

# Synthesis, Characterization and biological activity of Some Transition Metal Complexes with New Schiff Base Ligand Type (NNO) Derivative from Benzoin

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## Abstract

The research included preparation of New Schiff base by two steps: Preparation of Schiff base (HDEA) by react (4-aminoantipyrine) with (Benzoin) as first step then react the prepared Schiff base with (3-aminoacetophenone) to have the new Schiff base(HDEAP) as second step. The reaction was follow by TLC technic and the melting point was measure. The new Schiff base(HDEAP) was characterize by available spectral technics of Mass spectra , <sup>1</sup>H-NMR, Infra-Red spectra and Uv-Vis spectra. The complexes of new Schiff base(HDEAP) with Co(II) , Ni(II) and Cu(II) were prepare. The mole ratio was (1:2) (M:L) depend on Micro Elemental Analysis (C.H.N). The complexes characterized by spectral technics such as IR and Uv-Vis spectrums also by Magnetic, Molar Conductivity measurements and the percentage of metal ions calculations. Depending of these results the new Schiff base (HDEAP) behave as tri dentate ligand type (NNO) with mono negative charge. The suggested structure were Octahedral formula for all complexes. All these compounds were determined aligned with two classes of human pathogenic; bacteria gram positive and gram negative. The performance results explain that the metal complexes have greater antibacterial action over than the free ligand.

**Keywords:** Schiff base, Benzoin Derivative, Metal complexes, biological activity

## INTRODUCTION

Cobalt, Nickel and other transition metals complexes have brightly colored and are often strange empirical formula [1]. These compounds are called coordination complexes [2]. The presence of two, four, five, six and more chemical groups positioned geometrically around the metal ion to give their major proposed feature. These groups called coordinating groups change The chemical behavior of metal significantly such as electronic properties that the free metal or ion does not have and can adsorb visible light [3]. Large areas of biochemistry are applied transition metal chemistry [4]. Schiff base and their metal complexes are hugely widespread because of their miscellaneous chelating capabilities. They show significant function in analytical structural studies as a consequence of their preparative convenience and fundamental miscellany [5]. Towards a variety of metal atoms, the metal complexes of Schiff bases are expansively considered as a result of flexibility, sensitivity, synthetic and selectivity. They can be employed as catalysts, dyes , intermediates in biological formation and as polymer stabilizers. Numerous Schiff's base molecules have organic actions of antifungal, antiseptic, anti-inflammatory, antitumour, anti-proliferative, anticancer, anti-corrosion and antidiabetic performances [6].

Benzoin is one of  $\alpha$ - Hydroxy ketones compounds which is useful in the synthesis of Schiff base ligands that are well known of application in pharmaceutical , anti-microbial and industrial uses[7]. The spectroscopic and electro chemical performance of Schiff bases metallic compounds of benzoin moiety is yet considered [8]. As a consequence, this work has dedicated on chemical and spectroscopic properties of newfangled Schiff base ligand derivative from benzoin and its complexes with Co<sup>+2</sup> , Ni<sup>+2</sup> and Cu<sup>+2</sup>.

## MATERIALS AND METHODS

### Chemicals and Measuring Tools

Entirely used elements are of the maximum purity ((BDH , Fluka or merck) without using any refining.

Elemental analysis has been performed using EA300A Eurovector micro analytical unit of element analyzer .Absorption spectrums have been documented by Shimadzu UV-Vis 1700 spectrophotometer, for complexes solution in aqueous ethanol at room temperature with 1cm quartz cell. IR spectrums have been

documented with FT-IR-8000 Shimadzu, in (4000-400) cm<sup>-1</sup> range via KBr disc. Auto .Electrical conductivity determined by Alpha-800 conductivity measuring tool with solute concentration of 10<sup>-3</sup>M in ethanol at room temperature, Magnetic susceptibility magnitudes had been found at room temperature by the Gouy technique and Johnson Matthey Catalytic system. The metallic ratios have been measured using atomic absorption procedure by Shimadzu -AA-160. <sup>1</sup>H-NMR spectrum had been calculated on a DRX (300-MHz) spectrometer in DMSO, Mass spectrum in agilent mass spectrometer 5975 quadropoleanalyser and Thin Layer Chromatography (TLC) had been achieved on aluminum plates covered with gel of silica. Antibacterial screening was done at Biology Department, in University of Kufa.

