

# Synthesis, Characterization and Antibacterial Activity from Mixed Ligand Complexes of 8-Hydroxyquinoline and Tributylphosphine for Some Metal Ions

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*Co<sup>+2</sup>, Ni<sup>+2</sup>, Cu<sup>+2</sup> as well Zn<sup>+2</sup> compounds mixed ligand from 8-hydroxyquinoline (8-HQ) also tributylphosphine (PBu<sub>3</sub>) have been attended at aquatic ethyl alcohol for (1:2:2) (M:8-HQ:PBu<sub>3</sub>). Produced complexes have been identified by utilizing atomic absorption flame, FT-IR as well UV-Vis spectrum manners also magnetic susceptibility as well as conductivity methods. At addendum antibacterial efficiency from the ligands as well complexes oboist three species about bacteria have been as well examined. Ligands and their complexes show good bacterial efficiencies. Of the gained datum the octahedral geometry was proposed into whole prepared complexes*

**Keywords:** Mixed ligand, complexes, 8-hydroxyquinoline, tributylphosphine

8-Hydroxyquinoline has been reacted with many metal ions to form five stable membrane ring, it was chelated with metal ion by the oxygen atom after replaceable the hydrogen and heterocyclic nitrogen atom [1]. It has been found antibacterial activity and it used for determination of metal ions [2,3]. 8-hydroxyquinoline and their derivatives have been reported bioactivities containing anticancer, antibacterial, antidyslipidemic and antioxidative properties [4]. Mixed ligand complexes containing 8-hydroxyquinoline have been played an important area in the enzymes activation [5], these complexes have been appeared microbial activity against pathogenic microorganisms. Therefore, metal complexes which contain 8-hydroxyquinoline as ligand show biological activity [6]. The current paper refers to preparation of some metal complexes with mixed ligand from 8-hydroxyquinoline and tributylphosphine.

## Experimental part

### Instrumentation and materials

UV-Vis spectrum have been registered at a (Shimadzu UV-160 A) Ultra Violet-Visible Spectrophotometer. I.R-spectra were possessed at a (Shimadzu, FTIR-8400 S) Fourier Transform Infrared Spectrophotometer (4000-400) cm<sup>-1</sup>. Atomic Absorption was acquired through employing a (Shimadzu A.A-160A) Atomic Absorption/ Flame Emission Spectrophotometer. Conductors of 10<sup>-3</sup>M from complexes measured at DMSO on 25°C through employing (Philips PW- Digital Conduct meter). Magnetic characteristics were perfect through utilizing Auto Magnetic Susceptibility Balance Sherwood Scientific apparatus in 25°C. moreover, melting points were gained through employing (Melting Point Apparatus).

The following chemicals have been utilized like received of providers; cobalt chloride hexahydrate, nickel chloride hexahydrate, copper chloride dihydrate, zinc chloride (Merck), 8-hydroxyquinoline as well tributylphosphine (B.D.H).

### Study of bacterial activity

The ligands and their metal complexes were tested versus bacteria: *Staphylococcus aureus*, *E-coli*, *Pseudomonas* employing the paper disc mode. Ready compounds at DMSO solution have been utilized at vitro through paper disc mode. Every materials utilized have been sterilized at a hot air oven as well settlement from each to the screened micro organisms have been subcultured also brood around (8 h) before introducing to the agar plates. The discs (7 mm diameter) have been soaked for different exam samples (condensation 1000ug/mL) drained as well thereafter placed at agar plate utilizing sterilized tongs. Plates have been brood at 37°C with (48 h), at the end from nursery period; Zones from inhibition to the various bacteria have been completed [7-9].

