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Synthesis and characterization of copper (II) complexes of some 2-hydroxy-4,5-dimethyl acetophenone substituted Hydrazones.

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ABSTRACT

The present paper deals with synthesis and characterization of metal complex of some novel 2-hydroxy-4,5-dimethylacetophenone substituted hydrazones. The substituted hydrazones were prepared by reacting 2-hydroxy-4,5-dimethyl acetophenone with corresponding hydrazine, Phenyl hydrazine and (2,4-dinitrophenyl)hydrazine. The Copper (II) metal complexes of general formula ML_2 with newly prepared hydrazone synthesized and characterized by several physiochemical techniques like UV, Infrared, ^{1}HNMR, decomposition temperature/melting point, elemental analysis, molar conductance and magnetic susceptibility studies. The analytical data confirmed 1:2 stoichiometry of M:L and based on above analytical technique data suggest that each one Cu (II) complexes have octahedral geometry. The conductivity data show that each one these complexes are non-electrolytes.

Keywords: *Transition Metal Complexes, Hydrazone, Mutagenicity, Non-Electrolytes, Phenyl Hydrazine, and (2,4 Dinitrophenyl) Hydrazine*

1. INTRODUCTION

Metal complexes have significant role since early days of coordination chemistry. Indeed, tremendous deal of work has been controlled on the synthesis and characterization of transition metal compounds mostly due to their applications in several fields. However, the power of a metal ion to contribute in bonding to all or any possible coordination sites depends partly on its preferences for the donor atoms of the ligand, the pliability and conformational adaptability of the ligand used, also as on the competition from other Lewis acids and different entities capable of occupying the coordination pocket.

It is well recognized that copper is an important element in human normal metabolism. In biological system, copper occurs as a variability of complexes which due to that the coordinated forms of copper are more stable than the equivalent ionic species[1]. These complexes are more active and necessary drugs than the ligands themselves, suggesting that the activity of such metal based drugs may be due to the in vivo development[2] of metallic complexes.

Complexes of copper demonstration several geometries. Among them most common are tetrahedral, trigonal bipyramidal, square planar, square pyramidal and octahedral. First row transition metal ions, copper complexes appearance distortions[3]. For example, the hexa coordinated Cu(II) ion with d^9 configuration desires distorted octahedral geometry, which is a direct significance of Jahn–Teller effect4. Thus, octahedral complexes usually occur with a set of four strongly and two weakly coordinating ligands.

The study of mononuclear copper complexes has been inspired by a desire to 'mimic' the active sites of metalloproteins such as the enzyme galactose oxidase5 and nitrite reductase6. One of the comforts in the coordination chemistry is synthesis of high-nuclearity transition metal complexes[7]. These complexes are studied as models for the multimetal active sites of metal-storage proteins[8], and as single molecule magnets[9-10]. Dinuclear copper complexes appeal consideration as models of active centers of copper covering enzymes and as potential components of homogeneous catalytic systems11. Dinuclear copper complexes were investigated frequently, because of the attention in new inorganic materials viewing molecular ferro- or antiferromagnetic interactions[12].

Copper Schiff base complexes have establish wide use, since copper (I) can be stabilized by Schiff bases and used in nitrene transfer. Schiff bases derived from two types of diamine have also been defined by Muller and Fruit[13]. For the preparation of these complexes mostly used copper(I), but copper(II) sources have also been measured. Copper is a biologically significant element and many enzymes that depend on copper for their activity. Because of its biological importance, some complexes of copper(II) were prepared and explored for their biological activities [14-15].

Research on coordination chemistry has developed as an active and exciting field for chemists and biologists because of the reasoning that coordination compounds are basically operated within the gathering of pharmaceuticals, fragrances, semiconductors, ceramic precursors etc.[16] Copper complexes are studied extensively as functional and structural models of active centres of copper containing redox enzymes. Some copper complexes exhibit capabilities of superoxide dismutase and chemical nuclease.

Copper is an important constituent of many metallo proteins and enzymes. It shows a significant role in the utilization of iron and in the process of photosynthesis as an electron carriers. Copper shortage also causes anemia due to the impaired hemoglobin synthesis. Higher agglomeration of copper in liver, kidney, and brain causes Wilson's disease. Plastocyanin, containing copper is existing in chloroplasts of green plants. The growths in the field of bioinorganic chemistry have enlarged the attention in hydrazone complexes, because it has been known that many of these complexes may act as lead for biologically important species[17-20].

