

## Synthesis and Bacterial Evaluation of The V(IV), Cr(III), Fe(III) and Co(II) Ions Complexes with Mixed Ligands (2-hydroxybenzaldine)-4- Aminoantipyrine and 8-Hydroxyquinoline

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### Abstract

The primary ligand (2-hydroxybenzaldine)-4-aminoantipyrine (L) was prepared as a chelating ligand, which was treated with V(IV), Cr(III), Fe(III) and Co(II) ions in alcoholic medium in order to prepare series of new metal complexes in the presence of the Co-ligand 8-hydroxyquinoline.

These complexes were prepared by conventional method and characterized by available techniques, FT-IR, UV-Visible, magnetic susceptibility, flame atomic absorption technique as well as elemental analysis and conductivity measurements. From these studies measurements, a square pyramidal structure proposed for V(IV) while octahedral geometry for Cr(III), Fe(III) and Co(II) complexes. The bacterial activity of the prepared complexes has been determined and compared with the ligands.

**Keywords:** Mixed ligands, 4-aminoantipyrine, 8-hydroxyquinoline, antibacterial activity, transition elements.

### Introduction

The coordination compounds display a large diversity offered by the great variety of metal centers and ligand shapes [1]. In modern coordination compounds almost all organic and inorganic compounds can be used as ligands, then the design and synthesis of coordination compounds have attracted much attention from chemists [2]. The cyclic systems containing carbon atoms and at least one other element are called heterocyclic such as furan, pyrrole, oxazole, thiazole, thiazole, pyrazole, pyrrolidine [3] and other the heterocyclic compounds benzothiazoles are widely distributed in nature and essential to life in various ways [4-6].

8-hydroxyquinoline (oxine) behaves as a bidentate through (N and O) as a univalent ligand to form chelates with several metal ions [7]. Compounds containing quinoline moiety are of great interest to synthetic and medicinal chemists due to their unique chemical and biological properties [8].

Oxine is a bicyclic aromatic and is toxic if injected [9]. However its derivatives have long been used for their antibacterial and antifungal activity [10, 11] another applications of quinoline complexes, the quinoline ligands effectively sensitize neodymium ions emission following photo-excitation of polymer host [12] and also used as the electron transport

light emitting layer in such organic light emitting diodes [13].

### Experimental

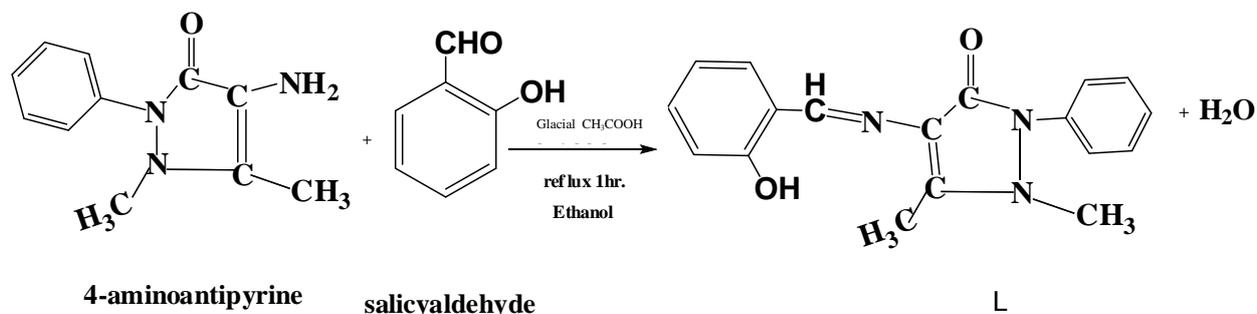
#### 1- Chemicals and Instrumentation

Chemicals which were used are  $\text{VOSO}_4 \cdot \text{H}_2\text{O}$ ,  $\text{Cr}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ,  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 1-10-phenanthroline, Ethanol absolute, salicylaldehyde, 4-aminoantipyrine and glacial acetic acid equipped from different companies FLUKA and BDH with high purity. The FT-IR for ligand and their metal complexes were recorded in the range  $4000\text{-}200\text{cm}^{-1}$  as a (CsI) disc on IR-Prestige-21, Single Beam Path Laser Shimadzu Infrared Spectrophotometer (FT-IR) -8300. UV-Visible spectra were measured by using UV-1650 PC Shimadzu in the range 1100-200nm. The magnetic susceptibility values of the prepared complexes were estimated at room temperature using Balance Magnetic Susceptibility Model MSB- MKT, the conductivity measurements of the prepared complexes were obtained using 0.001M in absolute ethanol solvent, Pw -Digital Meter of Conductivity. Elemental C.H.N analysis was carried out on a EM-101.mth instrument at Al-Bayt University. Atomic absorption measurements of the prepared complexes were obtained using Shimadzu Atomic Absorption 680 Flame Spectrophotometer. The melting