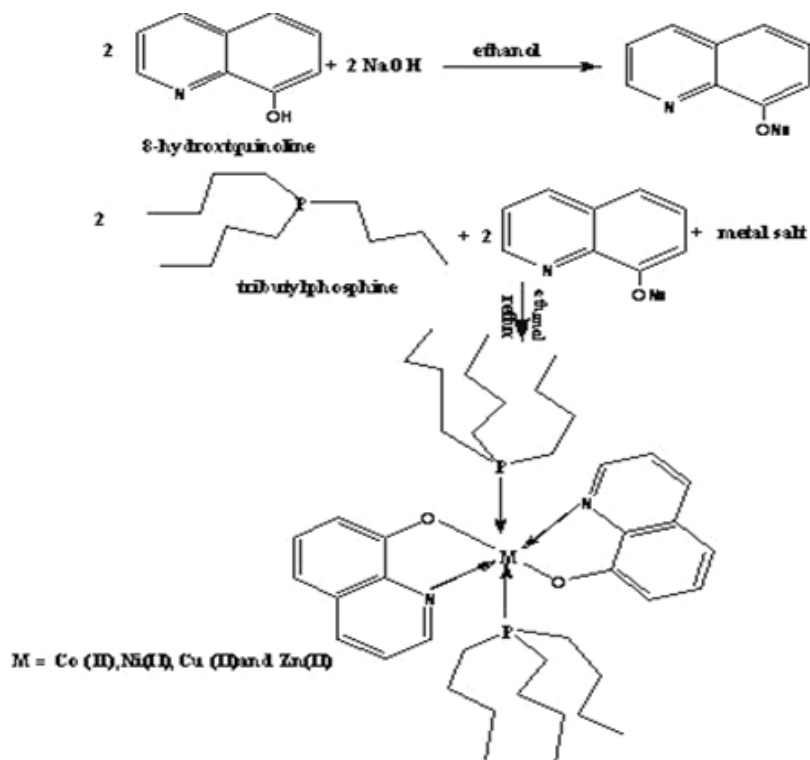
### Preparation of Metal Complexes

An aqueous solution at metal salts including 0.118g, 0.118g, 0.085g also 0.068g (1mmole) from cobalt chloride hexahydrate, nickel chloride hexahydrate, copper chloride dehydrate as well zinc chloride sequences has been added progressively for stirring to ethyl alcohol NaOH solution (0.140g, 2mmol) of 8-hydroxyquinoline. (0.5mL, 2mmole) of tributylphosphine (PBu<sub>3</sub>) was added into mix on the every status through employing stoichiometric amount (1:2:2) Metal:8-HQ:PBu<sub>3</sub> molar ratio. Mix has been refluxed for continuous stirring about an hour. Mix has been cooled in room temperature dark precipitate was created, filtered and recrystallized of ethanol, the following reaction according of scheme 1.

### Result and discussions

8-hydroxyquinoline ligand (8-HQ) has been specified through FT-IR as well UV-Vis. The solid compounds were ready by reaction from ethyl alcohol solution of the ligand for aqueous solution from metal ions and tributylphosphine into a (M:8-HQ:PBu<sub>3</sub>) from (1:2:2). Metal contents from complexes were into good correspondence with calculated values (table1) contains physical characteristics. Molar conductance from compounds like (10<sup>-3</sup>M) on DMSO

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Scheme 1. The expected geometry of the metal(II) complexes  $[M(\text{HQ})_2(\text{PBu}_3)_2]$

**Table 1**  
PHYSICAL CHARACTERISTICS INTO LIGAND AS WELL COMPLEXES

Compounds	Color	M.P <sup>o</sup> C	Yield%	M%	$\Delta_m$ (S.cm <sup>2</sup> .mol <sup>-1</sup> ) in DMSO(10 <sup>-3</sup> M)	$\mu_{\text{eff}}$ (B.M)
Ligand(8-HQ)	white	74	-	-	-	-
$[\text{Co}(\text{8-HQ})_2(\text{PBu}_3)_2]$	brown	212-215	77	8.66 (7.88)	10.45	4.84
$[\text{Ni}(\text{8-HQ})_2(\text{PBu}_3)_2]$	green	220-222	75	8.62 (7.79)	12.64	2.90
$[\text{Cu}(\text{8-HQ})_2(\text{PBu}_3)_2]$	dark blue	232-234	71	9.33 (8.93)	14.77	1.72
$[\text{Zn}(\text{8-HQ})_2(\text{PBu}_3)_2]$	pal yellow	227-279	75	9.46 (8.85)	15.33	Dia

**Table 2**  
THE FUNDAMENTAL FREQUENCIES TO THE LIGANDS AND THEIR COMPOUNDS (cm<sup>-1</sup>)

Compounds	$\nu(\text{OH})$	$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{C})$	$\nu(\text{M}-\text{N})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{P})$
Ligand(8-HQ)	3180 br.	1624 sho.	1577 sh. 1504 sh.	-	-	-
$[\text{Co}(\text{8-HQ})_2(\text{PBu}_3)_2]$	-	1639 s.	1577 sh 1500 sh.	513 w.	474 w.	435 w.
$[\text{Ni}(\text{8-HQ})_2(\text{PBu}_3)_2]$	-	1643 s.	1577 s. 1508 s.	513 w.	474 w.	450 w.
$[\text{Cu}(\text{8-HQ})_2(\text{PBu}_3)_2]$	-	1638 s.	1600 s. 1500 sh.	520 w.	466 w.	439 w.
$[\text{Zn}(\text{8-HQ})_2(\text{PBu}_3)_2]$	-	1639 s.	1577 s. 1500 s.	474 w.	455 w.	447 w.