### Formulation of newfangled Schiff base Ligand (HDEAP).

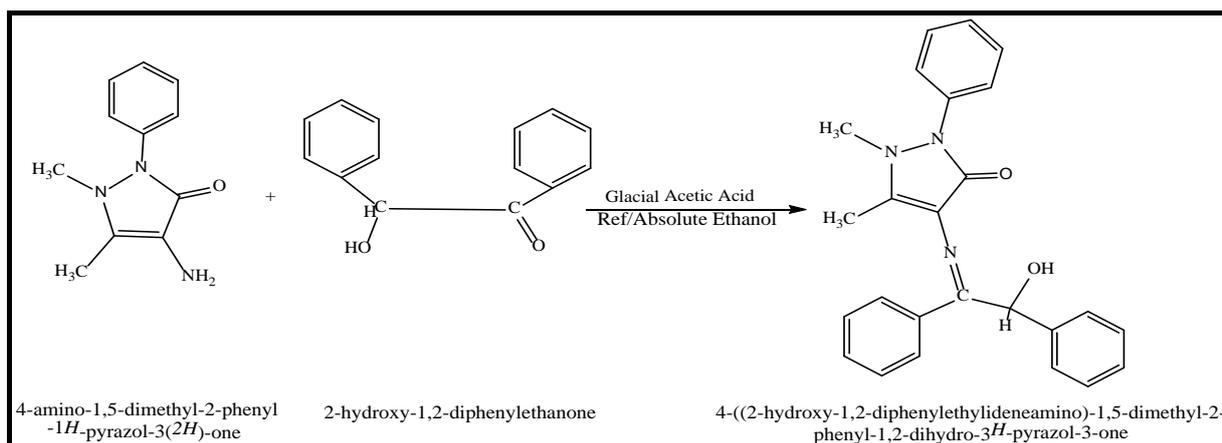
The preparation of the ligand (HDEAP) include two steps: The first one is preparation of the Schiff base (HDEA) which was resulted from condensation of 4-aminoantipyrine and Benzoin. The second step was preparing the new-fangled Schiff base ligand by acid catalyzed concentration of the product of step one (HDEA) with 3-aminoacetophenone in absolute ethanol.

### 1-Preparation of Schiff base (HDEA)

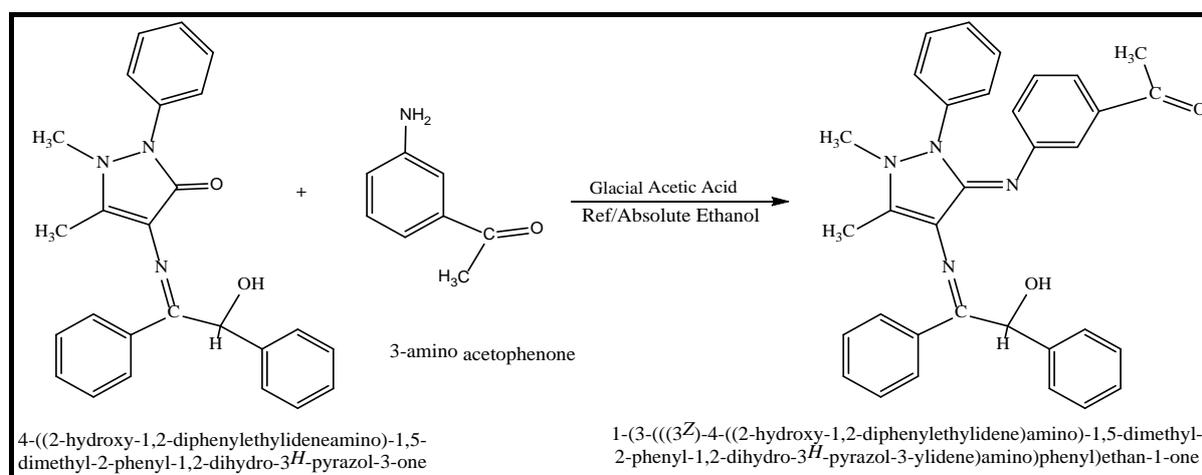
In a curved bottom container, 4-aminoantipyrine (0.01 mol, 2.032 g) in (15 mL) ethanol and benzoin (0.01mol, 2.122 g) in (15 mL) absolute ethanol have been inserted with limited drops of glacial acetic acid to solution. The mixture has been refluxed for 4 hours and the precipitous product has been gotten through separation and recrystallization from hot ethanol, and dehydrated over anhydrous CaCl<sub>2</sub>, the starting material; yield: 77.4%, m.p: (157-158) C, Rf: 0.52. The preparation showed in Scheme 1[9].