point of ligands and prepared complexes measured by using Stuart Melting Point Apparatus.

## 2- Preparation of 2-hydroxybenzaldine-4-aminoantipyrine (L)

According to the literature [14], the ligand 2-hydroxybenzaldine-4-aminoantipyrine was prepared as in the following scheme (1).



*Scheme (1): The route of preparation 2-hydroxybenzaldine-4-aminoantipyrine(L).*

## 3- Preparation metal complexes

(0.3 g, 1 mmole) of free Schiff base (L) was dissolved in absolute ethanol solvent and then added (L<sup>-</sup>) (0.14 g, 1 mmole) which dissolved in same a solvent. To the mixture added (1 mmole) of metal salts [(0.1 g VOSO<sub>4</sub>.H<sub>2</sub>O), (0.28g Cr (NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O), (0.34g Fe(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O) and (0.29 g Co(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O)] were mixed in 1:1:1 (M: L : L<sup>-</sup>). The resulting solution was refluxed for about (2-4) hrs. The colored precipitated was filtered and the recrystallized with ethanol and drying in oven 50 °C, some physical properties and analytical data are tabulated in Table (1).

properties of the complexes are summarized in Table (1). The molar conductance values of the complexes indicating that the complexes are ionic for chromium and iron while cobalt and vanadium complexes are non ionic.

## 4- Bactericidal activity

Bactericidal activity of the ligands and their complexes were evaluated against representative gram-positive and gram-negative bacteria by agar-plate method [15]. All the prepared compounds as drugs were prepared freshly by dissolving them in ethanol to obtain a final concentration of 1000µg/ml. All bacteria were cultivated in nutrient agar, the results are found in table (4).

## Result and Discussion

### A- Chemistry

The elemental analysis and atomic absorption shows 1:1: 1 (M: L: L<sup>-</sup>) stoichiometry for the prepared complexes. The analytical data together with some physical

**Table (1)**  
**Show some physical properties and analytical data for prepared compounds.**

Comp.	M.W g.mol <sup>-1</sup>	colour	M.P.C <sup>0</sup>	Yield %	Elemental analysis Found( Calc.)			Metal % Found (Calc.)
					C%	H %	N %	
L	307	yellow	239-241	72	70.65 (70.34)	3.65 (3.90)	12.92 (13.86)	-
L <sup>-</sup>	145	colorless	81-83	-	-	-	-	-
VL L <sup>-</sup>	526	light olive	170 d.	81	60.82 (61.29)	4.44 (5.32)	9.72 (10.64)	(8.88) (9.67)
CrL L <sup>-</sup>	609	green	217 d.	78	54.13 (53.20)	4.53 (4.59)	12.47 (11.49)	8.23 (8.52)
FeL L <sup>-</sup>	631	rednish brown	220 d.	61	51.68 (51.34)	4.51 (4.43)	10.84 (10.09)	8.03 (8.84)
CoL L <sup>-</sup>	581	light brown	129-131	72	54.81 (55.76)	5.70 (5.16)	9.92 (9.63)	9.57 (10.13)

### FT-IR Study:

FT-IR spectra for the ligand (L) [(2-hydroxybenzaldehyde)-4-aminoantipyrine] and Co- ligand L<sup>-</sup>(8-hydroxyquinoline) show bands at (3240 cm<sup>-1</sup>) and (3182 cm<sup>-1</sup>) which attributed to stretching frequency of  $\nu(\text{OH})$  phenolic group for (L) and (L<sup>-</sup>) respectively [16].

The medium bands which appeared at 1593 cm<sup>-1</sup> and 1577 cm<sup>-1</sup> both in ligands were assignable to stretching frequency of azomethane group (C=N) as in the literature [17].