sho=shoulder, s = strong, sh=sharp, w =weak, br = broad

consist of non-electrolytic style [10], the datum were tabulat at (table 1). The magnetic moments (table 1) from complexes lie at range (1.72-4.84) B.M. That value indicates to a paramagnetic (high spin) whom was reported with most octahedral structure. At case from Zn(II) complex due of the d-orbital filled, subsequently the magnetic moment ( $\mu=0$ ) is diamagnetic [11].

#### IR-Spectra

Vibration bands for ligands and their compounds were registered into KBr at the area 4000-400 cm<sup>-1</sup>. The assignments to the distinctive bands (FT-IR) spectrum to 8-HQ, PBu<sub>3</sub> and compounds are abbreviated at table 2. IR

spectral for ligand (8-HQ) (fig.1) show wide band in 3180 cm<sup>-1</sup>, whom has been qualified into stretching vibration for  $\nu(\text{OH})$  phenol, this band was vanish into the spectra (fig.2) for all generated complexes, whom referred coordination this band for metal ion [12]. Band in 1624 cm<sup>-1</sup> appointed into  $\nu(\text{C}=\text{N})$  stretching frequency [13], on complexation a shifting for alteration into form were noticed of these bands, whereas growing into density were observe, specified may be a outcome from coordination for metal ion. Bands into IR spectrum to the 8-HQ in 1577 and 1504 cm<sup>-1</sup> consequent into stretching vibration for  $\nu(\text{C}=\text{C})$  [14]. New bands watched in (435-513) cm<sup>-1</sup> are temporarily appointed into  $\nu(\text{M}-\text{N})$ ,  $\nu(\text{M}-\text{O})$  as well  $\nu(\text{M}-\text{P})$  (Metal-Ligands) stretching bands [15,16].

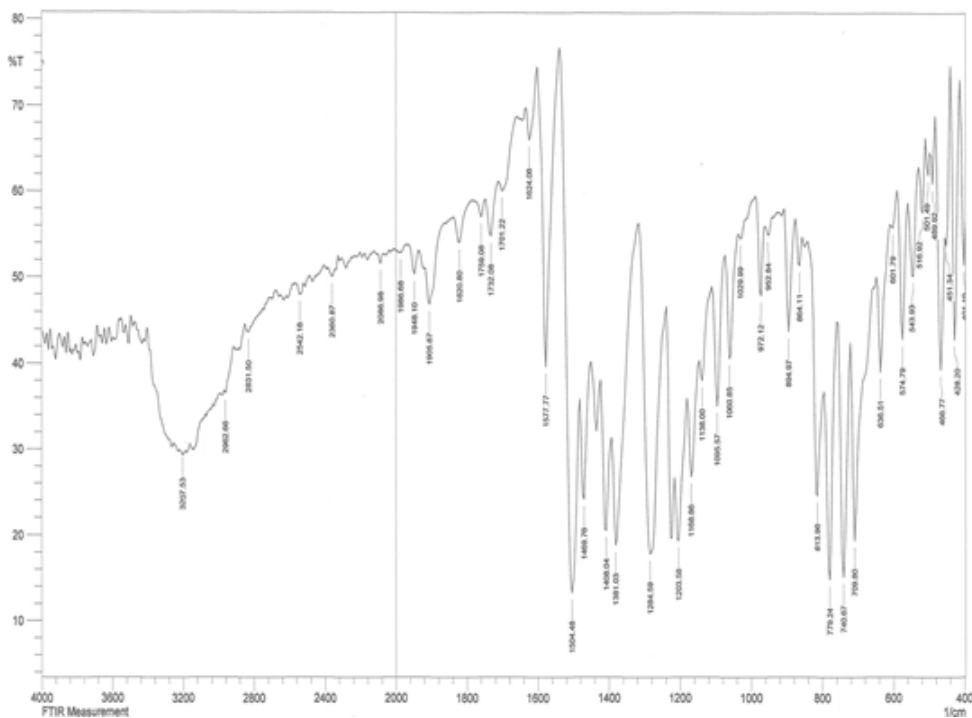


Fig.1. FTIR spectrum of 8-hydroxyquinoline.