### 2- Formulation of newfangled Schiff base Ligand (HDEAP)

HDEAP has been organized by condensing (HDEA) compound (0.01 mol, 3.974 g) that liquefied in (15 mL) absolute ethanol and refluxed with (0.01 mol, 1.351 g) of 3-aminoacetophenone for (3 hr) [10]. Addition of 3 drops from glacial acetic acid, a distinct colored solution can be gotten. The prepared Schiff base Ligand has isolated after the mixture volume has leessened to half through vaporization and recrystallization using hot absolute ethanol and dehydrated over anhydrous CaCl<sub>2</sub>, Scheme 2 shows the ligand formulation; yield: 79.8%, m.p: (169-170) C, Rf: 0.44 .



Scheme 1:- Preparation of the starting material (HDEA)



Scheme 2:- Formulation of the ligand (HDEAP)

### Formulation of metal complexes

The (1:2) chelate complexes of the metal and the ligand are prepared by dissolving (0.002 mol, 0.514 g) Schiff base (HDEAP) in (15 ml) of heated absolute ethanol. The corresponding hydrated metal chloride salts of ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  and  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ) of (0.001 mol) was dissolved in hot absolute ethanol (15 mL) was mixed with heated absolute ethanol solution of the ligand and refluxed for (1 hr) on a water bath and the contents was cooled after that. The complexes separated out in each case. The product has been filtered, washed with absolute ethanol and dried under vacuum. Table 1 illustrates the physical and analytical properties for those compounds.

### Biological activity study

All the newly synthesized ligand and all complexes have investigated alongside gram positive bacteria (*Staphylococcus aureus*, *Streptococcus pyogenes*) and gram negative bacteria (*Escherichia coli*, *Klebsiella pneumonia*) species using Kirby-Bauer paper disk diffusion method [11,12]. Muller Hinton Agar was used to culture the test bacteria. The chemical solutions used in the biological study are prepared by using dimethyl sulfoxide (DMSO) as solvent, where attended a single concentration (C)  $1 \times 10^{-3}\text{M}$ . The dishes are incubated at a temperature of  $37^\circ\text{C}$  for complete day. The formed inhibiting region by compounds against the specific tested bacterial strain can be used to evaluate the antibacterial activities of the synthetic compounds. The obtained mean magnitude for three individual replicates has been employed to compute growth inhibition zone of every sample.

## RESULTS AND DISCUSSION

### General

HDEAP Schiff base ligand is Brown crystal, that is partially soluble in water and soluble in universal biological solvents. Reacting this ligand with the metallic ions has different color crystals. All complexes are reasonably air-stable, insoluble in water, but solvable in most organic solvents as dimethyl sulfoxide, dimethylformamide, chloroform, acetone, methanol and ethanol.

### Physical Characteristics and Elemental Investigation

The physical characteristics and outcomes taken from C.H.N. investigation and metal substances of the arranged complexes are explained in Table 1. The investigative data had been acceptable with planned magnitudes. Molecular procedure of the ligand and its metal complexes had been proposed in relation to these data jointly with those acquired from spectral in addition to magnetic susceptibility of metal complexes. Every (1:2) metal to ligand solid complexes have been separated.

### Thin Layer Chromatography (TLC)

The ligand (HDEAP) solution and its complexes in ethanol as solvent have come into view as single spot. Each has confirmed that every one of these compounds are clear and have just single isomer [13]. Table 2 illustrates the Rf for the starting material, ligand and its complexes.