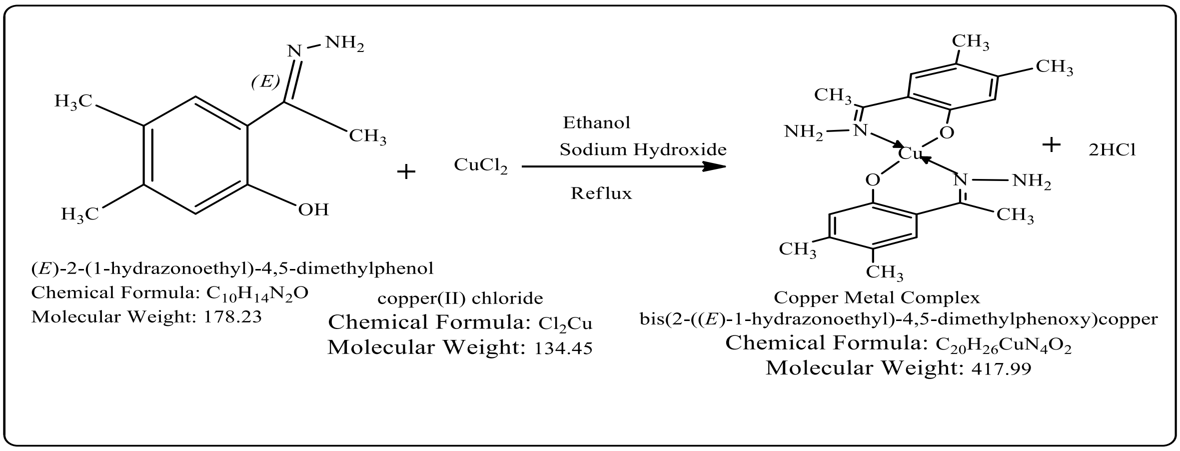
2. EXPERIMENTAL

2.1 Materials

The chemicals 2-hydroxy-4,5-dimethylacetophenone, (E)-2-(1-hydrazonoethyl)-4,5-dimethyl phenol, (E)-4,5-dimethyl-2-(1-(2-phenylhydrazono)ethyl)phenol, (E)-2-(1-(2-(2,4-dinitrophenyl) hydrazono)ethyl)-4,5-dimethylphenol, copper chloride, sodium hydroxide, ethanol, methanol, chloroform, DMSO, DMF etc., used in this work were of AR Grade, commercially available and used without further purification.

2.2 Preparation of Cu complex with (E)-2-(1-hydrazonoethyl)-4,5-dimethylphenol metal Complex or bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper

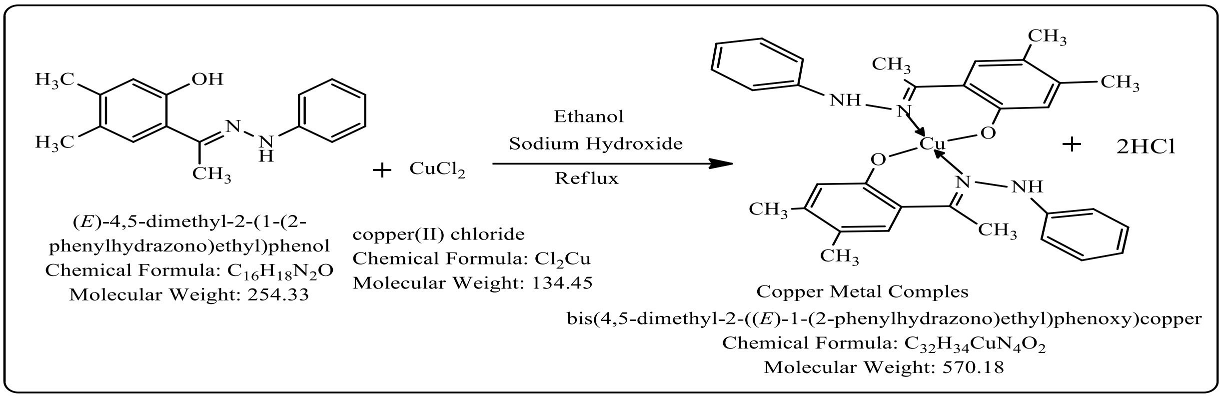
(E)-2-(1-hydrazonoethyl)-4,5-dimethylphenol were synthesized following published procedure[21]. The transition metal complex bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper were prepared by dissolution of (E)-2-(1-hydrazonoethyl)-4,5-dimethylphenol ligand (0.01M) in ethanol at hot condition, an ethanolic solution (0.005M) of the metal salt copper chloride was added drop wise with constant stirring and refluxed for 3 hrs. The resulting reaction mixture was cooled to room temperature and maintains pH 7 to 8 by addition of the 0.10 N sodium hydroxide. Thus light brown color precipitate was formed. The resultant product was filtered, repeatedly washed with ethanol and dried at 40°C to 45°C temperature. The compound was re-crystallized in ethanol for purification and improving description. Reaction scheme preparation of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper is given below Scheme 1.



Scheme 1: Reaction scheme preparation bis(2-(€-1-hydrazonoethyl)-4,5-dimethyl phenoxy) copper.

