixture was prepared over night. Afterward, the complexes have filtered off purified distilled water. Table.1 collects some physical properties of (SMAS) and its complexes.

Table 1: Some collected physical properties.

No.	Compound	Color	m.pC°
1	$C_{19}H_{17}N_7O_5S$	orang	230 – 231
2	$(C_{19}H_{17}N_7O_5S)_2Co$	Red	287-288
3	$(C_{19}H_{17}N_7O_5S)_2Ni$	Brown reddish	290-291

RESULTS AND DISCUSSION

pH Effect

Appropriate pH magnitudes for metal complex solutions have been within (5-10) range. In order to calculate the optimal pH magnitudes of metal compound solutions, the pH effect on the absorption was investigated.

Metal: ligand ratios

The metal: ligand ratio(M:L) of compounds has been evaluated using the mole ratio technique at (λmax), fixed pH and concentration. The results have high conformity with the reported values for some azo complexes[6,7], which indicate that the ligand (SMAS) has chelate complexes formations with the metal ions of Cu(II), Co(II).

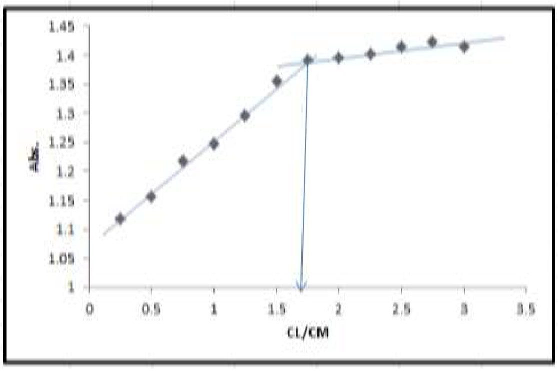

Figure 2: The (M:L) of Cu(II) with (SMAS).

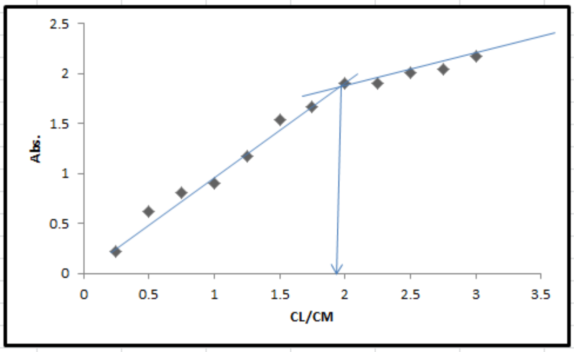

Figure 3: The (M:L) of Co(II) with (SMAS).

Absorption spectra

The ligand absorption spectrum (SMAS) and its complexes are investigated as shown in figures 6-8. The (max) of the ligand has been recorded at 423nm. The metal complex spectrums have been measured within (905 – 965) nm wavelength range. Additionally, the (max) of all compounds is illustrated in Table.2. Dual absorption bands have been come into view at the free ligand (SMAS) spectrum. The band at 260 nm is a sign of the $\pi \rightarrow \pi^*$ shifts of benzene ring, whereas the band at 423 nm is owing to the charge transfer complex.[8,9]

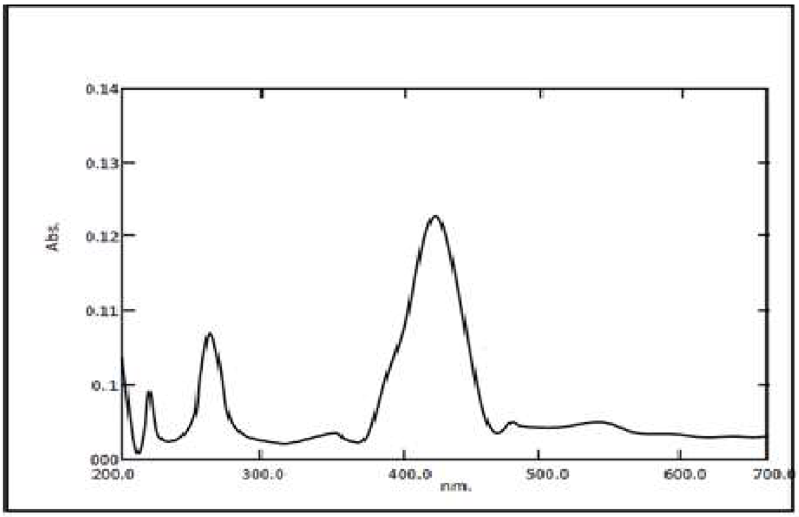

Figure 4: UV.-Vis. band ligand.