**Table 1: Physical characteristics and analytical details of HDEAP ligand and its complexes**

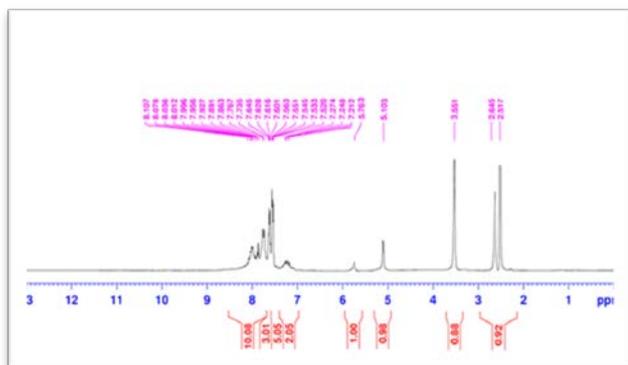
No.	Compounds	Color	Mwt	M.P C°	Yield, %	Found (Calc.)%			
						C	H	N	M
1	$C_{33}H_{30}N_4O_2$ (HDEAP)	Brawn	514.61	169-170	79.8	77.23 (76.95)	5.91 (5.82)	11.34 (10.88)	—
2	$[Co(C_{33}H_{29}N_4O_2)_2]$	Reddish-Brawn	1084.12	193-194	81.2	73.71 (73.05)	5.83 (5.16)	11.0 (10.33)	5.97 (5.43)
3	$[Ni(C_{33}H_{29}N_4O_2)_2]$	Dark Red	1083.88	188-189	78	74.21 (73.07)	5.74 (5.16)	11.10 (10.33)	5.90 (5.41)
4	$[Cu(C_{33}H_{29}N_4O_2)_2]$	Olive	1088.74	208-209	83.5	73.11 (72.74)	5.72 (5.14)	10.96 (10.28)	6.43 (5.83)

**Table 2: RF details for the starting material of Schiff base ligand and its complexes**

Compounds	Molecular weight (g/mol)	Rf
$C_{25}H_{23}N_3O_2$	397.48	0.52
$C_{33}H_{30}N_4O_2$	514.61	0.44
$[Co(C_{33}H_{29}N_4O_2)_2]$	1084.12	0.31
$[Ni(C_{33}H_{29}N_4O_2)_2]$	1083.88	0.35
$[Cu(C_{33}H_{29}N_4O_2)_2]$	1088.74	0.26

**NMR Spectra for the Ligand (HDEAP)**

The  $^1H$ -NMR band of ligand (Figure 1) exhibited two signals in high field at  $\delta=2.51$  and  $\delta=2.64$  ppm is attributed to (C-H) methyl antipyrine and (C-H) methyl carbonyl groups respectively (144,147), (C-H) methyl N-antipyrine appeared at (3.55) ppm [14], signals at (5.1, 5.76) due to (C-H and O-H) protons of ketol group in benzoin moiety[15]. The manifold peaks at  $\delta(7.21-8.1)$  ppm that had been attributed to chemical shifts of aromatic protons of phenyl rings [16],[17].

**Figure 1:  $^1H$ -NMR band of HDEAP Schiff base ligand****Mass Band of HDEAP Ligand**

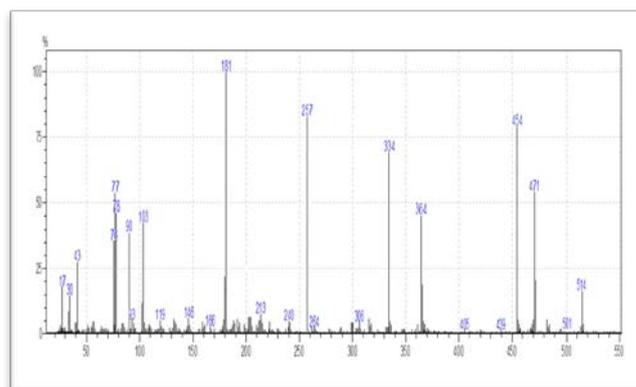
The mass spectrum of HDEAP Schiff base ligand as in (Figure 2) depicts a molecular ion peak at  $m/z$  514 related to  $[C_{33}H_{30}N_4O_2]$  ion. The peaks at  $m/z=471$ , 454, 364, 334, 257, 181, 181 and others are due to different fragments. The intensities of residual peaks are in line with their abundances. Other fragments are summarised in Table 3. [18,19].

**IR Spectrums of Ligand and its complexes**

The IR spectral data of Schiff base ligand (Figure 3) and their complexes have depicted in Table 4. IR spectrums of the complexes have been compared with the free ligand for evaluating the coordinating sites that involved in chelation. The free ligand spectrum has strong band in  $(3374) cm^{-1}$  which due to  $\nu(O-H)$  stretching vibration of Benzoin moiety [20], this band disappeared in each of Co (II), Ni(II) and Cu (II) complexes spectra specifies this band share in complexation in complexes [21]. The spectrum of the free ligand exhibit band at  $(1738) cm^{-1}$ ,

that can be related to carbonyl group of 3-amino acetophenone moiety. This band didn't change in the spectrums of complexes.