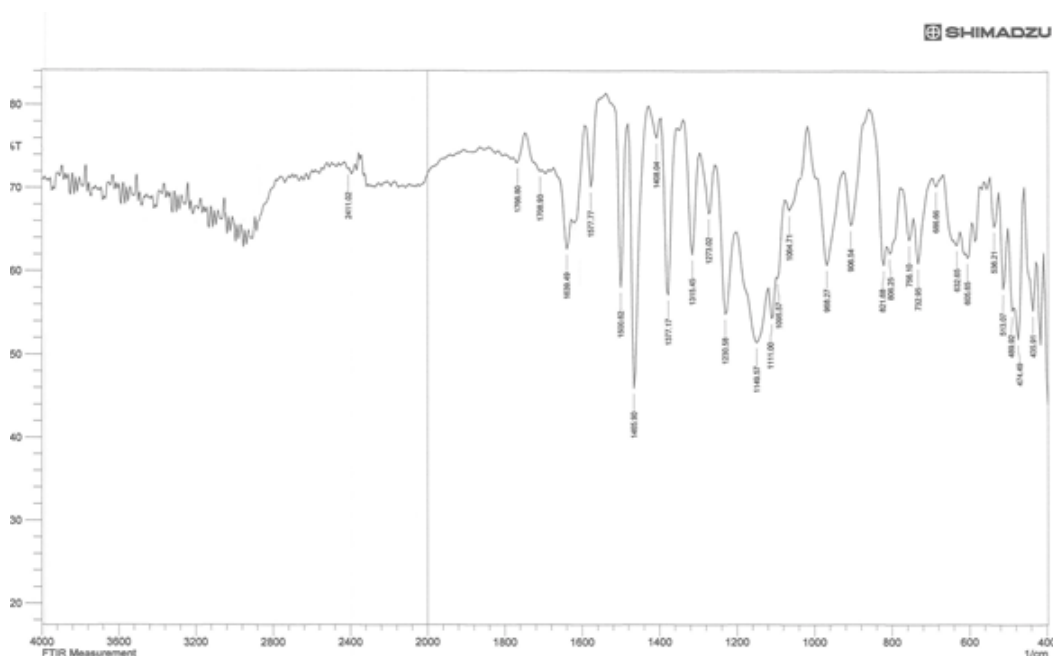


Fig. 2. FTIR spectrum of  $[\text{Co}(\text{8-HQ})_2(\text{PBu}_3)_2]$  complex.

### UV-Vis spectra

UV-Vis spectrum with ligands as well their compounds are included at (table 3). Spectrum of 8-HQ (fig.3) displays two peaks in 332 and 372 nm were appointed into  $(\pi-\pi^*)$  [17]. The  $\text{PBu}_3$  spectrum exhibit absorption peak in 297 nm was qualified into  $(\pi-\pi^*)$  [18]. Spectrum for  $\text{Co}^{+2}$  complex displays peaks, in 308 as well 398 nm were qualified into ligand field and charge transfer. Three peaks in 658, 732 and 920 nm were referred into electronic transition style  ${}^4\text{T}_{1g(\text{F})} \rightarrow {}^4\text{T}_{1g(\text{P})}$ ,  ${}^4\text{T}_{1g(\text{F})} \rightarrow {}^4\text{A}_{2g(\text{F})}$  as well  ${}^4\text{T}_{1g(\text{F})} \rightarrow {}^4\text{T}_{2g(\text{F})}$  respectively, as well the value from the magnetic moment in 4.84 B.M may be possessed as extra confirmation to octahedral geometry [19]. The electronic spectrum for  $\text{Ni}^{+2}$  complex gave two peaks, in 336 and 336 nm consequent into ligand field. Another three peaks in 537, 722 and 830 nm were appointed into electronic transition style  ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{1g(\text{P})}$ ,  ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{1g(\text{F})}$  as well  ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{2g(\text{F})}$  continuity. Magnetic moment for this complex in 2.90 B.M whom was much close to the octahedral