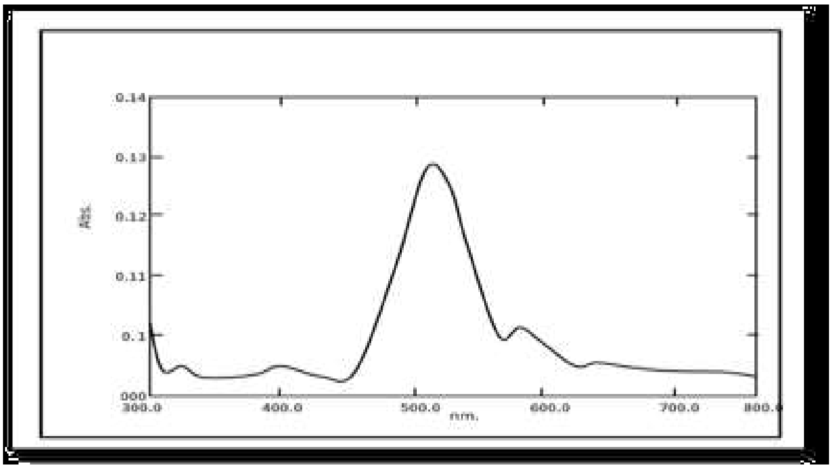

Figure 5: UV.-Vis. band of Co(II) complex with ligand ANBSA.

Table 2: The optimal pH values, concentration and wavelength (□□max) values of metal complexes.

Metal ions	Optimal pH	Optimal wave length (λ_{max})nm
Cu(II)	9	508
Co(II)	8	523

Infrared spectra

The infrared spectrum details of free ligand (SMAS) and its compounds with Cu (II) and Co(II) have been depicted in Figures 9-11 and Table.3. The shown spectrums are intricate in consequence of the wide-ranging overlap of bands number resulted from υ (N─H), υ(C═N), υ(N═N) and other bands owing to the phenyl in addition to benezensulfoamide ring as it is agreed with.[10]

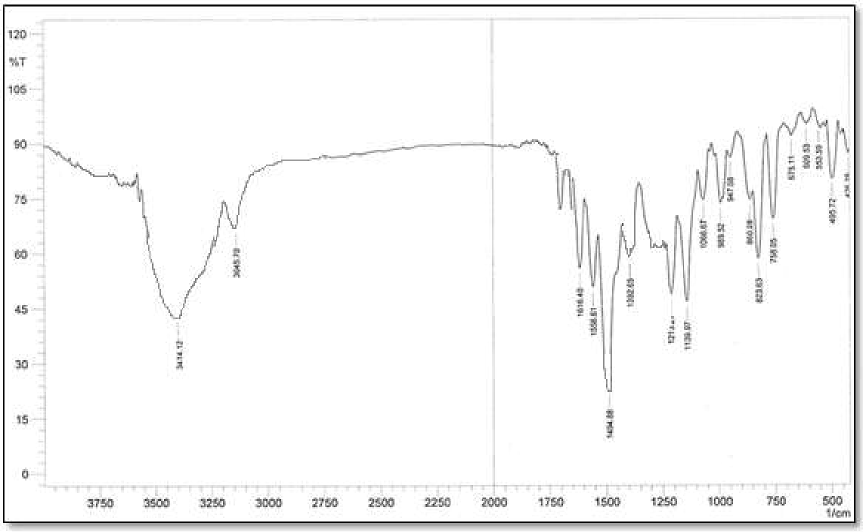

Figure 6: FT-IR spectrum of (SMAS).

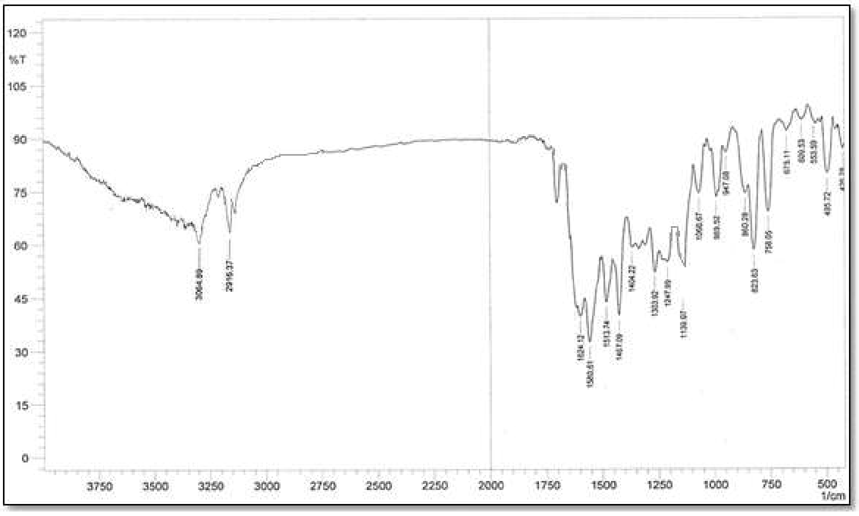

Figure 7: FT-IR spectrum of ion complex of Ni (II) with (SMAS).