In the free Schiff base ligand (L) present band at 1654 cm<sup>-1</sup> may be refer to the carbonyl group (C=O)[18] and slightly change about 5 cm<sup>-1</sup> when formation Vanadium, Chromium, Iron and Cobalt complexes, this result indicated their is nonparticipation of carbonyl group with all metal ions.

The deprotonation of phenol proton from free Schiff base and Co- ligand indicated the coordination of oxygen atom of phenolic group with the central metal atom [18]. The bands which appeared at 1269 cm<sup>-1</sup> and 1224 cm<sup>-1</sup> belonged to  $\nu(\text{C-O})$  in the free Schiff base ligand and Co-ligand respectively, these bands were shifted to lower frequency around (1210-1220) cm<sup>-1</sup> for all complexes, this shifted confirming the coordination of the oxygen atom of phenol group to the metal ions [20].

The bands azomethine group in both ligands were shifted to lower frequency when complexes formation. In the range (1500-

1558) cm<sup>-1</sup>, this can be indicated the coordination of the nitrogen of imine moiety to the all metal ions [21]. New weak bands which appeared in the complexes in the range (526-597) cm<sup>-1</sup> and (425-450) cm<sup>-1</sup> due to the stretching frequency of (M-N) and (M-O) bands respectively. The bands due to the ionic nitrate group which appeared almost in the range (1384-1022) cm<sup>-1</sup> with some higher or lower frequencies comparable to this range in some complexes, A broad bands observed around (3400-3440) cm<sup>-1</sup> in the spectra of metal complexes, assigned to the  $\nu(\text{OH})$  which refer to presence of water molecules. The most diagnostic bands are summarized in the Table (2).

**Table (2)**  
**The most characteristic bands of the compounds (cm<sup>-1</sup>).**

Compound	$\nu(\text{OH})$ Water	$\nu(\text{OH})$ Phenol	$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{O})$	$\nu(\text{C}-\text{O})$	$\nu(\text{M}-\text{N})$	$\nu(\text{M}-\text{O})$	Other bands
<b>L</b>	-	3240	1593	1654	1269	-	-	-
<b>E</b>	-	3182	1577	-	1224	-	-	-
<b>V L E</b>	3450	3062	1558	1650	1210	597	450	V=O =970 H <sub>2</sub> O=3430
<b>Cr L E</b>	3361	3062	1500	1655	1210	576	425	H <sub>2</sub> O=3400-3420 $\delta\text{OH} = 875$ NO <sub>3</sub> =1384, 1031, 1467
<b>Fe L E</b>	3390	3057	1551	1651	1220	526	455	H <sub>2</sub> O= 3400-3440 $\delta\text{OH} = 877$ NO <sub>3</sub> =1380, 1022, 1458
<b>Co L E</b>	3406	3051	1551	1654	1220	540	432	H <sub>2</sub> O =3440-3410 $\delta\text{OH} =865$

### Electronic Spectrum:-

Most of transition complexes are colored and their colors are different, then this is an important indication of coordination between ligands and metal ions, therefore the colored complexes showed different characteristic absorption bands in their positions and intensity [22].

The electronic spectrum of ligand (L) shows a absorption band in the ultraviolet region, the main band at 345 nm (28589 cm<sup>-1</sup>) which assignable to the (n→π\*) transition of imine group (C=N) [23]. The electronic spectrum of ligand (L)-8- hydroxyguinoline exhibits a very strong absorption band in ultraviolet region at 319 nm (31347 cm<sup>-1</sup>) due to the (π→π\*) transition for the intera ligand aromatic system (C=C) [23].

### Vanadium (IV) complex:-

The magnetic moment for the olive color VO(II) complex found to be 2.18 BM. at room temperature this value is higher than spin value of the vanadium ion only this result indicates a higher orbital contribution [24]. The diffuse reflectance spectrum of VO(II) complex displays three bands at 11037, 17241 and 21231cm<sup>-1</sup> which may be assigned to the <sup>2</sup>B<sub>2</sub>→<sup>2</sup>E (dxy→dxz-dyz), <sup>2</sup>B<sub>2</sub>→<sup>2</sup>B<sub>1</sub> (dxy→dx<sup>2</sup>-y<sup>2</sup>) and <sup>2</sup>B<sub>2</sub>→<sup>2</sup>A<sub>1</sub> (dxy→dz<sup>2</sup>) transitions respectively, and other band at 30487 cm<sup>-1</sup> which assignable to charge transfer C.T, when measured the conductivity for the complex it is found non ionic, These transitions and the magnetic moment value came in accordance with the published data for square pyramidal

geometry around VO(II)ion with a mononuclear structure [24].