2.3 Preparation of Cu complex with (E)-4,5-dimethyl-2-(1-(2-phenylhydrazono)ethyl) phenol metal complex Or bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl)phenoxy) copper

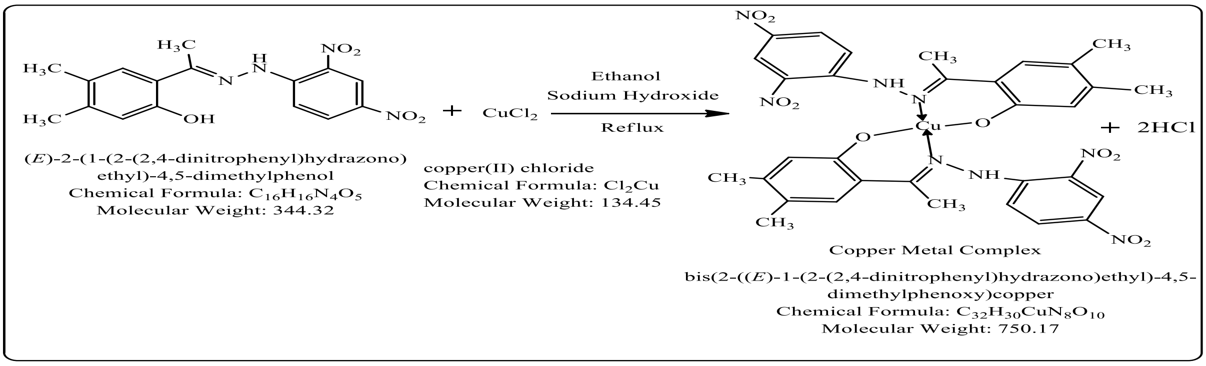
(E)-4,5-dimethyl-2-(1-(2-phenylhydrazono)ethyl)phenol were synthesized following published procedure[22]. The transition metal complex bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl)phenoxy)copper were prepared by dissolution of (E)-4,5-dimethyl-2-(1-(2-phenyl hydrazono)ethyl)phenol ligand (0.0069M) in ethanol at hot condition, an ethanolic solution (0.0035M) of the metal salt copper chloride was added drop wise with constant stirring and refluxed for 4 hrs. The resulting reaction mixture was cooled to room temperature and maintains pH 7 to 8 by addition of the 0.10 N sodium hydroxide. Thus light brown color precipitate was formed. The resultant product was filtered, repeatedly washed with ethanol and dried at 50°C to 55°C temperature. The compound was re-crystallized in ethanol for purification and improving description. Reaction scheme preparation of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl)phenoxy)copper is given below Scheme 2.



Scheme 2: Reaction scheme preparation bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl) phenoxy)copper.

2.4 Preparation of Cu complex with (E)-2-(1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenol metal complex Or bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenoxy)copper

(E)-2-(1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenol were synthesized following published procedure[23]. The transition metal complex bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono)ethyl)-4,5-dimethylphenoxy)copper prepared by dissolution of (E)-2-(1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenol ligand (0.005M) in ethanol at hot condition, an ethanolic solution (0.0025M) of the metal salt copper chloride was added drop wise with constant stirring and refluxed for 5 hrs. The resulting reaction mixture was cooled to room temperature and maintains pH 7 to 8 by addition of the 0.10 N sodium hydroxide. Thus reddish brown color precipitate was formed. The resultant product was filtered, repeatedly washed with ethanol and dried at 60°C to 65°C temperature. The compound was re-crystallized in ethanol for purification and improving description. Reaction scheme preparation of bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenoxy) copper is given below Scheme 3.



Scheme 3: Reaction scheme preparation bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenoxy)copper.

3. RESULTS AND DISCUSSION

The Cu-Metal complexes were analyzed by Infrared spectroscopy, Ultraviolet–visible spectroscopy, ^{1}HNMR, Carbon-Hydrogen-Nitrogen-Sulphur elemental analyzer, Decomposition temperature/Melting point, Molar Conductance, Magnetic susceptibility and Copper content by Gravimetric technique.Decomposition temperature / Melting points were recorded using Veego Scientific Device (Model: VMP-AD) in open capillaries and were uncorrected. Some physical properties of Cu-Metal complexes are mentioned in Table 1.

Table 1: Physical properties

Cu-Metal Complex	Chemical Formula and Molecular formula	Color	Solubility	Decomposition Temperature	Yield
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy)copper	$C_{20}H_{26}CuN_4O_2$ and 417.99	Light Brown	- Soluble in methanol, ethanol, Acetonitrile DMSO, DMF - Insoluble in water	> 300 °C	69.37%
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl)phenoxy) copper	$C_{32}H_{34}CuN_4O_2$ and 570.18	Light Brown	- Soluble in ethanol, methanol, DMSO, DMF - Insoluble in water	> 300°C	78.98%
bis(2-((E)-1-(2-(2,4-dinitrophenyl) hydrazono)ethyl)-4,5-dimethyl phenoxy)copper	$C_{32}H_{30}CuN_8O_{10}$ and 750.17	Reddish Brown	- Soluble in ethanol, methanol, DMSO, DMF - Insoluble in water	> 300°C	78.00 %

3.1 Infrared analysis

The IR spectra of Cu-Metal complexes were recorded in the region of 4000–400 cm^{-1} using FTIR spectrometer of model Agilent Resolutions Pro by direct sampling method.