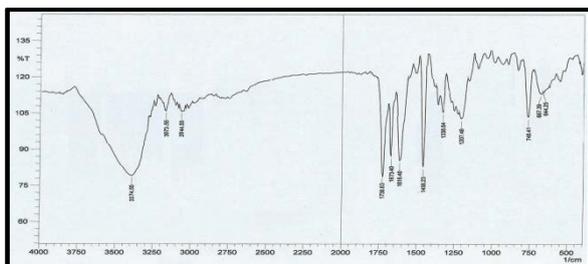
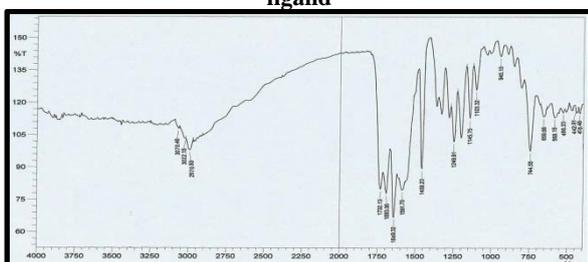
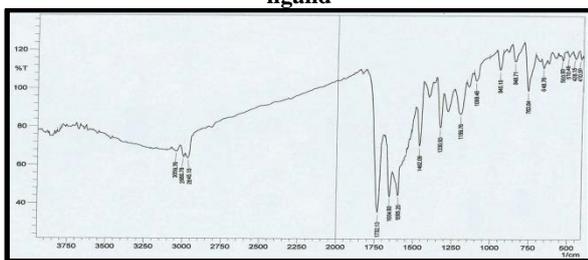
The two bands at  $(1673$  and  $1616) cm^{-1}$  in IR of free ligand are attributed to the stretching vibration of two imine groups  $\nu(C=N)$ , which were moved to minor frequencies in the IR spectrums of all complexes (Figures 4, 5 and 6) for Co(II), Ni(II) and Cu(II) complexes, these shifting indicated the coordination of the ligand with metal ion via nitrogen atoms for two imine groups [22,23]. The presence of a new two bands around  $(455-428) cm^{-1}$  and  $(558-509) cm^{-1}$  in IR spectrums of all complexes due to  $\nu(M-N)$  and  $\nu(M-O)$  substantiates respectively [20], it proved the metal complexes structure [17]. The ligand clearly acts as a tridentate ligand organized to metallic ions through oxygen atom of hydroxyl group of Benzoin and nitrogen atoms of two imine groups.

**Figure 2: Mass band of (HDEAP Schiff base ligand)****Table 3: Mass spectral details of HDEAP Schiff base ligand**

Fragments	Formula weight $g.mol^{-1}$	Relative abundance%
$C_{33}H_{30}N_4O_2$	514	21
$C_{31}H_{27}N_4O$	471	53
$C_{31}H_{26}N_4$	454	77
$C_{24}H_{20}N_4$	364	48
$C_{22}H_{14}N_4$	334	67
$C_{16}H_9N_4$	257	85
$C_{10}H_5N_4$	181	100
$C_7H_5N$	103	47
$C_7H_6$	90	46
$C_3N_3$	78	48
$C_6H_5$	77	52
$C_6H_4$	76	35
$CH_3O$	43	27
$C_2H_6$	30	14