### Chromium (III) complex:-

The green color for the chromium (III) complex spectrum exhibits three bands at 1818, 23923 and 38461 cm<sup>-1</sup> due to <sup>4</sup>A<sub>2g</sub>→<sup>4</sup>T<sub>2g</sub>, <sup>4</sup>A<sub>2g</sub>→<sup>4</sup>T<sub>1g</sub> and <sup>4</sup>A<sub>2g</sub>→<sup>4</sup>T<sub>1g(p)</sub> transitions respectively, with charge transfer which are assigned to spin allowed transition specific for Cr(III) in distorted octahedral symmetry[25]. All ligand filed parameters was calculated B', Dq, 10 Dq and β using Orgel diagram for d<sup>3</sup> system as illustrated in Table (3), the magnetic moment value is 3.3 BM., and this complex is ionic when conductivity measured. Thus the octahedral structure has been suggested for this complex

### Ferric complex:-

The spectrum of reddish brown color of iron (III) complex shows (10309, 20618 and 27322) cm<sup>-1</sup> which corresponding to <sup>6</sup>A<sub>1g</sub>→<sup>4</sup>T<sub>1g</sub>, <sup>6</sup>A<sub>1g</sub>→<sup>4</sup>T<sub>2g(G)</sub> and <sup>6</sup>A<sub>1g</sub>→<sup>4</sup>A<sub>1g</sub>+<sup>4</sup>E<sub>g(G)</sub> transition respectively and charge transfer at 31646 cm<sup>-1</sup>[26]. The ligand filed parameter was estimated as in Table (3), The magnetic moment μ<sub>eff</sub> value for Fe(III) complex is 5.12 BM indicating high spin for this complex. The complex is ionic, Electronic transition together with magnetic moment value suggested high spin octahedral geometry for ion complex.