The IR spectral data along with the possible assignments of Cu-Metal complexes are provided in Table 2, followed by IR spectra performed by IR direct solid method, use of Agilent Resolutions Pro and IR spectrum are showed in Figure 1, 2, 3.

Table 2: Hydrazone Copper metal complex IR frequency, cm-1

Bond/ functional group	**frequency, cm-1**		
	bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy)copper	bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono) ethyl) phenoxy)copper	bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethyl phenoxy) copper
N-H	3295	3392	3302
C–H	3023	3010	3084
C–H stretch aromatics	2963	2922	2922
C=N	1608	1620	1614
C–C	1510	1599	1592
C=C (aromatic ring)	1441	1467	1420
N-O	---	---	1331
C–O	1247	1248	1261
N-N	1119	1142	1115
O-Cu	Below 600	Below 600	Below 600

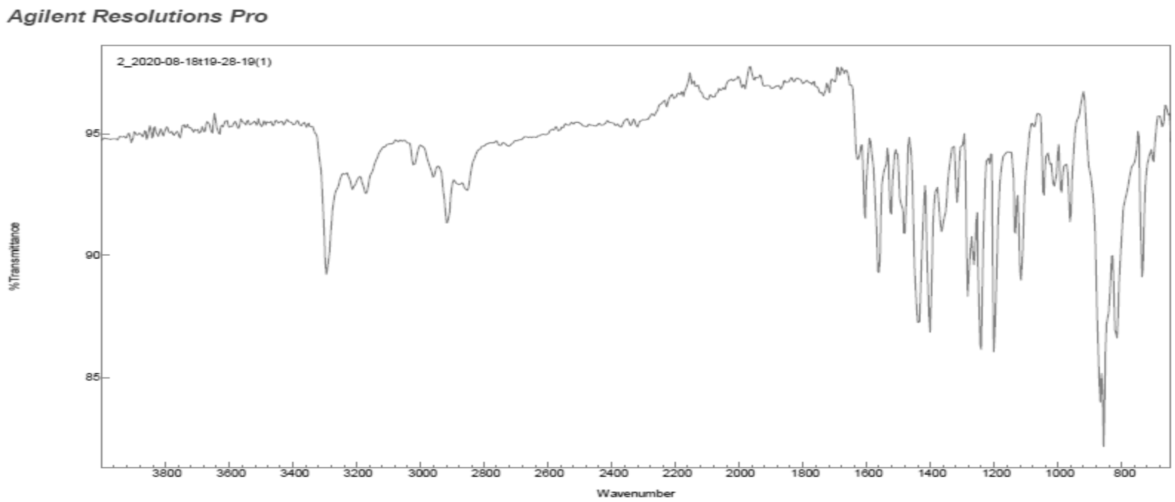


Fig. 1: IR of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper

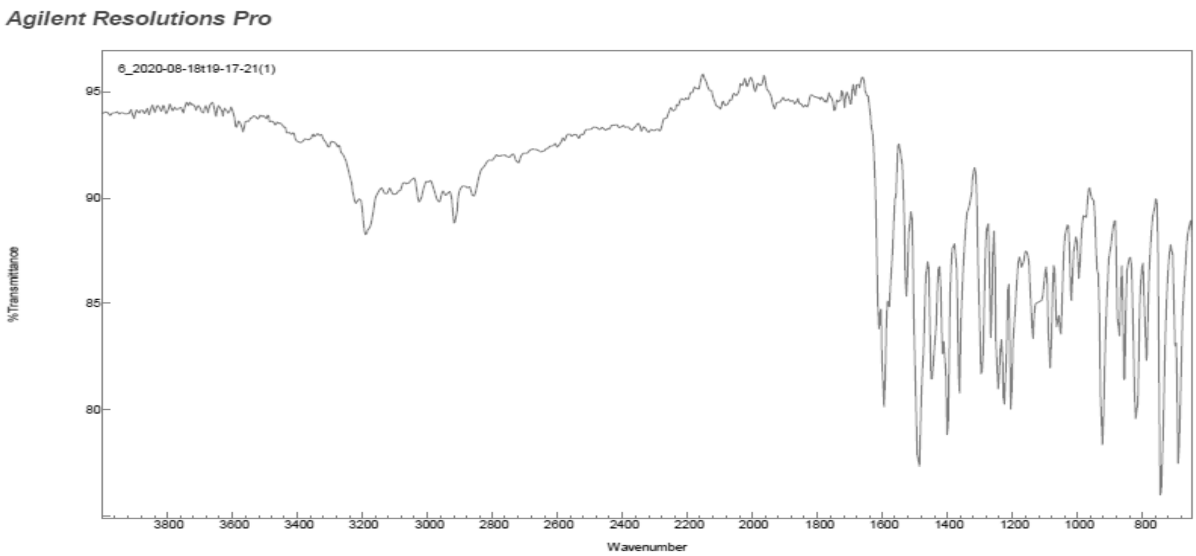


Fig. 2: IR of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl)phenoxy)copper