**Table 4: Spectral IR absorption details of the ligand (HDEAP) and its complexes in  $\text{cm}^{-1}$  units.**

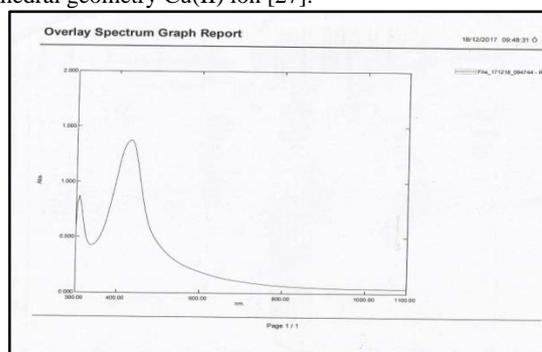
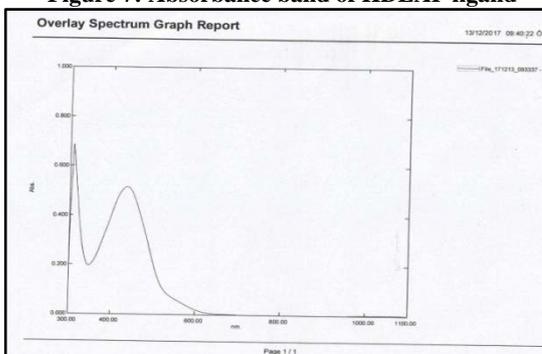
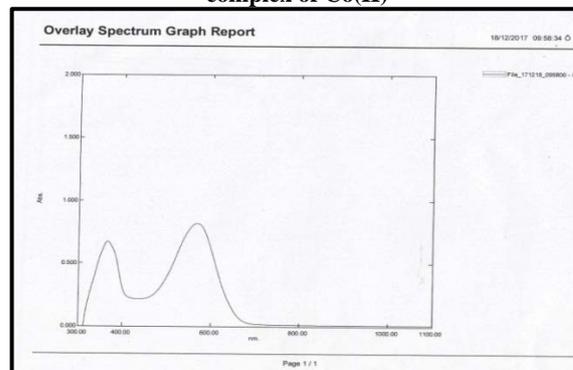
No.	Compound	$\nu(\text{O-H})$	$\nu(\text{C=N})$	$\nu(\text{C=O})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$
1	$\text{C}_{33}\text{H}_{30}\text{N}_4\text{O}_2$	3374	1616 1673	1738	-----	-----
2	$[\text{Co}(\text{C}_{33}\text{H}_{29}\text{N}_4\text{O}_2)_2]$	-----	1588 1650	1729	509	455
3	$[\text{Ni}(\text{C}_{33}\text{H}_{29}\text{N}_4\text{O}_2)_2]$	-----	1595 1654	1732	510	428
4	$[\text{Cu}(\text{C}_{33}\text{H}_{29}\text{N}_4\text{O}_2)_2]$	-----	1591 1649	1732	558	442

**Figure 3: IR band of HDEAP ligand****Figure 4: IR band of ion complex of Co(II) with HDEAP ligand****Figure 5: IR band of ion complex of Ni(II) with HDEAP ligand****Figure 6: IR band of ion complex of Cu(II) with HDEAP ligand**

### Electronic spectra

HDEAP spectrums of ligand (Figure 7) and its Co(II), Ni(II) and Cu(II) complexes in ethanol have been examined and the spectral details have been depicted in Table 5. UV-Vis spectra of Schiff base ligand can be described principally by three absorption peaks at (260 and 308) nm ( $38461$  and  $32467$ )  $\text{cm}^{-1}$  assigned to ( $\pi \rightarrow \pi^*$ ) and at (431) nm ( $23201$ )  $\text{cm}^{-1}$  due to ( $n \rightarrow \pi^*$ ) [24], these electronic transition were moved towards minor frequency in the electronic spectra of all prepared complexes, confirming the coordination of the ligand with metal ions.

The electronic spectrum of Co(II) complex (Figure 8) displayed new absorption peak (440) nm ( $22727$ )  $\text{cm}^{-1}$  which related to the  ${}^4\text{T}_{1g} \rightarrow {}^4\text{T}_{1g}(\text{p})$  ( $\nu_3$ ) transitions suggesting octahedral structure around Co(II) ion [25]. The electronic spectrum of Ni(II) complex (Figure 9) showed new absorption peak, the peak at (567) nm ( $17636$ )  $\text{cm}^{-1}$  that is as a result of  ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{1g}(\text{P})$  ( $\nu_3$ ) transition correspondingly of octahedral structure for Ni(II) complex [26]. The electronic spectrum of Cu(II) complex (Figure 10) showed broadband at (542) nm ( $18450$ )  $\text{cm}^{-1}$  assigned to  ${}^2\text{E}_g \rightarrow {}^2\text{T}_{2g}$  transition that denote Jahn-Teller distorting of octahedral geometry Cu(II) ion [27].