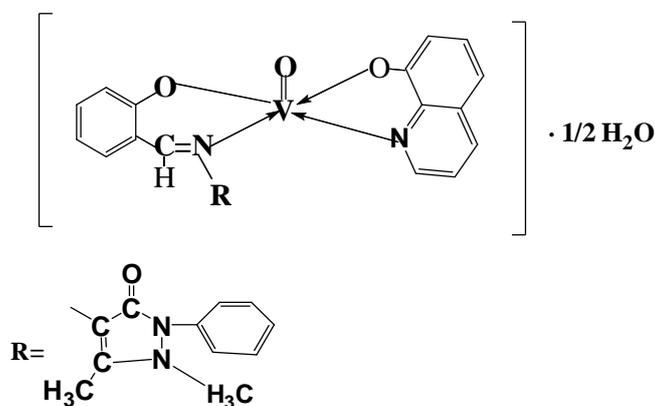
**Cobalte (II) complex:-**

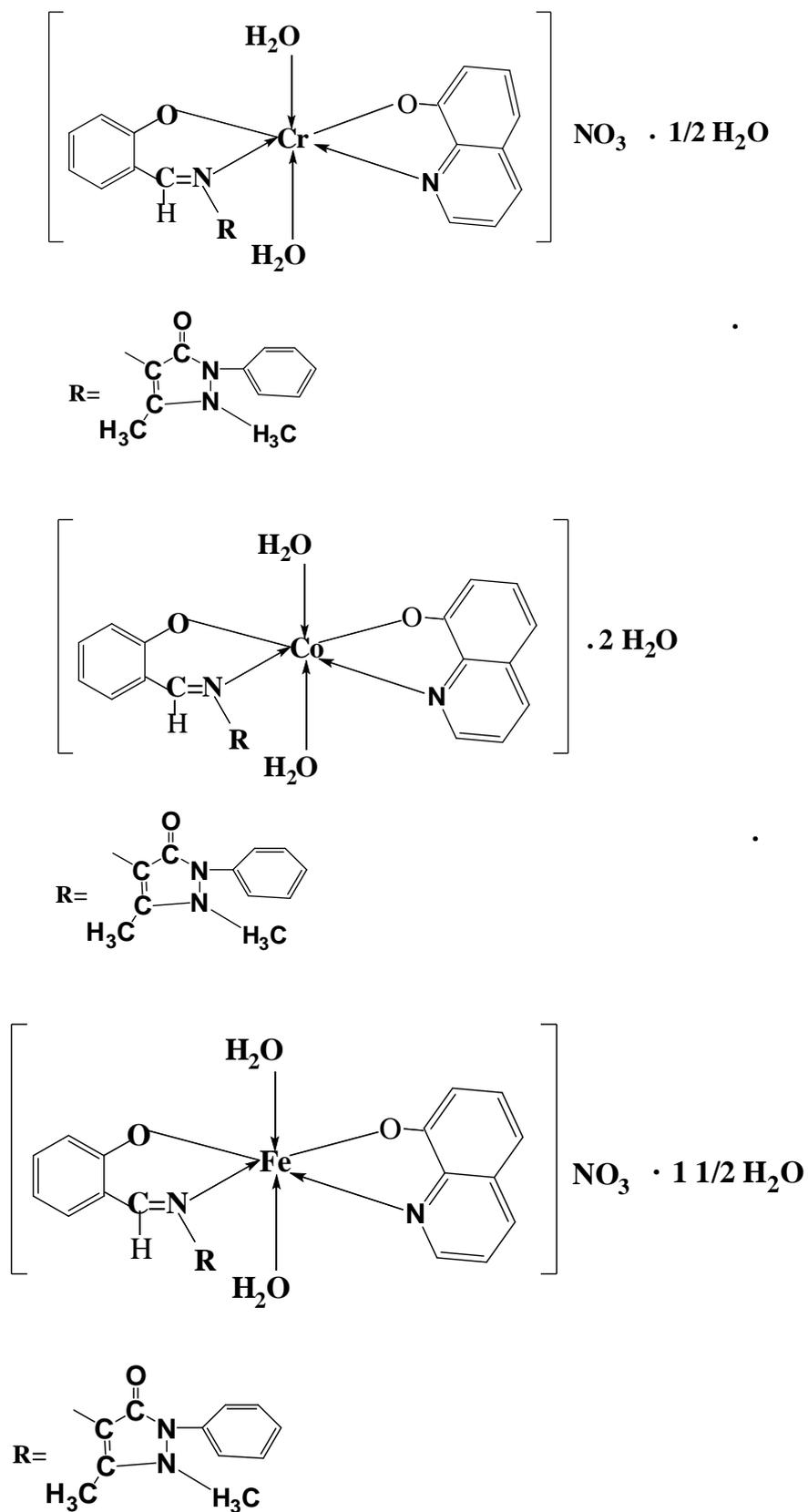
In the spectrum of brown Co(II) complex, exhibit two bands at 1088 and 18315  $\text{cm}^{-1}$  are associated to the transition  ${}^4T_{1g} \rightarrow {}^4T_{2g}$  and  ${}^4T_{1g} \rightarrow {}^4T_{1g(p)}$  [27] respectively, and charge transfer at 30769  $\text{cm}^{-1}$  these transitions are specified to the Co(II) ion in the field of octahedral symmetry, this complex is non ionic and the magnetic moment of 4.77 BM corresponds to this high spin geometry. The  $V_2$ , and  $Dq$ ,  $B'$ ,  $10 Dq$  and  $\beta$  was calculated using T.S.D for  $d^7$  system and this complex is non ionic nature. From these results, the distorted Oh structure was proposed for this complex.