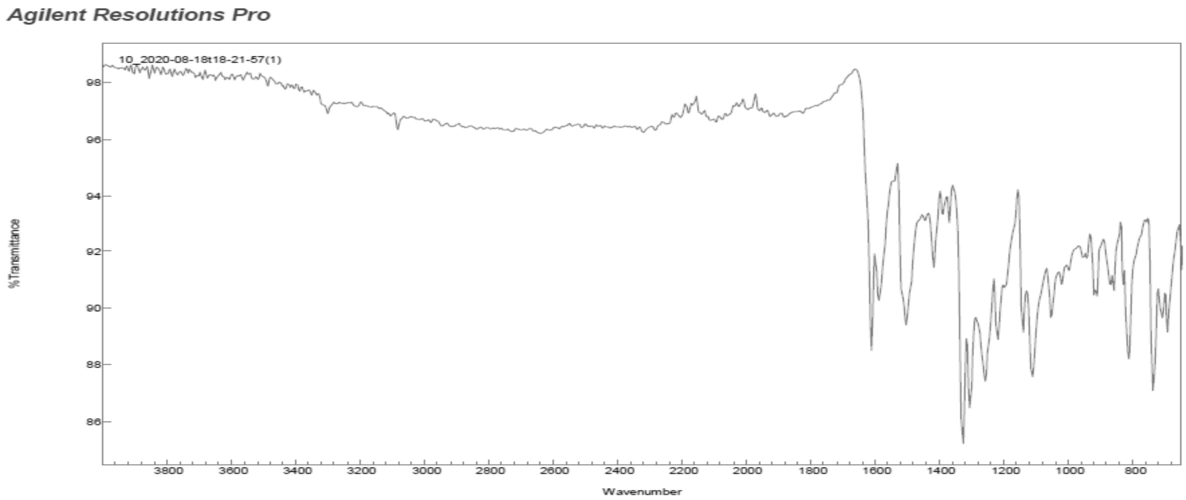


Fig. 3: IR of bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenoxy) copper

3.2 UV analysis

The UV spectra of the Cu-Metal complexes in methanol were recorded on SPECORD 50 PLUS-233 H 1409C Spectrophotometer, using a quartz cell of 1 cm optical path where methanol was used as a blank. The UV spectrum is shown in Figure 4, 5, 6. The spectra shows V (bands maximum in nm) are provided in Table 3.

Table 3: Cu-Metal Complex

Cu-Metal Complex	Wavelength nm
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethyl phenoxy)copper	277, 351, 551, 870
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl)phenoxy) copper	242, 305, 331, 468, 847
bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethyl phenoxy)copper	248, 316, 337, 862, 906