**Figure 7: Absorbance band of HDEAP ligand****Figure 8: Absorbance band of ligand (HDEAP) with ion complex of Co(II)****Figure 9: Absorbance band of ligand (HDEAP) with ion complex of Ni(II)**

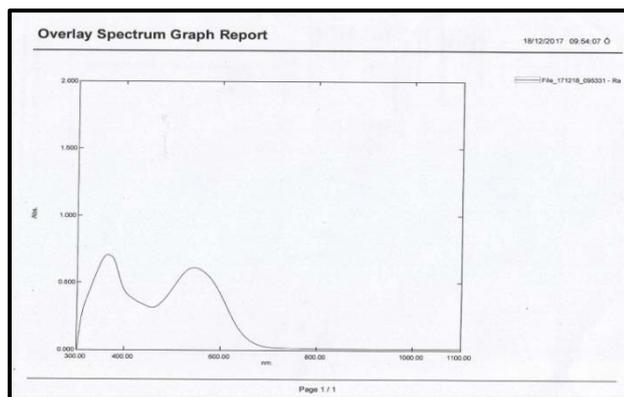


Figure 10: Absorbance band of HDEAP ligand with ion complex of Cu(II)

Table 5: The electronic spectrums of HDEAP ligand and its chelate complexes

Compounds	Band position, nm	Band position, cm <sup>-1</sup>	Transition	Suggested geometry
C <sub>33</sub> H <sub>30</sub> N <sub>4</sub> O <sub>2</sub>	260	38461	$\pi \rightarrow \pi^*$	
	308	32467	$\pi \rightarrow \pi^*$	
	431	23201	$n \rightarrow \pi^*$	
[Co(C <sub>33</sub> H <sub>29</sub> N <sub>4</sub> O <sub>2</sub> ) <sub>2</sub> ]	287	34843	Intra-ligand	Octahedral
	320	31250	Intra-ligand	
	440	22727	$^4T_{1g} \rightarrow ^4T_{1g}(p)$ ( $\nu_3$ )	
[Ni(C <sub>33</sub> H <sub>29</sub> N <sub>4</sub> O <sub>2</sub> ) <sub>2</sub> ]	281	35587	Intra-ligand	Octahedral
	352	28409	Intra-ligand	
	567	17636	$^3A_{2g} \rightarrow ^3T_{1g}(P)$ ( $\nu_3$ )	
[Cu(C <sub>33</sub> H <sub>29</sub> N <sub>4</sub> O <sub>2</sub> ) <sub>2</sub> ]	278	35971	Intra-ligand	Octahedral
	360	27777	Intra-ligand	
	542	18450	$^2E_g \rightarrow ^2T_{2g}$	

#### Magnetic measurements

(i) Co (II) complex displays magnetic moment  $\mu_{eff}$ . (4.19 B.M) based on three uncombined electrons. This refers to an octahedral environment around the Co (II) ion [28].

(ii) Ni (II) complex exhibits magnetic moment  $\mu_{eff}$ . (3.25 B.M) at room temperature based on dualistic uncombined electrons. This refers to an octahedral geometry nearby the Ni (II) ion [29].

(iii) The magnetic moment magnitude of  $\mu_{eff}$ . of the copper (II) complex (1.80 B.M) that refers to an octahedral geometry [30]. The magnetic moments  $\mu_{eff}$ . were explained in Table 6.

#### Conductivity measurements

All soluble complexes showed molar conductivity values at range (16.03 – 11.14) S.cm<sup>2</sup>. mol<sup>-1</sup> in ethanol solvent in 10<sup>-3</sup>M at room temperature. These magnitudes point to low conductivity and nonionic structure of these complexes [30]. The conductivity values were listed in Table 6.

Table 5: Molar Conductivity and Magnetic moment ( $\mu_{eff}$ .) of complexes

Complexes	Molar Conductivity S.cm <sup>2</sup> .mol <sup>-1</sup>	$\mu_{eff}$ . B.M
[Co(C <sub>33</sub> H <sub>29</sub> N <sub>4</sub> O <sub>2</sub> ) <sub>2</sub> ]	13.22	4.19
[Ni(C <sub>33</sub> H <sub>29</sub> N <sub>4</sub> O <sub>2</sub> ) <sub>2</sub> ]	16.03	3.25
[Cu(C <sub>33</sub> H <sub>29</sub> N <sub>4</sub> O <sub>2</sub> ) <sub>2</sub> ]	11.14	1.80