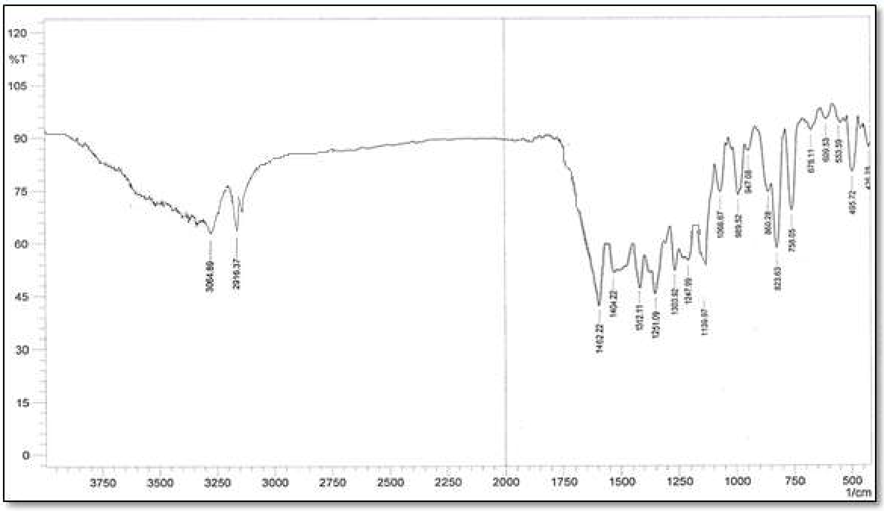

Figure 8: FT-IR spectrum of ion complex of Co (II) with (SMAS).

Table 3: Typical IR absorbance bands of the ligand (SMAS) and its compounds in units of cm-1.

Complex	υ(O─H)	υ(C=N)	υ(N=N)	υ(M─N)
$C19H17N7O5S$	3424.98	*1636.34*	*1484.98*	-
$(C_{19}H_{17}N_7O_5S)_2Cu$	-	1136.76	1475.35	445
$(C_{19}H_{17}N_7O_5S)_2Co$	-	1154	1480.21	490

Conductivity measurements

Based on the measurements, every complex has the conductivity values within arrange of (10.5– 10.6) S.cm2. mol-1 in ethanol solution. These measured magnitudes represent non-ionic structure of these complexes as it is agreed with[10] The values of measured conductivity are shown in Table 4.

Table 4: Conductivity measurements of complexes in Ethanol.

Complex	Λm ($S.mol^{-1}.cm^2$)in Ethanol
$[Cu\ L_2\ Cl_2]$	10.8
$[Co\ L_2\ Cl_2]$	15.5

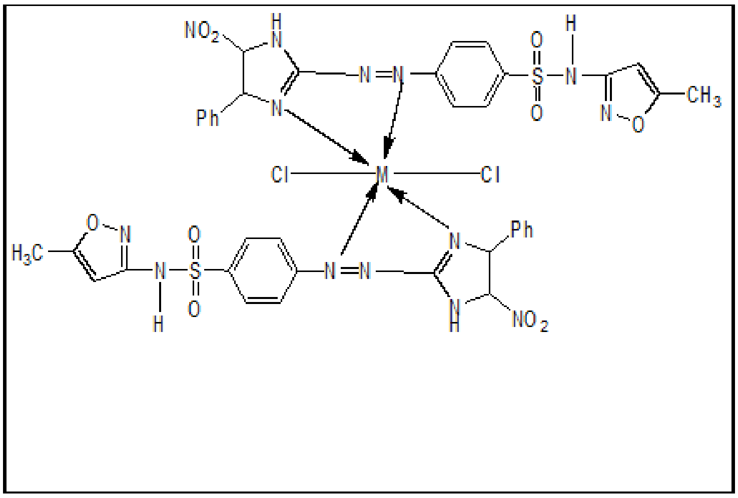

Figure 9: The structural formula of complex Ni(II).

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