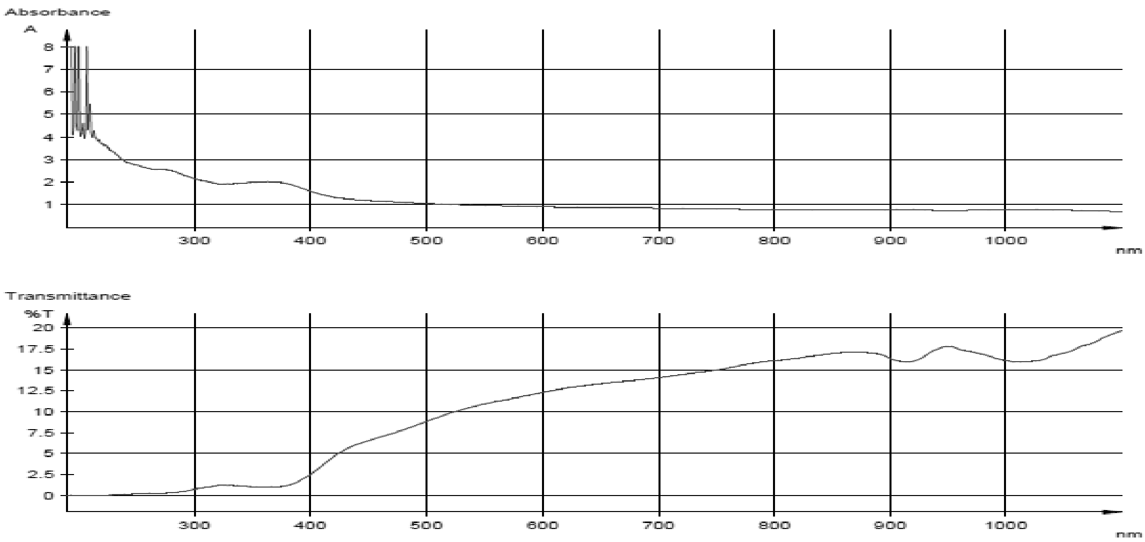


Fig. 4: UV spectra of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper

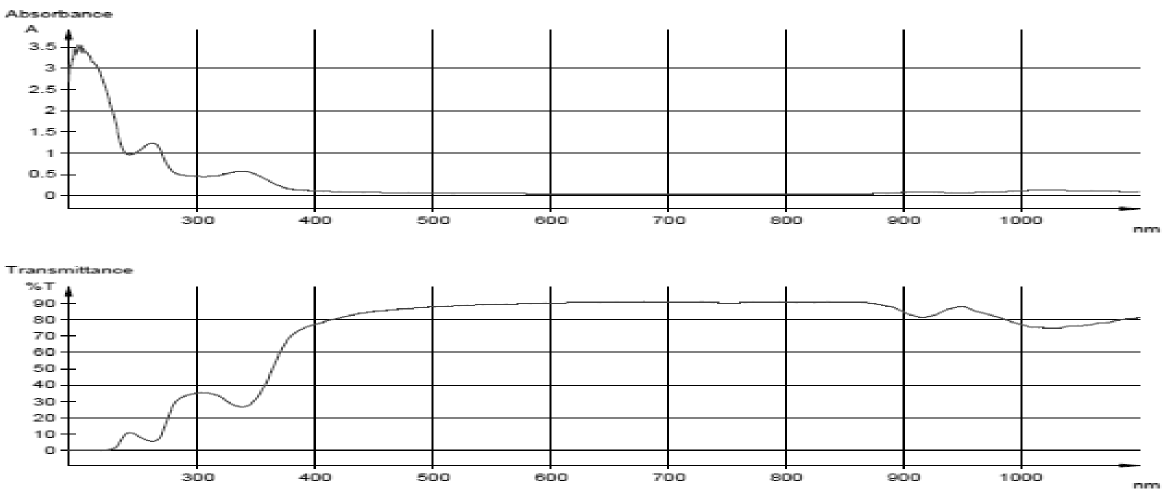


Fig. 5: UV spectra of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl)phenoxy) copper

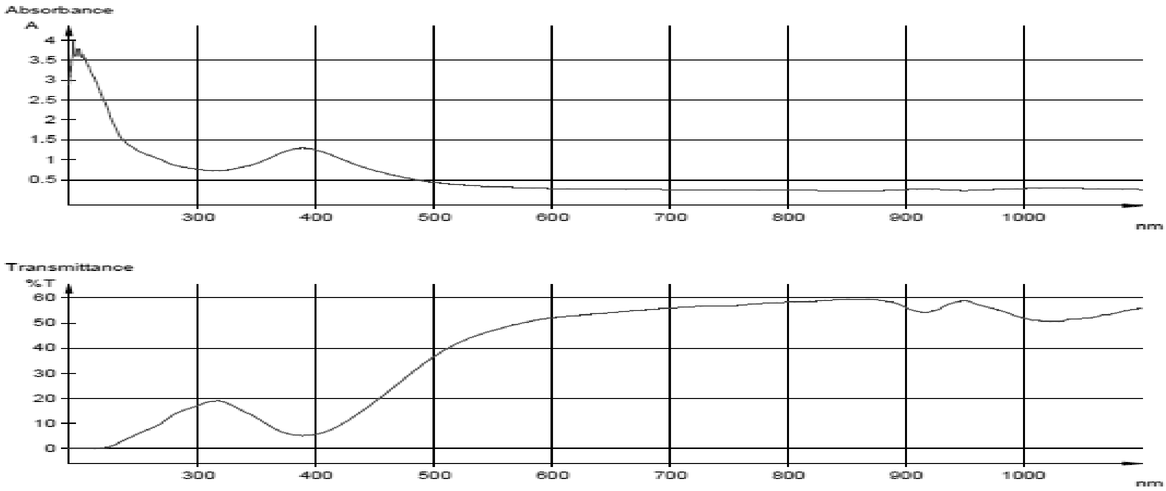


Fig. 6: UV-Visible Spectra of bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenoxy)copper

3.3 Elemental analysis (CHNS)

Formation of Cu-Metal complexes were further confirmed by elemental analysis which was recorded by Vario MICRO CHNS analyzer. The elemental analysis data of Cu-Metal complexes and Cu is confirmed by Gravimetric technique is summarized in Table 4.