#### Biological activity

The results are listed in Table 7, and its statistical presentation is shown in Figure 11. The ligand and its complexes have shown that *S. aureus* and *S. pyogenes* susceptible to ligand and its complexes while *E. coli* and *K. pneumonia* showed a reverse activity which resistance Co(II) complex but to ligand and Ni(II)

and Cu(II) complexes. Where *K. pneumonia* was resistance to ligand and Co(II) complex but sensitive to rest complexes [31]. Cu(II) complex showed highly activity and sensitivity against all bacteria while Co(II) complex which slightly activity towards all bacteria except the *S. aureus* more activity. The action mechanism of antibacterial drug is discussed under four headings, (1): inhibition of cell wall, (2): cell membrane function inhibition, (3): protein synthesis inhibition and (4): nucleic acid inhibition [32,33]. Finally, the toxicity of Co(II), Ni(II) and Cu(II) complexes were better than the ligand.

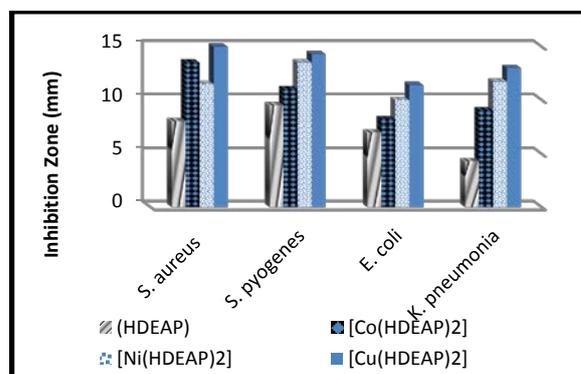


Figure 11: Statistical depiction for antibacterial action of HDEAP ligand and its metallic complexes

Table 7: Inhibition area of the bacterial sensitivity (zone of inhibition in mm) to ligand (HDEAP) and its metallic complexes

Compound	<i>S. aureus</i> G (+)	<i>S. pyogenes</i> G (+)	<i>E. coli</i> G (-)	<i>K. pneumonia</i> G (-)
(HDEAP)	+	++	+	-
[Co(HDEAP) <sub>2</sub> ]	+++	++	+	+
[Ni(HDEAP) <sub>2</sub> ]	++	+++	++	++
[Cu(HDEAP) <sub>2</sub> ]	+++	+++	++	+++

Not:

(0-6)mm =- (Non active)

(6-9)mm =+ ( Slightly active)

(9-12)mm=++ ( Moderately active) (12-17)mm=+++ ( Highly active)

#### Suggested Structure

On the basis of elemental microanalysis, proton-NMR, mass spectral, molar conductivity, magnetic moment, metal content and spectroscopic studies for the ligand (HDEAP) and Co(II), Ni(II) and Cu(II) complexes, the ligand seems to behave as tridentate on complexation with metal ions via the oxygen atom of hydroxyl group of Benzoin and nitrogen atoms of two imine group. we found that the mole ratio M:L was 1:2. According to these results, all complexes have octahedral geometry around metal ion Figure 12.

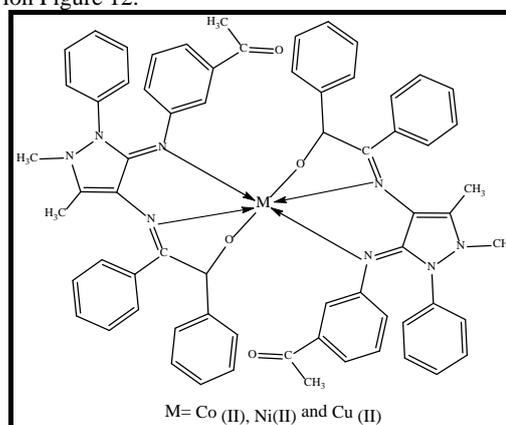


Figure 12: The suggested structure of Co (II), Ni(II) and Cu (II) with Schiff base ligand (HDEAP)

## CONCLUSION

In this research, the preparation and representation of Co(II), Ni(II) and Cu(II) complexes of ligand (HDEAP) were accomplished by means of elemental analyses, metal content, mass spectrum, <sup>1</sup>H-NMR, IR, and UV-Vis spectral measurements. Magnetic susceptibility measured results verify octahedral structure of the complexes. They are have greater biological actions than the respective ligand.

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