**Table (3)**  
**Electronic spectral data of L, L' and metal complexes and all ligand filed parameters, magnetic moment (BM) and conductance ( $\mu \text{ scm}^{-1}$ ).**

Comp.	Absorption $\text{cm}^{-1}$	Assignment	Dq	10 Dq	B	$B^0$	$B'$	15B'	$\mu_{\text{eff}}$	Molar Conductivity	Suggested Structure
L	28589	$n \rightarrow \pi^*$									
L'	31347	$\pi \rightarrow \pi^*$									
VL L'	11037	$B_2 \rightarrow {}^2E^2$									
	17241	${}^2B_2 \rightarrow {}^2B_1$									
	21231	${}^2B_2 \rightarrow {}^2A_1$							2.18	27	Sq.Py
	30487	C.T									
Cr L L'	18181	${}^4A_2g \rightarrow {}^4T_2g$	1728	17280	0.8	918	735	7841	3.3	40	distorted Oh
	23923	${}^4A_2g \rightarrow {}^4T_1g$									
	38461	${}^4A_2g \rightarrow {}^4T_{1g(p)}$									
Fe L L'	10309	${}^6A_1g \rightarrow {}^4T_{1g(G)}$	1272	12720	0.62	1300	848	17013	5.12	41	distorted Oh
	20618	${}^6A_1g \rightarrow {}^4T_{2g(G)}$									
	27322	${}^6A_1g \rightarrow {}^4A_1g + {}^4Eg(G)$									
	31646	C.T									
Co L L'	10881	${}^4T_{1g} \rightarrow {}^4T_{2g}$	1053	10530	0.8	1030	780	11700	4.77	29	distorted Oh
	26028 Cal.	${}^4T_{1g} \rightarrow {}^4A_2g$									
	18315	${}^4T_{1g} \rightarrow {}^4T_{2g(p)}$									
	30769	C.T									

According to these results in addition to spectroscopic and analytical data, the following structures can be suggested in Fig. (1).





*Fig.(1): Suggested structure of prepared complexes.*

## B - Bactericidal activity

Preliminary screening experiments which are intended to detect the *in vitro* activity of the ligands and their metal complexes show the complexation of the ligands with some studies metals exhibit highly significant activity against gram-positive and other gram – negative, This result can be attributed to be expected synergic effect between the metal and ligand which depend on, the chelate effect of the ligands, the nature of donor atoms, nature of the metal and the oxidation state and the type of the counter ions that the neutralize the complex and the geometrical structure of complexes [28].

**Table (4)**  
*Antibacterial activities of the ligands and their complexes showing inhibition zone in diameters (mm).*

Compounds	Bacterial types		
	<i>E.schriachia coli.</i>	<i>Staphlococcus aureus</i>	<i>Pseudomonasaerogenuosa</i>
L	+	++	+++
L <sup>-</sup>	+	-	+++
VL L <sup>-</sup>	+	++	+
CrL L <sup>-</sup>	+	+++	+
FeL L <sup>-</sup>	+	+++	+
CoL L <sup>-</sup>	+	+	++

(-) : No significant Zone

(+) : Inhibition Zone (4-10)

(+ +) : Inhibition Zone (10-18)

(+ + +) Inhibition Zone (18-25)

## Conclusion

The ligands 2-hydroxybenzaldine-4-aminoantipyrine and 8- hydroxyquinoline behaves as didentate ligand through (N, O) atoms. From the spectroscopy and analytical studies all complexes have Oh geometry except V (IV) complex have square pyramidal. From conductivity measurements showed the V(IV) and Co (II) complexes have nonionic nature while the Cr(III) and Fe(III) complexes have ionic nature and behaves as paramagnetism nature. The antibacterial activity of the complexes studies exhibit highly significant activity against gram. positive and other gram negative, this result can be attributed to be synergic effects.

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### الخلاصة

تم تحضير معقدات جديده بالطريقه التقليديه لليكاند (2- هايدروكسي بنزيلدين) -4- امينوانتيايرين مع بعض ايونات عناصر الفناديوم الرباعي، الكروم الثلاثي، الحديد الثلاثي والكوبلت الثنائي بوجود الليكاند المشارك 8- هايدروكسي كوينولين في وسط كحولي. شخصت المركبات المحضره بواسطة تقنيات الاشعه تحت الحمراء والاشعه فوق البنفسجيه - المرئيه والحساسيه المغناطيسيه والامتصاص الذري اللهبى وكذلك التحليل الدقيق للعناصر والتوصيليه الكهربائيه. من خلال هذه الدراسات تم اقتراح الشكل الهندسي لمعقد الفناديوم هرم مربع القاعده والشكل ثماني السطوح لكل من المعقدات الكروم، الحديد والكوبلت. كما تم تقييم البكتريا للمعقدات المحضره ومقارنتها مع الليكاندات المستخدمه.