Table 4: Carbon, Hydrogen, Nitrogen, Copper elemental analysis.

Metal Complex	Molecular formula	Molecular weight	Elemental data (required/found)			
			Carbon	Hydrogen	Nitrogen	Copper
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper	$C_{20}H_{26}CuN_4O_2$	417.99	57.15 (57.47)	5.95 (6.28)	13.05 (13.41)	14.79 (15.20)
bis(4,5-dimethyl-2-((E)-1-(2-phenyl hydrazono)ethyl) phenoxy)copper	$C_{32}H_{34}CuN_4O_2$	570.18	66.86 (67.40)	5.75 (6.02)	9.12 (9.83)	10.83 (11.15)
bis(2-((E)-1-(2-(2,4-dinitro phenyl) hydrazono)ethyl)-4,5-dimethyl phenoxy)copper	$C_{32}H_{30}CuN_8O_{10}$	750.17	50.95 (51.23)	3.65 (4.04)	14.41 (14.93)	7.99 (8.47)

3.4 Magnetic susceptibility

For copper complexes, many of researchers reported the value of magnetic moment (μeff) values at room temperature lies between 1.75 and 2.20 B.M.[24] which is reported in Table 5. The value of magnetic moment in our study lies between 1.77 and 2.05 B.M. which may be attributed to the orbital contribution and to the distortion of octahedral structure leading to a square planner geometry of the molecule[25]. Due to the polymeric species formation, a decrease in the normal magnetic moment value has been reported in most of the Cu(II) complexes. This subnormal value of magnetic moment of the Cu(II) complexes at room temperature indicated a partial spin.

Table 5: Magnetic susceptibility

Cu-Metal complex	μ_{eff} value
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper	1.78
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl) phenoxy) copper	1.86
bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenoxy)copper	2.02

3.5 Molar Conductance (mho.cm^2.mol^{-1}):

The molar conductance values at room temperature were measured in Methanol (10^{-3} M) on an Elico digital direct reading conductivity meter model CM-180 is summarized in Table 6.

Table 6. Molar Conductance

Cu-Metal complex	Molar Conductance mhos cm^2 mol^{-1}
bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper	0.082
bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl) phenoxy) copper	0.082
bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethyl phenoxy)copper	0.080

3.6 ^{1}H NMR:

We tried all Cu-complex 1HNMR and observe the spectrum of the complex differs from that of the free ligand in the following aspects:

1. Resolution is very poor due to the low solubility of Cu-Metal complexes in DMSO.
2. The disappearance of the signal due to the imine group, is attributed to its involvement in coordinating the copper ion[26].
3. The spectrum displays a multiple signal at δ (6.58–7.63 ppm) assigned to aromatic ring protons.

The 1H NMR Spectra of metal complexes are shown in Figure 7, 8, 9.