perimeter [20]. The spectrum for  $\text{Cu}^{+2}$  complex (fig.4) appears three peaks, the first also second at 308 as well 398 nm due to ligand field and charge transfer, the third pea seems in 658 nm was qualified into electronic transition style  ${}^2\text{E}_g \rightarrow {}^2\text{T}_{2g}$ . The magnetic moment for this complex was found in 1.72 B.M was much close to the octahedral environment [21]. Electronic spectral from  $\text{Zn}^{+2}$  complex do offer charge transfer, and the magnetic susceptibility seemed the complex has diamagnetic moments, result to d-d transition are not likely subsequently electronic spectrum did not confer any productive datum, on fact this outcome is a good agreement for former work from octahedral geometry [22]. Antibacterial activity of the ligands and their compounds have as well been examined oboist choice chosen species for bacteria, (table 4). Depend on the consequences gained and spectral analysis an octahedral geometry has been proposed of produced compounds.

Compounds	$\lambda_{\text{max}}$ (nm)	ABS	Wave number ( $\text{cm}^{-1}$ )	$\epsilon_{\text{max}}$ ( $\text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$ )	Remarks
Ligand(8-HQ)	332	0.819	30120	819	$(\pi - \pi^*)$
	372	0.861	26881	861	$(\pi - \pi^*)$
[Co(8-HQ) <sub>2</sub> (PBu <sub>3</sub> ) <sub>2</sub> ]	308	0.606	32467	606	L.F
	398	0.645	25125	645	C.T
	658	0.053	15384	53	${}^4\text{T}_{1g(\text{F})} \rightarrow {}^4\text{T}_{1g(\text{P})}$
	732	0.042	13661	42	${}^4\text{T}_{1g(\text{F})} \rightarrow {}^4\text{A}_{2g(\text{F})}$
	920	0.022	10869	22	${}^4\text{T}_{1g(\text{F})} \rightarrow {}^4\text{T}_{2g(\text{F})}$
[Ni(8-HQ) <sub>2</sub> (PBu <sub>3</sub> ) <sub>2</sub> ]	336	0.980	29761	980	L.F
	376	1.028	26595	1028	L.F
	537	0.028	18621	28	${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{1g(\text{P})}$
	722	0.013	13850	13	${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{1g(\text{F})}$
	830	0.011	12048	11	${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{2g(\text{F})}$
[Cu(8-HQ) <sub>2</sub> (PBu <sub>3</sub> ) <sub>2</sub> ]	308	0.606	32467	606	L.F
	398	0.645	25125	645	C.T
	658	0.053	15197	53	${}^2\text{E}_g \rightarrow {}^2\text{T}_{2g}$
[Zn(8-HQ) <sub>2</sub> (PBu <sub>3</sub> ) <sub>2</sub> ]	380	0.822	26315	0.822	C.T
	391	0.937	25575	937	C.T