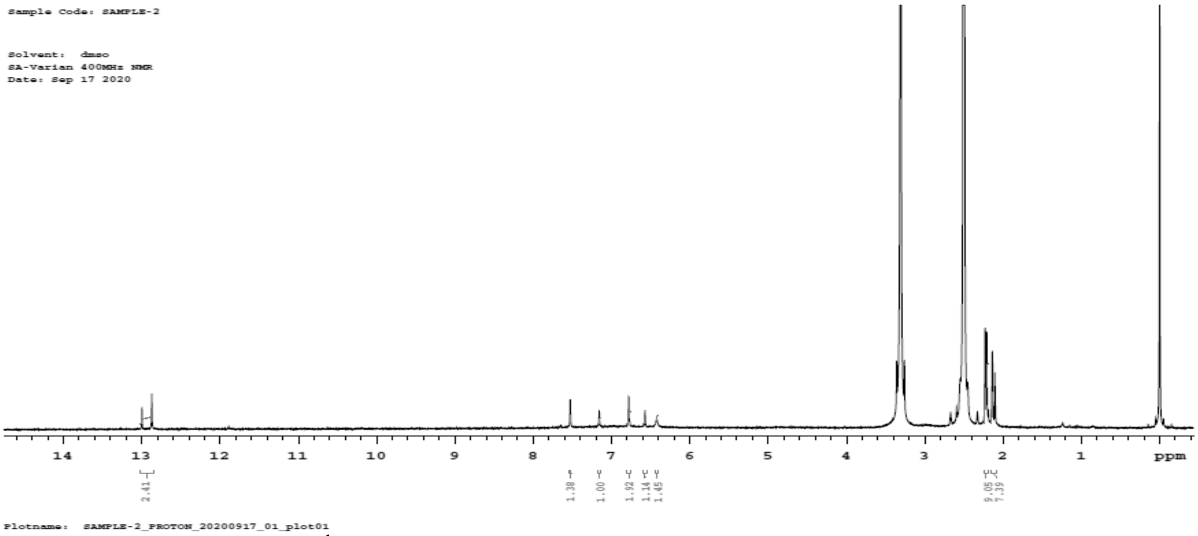


Fig. 7: ^{1}HNMR of bis(2-((E)-1-hydrazonoethyl)-4,5-dimethylphenoxy)copper

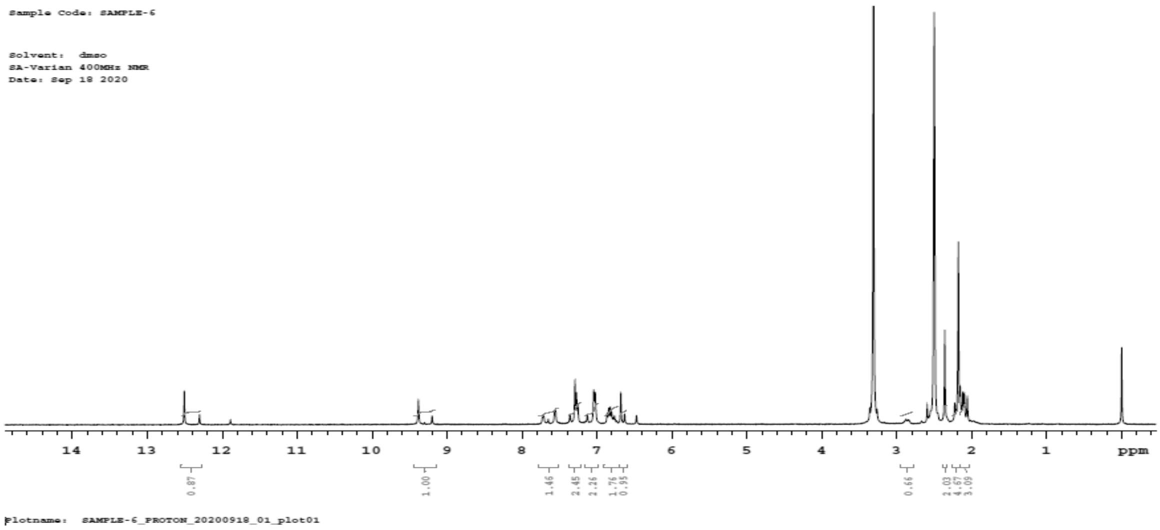


Fig. 8: ^{1}HNMR of bis(4,5-dimethyl-2-((E)-1-(2-phenylhydrazono)ethyl)phenoxy)copper

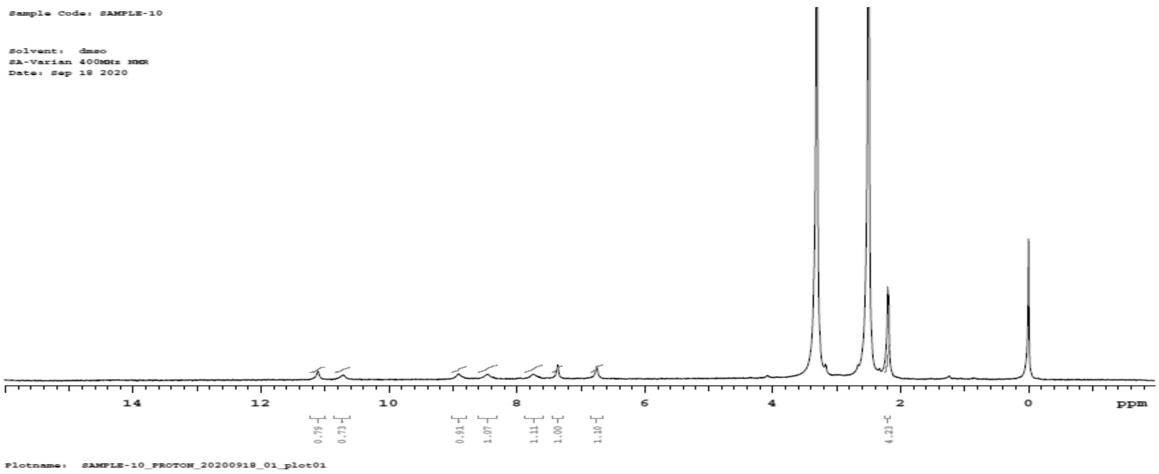


Fig. 9: ^{1}HNMR of bis(2-((E)-1-(2-(2,4-dinitrophenyl)hydrazono)ethyl)-4,5-dimethylphenoxy) copper.

4. CONCLUSION

All Cu complexes are characterized by elemental analysis, IR, UV Visible spectrophotometer, 1HNMR, Molar Conductance and Magnetic susceptibility measurement, gravimetric analysis. Based on above results it suggests the Octahedral geometry for the Cu(II) complexes and The conductivity data show that each one these complexes are non-electrolytes.

5. ACKNOWLEDGEMENTS

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