**Table 3**  
UV-Vis SPECTRUM DATUM  
TO THE LIGANDS AND  
THEIR COMPOUNDS

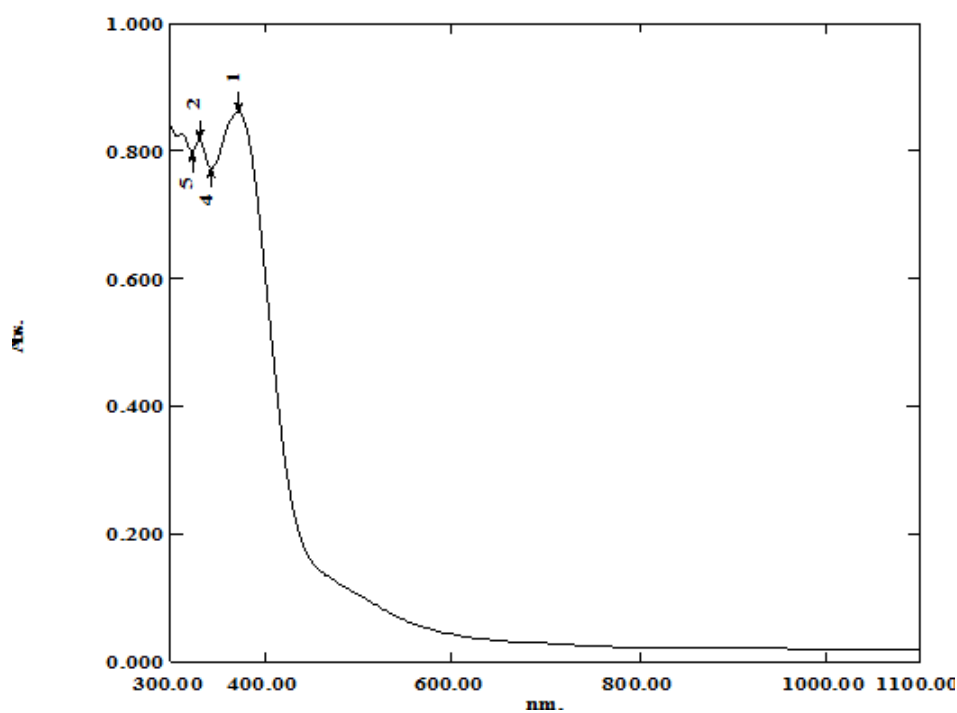


Fig. 3. UV-Vis spectrum of 8-hydroxyquinoline.

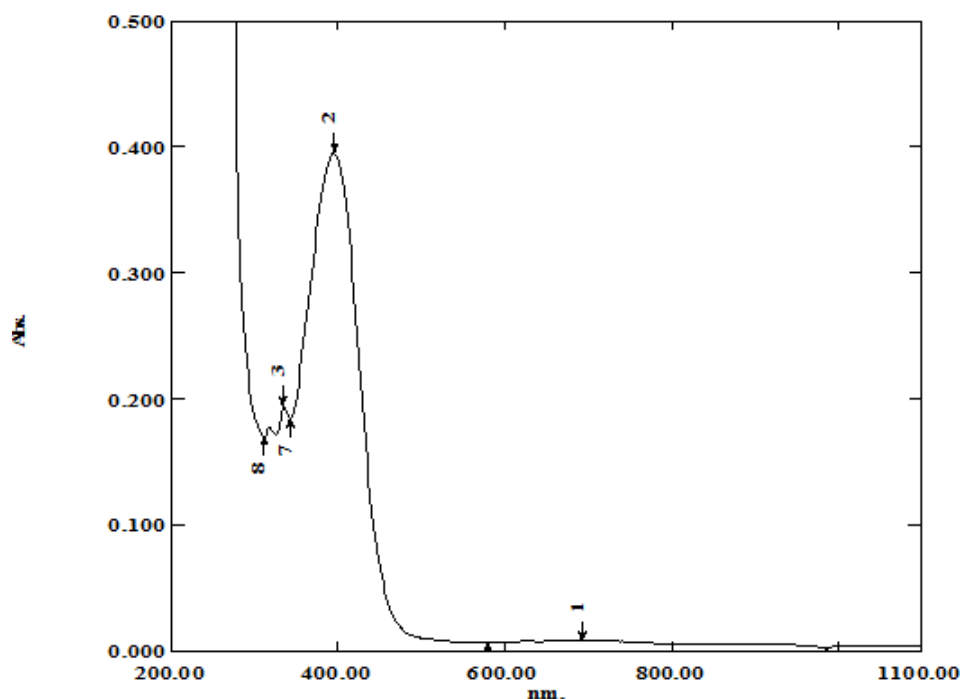


Fig. 4. UV-Vis spectrum of [Cu(8-HQ)<sub>2</sub>(PBu<sub>3</sub>)<sub>2</sub>] complex.

Compounds	<i>Staphylococcus Aurous</i>	<i>Escherichia Col</i>	<i>Pseudomonas</i>
Ligand(8-HQ)			
PBu <sub>3</sub>	22	25	25
[Co(8-HQ) <sub>2</sub> (PBu <sub>3</sub> ) <sub>2</sub> ]	20	22	20
[Ni(8-HQ) <sub>2</sub> (PBu <sub>3</sub> ) <sub>2</sub> ]	30	30	22
[Cu(8-HQ) <sub>2</sub> (PBu <sub>3</sub> ) <sub>2</sub> ]	26	22	20
[Zn(8-HQ) <sub>2</sub> (PBu <sub>3</sub> ) <sub>2</sub> ]	20	25	25
	20	20	20

**Table 4**  
DIAMETERS (mm) TO SUPPRESSION TO  
THE BACTERIA OF COMPOUNDS

## Conclusions

Mixed ligand complexes may be a synthetic defy to tune the characteristics from metal complexes as well were offered to display a wide range of. The probable geometry from synthesis complexes is octahedral as well it is six coordinated ligand metal complexes.

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